NASA Conference Publication 3259

National Educators' Workshop: Update 93

Standard Experiments in Engineering Materials Science and Technology

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Compiled by
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Proceedings of a workshop sponsored jointly by
the National Aeronautics and Space
Administration, Washington, D.C., the Norfolk
State University, Norfolk, Virginia, the United
States Department of Energy, Oak Ridge,
Tennessee, and the National Institute of
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and held in Hampton, Virginia
November 3–5, 1993

National Aeronautics and Space Administration
Langley Research Center • Hampton, Virginia 23681-0001

April 1994

PREFACE

NEW:Update 93 was held November 3 - 5, 1993, at the National Aeronautics and Space Administration, Langley Research Center, Hampton, Virginia. As with previous workshops, the theme was strengthening materials education. Participants witnessed demonstrations of experiments, discussed issues of materials science and engineering (MS&E) with people from education, industry, government, and technical societies, heard about new MS&E developments, and attended mini workshops in state-of-the-art NASA LaRC materials laboratories. Concurrent sessions were held in order to accommodate all of the demonstrations of experiments. Faculty in attendance represented high schools, community colleges, smaller colleges, and major universities. Some were the only materials educators on their campus, while others were from well established MS&E programs.

NEW:Update 93 marked the eighth annual National Educators' Workshop:Update. One hundred twenty participants, including first time attendees and those who attended previous New:Updates, aided in evaluating nearly thirty-five experiments that were presented before the group. Additional updating information relating to materials science, engineering and technology was also presented. Mini plenary sessions involved ASTM methods for composite characterization, recycling of automobiles, developments in carbon materials, new developments in aluminum for aircraft and automobiles, and adhesives and adhesion.

The experiments in this publication can serve as a valuable guide to faculty who are interested in useful activities for their students. The material was the result of years of research aimed at better methods of teaching materials science, engineering and technology. The experiments were developed by faculty, scientists, and engineers throughout the United States. There is a blend of experiments on new materials and traditional materials. Uses of computers in MS&E, experimental design, and a variety of low cost experiments were among the demonstrations presented. Transparency masters of technical presentations are also included.

An extensive peer review process of experiments was followed. After submission of abstracts, selected authors were notified of their acceptance and given the format for submission of experiments. Experiments were reviewed by a panel of specialists through the cooperation of the Materials Education Council. Authors received comments from the panel prior to NEW:Update 93, allowing them to make necessary adjustments prior to demonstrating their experiments.

Comments from workshop participants provided additional feedback which authors used to make final revisions for this publication.

Videotapes were made of the workshop by NASA Langley Research Center and Alfred E. McKenney. As with previous NEW:Updates, critiques were made of the workshop to provide continuing improvement of this activity.

The American Society of Engineering Education presented their Centennial Awards to James E. Gardner, Samuel Massenberg, and James A. Jacobs for their contributions to materials engineering education. Our host Co-Director, James E. Gardner, led an effort to produce a long overdue recognition of the invaluable contribution to the NEW:Update activities by Diana LaClaire, Assistant Director. Ms. LaClaire has provided invaluable support to the project. The Materials Education Council of the United States was represented again and will publish selected experiments in the **Journal of Materials Education**.

NEW:Update 93, and the **'86, '87, '88, '89, '90, '91** and **'92** workshops are, to our knowledge, the only national workshops or gatherings for materials educators that have a focus on the full range of issues on strategies for better teaching about the full complement of materials. Recognizing the problem of motivating young people to pursue careers in MSE, we have included exemplary pre-university activities such as Adventures in Science, ASM International Education Foundation's Career Outreach Program, Engineers for Education, and several programs run through high schools.

Through the workshops we have learned about ORNL Out Reach Programs, an NSF funded project to develop modules for pre-college materials education, and the Materials Science Technology (MST) Project at Richland High School (Richland, Washington) that has received support from Battelle PNL (Pacific Northwest Laboratory). An experiment was presented from the Richland High School's MST program. **NEW:Update 93**, with its diversity of faculty, industry, and government MSE participants, served as a forum for both formal and informal issues facing MSE education that ranged from the challenges of keeping faculty and students abreast of new technology to ideas to insure that materials scientists, engineers, and technicians maintain the proper respect for the environment in the pursuit of their objectives.
NEW:Update 93 resulted from considerable cooperative efforts by individuals in government, education, and industry. The workshop's goal is to maintain the network of participants and to continue to collect these ideas and resources to bring them together in a comprehensive manual of standard experiments in materials science, engineering and technology.

We hope that the experiments presented in this publication will assist you in teaching about materials science, engineering and technology. We would like to have your comments on their value and means of improving them. Please send comments to James A. Jacobs, School of Technology, Norfolk State University, Norfolk, Virginia 23504.

We express our appreciation to all those who helped to keep this series of workshops viable.

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Technical notebooks, videotaping, and announcements of the workshop were provided by
NASA LANGLEY RESEARCH CENTER

NASA Mini Workshops

Half-day workshops for small groups that provided participants in-depth view of materials science and engineering research at NASA-LaRC. Provides excellent technology transfer of NASA's research.

◆ **High Performance Composites - Resin Synthesis, Fabrication, Evaluation - Building 1293A**
Norman J. Johnston, Manager, Composites Technology, Materials Division
Steven P. Wilkinson, Polymeric Materials Branch

Chemistry Lab, prepregging lab, composite fabrication, nondestructive evaluation, and composite evaluation.

◆ **Non Destructive Evaluation Applications to Integrity - Building 1230B**
Joseph S. Heyman, Head, Non Destructive Evaluation, Science Branch IRD

Thermal NDE of bonds and corrosion in aircraft, ultrasonic scanning of defects in composites, micro-NDE of advanced composites, radiography-realtime and X-ray cat scans for integrity and design verification.

◆ **Advanced Fabrication Technology - Building 1232A**
John D. Buckley, Technical Assistant, Fabrication Division

Fabricating superconductors, resin transfer molding, no draft slip casting of high temperature ceramic structures, fabricating active structures of intelligent materials, and quality assurance techniques.

◆ **Materials Characterization for Durability and Damage Tolerance - Building 1205**
Charles E. Harris, Head - Mechanics of Materials Branch, Materials Division

Corrosion fatigue of aluminum and aluminum lithium, thermo mechanical fatigue of metal matrix composite for NASA, impact damage in composites, durability of composites for an SST.

◆ **Advanced Metals - Buildings 1148 and 1205**
W. Barry Lisagor, Head - Metallic Materials Branch, Materials Division

Advanced light metals and metal matrix composites, light weight, high strength structural alloys and composites to achieve thermal/mechanical performance, synthesis of new or improved ingot and powder metallurgy alloys, joining and forming processes for light-alloy material systems, near net shape processing, and advanced metallographic and test methods.

◆ **Superconductors - Building 1222**
Robert A. Hawsey and Richard Kerchner - Super Conducting
Technology Program, Oak Ridge National Laboratory
Robert W. Dull, Largo High School

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Moving to Workshops

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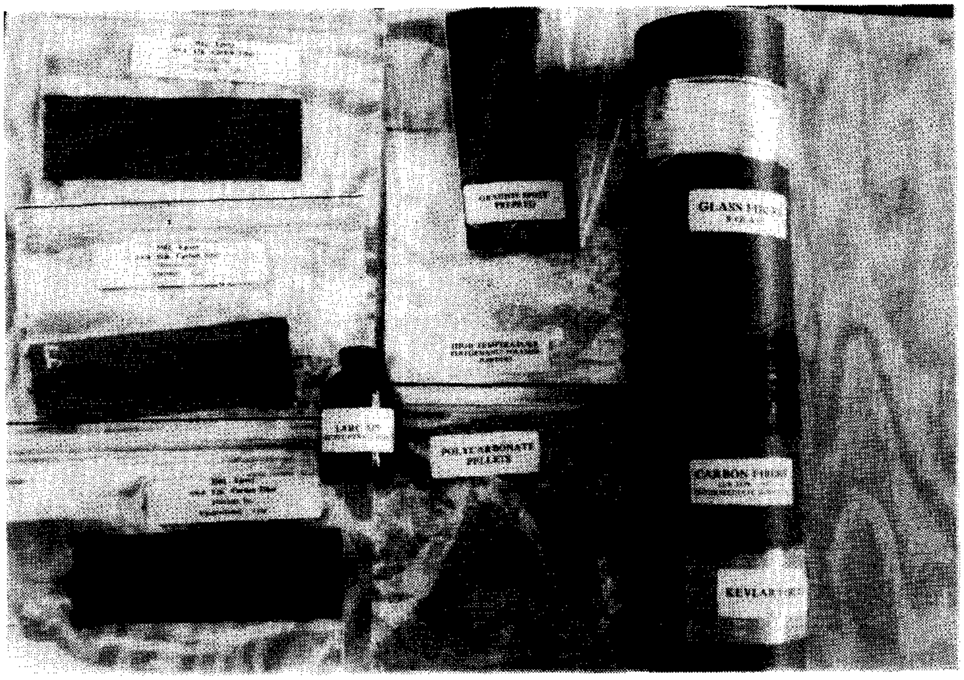

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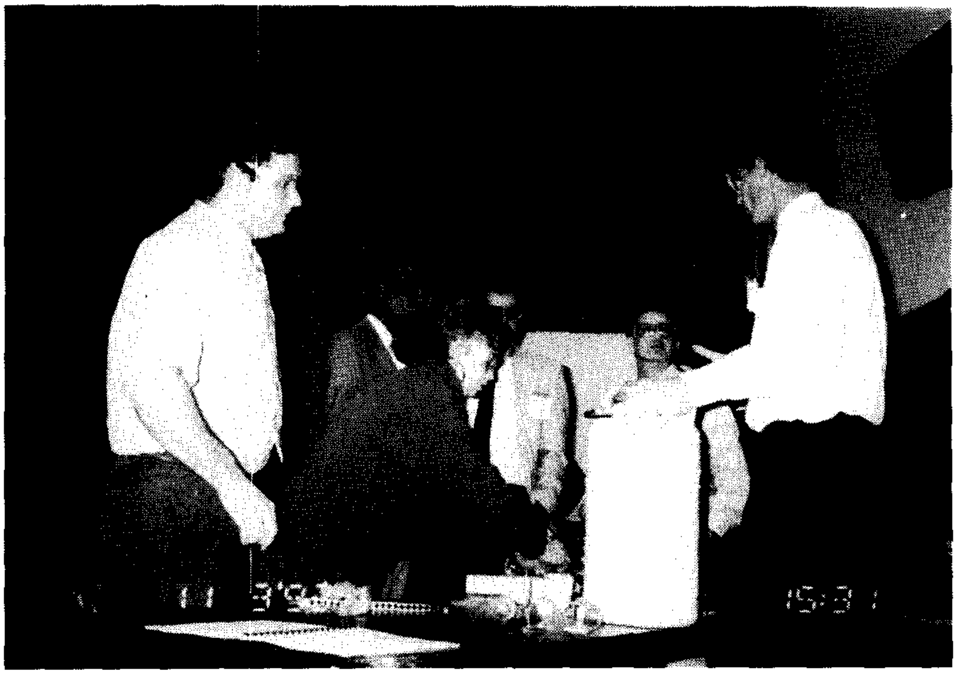

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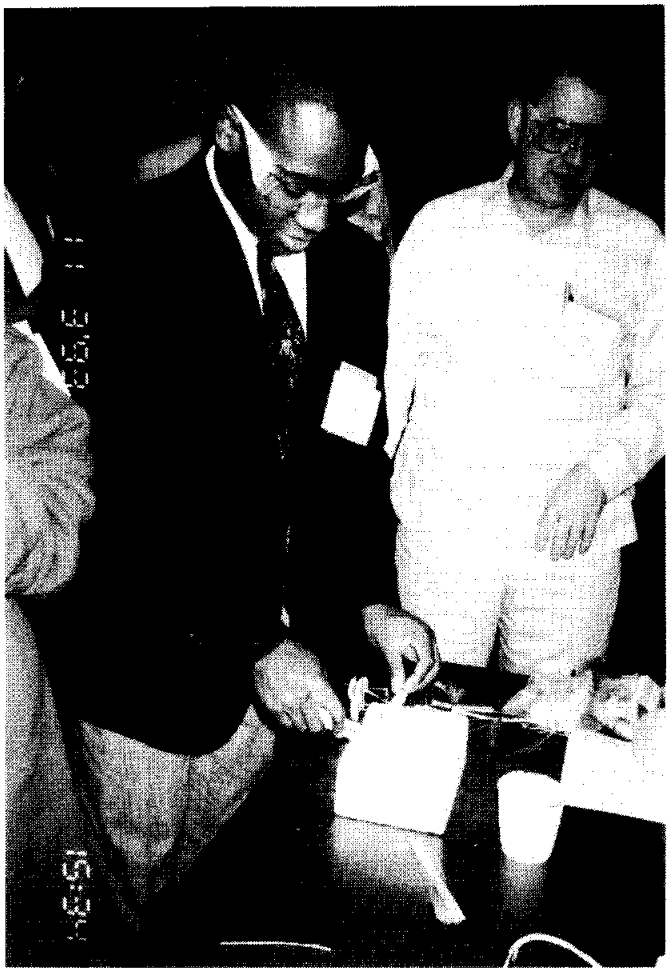

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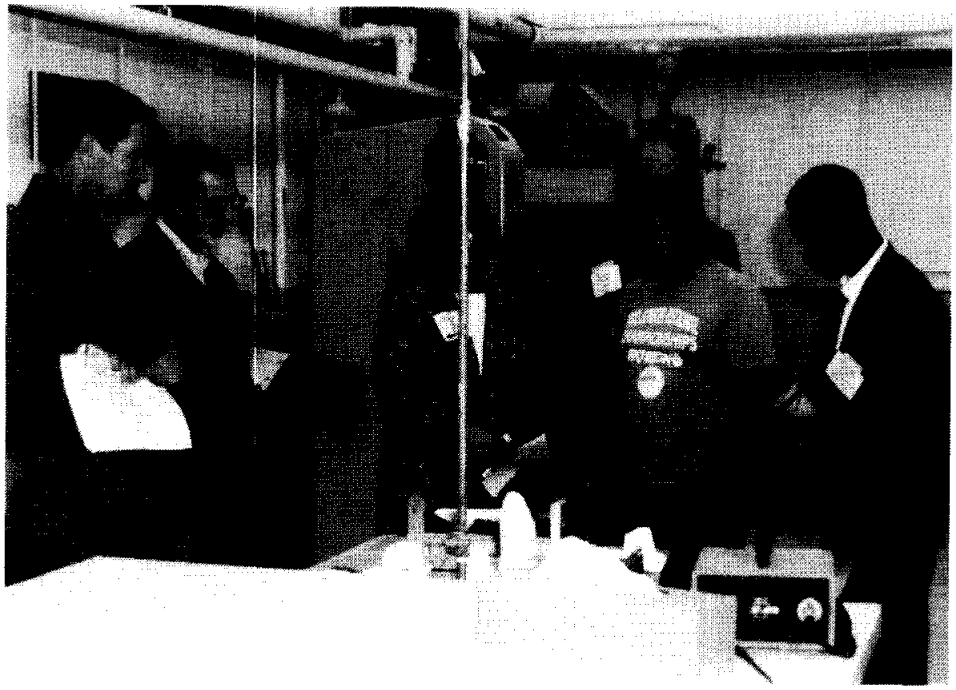

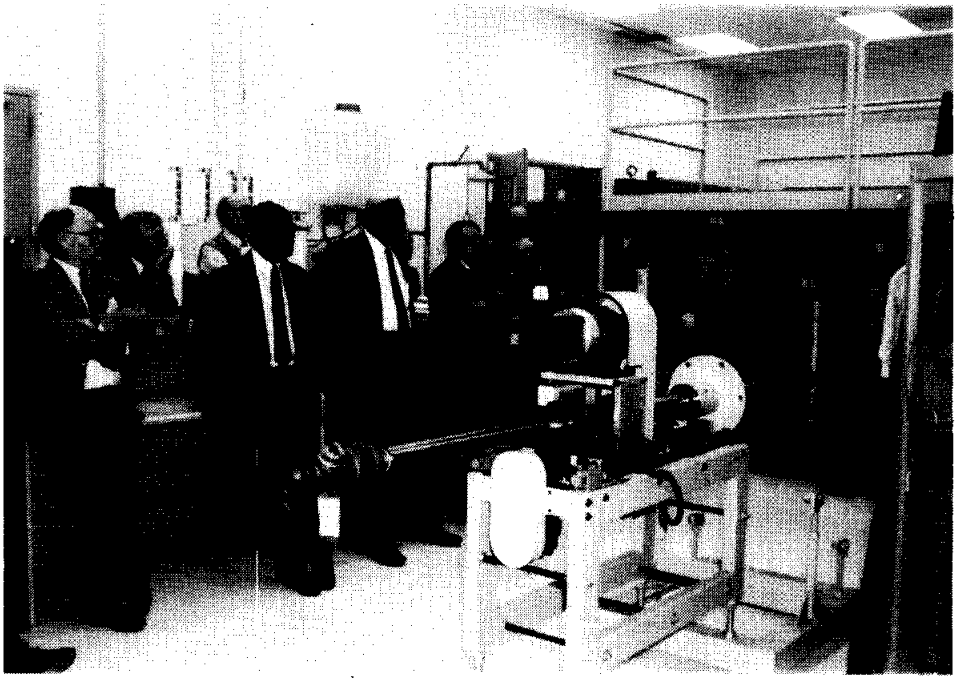

LISTING OF EXPERIMENTS FROM NEW:UPDATES

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NEW:Update 88 **NASA Conference Publication 3060**

Sastri, Sankar. "Fluorescent Penetrant Inspection"
Sastri, Sankar. "Magnetic Particle Inspection"
Sastri, Sankar. "Radiographic Inspection"

NEW:Update 89 **NASA Conference Publication 3074**

Chowdhury, Mostafiz R. and Chowdhury, Farida. "Experimental Determination of Material Damping Using Vibration Analyzer"
Chung, Wenchiang R. "The Assessment of Metal Fiber Reinforced Polymeric Composites"
Stibolt, Kenneth A. "Tensile and Shear Strength of Adhesives"

NEW:Update 90 **NIST Special Publication 822**

Azzara, Drew C. "ASTM: The Development and Application of Standards"
Bates, Seth P. "Charpy V-Notch Impact Testing of Hot Rolled 1020 Steel to Explore Temperature Impact Strength Relationships"
Chowdhury, Mostafiz R. "A Nondestructive Testing Method to Detect Defects in Structural Members"
Cornwell, L. R., Griffin, R. B., and Massarweh, W. A. "Effect of Strain Rate on Tensile Properties of Plastics"
Gray, Stephanie L., Kern, Kristen T., Harries, Wynford L., and Long, Sheila Ann T. "Improved Technique for Measuring Coefficients of Thermal Extension for Polymer Films"
Halperin, Kopl. "Design Project for the Materials Course: To Pick the Best Material for a Cooking Pot"
Kundu, Nikhil. "Environmental Stress Cracking of Recycled Thermoplastics"
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NEW:Update 91 **NASA Conference Publication 3151**

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Gorman, Thomas M. "Designing, Engineering, and Testing Wood Structures"
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Karplus, Alan K. "Determining Significant Material Properties, A Discovery Approach"
Spiegel, F. Xavier and Weigman, Bernard J. "An Automated System for Creep Testing"
Denton, Nancy L. and Hillsman, Vernon S. "Isotropic Thin-Walled Pressure Vessel Experiment"
Allen, David J. "Stress-Strain Characteristics of Rubber-Like Materials: Experiment and Analysis"
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NEW:Update 89 **NASA Conference Publication 3074**

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Ries, Heidi R. "Dielectric Determination of the Glass Transition Temperature"

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Molton, Peter M. and Clarke, Clayton. "Anode Materials for Electrochemical Waste Destruction"
Ries, Heidi R. "Dielectric Determination of the Glass Transition Temperature"

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Bunnell, L. Roy. "Temperature-Dependent Electrical Conductivity of Soda-Lime Glass
Dahiya, Jai N. "Phase Transition Studies in Barium and Strontium Titanates at Microwave Frequencies"

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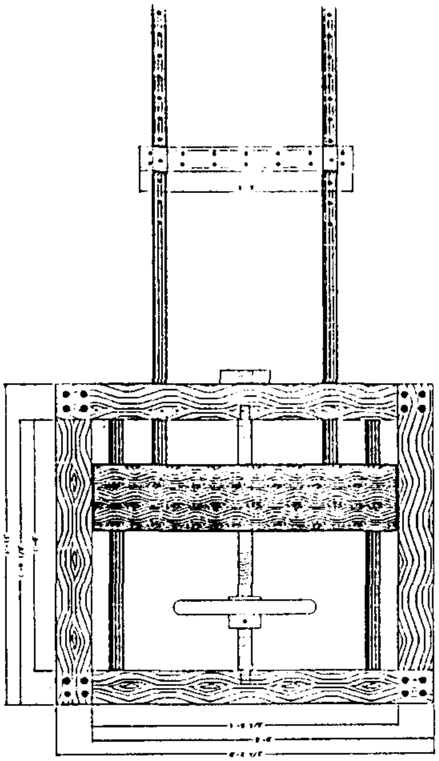
NATIONAL EDUCATORS' WORKSHOP

Update 93: Standard Experiments in Engineering Materials Science and Technology

November 3 - 5, 1993 - NASA LaRC, Hampton, Virginia



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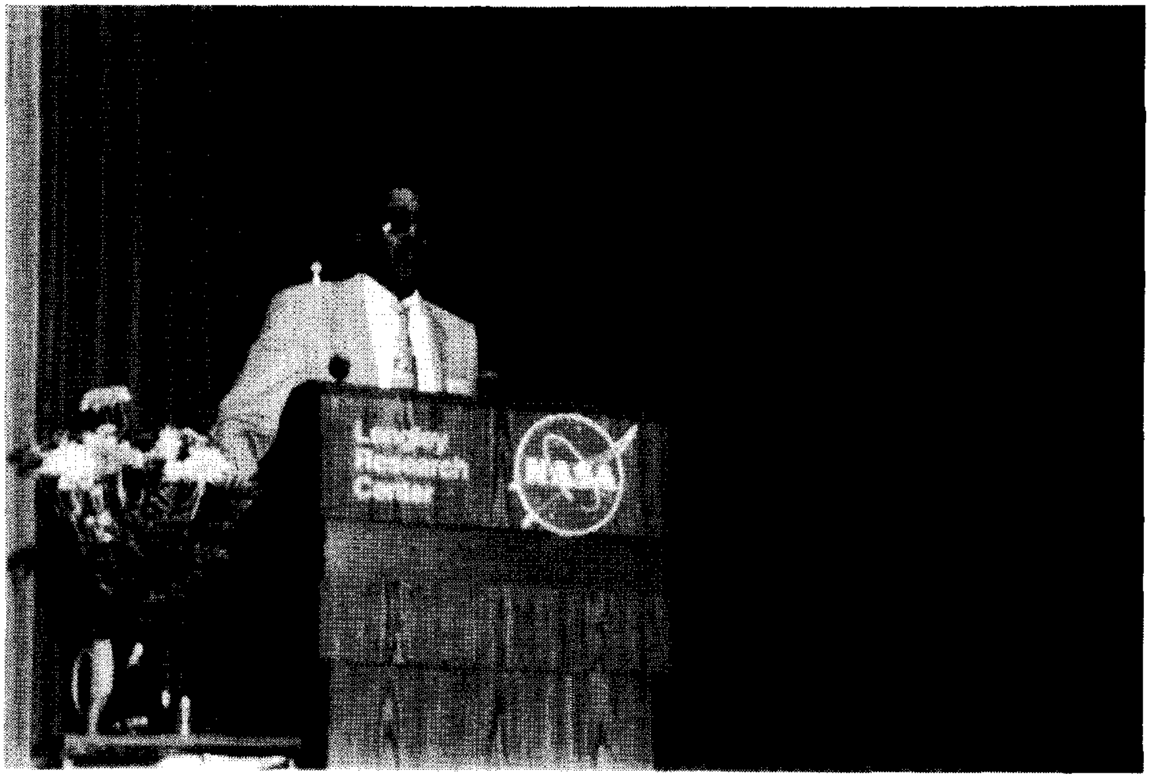
Welcome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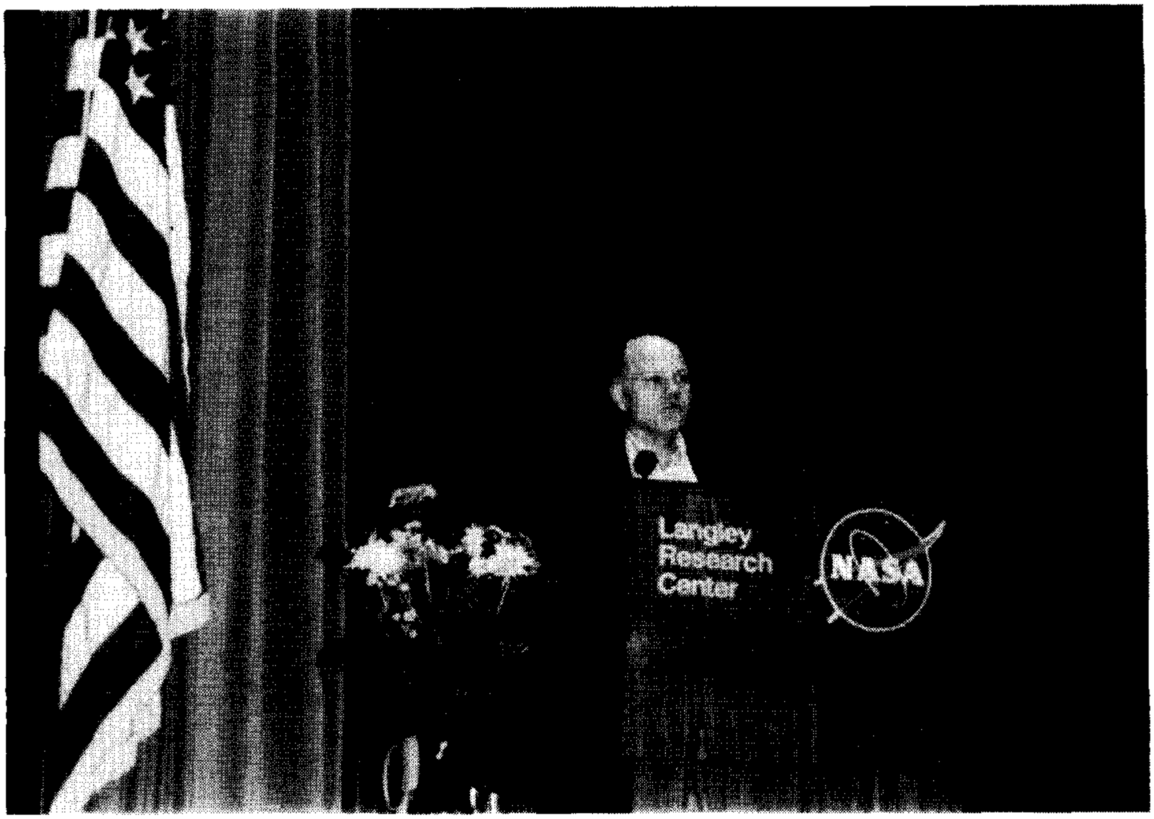
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Recognizing Contributions

Recognizing Contributions
(concluded)

Registration

ASTM TEST METHODS FOR COMPOSITE CHARACTERIZATION AND EVALUATION

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Outline of Presentation:

- Introduction
  - Objectives
- Discussion of ASTM
  - General Discussion
  - Subcommittee D-30
- Composite Materials Characterization and Evaluation
  - General Industry Practice
  - Test Methods for Textile Composites

Objectives :

- Introduce ASTM Organization and Activities
- Offer ASTM as a Resource
- Recruit New, Active Members

American Society for Testing and Materials

Definition :

A not-for-profit, voluntary, full-consensus *Standards Development Organization.*

ASTM publishes standards for ***Materials***, ***Products, Systems*** and ***Services***

Activities encompass ***Metals, Composites, Adhesives, Plastics, Textiles,*** paints, petroleum, construction, energy, the environment, consumer products, medical services and devices, computer systems, electronics, and many others.

American Society for Testing and Materials

Purpose :

"the Development of ***Standards*** . . . and the Promotion of ***Related Knowledge.***"

Promotion of ***Related Knowledge*** Accomplished through:

- Symposia and Workshops
- Technical Publications

American Society for Testing and Materials

ASTM produces six principal types of ***Standards.*** They are:

Standard Test Methods - a definitive procedure for the identification, measurement, and evaluation of one or more qualities, characteristics, or properties of a material, product, system, or service that produces a test result.

Standard Specification - a precise statement of a set of requirements to be satisfied by a material, product, system, or service that also indicates the procedures for determining whether each of the requirements is satisfied.

Standard Practice - a definitive procedure for performing one or more specific operations or functions that does not produce a test result.

Standard Terminology - a document comprised of terms, definitions, descriptions of terms, explanations of symbols, abbreviations, or acronyms.

Standard Guide - a series of options or instructions that do not recommend a specific course of action.

Standard Classification - a systematic arrangement or division of materials, products, systems, or services into groups based on similar characteristics such as origin, composition, properties, or use.

American Society for Testing and Materials

Technical Publications :

ASTM publishes a variety of technical documents other than standards. They include:

Special Technical Publications (STPs) - collections of peer-reviewed technical papers. Most STPs are based on symposia sponsored by ASTM Technical Committees.

Manuals, Monographs, and Data Series -

Technical Journals:

- *Journal of Composites Technology and Research*
- *Journal of Testing and Evaluation*
- *Cement, Concrete, and Aggregates*
- *Geotechnical Testing Journal*
- *Journal of Forensic Sciences*

Note: Papers presented in all publications are peer reviewed.

American Society for Testing and Materials

Facts and Figures :

- Organized In 1898.
- Membership Totals 34,000 Worldwide.
- 132 Standards-Writing Committees.
- Publishes 9000 ASTM Standards In The 69 Volume Annual Book Of ASTM Standards.
- Conducts Approximately 40 Symposia Annually.
- Publishes 40 To 50 Standard Technical Publications (STPs) Annually.

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Test Methods, Practices, Guides, and Terminology Documents:

High Modulus Fibers and Their Composite Materials

Test Methods:

D2344 - 84 (1989)	Apparent Interlaminar Shear Strength of Parallel Fiber Composites by Short-Beam Method
D2290 - 87	Apparent Tensile Strength of Ring or Tubular Plastics and Reinforced Plastics by Split Disk Method
D3410 - 87	Compressive Properties of Unidirectional or Crossply Fiber-Resin Composites
D3171 - 76 (1990)	Fiber Content of Resin-Matrix Composites by Matrix Digestion
D3553 - 76 (1989)	Fiber Content by Digestion of Reinforced Metal Matrix Composites
D3532 - 76 (1989)	Gel Time of Carbon Fiber-Epoxy Prepreg
D2586 - 68 (1990)	Hydrostatic Compressive Strength of Glass-Reinforced Plastic Cylinders
D2585 - 68 (1990)	Preparation and Tension Testing of Filament-Wound Pressure Vessels
C613 - 67 (1990)	Resin Content of Carbon and Graphite Prepregs by Solvent Extraction
D3531 - 76 (1989)	Resin Flow of Carbon Fiber-Epoxy Prepreg
D3529/3529M - 90	Resin Solids Content of Carbon Fiber-Epoxy Prepreg
D3552 - 77 (1989)	Tensile Properties of Fiber Reinforced Metal Matrix Composites
D3039 - 76 (1989)	Tensile Properties of Fiber-Resin Composites
D3479 - 76 (1990)	Tension-Tension Fatigue of Oriented Fiber Resin Matrix Composites
D4108 - 87	Thermal Protective-Performance of Materials for Clothing by Open-Flame Method
D3530/D3530M-90	Volatiles Content of Epoxy-Matrix Prepreg by Matrix Dissolution

ASTM Committee D-30, on High Modulus Fibers and Their Composites

Test Methods, Practices, Guides, and Terminology Documents (Cont.) :

Practices:

D2291 - 83 (1989)	Fabrication of Ring Test Specimens for Glass-Resin Composites
D3518 - 91	Inplane Shear Stress-Strain Response of Unidirectional Reinforced Plastics

Terminology Relating:

D3878 - 87	High-Modulus Reinforcing Fibers and Their Composites

Guides:

D4762 - 88	Automotive/Industrial Composite Materials, Testing of
D4255 - 83	Inplane Shear Properties of Composite Laminates, Testing

High Modulus Fibers

Test Methods:

D3800 - 79 (1990)	Density of High-Modulus Fibers
D4018 - 81	Tensile Properties of Continuous Filament Carbon and Graphite Yarns, Strands, Rovings, and Tows
D3379 - 75 (1989)	Tensile Strength and Young's Modulus for High-Modulus Single-Filament Materials
D4102 - 82 (1987)	Thermal Oxidative Resistance of Carbon Fibers

Terminology Relating:

D3878 - 87	High-Modulus Reinforcing Fibers and Their Composites

Guides:

D3544 - 76 (1989)	Reporting Test Methods and Results on High Modulus Fibers

ASTM Committee D-30, on High Modulus Fibers and Their Composites

Recent Special Technical Publications:

STP 1059 : Composite Materials: Test and Design (Ninth Volume)
S. P. Garbo, Ed. - 1990

STP 1080 : Thermal and Mechanical Behavior of Metal Matrix and Ceramic Matrix Composite Materials
J. M. Kennedy, H. H. Moeller, and W. S. Johnson, Eds. - 1990

STP 1110 : Composite Materials: Fatigue and Fracture (Third Volume)
T. K. O'Brien, Ed. - 1991

STP 1120 : Composite Materials: Testing and Design (Tenth Volume)
G. C. Grimes, Ed. - 1992

STP 1128 : Damage Detection in Composite Materials
J. E. Masters, Ed. - 1992

STP 1156 : Composite Materials: Fatigue and Fracture (Fourth Volume)
W. W. Stinchcomb and N. E. Ashbaugh, Eds. - 1993

STP 1174 : High Temperature and Environmental Effects on Polymeric Composites
C. E. Harris and T. S. Gates, Eds. - 1993

STP 1203 : Fractography of Modern Engineering Materials: Metals and Composites, Second Volume
J. E. Masters and L. N. Gilbertson, Eds. - 1993

STP 1206 : Composite Materials: Testing and Design (Eleventh Volume)
E. T. Camponeschi, Ed. - 1993

Composite Material : Characterization and Evaluation

A Survey Of Major Aircraft Manufacturers Indicates that:

- Procedures Are Designed to Minimize the Risk of Spending A Large Amount Of Funds On Materials Which Do Not Meet ***Structural*** or ***Processing*** Requirements.

- Materials Evaluation Conducted in Three Stages: ***Material Screening, Material Characterization***, and ***Development of Design Allowables.***

- Although The Tests Employed Were Not Identical, The Properties Measured At Each Level Of Investigation Were Similar From Company To Company.

- Majority Of Tests Focus On Obtaining The Mechanical Properties Which Are Most Useful To The ***Designer*** And The ***Structural Analyst*** But Which May <u>*Not*</u> Be Of Great Interest To The ***Material Scientist.***

- Three Major Design Factors that Control the Weight of an Aircraft: ***Stiffness, Damage Tolerance, and Stress Concentrations at Cut-Outs and Loaded Bolt Holes.***

Composite Material : Characterization and Evaluation

Screening Evaluation :

- First Step In the Material Characterization and Evaluation Process.

- Objective: Determine Material ***Acceptability*** for Aircraft Structural Applications.

- Compared Candidate Material To A Baseline Material To Determine if a More Extensive Evaluation Program is Warranted.

- 50 to 60 Tests Typically Performed.

Composite Material : Characterization and Evaluation

Screening Evaluation Tests :

A list of test methods commonly employed in screening evaluations is contained in the following table.

Test Type	Properties Measured	Environmental Condition
0° Tension	Strength, Modulus	RTA
0° Compression	Strength, Modulus	RTA, ETW
+/- 45° Tension	Strength, Modulus	CTA, RTA, ETW
Interlaminar Shear	Strength	RTA
Laminate Compression	Strength	RTA
Open Hole Tension	Strength	CTA, RTA, ETW
Open Hole Compression	Strength	CTA, RTA, ETW
Compression after Impact	Strength	RTA
Bolt Bearing Tension	Strength	RTA

Note: CTA indicates -65 ° F/ Ambient Moisture Conditions
RTA indicates Room Temperature/ Ambient Moisture Conditions
ETW indicates Elevated Temperature/ Saturated Moisture Conditions

Composite Material :
Characterization and Evaluation

Material Characterization :

- Objective: Establish ***Preliminary*** Design Properties for Design and Analysis of Test Components for Design Trade Studies.
- Measure Lamina Properties Required to Support Laminated Plate Theory and Failure Criteria.
- Measure Laminate Properties to Support Analysis and Design.
- 200 to 250 Tests Typically Performed.

Composite Material : Characterization and Evaluation

Materials Characterization Tests :

A list of test methods commonly employed in materials characterization tests is contained in the following table.

Test Type	Properties Measured	Environmental Condition
0° Tension	**Strength, Modulus, Poisson's Ratio**	CTA, **RTA**, ETW
90° Tension	Strength, Modulus, Poisson's Ratio	CTA, RTA, ETW
0° Compression	**Strength, Modulus**	CTA, **RTA, ETW**
90° Compression	Strength, Modulus	CTA, RTA, ETW
+/- 45° Tension	**Modulus**	**CTA, RTA, ETW**
In-Plane Shear	Strength	RTA
Interlaminar Shear	**Strength**	**RTA**
Interlaminar Tension	Strength	RTA
Laminate Compression	**Strength,** Modulus	CTA, **RTA**, ETW
Open Hole Tension	**Strength**	**CTA, RTA, ETW**
Open Hole Tension (Fatigue)	S - N Data	RTA
Filled Hole Tension	Strength	RTA
Open Hole Compression	**Strength**	**CTA, RTA, ETW**
Filled Hole Compression	Strength	RTA
Compression after Impact	**Strength**	**RTA**
Bolt Bearing Tension	**Strength**	**RTA**
Mode I Delamination Resistance	G_{IC}	RTA
Mode II Delamination Resistance	G_{IIC}	RTA

Note: **Bold Type** indicates tests performed in Screening Evaluation
CTA indicates -65 ° F/ Ambient Moisture Conditions
RTA indicates Room Temperature/ Ambient Moisture Conditions
ETW indicates Elevated Temperature/ Saturated Moisture Conditions

Composite Material :
Characterization and Evaluation

Development Of Design Allowables :

- Objective: Develop *Complete* Database for *Final Design* and *Certification*.
- Same Types of Tests Used in Materials Screening and Characterization Evaluations.
- Test Matrix *Expanded* to Include Additional Laminate Configurations, Alternate Specimen Geometries (e.g. Width/Diam. Ratios), Additional Environmental Conditions, More Replicate Tests on Samples taken from Several Batches of Material.
- Could Total Thousands of Tests Depending on Certification Requirements.

Composite Material :
Characterization and Evaluation

Tests Applied to Laminated Tape Composites :

TEST TYPE	TEST METHOD
• TENSION:	
Unnotched	ASTM D3039, D3518 MISC. COMPANY METHODS
Notched	SACMA SRM 5 NASA 1142- B9
• COMPRESSION:	
Unnotched	ASTM 3410 SACMA SRM1 NASA SHORT BLOCK MISC. COMPANY METHODS
Notched	SACMA SRM 3 NASA 1092 ST-4 MISC. COMPANY METHODS
• COMPRESSION AFTER IMPACT	SACMA SRM 2 NASA 1142 B11
• BOLT BEARING	MISC. COMPANY METHODS
• INTERLAMINAR TENSION	FLATWISE TENSION CURVED BEAM
• INTERLAMINAR SHEAR	ASTM D2344
• MODE I DELAMINATION	DOUBLE CANTILEVER BEAM
• MODE II DELAMINATION	END NOTCHED FLEXURE

Note: SACMA Indicates Test Methods Developed by the Suppliers of Advanced Composite Materials Association

Composite Material : Characterization and Evaluation

Physical Properties Measured :

Prepreg Tape:

Resin Content
Fiber Content
Volatile Content

Cured Laminates:

Resin Content
Fiber Content
Void Content
Density/Specific Gravity
Glass Transition Temperature (Dry and Wet)
Equilibrium Moisture Content
Thermal Conductivity
Heat Capacity
Coefs. of Thermal Expansion
Thermal Oxidative Stability

Development of Test Methods for Textile Composites

Program Objective :

As indicated below, the objective of this on-going effort, simply stated, is to develop a set of test methods and guidelines to be used to measure the mechanical and physical properties of composite materials reinforced with fibrous textile preforms. Investigations conducted to date have indicated that existing methods, which were developed largely to evaluate laminated tape type composites, may not adequately address the subtleties of these new material forms.

Develop And Verify Recommended Mechanical ***Test Procedures*** And ***Instrumentation Techniques*** For ***Textile Composites***

Development of Test Methods for Textile Composites

Statement of Problem :

The problem to be addressed is summarized in the two bullet statements given below. Simply stated, the test methods listed in the previous figures were developed to evaluate composite materials formed by laminating layers of pre-impregnated fiber-reinforced tape. The microstructure of these laminated composite materials differs significantly from the braided, woven, and stitched materials to be evaluated in this program. The fiber architecture will play a prime roll in determining the mechanical response of these textile composite materials. Will existing methods and practices accurately reflect the material response of these materials?

- **TEST METHODS DEVELOPED FOR LAMINATED TAPE COMPOSITES**

- **TEXTILE ARCHITECTURE CONTROLS MATERIAL RESPONSE**

Development of Test Methods for Textile Composites

Textile Composites Testing Issues :

It is not difficult to identify a number of specific testing issues relative to textile composites. Several of these concerns, which are applicable to virtually all of the test methods listed on the previous page, are listed below.

The first two reflect the unique size effects these materials may present. A unit cell is defined as the smallest unit of repeated fiber architecture. It may be considered the building block of the material. The size of the unit cell is dependent on a number of factors including the size of the yarns, the angle at which they are intertwined or interwoven, and the intricacy of the braid or weave pattern. A representative volume of material must be tested and monitored to accurately reflect true material response. Specimen geometry and strain gage sizes must be reexamined in terms of unit cell size. The effect of the sizes of the yarn bundles must also be considered since they may also affect the performance and the measurements. This is expressed in the third statement.

The final three items on the list reflect concerns over specimen geometry. Test specimen dimensions established for tape type composites may not be applicable to textile composites. The degree of heterogeneity present in the latter materials is quite different than that encountered in the former. The potential effects of these differences must be also quantified.

A limited amount of relevant data has been developed for 2-D triaxially braided textile composites. These results will be reviewed in the following section. They include Moiré interferometry and strength and modulus measurements.

- EFFECT OF UNIT CELL SIZE ON MECHANICAL PERFORMANCE
- EFFECT OF UNIT CELL SIZE ON STRAIN GAGE AND DISPLACEMENT MEASUREMENTS
- EFFECT OF TOW SIZE AND FIBER ARCHITECTURE ON MECHANICAL PERFORMANCE
- EFFECT OF FINITE WIDTH ON UNNOTCHED AND OPEN-HOLE SPECIMENS
- EFFECT OF EDGE CONDITIONS ON MECHANICAL PERFORMANCE
- EFFECT OF TEXTILE THICKNESS ON MECHANICAL PERFORMANCE

Development of Test Methods for Textile Composites

Program Approach :

A straightforward approach has been adopted to meet the objective outlined in the previous figure. An extensive test program will be conducted to gather data addressing the concerns listed earlier. The program, which will include a wide variety of woven, braided, and stitched preform architectures, will consider several loading conditions.

The general approach is outlined below. Details of material tested and test methods are supplied in the following pages.

- ➤ **IDENTIFY AND/OR DESIGN AND DEVELOP SPECIMEN CONFIGURATIONS AND TEXT FIXTURES**
- ➤ **CONDUCT MECHANICAL TEST PROGRAM**
  - **Variety of Test Methods**
  - **Variety of Instrumentation Techniques**
  - **Full Field Strain Measurements**
  - **Analytical Support**
- ➤ **IDENTIFY SMALLEST LEVEL OF HOMOGENEITY**
- ➤ **IDENTIFY APPROPRIATE TEST METHODS AND INSTRUMENTATION GUIDELINES**

Development of Test Methods for Textile Composites

Description of Material Tested : Preforms and Textile Parameters Studied

Fifteen woven, braided, and stitched preforms will be evaluated in the program. The preform types are listed below in the table; the number of each type to be tested is indicated in parentheses. The table also lists the braid parameter that will be varied for each preform type. The list of materials reflects the material forms that are being evaluated by the aircraft manufacturers in the ACT program.

TEXTILE PREFORM TYPES :

- ➤ 2-D TRIAXIAL BRAIDS - (4)
  - Tow Size
  - % Longitudinal Tows
  - Braid Angle
- ➤ 3-D INTERLOCK WEAVE - (6)
  - Weave Type - (3)
  - Warp, Weft, and Weaver Tow Size
- ➤ STITCHED UNIWEAVE - (5)
  - Stitch Material
  - Stitch Spacing
  - Stitch Yarn Size

MATERIALS :

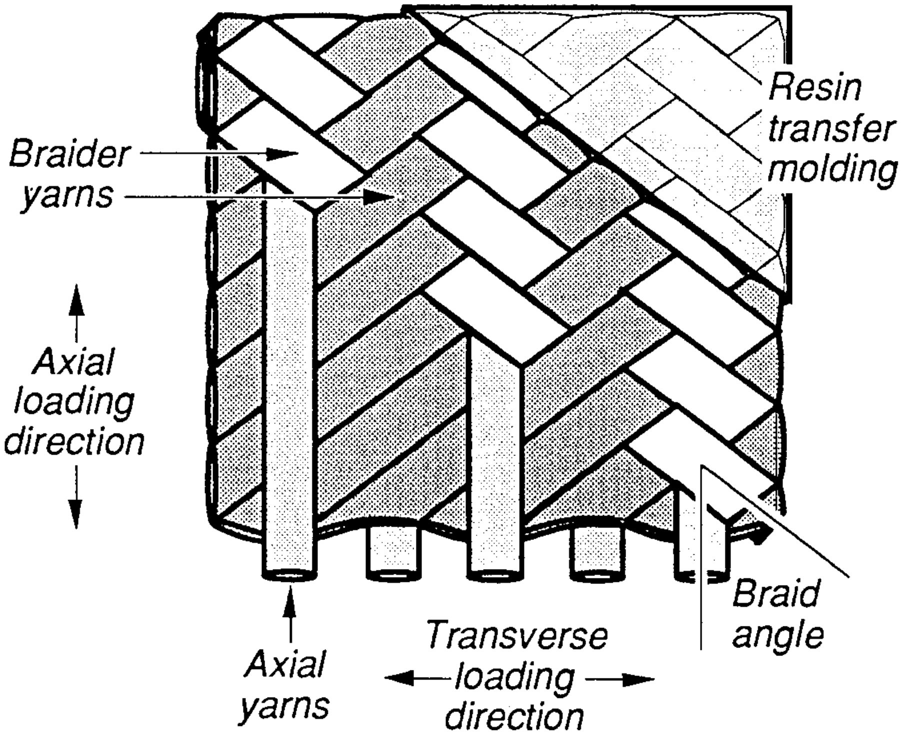
FIBER: HERCULES AS4
RESIN: SHELL 1895

Development of Test Methods for Textile Composites

Description of Material Tested :
Triaxial Braid Pattern

The specimens studied in this investigation featured 2-D triaxially braided AS4 graphite fiber preforms impregnated with Shell 1895 epoxy resin. In a triaxially braided preform three yarns are intertwined to form a single layer of 0°/± Θ° material. In this case, the braided yarns are intertwined in a 2 x 2 pattern. Each + Θ yarn crosses alternatively over and under two - Θ yarns and vice versa. The 0° yarns were inserted between the braided yarns. This yields a two dimensional material. The figure below schematically illustrates the fiber architecture and establishes the nomenclature used in the paper.

The yarns were braided over a cylindrical mandrel to a nominal thickness of 0.125 in. The desired preform thickness was achieved by overbraiding layers; there are no through-the-thickness fibers. After braiding, the preforms were removed from the mandrel, slit along the 0° fiber direction, flattened, and border stitched to minimize fiber shifting. The resin was introduced via a resin transfer molding process.



Development of Test Methods for Textile Composites

Triaxial Braid Configurations Tested :

Three preform parameters, braid angle, yarn size, and 0° yarn content, were varied in this study. The last parameter listed is typically expressed as a percentage of 0° yarns. It is the volumetric proportion of longitudinal yarns to total yarn content and is a function of braid angle and yarn size. Yarn size is expressed in terms of the number of filaments per yarn. The AS4 fibers used in these materials have a nominal diameter of 7 microns. The longitudinal yarns were larger than the braided yarns in all cases. The B1 and B2 architectures had the same yarn sizes; they differed in braid angle and 0° yarn content. The preform parameters are listed in the table.

The fabrics were formed with a 144 carrier New England Butt triaxial braider, incorporating 72 longitudinal yarns. The mandrel diameters varied for each architecture. Since the number of carriers was constant, this had the effect of changing the yarn spacing. These parameters are also listed in the table.

MATERIAL	BRAID PATTERN	BRAIDER YARN SIZE	0° YARN SIZE	0° YARN CONTENT (%)	0° YARN SPACING (Yarn/In.)	BRAID YARN SPACING (Yarn/In.)
A1	0/± 63°	12K	24K	31.5	4.17	9.16
B1	0/±66.5°	6K	18K	37.6	4.77	11.98
B2	0/±70°	6K	18K	34.0	4.37	12.74

Note: K indicates thousands. For the AS-4 yarns, each filament is 7 microns in diameter

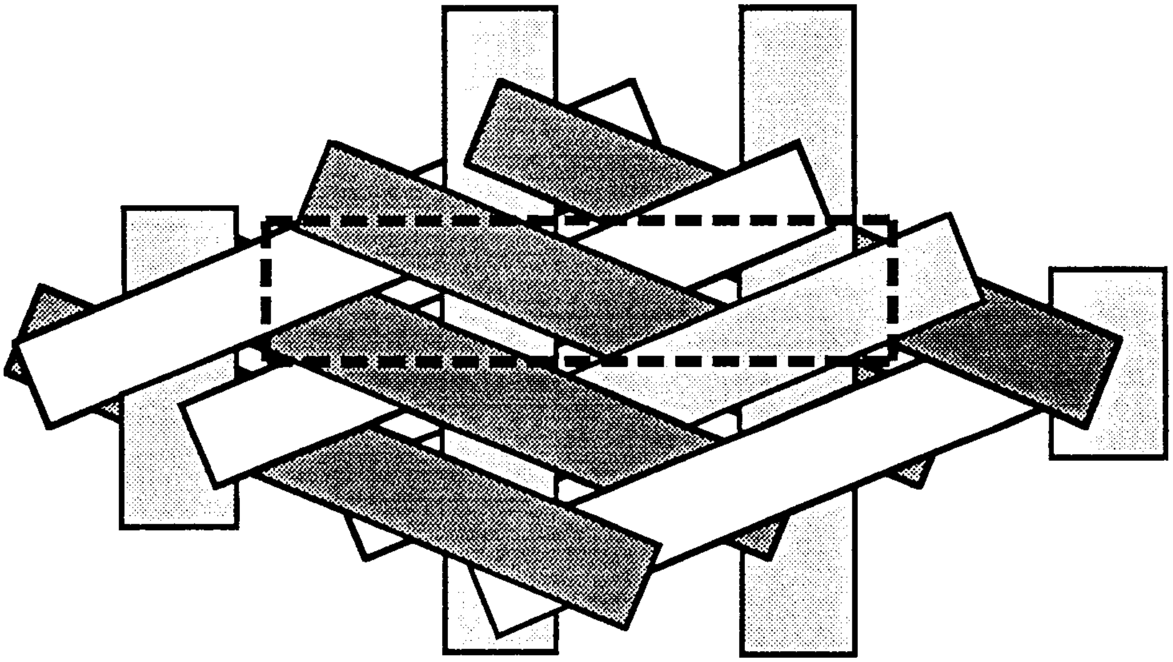
Development of Test Methods for Textile Composites

Unit Cell Definition :

A convenient way to describe textile preforms is to identify a unit cell of material - a repeatable unit of fabric geometry. The unit cell represents the complete yarn intertwinement pattern. The unit cell approach has become the foundation of textile analysis and serves as a convenient framework in which to interpret experimental data.

The rhombic frame show in the figure defines a unit cell for the 2-D triaxially braided material studied in this program. For computational purposes, it is desirable to define the smallest unit cell possible. In some analyses, rectangular unit cells are also required. The rectangular section shown in the figure represents the smallest unit cell identified.

The table shown below contains the dimensions of the unit cells for the three architectures tested. The unit cell width is dependent on the mandrel diameter and the number of yarns braided. The height of the unit cell is dependent on the cell width and the braid angle. Even though a conservative definition of the unit cell was applied in this case, the data in the table indicate that the unit cells can be quite large compared to typical specimen and strain gage dimensions.



UNIT CELL DIMENSIONS

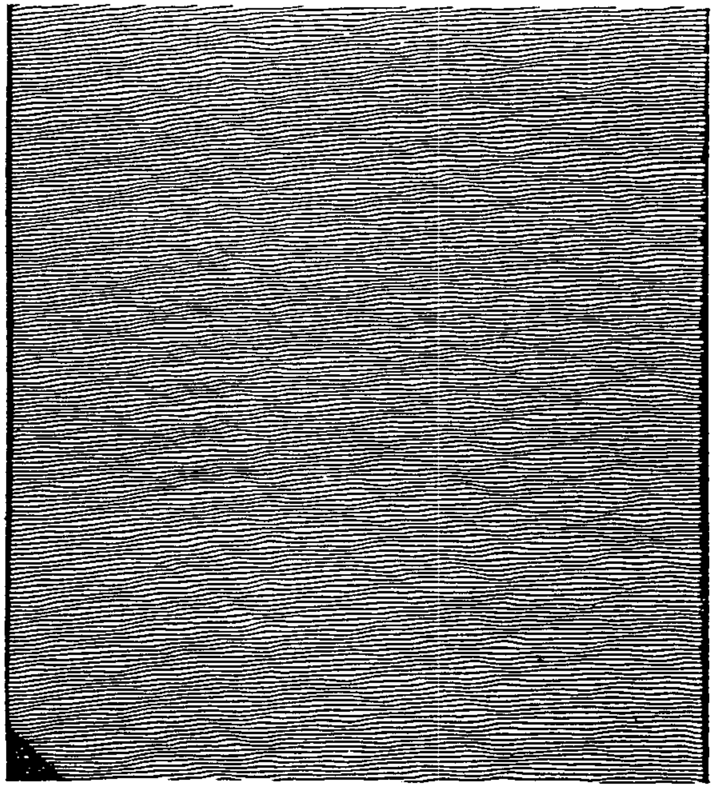
MATERIAL	WIDTH (in.)	HEIGHT (in.)
A1	0.48	0.12
B1	0.42	0.09
B2	0.46	0.08

MOIRÉ INTERFEROMETRY
Axial Load - Vertical Displacement Field

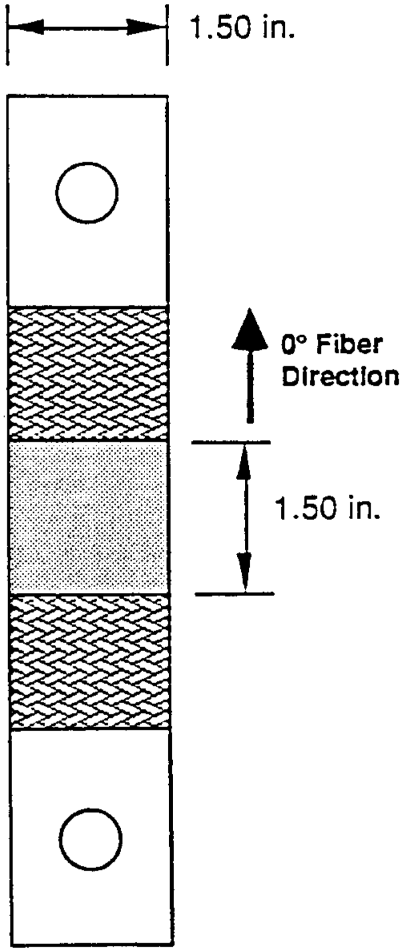
As indicated earlier, Moiré interferometry was used to define the full field strain distribution in these braided specimens. The technique defines deformation patterns in both the vertical and horizontal directions. The technique was applied to specimens subjected to longitudinal and transverse loading. These results are shown in this and the following figures.

The figure below illustrates the specimen geometry and highlights the section studied. The vertical displacement field that resulted when a specimen was loaded to 1200 micro-strain along the 0° fiber direction is also shown in the figure.

The vertical displacement fields (V fields) consist of basically horizontal fringes; this indicates specimen extension where points along one fringe have been displaced vertically with respect to points along a neighboring fringe. For a uniform extension the fringes should be evenly spaced and straight. The fringes for the specimens tested, however, are wavy and the spacing between them varies. The variation is cyclic and coincides with the repeated unit of the textile architecture.

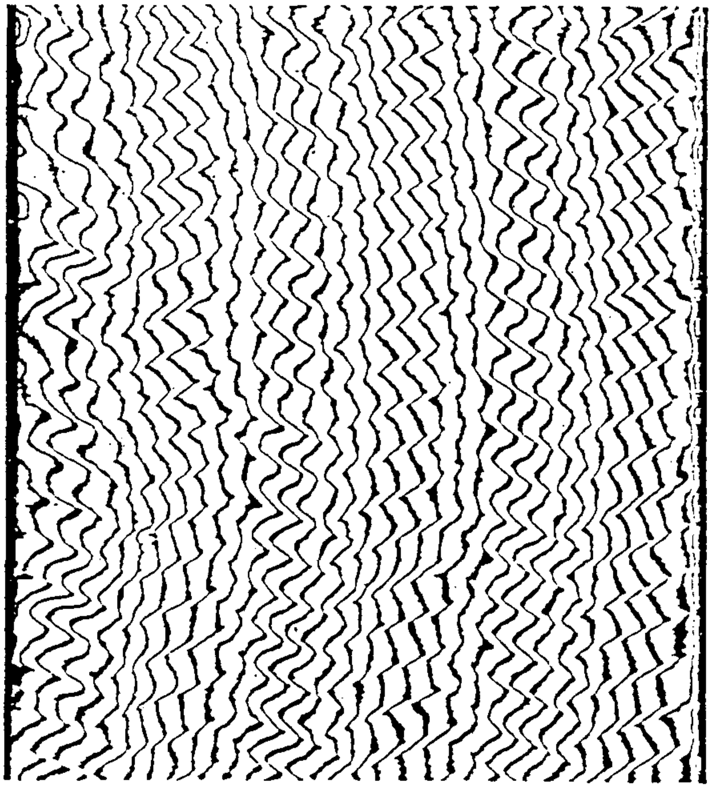


Vertical Displacement Field

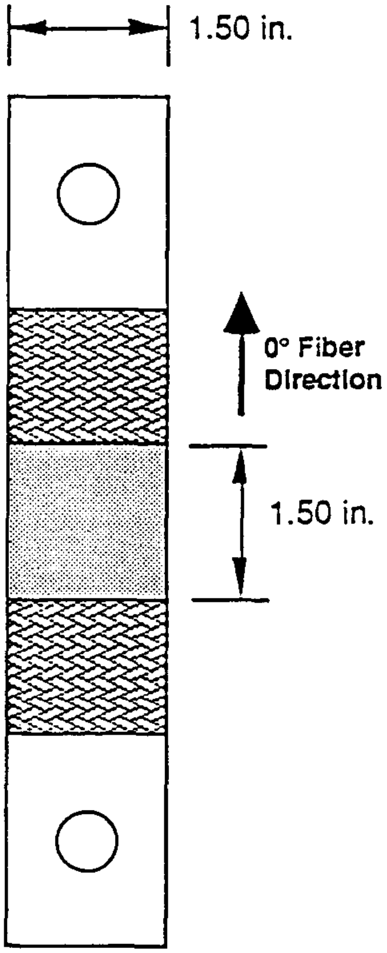


MOIRÉ INTERFEROMETRY
Axial Load - Horizontal Displacement Field

The horizontal displacement patterns (U fields) consist of zigzag vertical fringes that display the Poisson's effect. For uniform contraction the fringes should be straight and the spacing constant. The fringes however display a variation which is cyclic, and matches that of the braid geometry. The sharp kinks in the U field fringes reveal the presence of shear strains between the fiber bundles.

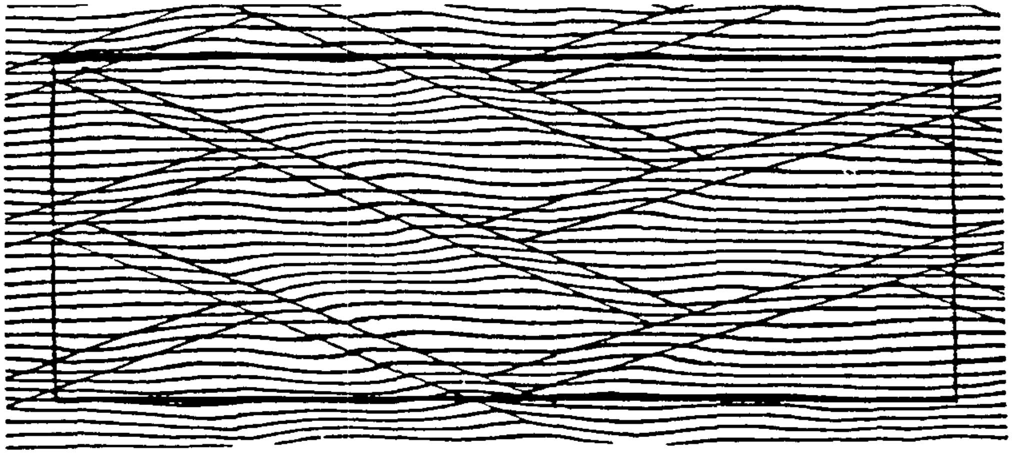


Horizontal Displacement Field

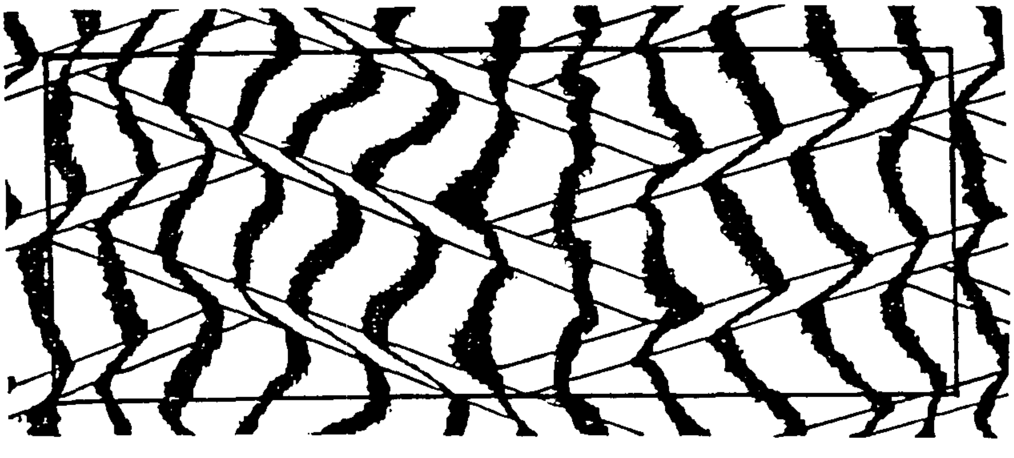


ENLARGED VIEW OF TWO UNIT CELLS OF SPECIMEN
(Axial Loading)

The figure shows the V and U fields of a highly magnified region of specimen that consists of two unit cells. The boundaries between adjacent fiber bundles and the outline of the cells are marked. It was revealed that the shear deformation at interfaces between the fiber bundles occurred over a finite width. This width is illustrated in the patterns as the distance between the closely spaced lines. This is consistent with the presence of the resin rich areas between the fiber bundles, which was on the order of one fifth of the width of the fiber bundle itself. The U field shows that the shear strain γ_{xy} in the resin rich zones was on the order of 0.5 times that of the average applied normal strain ε_y. Additionally, the U field shows that the Poisson effect was nearly constant across the unit cell. The V displacement pattern clearly shows that the strain ε_y varies significantly within each unit cell as can be seen by the nonuniform fringe spacing. The ratio of maximum strain ε_y to minimum strain was about 2 to 1. The normal strain varies on top of the fiber bundles and is nearly constant throughout all of the resin rich zones.



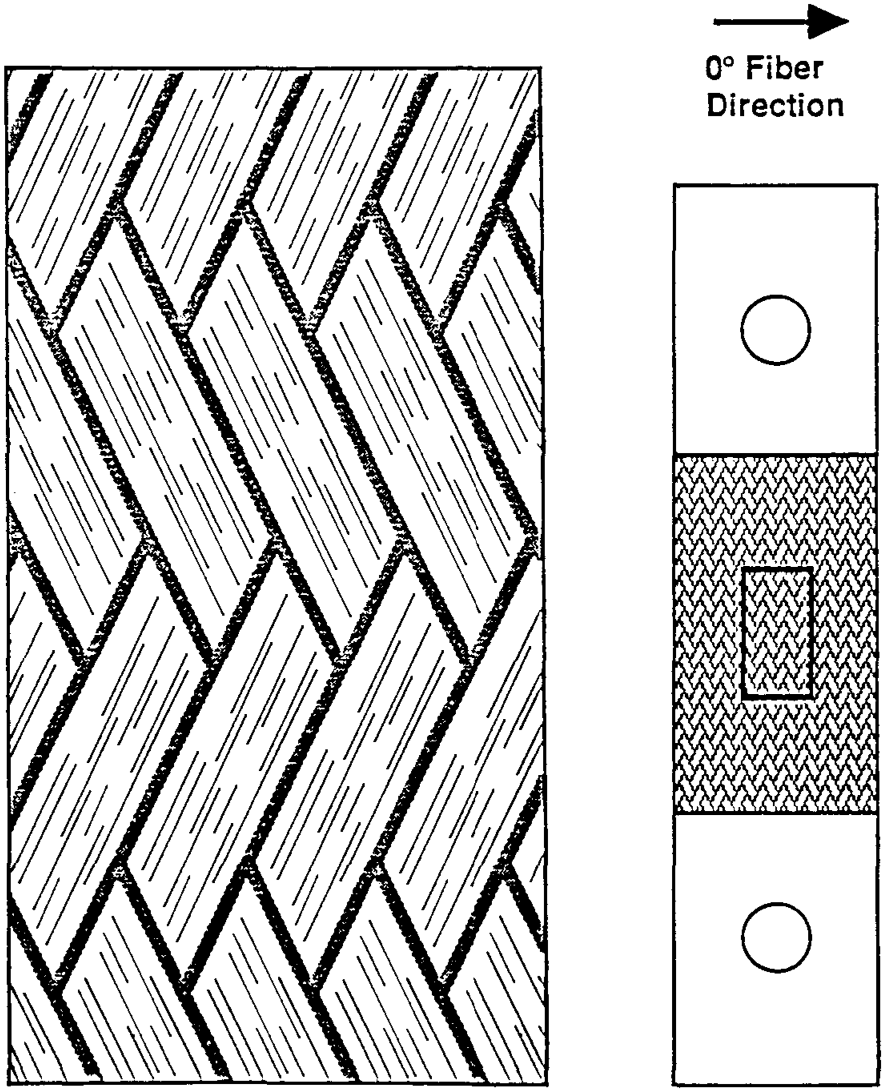
Vertical Displacement Field



Horizontal Displacement Field

SPECIMEN SECTION COINCIDING WITH MOIRÉ FRINGE PATTERNS
(Transverse Loading)

Interferometry was also performed on specimens loaded in the transverse direction (i.e. at 90° to the axial direction). This figure shows the region investigated in these specimens. The pattern of the surface braided yarns is shown schematically in the figure. The deformation fields that developed in these coupons are shown in the next two figures.

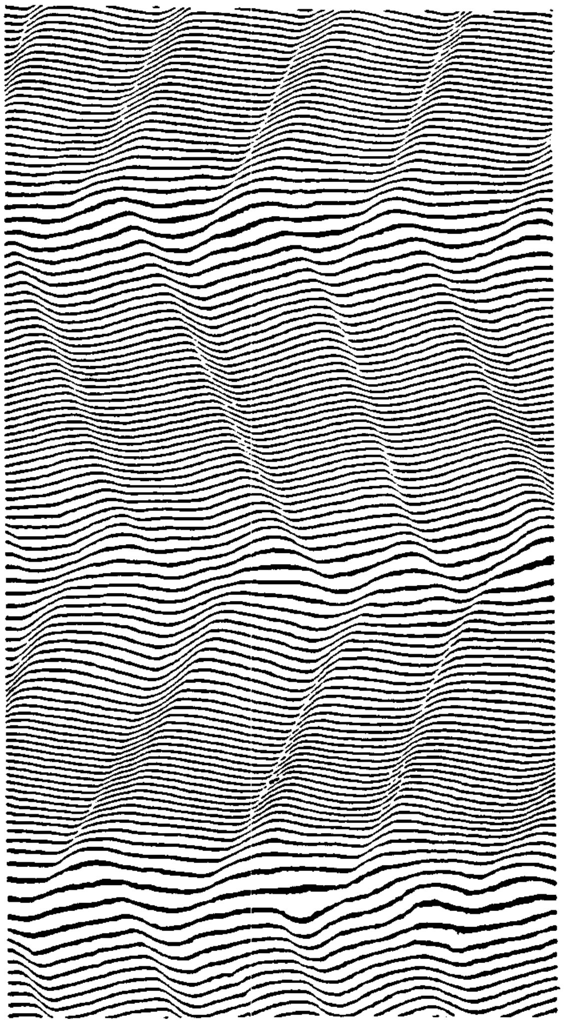


MOIRÉ INTERFEROMETRY
Transverse Load - Vertical Displacement Field

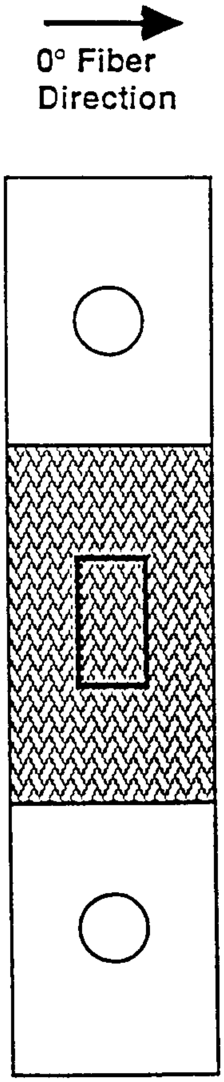
In general, the interferometry results indicate that greater variations in normal and shear strains existed in specimens loaded in the transverse direction than in the axial direction.

This figure displays the vertical displacement field for a coupon loaded in the transverse direction. The location of the yarns is evident in the vertical displacement fringe patterns, where sudden jogs in the fringes represent strong shear strains in the resin rich regions between the yarns. From the V displacement pattern, the spacing of the fringes in the vertical direction displays a cyclic variation. The strains are highest over the region where there are 90° fibers under the braider yarns. They are lowest over the regions where the braider yarns cross. The difference between the average strains in these areas is on the order of 3 times.

Unlike the axial loading case, the cyclic variation is not confined to the dimensions of the unit cell. The variation breaches the unit cell to form a global material response that covers the entire specimen. This is illustrated by the horizontal bands seen in the figure. They span several unit cells and extend across the specimen width.



Vertical Displacement Field



Development of Test Methods for Textile Composites

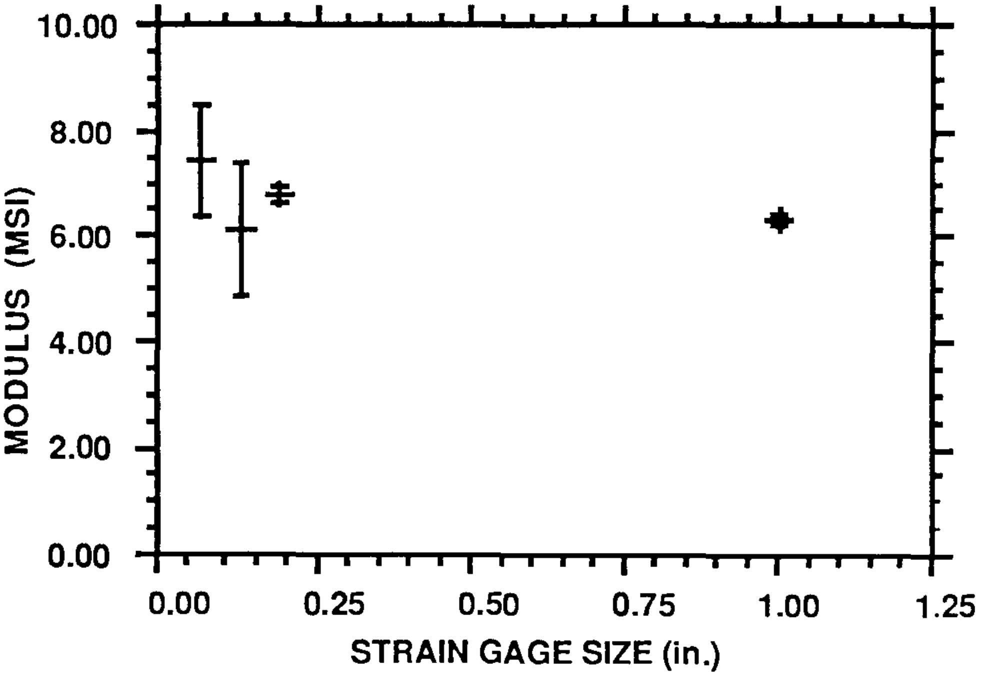
Effect of Strain Gage Size on Modulus :

The inhomogeneity in the strain fields demonstrated in the Moiré interferometric results discussed in the previous slides has significant implications with regard to specimen instrumentation. The large strain gradients seen within the unit cell graphically illustrate the need to measure strain over a truly representative volume of material to get an accurate determination of the global material response. Local strain readings can be misleading and confusing.

The data shown in the figure below demonstrate these points. The figure plots the measured transverse modulus of several B1 laminates vs. the size of the gages used to measure the strain. The gages ranged in length from 0.062 in. to 1.0 in.; the preform's unit cell measures 0.42 in. in this direction. The average modulus and the standard deviation of the data are shown in the figure. As the figure indicates, significant scatter was evident in the results obtained using the small gages. These effects are reduced as the length of the gage increased. The results also indicate that average value also decreased as strain gage size increased.

The results illustrate the need to consider the textile architecture when choosing instrumentation for a specimen.

Transverse Modulus vs Strain Gage Size

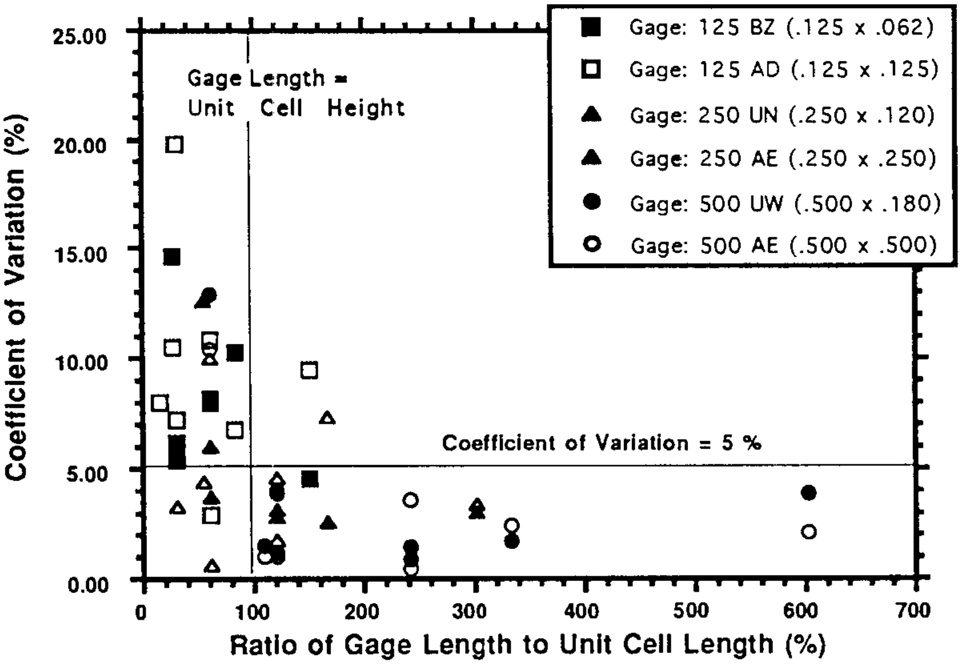


Effect of Strain Gage Size on Modulus :

The figure shown below presents results of a second evaluation of the effect of strain gage size on modulus measurements. The figure plots the coefficient of variation in the computed modulus measurements versus strain gage size (normalized to the dimension of the unit cell in the direction of loading). Six strain gage types were examined in these measurements. Their dimensions are indicated in the legend in the figure. A line indicating the point where the strain gage length equals the unit cell dimension has been added to the figure to aid in interpreting the data. Similarly, a line marking the point where the coefficient of variation equals 5% has also been added to the figure.

The figure again illustrates the need to consider the textile architecture when choosing instrumentation for a specimen.

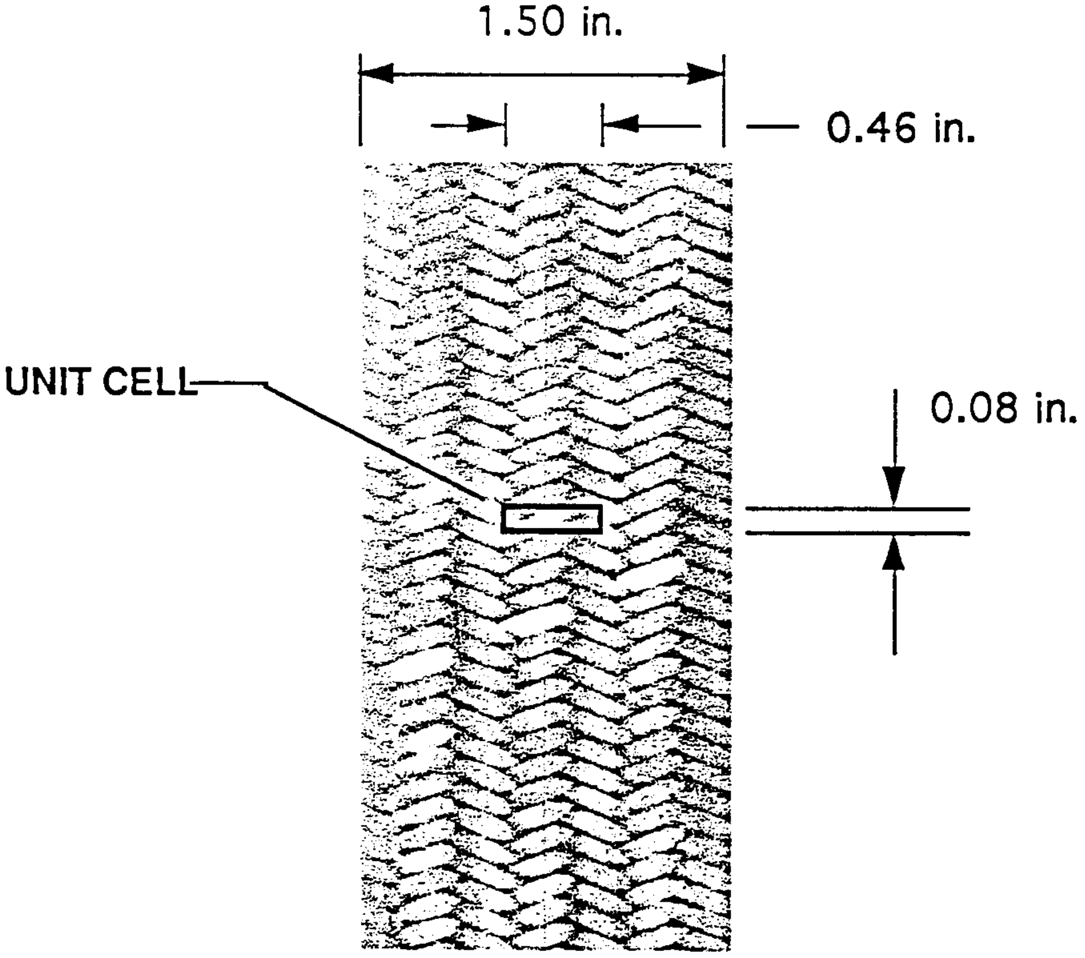
**Effect of Strain Gage Length on Modulus Measurements
(2-D Triaxial Braided Laminates)**



MATERIAL ARCHITECTURE AND TEST COUPON GEOMETRY

The preform architecture must also be considered when designing test specimens. The figure below contains a photograph of a tensile test coupon. The specimen, which is typical of those commonly used in screening and evaluation test programs, is 1.5 in. wide and 10 in. long. Superposed on the photograph are the B2 architecture's unit cell dimensions. As the figure illustrates, when oriented in this direction, the specimen is only three unit cells wide. This again raises the question of whether a representative volume of material is being sampled in the test.

Specimen width and thickness must be considered when designing test specimens to attain true measures of modulus and strength. Unfortunately, design criteria have not yet been established for these materials.

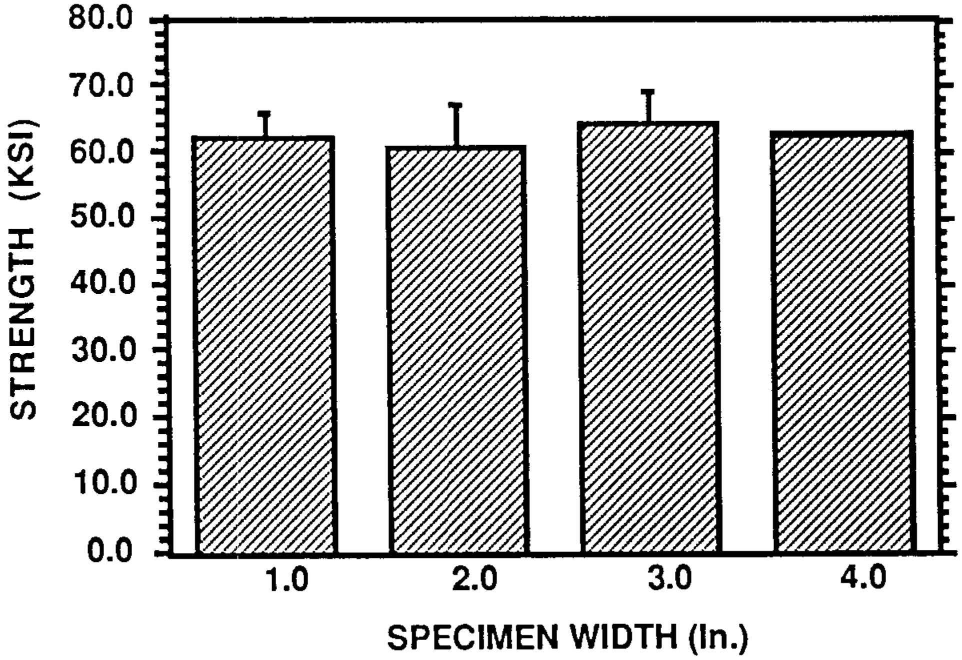


Effect of Specimen Width on Strength :

A series of longitudinal tensile tests were conducted to judge cursorily the effect of specimen width on strength of the B2 type 2-D triaxially braided laminates defined in an earlier figure. In these tests specimen width was varied from 1.0 in. (2 unit cells wide) to 4.0 in. (8 unit cells wide).

The results of these tests are shown in the figure below. The data, which have been normalized to 55% fiber volume to simplify the comparison, indicate that specimen width had no apparent effect on the test results for this architecture. The average strengths and the standard deviations of the results (indicated by the bars in the figure) were comparable for each group of tests (note: the 4.0 in. data represents the average of two tests; the standard deviation was not computed).

A larger, more complete, examination of the interaction of textile architecture and test laminate geometry is underway as a part of an effort to develop test methods for textile composites. This effort will be outlined in the following pages.



Development of Test Methods for Textile Composites

Summary Investigation of 2-D Braids:

A brief summary of the technical results reviewed in the presentation is given below. The experimental investigation conducted on 2-D braided materials indicated that significant strain gradients existed within the materials unit cell as a result of the braid architecture. This inhomogeneity in the strain field is an important factor that must be considered when choosing instrumentation for a test specimen. Although the 2-D braided laminates tested did not demonstrate a width effect, the size of the unit cell must also be considered when designing a test specimen.

Finally, the concerns discussed above and others listed in an earlier figure will be addressed in an on-going test method development effort.

- **MOIRÉ INTERFEROMETRY IDENTIFIED LARGE STRAIN GRADIENTS WITHIN THE UNIT CELL**
- **INHOMOGENEITY IN STRAIN FIELD EFFECTS INSTRUMENTATION**
- **UNIT CELL SIZE MAY AFFECT TEST RESULTS**
- **ON-GOING INVESTIGATION TO DEFINE TEXTILE TEST METHODS UNDERWAY**

CONTINUOUS UNIDIRECTIONAL FIBER REINFORCED COMPOSITES: FABRICATION AND TESTING

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CONTINUOUS UNIDIRECTIONAL FIBER REINFORCED COMPOSITES: FABRICATION AND TESTING *

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*Hauber Fellowship Funded

ABSTRACT: The study of the anisotropic mechanical properties of an inexpensively fabricated composite with continuous unidirectional fibers and a clear matrix was investigated. A method has been developed to fabricate these composites with aluminum fibers and a polymer matrix. These composites clearly demonstrate the properties of unidirectional composites and cost less than five dollars each to fabricate.

KEY WORDS: anisotropy, composite, fibers, matrix, reinforcement, strain, stress.

INTRODUCTION: A process was developed to make fiber composites with continuous fibers spanning the composite and aligned in one direction (Fig. 1). H.A. West and A.F. Sprecher have been studying the mechanical properties of this type of composite with samples of rubber reinforced with nylon cord.[1] These samples, however, are not readily available and require a hot press for consolidation and curing. It would be desirable to develop a system which sufficiently demonstrates the effect of fiber orientation, yet is easily fabricated from available materials. In addition to studying the parameters of this type of composite, a clear matrix was desired in order to view the fibers.

While developing this process to demonstrate anisotropic mechanical properties, it was very important to limit costs and to develop educational opportunities for composites in the laboratory and classroom. Composites are widely used in our society today; fiberglass, plywood, and concrete are just a few examples.[2]

PREREQUISITE KNOWLEDGE: The previous or concurrent study of composites, specifically continuous unidirectional composites, and their mechanical properties would be beneficial. The effect of fiber orientation on elastic modulus, Poisson's ratio, and state of strain can be investigated.

OBJECTIVE: An inexpensive fabrication process was developed for continuous unidirectional fiber reinforced composites with clear matrices in order to study material properties such as elastic anisotropy.

EQUIPMENT AND SUPPLIES: (1) Six 3" x 5 1/8" pieces of aluminum screening; (2) Utility knife; (3) Cutting board; (4) transparent or masking tape; (5) Manicure scissors or small wire cutters; (6) Three 200 mL plastic containers; (7) Plastic container 1" x 3" x 5" ; (8) Ferris see thru; (9) Por-a-mold;

(10) Release agent (Synair 1711); (11) Vacuum pump; (12) Oven (350 F); (13) Ferris jig (see Fig.3); (14) Two 4" x 6" x 1/8" glass plates; (15) Two clamps; (16) Isopropyl rubbing alcohol to clean containers.

PROCEDURE: Composite fabrication began with the preparation of either plain or anodized aluminum screening, the fibers, which were then set up for the matrix addition of either Por-a-mold or Ferris see thru. After the composite cured completely, its mechanical properties were tested.

Fibers

After cutting the screening with the fibers at the desired angle and affixing a 1/2" border of tape to all sides (to hold the screen together), a utility knife and a pair of manicuring scissors were used to cut out the unwanted fibers. By cutting the columns between every two desired fibers, the unwanted fibers slip away and leave the desired fibers intact (Fig. 2).

While preparing several layers, usually three or five, it was beneficial to leave three of the unwanted fibers intact near the center (support wires) and place the layer under a book while working on the others. Finally, the tape and support wires were removed; the preparation time ranged from 20-60 minutes per layer.

Matrix: Por-A-Mold

In the plastic container sprayed with the release agent, three layers of identical fibers were stacked flat. Then, the por-a-mold matrix was prepared by:

(1) stirring the curative thoroughly and measuring 75 mL in a plastic container
(2) stirring the prepolymer thoroughly and measuring 75 mL in a plastic container
(3) noting the time when adding the curative to the prepolymer (9-12 minutes working time)
(4) mixing it to a clear consistency (< 2 minutes)
(5) vacuuming it for 4 minutes
(6) pouring it evenly over the fibers
(7) spraying the release agent on the surface to remove additional air bubbles.

Finally, after allowing the matrix to cure at room temperature for 16 to 24 hours,[3] the composite was removed from the container and trimmed for testing.

Ferris See Thru

When a matrix of Ferris see thru was desired, three or five identical layers of fibers were fit into the grooves of the ferris jig before clamping a glass plate to each side (Fig. 3). Then, a small amount of the white ferris see thru liquid was dripped along the edges between the glass and jig to be heat cured at 350 degrees Fahrenheit until it turned clear (5-10 minutes). After a good seal was made, 175 mL of the ferris see thru was poured into the jig and vacuumed for five minutes. The Ferris see thru was then heat cured at 350 degrees Fahrenheit. After 50-60 minutes, the matrix became a clear solid with a slight medium yellow tint. The new composite was removed from the jig after it cooled.

Testing

Several composites were made using the two matrices, two types of fiber, and angles of 0, 15, 30, 45, 60, 75, and 90 degrees. Also, a few support wires were left in many of the first composites. Before testing, the supporting border of fibers was trimmed off to yield the continuous unidirectional fiber reinforced composite.

One purpose of combining two (or more) distinct materials to create a composite was clearly demonstrated by the first two samples tested under tension. A sample of solid por-a-mold elongated 7.5 inches with only 0.19 inches of plastic deformation and broke under merely 9.5 pounds. However, the por-a-mold and anodized Al fiber composite maintained its length up to 50 pounds and did not break under 110 pounds.

The most interesting tests depict elastic anisotropy. After a 90 degree angle is marked in the center of the sample, it is placed under tension while recording any angle change and the tension (Fig.4). The original 90 degree angle may or may not change, depending on the fiber orientation.

SAMPLE DATA: For a composite of por-a-mold and three layers of anodized Al:

30 degree fibers	@ 40 lbs.	90 degrees became 97
45 degree fibers	40 lbs.	90 degrees stayed 90

INSTRUCTOR'S NOTES: These composites can be used to demonstrate and study the Rule of Mixtures and Poisson's ratio[4] in addition to calculating percent fiber content by volume and observing deviations due to slight alterations. For example, if a 90 degree fiber composite is trimmed to leave only a few unwanted (0 degree) fibers along the length of one edge, it will torque under tension. However, once the 0 degree fibers are removed, it does not torque. At high forces, slippage and fiber pullout may occur. Also, the kinks in the fibers from being woven might want to be considered.

REFERENCES:

(1) West, H.A.; and Sprecher, A.F.: "Fiber Reinforced Composite Materials," National Educators Workshop: Update '90, NIST Special Publication 822, 1991.

(2) Askeland, Donald R.: The Science and Engineering of Materials, PWS-Kent,Boston, 1970.

(3) Product Bulletin Number CC-91-02, "Basic Techniques For Using Por-A-Mold, Clear-Cut," Synair Corporation.

(4) Agarwal, Bhagwan D.; and Broutman, Lawrence J.: Analysis and Performance of Fiber Composites, Second ed., John Wiley & Sons, Inc., New York, 1990.

SOURCES of SUPPLY: While the aluminum screening can be easily purchased at a hardware store (less than $0.15 sq. ft.), the Ferris see thru can be obtained through a jewelry dealer (approximately $55 per gallon). Unfortunately, por-a-mold is no longer on the market.

ACKNOWLEDGEMENTS: We would like to thank Mr. Howard Moore, Technical Assistant, Physics department, Loyola Còllege, for machining the ferris jig and allowing us the use of his tools. Also, we appreciate the interest and input of Jon Thompson, Technical Assistant, Engineering department, Loyola College, and many others at Loyola College and North Carolina State Univesity.

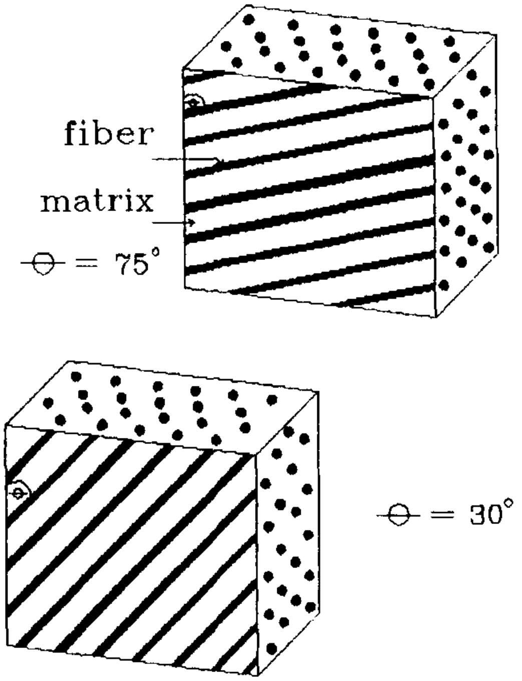


Figure 1. Continuous unidirectional fiber-reinforced composite

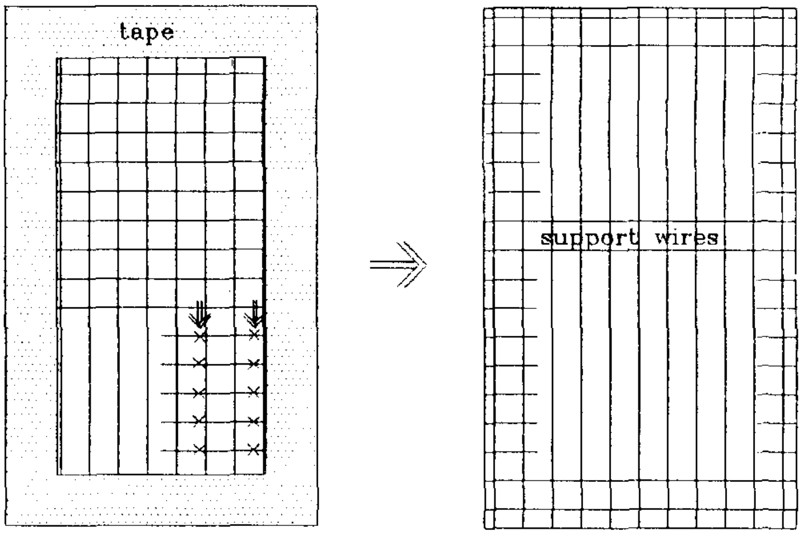


Figure 2. Fiber preparation of Al screening

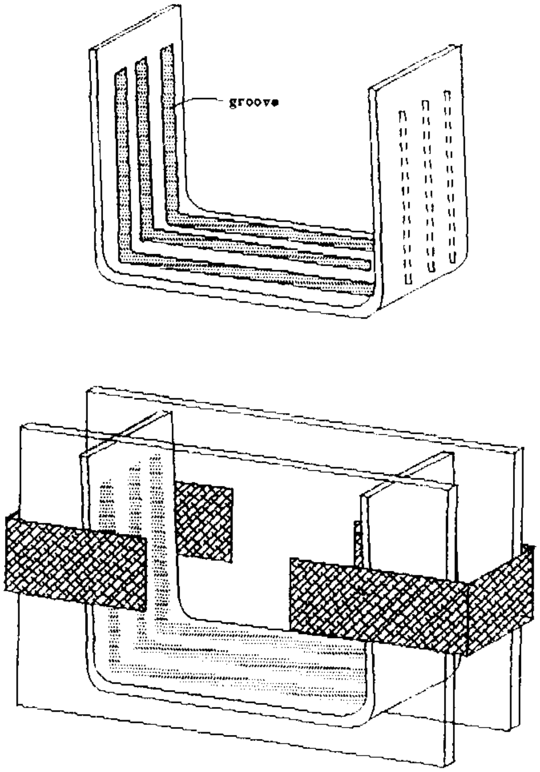
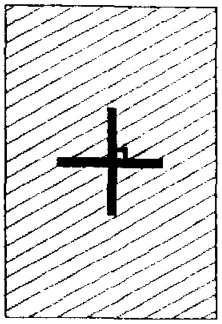
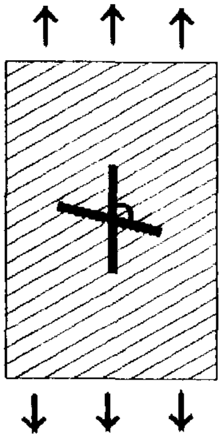


Figure 3. The Ferris Jig



Draw a 90° angle.



The angle may change.

Figure 4. Composite under uniaxial tension

DESIGN AND CONSTRUCTION OF A TENSILE TESTER FOR THE TESTING OF SIMPLE COMPOSITES

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Foreword

Materials testing has been an important part of engineering in America for over one hundred years. In 1832, the Franklin Institute began a systematic investigation into the cause of steam boiler explosions. This study was broken down into two parts. The first part was concerned with the operation of the boilers. The second part of the study, however, was concerned with the material used to construct the boilers. At the time, manufacturers of iron knew that their product was strong, but had no idea of how strong it was. The manufacture of iron was guided by rule of thumb and virtually nothing was measured. This ignorance concerning the production of iron did not have tremendous consequences until the arrival of the steam engine. Boilers proved the inadequacy of some irons by exploding violently. As a result, a method for testing materials was developed and initially executed by Walter R. Johnson, Professor of Mechanics and Natural Philosophy at The Franklin Institute in Philadelphia. This marked the first time that quantitative data on "proving tenacity" was gathered in America.[1]

Materials testing has grown considerably since Johnson's work and is now a major part of engineering. The material strength requirements for a steam engine pale in comparison to the requirements for the machines of today. From high speed electronic switches to space flight, the "boiler explosions" of today are much more costly, both in money and potential loss of life, than they were in Johnson's day. The explosion of the space shuttle Challenger is an example of the potential hazards waiting to happen. A single O-ring, cooled below an acceptable temperature, caused the loss of seven lives and billions of dollars. As a result, the strength and characteristics of a material must be fully understood before that material may be used in the manufacture of a product.

The following is a design for a tensile tester which will be used to test the tensile strength and anisotropic properties of simple composites. These simple composites are suspected to be anisotropic primarily in a single plane. When the composites undergo a tensile force, they will undergo deformation, causing movement either to the left or right. The composites are suspect due to their method of construction. Each sample has a single layer of unidirectional continuous fibers embedded in a rubbery resin. It has been well established that a serious limitation of unidirectional fiber composites is the very large in-plane anisotropy.[2]

The design presented here incorporates a single degree of freedom such that distortion (to the left or right) due to anisotropic tendencies may be measured. The device will spend the vast majority of its time in an undergraduate materials lab. As a result, ease of use and durability are valued more highly than research grade accuracy. Additional concerns focus on the fact that this machine will be built as a student project.

Issues which are dealt with during this design include:

1. Specimen configuration or shape.
2. Method of applying consistent, linear tension force.
3. Method of gripping specimen without affecting its overall properties.
4. Method of collecting data.
5. Repeatability of data.
6. Ease of use.
7. Ease of construction.
8. Cost.

After the device has been constructed, it will be used to test the simple composites which were fabricated in house. A comparison will be made between composites manufactured using aluminum screening as the strengthening fibers and those manufactured using fiberglass screening.

Design

Application of Force

The first matter which will be taken up is the application of force. A device is needed which can provide a consistent, linearly applied force, but still allow for lateral movement of the sample so that anisotropic properties as well as the modulus of elasticity and yield strength may be observed and measured. In addition, the machine must provide for an easy method by which several characteristics can be measured. These characteristics include:

1. Elongation
2. Lateral displacement as a result of anisotropic behavior.
3. Force applied.

A hand operated screw mechanism was chosen for the application of force. This provides a simple means of applying force in a single direction. In addition, the amount of force applied may be increased in a linear fashion with infinitesimal changes. The screw chosen was an Acme® threaded screw, one inch in diameter with a pitch of ten threads per inch. The Acme® thread is a square cut thread and is designed to support large amounts of force. This thread is commonly used in such high pressure applications as vices and large clamps. For each rotation of the screw, the sample will be elongated by one tenth of an inch. This provides a convenient point at which to take a reading of the force. As a result, measurements of force are taken every one tenth of an inch and the number of readings taken prove an accurate guide for the total elongation.

The screw is operated by means of a large hand wheel. The large wheel will provide leverage so that an adequate amount of torque may be easily applied to the screw which in turn will impart a large tensile force to the specimen which may be easily controlled by the operator.

In addition to being able to impart a tensile force to the sample, the screw must be able to support itself. This was accomplished by using a twenty inch long section of screw and turning down a one inch section on each end to 0.627" so that it would fit into two, flanged bushings. Figure 1 shows the hand wheel. Tensile force will be applied when the knob is turned clockwise. Most people associate a clockwise rotation of threaded objects with an application of force and hence operation of the wheel will be of second nature. The Acme® threaded rod appears in figure 2. Figure 3 shows the assembled screw mechanism. The screw and hand wheel are attached by means of a roll pin. A hole was drilled through the hand wheel and rod and the pin was then inserted.

Support and Load Transfer

Next to be considered is the frame of the machine. This is an important part since the frame not only supports the machine but facilitates the transfer of the load from the screw mechanism to the sample. In addition, it allows for lateral movement of the sample during testing. The frame is also the heart of the information gathering process. A load cell is mounted on the frame to allow for the measurement of the force being applied to the sample. The output from the load cell is read from a Fluke digital multimeter in milli-volts. Through information provided by the manufacturer as well as use of a standard, the relationship between the load cell's output in milli-volts to the actual pounds of force delivered was determined.

The support part of the frame was constructed from 1"x3" sections of maple (Note: These are mill dimensions). Maple was chosen because of its high strength and its beauty. The drive beam was constructed from oak. Maple would have been preferred, but a large enough piece was not available. The properties of these two woods appear in table 1 (all figures refer to forces applied parallel to the grain). Poplar is included in the table since it is of sufficient strength to use in the construction of the frame and drive beam. In addition, relatively large poplar beams (4"x4") are readily available and poplar is less expensive than maple or oak. Unfortunately, poplar has a yellowish green tint that renders it much less pleasing to the eye than either oak or maple. One should never neglect the aesthetics of a design.

Wood	**Specific Gravity**	**Modulus of Elasticity [lb/in^2 x 10^3]**	**Modulus of Rupture [lb/in^2]**	**Compressive Strength [lb/in^2]**
Maple, sugar	0.676	1,830	15,500	7,800
Oak, white	0.710	1,770	15,100	7,440
Poplar, yellow	0.427	1,500	9,200	5,540

Table 1: Properties of selected hard woods.[3]

The frame was constructed by using carpenter's glue to laminate five pieces of maple together to form four beams. The sections were offset such that finger joints were formed at the corners. These beams were clamped tightly and the glue was allowed to dry. After the glue had dried, holes were drilled as required using a drill press and the appropriate bits. Figure 4 shows the construction of the support beams. The drive beam appears in figure 5. All parts must be assembled within the frame before any final gluing can take place. This includes two steel rods, as well as the drive beam itself and the Acme® rod with hand wheel in place. Once assembled, the frame was measured to check for squareness. It was racked into shape by use of large wood working clamps which mount on standard one inch diameter black pipe. Once the frame was squared, four 1/2" holes were drilled at each corner and four 1/2" hardwood dowels which had been coated with glue were driven in place using a hammer. The resultant appears in figure 6 along with the two drive rods, rail assembly and mounted load cell.

The force from the drive beam is delivered to the sample by means of two, 36" long steel rods, 1" diameter. These drive rods support a rail and shuttle system which actually allows for the single degree of freedom. The rods have holes drilled in them such that the rail and shuttle system may be placed at different heights, thereby allowing for different length samples or grips (Figure 7).

A section of rail that was used appears in figure 8. The guide rail was commercially available in 6'-6" lengths. The rails were cut into four, 1'-3" sections and doubled as reinforcement to prevent any deformation during loading. The rails must remain straight if any anisotropic behavior is to be observed. If the rails bow, the shuttle system will remain in the trough of the bow. Figures 9 and 10 show how the rail assembly is constructed.

Grips and Shuttle system.

The grips were machined from aluminum and are straightforward in design. Each grip basically consists of a center, mounting plate and two gripping plates. The two gripping plates are fastened to either side of this center plate by means of four, knurled nuts. These nuts have four small holes on the knurled surface that allow for the insertion of a taper pin. This provides additional leverage in tightening the grips. The gripping plates were designed to be the exact same dimension as the sample to be tested. As a result, the plates alone are unable to grip the sample. This was done in order to minimize sample deformation from metal to composite clamping as this might affect the test. A thin sheet of rubber (obtained from the plumbing section of a hardware store) was attached to the face of the gripping plates using Duro® contact cement. This allowed the clamping surface to conform somewhat to the sample as opposed to pinching it. To provide additional gripping ability, pieces of 3M's Press'n Sand® "Sticky back" sanding sheets were cut to size and affixed to the rubber faces. The gripping plates are the same for the top and bottom (Figure 11). The bottom grip (Figure 12) screws into the load cell (Figure 13).

The top grip consists of two gripping plates identical to those on the lower grip. These plates mount on a shuttle system. The shuttle system consists of a center plate and four guide wheels. The guide wheels were obtained from the same company which supplied the guide rail. The unassembled shuttle system appears in figure 14.

Some comments concerning the machine's operation might prove helpful in improving on the design. The grips were insufficient in holding the samples without the sandpaper faces, and even then loads peaked at a maximum of just over 100 pounds before the samples began to pull free from the grips. Also, the sandpaper faces slipped during tests and had to be replaced periodically. A better idea might be to alter the design to accept commercial grips. This may not alleviate all the problems. It was noticed that when a series of samples 1" wide were tested (to match the 1" wide samples being run on the Chatillon LDX tensile tester since its grips could accommodate nothing larger) anisotropic tendencies did not appear consistently. The 2.5" wide samples, however, displayed excellent anisotropic tendencies. Still, the commercial grips might be capable of delivering enough of a load to a 1" sample such that its anisotropic characteristics are revealed.

Calibration and Verification of Devices Functioning

The load cell was connected to a Fluke digital multimeter as per manufacturer's instructions. Using the load cell's specifications sheet, a relationship was determined between the load cell's output in milli-volts and the corresponding force in pounds. The relationship between the load cell's output in milli-volts and the tensile force applied was calculated to be:

$$Lbf = 998.84\frac{Lbs}{volt} \times v \qquad \text{Equation 1}$$

Lbf = pounds force
v = volts

The first problem was that of collecting the data. After each full turn of the hand wheel (equivalent to an elongation of one tenth of an inch) the reading was taken from the multimeter and written down. In the time taken to write down the data, however, the sample had relaxed substantially. To prevent this, a small tape recorder was used to record data while the hand wheel was turned in a slow, constant fashion. This eliminated the problem of relaxation during data collection. Two people working together, as in an undergraduate lab, could also overcome the problem with one student turning the hand wheel and calling out the readings while the other student jots down the data.

The next problem encountered was the inaccuracy of the machine's output. Like samples of Ferris See-thru® (Neat, 0.3"x1"x5-3/4") were tested on the hand operated device and on a Chatillon LDX 500 pound capacity tensile tester. The data was off by an amount considered unacceptable (Figure 15). Two things are readily apparent in viewing figure 15. The first is that the hand operated device is reading ten pounds high (assuming the just purchased Chatillon was correct). The second is that the slopes, or moduli of elasticity, are similar. It was suspected that since the samples were being subjected to less than 100 pounds force, that the 5000 pound capacity load cell simply was not in its most effective range. A small, 50 pound capacity hand held spring scale (similar to a "fish scale") was used to check this. First, the scale was checked for accuracy using the Chatillon. While the scale's readings fluctuated high and low with respect to the Chatillon's output, a series of ten trials running from zero to forty-four pounds yielded an average that was reasonably accurate (Figure 16). The scale was then attached to the hand operated device. Ten sets of readings were taken ranging from zero to forty-four pounds. The data was averaged, zeroed and plotted (Figure 17). The relationship between the load cell's output in milli-volts and pounds force was determined to be:

$$Lbf = \frac{mV - mV_0}{0.963} \qquad \text{Equation 2}$$

Figure 18 shows the comparison of the data from figure 15 using Equation 2. The fit is still not perfect, but it is closer. In addition, the two plots in figure 18 are closer to being parallel at values above 3/4 pounds. The modulus of elasticity was determined by determining the slope of a hand fitted line. The results are as follows:

$$\text{Chatillon:} \quad \frac{(24.62-0)Lbf}{(2-0)inches} = 12.31\frac{Lbf}{inch}$$

$$\text{Hand Operated:} \quad \frac{(29.69-10.46)Lbf}{(2-0.5)inches} = 12.82\frac{Lbf}{inch}$$

$$\text{Difference:} \quad \frac{(12.82-12.31)}{12.31} \times 100 = 4.14\%$$

It is suspected that this small amount of error could be eradicated if a 500 pound load cell were used in the construction of the hand operated tensile tester. Unfortunately, none were available during the course of this project.

Samples

Many samples were tried during the course of working with this device. One inch wide samples were attractive at first because six were obtained from a single mold. Also, the Chatillon LDX's grips accepted nothing larger than a 1" sample. Unfortunately, 1" samples were very difficult to hold, slipping from the grips with as little as 40 pounds force. Also, the narrow span of fibers made the viewing of anisotropic properties difficult. Lateral displacement in samples which were obviously anisotropic was hit and miss. Data obtained from 1" samples will be ignored for the most part since like samples failed to respond in a consistent fashion.

Finally, 2.5" wide samples were used. The grips had originally been designed to hold samples of this size. The additional gripping surface proved invaluable, though slippage remained a problem throughout the tests. In addition, anisotropic properties were much more pronounced with the larger span of fibers. The bulk of data obtained from the 2.5" samples appears in the following brief discussion of tests.

Tests

The following is a series of eleven tests which were run on the completed and calibrated device. The composites which were tested were developed by M. D. Wampler and F. X. Spiegel of Loyola College, Maryland.[4] The original concept was modified by constructing a divider which allowed for two, 0.3"x2.5"x5.75" samples to be constructed at a time. Aluminum and fiberglass screening were used for the strengthening fibers. Strands of screen were selectively removed to produce composites with continuous unidirectional fibers at different angles to the horizontal (0°, 30°, 45°, 60° and 90°). The bits of screen were removed using either a fine pair of scissors or a modeling knife. A single layer of fibers was used for each sample.

Sample test numbers consist of the date and the order in which the tests were performed on that date. The samples used were 0.3"x2.5"x5.75". Two plots accompany each test. The first plot is of pounds force versus elongation. The modulus of elasticity was calculated from these. The second plot is of lateral displacement versus elongation. If a sample is anisotropic, a shear strain will be produced when a tensile stress is applied.[5] The shuttle system used in supporting the top grip allows this shear stress to be expressed in movement either to the left or right.

Test #0716931 - Ferris See-thru® Neat (Figures 19 & 20).
Test #0716932 - Ferris See-thru® with aluminum fibers at 0° (horizontal) (Figures 21&22)
Test #0716933 - Ferris See-thru® with aluminum fibers at 30° (Figures 23 & 24).
Test #0716934 - Ferris See-thru® with aluminum fibers at 45° (Figures 25 & 26).
Test #0716935 - Ferris See-thru® with aluminum fibers at 60° (Figures 27 & 28).
Test #0716936 - Ferris See-thru® with aluminum fibers at 90° (vertical) (Figures 29 & 30).
Test #0716937 - Ferris See-thru® with fiberglass fibers at 0° (horizontal) (Figures 31 & 32).
Test #0716938 - Ferris See-thru® with fiberglass fibers at 30° (Figures 33 & 34).
Test #0716939 - Ferris See-thru® with fiberglass fibers at 45° (Figures 35 & 36).
Test #07169310 - Ferris See-thru® with fiberglass fibers at 60° (Figures 37 & 38).
Test #07169311 - Ferris See-thru® with fiberglass fibers at 90° (vertical) (Figures 39 & 40)

None of the samples tested were brought to failure. In the force versus elongation plots, the drop off is due to the sample slipping from the grips. While the force existing as the sample slips from the grips is not important, what is important is the lateral displacement. Figures 26, 28, 30, 34, 36 and 38 clearly show the samples being laterally displaced as tension is applied. As the samples slipped from the grip, the lateral displacement reversed itself, with the samples moving back towards their original position. This data was retained since it further demonstrated the anisotropic properties of these composites.

Results

0716931 - Neat:

Modulus of Elasticity = $\frac{(48.22-20)Lbf}{(1.6-0.6)inch} = 28.22\frac{Lbf}{inch}$

The neat sample showed no surprises. It did not experience any permanent deformation, returning to its original dimensions after having slipped from the grips. The results appear in figures 19 and 20.

Aluminum

0716932 - Al fibers at 0° (Horizontal):

Modulus of Elasticity= $\frac{(58.57-0)Lbf}{(1.6-0)inch} = 36.60\frac{Lbf}{inch}$

The sample experienced some permanent deformation. Its final dimensions were 0.30"x2.42"x5.8". The aluminum wires were protruding from the sides of the sample as a result of the samples necking while under tension. It is believed that an uneven slippage rate of the aluminum wires caused the minimal amount of lateral displacement that was witnessed. The results appear in figures 21 and 22.

0716933 - Al fibers at 30°:

Modulus of Elasticity = $\frac{(62.57-0)Lbf}{(2-0)inch} = 31.29\frac{Lbf}{inch}$

The sample experienced some permanent deformation. Its final dimensions were 0.30"x2.48"x5.73". The aluminum wires were protruding from the sides of the sample as a result of the necking that occurred. In addition, the sample retained the shape of a parallelogram with an offset angle of 1°. The sample was originally 90° at the corner but has deviated to 89°. The results appear in figures 23 and 24.

0716934 - Al fibers at 45:

Modulus of Elasticity = $\frac{(59.43-0)Lbf}{(1.6-0)inch} = 37.14\frac{Lbf}{inch}$

The sample experienced permanent deformation. Its final dimensions were 0.30"x2.42"x5.80". The aluminum wires were protruding from the sides of the sample due to the necking which occurred while the sample was under tension. In addition, the sample overcompensated, returning slightly past its zero lateral displacement and retaining the shape of a parallelogram with an offset angle of 2.5° (87.5° at the corner). The results appear in figures 25 and 26.

0716935 - Al fibers at 60°:

Modulus of Elasticity = $\frac{(42.86-0)Lbf}{(0.75-0)inch} = 57.15\frac{Lbf}{inch}$

The sample experienced severe permanent deformation. Its final dimensions were 0.30"x2.40"x60". The aluminum wires were not protruding from the sides of the sample but there was a substantial amount of pull-out visible through the clear resin. In addition, the sample severely over compensated. As it slipped from the grips, it moved towards zero lateral displacement. It overshot, however, and came to rest -0.200" away from zero deviation. Its offset angle was 11° (79° at corner). The results appear in figures 27 and 28.

0716936 - Al fibers at 90° (Vertical):

Modulus of Elasticity = $\frac{(94.55-0)Lbf}{(0.2-0)inch} = 472.73\frac{Lbf}{inch}$

The sample experienced severe permanent deformation. Its final dimensions were 0.30"x2.40"x6.00". The aluminum wires had suffered pull-out and were protruding from the broad flats of the sample. The lateral movement was caused by slack in the wires. This slack arose during fiber orientation during flow.[2] When the liquid Ferris See-thru® was poured over the fibers, they shifted position and took on a bowed shape. As the curve was pulled out of the bow, the sample shifted laterally. The results appear in figures 29 and 30.

Analysis

In measuring the modulus of elasticity, a line was hand fitted to each plot. An attempt was made to include only points which were measured before any slippage occurred. The modulus of elasticity is directly related to the stiffness of a material.[6] A high modulus of elasticity indicates that a large amount of force is required to elongate a specimen. The results obtained here showed that the sample with vertical fibers (90°) had the highest modulus of elasticity. This was expected since the aluminum fibers receive the entire load. It would have been expected that the composite with horizontal fibers (0°) would have had the lowest modulus of elasticity. This was not the case, according to the results. The sample with fibers at 30° had the lowest at 31.29 *Lbf/inch* as compared to 36.60 *Lbf/inch*. The validity of this result is in question, but the percent error which was calculated during calibration does not account for it. The tendency of the samples to slip, however, would account for it.

The neat sample had the lowest modulus of elasticity of all. This was expected, since the chore of fibers within a composite is to provide strength while the resin's job is to support and protect the fibers.

It is suspected that the overcompensation displayed by the 45° and 60° aluminum was due to pull-out. Pull-out occurs when the fibers pull loose from the matrix.[2] Since the aluminum fibers are held in place by mechanical adhesion (mechanical interlocking of two surfaces) the bond between fiber and matrix is a weak one.[2] The hole left due to the pull-out of a fiber would neck down under tension, thus increasing the mechanical adhesion of the fiber. As the tension is released, the fiber does not slide entirely back into the hole from which it came. If this were the case, the array of aluminum fibers would now push the matrix in the opposite direction. This would explain the over compensation and the deformation which was so prominent in the 60° sample. Figure 41 shows a schematic of the distortion which took place in the aluminum fiber samples.

Fiberglass

0716937 - Fiberglass fibers at 0° (Horizontal):

Modulus of Elasticity = $\frac{(53.43-8.86)Lbf}{(1.6-0.2)inch} = 31.84\frac{Lbf}{inch}$

The sample experienced no permanent deformation. The fiberglass appears to have formed a more secure bond with the Ferris See-thru®. This was demonstrated by a lack of protrusion of fibers which was prevalent in the 0° with aluminum. The results appear in figures 31 and 32.

0716938 -Fiberglass fibers at 30°:

Modulus of Elasticity = $\frac{(55.43-10.86)Lbf}{(1.2-2)inch} = 44.57\frac{Lbf}{inch}$

The sample experienced some permanent deformation. Its dimensions remained the same, but it was offset by 1° (89° at the corner). Again, the fiberglass appears to have a more secure bond with the Ferris See-thru®. There was virtually no protrusion of fibers from the resin. The results appear in figures 33 and 34.

0716939 - Fiberglass fibers at 45°

Modulus of Elasticity = $\frac{(49.86-28.12)Lbf}{(0.8-0.4)inch} = 54.35\frac{Lbf}{inch}$

The sample experienced some permanent deformation. Its dimensions remained the same, but it was offset by 3° (87° at corner) due to over compensation. Again, the fiberglass appears to have a more secure bond with the Ferris See-thru®. This is demonstrated by a lack of protrusion of fibers which was more pronounced in the 45° with aluminum. The results appear in figures 35 and 36.

07169310 - Fiberglass fibers at 60°

Modulus of Elasticity = $\frac{(60-13.14)Lbf}{(0.8-0.2)inch} = 78.10\frac{Lbf}{inch}$

The sample experienced some permanent deformation. Its dimensions changed to 0.30"x2.42"x5.78". There was not as much offset due to over compensation as might be expected, only 2° (88° at corner). This is probably due to the comparably tenacious bond that the fiberglass fibers seem to have with the resin. The results appear in figures 37 and 38.

07169311 - Fiberglass fibers at 90°

Modulus of Elasticity = $\frac{(84.35-0)Lbf}{(0.2-0)inch} = 421.74\frac{Lbf}{inch}$

The sample experienced no permanent deformation, despite having experienced stresses in excess of one hundred pounds (by far the heaviest). The sample's dimensions remained the same and the fiberglass fibers showed no signs of pull-out. As with the aluminum at 90°, the lateral movement here is a result of fiber orientation during flow. The results appear in figures 39 and 40.

Analysis

The modulus of elasticity for the fiberglass composites behaved as expected with the 0° having the lowest modulus and the 90° having the highest. The composites whose fibers had higher angles of orientation had correspondingly higher moduli. This is as expected.

The fiberglass fibers form a bond with the resin that is far superior to the bond formed between the resin and aluminum fibers. The bond is still due to mechanical adhesion, but the fiberglass fibers have more surface rigosity. In addition, there are small "tabs" that remain on either side of the unidirectional fibers after the superfluous middle fibers are snipped free. This superior bond results in less sample deformation. Pull-out was virtually eliminated.

Overall, the fiberglass fibers proved superior to the aluminum fibers. Both pull-out and sample deformation were minimized with the use of fiberglass fibers. The aluminum was the stronger fiber, as evidenced by the modulus of elasticity for aluminum fibers arranged vertically. At 472.73 *Lbf/inch*, it was 12% higher than the modulus of elasticity for the corresponding fiberglass sample. The aluminum sample, however, emerged from the test severely deformed while the fiberglass sample appeared unscathed.

Closing Comments

While the device designed and constructed during this course is useful, many improvements could be made to increase its usefulness as an educational instrument. Below are several suggestions.

1) The addition of two LVDT's, one to measure lateral displacement and one to measure necking.
2) The addition of a device which could collect data from the load cell, convert it to pounds force and store it.
3) A redesigned grip, based on the commercial grips used by Chatillon, but capable of holding 2.5" wide samples.
4) Replace the 5000 pound capacity load cell with a 500 pound capacity load cell.

These are only suggested modifications. In its current state, the device is simple to learn and provides reasonable results. The best results are obtained when people work in teams of two or three. This is especially the case when lateral displacement readings are being made. One person operates the hand wheel, reading off the voltages to a note taker. As each reading is taken, a third student can make measurements of the lateral displacement by means of a small ruler clipped on the rail assembly.

Acknowledgments

Finally, in closing, we would like to thank all those who contributed time and advice during the design, construction, and testing of this device: special thanks to Walter Krug and Mike Francoviac, who were so helpful with advice concerning the machining of the grips; Bob Borst, whose woodworking tools and skill were invaluable; John Ward, who aided in the actual operation of the completed device. We could not have accomplished so much in so little time without their help.

<u>List of materials used in the construction of this device.</u>

1. Machinist's Labor (approximately 40 hours) $1000
2. Guide Rail A7C15-3065, one 6.5' section at $55.60 from Stock Drive Products
3. Guide Wheel A7Q16-2, four at $25.51 from Stock Drive Products
5. Flanged Bushing - 0.627" inner diameter, O.D. 0.878", Length 1", two at $2.04 from Stock Drive Products, A 7B 4-SF202808

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6. Alloy Steel Fully threaded rods, 1/2" -13, one at $4.78 McMaster-Carr, 98957A636
7. Alloy Steel Fully threaded rods, 1/2"-20, one at $8.75 McMaster-Carr, 92580A111
8. Round Drill Rod, 1/4", 3' long, one at $2.29 McMaster-Carr, 8893K36.
9. Steel Rod, 3/8" dia., 12" length, one at $5.95 McMaster-Carr, 6061K32
10. Guide Rod - 1" diameter, 20" length, two at $8.00 from McMaster-Carr
11. Load Rod - 1" diameter, 36" length, two at $12.00 from McMaster-Carr
12. Acme® threaded rod - 1" - 10 threads, 6' length, one at $55.00 from McMaster-Carr, 98935A219

13. Acme® Carbon Steel Flange - 1" -flange dia. 2.760", mounting hole dia. 0.266", from McMaster-Carr, 95082A644, $28.00

14. Acme® Bronze round nut, 1" - 10 threads, outer dia. 1.5", length 1.5" McMaster-Carr, 95072A116, $28.00

15. Knurled nut, 1/2"-13, four at $3.58 McMaster-Carr, 94775A033

16. Cast Iron Hand Wheel - 10" outer dia., 2-1/4" tall, 2-1/4" dia. cntr. 1" dia. grip, McMaster-Carr, 6025K14, $29.38

17. Lumber and other materials obtained from hardware stores, $100.00

Total cost of construction = $1478.19

Suppliers:

McMaster-Carr Supply Company
Dayton, New Jersey
TEL. (908) 329-6666
FAX. (908) 329-3772

Stock Drive Products
Box 5416
New Hyde Park NY 11042-5416
(516) 328-3300

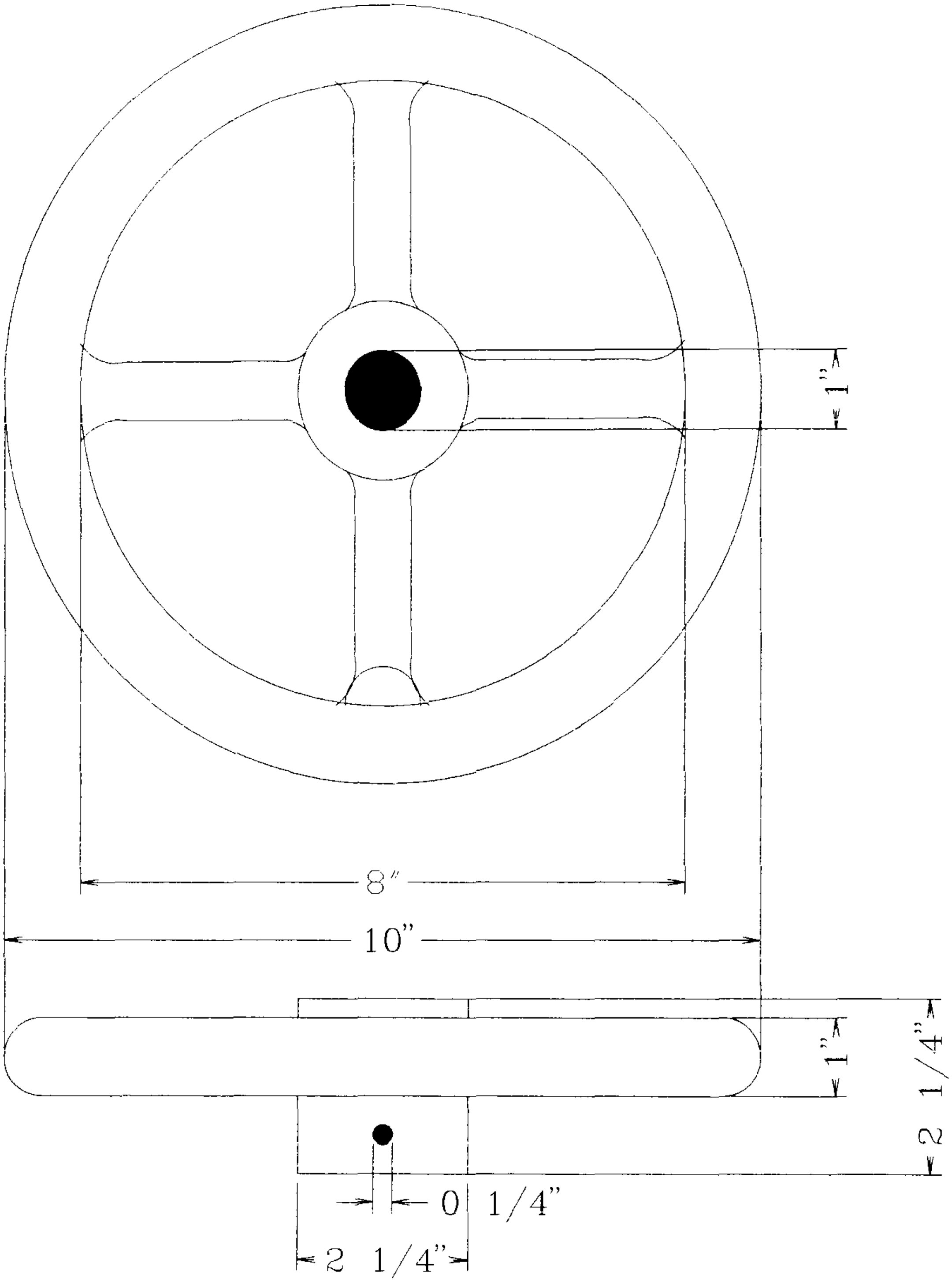
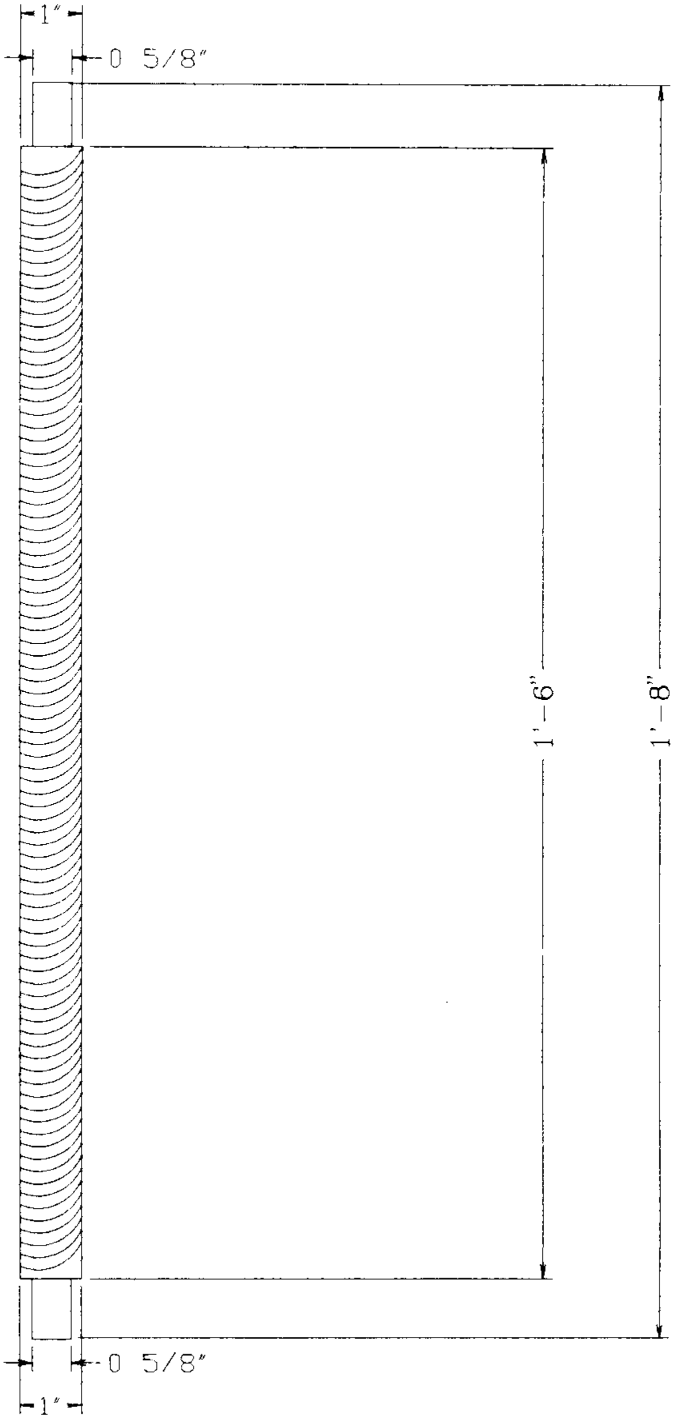


Figure 1. Hand wheel with through holes for Acme rod and roll pin.



Drive rod (1"-10) with ends turned down to 5/8".

Attach to hand wheel with roll pin four inches from bottom.

Figure 2. Acme threaded rod with ends turned down.

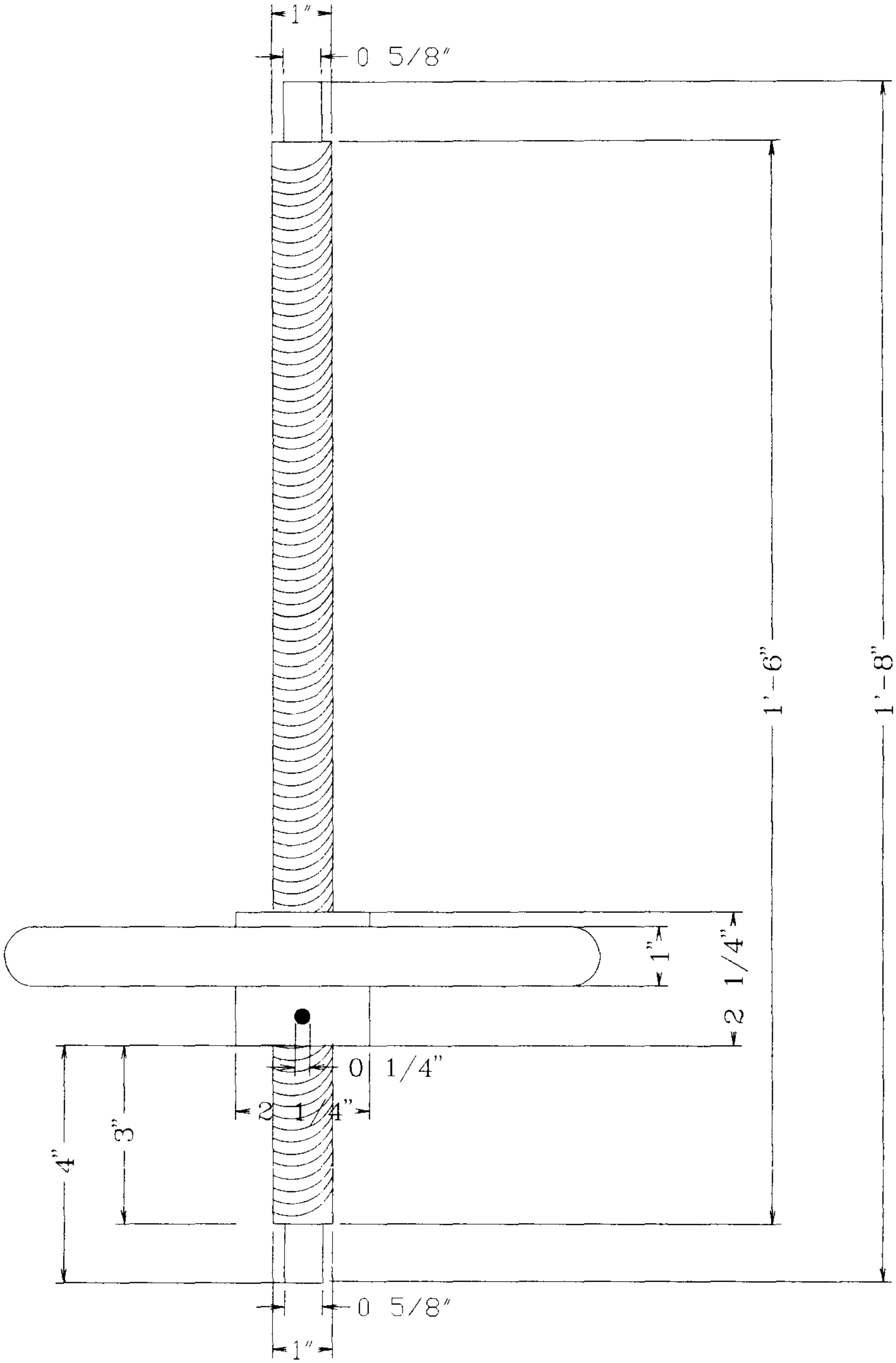
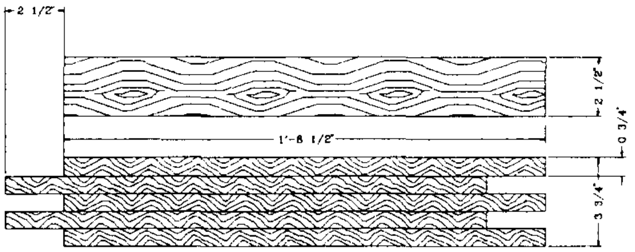
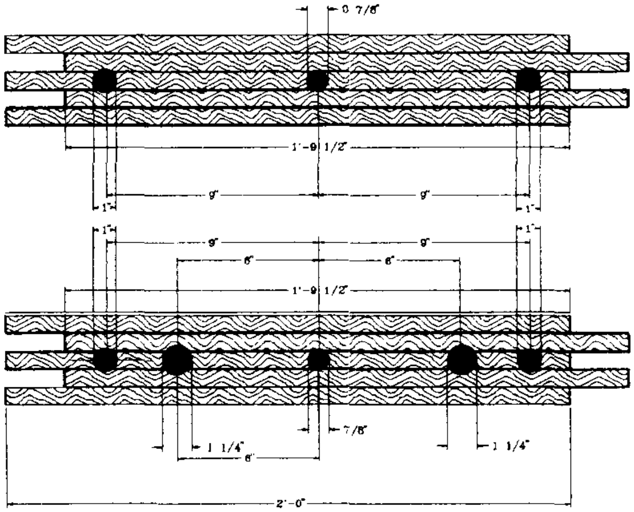


Figure 3. Acme threaded rod with hand wheel.

Laminated maple beam. Construct two horizontal beams with dimensions at right. Construct two vertical beams with the dimensions below.

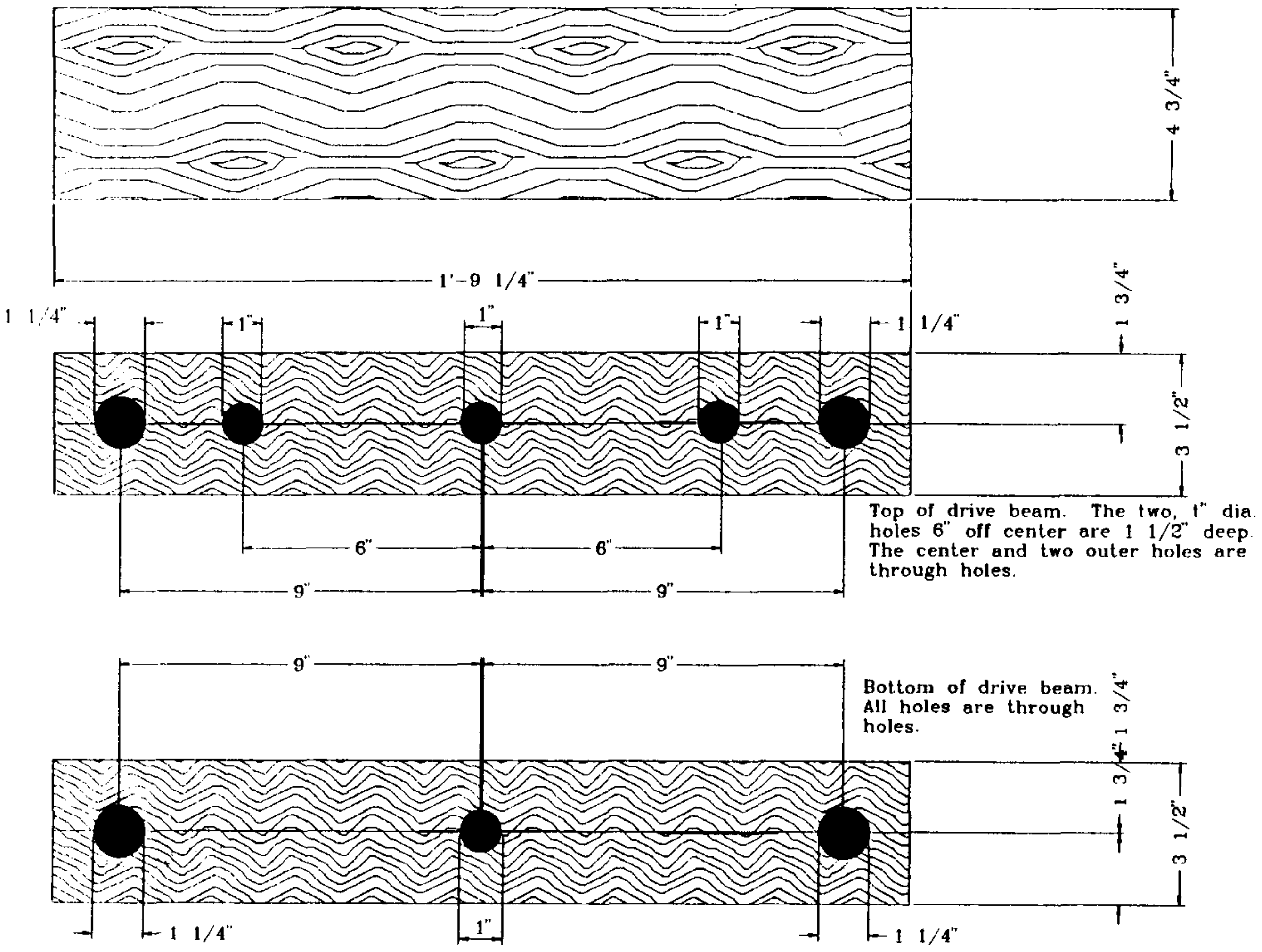




Top side of lower horizontal beam. All three holes are only 1" deep. The outer holes accept the 1" dia. guide rods while the center hole accepts the bushing which the Acme screw will ride in.

Under side of upper horizontal beam. The two outer holes, in addition to the center hole, are the same as on the lower, horizontal beam. The two 1 1/4" dia. holes that are 6" off center go all the way through the beam, allowing for passage of the two drive rods.

Figure 4. Construction of laminated maple beams.



Top of drive beam. The two, 1" dia. holes 6" off center are 1 1/2" deep. The center and two outer holes are through holes.

Bottom of drive beam. All holes are through holes.

Figure 5. Construction of laminated oak drive beam.

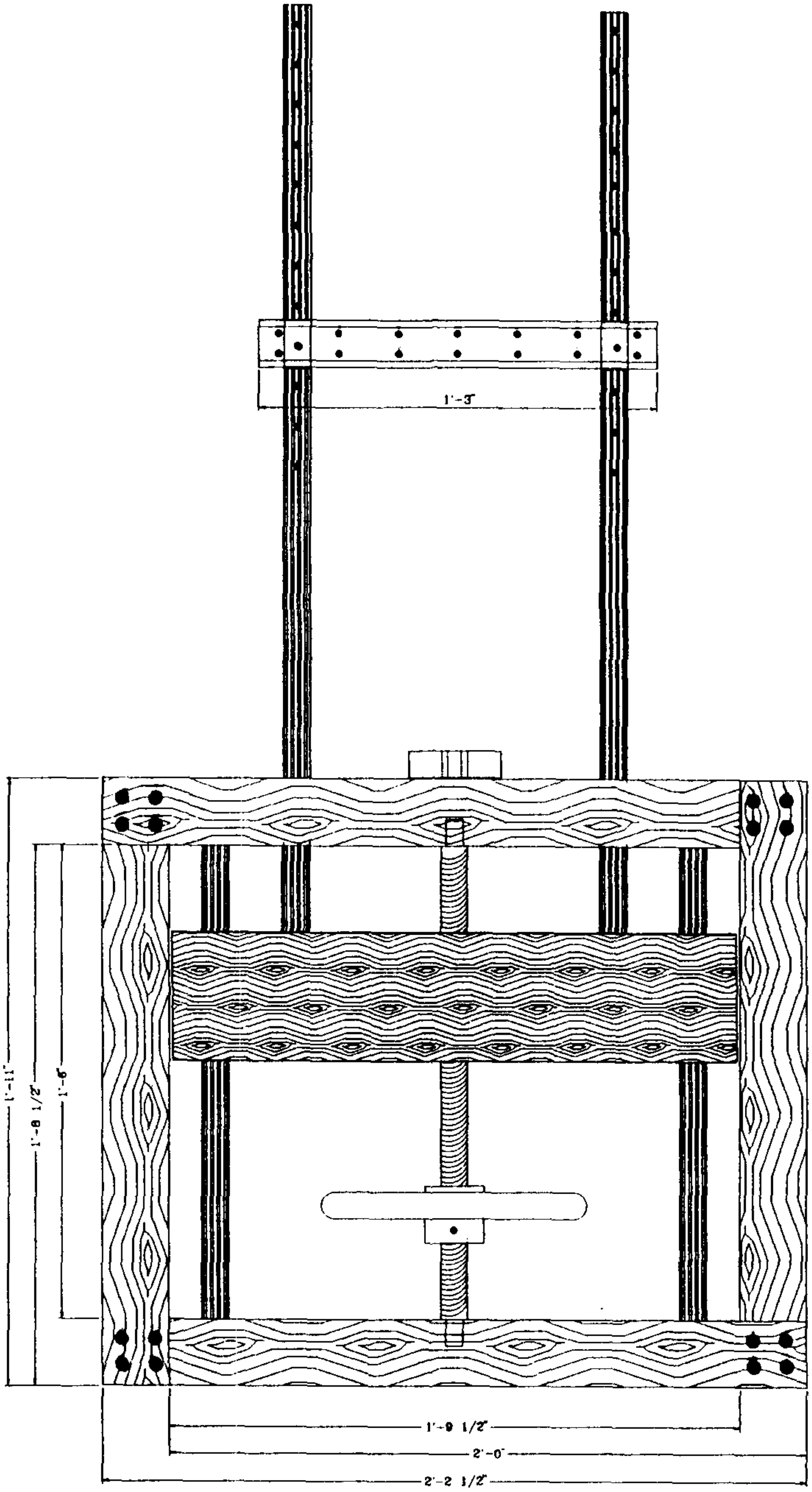


Figure 6. Maple frame with oak drive beam and Acme threaded rod. Two 1" diameter drive rods go up to the rail. The upper grip hangs from the rail while the lower grip screws into the load cell which is attached to the top of the frame by means of lag bolts.

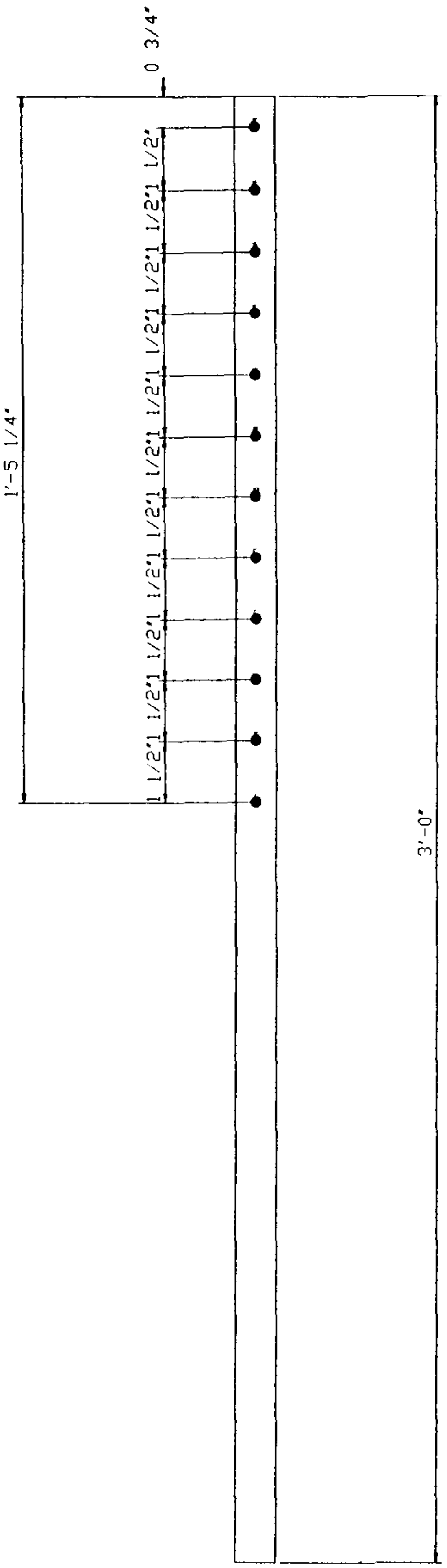


Figure 7. Drive rod (2) with drilled holes to accommodate 1/4" support pin.

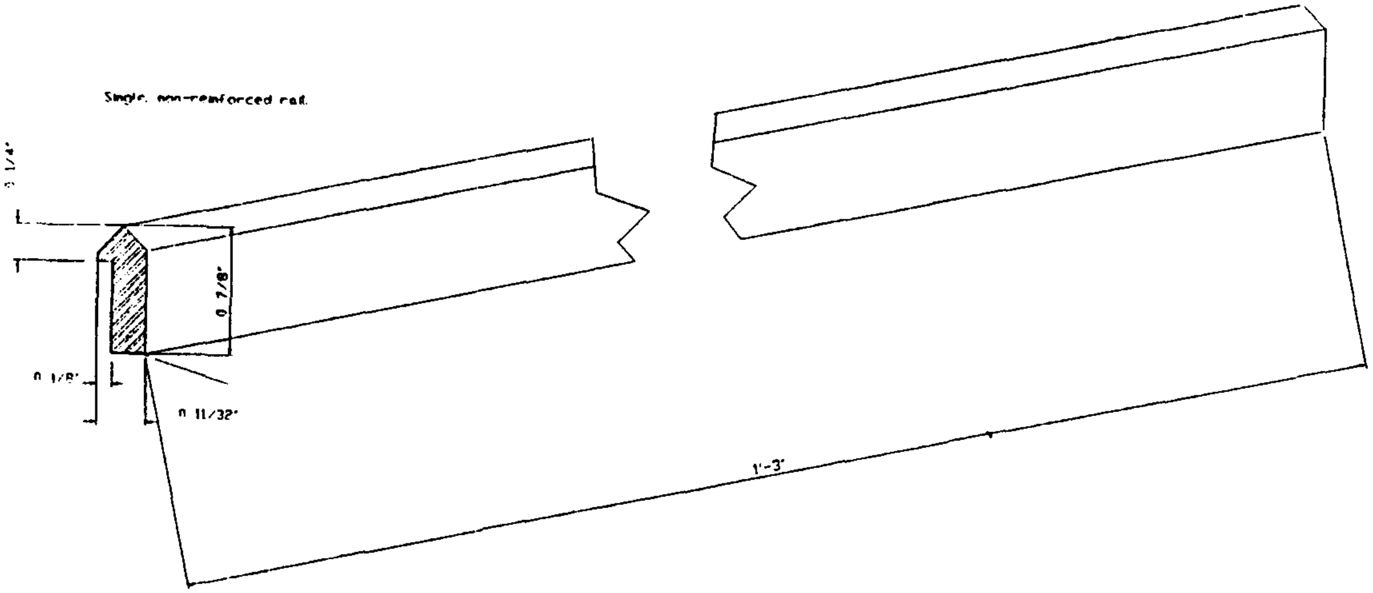


Figure 8. Single rail. Four are required.

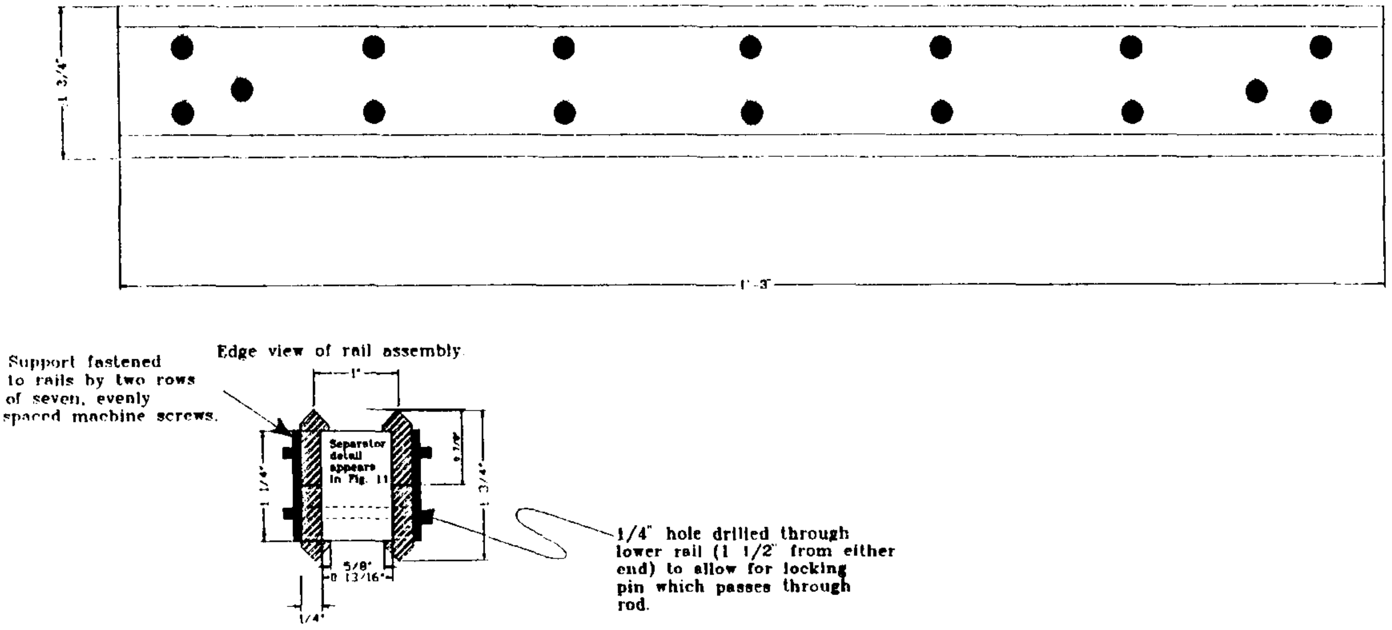
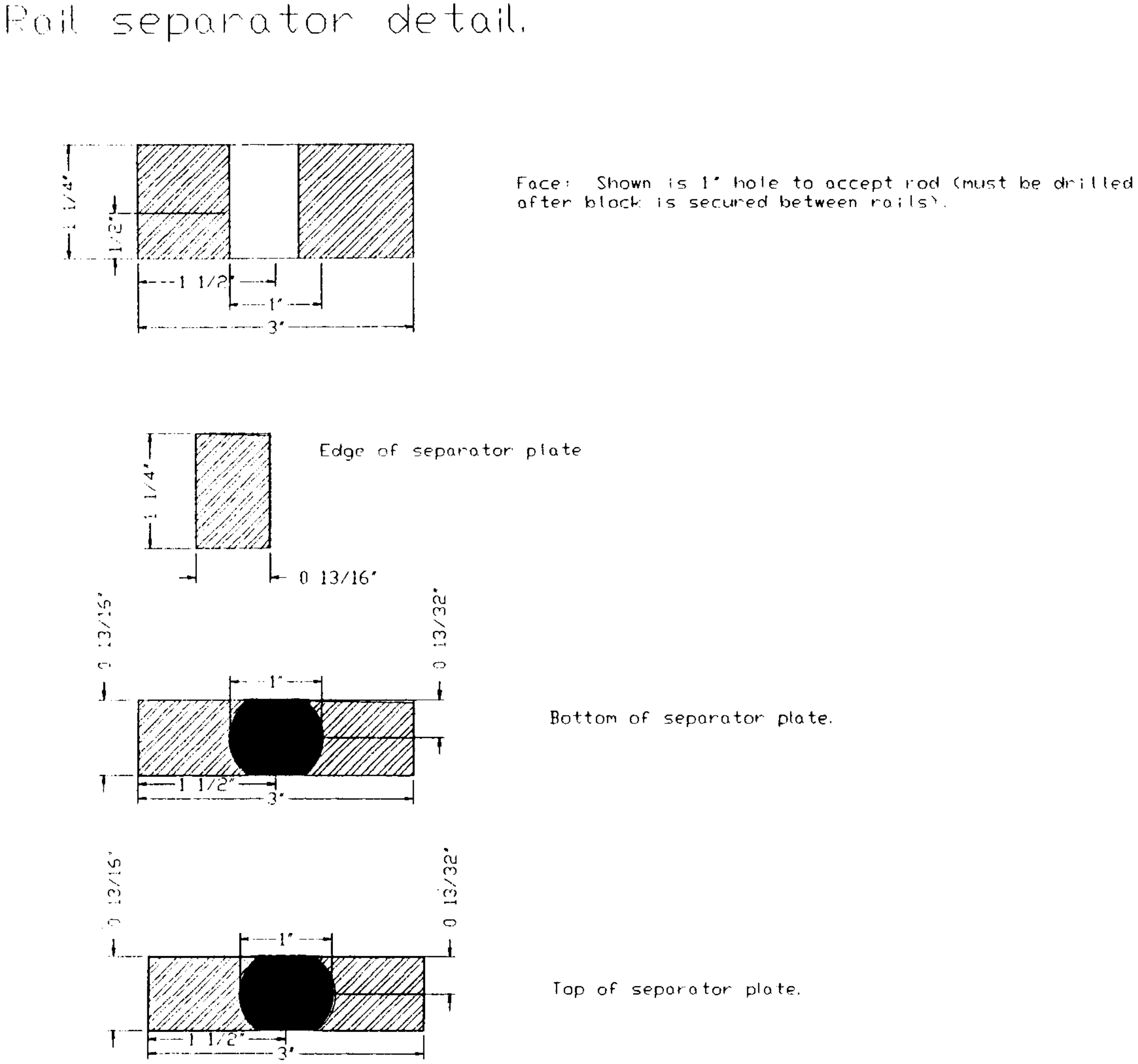


Figure 9. Rail assembly.

Rail separator detail.



Face: Shown is 1" hole to accept rod (must be drilled after block is secured between rails).

Edge of separator plate

Bottom of separator plate.

Top of separator plate.

Figure 10. Detail of rail separator.

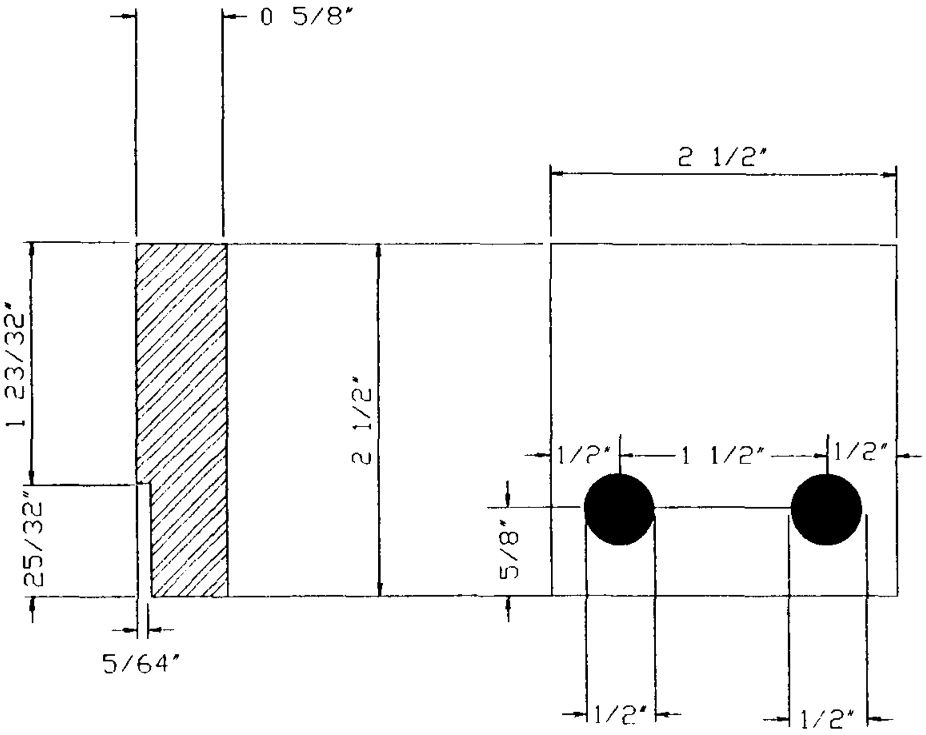


Figure 11. Gripping plate. Four are required.

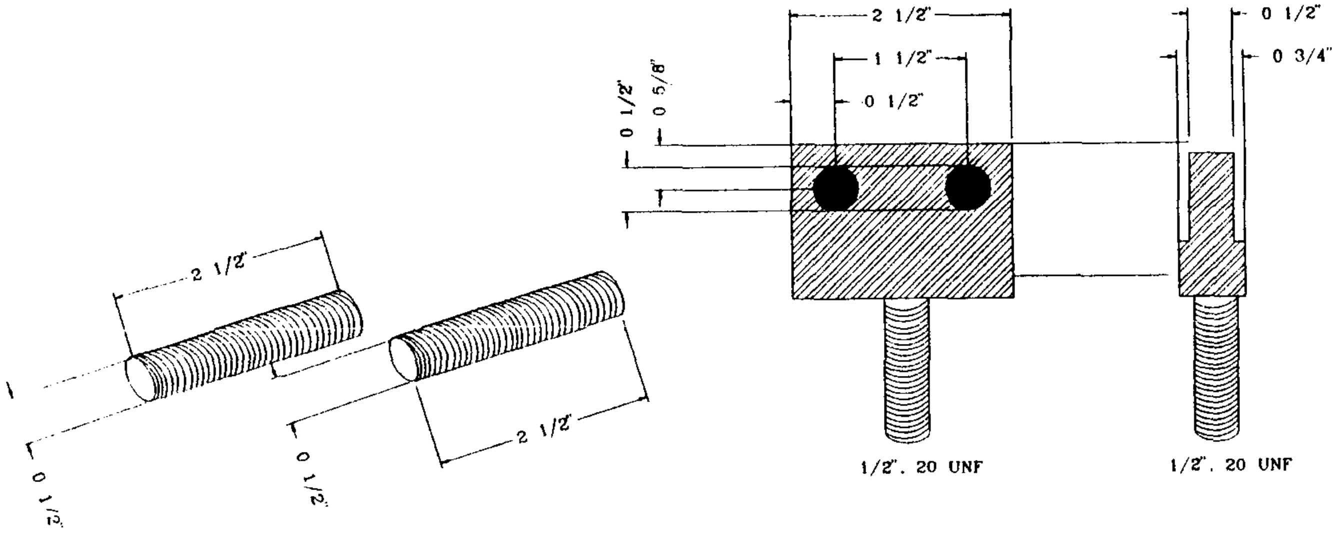
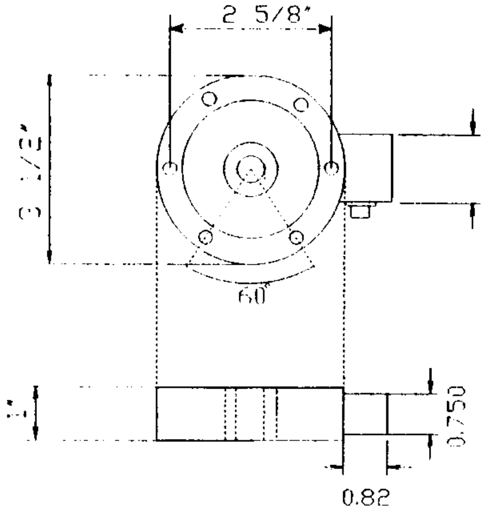


Figure 12. Bottom grip center plate. Two 1/2" dia. holes drilled through to allow passage of threaded rods. Gripping plates attach by nuts.



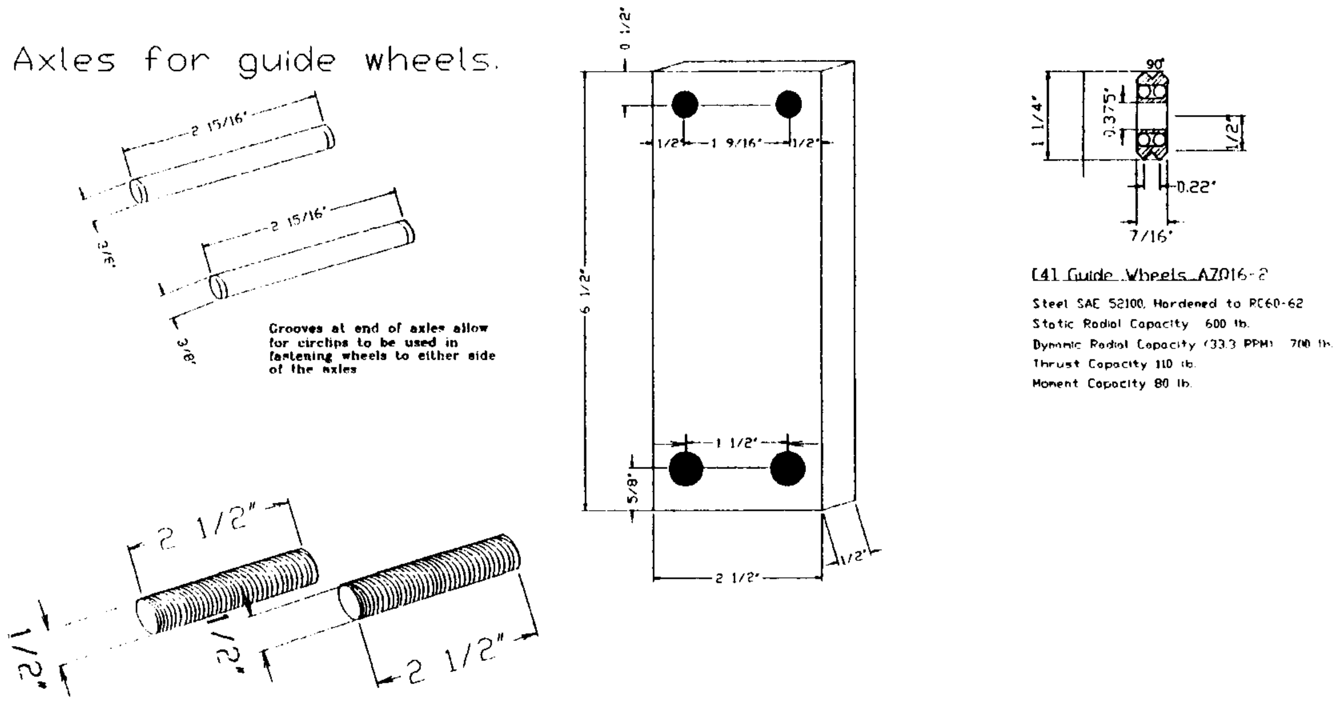
6 clearance holes equally spaced on 2.625" diameter B.C., 0.34" diameter thru.

Manufacturer's Specifications

Sensotec Model 41 Load Cell
Serial No. 296393
Part No. 572-05
Capacity: 0-5000 Lbs
Excitation Voltage: 10.0
T.C. Range: 60-160 degrees F.
Shunt Resistor Value: 59 kohms
Matching cable and connector (AA113)

With Amplifier: %Capacity	Output
0	0 volts
50	2.5004 volts
100	4.9996 volts
50	2.5027
0	0 volts

Figure 13. Sensotec load cell with manufacturer's specifications.



Grooves at end of axles allow for circlips to be used in fastening wheels to either side of the axles

[4] Guide Wheels AZ016-2
Steel SAE 52100, Hardened to RC60-62
Static Radial Capacity 600 lb.
Dynamic Radial Capacity (33.3 PPM) 700 lb.
Thrust Capacity 110 lb.
Moment Capacity 80 lb.

Same as threaded rods in bottom grip. Requires 4 wing nuts to attach gripping plates

Figure 14. Shuttle assembly for upper grip.

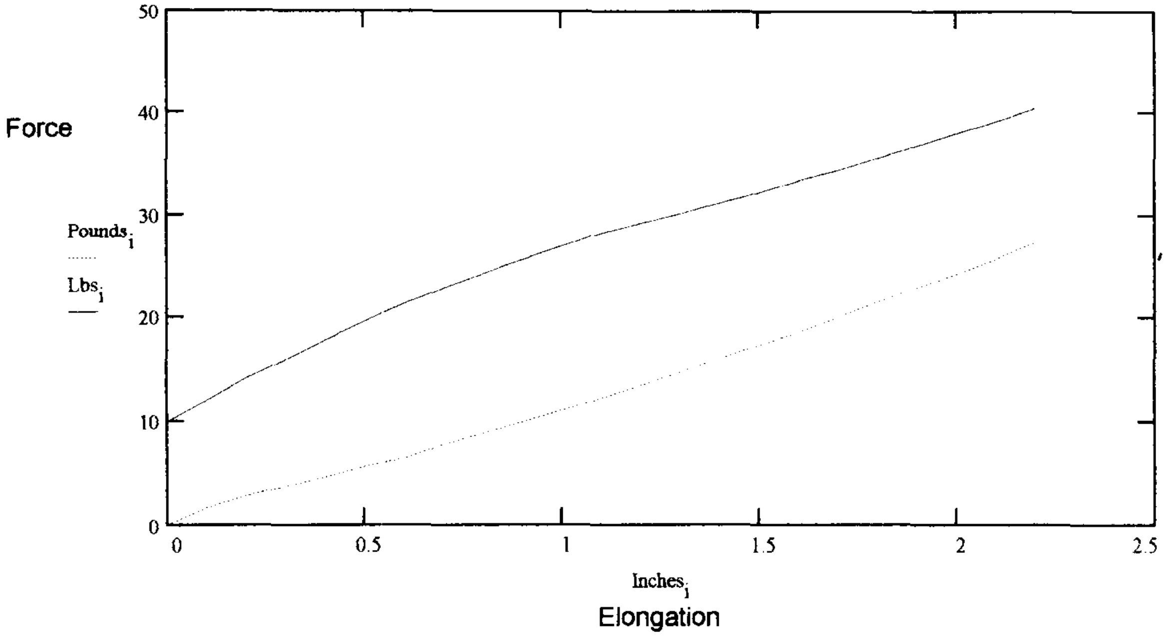


Figure 15. Neat samples of Ferris See-thru tested on Chatillon {Pounds} and hand operated device {lbs} using load cell manufacturer's specifications.

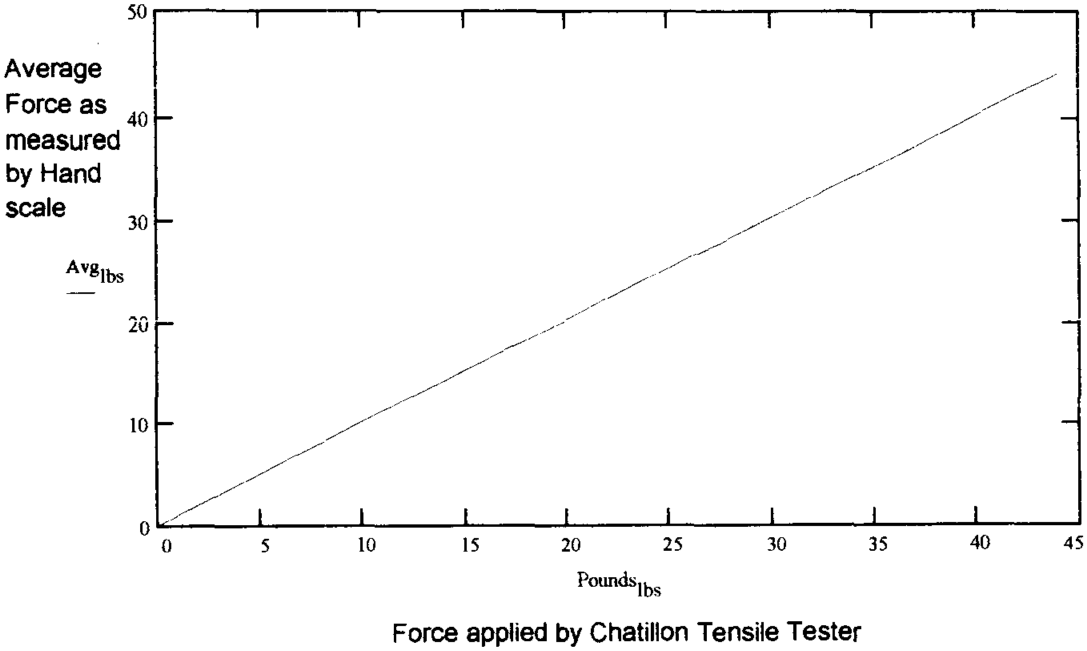


Figure 16. Average reading of hand scale (Chatillon Model IN-50) for ten trials, 0 - 44 pounds on Chatillon Tensile Tester.

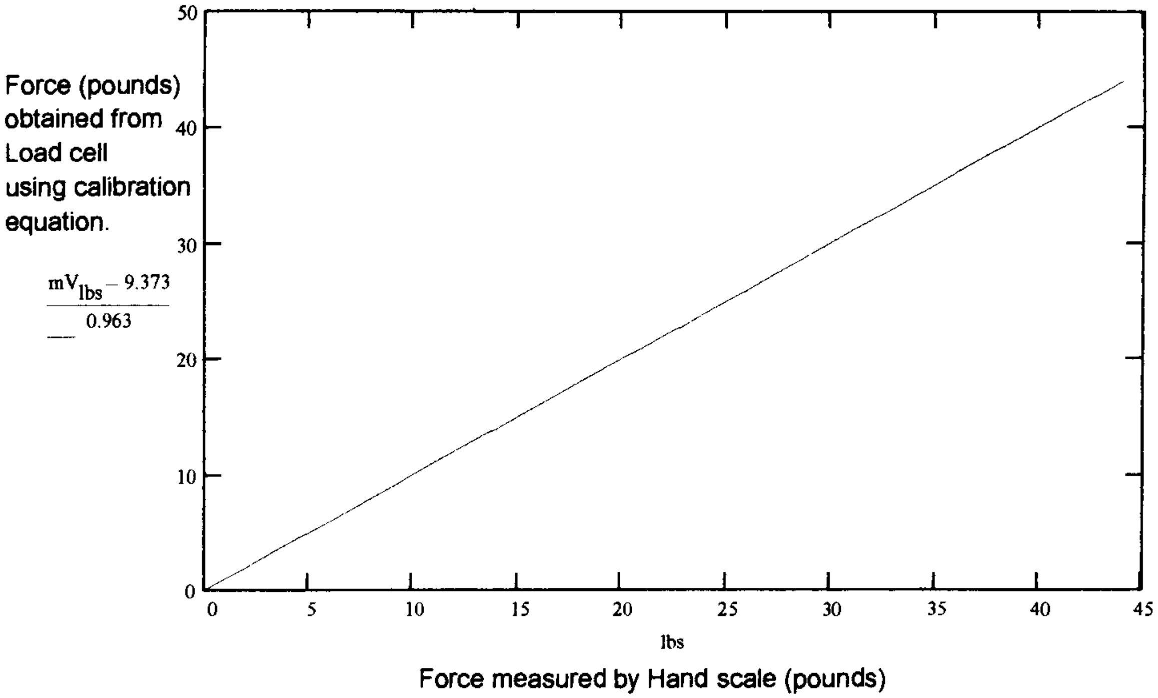


Figure 17. Calibration of load cell using Chatillon Model IN-50 Hand Scale. The load cell's average output in millivolts for ten trials is plotted against the hand scale's reading in pounds. The relationship between the load cell's output in millivolts to the force applied was determined to be. . .

$$\mathrm{Lbf} = \frac{mV - mV_0}{0.963}$$

mV = reading in milli-volts

mV_0 = initial reading [millivolts]

Lbf = Pounds force

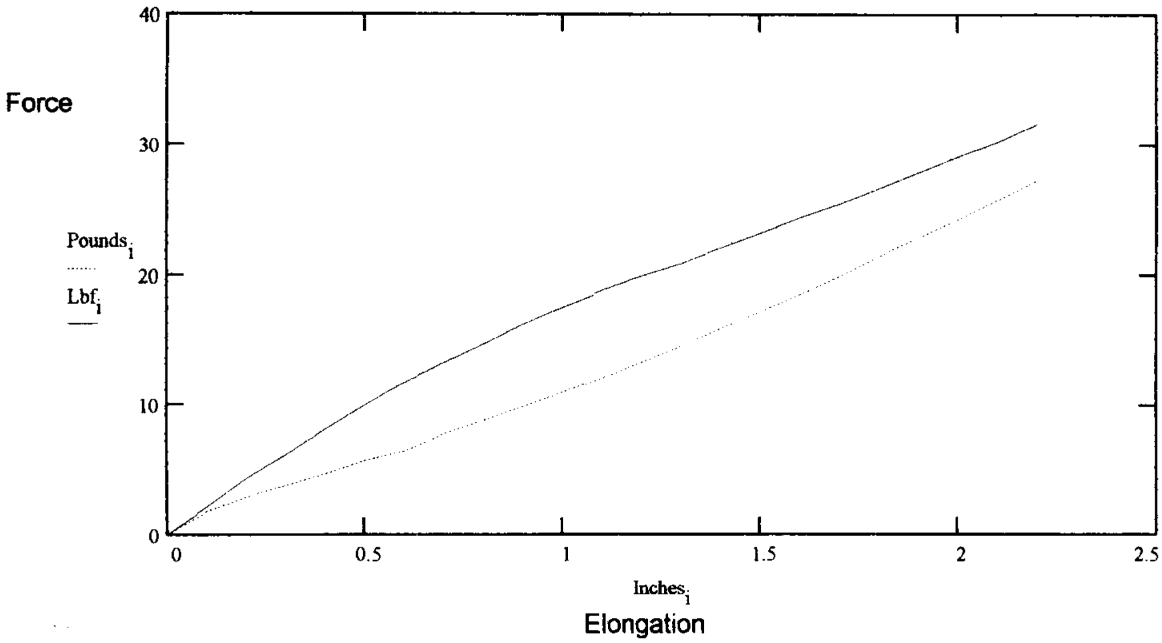


Figure 18. Neat samples of Ferris See-thru tested on Chatillon {pounds} and hand operated device (Lbf) using calibration data.

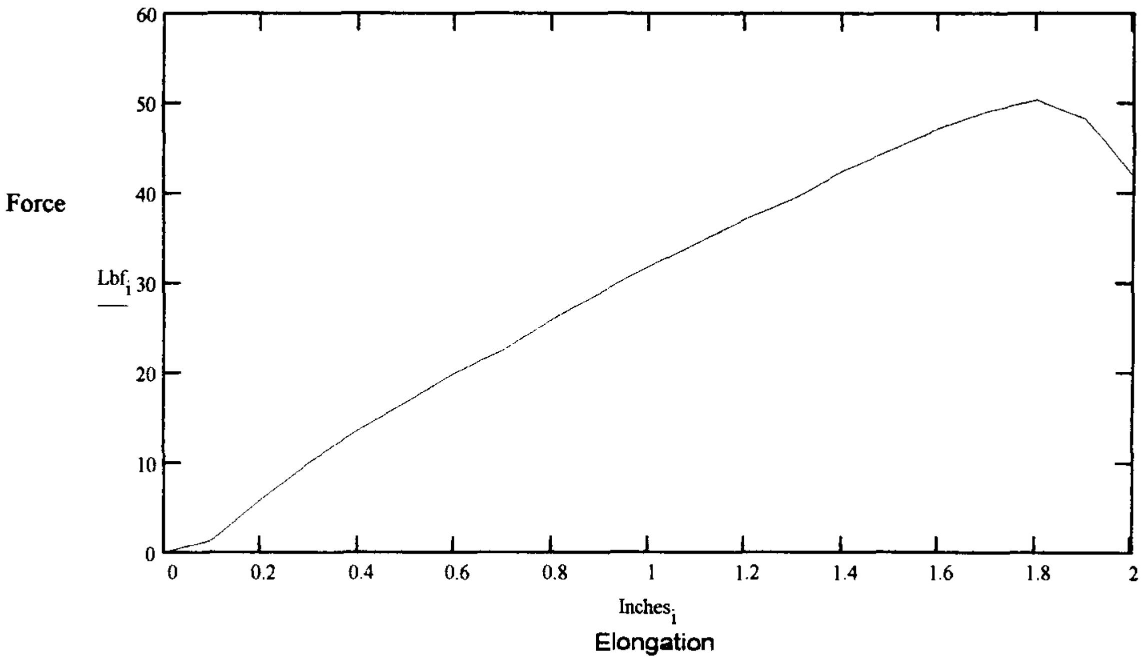


Figure 19. Force vs. elongation for Ferris See-thru Neat (0716931).

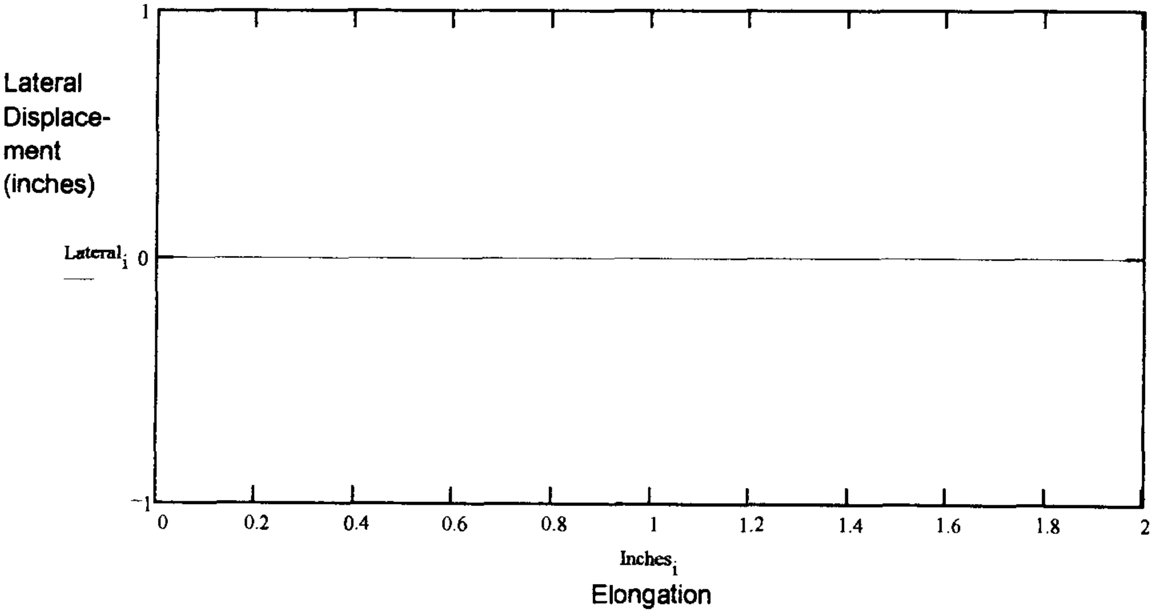


Figure 20. Lateral displacement vs. elongation for Ferris See-thru Neat (0716931).

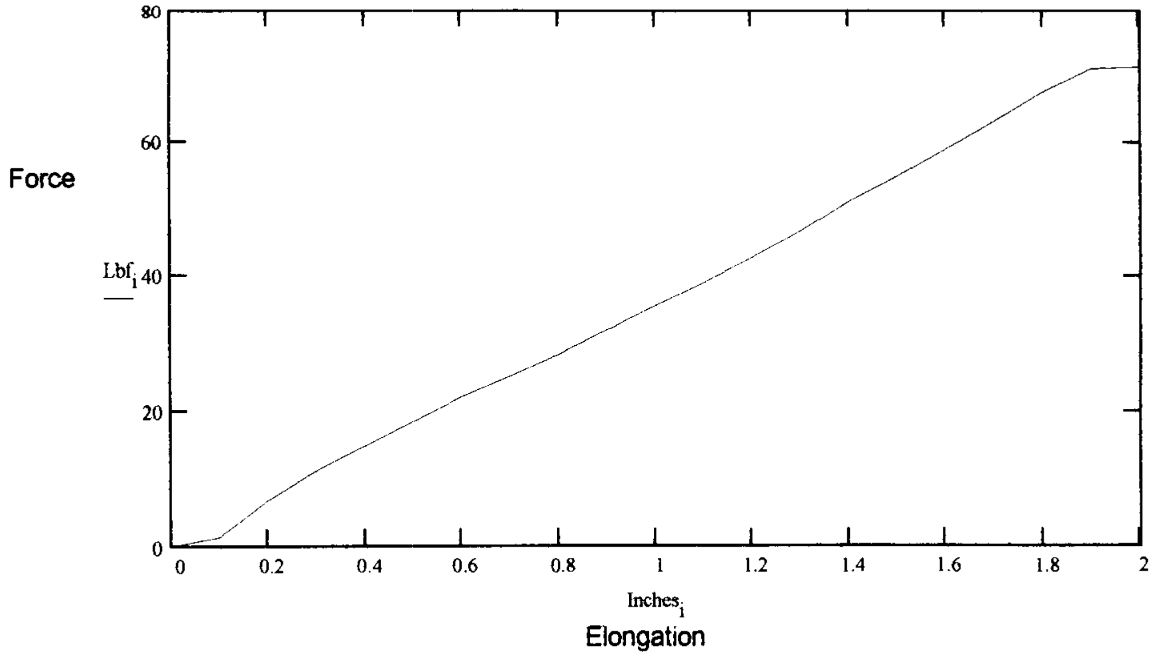


Figure 21. Force vs. elongation for Ferris See-thru Al 0° (0716932).

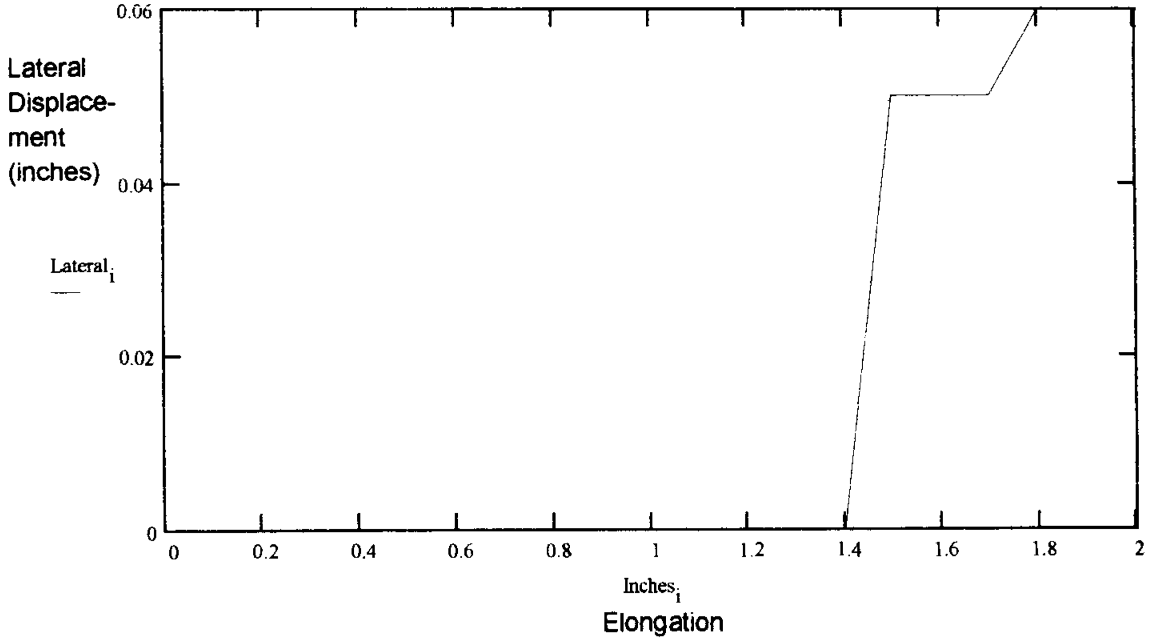


Figure 22. Lateral displacement vs. elongation for Ferris See-thru Al 0° (0716932).

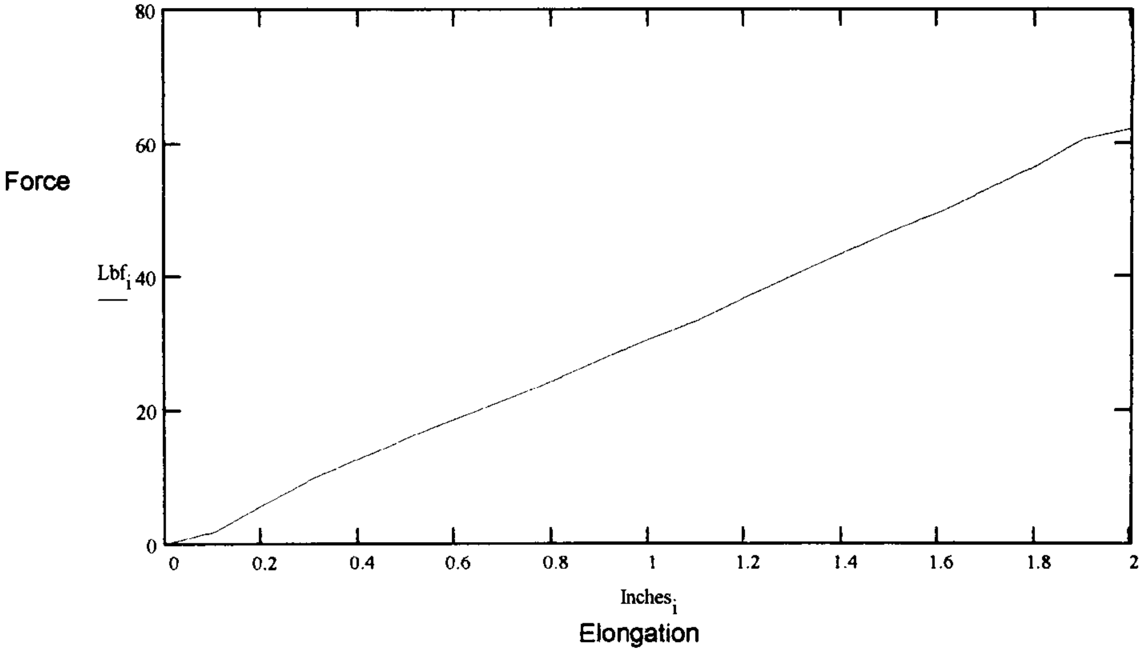


Figure 23. Force vs. elongation for Ferris See-thru Al 30° (0716933).

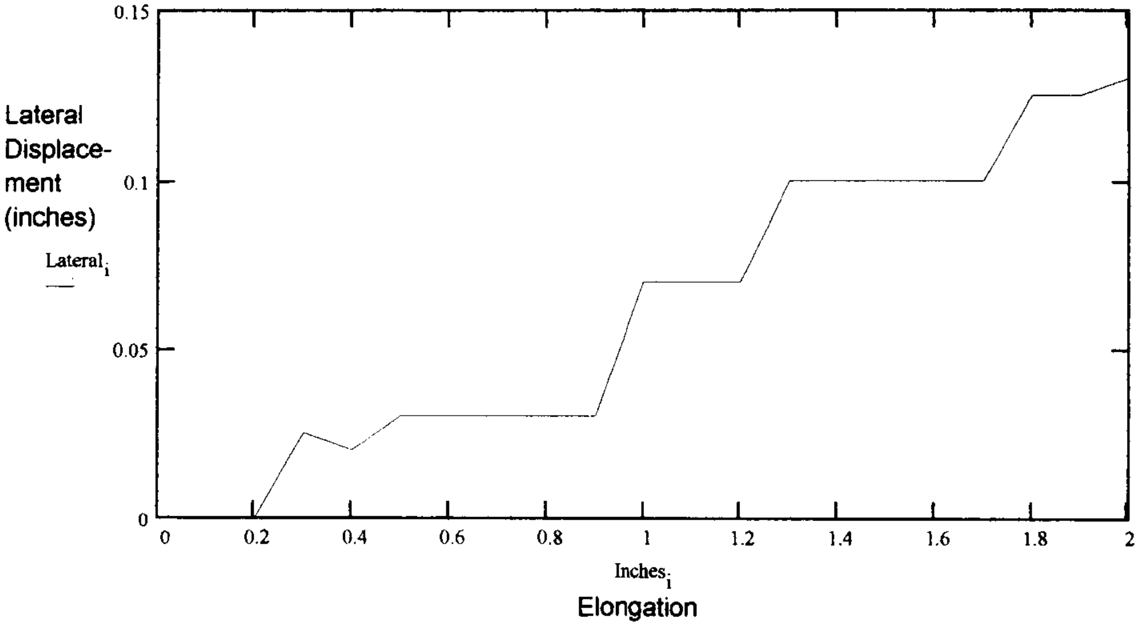


Figure 24. Lateral displacement vs. elongation for Ferris See-thru Al 30° (0716933).

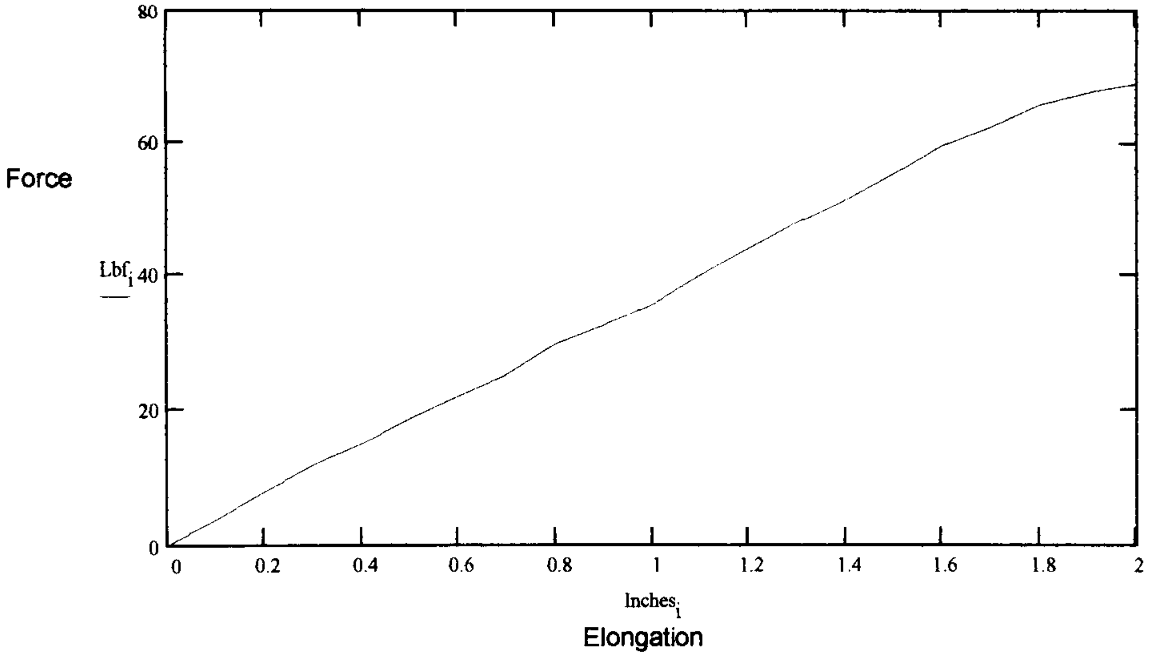


Figure 25. **Force vs. elongation for Ferris See-thru Al 45° (0716934).**

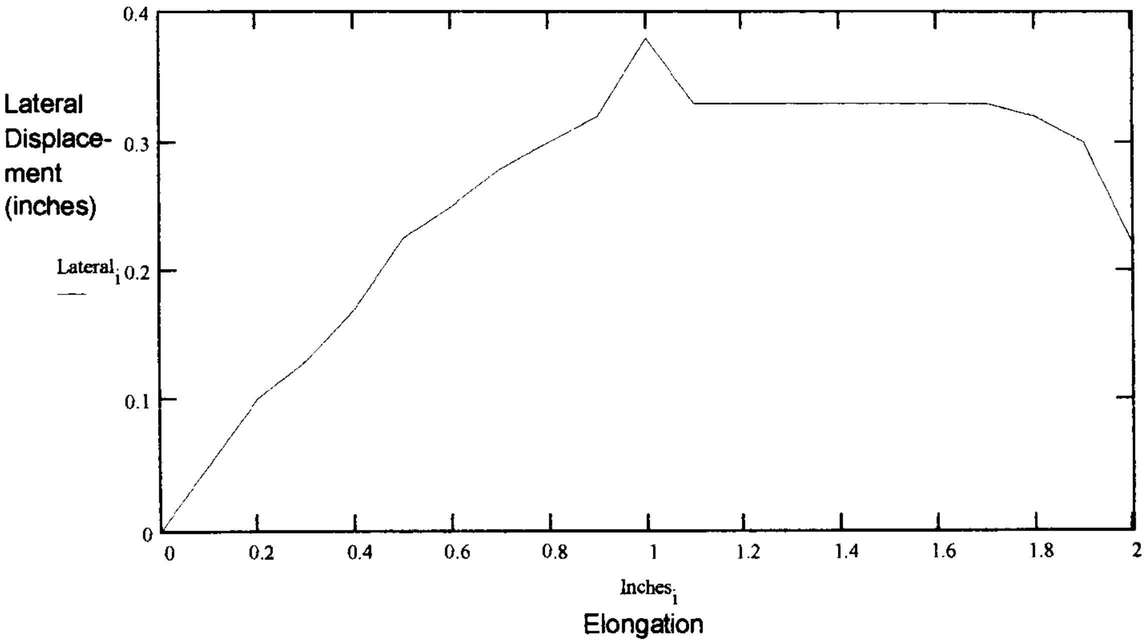


Figure 26. **Lateral displacement vs. elongation for Ferris See-thru Al 45° (0716934).**

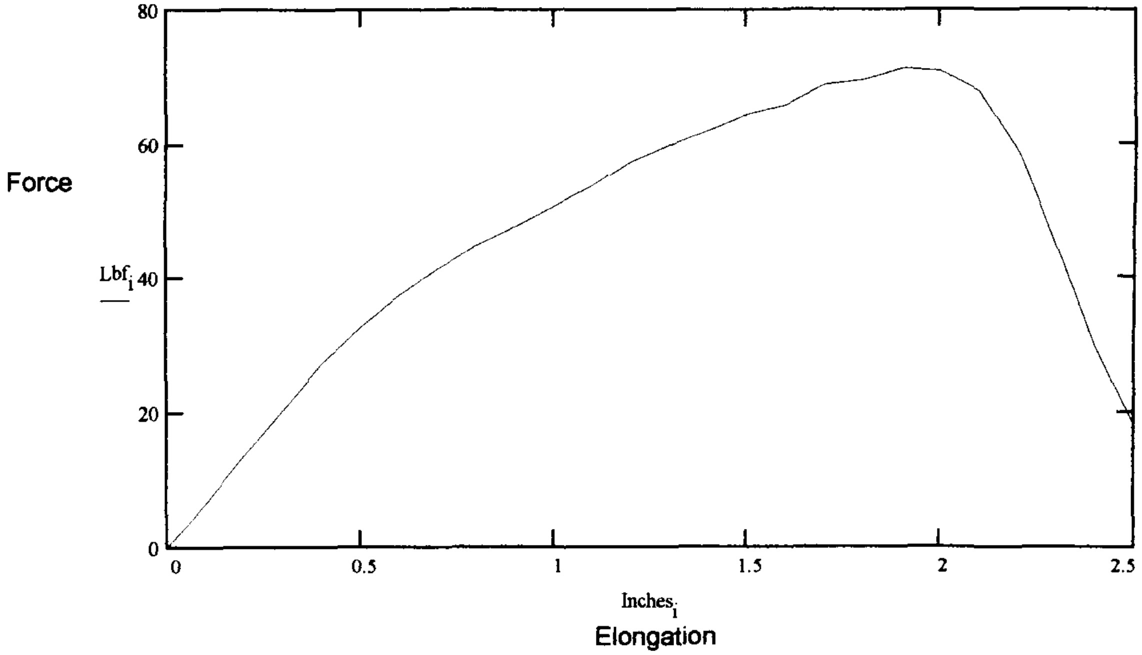


Figure 27. Force vs. elongation for Ferris See-thru Al 60° (0716935).

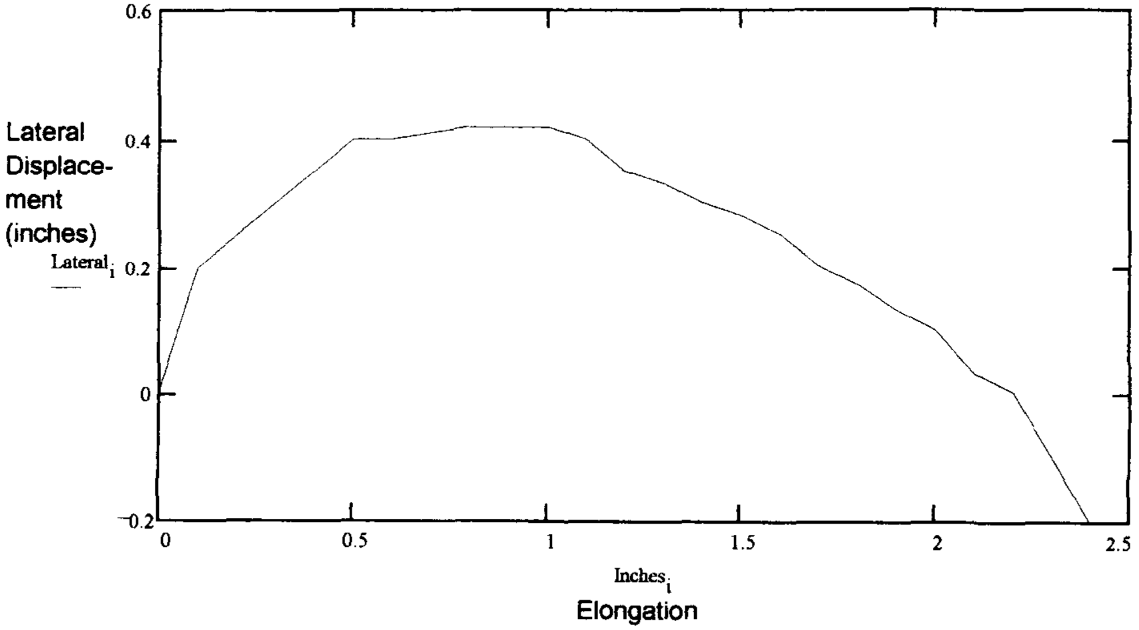


Figure 28. Lateral displacement vs. elongation for Ferris See-thru Al 60° (0716935).

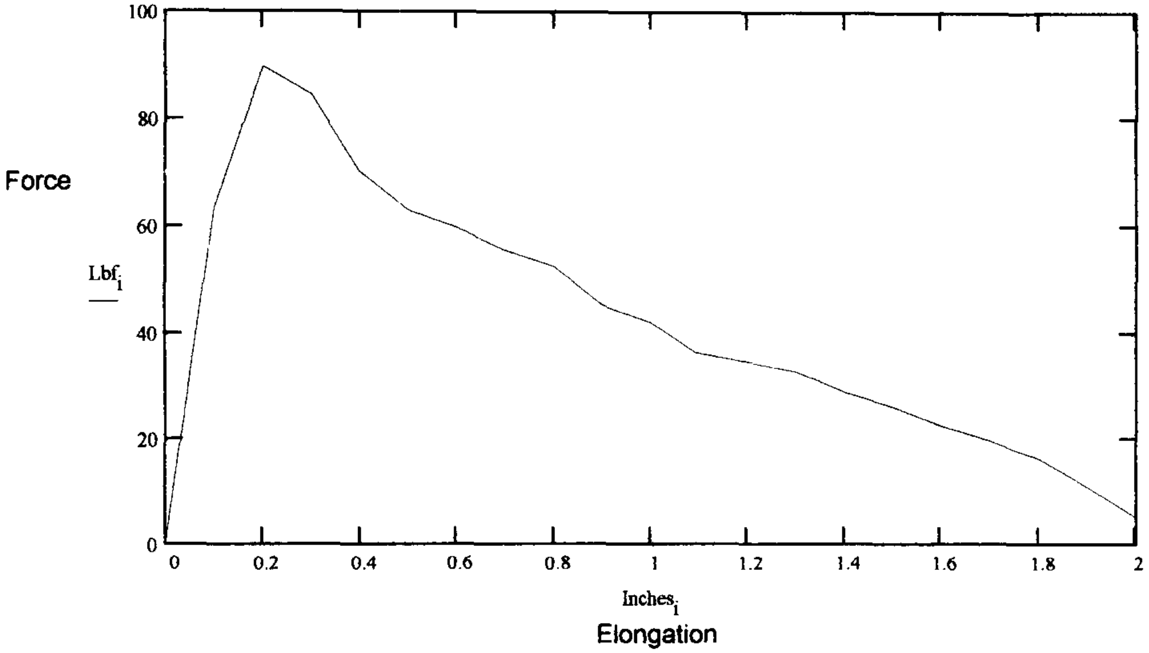


Figure 29. **Force vs. elongation for Ferris See-thru Al 90° (0716936).**

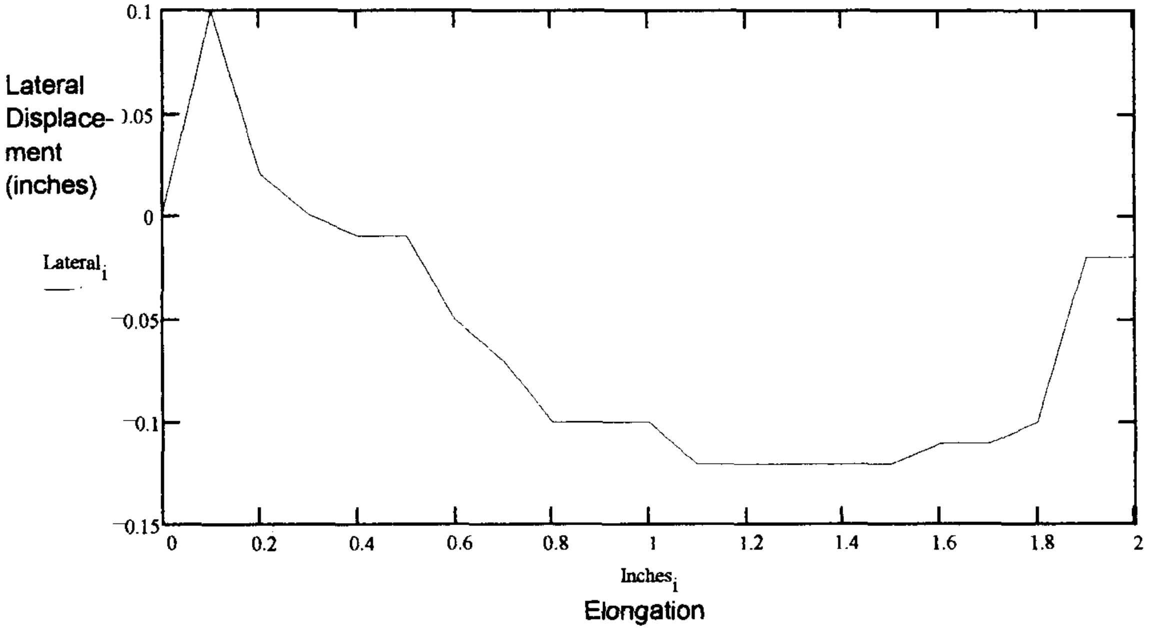


Figure 30. **Lateral displacement vs. elongation for Ferris See-thru Al 90° (0716936).**

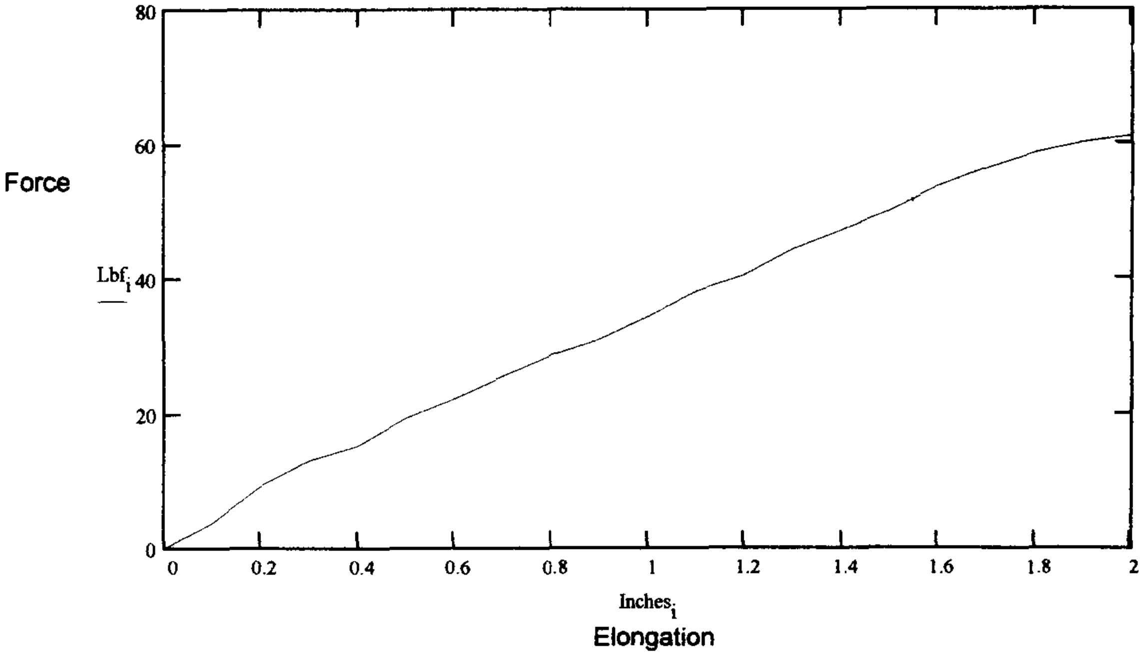


Figure 31. Force vs. elongation for Ferris See-thru Fiberglass 0° (0716937).

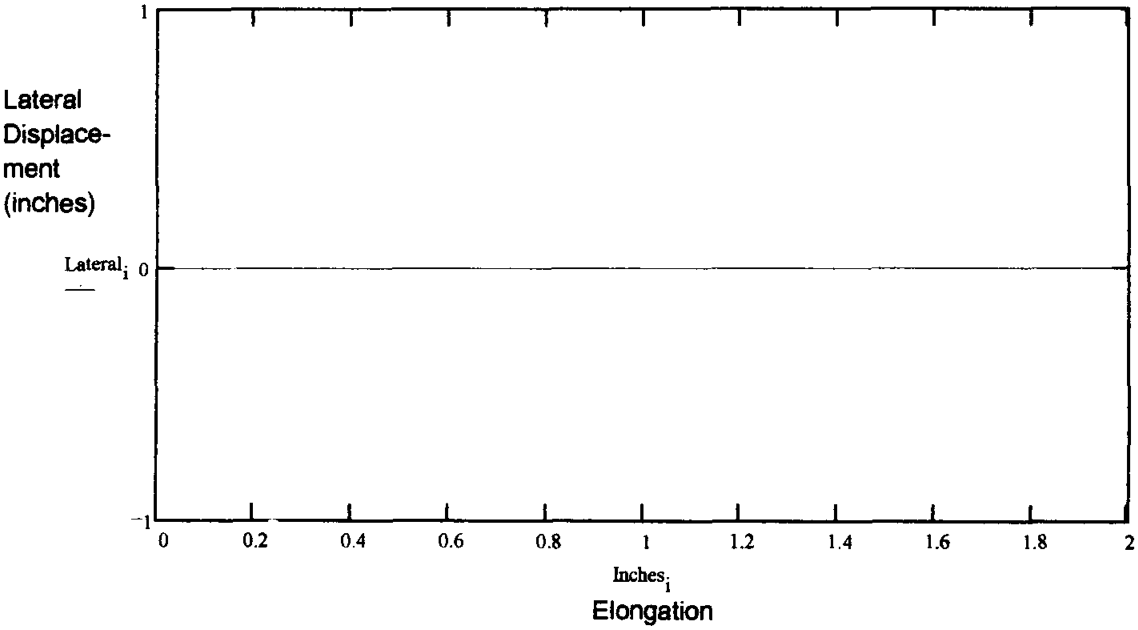


Figure 32. Lateral displacement vs. elongation for Ferris See-thru Fiberglass 0° (0716937).

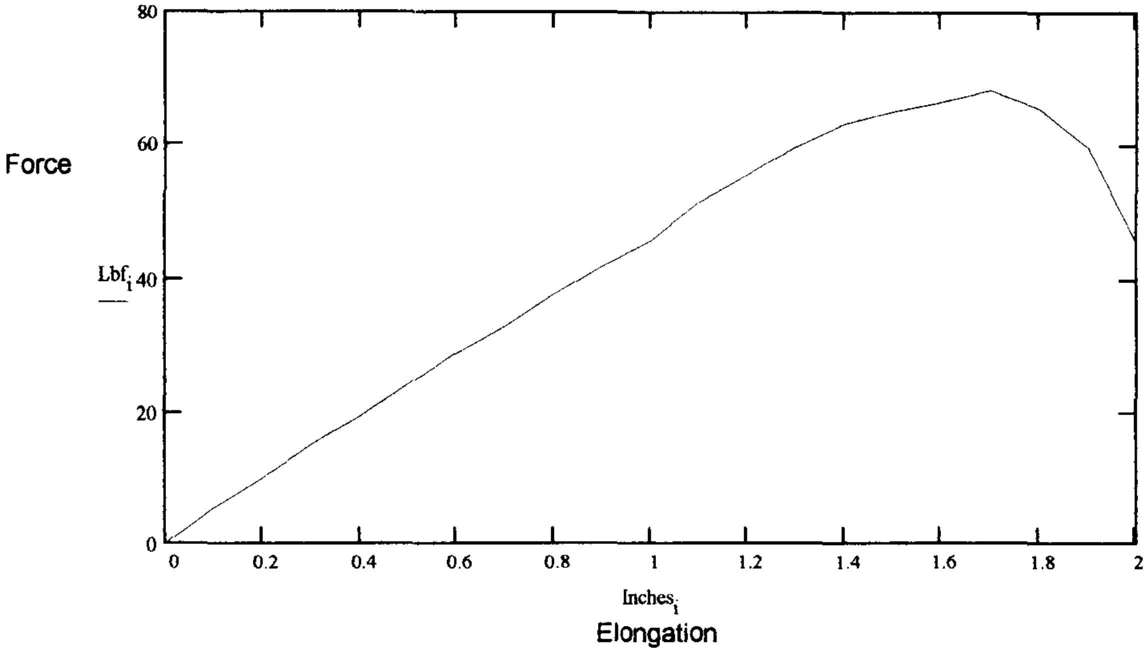


Figure 33. **Force vs. elongation for Ferris See-thru Fiberglass 30° (0716938).**

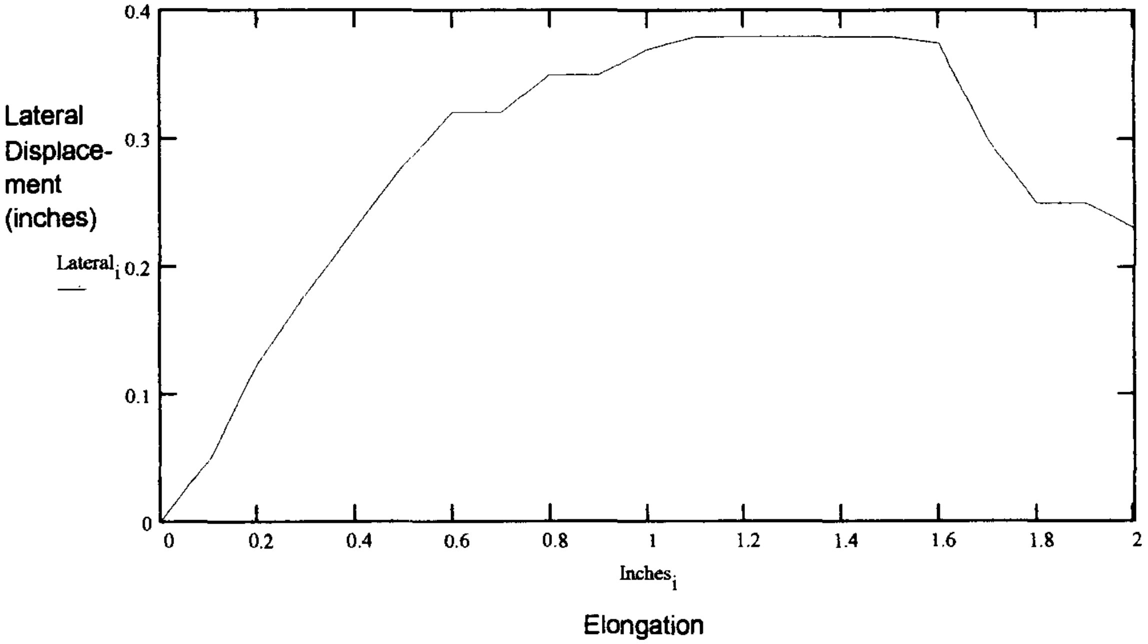


Figure 34. **Lateral displacement vs. elongation for Ferris See-thru Fiberglass 30° (0716938).**

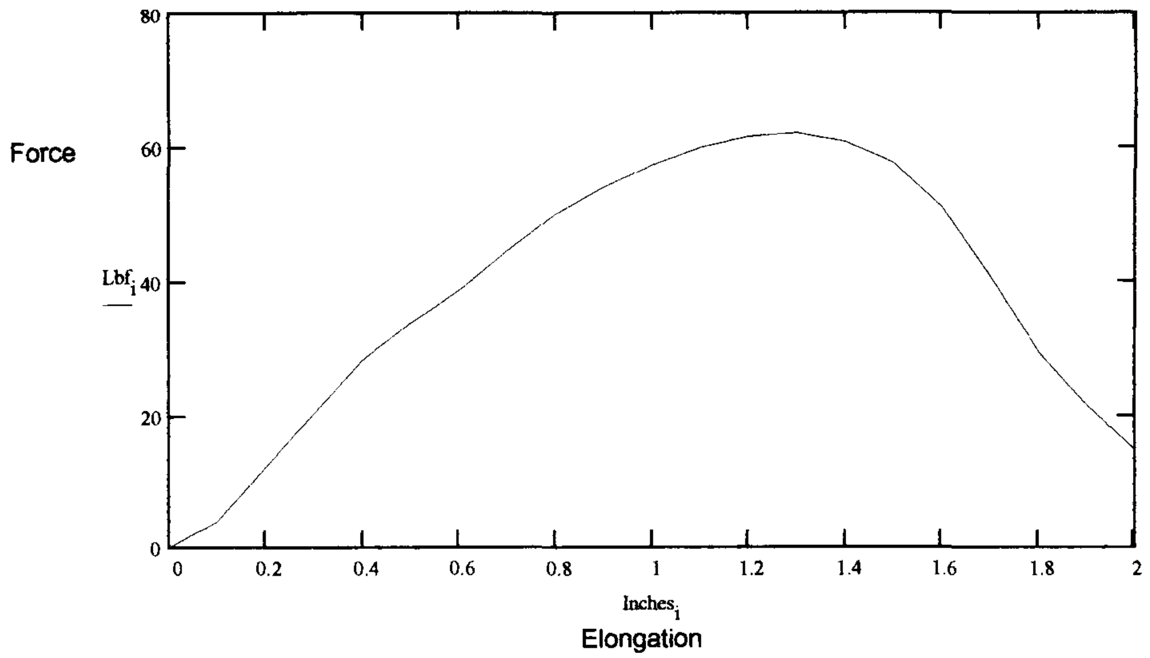


Figure 35. Force vs. elongation for Ferris See-thru Fiberglass 45° (0716939).

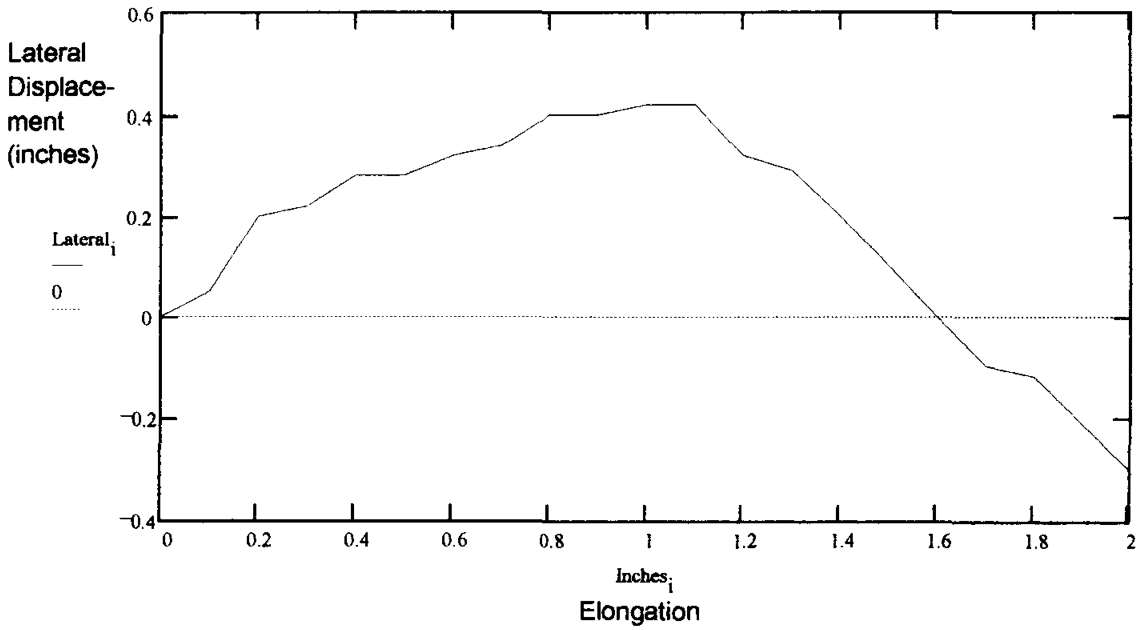


Figure 36. Lateral displacement vs. elongation for Ferris See-thru Fiberglass 45° (0716939).

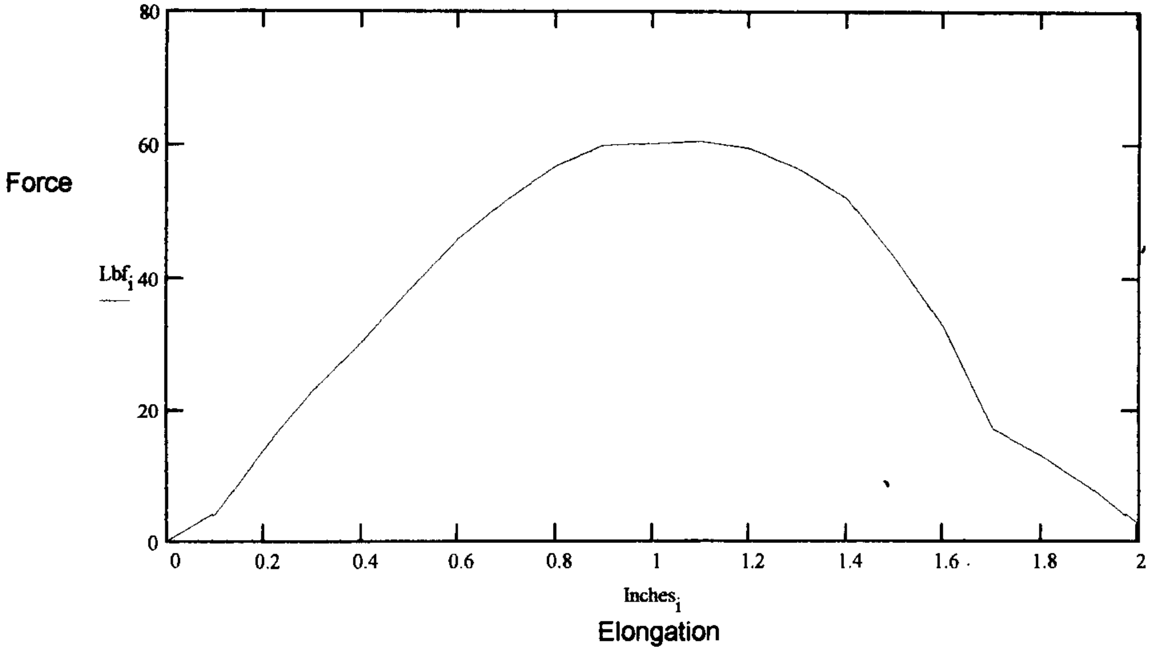


Figure 37. Force vs. elongation for Ferris See-thru Fiberglass 60° (07169310).

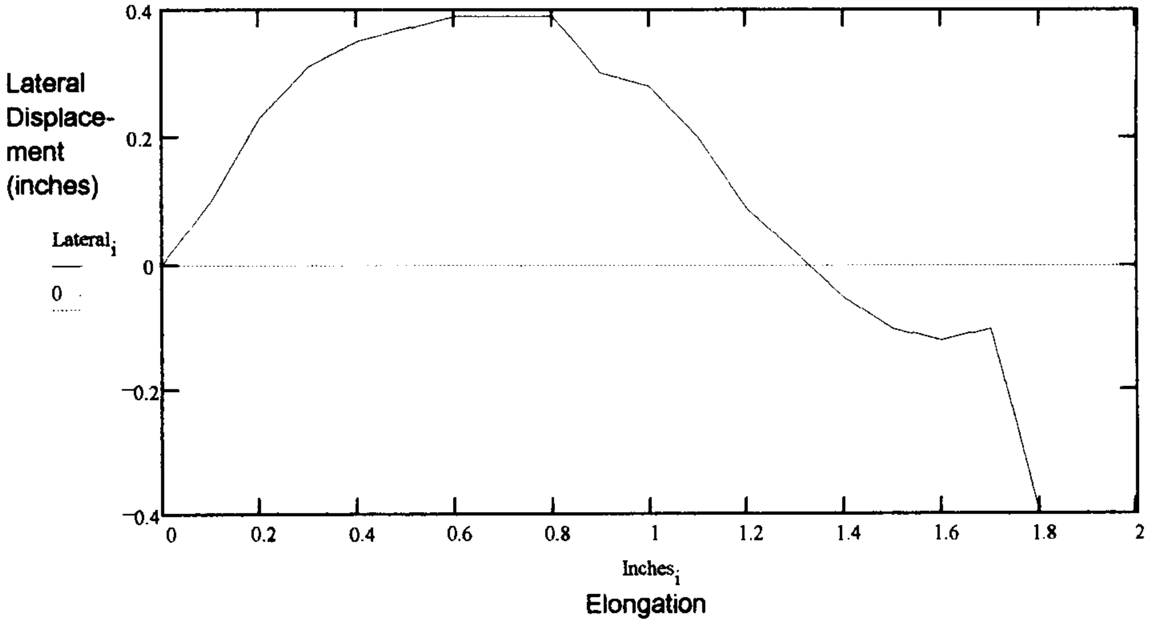


Figure 38. Lateral displacement vs. elongation for Ferris See-thru Fiberglass 60° (07169310).

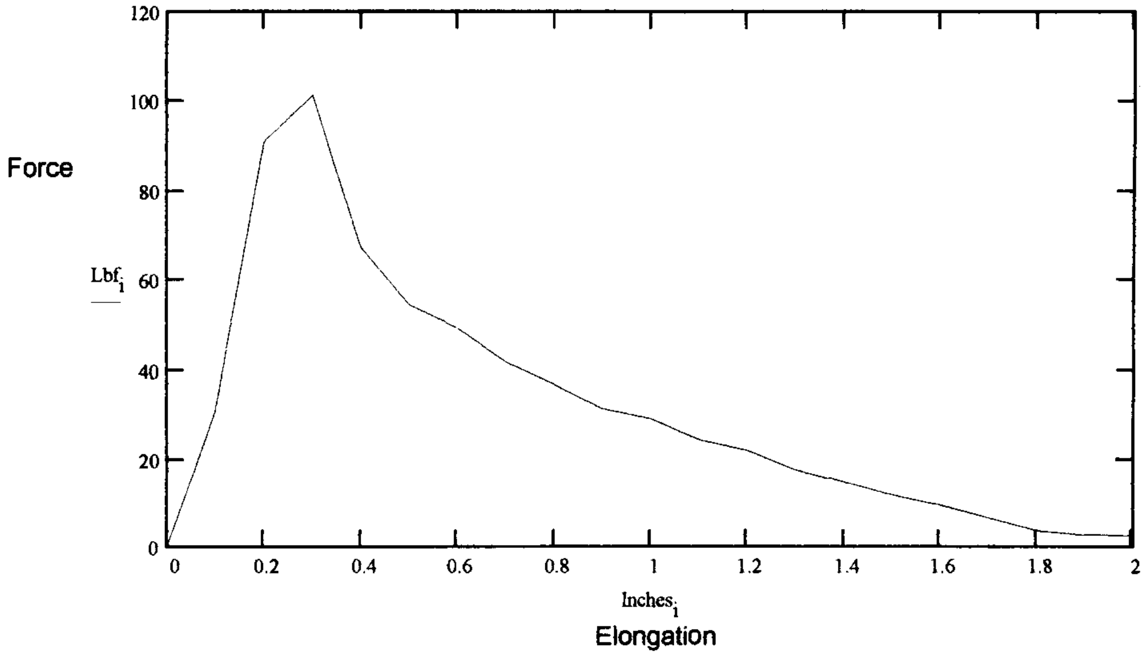


Figure 39. Force vs. elongation for Ferris See-thru Fiberglass 90° (07169311).

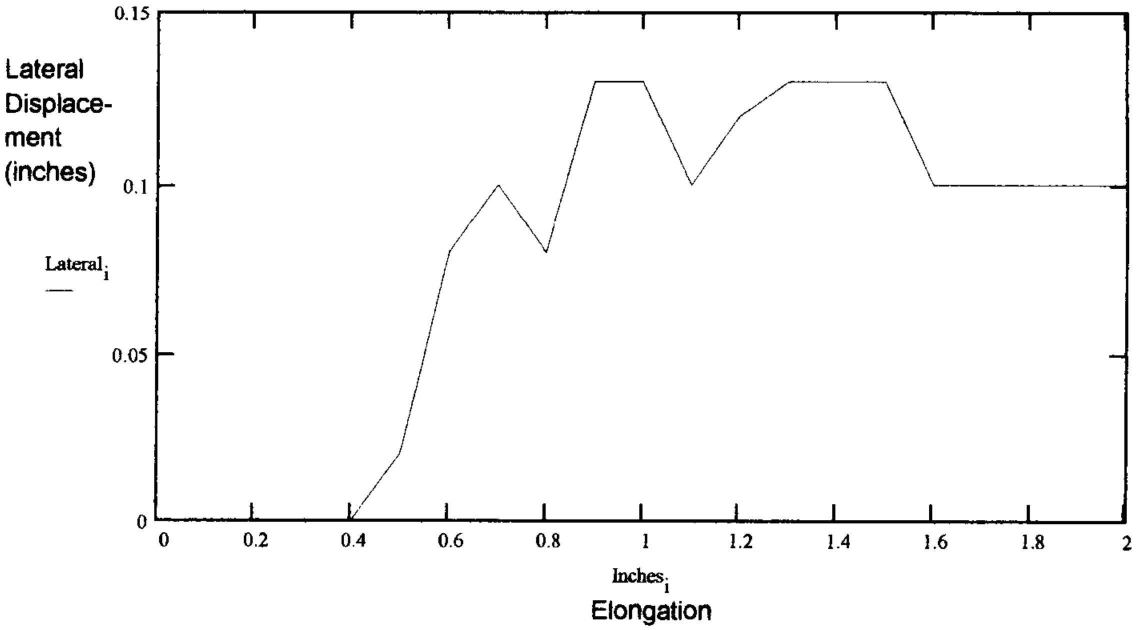


Figure 40. Lateral displacement vs. elongation for Ferris See-thru Fiberglass 90° (07169311).

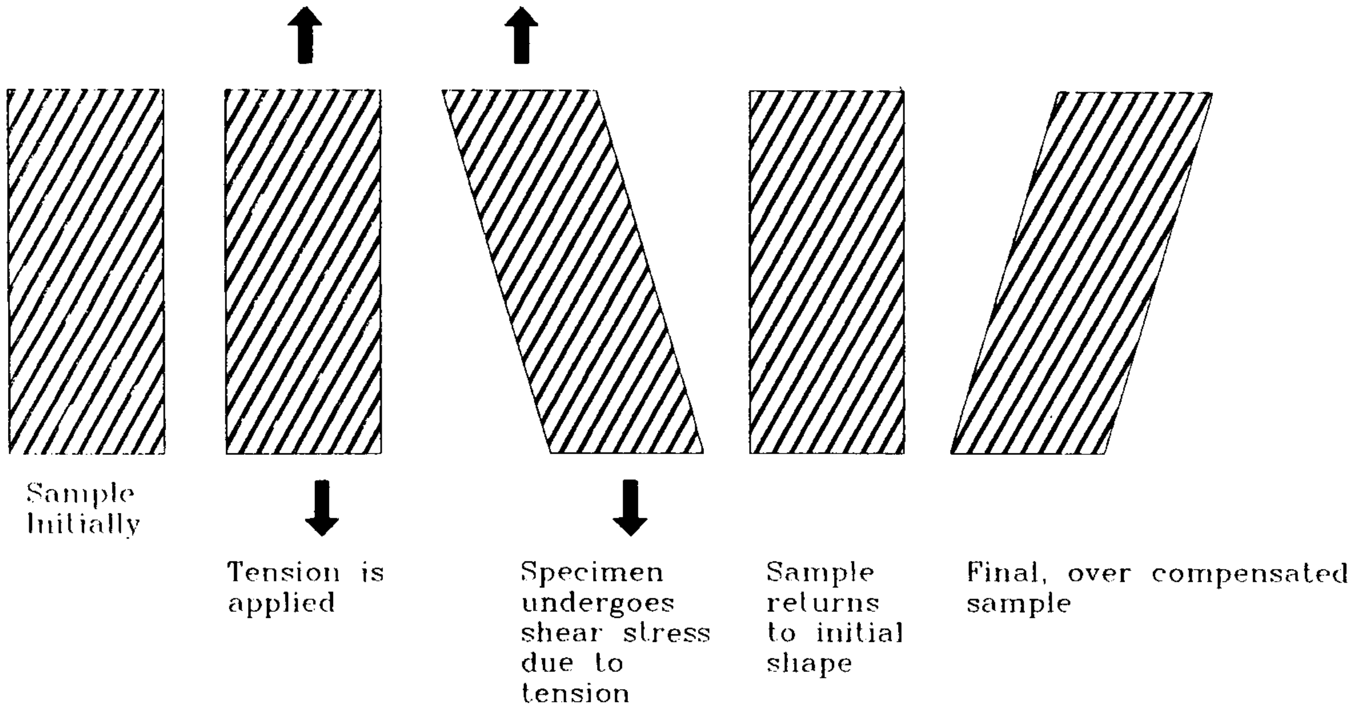


Figure 41. Schematic of sample deformation.

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APPLICATION OF MATERIALS DATABASE (MAT. DB.) TO MATERIALS EDUCATION

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Application of Materials Database (MAT. DB.) to Materials Education

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Charleston, IL 61920

KEY WORDS: material properties, strength, ductility, material data base, environment and working conditions.

PREREQUISITE KNOWLEDGE: The student should understand the fundamentals of materials behavior such as tensile strength, percent elongation, area reduction, manufacturability and formability. Some understanding of change in material behavior with different environments and loading conditions is expected. Knowledge of the basic application of a microcomputer and DOS commands are essential.

OBJECTIVES: To use the materials database MAT. DB. to search for useful data on materials behavior related to specific applications. With the materials database, design analysis, materials selection and manufacturing of products can be more efficient.

EQUIPMENT AND SUPPLIES:

1. IBM compatible 286 or higher microcomputer.
    a. 640 K RAM
    b. At least 15 M free hard disk
    c. Graphics card of CGA, Hercules, EGA, and VGA
    d. 4.25" or 3.5" floppy disk drive
    d. DOS 3.0 or higher.
2. MAT. DB and database files from ASM International.
3. Printer is optional for output.

INTRODUCTION:

Finding the right material for the job is an important aspect of engineering. Sometimes the choice is as fundamental as selecting between steel and aluminum. Other times, the choice may be between different compositions in an alloy. Discovering and compiling materials data is a demanding task, but it leads to accurate models for analysis and successful materials application.

Mat. DB is a database management system designed for maintaining information on the properties and processing of engineered materials, including metals, plastics, composites and ceramics. It was developed by the Center for Materials Data of American Society for Metals (ASM) International. The ASM Center for Materials Data collects and reviews material property data for publication in books, reports, and electronic database. Mat. DB was developed to aid the data management and material applications.

Mat. DB can provide the following functions to manage database for materials:

1. View information on specific material:

The material data are organized in material records, which include material designations, specifications, composition, product forms, classes, ranking, properties, graphs and notes. Once entering the database, the user can navigate among the nine categories of information for a specific material.

2. Searching:
One of the most powerful features of Mat. DB is its ability to search through megabytes of information to locate needed material data. The user begins a search session by creating a "folder", which contains all related results. The searched folder can be viewed in the same manner as above.

3. Editing:
Users can build their own database with the tools provided by Mat. DB. Sophisticated databases can be established to suit various applications for users.

4. Reports:
Mat. DB is capable of viewing and printing the results of a search in formats that help analyze search results.

PROCEDURE:

1. Start the Mat. DB and load the desired database file for the material of interest. A main manual will be prompted for each database file.

2. View data for a specific material:
After choosing "VIEW FOLDER" from the main manual, a material record manual will be shown on the screen. Mat. DB's basic building block is the *Material Record*, the file in which information on materials is organized. A separate record is devoted to each materials designation, e.g. AISI 1020, Ti-6Al-4V, etc. These records are then stored in Mat. DB databases. Each record can contain the following information for a given material:

Designation: The designations screen contains necessary information to identify the material and source of the data. It includes *Accession Number* (an identifying number unique to a material record), *UNS Number* (a cross-referencing number standardized by American Society for Testing and Materials (ASTM) and Society of Automotive Engineers (SAE)), *Material Group*, *Designation*, *Common Name*, *Manufacturer*, *Country* and *User.*

Specifications: Each material can be cross-referenced to a maximum of 50 alternate specifications. Each specification can be annotated with a short note.

Composition: Minimum and maximum percentages can be maintained for up to 20 elements. Additional comments on compositions can also be stored for display along with the element percentages.

Product Forms: The user may index the material by up to 50 different product forms.

Classes: The user may index each material condition by up to 50 application classes such as heat resistant, high strength, etc.

Ranking: Each material condition can be ranked for processing characteristics such as formability, weldability, machinability, hardenability, processing cost, and availability. The user can define up to 6 ranking categories.

Properties: Properties can be reported for up to 20 different conditions per material. For each condition up to 100 properties can be reported at as many as 20 temperatures. The properties are displayed on a screen that looks like a spreadsheet.

Graphs: Up to 20 graphs can be maintained and displayed for each material. Graphs can also be saved to a DOS file or printed directly to a laser or dot matrix printer.

Notes: Mat. DB can maintain up to 200 lines of comments for each material. The Notes section offers many standard word-processing features, making it extremely easy to add new comments to a record or edit existing comments.

3. Search information with "SIFT FOLDER" feature of Mat. DB.

Material information can be searched according to various combinations of material data on designation, specifications, composition, forms, classes, rankings, properties and so on. For example, in the folder of STARTER.DB (database supplied with Mat. DB), "sift folder" with the following conditions

CLASSES = CORROSION RESISTANT and
FORMS = BARS

results in the following three materials

316	STAINLESS STEEL
410	STAINLESS STEEL and
A3003	WROUGHT ALUMINUM.

All the material data on these three corrosion resistant bars can be found using the "VIEW FOLDER".

4. Build your own database with "editing" feature of Mat. DB.

If the database was opened in the *read-write* mode, a new material record can be created by moving the highlight to "new" at the bottom of the *List Screen* and pressing *enter*. This will open an empty material record.

The new material record will begin with "DESIGNATION" and data will be entered in the edit boxes. When finished, press the F10 function key and the editing will be saved.

5. Output the results:

The results can be printed with a dot matrix printer.

SAMPLE DATA SHEETS:

The following are two examples for the property data of AISI 4140 steel and high density polyethylene, respectively.

A. AISI 4140 Steel

Database: STARTER.DB
Folders: Metal Alloys (11)
View Folder: 4140 G41400 Alloy Steel

1. Designation

Accession Number	281189015
UNS Number	G41400
Material Group	Alloy Steel
Designation	AISI 4140
Common Name	4140
Manufacturer	
Country	USA
User	ASM

2. Specifications

Organization	Specification
AMS	6381 (*Aerospace Material Specification)
AMS	6382
AMS	6390
AMS	6395
ASTM	A322
ASTM	A331
ASTM	A505
ASTM	A519
ASTM	A547
ASTM	A646
MIL	S-16974
SAE	J404
SAE	J412
SAE	J770
DIN	1.7225

3. Composition

Element	Min.%	Max. %
C	.38	.43
Cr	.8	1.1
Mn	.75	1.
Mo	.15	.25
P	.	.035
S	.	.04
Si	.15	.3

4. Notes

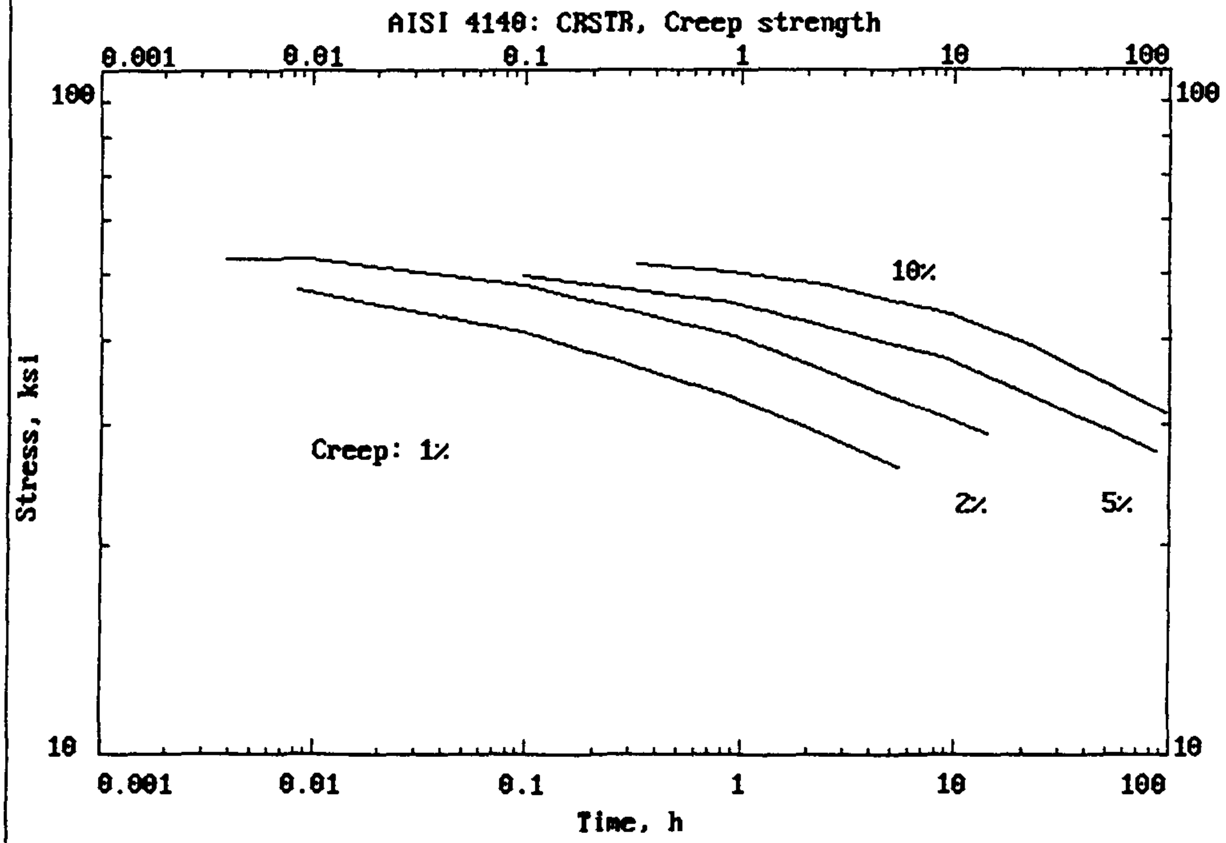
High-hardenability, medium-carbon steels; popular grades
DESCRIPTION: Grades 4140, 4140H, 4142 and 4142H are combined for discussion purpose because they are so similar in compositions and characteristics. Actually, they are so close that the composition could be either 4140 or 4142.

...

5. Properties

4140	Typical Properties		
	100 ^{o}C	200 ^{o}C	400 ^{o}C
ElResis n{o}*m	262.	326.	475.
ThCoefExp {u}m/m*K	12.2	12.6	13.7
ThConduct W/m*K	42.6	42.2	37.7
ThSpcHeat J/kg*K	.	473.	519.

6. Graphs: One example is shown below.



Sheet, 0.050 in., annealed at 1550 F, and tested at 1000 F.
Struct. Alloys Handbook, D.J.Maykuth, Ed., Vol 1 & 2,
p.16, Battelle, 1981

B. High Density Polyethylene (HDPE)

Database:	THERPLAS.DB
Folders:	Polyethylene (A-M) (63)
View Folder:	Marlex HXM 50100

1. Designation

Accession Number	81789333
Code Number	190100
Material Group	Polyolefin
Designation	Polyethylene
Common Name	Marlex HXM 50100
Manufacturer	Philips Chemical Co.
Country	USA
User	Engineering Thermoplastic

2. Notes

High-density polyethylene, use with food and drugs.
Uses: Large formed parts, cattle feeders, pallets, boats

FOOTNOTES
A Condition 190/2.16
B Type IV specimen, 2 per min.

3. Process: Thermoforming

4. Properties

	Typical Properties
	23 °C
Density, kg/m^3	950
FlexModul, GPA	2.08
StrElgYld, %	600
TenYldSt, MPa	26

5. Features:
Impact resistance
Food/drug use

6. Rankings
Chemical Resist.
Creep Resist.
Fatigue Resist.
Heat Resist.
Processability
Cost

INSTRUCTOR NOTES:

1. It is always a good idea for students to start with "Read-Only" mode for materials database folder. In the "Read-Only" mode, no change in any material record will be saved. This will prevent any useful information from being lost.

2. When searching materials with "SIFT FOLDER", students need to make sure to use "NEW" in the folder list. Otherwise, the existing folders will be replaced by the new folder. Some information could be lost.

3. Basic knowledge of DOS for microcomputer will be essential to the operation.

REFERENCES:

1. Puttre, Michael: Materials Data Bases are Key to Design Analysis, *Mechanical Engineering*, vol. 115 (5), 1993, pp 69-71.

2. ASM International, Materials Properties Database System: Mat. DB, ASM/Center for Materials Data, Materials Park, OH 44073, 1992.

SOURCES OF SUPPLIES: This is basically a computer software project. Microcomputers are readily available. However, the software is relatively expensive. The Mat. DB costs approximately $556-695, and material database files cost from $295.20 to 526 for each major category of materials. But, the software and database files can be purchased separately, which makes the cost planning a little easier.

ACKNOWLEDGMENT: Financial support for this project from the Council for Faculty Research (CFR) at Eastern Illinois University is greatly appreciated.

LAMINATED THERMOPLASTIC COMPOSITE MATERIAL FROM RECYCLED HIGH DENSITY POLYETHYLENE

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and

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Laminated Thermoplastic Composite Material from Recycled High Density Polyethylene

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Eastern Illinois University
Charleston, IL 61920

KEY WORDS: thermoplastic composite, high density polyethylene, laminate, glass fiber fabric, ASTM standards, tensile test, ultimate tensile strength.

PREREQUISITE KNOWLEDGE: The student should understand the fundamentals of polymer processing and mechanical property testing of materials. The ability to use ASTM standards is also necessary for designing material test specimens and testing procedures.

OBJECTIVES: To understand the concept of laminated composite materials, processing, testing and quality assurance of thermoplastic composites; to observe an application example of recycled plastics.

EQUIPMENT AND SUPPLIES:
1. Recycled one-gallon milk containers
2. One glass fiber fabric of 127 × 127 mm
3. Steel rules and compass (or a specimen template)
4. Scissors
5. Thermoforming or compression machine for plastics
5. Tensile test machine.

PROCEDURE:

1. Collect and clean recycled milk containers.

2. Cut four (4) flat sheets of high density polyethylene from the milk containers. The sheet size is approximately 130 × 130 mm.

3. Cut a piece of glass fiber fabric of 127 × 127 mm.

4. Place two sheets of high density polyethylene on both sides of the glass fiber fabric, as shown in Figure 1.

5. Compress the above combination of sheets under a normal load of 2/9 tons and a temperature of 135 ^{o}C for 5 minutes.

6. Turn off the heater of the mold, turn on the circulation pump and maintain the same normal load until room temperature.

7. Remove the laminated composite from the mold.

8. Cut the tensile test specimen from the laminate according to dimensions specified by ASTM standards [1], as shown in Figure 2.

9. Test the tensile strength of the laminated composite.

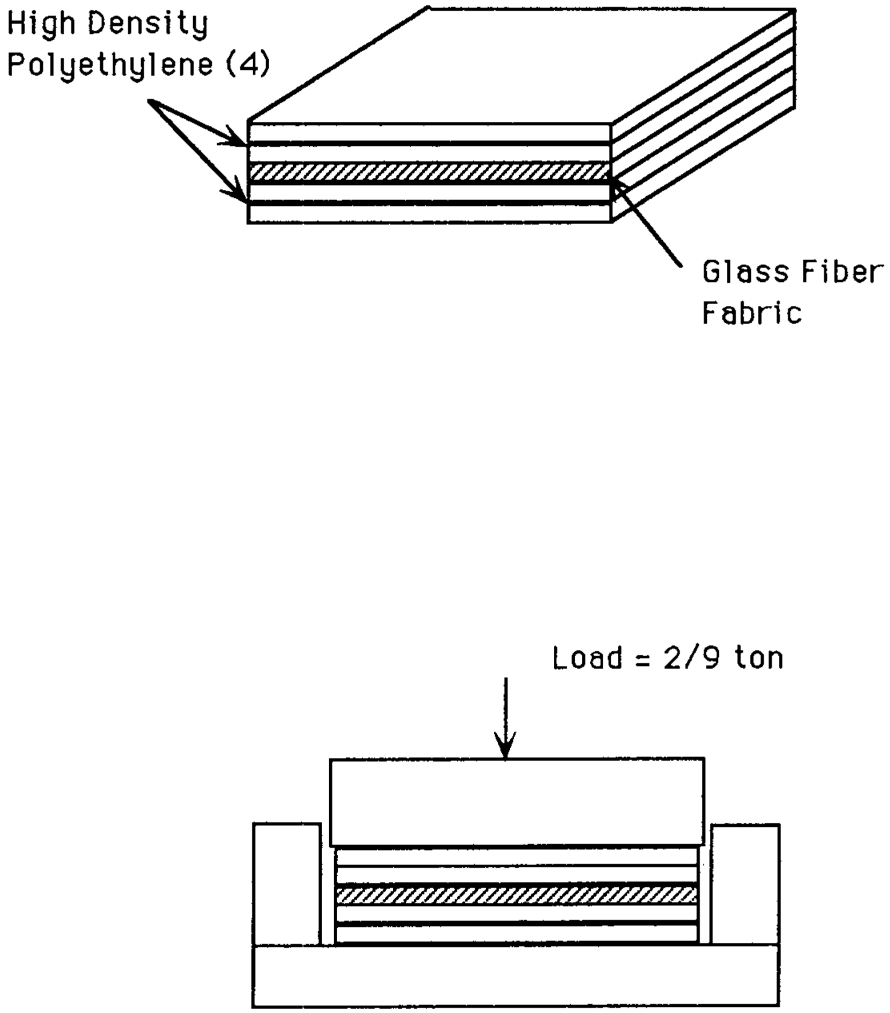

Figure 1. Configuration of laminated composites: two layers of HDPE on both sides of glass fiber.

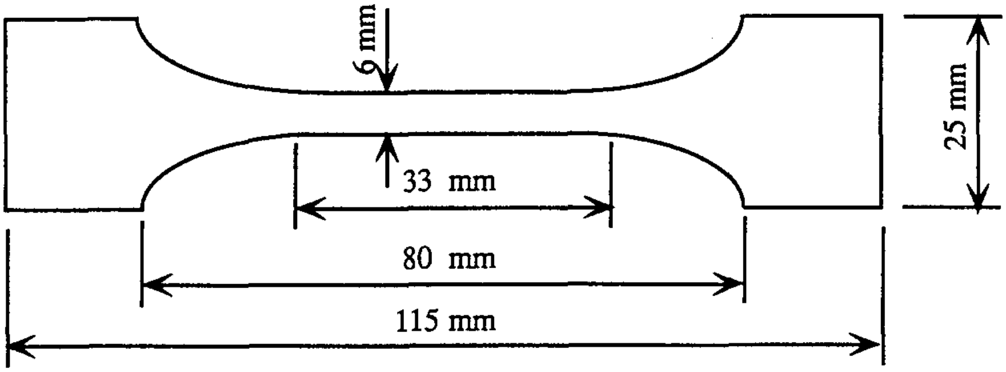

Figure 2. Dimensions of the tensile test specimen per ASTM standard D 638M-89.

SAMPLE DATA SHEETS

Students can record the experimental results using the following table. The data in the table are examples.

Sample #	Forming Load (t)	Temperature (°C)	Specimen Thickness (mm)	Specimen Width (mm)	Max. Load (N)	Tensile Strength (MPa)
1	2/9	135	1.44	6.46	237.9	25.6
2	2/9	135	1.28	6.43	191.9	23.3
3	2/9	135	1.30	6.62	284.7	33.1

INSTRUCTOR NOTES:

Students can perform investigative studies on the effects of forming temperature on the strength of the laminated composite [2,3]. Figure 3 shows a typical relationship between the compression temperature and ultimate tensile strength. As the temperature increases, the strength of laminated composite increases. This increase may be due to several reasons. One of the reasons is that as the temperature increases, better bonding may take place between the glass fibers and the plastic sheets. Another possible reason is that as the temperature increases, the thickness of the laminate decreases, which actually increases the percentage of glass fiber relative to the polymer. Glass fiber is stronger than high density polyethylene matrix. Therefore, material strength increases with increasing temperature.

The reinforcing effect of glass fiber is seen in Figure 3. The strength of recycled high density polyethylene is improved by laminating it with glass fiber. With the improved mechanical properties, the application of recycled plastics could be expanded, and therefore the amount of solid waste going to landfills can be reduced.

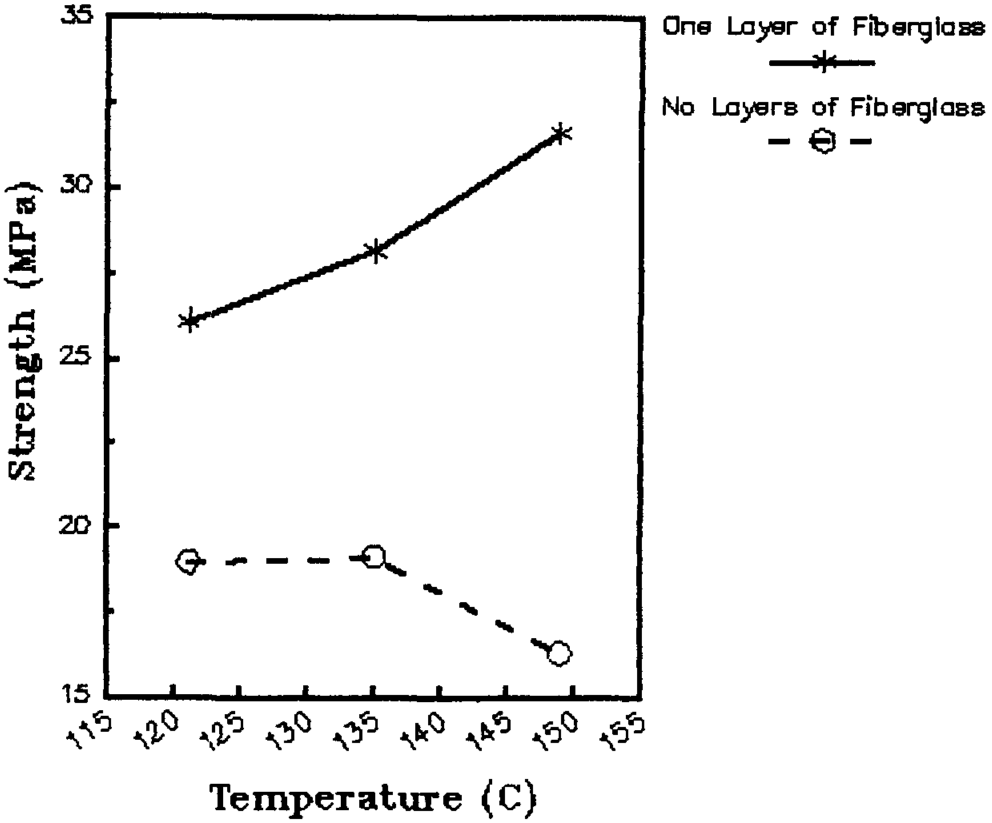

Figure 3. The relationship between the compression temperature and the tensile strength of recycled high density polyethylene laminated with glass fiber and recycled high density polyethylene without reinforcement.

It is also noted that the strength of the laminate increased with increasing temperature. To further investigate the reason, Figure 4 was created to show the relationship between the thermocompression temperature, specimen thickness and strength. It is believed that with increasing temperature the bonding between the fiber and the plastic was improved. As the thickness decreased with the increased temperature, the actual percentage of load-bearing glass fiber in the composite was increased. Therefore, the tensile strength increases with the thermocompression temperature.

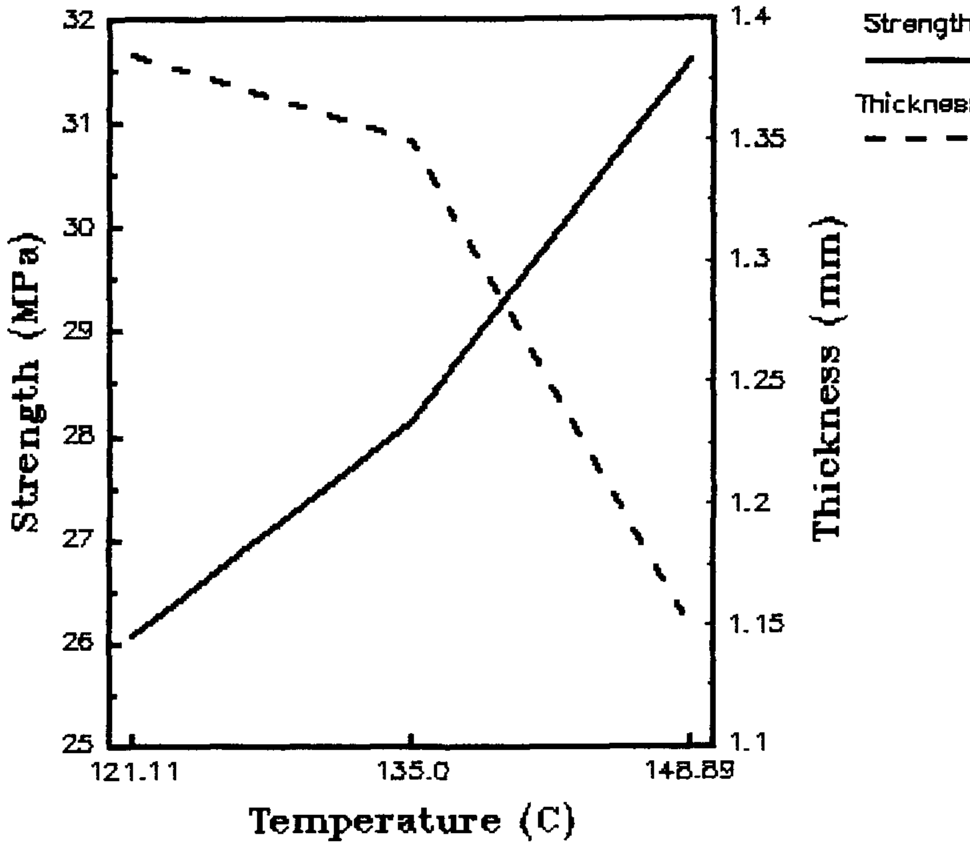

Figure 4. Variation of laminate strength and specimen thickness with thermocompression temperature.

In summary, this experiment provides an understanding of the reinforcing role of continuous fibers in thermoplastic composites. It also illustrates a possible application of recycled plastic, and increases the student awareness of the importance of materials recycling for solving solid waste crisis in the nation.

REFERENCES:

1. ASTM: 1989 Annual Book of ASTM Standards, Vol. 8.01 Plastics (I), American Society for Testing and Materials, Philadelphia, 1989.

2. Carlsson, Leif A.: Thermoplastic Composite Materials, Elsevier Science Publishers, 1991.

3. Agarwal, B.D.; and Broutman, L.J.: Analysis and Performance of Fiber Composites (2nd edition), John Wiley & Sons, Inc., 1990.

SOURCES OF SUPPLIES:

The high density polyethylene is obtained from milk containers which can be recycled by faculty and students without any cost. The cost of glass fiber fabric is the lowest among all engineering fibers, which is \$2.65 - \$6.25/ m^2 (Fibre Glast, 1-800-821-3283).

ACKNOWLEDGMENT:

Part of the financial support for this project is from the Illinois Department of Energy and Natural Resources through the Office of Solid Waste Research at the University of Illinois at Urbana-Champaign. Another part of the material is based upon work supported by the National Science Foundation under Grant No. DUE-9350629.

INEXPENSIVE MATERIALS SCIENCE DEMONSTRATIONS

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INEXPENSIVE MATERIALS SCIENCE DEMONSTRATIONS

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ABSTRACT: Readily available materials can be used to demonstrate such properties as anisotropy, stress concentration effects and composite behavior.

REQUISITE KNOWLEDGE: Curiosity.

KEY WORDS: Composite, Bi-metallic, Filament Tape, Anisotropy.

OBJECTIVES: The purpose of these demonstrations is to illustrate some interesting properties of composites. The following demonstrations have been performed by the author many times and have been received enthusiastically by audiences of all ages.[1,2,3,4]

EQUIPMENT AND SUPPLIES:

1) Filament Tape
2) Sheet of Paper
3) Index Card
4) Bi-metallic Disk *

FILAMENT TAPE: Filament tape is an excellent example of the anisotropy of a composite material. Filament tape consists of aligned parallel threads embedded in a polymer film. One side of this composite is coated with an adhesive. If you try to tear the composite in the direction of the threads the tape will separate very easily; however, if you try to tear the tape in any other direction you will find that it is almost impossible. This is an example of anisotropy or the directional dependence of a property of a material. In other words in this instance the mechanical property of tearing is different according to which direction you try to tear the tape. If you cut several pieces of the tape about 5cm long and stack them together so that different layers have their threads running in different directions, you will find that the anisotropic properties change. This technique is often used in industry to provide a more isotropic material. One major weakness of this type of composite is de-layering or delamination of the stacked layers.[1,2]

* Available from: Edmund Scientific Company
101 E. Gloucester Pike
Barrington, N.J. 08007-1380

SHEET OF PAPER: A sheet of paper can be used to demonstrate the action of an airfoil and also the effect of folding paper. If the sheet of paper is held in front of the mouth and you blow across the top of the paper the paper will rise demonstrating the way that faster moving air on the top of a wing surface than on the bottom of the wing surface causes the wing to experience lift.

Another interesting demonstration with a piece of paper (any size!) is that if you try to continually fold a piece of paper in half, eventually you will not be able to fold it again. You just are not strong enough. Ten folds cannot be done! This demonstrates how mechanical strength can be affected by composite folds.

INDEX CARD: A 3"x5" index card can be used to demonstrate crack deflection in a material. Crack deflection can be engineered into most materials and is not peculiar to composites. If two one inch slits are cut parallel to the 3 inch sides about one inch from either end and then a hole is punched at the bottom of one of the slits, the index card can then be pulled at the 3 inch sides and the index card will invariably fail not where the hole was punched but where the slit alone was made. This is an example of crack deflection. The energy supplied to fracture the card was deflected around the hole and was diminished whereas the same energy supplied to the slit alone was unimpeded and caused a fracture.[1,2]

BI-METALLIC DISK: Some composites are formed by joining two materials either mechanically or by some other means. The bi-metallic disk is a composite formed by joining stainless steel and Invar. The composite behavior of this combination arises from the fact that stainless steel and Invar have different coefficients of expansion. When the disk is received it is concave on one side and convex on the other. If the convex side is rubbed with the thumb and then pressed, the convex side will become concave and vice-versa. If the disk is then placed concave side down on a surface after a short time it will jump off of the surface as it returns to its original state. Some thermostats take advantage of this property to accurately determine small temperature changes. Of course, nothing in a thermostat jumps since the metals joined are strips, are not discs, and one end is fixed.

CONCLUSIONS: The author has had enthusiastic response from audiences of all ages, with each of these demonstrations. Quantitative results can be measured and each demonstration can be expanded and/or altered. You are only limited by your imagination and curiosity.

REFERENCES:

1. Jacobs, James A. and Kilduff, Thomas F.: Engineering Materials Technology, Prentice-Hall 1985.

2. Fellers, William O.: Materials Science, Testing and Properties for Technicians, Prentice-Hall 1990.

3. Spiegel, F. Xavier: Five experiments in Materials Science for less than $10.00, NASA Conference Publication 3151 November 1991 263-265.

4. Spiegel, F. Xavier: Experiments in Materials Science From Household Items, NASA Conference Publication 3201 November 1992 373-375.

ACKNOWLEDGEMENT: The author wishes to thank Mary L. Spiegel for typing this manuscript.

RECYCLING OF AUTOMOBILES AN OVERVIEW

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RECYCLING OF AUTOMOBILES
AN OVERVIEW

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FORD MOTOR COMPANY
DEARBORN, MICHIGAN

SCOPE

TOTAL SOLID WASTE: 326 Billion Pounds In 1988

PLASTICS: 7% By Weight
30% By Volume

Plastics Projected To Increase 10-11% By Weight By The Year 2000.

80-85% Of Solid Waste Is Landfill.

Household Plastic Waste Is Six Times The Automotive Plastics Waste.

PLASTICS WASTE
USA 1990

TOTAL PLASTICS WASTE	**12.0 MILLION TONS**
WEIGHT % OF TOTAL	**7.5**
VOLUME % OF TOTAL	**19.0**
POLYMER PRODUCTION	**30.0 MILLION TONS**

YEAR 2000 PROJECTION
PLASTICS WEIGHT 11%

LAND FILLS IN USA

YEAR	NUMBER
1979	18000
1989	6300
1995	3800

LANDFILL TIPPING FEES

$/TON BY REGION

REGION	AVERAGE	MAXIMUM
NORTHEAST	64.76	120.00
MID-ATLANTIC	40.75	89.00
SOUTH	16.92	40.00
MIDWEST	23.15	50.00
WEST CENTRAL	11.06	13.50
SOUTH CENTRAL	12.50	26.25
WEST	25.63	55.00
NATIONAL	26.56	120.00

SOURCE: NSWMA

DISPOSAL COSTS

	$/TON
LANDFILL	12 --- 120
INCINERATOR	30 --- 70
RECYCLING	100 --- 300

RECYCLING RATES
USA 1992

MATERIAL	RECYCLED, %
ALUMINUM	33
IRON & STEEL	39
PAPER	22
GLASS	8
PLASTICS	1

MAGNITUDE

AUTOMOTIVE RECYCLING

NO. of Cars Shredded	10 Million
Ferrous Metals	11.2 Million Tons
Non Ferrous Metals	0.8 Million Tons
Shredder Residue	3.8 Million Tons 6.7 Million Cu. Yds.
Landfill Cost	\$20 – 100 Per Ton
Heating Value	5000 – 7000 btu/lb

COMPOSITION OF US BUILT CAR

lbs/car

	1990	1980
Steel		
Conventional	1246	1697
High Strength	233	169
Stainless	32	28
Other	53	54
Iron	398	480
Aluminum	158	123
Copper	46	37
Zinc Die Cast	19	21
Powder Metal	23	17
Rubber	128	130
Plastics/Composites	222	196
Fluids	167	181
Other Materials	88	90

Source: Wards Reports

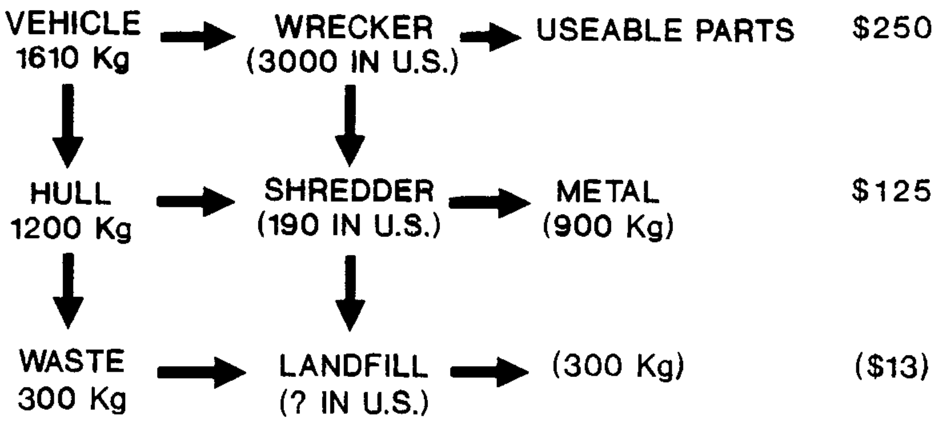
AUTOMOTIVE POLYMER USE

USA (KG PER VEHICLE)

	1989	1995
THERMOSETS		
POLYURETHANE	34.5	44.0
SMC AND OTHERS	13.0	14.8
RUBBER	50.0	47.0
OTHERS	9.1	9.7
THERMOPLASTICS		
POLYPROPYLENE	20.2	21.4
STYRENICS, ABS	13.3	15.5
POLYVINYL CHLORIDE	12.7	13.0
NYLON	9.6	10.8
POLYETHYLENE	8.8	9.6

U. S. VEHICLE DISPOSAL

SCHEMATIC



VEHICLE 1610 Kg → WRECKER (3000 IN U.S.) → USEABLE PARTS $250

HULL 1200 Kg → SHREDDER (190 IN U.S.) → METAL (900 Kg) $125

WASTE 300 Kg → LANDFILL (? IN U.S.) → (300 Kg) ($13)

1988 TOTAL VEHICLES DISPOSED: 12 MILLION

DISMANTLING TIMES

COMPONENT	WT. kg	TIME, SEC.
SIDE MOLDING	0.5	18
DOOR INNER	0.6	32
BUMPER	12.2	90
SEAT	19.5	100
INSTRUMENT PANEL		4800

VEHICLE DISPOSAL

PARTS TAKEN OFF BY DISMANTLERS

- Radiator
- Battery
- Gas Tank
- Tires
- Catalytic Converter
- Usable Parts

FLUID DRAINAGE

- ENGINE OIL
- BRAKE FLUID
- RADIATOR COOLANT
- WASHER
- FUEL

VEHICLE DISPOSAL

SHREDDER FRACTIONS

- Ferrous Metals
- Non-Ferrous Metals
- Fluff
- Dirt

VALUE OF CAR HULK

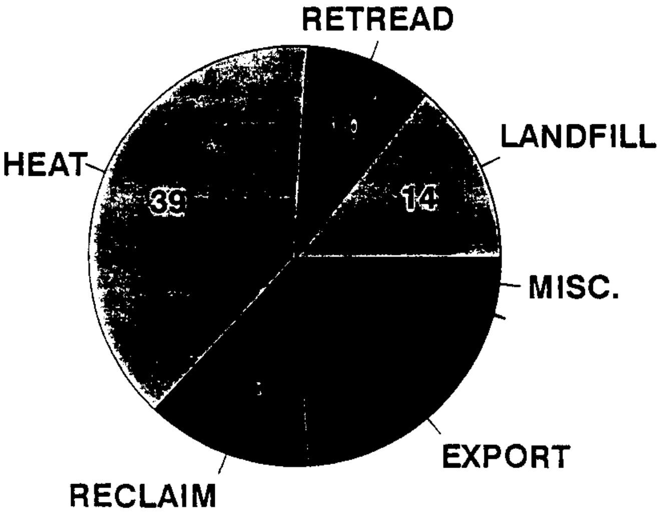
Material	Wt. lbs	Value $
Steel	2250	135.00
Nonferrous Metals	150	18.00
Fluff	750	(47.00)
Freight		(10.00)
Processing		(47.00)
Net Value	3 150	49.00

Source Waxman Metals Group

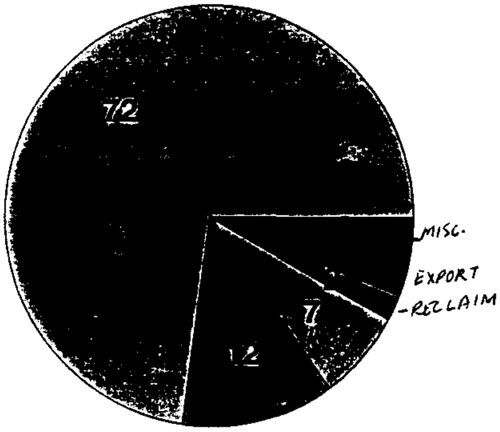
SHREDDER RESIDUE

PLASTICS	**34 %**
FLUIDS	**17%**
RUBBER	**12%**
GLASS	**16%**
OTHER	**21%**

TIRE RECYCLING



JAPAN

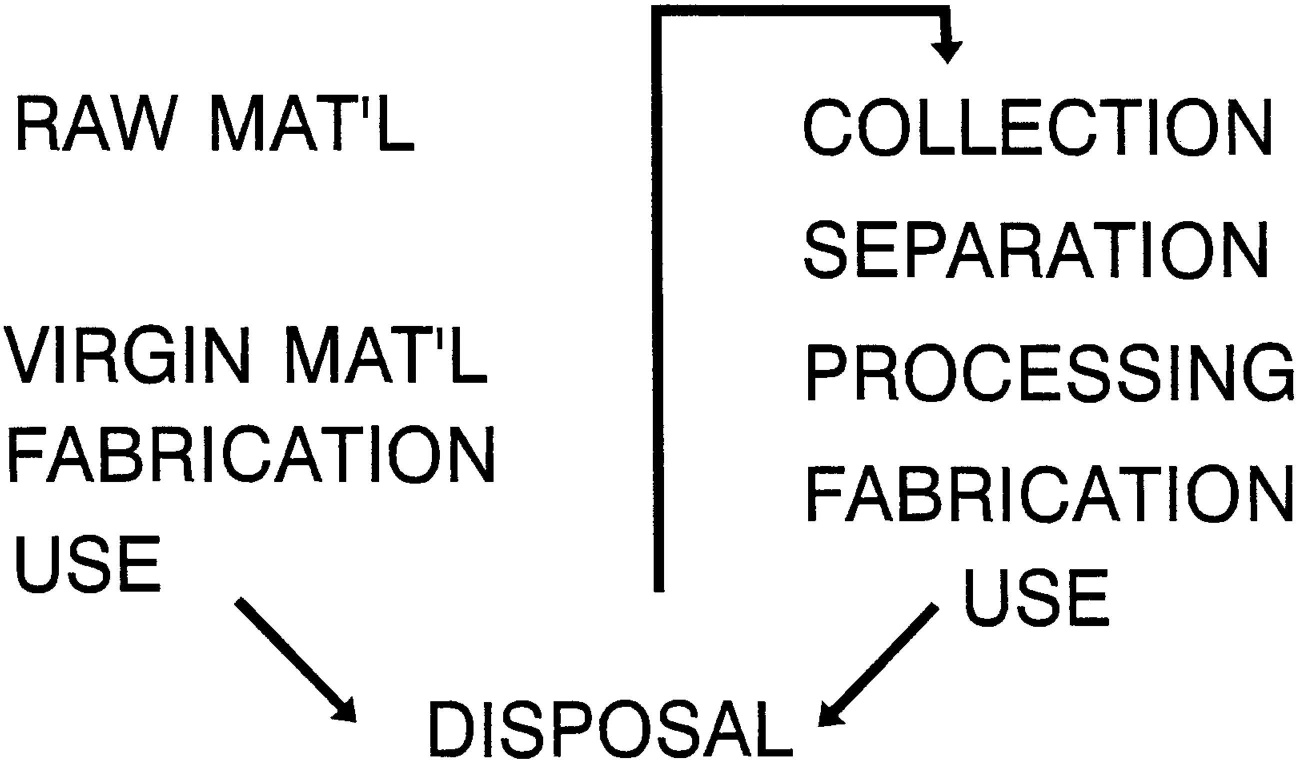


USA

Tire Recycling

NO. of Scrap Tires per yr.	234 Million
Scrap Tires in Environment	3 Billion
Fraction Recycled	20 Percent
Retread	
Reclaim	
Crumb Rubber	
Misc. Products	
Asphalt Paving	
Tire Recycle Incentive Act	S.2462 / HR 4147

COST BALANCE



RAW MAT'L

VIRGIN MAT'L
FABRICATION
USE

COLLECTION
SEPARATION
PROCESSING
FABRICATION
USE

DISPOSAL

Support new recycling efforts

Baucus Offers New Recycling Proposal

WSJ 7/16/93

Trashed

Just as Vice President Gore's disciples are gearing up to copy Germany's ambitious trash recycling program, the Germans themselves are deeming the program there *kaputt*.

Dumpers bury city in worn-out rubber

City caught in trash avalanche

MAILBAG

Incineration no solution

Plastics recycling sham

Hoechst Celanese Suspends a Project On Improving Plastic Recycling Process

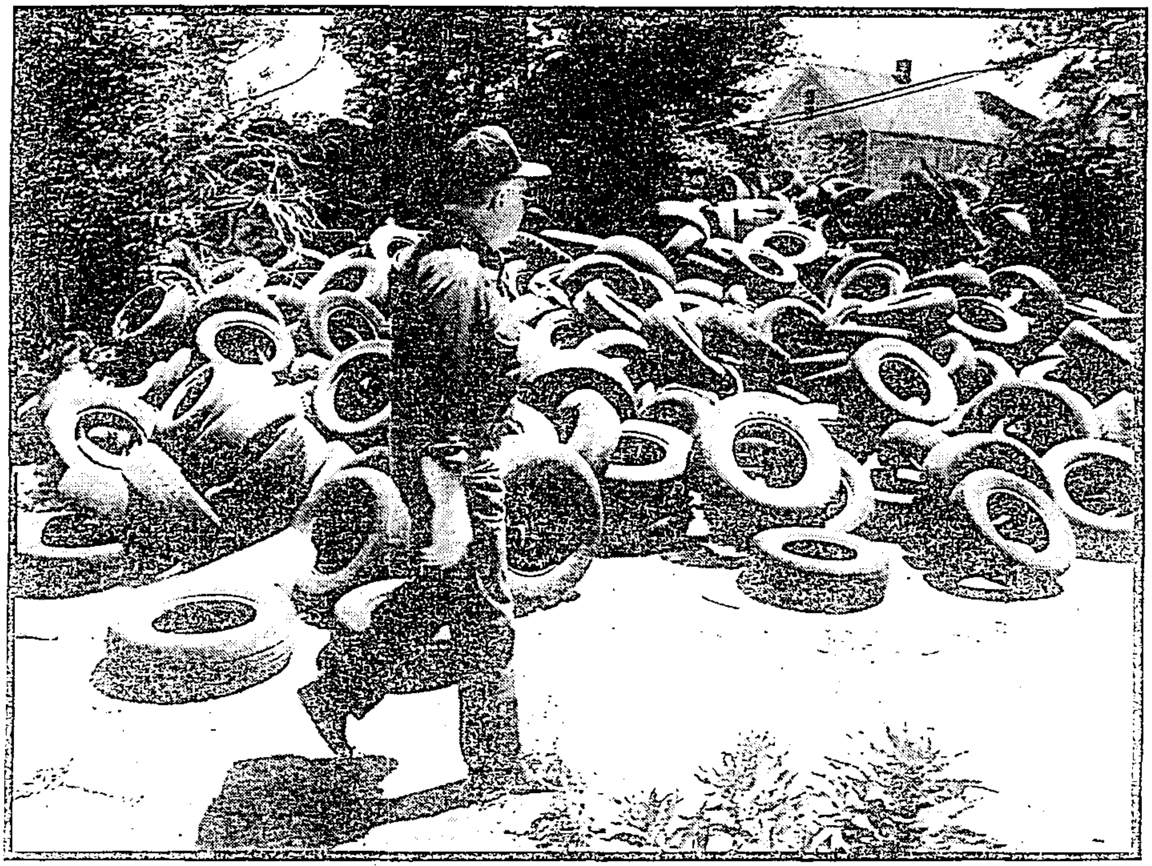
WSJ 6/11/93

WSJ 9/21/92

Lag in Plastics Recycling Sparks Heated Debate

PLASTICS recycling is going strong, according to the Partnership for Plastics Progress, a trade group.

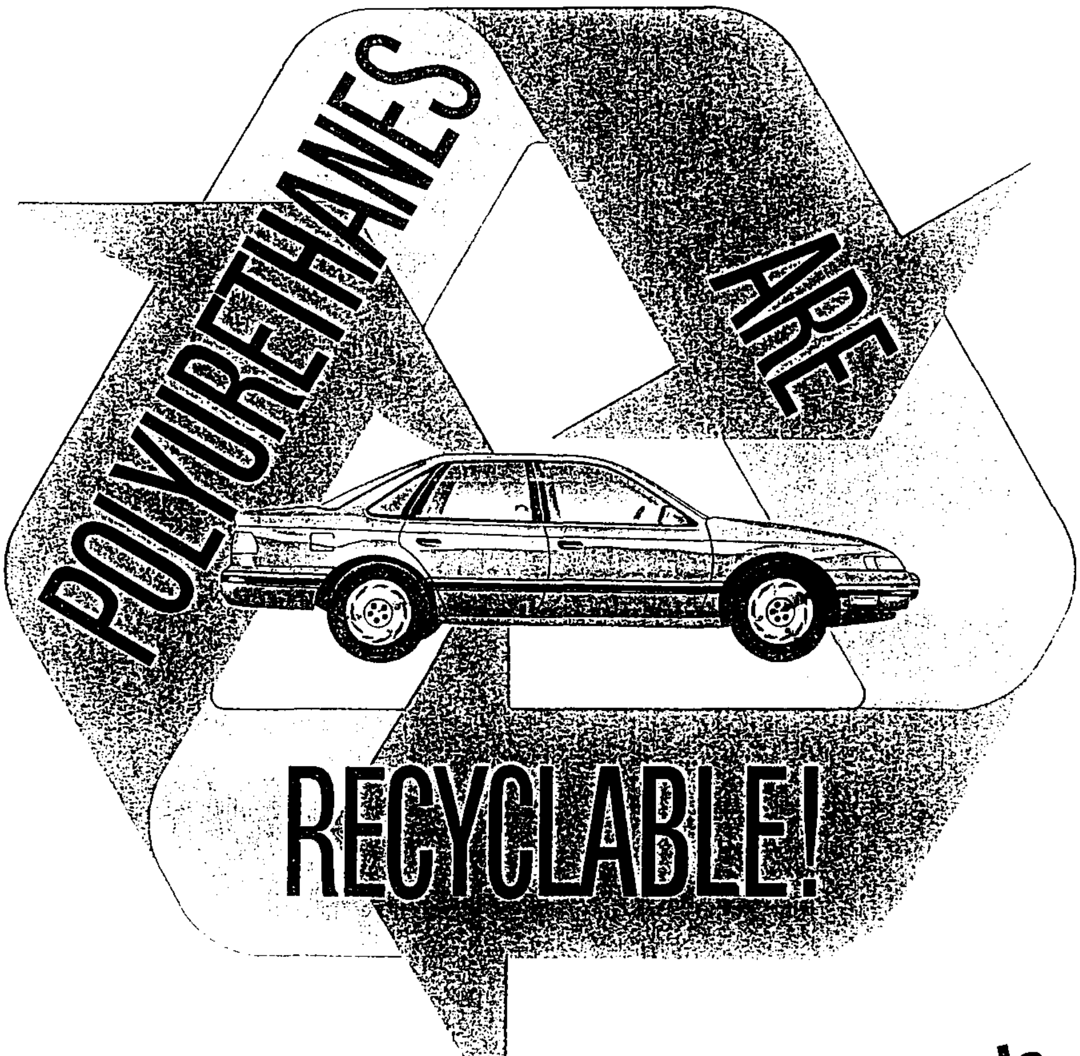
Hogwash, counter environmental groups. "The plastics industry is



DANIEL MEARS/The Detroit News

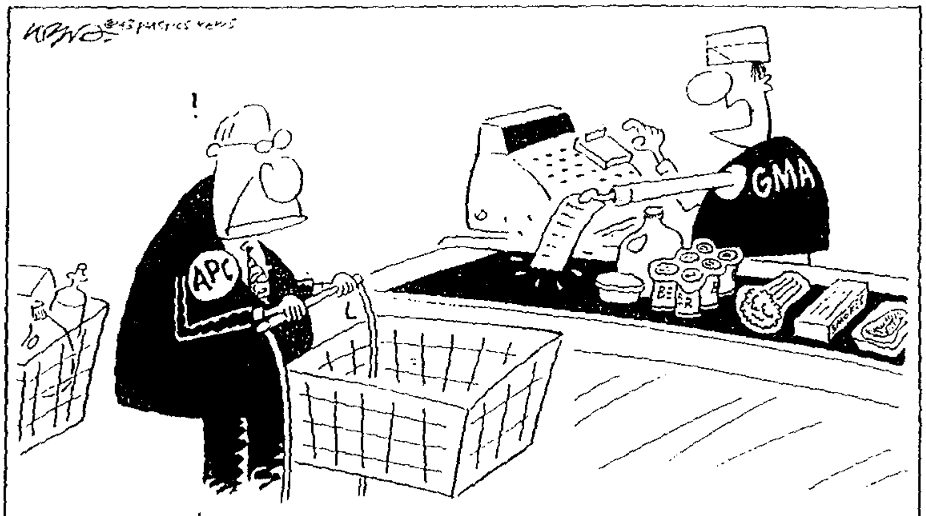
Charles Grimmer, 70, his house visible in the background, walks by the ever-growing mountain of tires dumped in his east side neighborhood near Van Dyke and Mt. Olivet.

Thermoset recycling may be possible



Firm finds way to recycle circuit boards

Is pyrolysis breaking down coalition?



"THAT'LL BE $12.99 FOR THE GROCERIES, AND A COUPLE GAZILLION FOR THE COST OF RECYCLING..."

Trash That Recycling Plan

VIEWPOINT

What is the true cost of recycling?

Here's the inside story on recycling our automobiles

ALBANY TIMES-UNION 8/6/92

veryone's heard of recycling

At Ford, paper and other materials from its many plants and offices across

Automakers want to recycle all of the car

Environmental pressures dictate recycling more auto materials, but costs make total disassembly unlikely anytime soon.

By BRYAN BERRY
Automotive Editor

Auto-part recycling is a top industry priority

Ford Fund Promotes Recycling

Union Carbide Marks Recycling Milestone

AUTOMOTIVE PLASTICS

AUTOMOTIVE PLASTICS: TAKING APART THE PUZZLE

The automotive and plastics industries, jointly and individually, seek greater recyclability of plastic automotive components. The challenge: getting the material out of the car and into the recycling loop.

Recyclers Add Capacity Throughout the Country

GM using recycled SMC in '93 Corvette

By Roger Rowand

around the rear window that sup- activity of the Society of the Plas- ages the company's GM account. environmentally correct parts

NEW corporate facilities programs en cycli Chemical panies account for several major efforts. New recycling plants owned by Union Carbide, Quantum Chemical and Phillips Petroleum add significant

VEHICLE RECYCLING PARTNERSHIP PARTNERS

CHRYSLER MOTOR COMPANY

FORD MOTOR COMPANY

GENERAL MOTORS CORPORATION

VRP Objectives

- **Understand Issues Involved with Vehicle Recycling**
- **Interact with Other Entities Involved in Recycling Research**
- **Conduct Research and Development to Recycle Materials and Components from Scrap Vehicles**
- **Develop Guidelines for Design and Material Selection to Facilitate Recycling**

NEEDED FOR RECYCLING

- RECYCLABLE MATERIALS
- RECYCLING TECHNOLOGY
- DISMANTLING OR SEPARATION
- DESIGN FOR DISASSEMBLY
- MATERIALS IDENTIFICATION CODES
- DEVELOPMENT OF RECYCLING BUSINESS

RECYCLING

THERMOPLASTICS

- Reuse
- Compatiblize
- Reactive Processing
- Incinerate
- Pyrolyse
- Depolymerize

RECYCLING THERMOSETS

- INCINERATION
- PYROLYSIS
- GRIND, USE AS FILLER
- CHEMICAL CONVERSION

AN INTRODUCTION TO STRENGTH OF MATERIALS FOR MIDDLE SCHOOL AND BEYOND

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An Introduction to Strength of Materials For Middle School and Beyond

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KEY WORDS: stress, strain, stability, bend, crush, shear, buckle, axial force, shearing force, bending moment, torque.

PREREQUISITE KNOWLEDGE: The students should be able to follow directions regarding safety of themselves and of those around them. No technical knowledge is required.

OBJECTIVES: To gain familiarity with the basic concepts of strength of materials; strength, stiffness and stability. To observe how materials respond to various types of loading. To promote a better understanding of technical professions.

EQUIPMENT AND SUPPLIES:

For specimens and demonstrations:
(1) Craft ("popsicle") sticks - 50 or more per student
(2) Circular cross-section plastic straws - up to 50
(3) Fishing line - approximately 0.5 m per student of two or more strengths. All should be of a single material.
(4) Wood model glue - at least one tube per student team
(5) Small hex head bolts - preferably of several materials
(6) Foam bar with "stress element" - rectangular cross-section length of foam at least 50mm x 60mm x 250mm
(7) Tongue depressors - at least 4; with pairs glued lengthwise to form double-thickness "beams"
(8) Utility knife - for the instructor's use only!
(9) Flat plastic scale
(10) Safety glasses - one pair per person

For testing:
(1) Digital torque wrench with maximum value storage
(2) Small vise

(In the materials/mechanical testing laboratory)
(3) Universal test machine fixtured with flat plates (about 250 mm x 500 mm)
(4) Metal rods - 3 of same diameter and material, about 175 mm long
(5) Instrumentation to measure test machine loading (such as load cell and strain indicator)

(At any other location)
(3) Lightweight bucket with round metal handle
(4) Metal rods - 2 of diameter similar to the bucket handle
(5) Loading material such as sand or water
(6) Small amount of clay, putty, beeswax, or similar material
(7) Weight scale

INTRODUCTION: Strength of materials is a subject that affects all walks of life, and even children have some inherent qualitative understanding of strength, stiffness, and stability. In a faculty-led workshop, precollegiate students consider how common items withstand loads due to axial forces, shear forces, bending moments, and torques. The effects of geometry and material properties are investigated while students crush, shear, buckle, bend, stretch and twist various items. For students who have studied basic algebra, the simplest equations governing the resulting stresses and deflections (or strains) can be presented. For all students, the general concepts of strength, stiffness, and stability are demonstrated and engineering concerns related to design, testing, and manufacturing are discussed throughout. Experiential learning is emphasized in all activities of the workshop.[1]

PROCEDURE: Note that the foam bar is used to illustrate each of the following concepts as it is presented, and thus, should be near the instructor at all times.

Axial Force and Deflection: Pull on the foam bar to show how tensile axial loads cause it to deflect. For older students, show the following equations:

Axial Stress = P/A
Axial Deflection = PL/AE

where P is axial force, L is specimen length, A is cross-sectional area, and E is Young's modulus.

Next, show the students the lengths of fishing line and point out that the only difference is in cross-sectional area. Have the students slowly pull each line until it snaps. Discuss how the line lengthens before breaking. Have them compare the force needed to break each line. This test demonstrates both strength and stiffness (deflection) for axial loading. (Ideally, there will be a sufficient number of lengths of fishing line for each student to break a pair of lines).

Ask the students for examples of materials that stretch only a little or a lot. Discuss how some of them are used, and explain that designers usually consider both elasticity and strength when selecting a material for a product.

Stability/Buckling: Apply a longitudinal compressive axial load to the foam bar and show that it bows out (buckles) in the center of its length. Apply a similar load to the center of a transverse side to show that the bar crushes when the length and cross section proportions are reversed. Note that the buckling occurs at a much lower load than would be required to crush the material in compression. Provide and discuss Euler's equation for buckling of pinned-end columns:

Critical Buckling Load = $\pi^2\ EI/(L_e)^2$

where E is Young's modulus (material stiffness), I is the area moment of inertia of the cross-section (shape stiffness); and L_e is the effective length of the column.

To illustrate the concept of area moment of inertia (I), apply a similar compressive load to a scale (ruler) of rectangular cross-section, and note that it buckles in the direction with material occurring closer to its center, its weak direction. Show the equation:

Rectangle's area moment of inertia: $I = bh^3/12$

where b is the base of the rectangular cross-section and h is the height. Based on the Euler equation, the scale will buckle in the direction of the lowest area moment of inertia, which is calculated using the smallest side dimension of the rectangular cross-section as the height, h.

Take a single full-length straw; install it between the flat plates on the pre-instrumented universal test machine; and have a pair (or team) of students measure the straw's buckling load. Next, cut a straw in two. Have the students guess if the buckling load should be higher or lower. Have another pair of students measure the load to determine who is correct. Finally, bundle two or more straws together; ask the students how much load should be needed for buckling; and have a third pair measure the buckling load.

The straws typically vary noticeably in geometry and stability. This provides an opportunity to discuss manufacturing issues such as quality control and reasonable tolerances for an item based on its expected use. For example, for some straws, the side may collapse before the length buckles due to variations in material thickness.

Torsion: Start by slowly and carefully twisting the foam bar about its longitudinal axis. Show that the center of the bar does not move, while the corners rotate through a significant angle. While holding one end in place, again twist the bar to demonstrate that the corners at the fixed end of the bar do not move, but the corners at the bar's free end can experience a great deal of deflection. Explain that in addition to motion, the bar is experiencing torsional shear stress, or stress due to slipping between "slices" of bar. At the center of the bar, because no motion occurs, there is no slipping, and therefore, no shear stress. At the corners, the opposite is true, so there can be a large amount of torsional shear stress. The related equations (for a circular cross section rod) are:

Torsional Shear Stress = TR/J

Torsional Deflection = TL/JG

where T is torque, R is the radial distance from center, J is the area polar moment of inertia (shape stiffness), L is the length being twisted, and G is the shear modulus of the material (material stiffness which resists twisting).

Next, move to the vise and tighten about the threads of one bolt. Have a pair of students use the torque wrench to SLOWLY twist the bolthead off while measuring the torque applied. (Note that the torque required will generally be low, so that torque measurement is difficult without the peak hold option on the torque wrench). Use other students to remove the bolt and install one made of another material. Have another pair of students twist the second bolthead. Repeat additional times with bolts of different materials. Compare the torques required for failure, and discuss the benefits and limitations for use of each (i.e., nylon versus steel).

Bending: Begin by showing how the shape of the stress element on the foam bar changes when subjected to three point bending. Bend the foam beam so that its shape appears concave up, and have the students note the changes to the lengths of the lines at the top, bottom and center of the stress element. The top line will appear shorter, so the top of the beam is in compression. The bottom line will appear longer, which is evidence of tension on the bottom of the beam. The line in the center of the stress element will remain unchanged, which relates to zero stress.

The equations used to quantify three point bending are as follows:

Flexural Stress at center = $PLy/4I$

Deflection at center = $PL^3/48EI$

where P is the load applied at the center of the beam, L is the beam length, y is the distance from the neutral axis to the top or bottom surface of the beam, E is the modulus of elasticity and I is the area moment of inertia of the beam cross-section.

For workshops conducted in the mechanical/materials testing laboratory, explain that the students will be building a structure of their choosing during the next fifteen minutes, then bending it until it breaks. The size of the structure is limited by the size of the flat plates used for loading. The load required to break each structure will be recorded, with recognition given to the student(s) who build the strongest structure. Then distribute about 50 craft sticks (and glue) to each student, or team, and let them build their structures. Assist them as needed with ideas, cutting the sticks, etc. While the structures dry (about 5 minutes), proceed with other bending tests.

Two rectangular cross-section beams, each made of a pair of glued tongue depressors, are tested in three point bending to show the effect of the orientation of the cross-section on beam deflection and load carrying capability. Bend each to failure using three point bending on the universal test machine, fixtured to obtain a concave up shape as shown with the foam beam. Measure the load required to bend about the weak axis and about the strong axis. Discuss the concept of area moment of inertia (shape stiffness) again and point out how structures are designed to take advantage of this property.

When the glue on the students' craftstick structures is dry and "cured," have each install their structure in the universal testing machine for three point bending (or crushing, if more appropriate for the structure). Assist them in slowly loading the structures until failure occurs. Record the peak load each structure withstands, and recognize the student(s) who designed and built the strongest structure. Discuss how construction techniques affected the structures' ability to withstand load, just as they can affect the many structures used in daily life.

For workshops held outside of the mechanical/materials testing laboratory, it may not be possible to test student-built structures. As an alternate method of illustrating three-point bending, tongue depressor "beams" can be mounted across two metal rods, with an empty bucket across the center of each "beam." Load can be applied by slowly adding water or sand to the bucket until the "beam" fails. The bucket can then be weighed to determine the load required to break beams of various cross-sections. Preparation for this variation includes setting up a test frame and gluing tongue depressor "beams" of several thicknesses. The test frame can be set up many ways. A simple method is to move tables close together, lay one metal rod on each, and apply a small amount of clay or putty to prevent rolling. Sufficient beams should be prepared to facilitate a discussion of shape stiffness, demonstrate how shape stiffness affects the amount of load required for failure, and to permit small teams of students to each test at least one "beam."

Conclusion: Summarize the basic concepts of strength of materials: strength, stiffness, and stability. Review the types of loading applied, and how items responded to each. Encourage the students to provide examples of each, and to think about how the work of engineers and technologists affects them every day. Experiences like these illustrate the practical applications of mathematics and the physical sciences while helping students understand why "to engineer is human."[2]

INSTRUCTOR NOTES: Students are more interested, and retain more of what they are taught when they are actively participating in the learning process.[3] If possible, students should do the setup and testing themselves. In order to facilitate that type of participation, the group size needs to be limited to about 8 students per instructor. If two instructors lead the workshop, a maximum of 15 students is recommended.

Safety must always be a top priority, and students should be warned about:

(1) Continued inhalation of fumes from the glue can cause a variety of physical problems including dizziness and difficulty breathing.

(2) The typical universal testing machine is capable of applying great force onto fingers or whatever gets in its path.

(3) When twisting the bolts, it is possible that the bolthead and/or torque wrench could fly loose.

(4) Safety glasses should be worn during all tests.

REFERENCES:

1. National Council of Teachers of Mathematics. (1989). Curriculum and Evaluation Standards For School Mathematics. The National Council of Teachers of Mathematics, Incorporated, Reston, VA.
2. Petroski, H. To Engineer is Human: The Role of Failure in Successful Design. St. Martin's Press.

3. Resnick, L. B. (1987). Education and Learning to Think. Washington, D.C., National Academy Press, 1987.

SOURCES OF SUPPLY: The local hardware store or craft store will stock most items required for the workshop. It may be necessary to obtain tongue depressors from a pharmacy, and straws from a supermarket.

ACKNOWLEDGEMENT: The thoughtful and timely assistance of James D. Osborne, department mechanician, in the development of this workshop is gratefully acknowledged.

SIMULATION OF MATERIALS PROCESSING: FANTASY OR REALITY?

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Simulation of Materials Processing:
Fantasy or Reality?

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KEY WORDS: computer-aided design (CAD), microelectronics, oxidation, computer simulation.

PREREQUISITE KNOWLEDGE: The students should understand the concepts associated with the oxidation of silicon as applied to integrated circuit fabrication. In addition, the students should be familiar with the operating system of the computer, which will be executing the software.

OBJECTIVES: The objectives of this experiment are the following:

1. Introduce the general topic of simulation of materials processing as it relates to the fabrication of microelectronic circuits,

2. Familiarize the students with the use of the specific process simulation tool, which they will be required to use during subsequent semiconductor experiments, and

3. Introduce three independent measurement techniques which are used to characterize oxide thickness and to verify computer simulations.

EQUIPMENT AND SUPPLIES: The equipment required to implement this experiment is listed in Table I. The oxidation furnace is used to oxidize the silicon wafers. The reflectance spectrophotometer, ellipsometer and profilometer are required to measure the thickness of the resulting oxides.

The *TSUPREM-4* (Technology Modeling Associates, Inc., Palo Alto, CA) simulation software[1] is required. The students will use this software to simulate the various oxidation furnace conditions utilized during the oxidation process.

The required processing supplies are those commonly found in a silicon-based semiconductor research laboratory. These supplies are listed in Table II. All of the chemical supplies should be standard electronics grade. The specific silicon wafers utilized in this experiment are prime grade, (100)-oriented, p-type (boron doped) with a nominal resistivity of 6 - 18 Ω·cm.

PROCEDURE: The general procedure of this experiment consists of four steps: 1) simulation of the oxidation process, 2) thermal oxidation of silicon wafers, 3) measurement of the resulting oxide thickness, and 4) comparison of the simulation to the actual results of the oxidation.

1. Simulation of Oxidation Process.

To accomplish the computer simulations, the students are provided a user's guide[2] and an introduction to the use of the *TSUPREM-4* software. The user's guide briefly describes the fundamental processes which can be simulated with the software. Practical information concerning the trade-offs between accuracy and simulation time, as well as the basic structure of the simulation input files, is presented. The introduction is accomplished by executing a simple example with the students observing the flow of the simulation process. A commentary is provided during the simulation process.

After familiarizing themselves with the software, the students simulate the thermal oxidation of silicon for various process conditions. The various oxidation conditions are listed in Table III. The wet oxygen ambient consists of atmospheric-pressure oxygen bubbled through water containing 2% (by volume) HCl. In addition, the furnace pressure is maintained at one atmosphere during all oxidation conditions. A sample simulation file is presented in Figure 1, and portions of the generated output are depicted in Figures 2 and 3.

2. Thermal Oxidation of Silicon Wafers.

The students implement the thermal oxidation which they previously simulated. To accomplish this process, the students must clean the wafers and expose them to the appropriate oxidation conditions.

(A) Cleaning:

Since undetectable traces of contaminants can be catastrophic to both the wafer and the oxidation furnace, proper cleaning is essential before exposing the wafers to the elevated temperatures in the oxidation furnace.

(1) The wafers should be immersed in a modified piranha solution (H_2SO_4 : H_2O_2, 3:2). The wafers should be cleaned for 20 minutes and rinsed thoroughly with deionized (DI) water to at least a 10 MΩ·cm standard.

(2) Then, after blowing the wafers dry with N_2, they should be transported to the oxidation furnace in a covered container.

(B) Oxidation:

Two types of oxygen ambients (dry and wet) will be used by the students. The wet ambient should be realized by flowing the dry oxygen through a 2% (by volume) solution of HCl and DI water, which is heated to 95°C.

(1) The wafers should be loaded into a quartz oxidation boat.

(2) A furnace setting of 900°C and an oxygen flow of one liter per minute (lpm) should be verified.

(3) The wafer boat should then be slowly pushed (1 inch per minute) into the furnace's center hot-zone. If a wet oxidation is to be done, the wafers should be exposed to the dry oxygen for 5 minutes prior to flowing the wet oxygen through the furnace. This brief dry oxidation will produce an initial thin oxide. This initial oxide will prevent the introduction of defects in the silicon wafer which may be caused by the exposure to HCl.

(4) Upon completion of the oxidation, the wafer boat should be withdrawn from the furnace at the rate of 1 inch per minute. The wafers should be removed from the boat and placed in a covered container.

(5) The oxidation process is repeated for each of the process conditions.

3. Measurement of Oxide Thickness.

After completing the oxidation, the students measure the oxide thickness which was produced during the various processing conditions. The thickness of the oxides is determined by using a reflectance spectrophotometer, an ellipsometer and a profilometer. The reflectance spectrophotometer and ellipsometer introduce the students to two types of non-contact, nondestructive measurement techniques, whereas the profilometer introduces the students to a destructive measurement technique.

While reflectance spectrophotometric and ellipsometric measurements do not require specific post-processing of the oxidized wafers, profilometry requires the fabrication of mesas within the oxide layer to be measured. This requirement makes profilometry destructive. To fabricate the mesas, a simple multistep process is used. Portions of the oxide are selectively etched. The remaining oxide forms mesas of silicon dioxide upon the silicon wafer's surface.

These mesas are subsequently measured with the profilometer to determine the oxide thickness. The mesa fabrication consists of the following steps:

(A) Masking:

(1) Waycoat HR200 negative photoresist is dabbed onto the oxidized wafer's surface. A sterile cotton Q-tip is used to apply the photoresist. The diameter of the dots or speckles of photoresist should be less than 5 mm.

(2) The patterned wafers are placed in an oven at 135°C for 20 minutes to evaporate the solvents from the photoresist and harden it. If an oven is not available, a hot plate can be substituted.

(B) Etching:

(1) After allowing the wafers to cool, the wafers are etched using a buffered HF (NH_4F : HF, 4:1) solution. Fresh etchant should be mixed sufficiently early to let it stabilize (2 hours) before it is used. Nevertheless, it must be used within 6 hours after mixing. The wafers are etched until the exposed portions of the wafer become hydrophobic and all unmasked portions of the oxide are completely etched away. Students can anticipate an etch rate of 110-150 nm/min.

(2) The etched wafers are rinsed in DI water for 2 minutes after etching.

(C) Stripping:

After etching, the remaining photoresist must be stripped from the wafers' surface.

(1) The wafers are immersed in a modified piranha solution (H_2SO_4 : H_2O_2, 3:2). The wafers should be cleaned for 20 minutes and rinsed thoroughly with DI water.

(2) Then, after blowing the wafers dry with N_2, the resulting mesas of silicon dioxide can be characterized by profilometry.

The results of reflectance spectrophotometer, ellipsometer, and profilometer measurements of a typical experimental trial are depicted in Table IV. The reflectance spectrophotometer results are the average of ten measurements of two similarly oxidized wafers. Likewise, the ellipsometer results are the average values of two similarly oxidized wafers. The profilometer results are the average of multiple mesas on one of the two wafers, which were previously characterized during ellipsometry.

4. Analysis of the Results.

The students compare the measured oxide thickness to the simulations of the actual fabrication process. In their analysis, the students use estimated oxide thicknesses from the *TSUPREM-4* results and oxidation charts from textbooks. As part of their analysis, the students need to consider ease of use, accuracy, and precision of the computer simulations.

The students should "discover" the utility of CAD tools designed to model technological processes. For example, the wet oxidation is not readily determined from simple oxidation charts. Table V depicts the information which can be found in most oxidation charts. While the use of HCl in the steam yields oxides with superior electrical properties, the HCl modifies the oxidation rate of silicon. Also, the initial dry oxidation for 5 minutes (before introducing the steam) produces an initial thin oxide which is not accounted for in most charts.

Hence, multi-step processes are more easily handled with CAD tools. Without CAD, the students must compute the expected oxide thickness by consulting numerous tables of parameters, such as linear and parabolic rate constants. This process can be tedious, repetitive, and prone to errors; thus, the students realize the value of CAD.

In addition, the use of charts and other graphics requires interpolation of parameters to account for the actual process conditions. Small deviations can affect the accuracy of the process. For example, a deviation of 5 minutes and 10^{o}C may yield an error of more than 10 nm. This error can be significant when working with typical microelectronic devices which have critical oxide thicknesses of about 20 nm. These small deviations are easily entered into CAD tools, whereas the interpolation errors in simple charts can be as large as the process deviations. These errors can affect accuracy and precision.

The accuracy and precision of a calibrated simulation are readily obvious to the students after they compile figures or tables comparing simulations to measurements. Examples of typical results are depicted in Figures 4-7 and Table VI. The dry oxidation results are depicted in Figures 4 and 5. Additionally, the wet oxidation results are illustrated in Figures 6 and 7. Within each figure, the simulation results are compared to the measured reflectance spectrophotometric results. The mean measured value is depicted with a bar, while the maximum and minimum measured values are depicted with unconnected cross symbols. The corresponding simulated values are depicted with a connected line.

The typical magnitudes of the deviation among the simulations and measured results are depicted in Table VI. As depicted in Table VI, the deviations among the simulations and the nondestructive techniques of measurement are consistently less than four percent. From simple analyses, such as these, it should be readily apparent to the students that a great deal of variability exists. However, it should also be evident to the students that CAD produces a result comparable to the measured values with relative ease of use.

SAMPLE DATA SHEETS: Self-Evident.

INSTRUCTOR NOTES: This experiment introduces students to the application of computer-aided design (CAD) and analysis of materials processing in the context of integrated circuit (IC) fabrication. The fabrication of modern ICs is a complex process which consists of several sequential steps. These steps involve the precise control of processing variables such as temperature, humidity, and ambient gas composition. In essence, the particular process employed during the fabrication becomes a "recipe." Due to economic and other considerations, CAD is becoming an indispensable part of the development of new recipes for IC fabrication.

In particular, this experiment permits the students to explore the CAD of the thermal oxidation of silicon. After the students simulate dry and wet oxidation processes, they implement the simulated processes and measure the thickness of the oxide actually realized with their recipes. The students conclude the experiment by reconciling the differences between the simulated and real oxide.

The students should be divided into at least four groups. The groups are represented in Table III. By assigning each group a particular oxidation condition, the individual groups of students can complete the time consuming oxidations relatively quickly (during a single laboratory period of three to four hours). Another laboratory period is required to complete the measurements of the oxidation thickness. Upon completing the oxidations in the laboratory, the four groups can share their measured results. Another laboratory period is required to complete the simulations. The entire experiment can be easily incorporated within a lecture course on integrated circuit technology. The integration of the experiment within a technology course can provide a hands-on realistic appraisal of the utility of CAD tools in materials processing.

The students should be cautioned about the hazards. When working with the acids, the students should wear eye protection and gloves. Particular care should be exercised while working with HF. When working with the ellipsometer, the students should be warned not to stare into the laser.

Upon completion of the experiment, the students gain an insight into the role of CAD in material processing. Hence, they can begin to discern when CAD may be intelligently applied to processing so as not to obfuscate, but to enlighten. That is, the student will be able to distinguish between the fantasies and the realities associated with semiconductor-material process simulation. In addition, the student will be prepared to apply the specific process simulator in other academic studies.

REFERENCES:

1. User's Manual: *TMA TSUPREM-4 Two-Dimensional Process Analysis Program*, Version 9035. Technology Modeling Associates, Inc., 300 Hamilton Avenue, Third Floor, Palo Alto, California 94301, September 1990.

2. Thomas Jenkins: *AFIT User's Guide to TSUPREM4*, Student Handout, Department of Electrical and Computer Engineering, Air Force Institute of Technology, Wright-Patterson Air Force Base, Ohio 45433, December 1991.

3. Roy Colclaser: *Microelectronics Processing and Device Design*, John Wiley & Sons, New York, 1980, pp. 92-93.

SOURCES OF SUPPLY: Other than the photoresist, all laboratory chemicals required for this experiment are available from Mallinckrodt, Inc., Science Products Division, Paris, Kentucky. The photoresist is available from Olin Hunt Specialty Products, Inc., 5 Garret Mountain Plaza, West Paterson, New Jersey. The silicon wafers can be obtained from Ziti, Inc., 14755 Preston Road, Suite 421, Dallas, Texas.

Table I. Laboratory Equipment

Diffusion/Oxidation Furnace
Model 4100, Thermco Products Corp., Orange, CA

Ellipsometer
Model L117, Gaertner Scientific Corp., Chicago, IL

Oven
Model Imperial IV 3450M, Lab-Line Instruments, Melrose park, IL

Profilometer
Model Dektak IIA, Sloan Technology Corp., Santa Barbara, CA

Reflectance Spectrophotometer
Model LTS-M/SP, Leica Inc., Deerfield, IL

Table II. Laboratory Supplies

Ammonium Fluoride	NH_4F	40%	(mixed with H_2O)
Hydrochloric Acid	HCl	37%	(mixed with H_2O)
Hydrofluoric Acid	HF	49.2%	(mixed with H_2O)
Hydrogen Peroxide	H_2O_2	30%	(mixed with H_2O)
Sulfuric Acid	H_2SO_4	96%	(mixed with H_2O)
Waycoat HR200 Negative Photoresist			

Table III. Conditions of Thermal Oxidation

	Time (min)	Temperature (°C)	Oxygen Ambient
Group A			
	45	900	dry
	60	900	dry
	120	900	dry
Group B			
	45	1100	dry
	60	1100	dry
	120	1100	dry
Group C			
	45	900	wet
	60	900	wet
	120	900	wet
Group D			
	45	1100	wet
	60	1100	wet
	120	1100	wet

Table IV. Typical Measurements

	Time (min)	Thickness (nm)		
Dry Oxidation:		Reflectance	Ellipsometer	Profilometer
900°C	45	23	23	30
	60	29	29	26
	120	43	42	64
1100°C	45	103	103	147
	60	137	141	186
	123	212	214	253
Wet Oxidation:				
900°C	45	104	102	139
	60	135	134	177
	120	235	235	298
1100°C	46	485	490	578
	63	591	594	733
	124	839	846	990

Table V. Oxide Thickness Estimated From Oxidation Charts[3]

Time	900°C Dry	900°C Wet	1100°C Dry	1100°C Wet
45 min	29 nm	150 nm	110 nm	550 nm
60	31	200	130	650
120	50	300	190	900

Table VI. Deviations Among Simulated and Measured Results

	Time (min)	Deviation (%)			
Dry Oxidation:		Reflectance	Ellipsometer	Profilometer	Chart
900°C	45	2.1	2.1	28	23
	60	1.0	1.0	9.4	8.0
	120	1.2	3.4	47	15
1100°C	45	0.0	0.0	43	6.8
	60	0.29	2.6	35	5.4
	123	1.9	2.9	22	8.6
Wet Oxidation:					
900°C	45	0.19	1.7	34	45
	60	1.7	0.98	33	51
	120	0.56	0.56	28	28
1100°C	46	0.16	0.86	19	13
	63	2.2	2.7	27	12
	124	0.12	0.71	18	7.1

```
$ TMA TSUPREM-4 -- Oxidation Simulations for NEW'93
option    device=x
$ Define the simulation grid and initialize
line x    loc=0.0  spac=0.5
line x    loc=1.0  spac=1
line y    loc=0    spac=0.01
line y    loc=0.5  spac=0.1
line y    loc=1.0  spac=0.1
line y    loc=2.0  spac=1.0
line y    loc=10.0  spac=5.0
initialize <100> boron=3E15
$ Plot the initial grid
select    title="Initial Grid"
plot.2D   grid  y.max=10
pause
$ Select oxidation model
method    vertical  grid.oxi=4.0
$ thermal oxidation
diffusion  temp=630 time=15 t.final=930 dryo2
diffusion continue temp=930 time=120  pressure=1.0 dryo2
diffusion continue temp=930 time=15 t.final=630 dryo2
$plot oxide-mask thickness
select   z=1
print layers x.v=0
pause
select title="Oxidation Results"
plot.2d y.max=1
color oxide color=4
color silicon color=2
stop
```

Figure 1. *TSUPREM-4* sample input file to simulate thermal oxidation.

```
Num   Material    Top          Bottom       Thickness    Integral
1      oxide      -0.0251      0.0184        0.0435      4.35e-06
2      silicon    0.0184       200.0000     199.9816     1.9998e-02
```

Figure 2. Portion of simulation output: printing oxidation thickness.

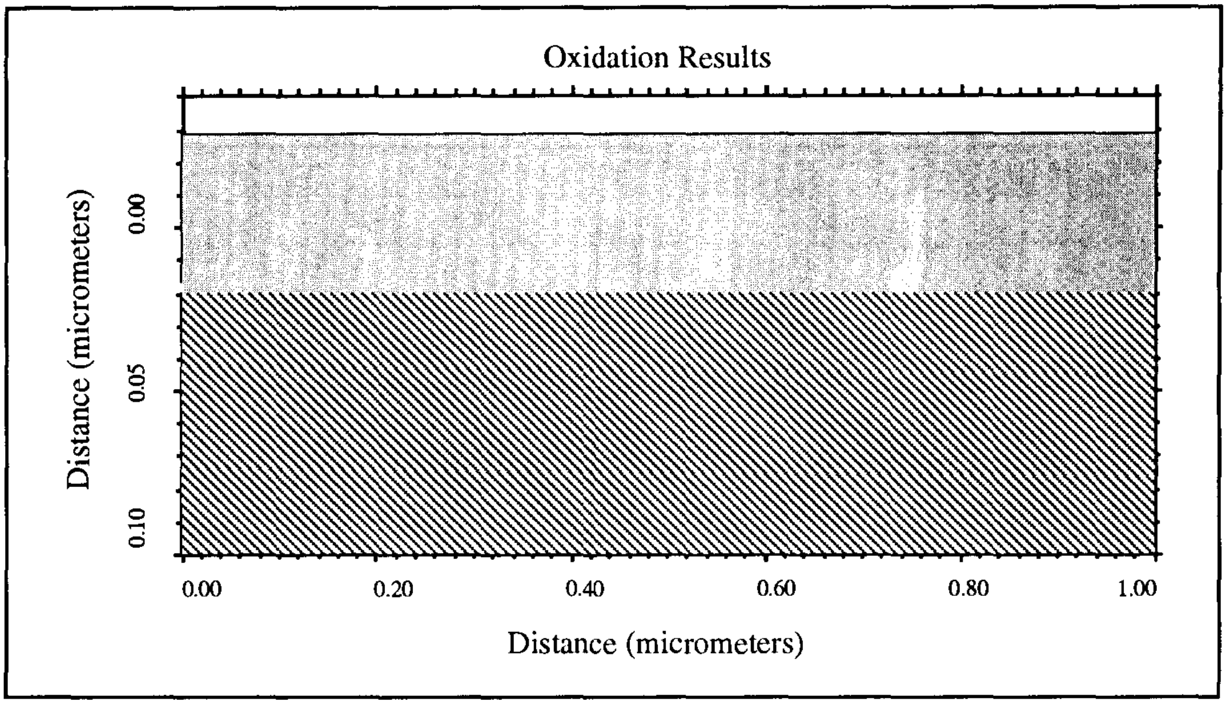

Figure 3. Portion of simulation output: two-dimensional view of oxidized wafer.

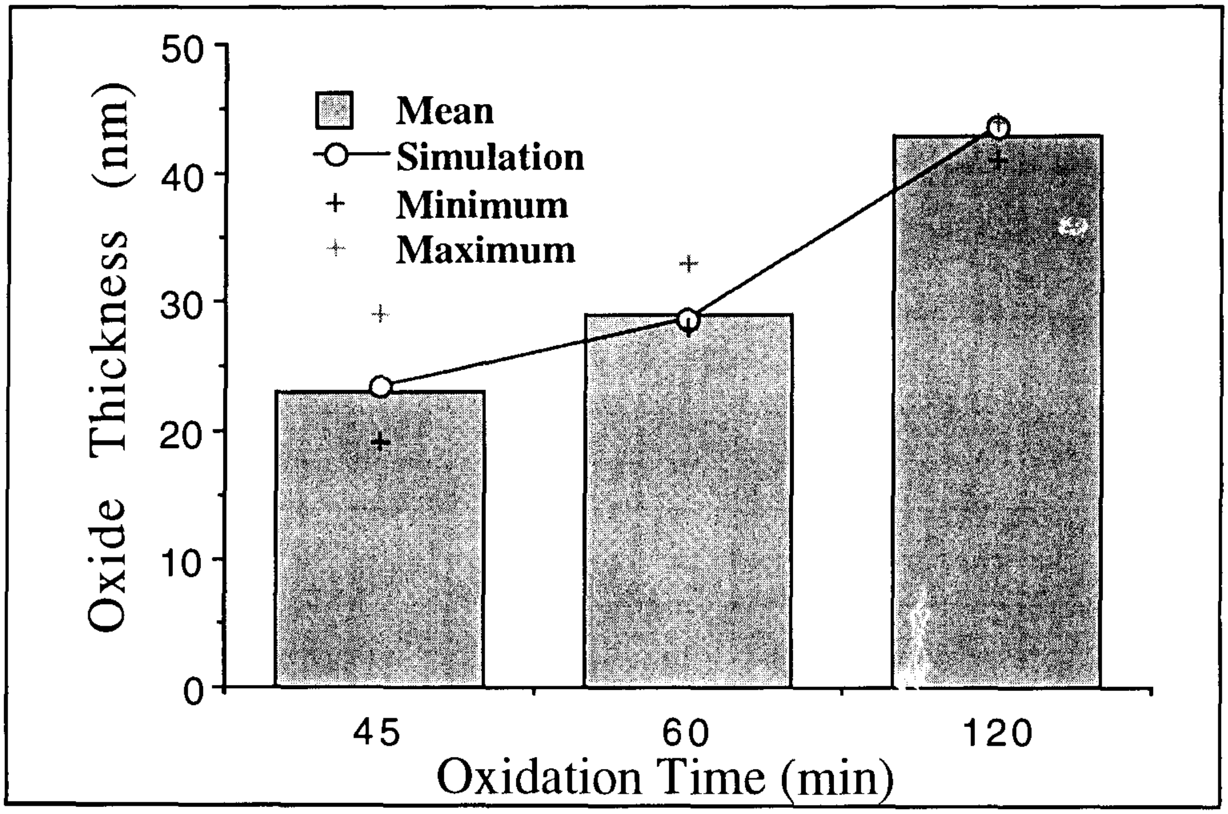

Figure 4. Dry thermal oxidation of silicon at 900°C.

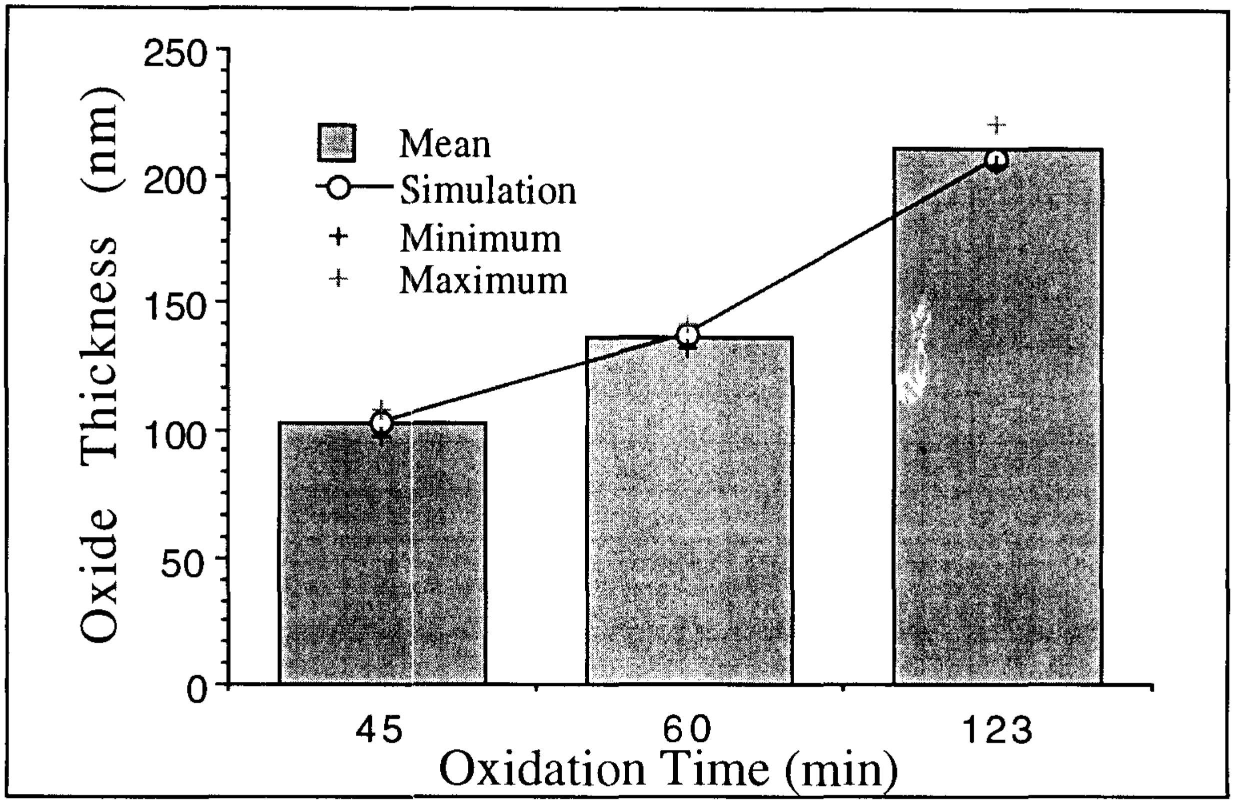

Figure 5. Dry thermal oxidation of silicon at 1100°C.

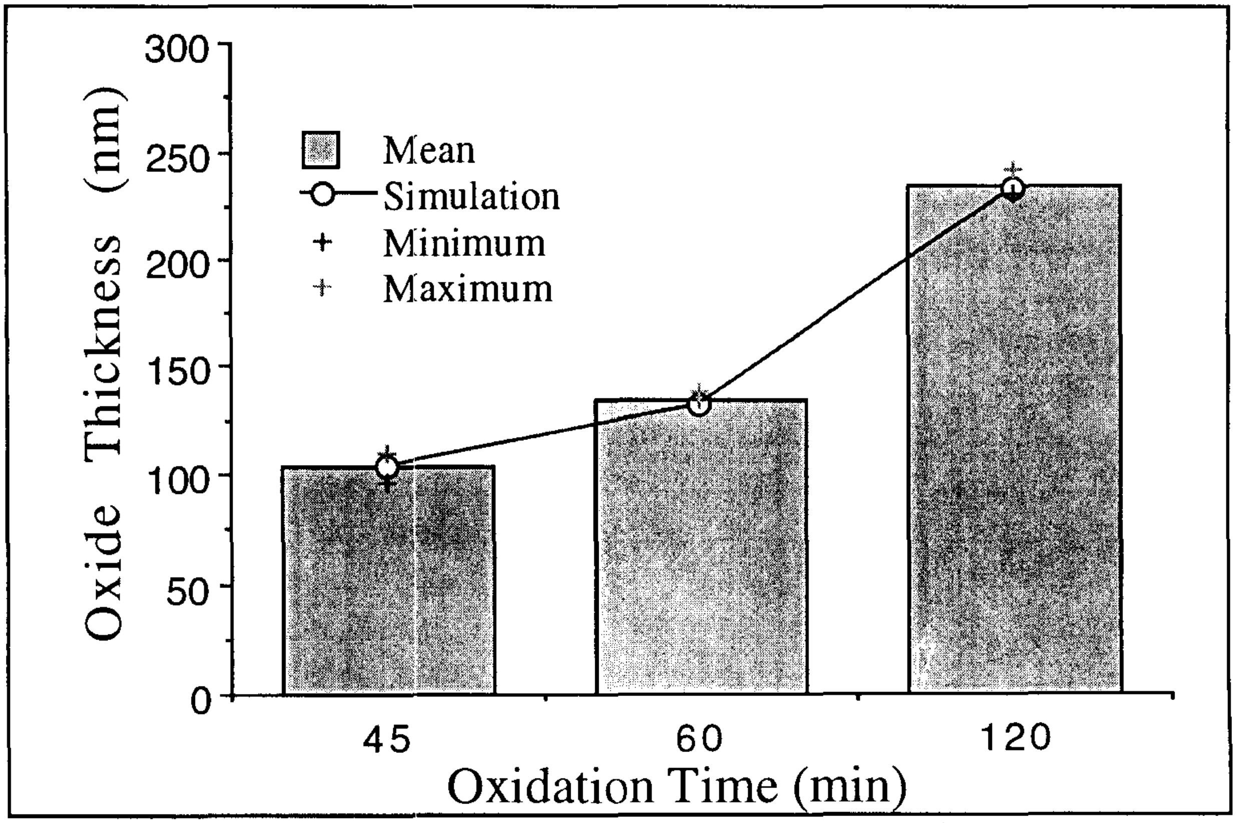

Figure 6. Wet thermal oxidation of silicon at 900°C.

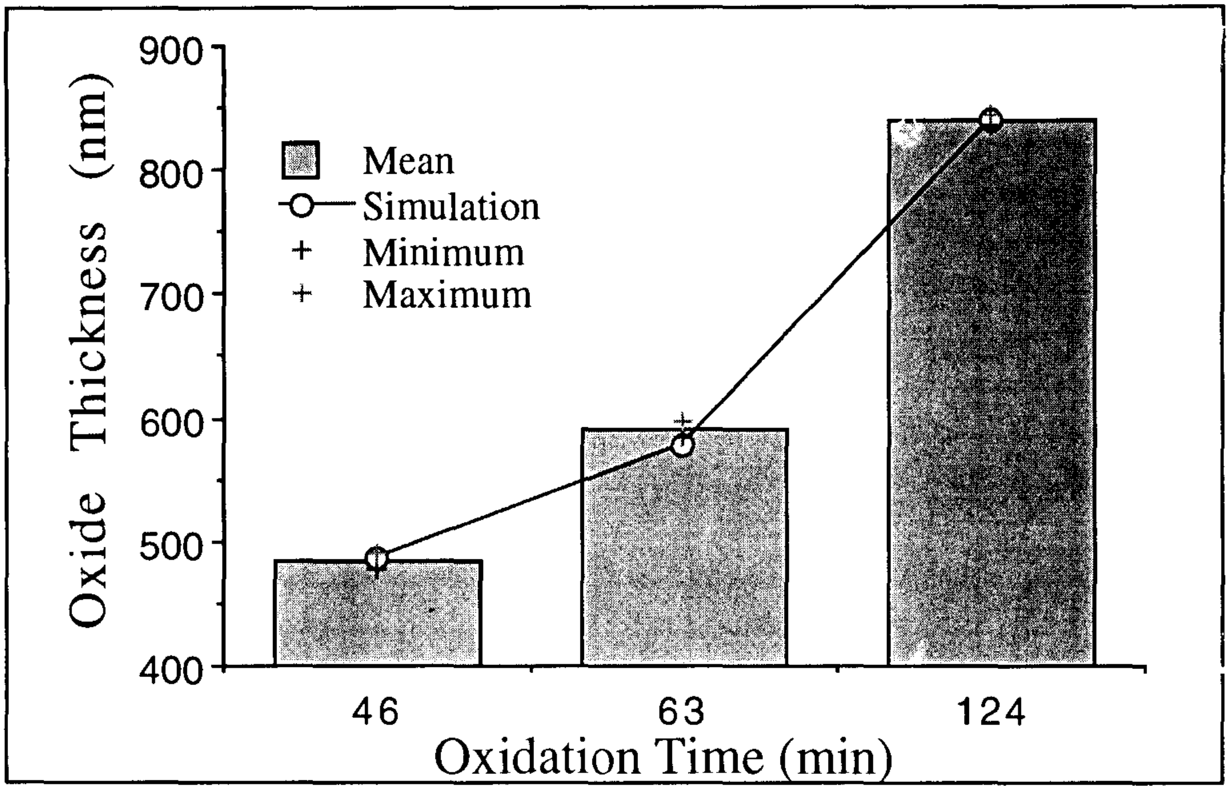

Figure 7. Wet thermal oxidation of silicon at 1100°C.

USING EXPERIMENTAL DESIGN MODULES FOR PROCESS CHARACTERIZATION IN MANUFACTURING/MATERIALS PROCESSES LABORATORIES

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Using Experimental Design Modules for Process Characterization in Manufacturing/Materials Processes Laboratories

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Abstract: Modules dealing with statistical experimental design(SED), process modelling and improvement, and response surface methods have been developed and tested in two laboratory courses. One course was a manufacturing processes course in Mechanical Engineering and the other course was a materials processing course in Materials Science and Engineering. Each module is used as an 'experiment' in the course with the intent that subsequent course experiments will use SED methods for analysis and interpretation of data. Evaluation of the modules' effectiveness has been done by both survey questionnaires and inclusion of the module methodology in course examination questions. Results of the evaluation have been very positive. Those evaluation results and details of the modules' content and implementation are presented. The modules represent an important component for updating laboratory instruction and to provide training in quality for improved engineering practice.

Introduction (history): What follows is a brief discussion of the history of the development of the SED and related topics modules in ME310 and MS&E370. The actual ME310 module on SED is then presented in its format as one lab experiment followed by an example of the SED applications from one of five subsequent experiments which rely on the module for data analysis. A discussion of the comparison of the ME310 and MS&E370 modules is given next. Finally, the results of the evaluation of the effectiveness of the ME310 and MS&E370 modules are presented which are based on measurements of student skills (ME310) and student opinion surveys (ME310 and MS&E370).

ME310 Lab: The ME310 course (Manufacturing Processes I) has been a requirement in the ME curriculum for decades. Industrial Engineering students also may take the course as an option and many usually do so. It is a three credit per semester course with the course grading scheme effectively making for a division of student effort into two-thirds lecture and one-third lab. The labs in the current version of the course are very practice oriented with emphasis placed on principles and practices of machining operations and deformation processing, e.g., extrusion and sheetmetal forming. The SED module occupies one lab period near the beginning of the course.

ME310 Lecture: Although the focus of the module development and applications has been the ME310 laboratory the lecture portion has also been a source of information on SED methods and related quality improvement tools. During 1991-92, for example, Visiting Professor John Corbett used the lecture to introduce quality function deployment (QFD) as a component of the broader topic, "design for competitive manufacturing". In 1992-93, one of the authors (JAC) added presentations on fractional factorial designs, Taguchi methods, and exploratory data analysis tools (check sheets, cause-and-effect diagrams, flow charts, etc.) to the lectures.

MS&E370 Lecture: The module for this course was developed in 1992-93 with the intent of being more comprehensive than the ME310 SED module. The reason for the change of focus and format was two-fold: (1) MS&E370 was undergoing revision and the applications of SED in the course's laboratories would be undertaken as a second stage of the course revision; (2) unlike students in ME310 those in MS&E370 would not likely have completed an introductory statistics or quality engineering course prior to the 370 course.

Historically, the course MS&E370 (Materials Processing-Unit Operations) has been a four credit per semester course with division of effort making the lab count effectively as one credit. In the 1992-93 academic year the course was being revised to make it fit, as an approved option, into the Mechanical Engineering curriculum. Thus, there was an increased interest to incorporate topics such as SED and process characterization and improvement tools in the revision of MS&E370 arose to make it similar to ME310.

Lab Module(s): The actual ME310 Lab module on Statistical Experimental Design is given in Appendix I. Also in that Appendix is an SED Project exercise which is the basis of the student reports for the SED lab session. Subsequent to the SED lab session there are five (5) SED-based lab sessions: Extrusion, and Forces and Power in Metal Cutting I and II (drilling; grinding; milling; turning). The five applications of SED along with the SED module itself comprise approximately 40%, or 110 points out of 250, of the value allocated to ME310 Lab reports. A copy of the ME310 Lab data sheet used in the SED application for the Extrusion experiment is included in Appendix I. That data sheet is typical of the forms used in the five SED applications labs.

The module on Process Characterization Tools prepared for MS&E370 is excerpted in Appendix II. Shown there are: (i) the cover page for the module which outlines the module's intent, and (ii) a summary figure from the module, "PROCESS IMPROVEMENT TOOL BOX". As the cover page "Abstract" indicates the MS&E370 module covers a wider range of topics than the ME310 SED module. Its future application in the revised MS&E370 was the basis for such a range of topics being introduced. An exercise was included in the MS&E370 module which is similar to the 2^3 design analysis of the ME310 SED module. However, in the first semester of its use none of the other lab experiments in MS&E370, e.g., casting, heat treating and joining, were modified to take advantage of the new process characterization/improvement module.

Evaluation of Effectiveness: Two methods of evaluation were used to determine the effectiveness of the ME310 module on SED. These methods were: (1) evaluation of each student's skill level in designing, analyzing and interpreting a standard 2^3 factorial experiment during an exam; (2) surveying each student to gain information about their attitudes towards the perceived general usefulness and possible future career applications of SED methods. Only the survey method was used with the students in MS&E370 since they did not have the opportunities for practice of the skills afforded in ME310.

Skill Level - ME310: As a part of one of the ME310 lecture examinations a problem was given which asked the students to compare their actual lab experiment on sheetmetal forming (done in sequence prior to their SED lab, and also done without regard to balancing the pattern of investigation of the three test factors of: alloy type, sheet thickness and lubrication) to data from a standard 2^3 factorial design of the same experiment. Of the sixty-five (65) students taking the exam 62 got the problem completely correct, except for some math errors in calculating the predictive model residuals. The three remaining students' error was in writing the standard design in reverse order, after which the rest of their calculations were correct except for the wrong signs in the calculations of the predictive models. The exam was closed book and closed notes. They, as a group, had clearly developed the skill of being able to design and analyze simple factorial designs. Of equal importance, they were able to recognize the "bad" design of the original sheetmetal forming experiment.

Attitudes Survey - ME310 and MS&E370: At the end of Spring Semester 1993 the 65 students in ME310 and 6 in MS&E370 were surveyed to determine how effective they felt SED was as an experimental tool. The summary results from three questions relating to usefulness of SED are presented below. The percentages are based on the total number of responses who either ***agree***, or ***strongly agree*** with the statement.

Statement	ME310	MS&E370
I would recommend that SED methods be incorporated in all lab courses.	82.5%	66.7%
I think SED is a tool that I will find useful in future applications.	95.2%	100%
I would like to gain more experience in SED methods	88.9%	83.3%

The survey results provide a strong signal for those of us developing laboratory courses and experiments. It is worth adding at this point that several students in both courses were co-op students. Their comments on the surveys were that they had already been introduced to these experimental planning and analysis tools during their co-op. Unfortunately, except for these instances (ME310 and MS&E370) they were not being given regular access to such modern laboratory analysis methods in their engineering education. (We should note here that the University of Wisconsin - Madison's Plasma Processing Laboratory offers SED instruction as part of the plasma lab course at the graduate level. An SED module was written by Prof. Soren Bisgaard for that course.)

Summary: We have outlined an example of an instructional plan for incorporating statistical experimental design methods into existing laboratory courses. The incorporation of these techniques has been shown to be effectively accomplished in terms of both student skill development and attitudes regarding experimentation. Furthermore, this procedure of making the SED topic an integral part of the overall lab operation strengthens the entire course with minimal effect on the curricular focus of the lab course.

Instructor Notes: The major impediment to using SED methods in existing laboratory courses is lack of instructor familiarity with the concepts. We offer two recommendations for overcoming that barrier. The first suggestion is to enlist the aid of a colleague at your institution who has a background in statistical methods to help you write your own module(s) using this module as a guide. In this approach you will find a willing support person with statistics as a background and you will 'learn by doing' as you develop the module to fit your specific circumstances. A second way to modify your course(s) to include the SED techniques is to attend formal workshops or short courses offered by a variety of organizations. We will not list any of those offerings here, but can provide you with suggestions if you contact us.

Acknowledgements: There have been many individuals responsible for the successful development of the ME310 SED modules. Particular mention should be made of the Teaching Assistants who have taught the lab course and specifically contributed to the lab's success. They are, in alphabetical order: John Bashel, Dan Bee, Dave De Haan, Bill Durkin, Pat Galecki, Bob Gustafson, Jim Rink, Russ Tilsner, Dave Van Zuest, and Jim Witte. The encouragement of several faculty also was essential to implementing these changes. The faculty to be acknowledged include, in alphabetical order: Prof. Soren Bisgaard (IE and Associate Director of the Center for Quality and Productivity Improvement), Prof. Marvin DeVries (Chairman of ME and a long time proponent of use of SED in manufacturing processes), and Prof. Slawomir Spiewak (ME and faculty in charge of ME310 during these changes). Two of the authors (BA and JC) also wish to recognize the support to CQPI by a grant from the Alfred P. Sloan Foundation during 1992-93 when this report was prepared.

(*Appendix I*)

ME310 Lab
STATISTICAL EXPERIMENTAL DESIGN

Key Words: Statistical Experimental Design; Factorial designs; Randomization; Interaction Effects; Predictive models; Process characterization; Continuous Improvement; Response surfaces; Exploratory data analysis.

Prerequisite Knowledge: You are expected to have taken an introductory materials science/engineering course and math through differential equations. No prior course work in statistics is required.

Purpose and Learning Objectives: The *Purpose* of this instructional module is to introduce you to an effective method of planning, conducting, analyzing and interpreting experiments. This module will be followed by a series of laboratory modules where practice of the methods will help to develop skills The methodology of statistical experimental design (SED) is especially useful for the initial characterization and continuous improvement of processes, particularly industrial processes.

Learning Objectives After completion of this module you should have accomplished the following *knowledge*, *skill* and *attitude* objectives:
Knowledge Objectives Know about the use of: *Factorial SED techniques and their capability to determine the effects of major process control parameters and interactions between parameters; randomization,replication, blocking, and confounding in conducting and analyzing experiments; Exploratory data analysis tools;*
Skill Objectives Be able to: *Plan, and properly conduct a full 2^n factorial design; Calculate contrasts and effects from standard results; Determine significant effects; Construct empirical predictive model of behavior based on significant effects; Construct contour diagram (preliminary response surface) from predictive behavior model; To aid in problem solving: Construct check sheets, Pareto diagrams, histograms, flow charts, cause-and-effect diagrams, scatter diagrams for a given data set;*
Attitude Objectives *Actively promote the application of: Designed experiments in the determination of major process effects for quality improvement and optimization; Simple graphical methods of data analysis;*

Equipment and Supplies: The SED modules require only data from real experiments or industrial operations. Access to a computer software package with capability to do graphics manipulations such as cube plots, normal probability plots, flow chart construction, etc. is desirable. Such software is best presented as a demonstration (needing appropriate hardware for overhead projection from computer screen) initially with subsequent student access to the software for report calculations.

Introduction to Statistical Experimental Design

Statistical Experimental Design is a method of experimentation which provides the experimenter with tools to accurately and efficiently collect and analyze data. *A factorial experiment* usually involves several factors which are varied by the experimenter and one or more responses which are to be optimized. In the past, engineers and scientists have relied on one-factor-at-a-time experiments. These are experiments in which each factor in the experiment is varied independently while the other factors are kept constant. However, this type of experimentation has been found to be inadequate to produce the speed and accuracy needed to develop new products and processes. The most notable short coming of the one-factor-at-a-time approach is that the interaction that factors often have with each other cannot be estimated. For example, in a sheetmetal forming operation, it may be found that at one thickness of material, lubrication has a large effect on the formability of the material, but at another thickness level the lubrication has almost no effect. A one-factor-at-a-time experiment will not provide any information about this interaction. However, a factorial experiment based on modern, statistical principles of experimental design will be able to detect such interaction effects.

Two-Level Factorials

Although there are many types of statistically designed experiments, some of the most useful are called two level factorials. For a two level factorial design each factor is set at a high and low level (usually a small amount above and below what is currently considered the best setting of that factor). Each possible combination of settings of the factors is then run. Due to the properties of the experiment, the experimenter can determine which factors have the most effect on the response, which factors interact, and build an approximate model relating the factors to the response. This information can then be used to optimize the response. Unreplicated two-level, three factor experiments, called 2^3 factorials, are especially useful for efficiently investigating a process about which very little is known. These experiments only require 2^3 or 8 experimental trial runs and the results can be used to get ideas about the underlying relationships of the factors with the response during the exploratory phase of an investigation. To confirm these results for publication or other such purposes, replications would be needed.

As an example, a 2^3 factorial mill experiment will be designed and analyzed. The three factors will be the Speed of the cutter (RPM), the Feed rate (in./min.), and the Depth Of Cut or D.O.C. (in.).

Coding of the Factor Levels

For ease of analysis, it is convenient to code the factors into high and low levels as follows:

Factor	Low Level (−1)	High Level (+1)	Units
Speed (S)	50	75	RPM
Feed (F)	2.5	3.5	in./min
D.O.C. (D)	.1	.2	in.

Using the codes "–1" and "+1" all the possible combinations of levels must now be listed. For simplicity we will use "–" to show the "–1" level and "+" to show the "+1" level. The order listed below is called the standard order and you can quickly see that the pattern of the standard order makes it easy to detect whether all 2^3 possible combinations of low and high factor levels are present.

Standard Order	S	F	D
1	–	–	–
2	+	–	–
3	–	+	–
4	+	+	–
5	–	–	+
6	+	–	+
7	–	+	+
8	+	+	+

Randomization

It is unwise to run the actual experiment in the standard order. For example, if the experiment were run in standard order and the calibration or any other uncontrolled factor changed halfway through the experiment, it would be impossible to tell the difference between the effect of the uncontrolled change and the effect of depth of cut (D) since this factor changes only once at the midpoint of the experiment. This leads to the idea of Randomization. *Randomization* is a principle which states that if the experimental trials are run in random order, it is very unlikely that any uncontrolled factor change will exactly correspond to any of the controlled factor changes and therefore the effect of the uncontrolled factors should not significantly bias the experimental results. For this reason, the trials in this experiment will be run in a random order. To determine the random order, the numbers 1 through 8 should be written on small pieces of paper and drawn from a hat or a bowl.

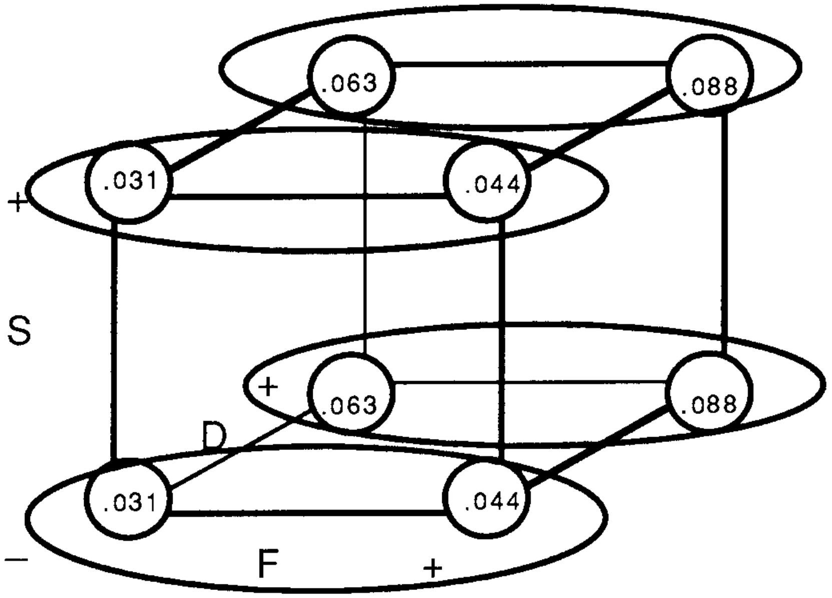
Cube Plots

A *cube plot* is a method of plotting the data from a 2^3 factorial. The plot consists of a cube with a response value located at each corner. Each dimension of the cube represents the coded scale of one of the factors from the low level (–) to the high level (+). An example is given below using metal removal rate (Q) of the mill as the response plotted versus the factors; Speed (S), Feed (F), and D.O.C.(D).

This plot is useful because one can easily compare the four responses with low F on the left side of the cube (.031, .031, .063, .063) with those with high F in the corresponding location on the right side of the cube (.044, .044, .088, .088) to see the effect of F on metal removal rate,(Q).

In the figure below you will notice that there are four separate comparisons which can be made and in each comparison the only factor changed is F. These separate comparisons are called *hidden replications* and are the reason that, even when unreplicated, 2^3 factorials can be relied upon to convey a great deal of information. Similarly one can compare the front and back of the cube to see the effect of D on Q or the top and bottom of the cube to see the effect of S on Q. In the cube shown, one can see that the responses on the top of the cube are identical to those in the corresponding position on the bottom of the cube, which shows that Speed has no effect on Q. This confirms the already known relationship, Q = (D.O.C.)*(Feed)*(Width of Cut), in which Speed is not a factor.

Metal Removal Rate of the Mill



Calculation and Meaning of the Effects

To this point, the effect of each factor has been spoken of in a qualitative way. However, the specific quantitative definition of the *main effect* of a given factor on a given response can now be defined as the average difference between the responses at the high level of the factor and the responses at the low level of that factor. Thus, the effect of Feed on Q is the average of the differences (.044 – .031), (.044 – .031), (.088 – .063), and (.088 – .063). Therefore, for the ranges used in this experiment the effect of Feed (F) on Q is .019, the effect of D.O.C. (D) on Q is .038, and the effect of Speed (S) on Q is zero.

The *interaction effect* between two factors is the average amount that the effect of a factor changes when another factor is varied. For example, the effect of F on Q at the low level of D is the average of (.044 – .031) and (.044 – .031) or .013, but at the high level of D the effect of F on Q is .025. The interaction effect then is (.025 – .013)/2 or .006. These interaction effects are most easily calculated using the table shown below, where the interaction effect between Feed (F) and D.O.C.(D) is called FD and the other interaction effects are similarly called SF and SD. SFD is the three way interaction effect. The contrast for each column is the sum of the data when the data is given the signs of that column as shown in the example column for FD. All effects, including the main effects, are calculated as shown by dividing the contrast for each column by 4*. In this case the only interaction effect is the interaction between the Feed and the D.O.C. (FD); all other interactions show values of zero.

	Main Effects			Interaction Effects					
Trial #	S	F	D	SF	SD	FD	(example FD)	SFD	Q
1	–	–	–	+	+	+	(+.031)	–	.031
2	+	–	–	–	–	+	(+.031)	+	.031
3	–	+	–	–	+	–	(–.044)	+	.044
4	+	+	–	+	–	–	(–.044)	–	.044
5	–	–	+	+	–	–	(–.063)	+	.063
6	+	–	+	–	+	–	(–.063)	–	.063
7	–	+	+	–	–	+	(+.088)	–	.088
8	+	+	+	+	+	+	(+.088)	+	.088
Contrast	0	.076	.152	0	0	.024	(.024)	0	
Effects	0	.019	.038	0	0	.006	(.024/4=.006)	0	

*4 equals the number of differences being averaged, and can be easily determined by the number of + and – pairs in the column.

Model Building

Once the important effects have been identified, a model can be created which will estimate the response, in this case Metal Removal Rate, at locations in or on the cube which were not specifically run in the experiment. The model uses the coded scale –1 to +1 for each of the factors and includes only the strong effects. The coefficients for the polynomial model are exactly one half of the effects previously calculated, so the model we will use is:

$Q = (.0095)F + (.019)D + .0565$ (Model 1)

where .0565 is the average value of the Metal Removal Rate data. If we consider the FD interaction to be important, the model would then be:

$Q = (.0095)F + (.019)D + (.003)FD + .0565$ (Model 2)

Based on Model 1, we would predict a Q of .07075 for F=.5 and D=.5.
Model 2 would predict a Q of .070825, which shows that the interaction term is indeed quite a small effect. Using the cube plot and the model we can see that if Q is to be increased, Feed and D.O.C. must be increased and Speed has no influence on Q.

References

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ME 310 Project II

Given the data in the standard order table below, perform the following analysis: (Note: This experiment was performed in random order.)

1. Calculate the contrasts and main effects of speed (S), feed (F), and depth of cut (D). *Note: Show all calculations, no spread sheet analysis is allowed.*
2. Calculate the contrasts and interaction effects of <u>all</u> control factors. *Note: Show all calculations, no spread sheet analysis is allowed.*

	Main Effects			Interaction Effects				
Trial #	S	F	D	SF	SD	FD	SFD	Hpu
1	–	–	–	+	+	+	–	29.5
2	+	–	–	–	–	+	+	40.7
3	–	+	–	–	+	–	+	28.6
4	+	+	–	+	–	–	–	40.2
5	–	–	+	+	–	–	+	21.3
6	+	–	+	–	+	–	–	34.1
7	–	+	+	–	–	+	–	21.6
8	+	+	+	+	+	+	+	34.0
Contrast								
Effects								

3. Draw the cube plot with the appropriate response at the corners of the cube.
4. On your cube plot, circle the four (4) hidden replications of feed (F).
5. Rank effects in ascending order, or lowest to highest, and calculate the cumulative probabilities for each effect given by the equation:

   $$P(i) = 100 (i - .5) / n$$

   where:
   i = ascending order number associated with each piece of data.
   n = size of the sample or number of effects.
6. Plot the cumulative probabilities on the normal probability plot provided.
7. Determine which main effects and/or interaction effects are significant from the above analysis.
8. Develop the coded model for Hp_u using the significant effects.
9. From the coded equation, determine the actual value for speed if it is desired to maximize depth of cut and have a Hpu value of 30.2. You will need to use the table for coded factors on page 2 of the SED Lab Notes.

NOTE: **For further examples of the graphical methods of using cube plots to analyse factorial designed experiments please refer to the following References. To see how an additional problem is analyzed see the Box and Bisgaard 1988 article in *Mechanical Engineering*. For a more complete, but very readable description of cube plots refer to the textbook by Box, Hunter and Hunter (1978).**

Copies of the ME310 Lab Manual may be requested from Prof. Donald Ermer at the Mechanical Engineering Department, Univ. of Wisconsin-Madison, 1513 University Ave, Madison, WI 53706. New copies will, however, not be printed after Fall Semester 1993

Extrusion Summary Sheet

	S.O. 1*	S.O. 2	S.O. 3	S.O. 4	S.O. 5	S.O. 6	S.O. 7	S.O. 8
Random Order								
Direction	Forward	Reverse	Forward	Reverse	Forward	Reverse	Forward	Reverse
Die Geometry	Square	Square	Round	Round	Square	Square	Round	Round
Lubrication	No	No	No	No	Yes	Yes	Yes	Yes
Break Through Force (lbs)								

SQR= -	FWD= -	N/L= -
RND= +	REV= +	LUB= +

* S.O. represents "standard order"

	Main Effects			Interaction Effects				
S.O. #	D	G	L	DG	DL	GL	DGL	B.T. Force
1	−	−	−	+	+	+	−	
2	+	−	−	−	−	+	+	
3	−	+	−	−	+	−	+	
4	+	+	−	+	−	−	−	
5	−	−	+	+	−	−	+	
6	+	−	+	−	+	−	−	
7	−	+	+	−	−	+	−	
8	+	+	+	+	+	+	+	
Contrast								
Effects								

Sheet Metal Forming Experimental Design

	Main Effects			Interaction Effects				
Std. order				**Axt**	**AxL**	**txL**	**AxtxL**	Bulge
1				+	+	+	–	34.0
2				–	–	+	+	21.3
3				–	+	–	+	34.3
4				+	–	–	–	28.6
5				+	–	–	+	40.2
6				–	+	–	–	21.6
7				–	–	+	–	40.7
8				+	+	+	+	29.5
Contrast								
Effects								

Coding of the Factor Levels

For ease of analysis, it is convenient to code the factors into high and low levels as follows:

Factor	Low Level (–1)	High Level (+1)	Units
Alloy (A) thickness(t) Lube (L)	2024-0 0.05 No lube	2024-T3 0.125 Lube	treatment in. lubricant

NOTE: This page is from ME310 Exam #2. It is the data sheet for a question about how to reevaluate the Sheet Metal Formability Lab if a statistically designed experiment had been conducted. In addition to completing the data table a cube plot and process model equation was required as part of the exam question.

<u>*(Appendix II)*</u>

MS & E 370

Process Design and Control Toolbox: I
Exploratory Data Analysis Tools and Statistically Designed Experiments

Key Words: Statistical Experimental Design; Factorial designs; Randomization; Interaction Effects; Predictive models; Process characterization; Continuous Improvement; Response surfaces; Exploratory data analysis.

Prerequisite Knowledge: You are expected to have taken an introductory materials science/engineering course and math through differential equations. No prior course work in statistics is required.

Purpose and Learning Objectives: The <u>*Purpose*</u> of this instructional module is to introduce you to an effective method of planning, conducting, analyzing and interpreting experiments. This module will be followed by a series of laboratory modules where practice of the methods will help to develop skills. The methodology of statistical experimental design (SED) is especially useful for the initial characterization and continuous improvement of processes, particularly industrial processes.

<u>Learning Objectives:</u> After completion of this module you should have accomplished the following *knowledge*, *skill* and *attitude* objectives.

<u>*Knowledge Objectives*</u> Know about the use of: Factorially Designed experiments; Blocking, confounding and randomization in conducting and analyzing experiments; Exploratory data analysis tools;

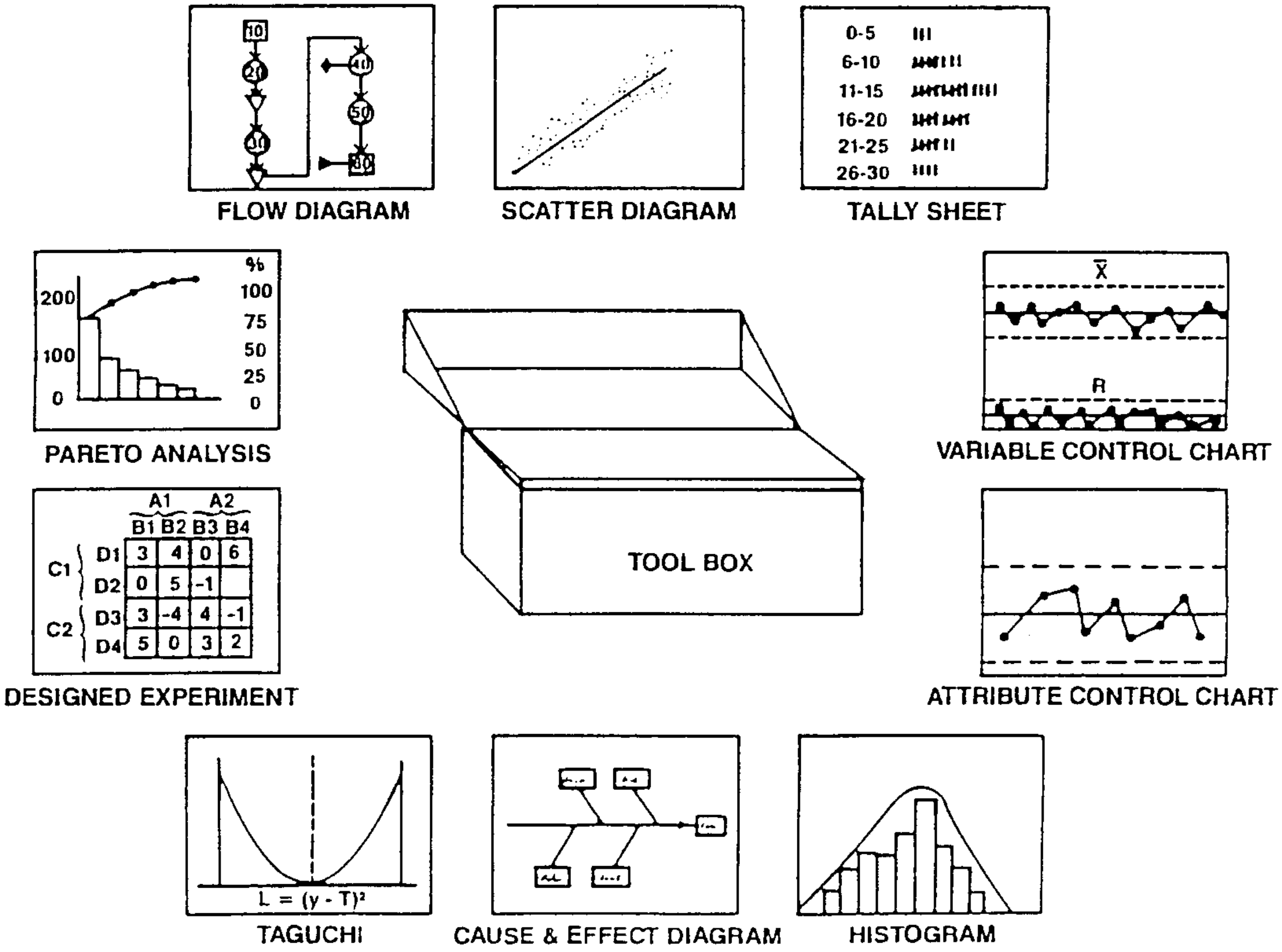
<u>*Skill Objectives*</u> Be able to: Plan, and properly conduct a full 2^n factorial design; Calculate contrasts and effects from standard results; Determine significant effects; Construct empirical predictive model of behavior based on significant effects; Construct contour diagram (preliminary response surface) from predictive behavior model; Construct check sheets, Pareto diagrams, histograms, flow charts, cause-and-effect diagrams, scatter diagrams for a given data set;

<u>*Attitude Objectives*</u> Promote the application of: Simple graphical methods of data analysis; Using designed experiments in the determination of major process effects for improvement and optimization.

Equipment and Supplies: The SED modules require only data from real experiments or industrial operations. Access to a computer software package with capability to do graphics manipulations such as cube plots, normal probability plots, flow chart construction, etc. is desirable.

<u>Abstract:</u> Traditional methods of designing and improving processes have been based on one-factor-at-a-time (***1-faat***) procedures for obtaining information about the processes. Such methods are known to be inefficient and often misleading with regard to the way process variables are determined to affect the output. In this module we will describe and illustrate the use of a variety of tools, or techniques, which are especially easy to use for exploring existing data, and for obtaining and interpreting new data. The tools which will be introduced are: *flow diagrams; check sheets; Pareto charts; histograms, control charts, cause-and-effect diagrams; designed experiments; response surfaces*. These tools place strong emphasis on visualization of data rather than on numerical descriptions of a system. They can, consequently, be effectively used by a wide range of personnel involved with design and monitoring of processes. Special emphasis will be given to factorial methods of experimental design since those methods provide one of the best techniques for acquiring data and converting it into useful information.

PROCESS IMPROVEMENT TOOL BOX



PROCESS CAPABILITY DETERMINATION OF NEW AND EXISTING EQUIPMENT

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KEY WORDS: Process capability, statistical process control, statistics, equipment characterization.

PREREQUISITE KNOWLEDGE: Introductory statistics, some knowledge of manufacturing processes and equipment. This will generally be an experiment for an advanced class.

OBJECTIVES: To illustrate a technique for determining the process capability of new equipment, existing equipment, or testing laboratories. To illustrate a method of objectively determining wear and other changes in equipment used for small quantity production over a period of time. To present an example of the application of the technique.

INTRODUCTION

The objective of this paper is to illustrate a method of determining the process capability of new or existing equipment. The method may also be modified to apply to testing laboratories. Long term changes in the system may be determined by periodically making new test parts or submitting samples from the original set to the testing laboratory.

The technique described has been developed through a series of projects in special topics manufacturing courses and graduate student projects. It will be implemented as a standard experiment in an advanced manufacturing course in a new Manufacturing Engineering program at the University of Wisconsin-Stout campus.

Before starting a project of this nature, it is important to decide on the exact question to be answered. In this case, it is desired to know what variation can be reasonably expected in the next part, feature, or test result produced. Generally, this question is answered by providing the process capability or the average value of a measured characteristic of the part or process ± three standard deviations. There are two general cases to be considered: (1) the part or test is made in large quantities with little change or (2) the process is flexible and makes a large variety of parts. Both cases can be accommodated; however, the emphasis in this report is on short run situations.

GENERAL PROCEDURE

The first step in any investigation is to clearly define what is desired. Specifically, what measurements will give the most information about the process, can be measured with relative ease, will continue to be made for the foreseeable future, and relate directly to the quality of the part. Quite often, this step is done too quickly and the results do not give the desired information. It is suggested that extra time be taken to clearly define the specific problem.

Selecting the proper sample is the next step. For large runs of production parts or routine tests, the normally measured features of the part or the standard test samples can be used. A special part may have to be developed for short run, flexible machinery. This part should include a variety of features which incorporate as many of the functions of the machine as possible. One such part is illustrated later. For short run situations, a method can be used which focuses on the process, not the product.[1] One technique is to use the deviation from the nominal dimension as the measured characteristic rather than the actual dimension.[2] This allows measurements of various depth holes or different diameter rounds, etc. to be used to generate sufficient numbers for reasonable process capability estimates. These comparison procedures (comparing deviations from nominal dimensions for similar features) can be used to give a running process capability. Periodically, an original test sample can be produced to evaluate any undetected changes with time in the machine performance.

When testing laboratories are to be evaluated, a large number of identical samples are obtained from well characterized material. If more than one type of material is tested routinely, then representative samples of each should be included. About one-fourth of these are used initially and the rest are submitted over a period of time.

Each part produced may be measured but often a sample of the production lot is measured. In the latter case, it is important to randomize the part selection so the Laws of Chance will apply and the results will be valid. All of the testing laboratory specimens should be numbered sequentially (include all different materials) and a random sample selected comprising the initial one-fourth or so of the specimens. These should be submitted to the laboratory with instructions to run the specimens in the order listed. If the materials are sufficiently different, such as those requiring different load cells on a tensile machine, a stratified random sample may be required. The data from the initial measurements or test results can be used to determine the current, initial process capability. The process capability can be updated as new measurements are obtained.

For testing laboratories, a small number of original "standard" specimens can be blended into the normal production tests and the variation of test lab results with time evaluated. These results can be combined with the results for the normal production specimens to give a continuously updated process capability. It is suggested that test laboratory evaluations be discussed with the lab manager after the initial results are obtained before being generally distributed. Quite often there are explanations for unexpected results of which the investigator may be unaware. This also helps keep political peace within the company.

APPLICATION TO A MACHINE TOOL

Objective:

The Manufacturing and Industrial Technology Department of Arizona State University recently obtained a Fadal VMC-20 vertical machining center. Mr. Su was assigned to determine baseline measurements of the dimensional reproducibility of this machine as part of his Master of Technology project. Sample materials were two alloys commonly used in the department: 1018 cold rolled steel and 6061-T6 aluminum. The objective was to determine the initial dimensional reproducibility of the new machine, to determine if a difference exists between the two materials, and to quantify such a difference if one exists.

Procedure:

The machining center was obtained as a flexible component of a manufacturing cell and is not part of a line producing many of the same component. For this reason, a special part was designed to give a measure of the various machine capabilities and this part is shown in Figure 1. The overall nominal dimensions are 102 mm x 102 mm x 44.5 mm (4 inches x 4 inches x 1.75 inches). Each feature is dimensioned with respect to the center line or top surface but the numbers are not shown because of the clutter. The parts were machined to the same programmed nominal dimensions and these dimensions were then measured on a coordinate measuring machine. The deviations from the nominal dimensions were the characteristics of interest. In this way, the various linear dimensions could be combined with each other and likewise with the various circular dimensions. Each part is therefore capable of giving numerous samples for the calculation of the process capabilities.

Six machining processes were evaluated using the designed part:

1. End milling of the steps to preset depths.
2. End milling of steps to preset peripheral dimensions.
3. End milling circular interpolation clockwise in to preset outside diameters of cylinders and round corners.
4. End milling circular interpolation clockwise out for circular pockets and round corners.
5. Reaming the holes to preset diameters after center hole and pilot hole drilling.
6. The relative positions of the holes from the center lines of the part after reaming.

Two materials were used: 1018 cold rolled steel and 6061-T6 aluminum. The same basic program was used for each with variations based on published optimum machining conditions for each material.[3] These conditions were modified somewhat for the steel as a result of chatter problems with the initial part. The Fadal machining center was capable of holding the complete tool sets needed for both parts and, thus, loading tooling into the machine was not a factor in the results.

The initial experiment was designed to determine if there was a difference in dimensional reproducibility between the aluminum and steel at the 95% confidence level. A difference of 0.0076mm (0.0003 inches) was considered to be of practical significance and an initial standard deviation of 0.0051 mm (0.0002 inches) was used for the initial calculations. These values were estimated from experience and guessing. The standard deviations were checked after the first parts were machined and found to be higher for these parts, to vary with the process in question, and with the material. The number of test parts required was then recalculated. The calculations considered the number of features to be combined on each part and showed that six parts of each material were required to determine if a difference exists between the steel and aluminum. The machining order for two materials was determined by randomization. Each part blank was placed into the machine by hand.

Results:

The results of the machining tests are summarized in Table I.

TABLE I
Summary of Machining Results
Values are Variations from Nominal Dimensions mm (inches)

Aluminum--6061-T6:

Process	Average	Std Dev (σ)	Initial PC ($\pm 3\sigma$)	#meas
1	-0.0061 (-0.00024)	0.0053 (0.00021)	± 0.015 (± 0.0006)	36
2	0.0322 (0.00125)	0.0990 (0.00039)	± 0.0305 (± 0.0012)	36
3	0.0236 (0.00093)	0.0104 (0.00041)	± 0.0305 (± 0.0012)	36
4	0.0142 (0.00056)	0.0127 (0.00050)	± 0.0381 (± 0.0015)	36
5	-0.0338 (-0.00133)	0.0142 (0.00056)	± 0.0457 (± 0.0018)	24
5*	0.0310 (0.00122)	0.0051 (0.00020)	± 0.0152 (± 0.0006)	23
6	-0.0079 (-0.00031)	0.0135 (0.00053)	± 0.0419 (± 0.0017)	48

One hole in one part had a very high variation in process 5 and no assignable cause was found for this variation. The data for 5 were calculated using all results and 5 dropped the questionable reading. All of the readings were used in the comparisons.

Steel--1018 cold rolled

Process	Average	Std Dev (σ)	Initial PC (± 3σ)	#meas
1	0.0117 (0.00046)	0.0117 (0.00046)	± 0.0356 (± 0.0014)	36
2	-0.1540 (-0.00607)	0.0991 (0.00390)	± 0.2970 (± 0.0117)	36
3	-0.0478 (-0.00188)	0.0937 (0.00369)	± 0.282 (± 0.0111)	36
4	0.1460 (0.00573)	0.1430 (0.00562)	± 0.4270 (± 0.0168)	36
5	-0.0277 (-0.00109)	0.0610 (0.00024)	± 0.0152 (± 0.0006)	24
6	-0.0157 (-0.00062)	0.0236 (0.00093)	± 0.0419 (± 0.0017)	48

Each average is calculated from 24, 36, or 48 individual readings depending on the type of measurement. For example, there are six steps (one inside and five outside on each of the six parts) which were measured for Process 1 so 36 measurements were averaged. There are four holes in each part so Process 5 is the average of 24 readings. Two measurements were made for the position of each hole (x and y) with respect to the center of the part so 48 measurements were averaged for process 6.

The data were compared using the "z Test for Measurements" and the "Student's t Test"[4] and it was found that a significant difference existed between the aluminum and steel at the 95% confidence level for all processes measured except for processes 5 and 6. It should be noted that process 5 was the process with the one unexplained high reading and the comparison statistic was very close to the decision point. This would normally indicate that another one or two parts should be run but, at the time of writing this experiment, no free machine time was available to run these parts.

Thus, except for processes 5 and 6, the two populations cannot be combined and the process capability must be determined for each material. These data are listed in Table I.

Discussion:

It should be emphasized that the process capability of the machine in making a specific part will be greatly influenced by the cutting conditions used. This machine will be used for a variety of parts under a variety of cutting conditions as a class room teaching tool and it is difficult to derive process capability values for each situation. The cutting conditions used in this study were arbitrarily chosen using handbook data and may not represent the best conditions for precision cutting of a particular part for a specific application. These data do give a baseline to use for determining changes in the machine due to wear or other factors as the machine is used. The programs generated in this study can be used with the same, readily available materials at various future times and the process capabilities compared to the initial data presented above. Any wear should manifest itself as increases in the ± 3σ values.

Because no assignable cause could be found for the high reading in process 5 for the aluminum, the data point was included in the comparison between the steel and aluminum for this process.

Many authors consider a minimum of 50 readings to be necessary for an accurate process capability determination.[5] For the situation in this study, 24 to 48 readings were considered sufficient for initial process capability values. Additional readings will be obtained from the parts made in the classes which can be measured using the same techniques described here and the data combined with the appropriate process category. This will give a running process capability with the test parts being machined at regular intervals of machining time to spot check for wear or other changes in the equipment.

CONCLUSIONS

A method has been presented by which the process capability of machine tools, manufacturing processes, or testing laboratories can be evaluated. The method allows the continuous monitoring of the process capability over an extended time period by running similar standard parts periodically mixed in with normal production.

An example was presented for a Fadal VMC-20 vertical machining center. Initial process capabilities for six individual machining processes were determined using 6061-T6 aluminum and 1018 cold rolled steel and a specially designed part. It was also determined that 1018 cold rolled steel test parts had a statistically higher variation in machined dimensions than 6061-T6 aluminum at the 95% confidence level when each were machined using handbook values to establish the cutting conditions. The data generated can be used as a baseline to check for later wear or other machine changes by periodically machining similar parts from similar materials using the same NC program. Short run statistical techniques were used because this machine was purchased as a flexible tool for making a variety of parts in a classroom situation.

The process capabilities determined apply only to the test parts manufactured but additional data can be obtained from class projects and combined to give running process capability measurements for each individual function of the machine.

SAMPLE DATA SHEETS: Data should be gathered in a manner compatible to analysis using a computer spread sheet or database program. One suggestion is as follows:

Material	Process	Nominal meas	Actual meas	Difference
----------	---------	----------	----------	--------------
----------	---------	----------	----------	--------------
----------	---------	----------	----------	--------------
"		"	"	"

SAFETY CONSIDERATIONS: There are no special safety considerations for this experiment but all safety practices common to the equipment or processes studied should be followed.

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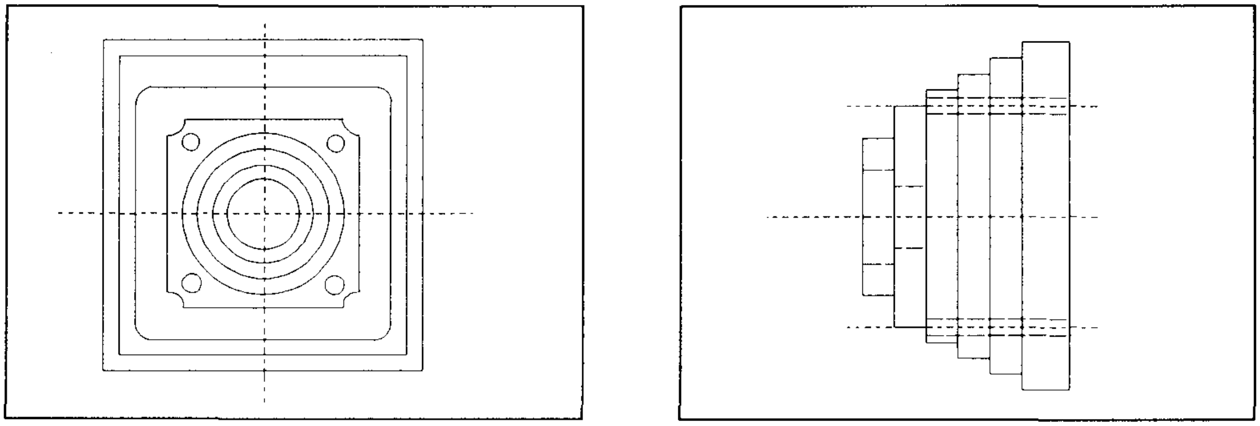

Figure 1: Top and side views of the specially designed part for the machining study.

INTRODUCTION TO USABLE STATISTICAL METHODS

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INTRODUCTION TO USABLE STATISTICAL METHODS

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KEY WORDS: Statistics, Process analysis, Statistics courses.

PREREQUISITE KNOWLEDGE: College algebra, Introductory manufacturing course helpful but not mandatory.

OBJECTIVES: Provide an introduction to statistics, experimental design, and data analysis for engineering and engineering technology students who do not have a full statistics course in their curriculum or who are not exposed to experimental design.

INTRODUCTION

The purpose of this paper is to present a description of a short course on usable statistical methods for students who are not exposed to a full statistical course. The course is designed to take approximately one fourth to one third of a semester (10 - 16 class hours) as part of a more general technical course such as a selected topics course in manufacturing. The need for a course of this nature is apparent when it is realized that many engineering programs do not require a statistics course and those courses which are required often do not include experimental design. Often, both students and faculty are not aware of basic statistical principles.

There are many introductory statistics courses offered by various departments with some campuses having four to seven seemingly similar courses.[1,2] A nonscientific, personal poll of students at two universities taking a variety of the introductory statistics courses of which I was aware all described the same course syllabi. A considerable amount of time is spent on basic number theory (mostly manipulating numbers which is not the same as understanding what the numbers mean) and more time on permutations and combinations. These are all good subjects but no time remained for discussing the basics of experimental design, random sampling, or the statistical evaluation of experimental data.

Personal experience in industry has shown that most manufacturing applications of statistics involve determining baseline process information (process capabilities) or comparing baseline information with the process performance after a controlled change. Generally, the results of the desired changes must be fairly large to be justified economically, particularly where new equipment must be purchased. Thus, techniques aimed at finding subtle differences among the data are usually not required.

The course described in this paper covers only the basics of probability and emphasizes experimental design and analysis.

GENERAL COURSE DESCRIPTION

Practical Statistics, Simply Explained by Langley[3] is the recommended text. This is a basic text which covers the fundamentals very well while the examples are easily updated, where necessary, by incorporating items from current news sources. Following the text, the course covers how to understand and interpret numerical data, basic probability theory, sampling, averages and scatter, experimental design, and significance tests.

This is a course in which items from current events add a generality to the topics which helps the students understand that the basic concepts of interpreting numbers are widely applicable. Occasionally some major events such as the oil crises of the seventies and eighties lead to many years of examples of media misuse of numbers on an almost daily basis. Currently, the debates over mandatory AIDS testing, gun control, and the President's budget offer regularly occurring topics. Sometimes it requires a bit more searching for appropriate examples. If you are teaching this course on a regular basis, you may pull examples from the news media as they appear over the year and save them for the class.

The concepts discussed in the early portion of the course are those which, in my experience, students have not understood during their earlier education. Students often become quite adept at manipulating numbers and equations without gaining any insight into what the numbers mean. The basic approach of the Langley text is how to avoid being misled by numbers. Students often find the other side of this topic to also be of interest, i.e. how to mislead others by the misuse of numbers. Either approach can be used to get the concepts across.

SPECIFIC TOPICS

Each of these topics can be covered in one or two 1 hour lectures.

Numbers:

The first chapter deals with being misled by arithmetical errors, false percentages, fictitious precision, misleading presentations, incomplete data, and faulty comparisons. Each of these topics can be related to other courses which the students are taking, particularly those with laboratory components such as physics or chemistry. For example, excessive significant digits resulting from students copying the answer directly from their calculators can be used as a prime example of fictitious precision. Generalizing mundane laboratory data to earth shaking, socially significant conclusions can be used to explain incomplete data or misleading presentations. Misleading presentations can also be illustrated by reference to the "low fat" competition going on in the grocery stores.

Langley states that these are all important causes of being misled by numbers but that the usual causes are biased samples and not considering the effect of chance on the numbers.

Probability:

Probability is introduced by a discussion of the Laws of Chance. This leads into the Binomial and Poisson's probability formula for describing the proportions of occurrence of two classes of events, i.e. on/off, go/no-go, yes/no, happens/doesn't happen. The normal probability formula is introduced to deal with continuous data. A brief discussion at this point covers the mean of the probability distributions and the scatter or variation about the mean. The main objectives for this topic are to introduce the probability distributions and the reasoning behind them rather than memorizing a formula.

Experimental Planning and Sampling:

I spend a considerable amount of time discussing the statement of the problem, planning the experiment, and devising an appropriate sampling plan. The problem statement is extremely important and is often made with little thought. A proper problem statement prevents wasted time and effort finding an possibly elegant solution to a non-problem or solving only a part of the problem. It also allows the proper generalization of the results. The importance of phrasing the problem in terms such as "...to determine the effect of.." rather than "...to prove the effect of..." is emphasized. This allows negative results (i.e. no difference with the treatments) to still be valid. Another important point is to state the problem so a path to the solution is apparent. If no path to the solution is apparent, restating the problem in different forms until a solution path is obvious is discussed. This also allows a better definition of what is sought.

The next step is to select the method of data analysis before the data are taken. In this manner, the data can be obtained in a form which fits the analysis method. An example of a lack of agreement would be to assume that the analysis will involve the binomial distribution but the experiment measures a continuously changing series of events which requires a normal distribution based analysis. The result of this would be an extra amount of data manipulation and less than satisfactory conclusions.

Sampling is a big area of misunderstanding. I have often seen situations where the student is simply told to go and take an appropriate sample with no further instructions. The major objective in this section is to emphasize the importance of a properly designed sampling procedure.

If the whole population is measured, there is little need for a formal sampling procedure. In many cases, however, the population is very large and conclusions regarding this population are desired using a minimum of measurements (and time and cost). Considerable time is spent discussing the need for a random sample if the Laws of Chance are to be satisfied and the reliance of the various statistical analysis procedures on the

Laws of Chance is emphasized. The definition of a random sample is that each possible outcome of an event has an equal chance of being measured. This can only be achieved by using a random number generator of some sort which covers the entire population.

One further point discussed on the Experimental Planning topic is the personal recommendation of keeping the studies simple. Many small studies concerning two or three factors are easier for the student to comprehend than trying to measure everything in one big study. Computer programs exist which can analyze large amounts of experimental data but the human mind is much more limited in trying to fully understand and document the results of these studies.

The importance of documenting and communicating the results is discussed in terms of what the student will be expected to do upon graduation. Someone can be hired at minimum wage who cannot tell the manager what the results mean in a clear manner; why pay someone engineering wages for the same lack of information. This leads naturally into a plug for the writing and speaking courses which most students dislike.

I generally spend about half the course on the topics of this section.

Analysis of the Results:

Once the data are obtained, the previously determined procedure is used for analysis. I generally spend time discussing the general features of the various common procedures with a brief worked example of one or two. Computer usage is encouraged in the planning and analysis stages if the programs are available, with an understanding of the procedure determined through a separate in-class test.

The Langley text is very good in this regard. The last half of the text contains a brief description of many common analysis procedures with the required tables and worked examples. This text was pre-computer so the mathematical manipulation is simplified. These descriptions can be used to guide the student in a project.

Project:

The experimental project is key to illustrating the important points of the course. The general ground rules are that the student states a problem which involves a one-on-one comparison, designs an appropriate experiment, and analyzes the results using a standard, preselected technique. A simple one-on-one comparison allows the student to complete the project in a reasonably short time of two to three weeks.

Topics for the comparison are selected by the student with the problem statement reviewed by the instructor prior to any data collection. The review is to insure that the problem statement is to the point and does not involve too much work. Most students greatly underestimate the amount of time involved in properly gathering data and trying to solve a problem of this nature. A discussion of the amount of generalization which can derive from the completed project based on the problem statement is included in the review. Allowing the students to choose their topics leads to many similar topics such as how many red versus green M&M's are there in a package but also leads to useful information such as which brand of popcorn gives the most old maids or which bartender at the local hangout fills the glasses the fullest. One or two times through the course will give you great insights on which local store has the best price on beer or which hunting ammunition gives the most accurate and precise patterns.

The project is documented in a formal engineering technical report and I normally grade on content, spelling, and English usage.

SUMMARY

An outline for an introductory statistics course has been presented which is designed for students who do not take a full semester statistics course. The course is designed to be taught in 10 - 16 class hours and covers topics of most use to working engineers and engineering technologists. The understanding of numerical information is presented followed by an introduction to probability and probability distributions. Considerable time is spent on the statement of the problem and designing a proper data collection method. Statistical analysis techniques are discussed and a final project using student selected topics reinforces the main points of the course.

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3. Langley, R, Practical Statistics, Simply Explained, Dover, 1971.

The above represents views based on my experience. I am very interested to hear from others as to their experiences and welcome suggestions on course improvements. Please contact me at:

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MEASUREMENT OF SURFACE HEAT FLUX AND TEMPERATURE

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Measurement of Surface Heat Flux and Temperature

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KEY WORDS: heat flux, temperature, computer data acquisition, experimental statistics, microsensor, combustion flame

PREREQUISITE KNOWLEDGE

Students should have an undergraduate course in heat transfer to understand the fundamentals of conduction, convection, and radiation heat transfer. An understanding of transient conduction is important and a background including semi-infinite conduction solutions is helpful. An introduction to elementary experimental statistics is necessary.

OBJECTIVES

The experiment demonstrates a new sensor technology. The students use the sensor to observe details of a combustion flame and allows them to experience the application of modern research. The data processing clearly demonstrates the different effects of integrating versus differentiating data.

EQUIPMENT AND SUPPLIES

1. Heat Flux Microsensor and associated amplifier
2. Self-igniting propane torch
3. TEGAM thermocouple read out
4. DC power supply
5. Elenco Precision Bread board
6. Texas Instruments 741 OP-AMP
7. Voltmeter
8. Macintosh Quadra 700 computer
9. LabVIEW software
10. Leather gloves

INTRODUCTION

The Heat Flux Microsensor is a new sensor which was recently patented [1] by Virginia Tech and is just starting to be marketed by Vatell Corp. The sensor is made using thin-film microfabrication techniques directly on the material that is to be measured. It consists of several thin-film layers forming a differential thermopile across a thermal resistance layer. As illustrated in Fig. 1, the measured heat flux q is proportional to the temperature difference across the resistance layer [2]

$$q = \frac{k_g}{\delta_g}(T_1 - T_2) \tag{1}$$

where k_g is the thermal conductivity and δ_g is the thickness of the thermal resistance layer. Because the gages are sputter coated directly onto the surface, their total thickness is less than 2 μm, which is two orders of magnitude thinner than previous gages. The resulting temperature difference across the thermal resistance layer ($\delta < 1$ μm) is very small even at high heat fluxes. To generate a measurable signal many thermocouple pairs are put in series to form a differential thermopile [2], as illustrated in Fig. 2.

The combination of series thermocouple junctions and thin-film design creates a gage with very attractive characteristics. It is not only physically non-intrusive to the flow, but also causes minimal disruption of the surface temperature. Because it is so thin, the response time is less than 20 μsec [3]. Consequently, the frequency response is flat from 0 to over 50 kHz. Moreover, the signal of the Heat Flux Microsensor is directly proportional to the heat flux. Therefore, it can easily be used in both steady and transient flows, and it measures both the steady and unsteady components of the surface heat flux.

A version of the Heat Flux Microsensor has been developed to meet the harsh demands of combustion environments. These gages use platinum and platinum-10% rhodium as the thermoelectric materials. The thermal resistance layer is silicon monoxide and a protective coating of $Al_2 0_3$ is deposited on top of the sensor. The thin-film pattern of all six layers is superimposed in Fig. 3. The large pads are for connection with pins used to bring the signal out the back of the ceramic. In addition to the heat flux measurement, the surface temperature is measured with a platinum resistance layer (RTS). The resistance of this layer increases with increasing temperature. Therefore, these gages simultaneously measure the surface temperature and heat flux. The demonstrated applications include rocket nozzles, SCRAM jet engines, gas turbine engines, boiling heat transfer, flame experiments, basic fluid heat transfer, hypersonic flight, and shock tube testing. The laboratory involves using one of these sensors in a small combustion flame. The sensor is made on a 2.5 cm diameter piece of aluminum nitride ceramic.

Heat Transfer

The fast time response of the temperature and heat flux sensors allows the comparison of time-resolved measurements. The temperature rise of the ceramic substrate is caused by the heat transfer from the flame to the surface. Modeling of the heat transfer allows prediction of the temperature rise for the measured heat flux. Alternatively, the temperature measurement can provide an independent measure of the surface heat flux. If the ceramic piece is considered to be a one-dimensional semi-infinite solid as sketched in Fig. 4, the temperature history is

$$T_s - T_i = \frac{2 q \sqrt{t}}{\sqrt{\pi}\sqrt{k \rho C}} \tag{2}$$

for a constant surface heat flux starting at time t=0. The values of the thermal conductivity (k), the density (ρ), and the specific heat (C) of the ceramic substrate are listed below.

$k = 165$ W/m·K
$\rho = 3290$ kg/m^3
$C = 713$ J/kg·K
$\alpha = 7.03 \times 10^{-5}$ m^2/s

The surface temperature predicted by the semi-infinite solution is correct to within 1.0% for times less than [4]

$$t = 0.3 \frac{L^2}{\alpha} \tag{3}$$

where α is the thermal diffusivity and L the thickness of the substrate, which is 6.35 mm for the given substrate. For times longer than given by eq. (3) the analytical solution loses accuracy. Experimentally the temperature will rise faster than the semi-infinite solution predicts. For the present case the effect should be small for times less than 1 second. A good additional assumption is that the presence of the gage itself does not affect the substrate response of eq. (2).

The sensitivity of the Heat Flux Microsensor, S, is used to convert the voltage signal, E, to heat flux.

$$q = \frac{E}{S} \tag{4}$$

Calibrations give a gage sensitivity of $S=23.2$ mV/(W/cm^2) for the signal from the amplifier box (at 1000X amplification). The equation to convert the RTS signal (at the 500X setting of the amplifier box) to temperature is

$$T_s = (132.33 \frac{^\circ C}{V}) E - 286.06^\circ C \tag{5}$$

Data Processing

Equation (2) relates the temperature change of the surface of a material to a single step change of the surface heat flux. Manipulation of this type of equation gives the temperature response to a varying heat flux signal as a series solution [5]. This can be in the form of a series of impulse functions as shown in Fig. 5. The solution for the temperature response to a varying heat flux input is

$$T_s(t_n) - T_i = \frac{2}{\sqrt{\pi}\sqrt{k\rho C}} \sum_{j=0}^{n-1} q_j \left[\sqrt{t_n - t_j} - \sqrt{t_n - t_{j+1}}\right] \tag{6}$$

Note that this is similar to integrating the heat flux measurements.

In similar fashion the solution for determining the heat flux from a given set of surface temperatures is [4]

$$q(t_n) = \frac{2\sqrt{k\rho C}}{\sqrt{\pi}} \sum_{j=1}^{n} \frac{T_j - T_{j-1}}{\sqrt{t_n - t_j} + \sqrt{t_n - t_{j-1}}} \qquad (7)$$

Note that this is similar to differentiating the surface temperature measurements.

Both of these expressions are in the form needed for digital signal processing. They have been programmed on a computer disc for subsequent processing of the data. A listing of the FORTRAN code is attached. By calculating the corresponding surface temperature response to the measured heat flux and comparing with the experimental temperatures, additional confidence in the measurements can be established. The data processing task is to do this and to quantify the results.

As an additional check of the program for data processing, a manual comparison can be performed using eq. (2). Simply input an average heat flux over the time of the heated portion of the test and compare the surface temperature rise with that calculated with the program. It should be reasonably close.

Data Acquisition

The LabVIEW software (a product of National Instruments) allows the student to formulate an instrument in software. This instrument, referred to as a virtual instrument, incorporates the data acquisition with drivers to support high speed A/D boards, preprocessing algorithms, such as digital filters, and post processing capability. The post processing for this experiment may include the differentiation, or integration of the digital data. This software also provides data formats for storage. Although the LabVIEW software is not required to perform this experiment, the iconified format and the flexibility permit numerous variations in the experiment without extensive software revision. This software is used extensively throughout the laboratory experience to promote the concept of integrated data acquisition, signal conditioning, post-processing and graphical displays.

PROCEDURE

The flame is provided by a propane torch which is aimed directly at the heat flux gage, as illustrated in Fig. 6. The torch has a self igniter which is activated by a button on the front. To ignite the torch the gas is turned fully on by the knob on the back and then the button is pressed. After the flame is adjusted to the desired level the test is started by opening the shutter to allow the flame to impinge on the heat flux gage. A chromel/alumel thermocouple is provided to measure the flame temperature. An amplifier box is provided with the gage to boost the heat flux signal and convert the resistance of the surface RTS to a voltage.

Safety Considerations

Because the students are dealing with a high temperature flame test (1200°C), they need to be reminded of a few safety factors.

1. Do not touch the upper part of the apparatus. When opening and closing the gate, leather gloves may be worn as added protection.

2. Do not leave the flame on for longer than necessary. Always turn it off between tests. Because it does not have active cooling, the entire apparatus will become excessively hot with only a few minutes continuous operation.

3. Do not run the flame on the gage for over a few seconds at a time. Leave the shutter closed at all times except when actually taking data. This should keep the temperatures well below 100°C.

Zeroing the Amplifier

After the amplifier is turned on and allowed to stabilize, both channels need to be zeroed. Set the switch on the amplifier to the zero position. Connect the output from heat flux channel to a voltmeter. Adjust the appropriate pot so that the output reads 0.0 V. Connect the output from the RTS channel (surface temperature) to the voltmeter. Adjust the appropriate pot so that the output reads 0.0 V. When the switch is flipped from zero to test, the output of the heat flux channel should be 0.0 V, but there will be a DC offset of 2 or more volts on the temperature channel. This reflects the resistance of the RTS at room temperature. Because the change in resistance is only millivolts, this offset must be removed from the RTS output before being processed by the computer to maximize the resolution of the A/D conversion. Therefore, a differential amplifier should be used to remove 2 volts from the signal.

Building a Differential Amplifier

Construct the amplifier on the breadboard as illustrated in Fig. 7. An accurate reading of V_{in1} (~ 2 V) should be taken by the voltmeter and recorded to be re-inserted into the computer.

Experimental Tests

Connect the heat flux and differential amplifier outputs directly into the interface box (A/D board). Start the VI on the computer. Adjust the gain and offset for each channel (Hint: Remember the 2 V that was measured and subtracted previously). Adjust Time Base, Interval and Samples/channel accordingly. Record the values for Interval and Samples/channel for each test for use in the FORTRAN code for data processing.

When ready to acquire data, light the propane torch. Immediately after pressing the Acquire Data button open the gate. Each test should last no more than 2 seconds. Save the data and repeat the test until you have acquired at least five sets of data. Try to use varying flame heights and conditions for each test. Observe the flame and make notes to explain the observed heat flux response.

Statistical Treatment of Data

For each test use the FORTRAN code to calculate the surface temperature and heat flux values to compare with the corresponding measured values. Compute the residuals of the measured and calculated data for both the heat flux and temperature responses. Plot the residuals and perform a visual analysis. Discuss whether the data looks random or if there appears to be some bias error present.

Compute the mean and the standard deviation of the residuals. Perform a 95% confidence interval for the mean. Discuss results. Are the values different when computed for the first 1 second of the flame test than for the entire sampling period?

Given that the estimated uncertainty for heat flux is 0.05 W/cm^2 and for temperature is 0.2°C, are the methods presented in this lab acceptable? Use the uncertainty to discuss your error, and discuss which assumptions made in the calculations most likely lead to this error. Provide a plot of the residuals and a tabulated summary of the statistical data.

From the calculated values of the heat flux and surface temperature determine which is a better approximation of the measured data. Discuss the advantages and disadvantages of each method.

Sample Results

Each student should plot comparisons of their measured and calculated data versus time for both temperature and heat flux and the resulting residuals from these comparisons. Sample results are illustrated in Figures 8-11. Students are expected to complete a statistical analysis of their data and present it in tabulated form as shown in Table 1 for the results in the figures.

Table 1 Statistical Results of Temperature and Heat Flux Residuals

	Total Test	Total Test	First Second of Flame	First Second of Flame
	T_s (°C)	q (W/cm^2)	T_s (°C)	q (W/cm^2)
Mean	1.41	-2.69	0.25	-1.86
Standard Deviation	1.80	4.15	0.57	3.62
95% Confidence Interval	±0.25	±0.59	±0.16	±1.0

INSTRUCTOR NOTES

1. Care should be exercised once the test has been run several times because the Heat Flux Microsensor housing becomes hot. Leather gloves can be worn as a precaution when opening and closing the gate.

2. Due to 60 Hz noise from surrounding equipment, a high sampling frequency should be used on the LabVIEW software or an anti-aliasing filter should be used.

3. Results of the sample test are typical. The means of the residuals are close to zero (based on the 95% confidence intervals) only for the first second of the flame. The semi-infinite assumption of the model breaks down for the four seconds of the test time. This is also evident in the large increase of standard deviation of the temperature residuals going from the one second to the four second time interval. Because of the small change in standard deviation of the heat flux residuals, it can be concluded that the data set is ergodic and the residuals are obtained from the sample population throughout the test period. This is not true for the temperature residuals.

4. The students should be able to identify that the calculation from the measured heat flux to the surface temperature (an integration type process) is preferable to the temperature to heat flux calculation (a differentiation type process).

REFERENCES

1. Diller, T. E., and Onishi, S.,"Heat Flux Gage," U.S. Patent No. 4,779,994, Issued 25 October 1988.

2. Hager, J. M., Onishi, S., Langley, L. W., and Diller, T. E., "High Temperature Heat Flux Measurements," **AIAA Journal of Thermophysics and Heat Transfer**, Vol. 7, 1993, pp. 531-534.

3. Hager, J. M., Simmons, S., Smith, D., Onishi, S., Langley, L. W., and Diller, T. E., "Experimental Performance of a Heat Flux Microsensor," **ASME Journal of Turbomachinery**, Vol. 113, 1991, pp. 246-250.

4. Diller, T. E., "Advances in Heat Flux Measurement," **Advances in Heat Transfer**, Vol. 23, 1993, pp. 279-368.

5. Baker, K. I. and Diller, T. E., "Unsteady Surface Heat Flux and Temperature Measurements," ASME Paper No. 93-HT-33, 1993.

SOURCES OF SUPPLIES

1. Heat Flux Microsensor - $1500, Vatell Corp., Blacksburg, VA 24060, (703) 961-3576
2. Heat Flux Amplifier Box - $1500, Vatell Corp., Blacksburg, VA 24060
3. Thermocouple read out - $100, TEGAM, Madison, OH 44057, (216) 428-7505
4. BernzOmatic 2-piece Propane Torch Kit, Model TS 2000K - $18, WalMart
5. LabVIEW - $500 (Educational discount), National Instruments
6. Elenco bread board - $35, Radio Shack
7. Macintosh Quadra 700 computer - $4,000, Apple Computer
8. Texas Instruments 741 OP-AMP - $1, Radio Shack

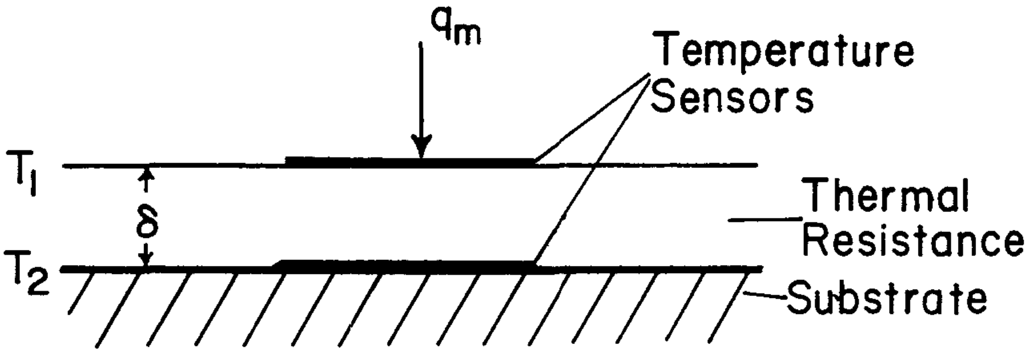

Fig. 1 Measurement Concept of Heat Flux Microsensor

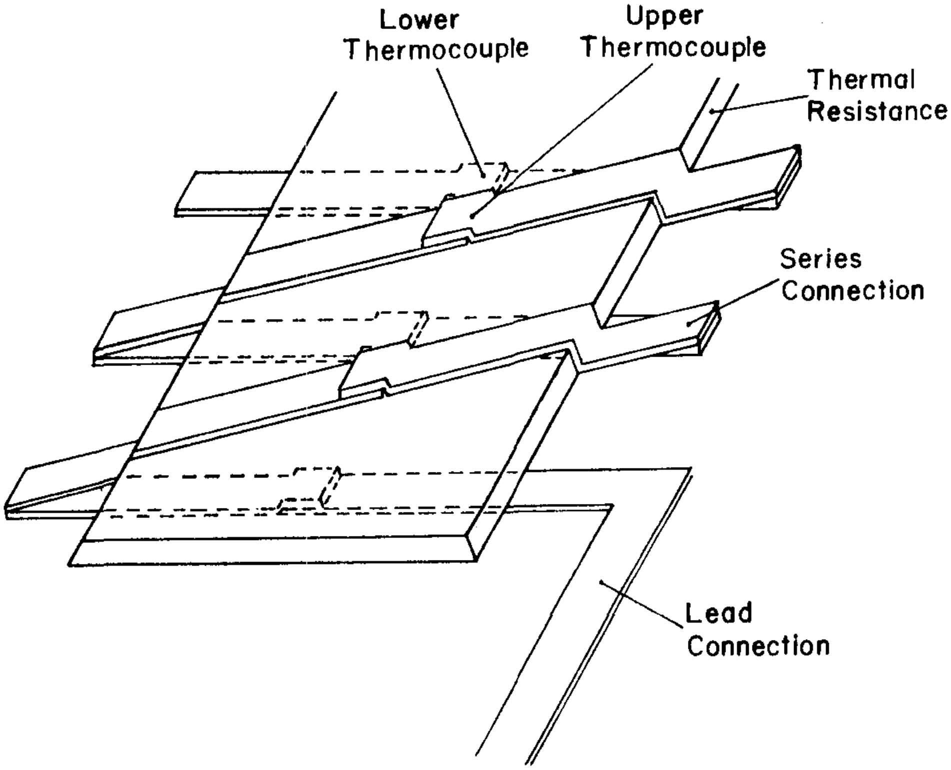

Fig. 2 Differential Thermopile

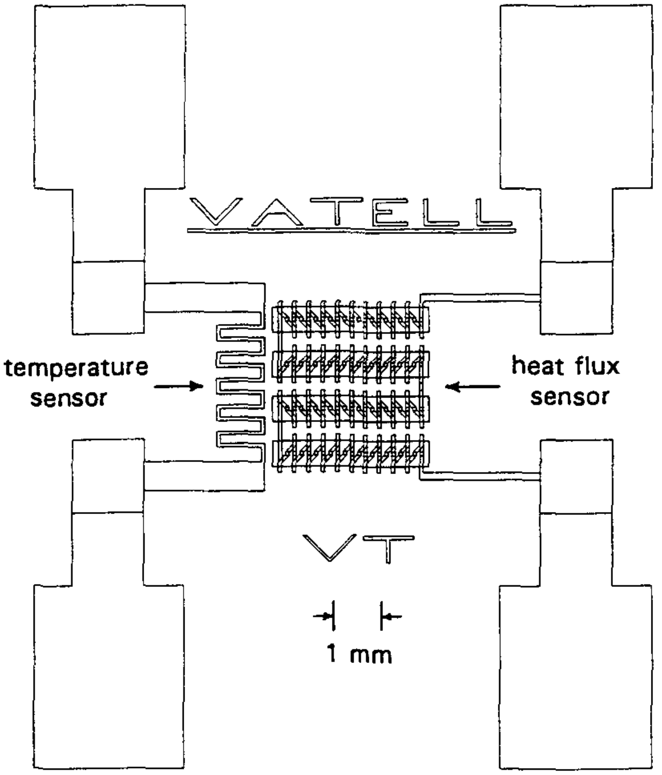

Fig. 3 Overlay Pattern of Heat Flux Microsensor

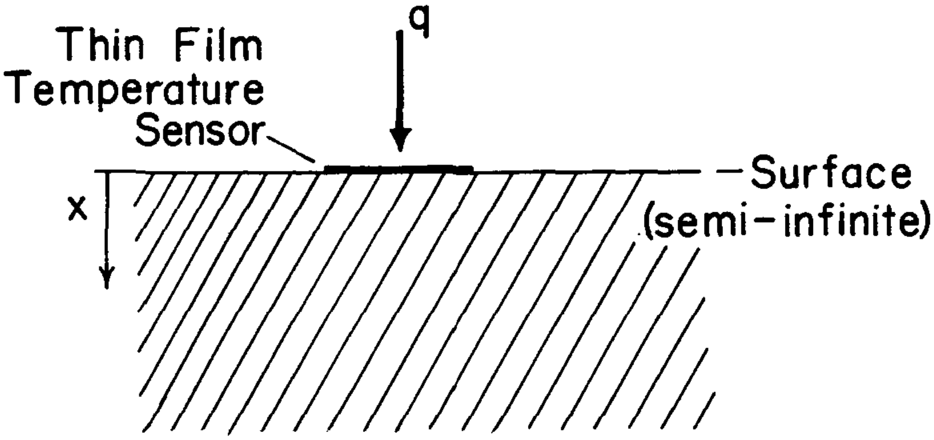

Fig. 4 Semi-Infinite Conduction Geometry

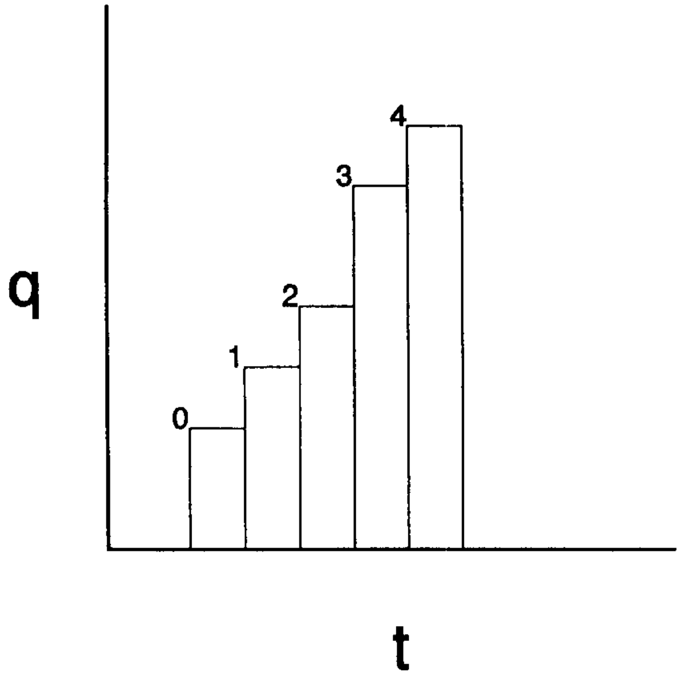

Fig. 5 Impulse Representation of Heat Flux

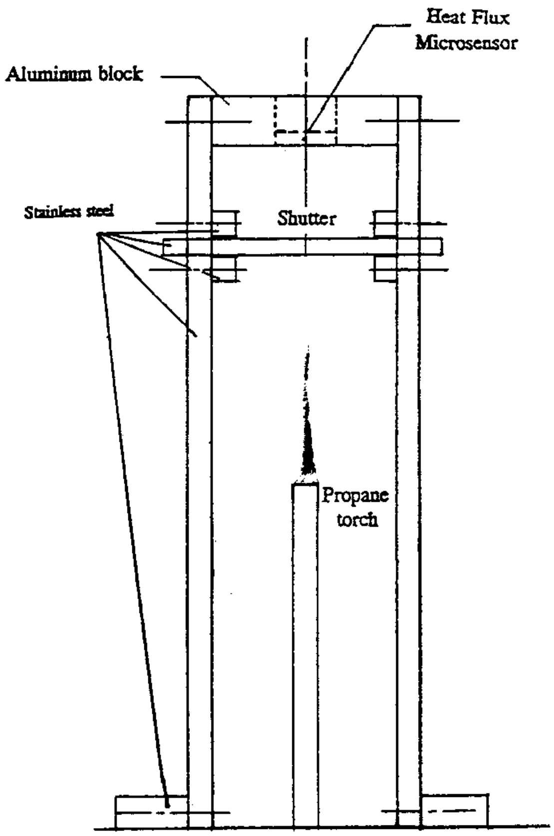

Fig. 6 Experimental Flame Apparatus

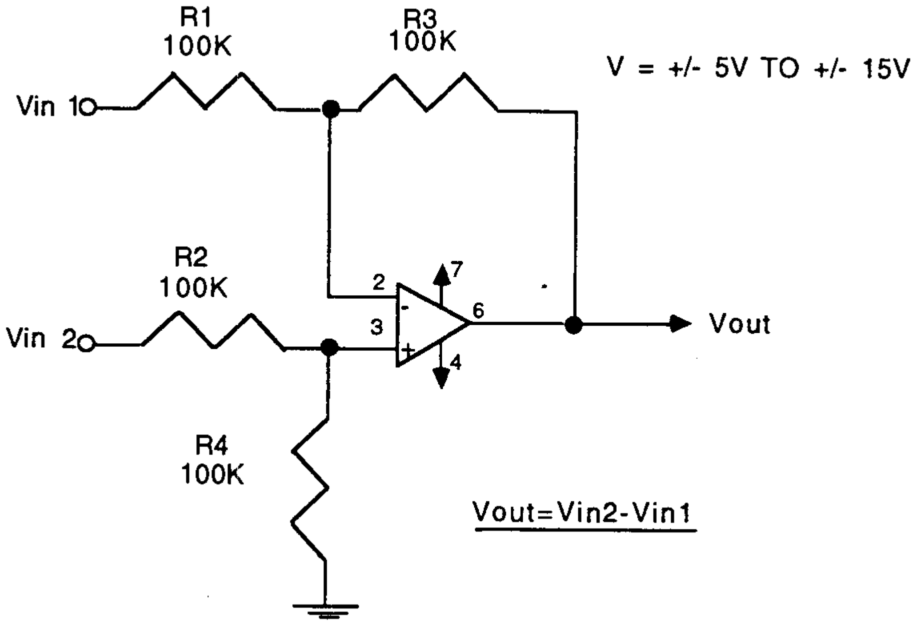

Fig. 7 Differential Amplifier

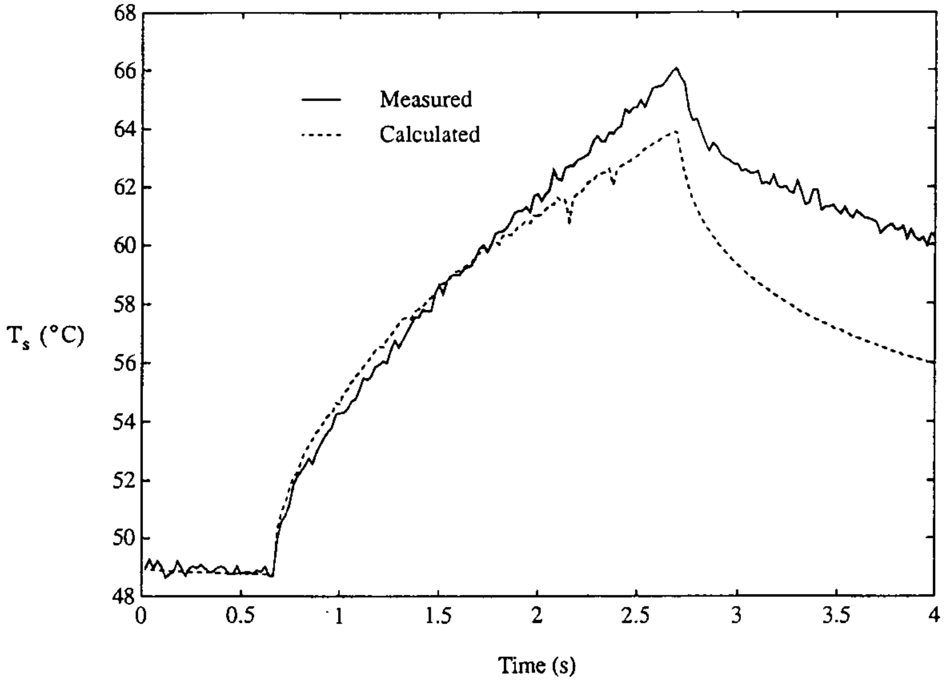

Fig. 8 Measured and Calculated Temperature

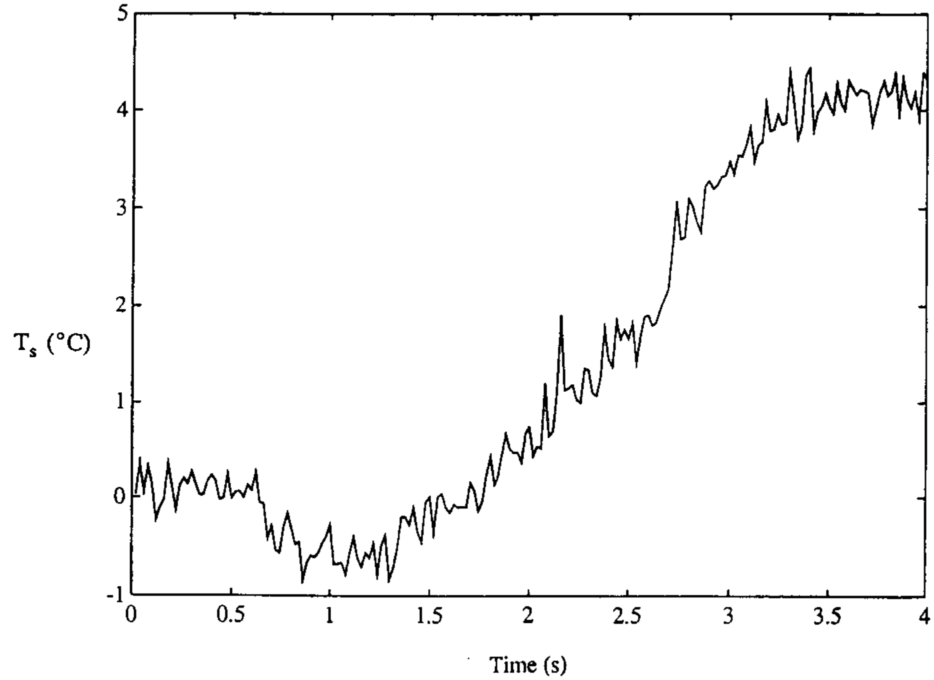

Fig. 9 Temperature Residuals (Meas.-Calc.)

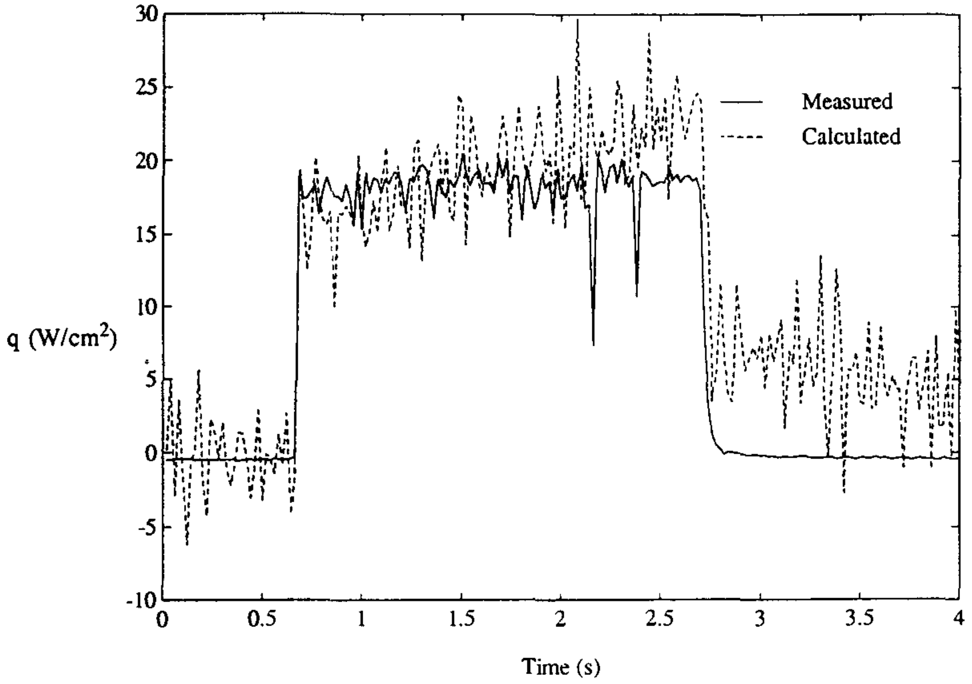

Fig. 10 Measured and Calculated Heat Flux

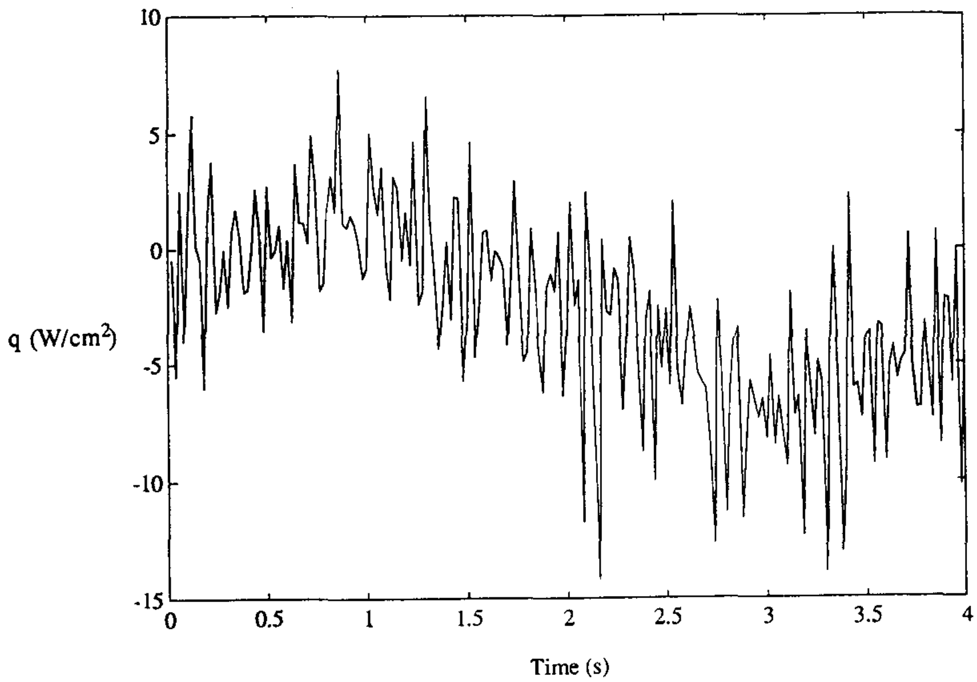

Fig. 11 Heat Flux Residuals (Meas.-Calc.)

```
***********************************************************************
*   This program converts the voltages obtained from the heat flux         *
*   sensor to their corresponding heat flux and temperature                *
*   responses. It also utilizes the Cook and Felderman method to           *
*   differentiate temperature with respect to time to obtain the           *
*   corresponding heat flux and Green's function to integrate the          *
*   heat flux with respect to time to yield temperature. Variables         *
*   used are:                                                              *
*   K...thermal conductivity of resistance layer (W/cmK)                   *
*   RHO...density of resistance layer (kg/cm^3)                            *
*   CP...specific heat of resistance layer (J/kgK)                         *
*   S...sensitivity of sensor (mV/W/cm^2)                                  *
*   ALPHA...thermal property of resistance layer                           *
*   TIME()...time interval                                                 *
*   QVOLT()...voltage corresponding to measured heat flux                  *
*   TVOLT()...voltage corresponding to measured temperature                *
*   QEXP()...heat flux calculated from experimental voltage                *
*   TEXP()...temperature calculated from experimental voltage              *
*   QCALC()...heat flux calculated from temperature                        *
*   TCALC()...temperature calculated from heat flux                        *
*   TIMEF...number of data points taken                                    *
***********************************************************************

      REAL K, RHO, CP, S, ALPHA, TIME (201), QVOLT(201), TVOLT(201),
     +QEXP(201), TEXP(201), QCALC(201), TCALC(201)
      INTEGER TIMEF
      PARAMETER (PI=3.141592654)

***********************************************************************
*   The ASCII data file is opened and an output file is created            *
***********************************************************************

      OPEN (9, FILE = 'RUN3.DAT')
      OPEN (6, FILE = 'RUN3.OUT')

***********************************************************************
*   Definition of thermal properties of resistive layer                    *
***********************************************************************

      K=1.65
      RHO=3.29E-3
      CP=713
      S=23.1765
      ALPHA=K/RHO/CP
      TIMEF = 200

***********************************************************************
*   Do loop to read ASCII file and convert voltages to corresponding       *
*   heat flux and temperature values                                       *
***********************************************************************

      DO 1 I = 1, TIMEF
       READ (9, *) TIME(I), QVOLT(I), TVOLT(I)
       QEXP(I) = QVOLT(I)*1000/S
```

```
        TEXP(I) = 132.33*TVOLT(I)-286.06
  1     CONTINUE

************************************************************************
*     Do loop to perform numerical expression developed by Cook and        *
*     Felderman to calculate heat flux from surface temperature            *
************************************************************************

       DO 2 M = 1, TIMEF
        QCALC(M) = 0
       DO 3 L = 2, M
        QCALC(M) = QCALC(M) + (TEXP(L) - TEXP(L-1))/(SQRT((M -L)*.02)
     + +SQRT((M-L+1)*.02))
  3     CONTINUE
        QCALC(M) = 2.*SQRT(K*RHO*CP)/SQRT(PI)*QCALC(M)
  2     CONTINUE

************************************************************************
*     Do loop to perform method of Green's function to calculate           *
*     temperature from heat flux and write all previously calculated       *
*     values to the output file created above                              *
************************************************************************

       DO 4 N = 1, TIMEF
        TCALC(N) = TEXP(1)
       DO 5 J = N, 1, -1
        IF (J.EQ.1) THEN
          TCALC(N) = TCALC(N) - 2.*SQRT(ALPHA)/K/SQRT(PI)*QEXP(J)
     +    *(SQRT(((N-.5)-(J-.5))*.02)-SQRT((N-.5)*.02))
        ELSE
          TCALC(N) = TCALC(N) -2.*SQRT(ALPHA)/K/SQRT(PI)*QEXP(J)
     +    *(SQRT(((N-.5)-(J-.5))*.02)-SQRT(((N-.5)-(J-1.5))*.02))
        ENDIF
  5     CONTINUE
  20    FORMAT (1X, F16.5, 2X, F16.5, 2X, F16.5, 2X, F12.8)
       WRITE (6, 20) QEXP(N), TEXP(N), QCALC(N), TCALC(N)
  4     CONTINUE

       STOP
       END
```

MICROSCALE SYNTHESIS AND CHARACTERIZATION OF POLYSTYRENE: NSF-POLYED SCHOLARS PROJECT

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Microscale Synthesis and Characterization of Polystyrene: NSF-POLYED SCHOLARS PROJECT

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KEY WORDS: Polystyrene, polymers, film-casting, molecular weight determination, film thickness determination.

PREREQUISITE KNOWLEDGE: The student should be familiar with the care and handling of small amounts of hazardous materials and with simple laboratory techniques normally obtained in a first year chemistry course.

OBJECTIVES: To synthesize a polymer (polystyrene) using laboratory techniques typically introduced in the first year chemistry course. In addition, the concepts of number average molecular weight and weight average molecular weight in polymeric materials, polydispersity, methods of film casting, and film thickness determination are introduced.

EQUIPMENT AND SUPPLIES:

Polystyrene Synthesis:
100 mL corn oil#
1 mL styrene
1-4 mg azobis(isobutyronitrile) commonly called AIBN
2 g Alumina Adsorption 80-200 mesh (Fisher Cat no. A540-500)
50 mL methanol
thermometer#
250 mL beaker#
50 mL beaker
2 small Teflon coated magnetic stir-bars
hot plate equipped with stirring rotor#
small vacuum flask
small vacuum filter funnel
10 X 75 mm test tube
slotted cork
3 Pasteur pipettes

Determination of Molecular Weight by Selective Precipitation (TLC):
Reverse phase thin layer chromatographic plate treated with indicator (Whatman 4803-425)
Polystyrene standards (2K-100K)#
polystyrene sample(s)
5 capillary tubes
1000 mL beaker
plastic ruler
UV lamp#
50 mL graduated cylinder

100 mL stopper glass bottle
Bunsen burner

Film Casting:
0.5g Styrofoam
2 mL chloroform or toluene
1 mL of working solution composed of 40% dichloromethane and 60% diethyl ether
glass rod
watch glass
NaCl or KBr infrared plate
Microspatula or scalpel
glass plate approximately 8 X 8 cm.

Film Thickness Determination:
polymer films of uniform thickness
Infrared Spectrophotometer or Spectrometer#

Quantities are estimated on a per student basis unless otherwise indicated
More than one student may share this equipment

INTRODUCTION: Polystyrene is a familiar polymer with many commercial uses. Its applications range from the clear, high index of refraction, brittle plastic used to form audio cassette and CD cases to the foamed material used in insulated drink cups and packaging material. Polystyrene constitutes 11% of the plastics used in packaging with only High Density Polyethylene (HDPE) and Low Density Polyethylene (LDPE) contributing a larger share (1). So much polystyrene is used today, it is one of six common plastics that manufacturers have assigned an identification code. The code helps recycling efforts. Polystyrene's code is (PS code 6).

During the summer and fall of 1992 several new polymeric experiments were developed by the NSF POLYED Scholars for introduction into the chemistry core curriculum. In this presentation one such project will be discussed.

This laboratory project is recommended for a first or second year laboratory course allowing the introduction of polymeric science to undergraduates at the earliest opportunity. The reliability of the experiments which make up this project and the recognition factor of polystyrene, a material we come in contact with everyday, makes the synthesis and characterization of polystyrene a good choice for the introduction of polymerization to undergraduates. This laboratory project appeals to the varied interests of students enrolled in the typical first year chemistry course and becomes an ideal way to introduce polymers to a wide variety of science and engineering students.

In this sequence of experiments, students will:

a. Purify commercially available styrene by removing its inhibitor by small column chromatography.
b. Prepare polystyrene by using different amounts of inhibitors at constant temperature and time.
c. Determine the molecular weights of the synthesized polystyrene by Selective Precipitation (TLC) using a calibration curve obtained from polystyrenes of known molecular weights.
d. Prepare and evaluate polystyrene films formed by casting methods described below.

Procedures for the synthesis have appeared in the literature and several undergraduate laboratory textbooks and manuals (2-5). However, the authors have found several problems with these procedures. This modified procedure is introduced to improve the experiment and make it more applicable to the lower level undergraduate laboratory curriculum.

First, the scale of many referenced procedures is such that students work with larger amounts of the hazardous material styrene than either of the authors felt was necessary. This is consistent with current trends to adapt experiments to microscale in the interest of safety and cost reduction (4,6,7).

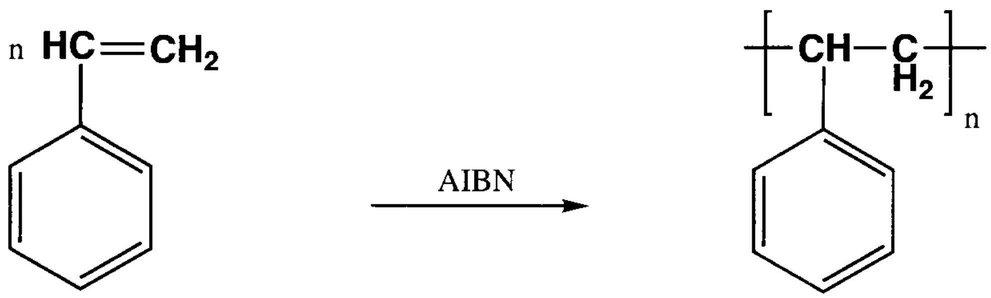
Second, the use of the reagent benzoyl peroxide as an initiator is considered too hazardous for lower level undergraduate laboratories. A suitable substitute is needed to lessen the risk.

Third, in order to accommodate the larger scale of the experiment reported in the literature, the inhibitors present in commercially available styrene are usually removed by distillation or extracted with sodium hydroxide prior to the formal laboratory time. Students lose the pedagogical opportunity to remove the inhibitors from their own sample and thus lose the opportunity to understand its presence in the commercially available sample. The inhibitor, typically 10 to 15 parts per million of 4-*tert*-butylcatechol, is present to prevent the styrene from polymerizing in the bottle. The hazards of working with styrene are also minimized by the microscale of this experiment. In addition, some procedures recommended using an excess of benzoyl peroxide to overwhelm the inhibitors present in the polystyrene. Given our reluctance to use benzoyl peroxide, we were even more reluctant to use an excess. The authors feel that the column method suggested by Williamson (4) has the advantages of being small scale, rapid, and introduces chromatography as a method of purification.

Lastly, the time and temperature required for some of the procedures yield product in the form of a solidified glass plug of polystyrene. The formation of the plug shows the consequence of extensive polymerization, but the solidified product is more difficult to characterize by the equipment available in the lower level undergraduate laboratory. The plug often needs to be removed from the reaction test tube by wrapping the test tube in a cloth and shattering both the test tube and the polystyrene plug with a hammer. In addition, the time needed for the polystyrene plug to form uses too much valuable student and instructor time. Conditions are needed that stop the synthesis short of solidification to allow characterization and make more effective use of laboratory time. By purposely stopping the experiment before a solid plug of polystyrene is formed, the reaction yields a solution that is more easily manipulated than the solid plug often seen in bulk polymerization of styrene. The viscous liquid that forms can easily be transferred and precipitated. Precipitation of the polymer in methanol (a non-solvent) illustrates a common method of isolation and purification of a polymer. If a solid plug forms, the student should grind it with a mortar and pestle to obtain a dry powder.

Experience has shown us that the most successful laboratory synthesis experiments are those coupled with an additional goal besides the formation of the product itself. In this synthesis, an additional aspect of polymerization can be illustrated and the pedagogical function of the experiment and the utility of the students' time are enhanced. For example, by using different amounts of initiator in the synthesis, the effect of initiator on the yield can be investigated. Characterization of the polystyrene is accomplished by film casting and identification by infrared spectroscopy.

In this experiment styrene is polymerized in the absence of solvent. Such a reaction is called a bulk polymerization. The polymerization proceeds as a free radical polymerization of styrene initiated by 2,2'-azobis-(2-methylpropionitrile), more commonly known as azobis(isobutyronitrile), AIBN.



The value n is a function of the experimental conditions and will determine the molecular weight of the resulting polymer. The molecular weight and polydispersity of a polymer greatly affect its physical properties and commercial applications. The molecular weight of small molecules is easily defined. The definition of molecular weight for polymers is not as easily defined because each polymeric chain constitutes a molecule and each chain can be of different length. Definitions and methods of molecular weight determination used for small molecules are not directly applicable to polymers.

Molecular Weights for polymers typically range from 5,000 to greater than 1,000,000. Students need to know how molecular weight differs for small and large molecules (polymers) to understand the effect that molecular weight has on the properties of all molecules. The ability of polymer chains to exist in varying lengths gives rise to a distribution of molecular weights within a polymeric sample. Several types of molecular weights have been defined. Two of the more frequently encountered are the **number-average molecular weight** (M_n) and the **weight-average molecular weight** (M_w). The **number-average molecular weight** is defined as the total mass of polymer molecules divided by the total number of moles of molecules present.

$$M_n = \sum_i n_i M_i \Big/ \sum_i n_i \qquad (1)$$

Molecular weights of this type are typically obtained by methods involving effects on colligative properties, GPC (gel permeation chromatography), and end group analysis.

Another definition of molecular weight is **weight-average molecular weight** which is defined as:

$$M_w = \sum_i n_i M_i^2 \Big/ \sum_i n_i M_i \qquad (2)$$

This value can be obtained by light scattering techniques. Due to the square of the M_i term, this value is biased towards the contribution of the higher molecular weight chains.

The ratio of the number-average molecular weight and the weight-average molecular weight is representative of the molecular weight distribution and is referred to as a sample's molecular weight polydispersity.

$$\text{Polydispersity} = M_w/M_n \qquad (3)$$

This value is related to how broadly the chain lengths are distributed in the polymeric sample. When M_w approaches M_n, their ratio approaches 1, and the sample approaches **monodispersity**. Synthetic polymers exhibit varying degrees of **polydispersity**, with vinyl polymers typically having ratios from 2-10. Many biological macromolecules (polymers) exhibit monodispersity. Determination of the polydispersity of a polymeric sample is important because even small changes in polydispersity can affect the properties of a polymeric sample.

Molecular weight is one of the most important characteristics of a polymer and as previously mentioned, is typically determined by GPC or light scattering techniques. Each of these methods gives meaningful molecular weights but these methods are not accessible to most lower level undergraduate students. An alternative method for determining molecular weight of polystyrene has been reported using Selective Precipitation by thin layer chromatography (TLC) (2,5,8,9). In the TLC procedures, polystyrene samples and standards, over a wide range of molecular weights, are spotted on a reverse phase TLC plate and eluted using a dichloromethane/methanol solution. The Rf is related to the molecular weight of the sample. Using a calibration curve obtained from the polystyrene standards, the molecular weight of the unknown sample is determined.

The coupling of a polystyrene synthesis with a TLC determination was reported by Armstrong (2). Experience has uncovered several problems with these procedures. First, various procedures report that the reproducibility of these methods is very sensitive to the conditions. Yet each procedure requires slightly different conditions. Second, the reverse phase TLC plates are expensive and the cost per student too high for some departments. Third, molecular weights above 100,000 give significantly poorer results than polystyrene samples between 2,000 and 100,000. Many commercial samples of polystyrene have molecular weights above 100,000. Lastly, many polystyrene samples are polydispersed resulting, unfortunately, in a diffuse spot on the TLC plate. The indistinct nature of the spot makes it difficult to assign the "center" and determine the molecular weight.

Our goals for improving this procedure included optimizing conditions, developing a method to recycle any reusable plates, and determining how closely the results obtained by TLC correspond with the molecular weight determined by GPC for the same polystyrene samples. In addition, a method for casting film from the microscale synthesis and assessing its quality was desired.

The experiment discussed in this paper uses thin layer chromatography to determine the molecular weight of polystyrene samples obtained from the microscale bulk polymerization synthesis. The separation of polystyrene by molecular weight is due to selective precipitation of the polymer from the mobile phase. The mobile phase for this elution consists of dichloromethane and methanol. Polystyrene is soluble in dichloromethane and insoluble in methanol. As the chromatogram develops, a solvent gradient is established along the length of the plate. The bottom of the plate is rich in the polymer soluble solvent dichloromethane. As the chromatogram develops, the concentration of dichloromethane decreases and the relative concentration of the non-solvent methanol increases. The less soluble, high polymer weight fractions precipitate first, and as the solvent concentration changes, the lower molecular weights precipitate out at various Rf's.

The developing chamber should be open to the air, away from drafts and without a wick to insure non-equilibrium conditions while the TLC plate is developing. Considerable care must be used in preparing the eluting solvent because even small deviations from the suggested concentrations alter the results. A fresh portion of eluting solvent must be used for each plate because the relative concentration of the two solvents in the reservoir at the bottom of the developing chamber changes during the developing process. A starting concentration of 71% dichloromethane/29% methanol (V/V) as reported by Armstrong (2) gives the best results. This concentration must be maintained to insure reproducibility. A more extensive application of this technique was applied to 47 different samples of polystyrene having molecular weights between 2,000-100,000. Gel Permeation Chromatographic analysis of the same samples showed a correlation (96%) between the bottom of the TLC spot and the weight average molecular weight determined by GPC. Additional correlation (85%) was observed for the midpoint of the TLC spot and the number average molecular weight. The poorer M_n correlation is attributed to the difficulty determining the midpoint of a spot whose top may be somewhat vague. A similar difficulty is not encountered when determining the bottom of the TLC spot.

A polydispersity index for the polystyrene sample can be calculated based on the ratio of M_w/M_n determined by this method.

There has been much concern in the past over the reproducibility and the cost of this experiment. The key to reproducibility is to keep the conditions as nearly identical as possible from plate to plate. If care is taken, this experiment gives reliable results. One method to minimize variation is to divide a 10 cm X 10 cm reverse phase TLC plate into ten lanes of 1 cm each. Assign the first five lanes to standards and the remaining five lanes to students (See Fig. 1). This allows five students to have identical conditions and results in considerable cost savings.

The ability to form films in high molecular weight compounds is one of the characteristic differences between high and low molecular weight compounds. Under similar conditions, high molecular weight compounds form films where low molecular weight compounds form crystalline deposits or oils. The quality of the films formed from polymeric material is proportional to the molecular weight. The formation of a high molecular weight polymer in a reaction is often confirmed by the formation of a cohesive film. Films may be made by either melt techniques or solution casting techniques (10,11). This procedure discusses two methods of solution casting that are applicable to a wide variety of polymers, and is easily adaptable to the undergraduate laboratory. Finding a suitable solvent is the most difficult part of the technique. A suitable solvent is volatile enough to evaporate in a reasonable amount of time but not too volatile so that the film forms holes, bubbles or precipitates before the film formation is complete.

Polystyrene is a glass at room temperature. A piece of clear brittle polystyrene produces sharp edges when broken. If the film is thick enough, it will appear brittle. When warmed in boiling water, the glass transition temperature of 100°C is approached. At the glass transition temperature, polystyrene undergoes a characteristic change from a glass to a flexible thermoplastic. Above the glass transition temperature, T_g, polymer chains have enough energy to undergo conformational changes, resulting in a more flexible polymer. Flexibility on the macroscopic scale is related to torsional mobility on the molecular level (10).

There are several options available in this experiment. The effect of initiator concentration on yield and molecular weight distribution can be determined by running identical reactions using three different initiator concentrations. Each student can be assigned all three concentrations or only one of the three variations. The class results are then pooled to determine the effects of initiator concentration on yield and molecular weight distribution.

PROCEDURE: Microscale Bulk Polymerization of Styrene

Safety Statements:

Styrene is a hazardous chemical with an unpleasant odor. Work in the hood at all times and avoid skin contact with this chemical. The flammability of this chemical should also be respected. There should be no open flames in the vicinity.

Waste Disposal:

All the alumina used to remove the inhibitors from the styrene should be placed in a jar labeled styrene contaminated alumina. All organic solvents should be disposed of in properly labeled waste containers.

Equilibration of Constant Temperature Bath:

1. Fill a 250 mL beaker 1/3 full with oil and place a magnetic stirring bar in the beaker.
2. Heat the oil bath to 80°C on a hot plate equipped with a stirring rotor. In the interest of time, the oil bath may be turned on and equilibrated prior to the beginning of the laboratory period.

Preparation of Styrene: Removal of Inhibitors in Styrene

1. Prepare the mini-column by placing a small glass wool plug in the bottom of a Pasteur pipette and fill it one half full with alumina.
2. Place the mini-column in a hood and add styrene drop wise to the top of pipette. It takes about 4 minutes to elute 2 mL of styrene. If it takes much longer than 4 minutes, the styrene may be contaminated with some polymeric material and very little monomeric styrene will be collected in a reasonable amount of time. A new bottle of styrene should be substituted.

Preparation of Polystyrene:

3. Weigh 0.250 g of freshly eluted styrene and 1, 2 or 4 mg of the initiator, AIBN, in a 10 X 75 mm test tube.
4. Cover the test tube(s) with a slotted cork.
5. Place the test tube(s) in the oil bath at the same time and record time and temperature.
6. Remove the test tubes from the oil bath after 45 minutes.
7. Pour the reaction mixture, immediately after removal from the oil bath, into a beaker containing 20 mL stirred methanol. A white precipitate should settle out.

7a. If the product is difficult to pour, add a small amount of toluene (~0.5 mL) to dissolve it and then pour as described in 7 into 40 mL of methanol.

8. Continue to stir in methanol for another 10-20 minutes.
9. Vacuum filter the solid polymer and wash with methanol several times.
10. Air dry the polymer or dry the polymer in a vacuum oven at room temperature until constant weight.
11. Weigh and save the polymer in a vial. Yields may be calculated on dried samples.

Plot % yield of polymer vs. mg of AIBN and note the effect of initiator concentration on the yield of polystyrene.

The polystyrene may be characterized by infrared spectroscopic identification of a film cast on glass, water, or a NaCl or KBr plate and the molecular weight distribution may be determined by selective precipitation (TLC) or GPC techniques.

PROCEDURE: Determination of Molecular Weight by Selective Precipitation

Safety Statements:
Dichloromethane and methanol are hazardous solvents. Avoid skin contact and breathing of the vapor. Do not look directly at the UV lamp.

Waste Disposal:
Dispose of all organic solvents in a properly labeled container. Dispose of all glass waste in a properly labeled container.

1. Prepare 100 mL of 71:29 stock solution of dichloromethane/methanol by pipetting 71 mL of dichloromethane in a clean, dried stoppered bottle and adding 29 mL of methanol. Mix the solution thoroughly and keep stoppered when not in use. This stock solution is enough for 2 separate slide developments, as 50 mL is needed for each.
2. Using a pencil, very gently, without disturbing the adsorbent, draw a baseline across the width of the TLC plate, 1 cm from the bottom of the plate.
3. Prepare a capillary spotter for each standard or sample that will be examined. Using capillary tubing, draw out the tubing into a fine narrow spotter using a Bunsen burner.
4. Place the plate on a piece of wide ruled paper. This allows for easy visualization of lanes on the TLC plate. If care is taken during the spotting procedure, 10 lanes can easily be run on a 10 cm plate without risk of overlap. This allows for several permutations of standards and samples on the same plate. Typically, 5-7 standards are needed for the calibration curve, leaving 3-5 lanes for samples to be analyzed. The standards and samples should have molecular weights between 2,000 and 100,000 and be prepared by dissolving 5 mg of polystyrene in approximately 1 mL of dichloromethane.
5. Place a standard or sample spot 1 cm from the bottom of the plate in the center of a lane. Care should be taken to avoid overloading the plate at any one site. Check the standards and sample spots with the UV lamp. Care should be taken not to overload the spot but make sure the spots can be clearly visualized under the UV lamp. As the spot develops, the material spreads out and leaves a very diffuse spot after development.
6. Place 50 mL of the stock solution in a clean and dry 1,000 mL beaker. This amount of solvent should be sufficient to cover the bottom of the 10 cm X 10 cm reverse phase TLC plate without being above the spots. Place this beaker or an equivalent developing chamber in a location free of drafts. Do not allow the solvent to sit too long in the developing chamber.
7. Place the TLC plate in the developing chamber gently. Lean the plate against the side of the beaker so that the plate only touches the beaker at the top and the bottom. Do not cover.
8. Do not disturb the plate while it is developing. Remove the plate when the solvent comes within 5 mm of the top of the plate and gently mark the solvent front.
9. Place the TLC plate under a UV lamp. Circle each spot with pencil and record the distance from the baseline to the bottom, the midpoint, and the top of each spot. (See Fig. 1).
10. Determine the R_f of each of the recorded points for each spot by dividing the distance from the baseline by the distance from the baseline that the solvent traveled.
11. Plot log MW of each standard versus R_f for the bottom and the midpoint of each corresponding spot. Obtain 2 calibration curves (See tables 1 and 2). The results of each should correlate best with a second power polynomial fit. If access to a curve fitting program is limited, a reasonable linear fit may be obtained using graphs of Log MW versus R_f bottom and R_f midpoint.
12. Using the calibration curves produced in step 11, determine the molecular weights of the bottom and midpoint for each of the unknown polystyrene samples.
13. Under the conditions discussed above, the molecular weight derived from the bottom of a spot correlates with the weight average molecular weight (M_W) for the sample. The molecular

weight derived from the midpoint of the spot correlates with the number average molecular weight (M_n) for the sample.

14. Calculate the polydispersity index for each sample by dividing M_w by M_n.

PROCEDURES: Film Casting

Safety Statements:

The solvent used to cast the film is hazardous and the procedure should be carried out in the hood.

Waste Disposal:

Dispose of all organic waste in a properly labeled container.

Method 1. Traditional Film Casting on Glass

This method requires significantly more samples than methods 2 and 3 and may not be suitable for small samples. The following procedure is adapted for a polystyrene sample.

1. Place approximately 0.5 g of pieces of polystyrene in a test tube and add 2 mL of chloroform or toluene. Stir until completely dissolved.
2. Pour the viscous solution near the edge of a glass plate. Spread the solution out by rolling a glass rod or Pasteur pipette, whose edges have been wrapped in a layer of tape to allow for a space between the rod and the glass plate, once across the surface of the solution in an effort to form a film of uniform thickness. Allow the solvent to slowly evaporate in the hood. Complete drying may take overnight.
3. Remove the film by lifting the edge of the film from the glass using a sharp instrument (a knife, microspatula, or scalpel). If the film does not lift off the glass surface, the glass plate may be partially submerged in a pan of water and the film should float off the glass plate. Allow the film to air dry after excess water has been removed by gently patting with a paper tissue.
4. Examine the film and assess its quality. Does it appear uniform? Are there any weaknesses or holes in the film? Is it flexible?
5. The film may be mounted on a cardboard holder and its infrared spectrum obtained. If the film is uniform in thickness, then interference fringes will be evident and the film thickness may be calibrated.

Method 2. Film Casting on Water

This method is applicable to small samples and limited to polymers which dissolve in solvents or mixtures of solvents that are immiscible and less dense than water. The following procedure is adapted for a polystyrene sample.

1. Prepare a working solution by dissolving approximately 10 mg of the polystyrene sample in 1 mL of a 40% dichloromethane/60% diethyl ether (V/V) solution. Allow the polystyrene sample to completely dissolve.
2. Add a small amount of water to a clean watch glass to make a puddle approximately 5 cm in diameter. As an alternative, half fill a small recrystallizing dish with water.
3. Add the entire polystyrene solution dropwise to form a layer on top of the water.
4. Observe the surface of the water closely. Almost immediately a film will begin to form. After several minutes the film should have set enough to allow for a qualitative evaluation of the film. If it proves cohesive enough, gently remove the film from the surface of the water and place on a paper towel to dry for 10 minutes. Using the alternative procedure, a glass microscope slide may be dipped under the fragile film in the recrystallizing dish, and raised at an angle to aid in removal of the film from the surface of the water.
5. Examine the film and assess its quality. Does it appear uniform? Are there any weaknesses or holes in the film? Is it flexible?
6. The film may be mounted on a cardboard holder and an infrared spectrum obtained.

Method 3. Film Cast Directly on Warm NaCl or KBr Plates

This method is applicable to solutions of polymeric materials with low molecular weight which do not form quality films and for microscale samples.

This method usually yields the poorest quality films because the polymeric material often precipitates on the plate prior to film formation. If this occurs, substitute a room temperature salt plate to see if this improves the film quality. Fortunately, even if the film quality is poor, an infrared spectrum may be recorded and the material characterized by this method.

1. Dissolve the sample in an appropriate solvent.
2. Add the solution to the center of a NaCl or KBr plate that has been warmed in a 60°C oven. The solvent will flash evaporate leaving behind a film residue of the polymeric material.
3. After the plate is dried, record the infrared spectrum.

PROCEDURE: Determination of the Thickness of a Polymer Film by the Interference Fringe Technique (12)

1. Place the polymer film in the sample beam of an infrared instrument and record its spectrum from 4000 cm^{-1} to 400 cm^{-1}.
2. Determine if any wavy interference pattern emerges in the spectrum. Such a pattern emerges ONLY if the film is of uniform thickness.
3. Using the interference pattern, the thickness of the film can be calculated using the following equation:

$$d = \Delta m/2n(v_1-v_2) \qquad (4)$$

where:

d = thickness of film in cm

Δm = a whole number of complete fringe maxima (or minima) in the interval from u_1 to u_2.

v_1 = frequency at which first maximum (or minimum) occurs, in cm^{-1}.

v_2 = frequency at which last maximum (or minimum) occurs, in cm^{-1}.

n = the index of refraction for the film.

INSTRUCTOR'S NOTES:

This sequence of experiments can be done in teams of two students.

Microscale Bulk Polymerization of Styrene

The mechanism of the free radical polymerization of styrene can be found in the section on polymers and/or vinyl polymerization in any undergraduate organic chemistry text.

The AIBN and Styrene should be keep in the refrigerator when not in use.

The styrene contaminated alumina and pipettes should be placed in a beaker labeled **styrene contaminated alumina and pipettes** and left in the hood until it can be disposed of properly as hazardous waste.

The synthetic experiment should be performed in the hood. Each student is only manipulating one test tube and/or pipette column so that all students should be accommodated in the various hoods. Styrene is extremely flammable and toxic so encourage the use of gloves when handling the monomer. The polymer does not exhibit these health hazards. The difference in toxicity between the monomer and polymer illustrate just one of the many differences between these two compounds.

Benzoyl peroxide is traditionally used to initiate free radical polymerization of styrene, but the explosive hazard of this chemical is considered too risky for a first year experiment. The substitution of AIBN proved acceptable and significantly less hazardous for undergraduates.

All reactions were run with and without a nitrogen purge. The yields without the nitrogen purge were higher and were believed to be due to the loss of monomer during the purging process. The presence of oxygen does not seem to adversely affect the outcome of the free radical reaction on a microscale, although a small induction period is needed before any polymeric material can be isolated.

It is essential the oil bath (corn oil, cooking oil) be equilibrated before the beginning of the lab. I provide settings on various hot plates that should equilibrate close to 80°C. If the temperature goes much higher than 80°C, the polymerization will yield a solid plug of polymer. This material, although polystyrene, is not easily manipulated by equipment available to first and second year students. The precipitation of the polymer in methanol is a classic method for handling polymeric material. Polystyrene is soluble in toluene and dichloromethane but not in methanol or diethyl ether. If a student does form a polystyrene plug (temperature too high or heated too long) and it does not easily slide out of the test tube, try adding a small amount of toluene to dissolve the polymer. If that does not work, then the only method opened to them is to wrap the test tube in a towel and crack it open with a hammer. Unfortunately it is often difficult to differentiate between the silica glass of the test tube and the organic glass of polystyrene when it is all commingled in pieces on the paper towel.

Once precipitated, the polystyrene is filtered using a vacuum filtration setup. The white powder (it does not precipitate as a transparent, amorphous glass) is polystyrene. The yields are obtained by weighing the product. By combining students' data, the class can determine if there is any correlation between the amount of initiator used and the yields obtained. If vacuum filtration is not possible, then gravity filtration can be substituted. This reaction is a chain-growth polymerization and as such will always have monomer present until polymerization is complete. Therefore the precipitation, and, if possible, the filtration (vacuum or gravity) should be done in the hood to minimize the amount of styrene introduced into the laboratory.

Further Extensions: Variation of yields and molecular weight distribution with temperature may be investigated. The authors investigated 60°, 70° and 80°C and found that 80°C gave the highest yields.

Molecular Weight Determination by Selective Precipitation

The characterization by TLC is straightforward. The TLC is best run in the hood in a 1 L beaker that is clean. Soap residue interferes with the separation and gives uninterpretable results. Once the students have obtained the R_fs of both the midpoints and the bottoms of all spots they should plot the results using a plot program, if they have access to one, or using graph paper. A correlation coefficient of 1.0 was obtained for a second order polynomial fit, whereas correlation coefficients of .96-.99 were obtained for a linear fit of the same data (See Tables 1 and 2). This method of determination of molecular weight of polystyrene is applicable to all polystyrene samples whose molecular weight falls between 2,000-100,000. Samples whose molecular weight falls outside this window give significantly poorer correlations with their corresponding GPC data.

From their results, students should be able to determine the M_n, M_w and the polydispersity of their polystyrene sample. By combining students' data, the class can determine if there is any correlation between the amount of initiator used and the molecular weight obtained. As the concentration of initiator increases, the yield of polystyrene should increase. More initiator means more chains started. There should be a corresponding decrease in the molecular weight obtained. There is an inverse square root relationship between the molecular weight and the initiator concentration. It is the authors' observations that students do not get precise enough results to support this mathematical relationship, but correlating trends are observed. The physical justification for this relationship lies in the frequency of termination steps. With more initiator present, more chains start, resulting in more chains termination steps, which causes the average molecular weight to stop increasing.

Recycling of the reverse phase TLC plates results in a significant cost reduction. Recycling can be easily accomplished with only minor modifications in the procedure. These modifications are time costly, as the student is required to spend more time using the UV lamp. The laboratory directors will determine which procedure best serves their needs. Reverse phase TLC plates can be recycled by a method discussed in a separate procedure (13).

Film Casting

Another aspect of this experiment is to examine the relationship between molecular weight and film quality. Film formation is dependent on molecular weight. To form a film, polymer chains must be of sufficient length to have a large amount of chain entanglements. The longer the chains (the higher the molecular weight) and more extensive the entanglements, the stronger the film. For these samples, the students will see film formation on the surface of the water. When they try to remove their samples they will find that the film is extremely fragile and may not maintain its integrity. For comparison, samples of polystyrene made from packing peanuts can be made in a similar manner. These films are normally stronger than the student samples because they are made from polystyrene of higher molecular weight. If a film can be lifted from the surface of the water and placed on a paper towel to dry it can be suspended in the infrared beam and a spectrum obtained. This spectrum should be identical to the polystyrene film in the calibration strip including the distinctive 1601 cm^{-1} peak.

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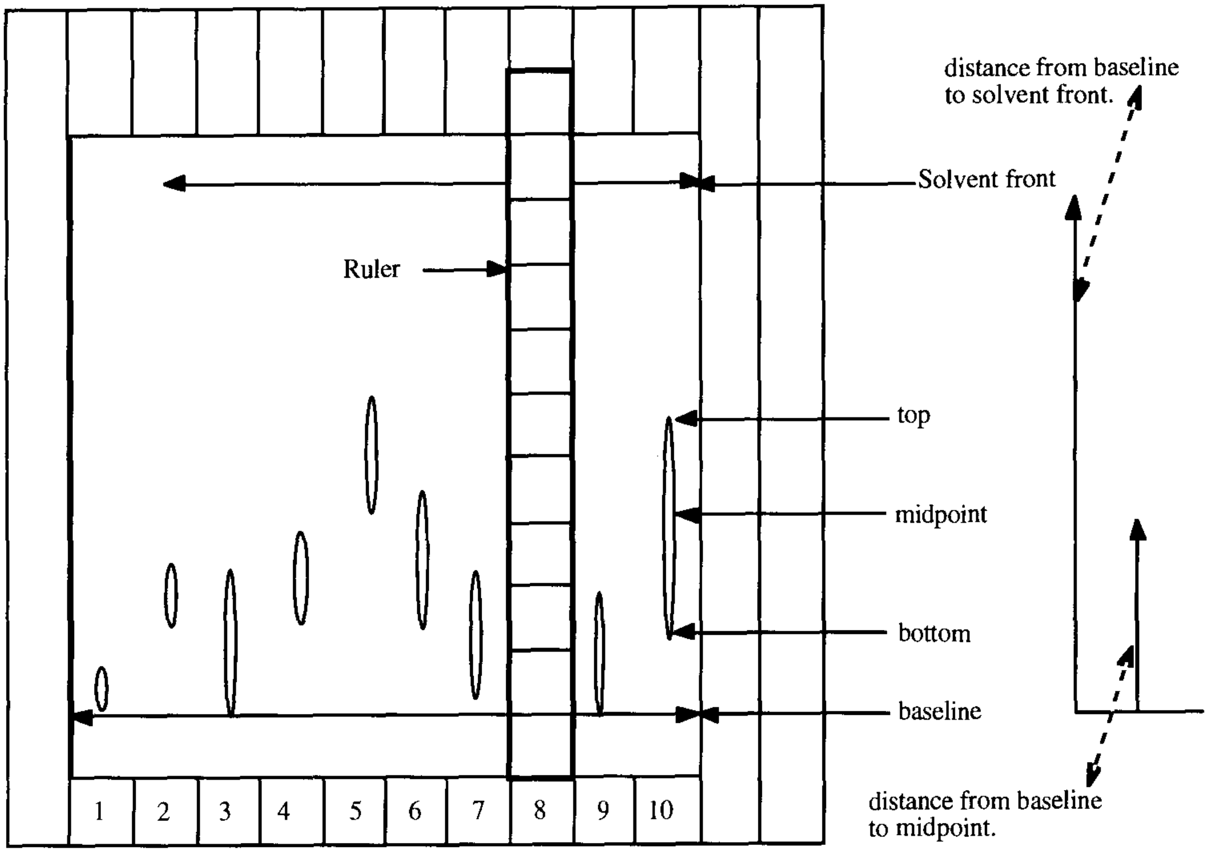

Figure 1. Reverse Phase TLC plate on top of wide ruled paper. The paper serves as a guide for sample lanes. Rf is calculated by dividing the distance travelled by the spot by the distance travelled by the solvent front.

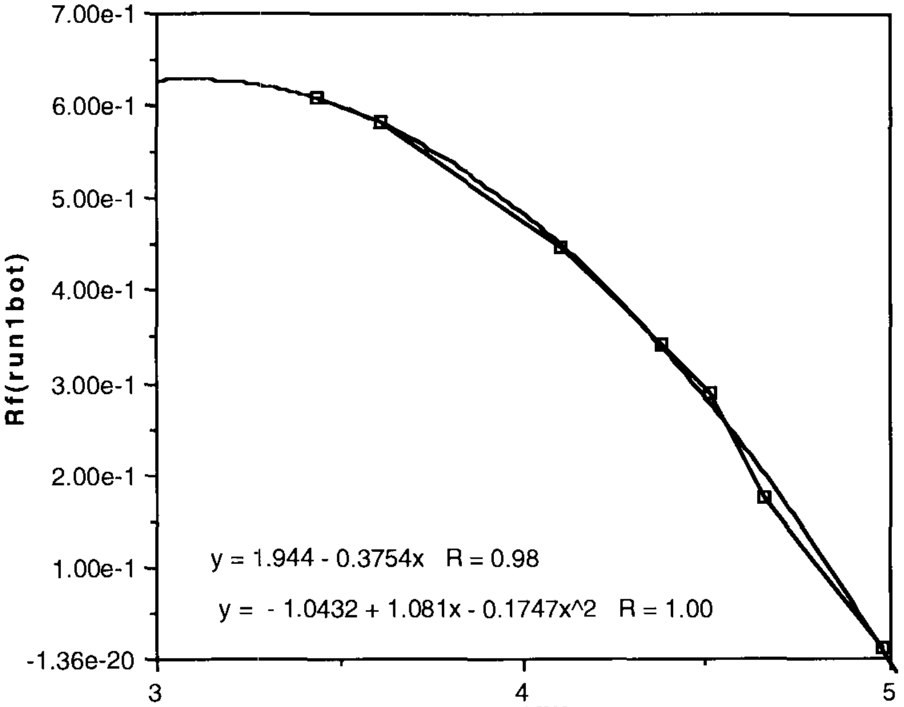

Table 1. Plot of log MW vs. Rf_{bottom} which serves as a calibration curve used to calculate the number average molecular weight using Rf values obtained by TLC. Correlation coefficient for first order fit is .98, and for a second order polynomial fit is 1.

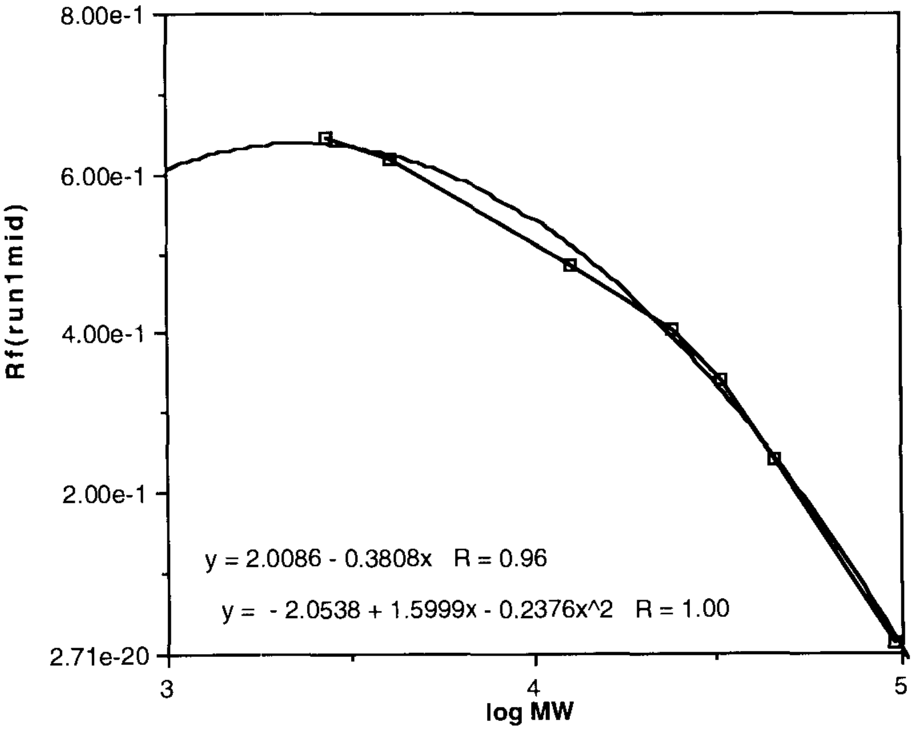

Table 2. Plot of log MW vs. $Rf_{midpoint}$ which serves as a calibration curve used to calculate the weight average molecular weight using Rf values obtained by TLC. Correlation coefficient for first order fit is .96, and for a second order polynomial fit is 1.

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CRATER CRACKING IN ALUMINUM WELDS

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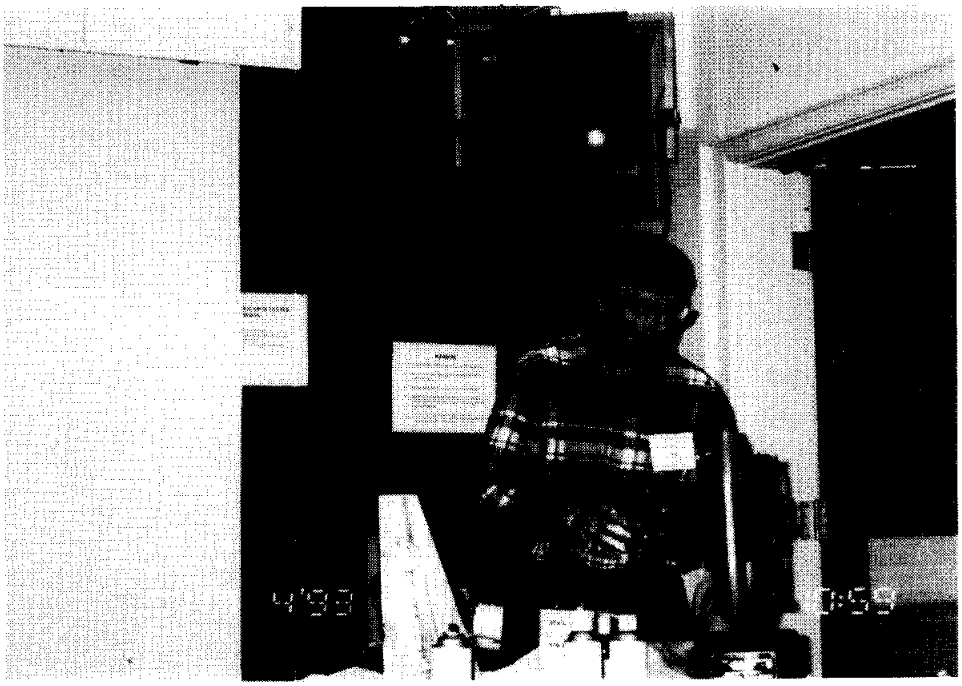

CRATER CRACKING IN ALUMINUM WELDS

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KEY WORDS: Crater, crater crack, spider cracks, hot crack propagation, capillary action, strain hardening, hot short, T-6 Temper, dye penetrant, non-aqueous, heattreatable, dwell time.

PREREQUISITE KNOWLEDGE: The students should have a definitive knowledge of capillary action. The understanding of the non-ferrous characteristics of aluminum will be helpful.

OBJECTIVES: To observe the importance of non-destructive testing even after visual examination. To determine the extent of damage which can be caused by crater cracking.

EQUIPMENT AND SUPPLIES:

(1) Safety glasses, gloves and a vapor mask should be worn.
(2) Fractured aluminum material.
(3) Solvent cleaner. (Aerosol or liquid).
(4) Fluorescent Penetrant. (Aerosol or liquid).
(5) Developer (non-aqueous or powder).
(6) Lint free cloths or paper towels.
(7) Black light.
(8) Three acid swabs. (Brush type is preferred).

NOTE: A tee joint with fillet welds on both sides is the most desirable configuration.

INTRODUCTION: The non-heattreatable alloys, which are cold work tempered, are much less difficult to weld than the heattreatable alloys. Alcoa classifies weldability beginning with "A" as the easiest to "D" the most difficult. The 6061-T6 alloys rate a "D".

PREPARATION FOR WELDING: All aluminum alloys require removal of oxidation. A mix of hydrofluoric or hydrochloric acid with nitric acid and water works very satisfactorily. This should be applied at 70° to 80° F and followed by a thorough rinse. A 5% sodium hydroxide solution may also be used and followed by a thorough cold water rinse. A simple volt OHM meter may be used to determine whether the part is cleaned sufficiently. A high resistance reading will indicate a need for more cleaning.

PERFORMING THE FIRST SIDE WELD: A wire such as ER 5356 may be used to weld the first side which should be a good weld for proving our point. The proper amps and volts should be used. The travel speed should be sufficient to fill the joint on the first side to obtain a .1875" throat depth. A pause at the terminal point of the weld will fill the crater and assure a gas shield. Preheat to 350° F and slow cool.

PERFORMING THE SECOND SIDE WELD: Since we want the second side weld to fail, the procedure will be changed to cause the failure. The precleaning is still a must since an arc cannot be established through a heavy aluminum oxide film. The elimination of preheat will cause the material to receive a thermal shock. The second error to be made is to increase the amperage and voltage to about 30% above the recommended parameters. The travel speed will be varied to cause uneven heat distribution in the base material. The sure fire killer will be provided by a sudden stop and immediate withdrawal of the welding gun from the weld. This assures that a crater will form and no gas shield will be in place to protect the molten, cratered weld pool. The crack may or may not be visible to the eye, but a quick penetrant examination will reveal the spider like fracture.

EXPERIMENT:
PERFORMANCE OF THE DYE PENETRANT EXAMINATION:

STEP 1: Preclean the complete specimen using solvent type, rapid evaporating solvent. Aerosol is faster than plain liquid.

STEP 2: Using the acid swab brush, apply a heavy coat of fluorescent penetrant covering the welds on both sides.

STEP 3: Allow a 7 to 10 minute dwell time.

STEP 4: Spray the solvent cleaner on a lint free cloth or paper towel and remove all the excess penetrant. Do not spray directly on the part to be examined. This will wash out the penetrant.

STEP 5: Apply the developer.

STEP 6: Allow a 7 to 10 minute dwell time.

STEP 7: Turn on the black light and observe the results.

SUMMARY: The black light should show that even though the first side was welded correctly, the propagation has carried the crack from the correctly welded side through to the side that contained the crater.

The student should be made aware that although a failure was depicted in the base material and the weld, aluminum is still a very good weldable material. Any material that is fabricated improperly may fail. Proper procedures are necessary even for the most simplistic forms of metal processing.

SAFETY PRECAUTIONS: Nearly all good penetrant products are flammable. They should not be used on materials hotter than 120° F. No fires or flames should be allowed in the area of use. The vapors are not extremely toxic but prolonged exposure to the products can be harmful. Contact with eyes and skin should be minimized, if not avoidable. Flushing with water and soap will remove the product from skin. A thorough water flush should be used for eye washing. If swallowed, do not induce vomiting. See a physician as soon as possible. Keep all penetrant products in cool storage (around 70° F) and away from possible fires. Secure the cabinet to prohibit "sniffing" by those persons who indulge in such practices.

BRIDGMAN SOLIDIFICATION EXPERIMENT TO ASSESS BOUNDARIES AND INTERFACE SHAPE

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Bridgman Solidification Experiment to Assess Boundaries and Interface Shape

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KEYWORDS: Bridgman solidification, solid-liquid interface shape, directional solidification, thermal conductivity, crucible, II-VI semiconductors, III-V semiconductors, IV semiconductors, and melt growth.

PREREQUISITE KNOWLEDGE: The student should understand the fundamentals of thermal transport phenomena and the fundamentals of solidification processes, specifically directional and Bridgman solidification. This material would typically be covered during a materials science course, an introduction to solidification processes course, introduction to metallurgy, or crystal growth techniques course. The growth interface shape and its relationship to thermophysical properties is the key part of the experiment. [Ref. 1]

OBJECTIVES: To understand the basics of Bridgman-Stockberger solidification using an experimental model that is directly related to II-VI, III-V, and IV semiconductors melt growth like Mercury Cadmium Telluride, Silicon or other similar materials. To observe and understand the interface shape and its relationship to thermal transport and thermophysical properties of the solid, liquid, and crucible materials. NOTE: This lab can be taught at various levels of understanding. The following will simply discuss interface shape models, specifically the Holland effect. The Holland effect relates the solid-liquid (or melt) interface to the thermal conductivity of the solid and liquid (see equation 2). Other topics such as solid interface growth rate and temperature profile within solid - liquid during solidification will be addressed.

EQUIPMENT AND SUPPLIES: [Ref. 2]

(1) Plexiglas crucible (25 mm inner diamter (ID) approximately 600 mm in length)
(2) Aluminum or alloy cold finger (25 mm ID that fits into the Plexiglas crucible)
(3) Liquid Nitrogen dewar (20 liter) or other cryogenic source

(4) Styrofoam dewar-cold finger adapter 2 7/16" outer diameter (O). Anything that would secure the cold finger in the dewar would be acceptable
(5) De-ionized water
(6) Aluminum welding rod-optional (3.175 mm or 1/8")
(7) A meter stick or other measuring device
(8) Stop watch

INTRODUCTION: The thermal conditions in Bridgman-Stockberger (denoted Bridgman for the remainder of the paper) growth solidification configurations can be used to understand the interface shape between the solid, liquid, and crucible wall. The theory proposed by Holland can be used to define the solid-liquid interface shape.[Ref. 1] The Bridgman growth can be modeled using the solidification of water. For good crystallinity, a convex crystal face is required as show by the solidification of lead. Holland's theory may be used to understand the problems of II-VI, III-V, and IV semiconductor crystal growth whose interface propagates with a concave shape.

Bridgman Solidification System Boundary Conditions: A typical Bridgman solidification system is illustrated in Figure 1. Bridgman solidification is directional crystal growth where the loaded crucible moves in the direction from the hot zone into the cold zone where the melt or liquid solidifies. The hot and cold zones are quasi-static for slow solidification. The crucible moves through the stationary furnace at a predefined velocity that defines the growth or solidification rate. A thermal resistance is encountered as the heat flux propagates into the cold zone due to the change in thermal conductivity as illustrated in Figure 1. The corresponding heat flow diverges outward through the walls of the crucible. When the heat flux diverges, the surfaces of constant temperature isotherms must curve up at the edges. The growth face occurs at the freezing temperature isotherm. It should be noted that crystal growth propagates perpendicular to the isotherm representing the solidification temperature[Ref. 3]. The crucible walls form a thermal "short circuit" in the system.

The thermal flux and thermal gradient can be used to explain this phenomenon in a more quantitative way. The thermal flux and thermal gradient boundary conditions at boundaries 1, 2, and 3 (from Figure 1) can be used to define the ruling relationship. Within the crucible wall, sufficiently close to the melt solid interface, the thermal gradients obey (the subscripts m and s refer to the melt and solid phase of the load):

$$\left(\frac{\partial T}{\partial z}\right)_m = \left(\frac{\partial T}{\partial z}\right)_s \qquad (1)$$

The z-component of the thermal flux, the thermal conductivity (k) times the thermal gradient, at the solid-melt interface obeys:

$$k_m\left(\frac{\partial T}{\partial z}\right)_m = k_s\left(\frac{\partial T}{\partial z}\right)_s \qquad (2)$$

Both equations 1 and 2 are valid only if either $k_m=k_s$ or $\partial T/\partial z = 0$ in both media. We know that k_m is not equal to k_s. So, if k_m is greater than k_s, then at the line of contact of the melt-solid interface with the crucible the heat flux is strictly horizontal, into the crucible wall ($\theta=0°$). Similarly, if k_m is less than k_s, as the case with a metal, then the interface turns down at the edge, and $\theta=180°$.

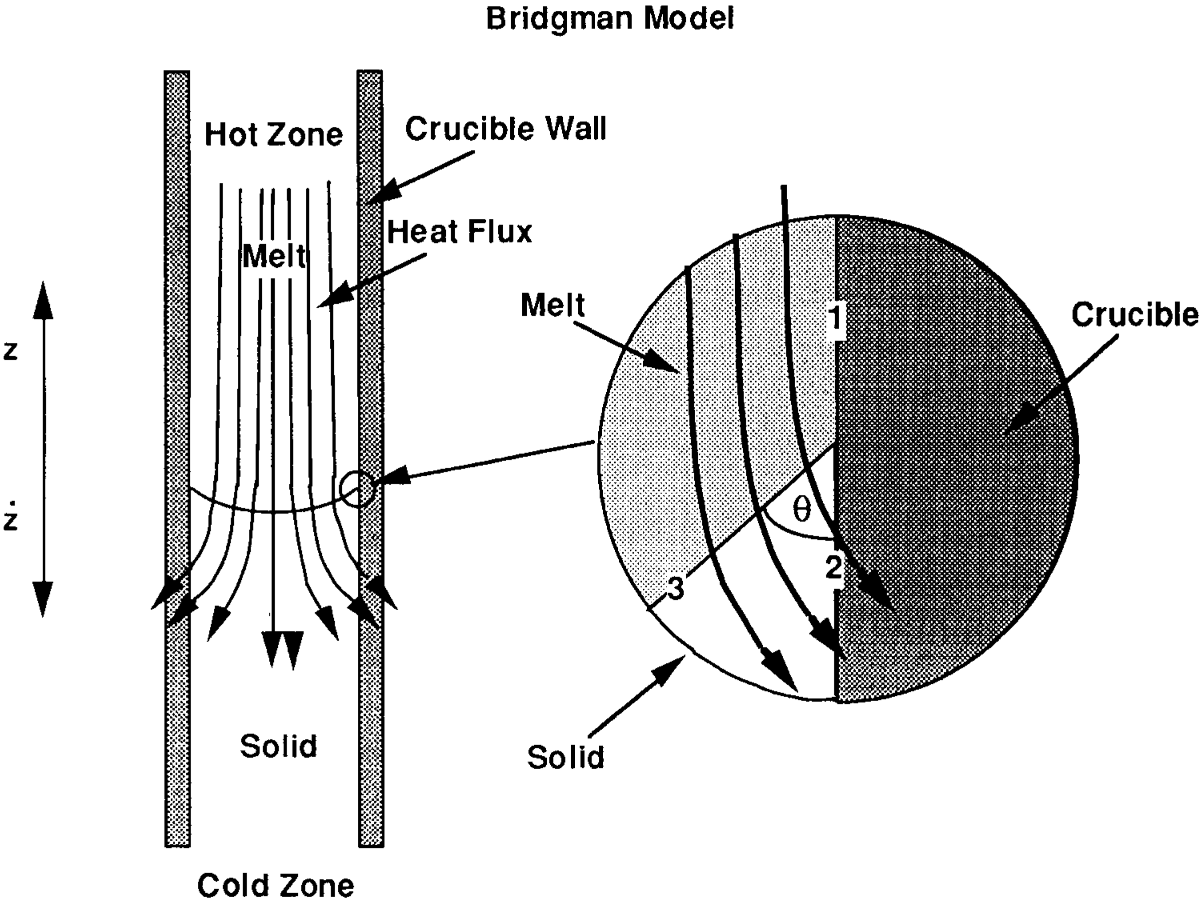

Figure 1. Steady State Bridgman Model Schematic

PROCEDURE:

Safety considerations:

1. Explain the precautions of the use of nitrogen to the student. The laboratory should have proper ventilation.

Sample Preparation: This can be a class demonstration or done by each laboratory team (i.e., you are probably equipment limited).

1. Insert the cold finger into the dewar adapter. [Note: Fabrication drawings for the aluminum cold finger, dewar adapter, and crucible assembly can be obtained from author.]
2. Slip the transparent crucible over the cold finger until it fits over the o-ring. Note: the meter stick does not need to be affixed to the crucible.
3. Fill the crucible with de-ionized water.
4. Insert the cold finger, crucible, and adapter assembly into the LN_2 dewar or other available cryogenic source. Note the starting time in the sample data sheets.
5. Upon solidification of the water note the morphology of the ice. Take measurements of the ice growth length each minute after the ice has fully solidified past the cold finger. Notice the interface shape at the wall of the crucible. Does the ice grow with a concave or convex interface? Predict whether liquid water has a larger thermal conductivity than solid water. Does the solid or liquid have a greater density? Does a relationship exist between the density and the thermal conductivity of the solid and melt material? Consider the packing fractions for the different states of matter.
6. [Optional] Insert an aluminum or other metallic rod into the crucible after the ice has solidified past the cold finger. Let the rod rest on the ice. What happens at the point of contact between the metallic rod and ice? Again, notice the interface shape as the water solidifies up the metallic rod. Again, predict whether liquid water has a larger thermal conductivity than solid water. Does this result agree with the conclusion from step 5.
7. The experiment or demonstration should last at least 30 minutes. Stop the experiment. Remove the cold finger, crucible, and adapter assembly from the LN_2 dewar. Cap the LN_2 dewar.
8. Complete the data sheet. Was the growth rate constant, increasing or decreasing?

SAMPLE DATA SHEETS: Each student should answer all questions given in the sample preparation steps and complete the following data sheet. The growth rate as a function of time should be plotted.

Start time: ________________

Elapsed Time for first solidification: _____________

Elapsed Time (seconds)	Solid Zone Length (cm)	Growth Rate (cm/sec)	Observations
60			
120			
180			
240			
300			
360			
420			
480			
540			
600			
660			
720			
780			
840			
900			
960			
1020			
1080			
1140			
1200			
1260			
1320			
1380			
1440			
1500			
1560			
1620			
1680			
1740			
1800			

Does the ice grow with a concave or convex interface?_____________________________

____

The thermal conductivity of the liquid water is less than or greater than the thermal conductivity of the solid water. Explain your answer.

Predict the growth interface shape assuming the thermal conductivity of the liquid opposite to the observed shape.____

How does the thermal conductivity of the crucible wall matter to this solidification process?___

Was the growth rate constant, increasing or decreasing?______________________________

INSTRUCTOR NOTES:

1. The laboratory instructor should review reference 1. The derivations for equations 1 and 2 are described in this article.

2. The most important lesson from this laboratory is the relationship between interface shape and the thermophysical properties of the solidifying material. Advanced undergraduates or graduate students should relate the growth kinetics to theoretical predictions. Much of the background material for this can be found in references 1 and 3.

3. Other experiments such as that first performed by Azouni [Ref. 2] can be done to understand the phenomena of convection in solidification systems. The Azouni experiment requires a multi-channel (16) temperature measurement capability.

REFERENCES:

1. Holland, L.R., *Crucible Surface, Thermal Refraction, Boundaries, and Interface Shape in Melt Growth,* Journal of Crystal Growth 96 (1989) 577-583.

2. Azouni, M.A., *Helical Impurity Distribution Produced By a Hydrodynamic Instability,* Journal of Crystal Growth 42 (1977) 405-410.

3. Kurz, W. and Fisher, D. J., *Fundamentals of Solidification,* 3rd. ed., Trans Tech Publications, 1989.

SOURCES OF SUPPLY: The plexiglass crucible will have to be purchased from your local plastics retailer. The plexiglass tube should cost less than $10.00 or you may obtain for free as remnant stock. The 1 inch cylindrical Aluminum stock can be obtained and fabricated by your department machine shop. A nitrogen dewar can be found in most Physics departments. The styrofoam adapter can be cut into form using a lathe and knife (or simply cut it into shape) from commonly available packing block material. The de-ionized water may be obtained from the Chemistry department.

PREPARATION OF SIMPLE PLASTER MOLD FOR SLIP CASTING

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PREPARATION OF SIMPLE PLASTER MOLD FOR SLIP CASTING

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KEY WORDS:
model
plaster mold
parting agent
simple mold
cottle
slake
working mold
gypsum

ABSTRACT:

The manufacture of permeable gypsum molds is an important phase of the slip casting process, a process which can economically produce complex shapes. Controlling the water content during the preparation of the mold can affect its porosity, which in turn affects the strength and wear resistance of the mold, casting rate, and ability to transfer detail. This demonstration of making a simple plaster mold involves setting the cottle and model, mixing the plaster, and pouring the plaster.

PREREQUISITES:
Ability to weigh out raw material.
Ability to operate a power mixer.
Ability to slake.

OBJECTIVES:

1. Define keywords
2. Build a cottle.
3. Produce a simple plaster mold.

MATERIALS:
U.S. #1 pottery plaster
20oz. styrofoam cup
parting agent (mold soap)
water
clay

EQUIPMENT:
safety glasses
power mixer
dowel rod
cottle boards with angle irons
carpenter's square
two 5-gal. buckets
modeling clay
wedges
paint brush
rasp

INTRODUCTION:

Many sophisticated methods of manufacturing have been introduced into the ceramics industry, yet complex shapes of products can still be done economically only by slip casting. Slip casting requires a mold which is highly permeable to draw excess water from a liquid clay slurry and leave behind a rigid clay piece which is subsequently fired. This water permeability is a function of the number, size and shape of the pores in the mold material. A second requisite is that the mold material be easily worked, or tooled, into the desired shape.

Gypsum plaster best fulfills these requirements. Naturally occurring raw material gypsum ($CaSO_4 2 \cdot H_2O$) can be heated to a high temperature to yield a material containing less water of hydration. This dehydrated material is called plaster of Paris ($CaSO_4 \cdot \frac{1}{2} H_2O$) and is the starting ingredient for making molds. It is mixed with water forming a creamy slurry which can be easily shaped by casting. The plaster of Paris dissolves in the water and gypsum nucleates and grows from solution. Only 18.6 pounds of water are required for chemical hydration of 100 pounds of plaster, but excess water is required to allow one to work the mixture. Upon drying, the excess water above that required for crystallization is evaporated away leaving behind interconnected pores. These interconnected pores, some of which are open at the surfaces of the mold, create the necessary sponge-like microstructure needed for slip casting.

The manufacture of gypsum molds is an important phase of production, and preparing complicated molds is a highly skilled process. Preparation of a complicated mold involves preparing four types of molds: 1) model; 2) block; 3) case mold; and 4) working mold. The model usually has larger dimensions than the final product to allow for shrinkage which occurs during drying and firing. The block mold is a negative of the model. The case mold is composed of several interlocking pieces formed by casting plaster around the block mold. The working mold is actually used for slip casting the product. The block and case molds are made from harder and stronger gypsum materials to minimize wear and cracking. Gypsum plaster molds are not strong and have a limited life; therefore, a plaster shop maintains a steady production of molds.

This lab will involve producing a simple working mold directly from the model. A simple mold consists of only one part. A complex mold has two or more parts.

PROCEDURE:

A. Calculation of Material Amounts

1. Volume of cottle (V_c):

$$V_c = \text{length} \times \text{width} \times \text{height}$$

2. Volume of styrofoam cup (V_m):

$$V_m = (h\pi/3)\ (R_1^2 + R_1R_2 + R_2^2)$$

3. Volume of plaster mold (V_p) required:

$$V_p = V_c - V_m$$

4. Amount of plaster (P) required is calculated by multiplying the plaster mold volume (Vp) by the density of the plaster (D). The density of the U.S. #1 pottery plaster is 20 g/in^3.

$$P = V_p \times D$$

5. Amount of water (w):

The water to plaster ratio is commonly between 1: 1 $^3/_8$ and 1: 1 $^1/_2$.

$$\frac{w}{P} = \frac{1}{1\ ^3/_8} \quad \text{or} \quad \frac{w}{P} = \frac{1}{1\ ^1/_2}$$

B. Making simple mold

1. Set up cottle by positioning boards using wedges to secure the proper dimensions.
2. Use clay to seal (caulk) the cottle sides and bottom.
3. Fill model with clay to maintain shape and keep from floating.
4. Coat cottle and model with parting agent.
5. Position model in cottle with widest end on table top.
6. Weigh out plaster amount (P).
7. Weigh out water amount (w) separately.
8. Slake the plaster into the water by forming a cone which allows the air to escape. This process ought to take about two minutes.

NOTE: Plaster set-up time is about six minutes total.

9. Mix with a power mixer for 1.5 to 2 minutes. WASH the mixer blade immediately.
10. Insert hand into mixture to check for lumps and release air bubbles.
11. Watch for waves to form as you hand mix. This wave formation indicates plaster is setting up.

NOTE: If you pour too soon, plaster will settle out of the water and the mold will be more dense at the top.

12. Pour plaster into the cottle along its side. Pour enough to cover model. Use dowel rod to keep model in place.
13. Gently shake cottle to release air (rock table). Air bubbles are the enemy of the mold maker.
14. Set for forty (40) minutes.
15. Remove model and wash mold.
16. Use rasp to plane bottom of plaster mold if it is uneven.

INSTRUCTOR COMMENTS:

- Boards nailed together could be used as a cottle.
- New plaster sets up faster, therefore one may need to decrease the time of mixing with the power mixer and by hand.
- To allow for set-up time (in a limited laboratory period) may start students making for which the amounts are previously calculated and given to the students. While the mold sets then show the students how to calculate the amount of gypsum and water needed.
- May divide class into two groups:
    - a) prepare cottle
    - b) weigh materials
- Density varies with ratio of water and the type of plaster.
- Use a three (3) inch blade on the power mixer.

REFERENCES:

1. "The Gypsum Mold", *Ceramics and Glasses, Engineered Materials Handbook, Vol.4*, S.J. Schneider, Jr., ed.; ASM: 1991; pp. 157-158.
2. F.H. Norton, *Elements of Ceramics,* 2nd ed., Addison-Wesley Pub. Co., 1970, pp.99-100.
3. W. Ryan, *Properties of Ceramic Raw Materials,* Peramon Press, 1978, pp.99-100.

SLIP CASTING

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SLIP CASTING

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KEY WORDS:
drain casting
solid casting
reservoir
slip casting
deflocculants
working mold
simple mold
complex mold
electrolytes

ABSTRACT:

The production of highly complex shapes can be accomplished by slip casting. A slip is a suspension of colloidal powders in an immiscible liquid (usually water). Slip casting entails pumping or pouring the slip into a permeable mold (usually gypsum). The capillary suction of the mold causes the liquid to be filtered from the suspended medium and a densely packed layer of particles to be deposited against the mold wall. This demonstration includes the preparation of a slip, evaluating its specific gravity and viscosity, casting simple hollow ware shapes, and solid casting.

PREREQUISITES: Ability to weigh raw material.
Ability to operate a power mixer.
Ability to slake clays.

OBJECTIVES:

1. Define keywords
2. Drain cast.
3. Solid cast.
4. Evaluate viscosity with Zahn cup.
5. Determine specific gravity with pycnometer bottle.

MATERIALS:
ball clay
sodium silicate
kaolin
flint
feldspar
water

EQUIPMENT:

- safety glasses
- power mixer
- two 1-gal. jars
- stop watch
- simple plaster mold
- #1 Zahn cup
- five 2-3gal. buckets
- five 100ml beakers
- pycnometer bottle
- complex bar molds
- dinner plates
- weighing scales
- scoop
- scrapers
- popsicle stick
- calipers

INTRODUCTION:

The term "slip" in the ceramics field refers to a fluid suspension or slurry (a pourable suspension of a high content of insoluble particulate solids in a liquid medium, most often water). All slips require three basic ingredients: a suspension medium or vehicle, fine powdered solid material, and electrolytes or deflocculants.

The suspension medium is usually water and suspended solids are the ceramic raw materials. For ease of casting, the slip must be fluid enough to pour easily but yet be of sufficient viscosity to prevent rapid settling of the suspended solids. This balance is usually attained by the addition of small quantities of selected electrolytes such as sodium silicate, sodium polyphosphate, or many others to the slip.

The electrolytes are charged particles which assist in suspending the solid material in the vehicle. The electrolytes are also called deflocculants. For high clay slips, sodium silicate works quite well.

The ceramics industry uses the terms density and specific gravity interchangeably when discussing slip. The specific gravity (S.G.) of a liquid is the ratio of the liquid density to the density of pure water at 20°C (1g/cc).

One standard test used to evaluate slip is the apparent viscosity which can be performed with a Zahn cup. The Zahn cup method evaluates the number of seconds it takes a liquid to empty from the cup through an orifice of definite diameter.

Slip casting may be used to form hollow ware by draining the excess slip out of the mold when the desired wall thickness is obtained. Solid casting is similar to metal casting in a sand mold. As the water is drawn out of the slip by the plaster mold, the volume of the slip inside the mold will shrink, requiring an ample supply of slip during the time of casting or the piece will be hollow.

PROCEDURE:

A. Slip Batch Preparation

1. Calculate the amount of each of the following raw materials required to yield a dry batch weighing 3000 grams.

Material	%	Chemical composition	Amount
ball clay	30	$Al_2O_3\cdot 0.1K_2O\cdot 4SiO_2\cdot 2H_2O$	
kaolin	18	$Al_2O_3\cdot 2SiO_2\cdot 2H_2O$	
feldspar	32	$Al_2O_3\cdot 0.5K_2O\cdot 0.5Na_2O\cdot SiO_2$	
flint	20	SiO_2	

2. Calculate the amount of the liquids required based on the dry batch weight.

Material	%	Chemical composition	Amount
water	45-50	H_2O	
sodium silicate	0.4-0.7	$Na_2O\cdot xSiO_2$ ($x = 3$ to 5)	

3. Weigh the water into a suitable container.
4. Weigh the sodium silicate which is a liquid. Dip the entire beaker containing the sodium silicate into the water making sure all of it dissolves.
5. Mix the solution with a power mixer.
6. Weigh the ball clay.
7. Slake the ball clay into the liquid mixture and then mix it with a power mixer.
8. Weigh the kaolin.
9. Slake the kaolin into the mixture and then mix it with a power mixer.
10. Weigh the feldspar.
11. Add the feldspar to the mixture and mix it with a power mixer.
12. Weigh the flint.
13. Add the flint to the mixture and mix it with a power mixer.

NOTE: MIX UNTIL SMOOTH OR UNTIL NO LUMPS PERSIST.

B. Slip casting

1. *Simple plaster crucible mold*
    - a) Pour the slip into simple plaster mold.
    - b) Allow the mold to soak while maintaining a "cap" (an excess of material at the top of the mold).
    - c) Pour off the slip and invert the mold on a dinner plate for two (2) minutes.
    - d) Position mold upright for one minute.
    - e) Re-invert for two (2) minutes.
    - f) Remove crucible from plaster mold after approximately 45 minutes.
    - g) Measure wall thickness with calipers.
    - h) Weigh crucible.

2. *Solid casting with a complex bar mold*
   - a) Pour slip into a complex bar mold.
   - b) Continue adding slip to the reservoir (fill chamber) until the mold becomes solid.
   - c) Allow molds to set (approximately two hours).
   - d) After about an hour poke with stick to determine if bars are setting up.
   - e) Separate molds to release bars after slip has hardened (cured).

C. <u>Density and Specific gravity</u>

1. Weigh pycnometer bottle empty.
2. Weigh pycnometer bottle full of water, being sure that the water comes out of the hole in the top of the cap as the cap is positioned. Dry off the bottle.
3. Weigh the pycnometer bottle with slip again being sure that the slip has come out the hole in the top of the cap as the cap is positioned. Clean and dry the outside of the container.
4. Calculate the specific gravity using the following formula:

$$\text{Specific gravity} = \frac{(\text{bottle plus slip weight}) - (\text{bottle weight})}{(\text{bottle plus water weight}) - (\text{bottle weight})}$$

D. <u>Viscosity Measurement with Zahn Cup</u>

1. Submerge the Zahn cup into the slip.
2. Fill the cup.
3. Raise the cup out of the slip hanging vertically from finger and start timing with a stop watch. Hold the draining cup about six inches above the liquid level in the container.
4. Allow the slip to flow out of the cup until you can see break in the flux stream.
5. Record the Zahn cup number and time it takes to empty the cup.

INSTRUCTOR COMMENTS:

* Suggested batches to assign to each student:

	% water	% sodium silicate
1	45	0.7
2	50	0.7
3	45	0.6
4	50	0.6
5	45	0.5
6	50	0.5
7	45	0.4
8	50	0.4

* Slip casts better after it has aged about a week.

* Suggested slip casting soak times: five or ten minutes

- Use simple plaster molds of different densities and compare the hollow ware wall thickness and the weights of the castings made in each mold.

REFERENCES:

1. "Slip Control", *Ceramics and Glasses, Engineered Materials Handbook, Vol.4,* S.J. Schneider, Jr., ed.; ASM: 1991; pp. 156-157.
2. F.H. Norton, *Elements of Ceramics,* 2nd ed., Addison-Wesley Pub. Co., 1970, pp. 95-96.
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FROM SAND CASTING TO FINISHED PRODUCT (A BASIC UNIVERSITY-INDUSTRY PARTNERSHIP)

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KEY WORDS: High density green sand molding, green sand, castings, match plates, cores, patterns, foundry sands, fillets, radii, parting line, shrink rules, draft, runners, gates, core prints, core boxes, loose pieces, shell molds, the mold cavity, cope, drag, sprue, riser, permeability, green strength, durometer, the muller, binders, chaplets, the follow board, the bottom board, chills, the pouring basin, vents, qualifying surfaces, and machine locators.

PREREQUISITE KNOWLEDGE: The student should have a basic understanding of metals and basic machining concepts. No in depth knowledge is required.

OBJECTIVES:

1. To understand green sand mold casting with sand cores.
2. To understand permanent mold castings with collapsible metal cores.
3. To demonstrate current technology of high density green sand molding and the foundry's application of just-in-time manufacturing concepts in foundry operations by a plant tour of local foundry.
4. To demonstrate machining operations of castings utilizing current workcell and just-in-time manufacturing concepts by a plant tour of local manufacturing facility.

EQUIPMENT AND SUPPLIES: Castings, match plates, cores, shell cores, patterns, core box, die casting, foundry sands, flask, and tools used in hand casting process. This is a classroom demonstration only showing finished casting and machine part operations (use video to show pouring molten metal).

PROCEDURE:

PHASE I - The professor will demonstrate the making of a mold by hand-ramming of sand and pour an aluminum casting to show the procedures necessary to go from an ingot to a finished casting for shipment (including removal of gate and runner systems). Safety precautions must be taken when operating the furnace to melt the aluminum. When taking the crucible from the furnace, care must be taken not to spill the molten aluminum. A sand pit should be used for the pouring area. Protective clothing must be worn when handling the molten metal. The metal will remain "hot" upon solidification - DO NOT PICK-UP WITH BARE HANDS, USE TONGS. Wait until the casting has cooled to remove the gate and runner systems.

DISCUSSION: Sand castings are produced in large numbers; the molds are made on molding machines. However, in order to design castings, patterns, and all the component jigs, fixtures, and core boxes, one must understand the fundamentals of molding.

Making a sand mold involves the proper ramming of molding sand around a pattern. After the pattern is removed from the sand and the gating arrangement is completed, the mold cavity is filled with molten metal to form a casting. The sand used is a mixture of sand grains, clay, water, and other materials added for specific purposes or properties.

Green-sand molding begins with inverting a loose pattern on a mold board and placing a suitable-sized flask on the board. Loose patterns are used when relatively few castings are needed. A large production rate would call for match-plate or cope-and-drag-plate patterns.

Steps in the construction of a mold made by hand-ramming of sand. (FIGURE 1)

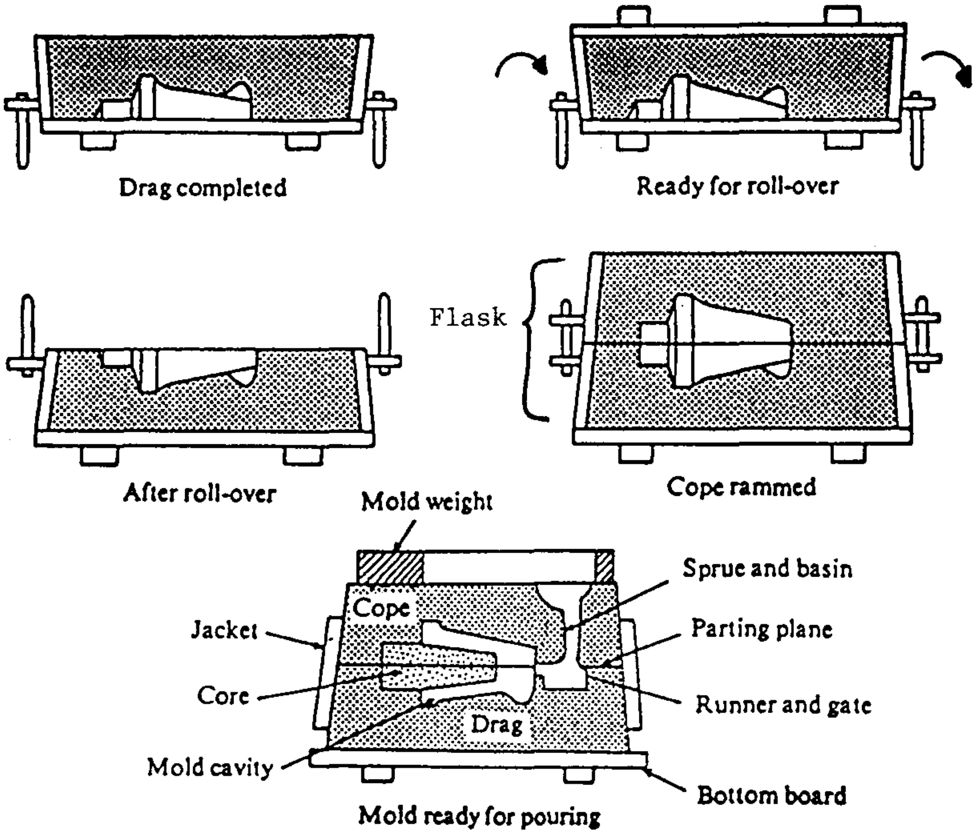


FIGURE 1

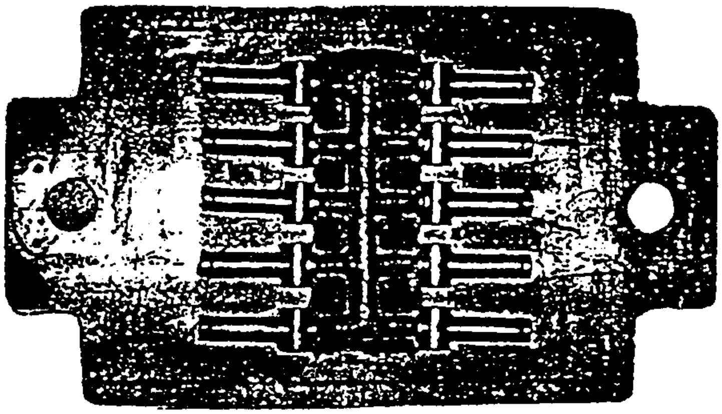
The making of the mold requires the ramming of sand around the pattern within the confines of the flask (cope and drag assembly). As the sand is rammed, hardness and strength are developed making the sand firm and rigid. This makes it possible for the walls of the mold to retain their shape and not collapse or erode when molten metal is poured into the mold cavity.

Steps in mold making using a Match Plate Pattern briefly are:

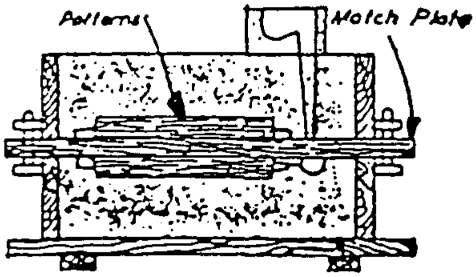
A. Place flask (drag or cope) on match plate, place sprue and riser, (if cope), riddle sand over pattern, rap lightly over pattern and firmly at flask. Strike off, place follow board and roll-over.

B. Remove match plate, inspect or repair and assemble with other mold half.

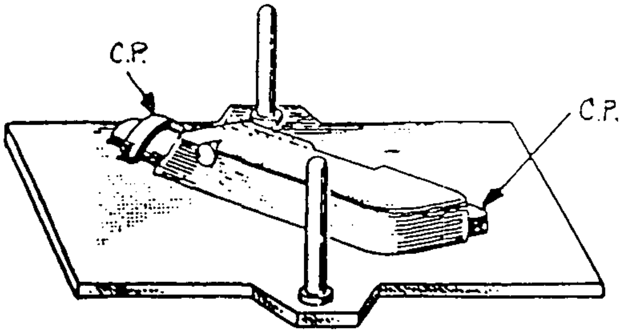
NOTE: Runners and gates are part of match plate. Multiple cavities are possible. (FIGURE 2)



Match plate pattern.



C.P. = Core Print



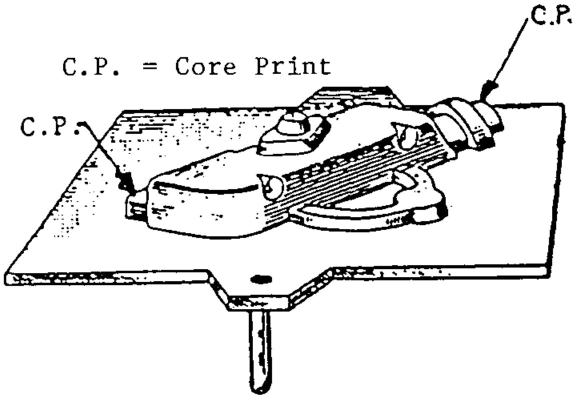


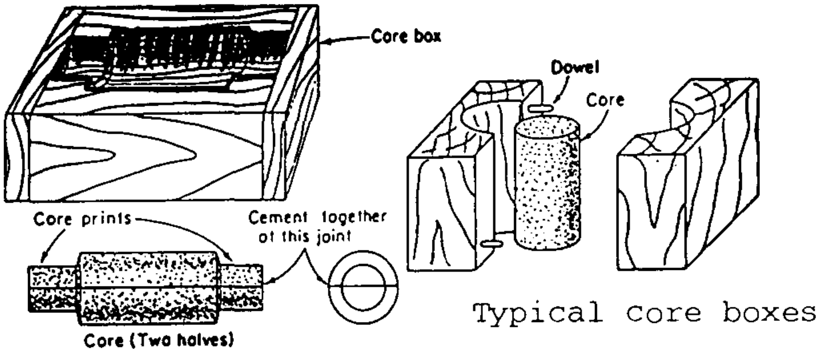
FIGURE 2

C. No parting compound is required with the match plate which is an advantage as it tends to POISON the sand by reducing its bond strength due to its contamination.

Steps in Sand Core Making briefly are:

A. Apply small amount of <u>parting compound</u> to inside of <u>core box.</u>

B. Fill with sand and ram with fingers or hand bench ram to ensure sharp and accurate detail.



Typical core boxes

FIGURE 3

C. Invert onto <u>follow board</u> and rap carefully but firmly on back of core box.

D. Lift core box to expose <u>sand-core.</u>

E. Two sand cores generally are made per casting, are baked and glued together prior to their use in a green sand mold or a permanent mold, just before the mold is assembled for pouring. The core may be "washed" or "dipped" to provide improved casting finish. (FIGURE 3)

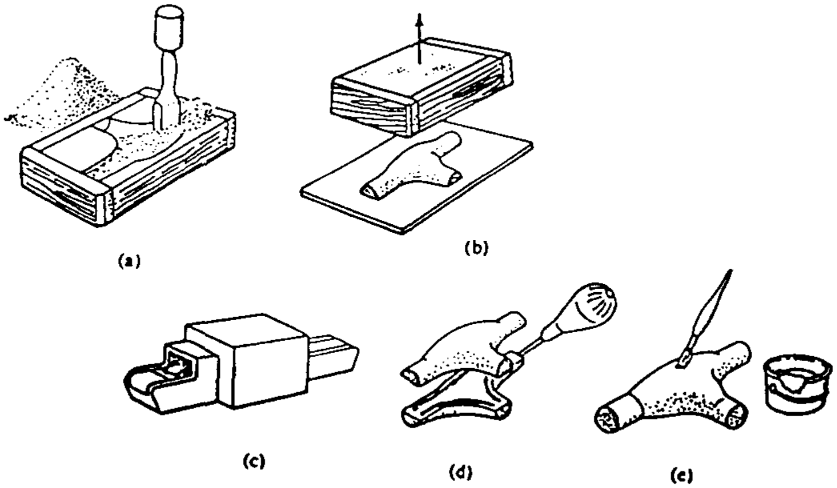


FIGURE 4

Making a core. (a) Ramming core sand, (b) drawing the core box, (c) baking the core half (in a dielectric oven), (d) pasting the core halves, (e) "washing" the core with refractory slurry to improve finish of casting surface. (FIGURE 4)

Importance of Gating Systems:

To increase production and improve the quality of castings produced with the use of molding machines, gating systems are prefabricated and used directly with the patterns. Or a gating system may be an integral part of a match plate, thus eliminating variations in the system. This method is far more reliable than that of hand-cutting the gating system. Once the desired runner system is determined, the designer can create a uniform system for all castings to be produced. Such a system must be carefully engineered to develop the desired characteristics. Various systems of gating are constantly being applied in the production of quality castings of all sizes.

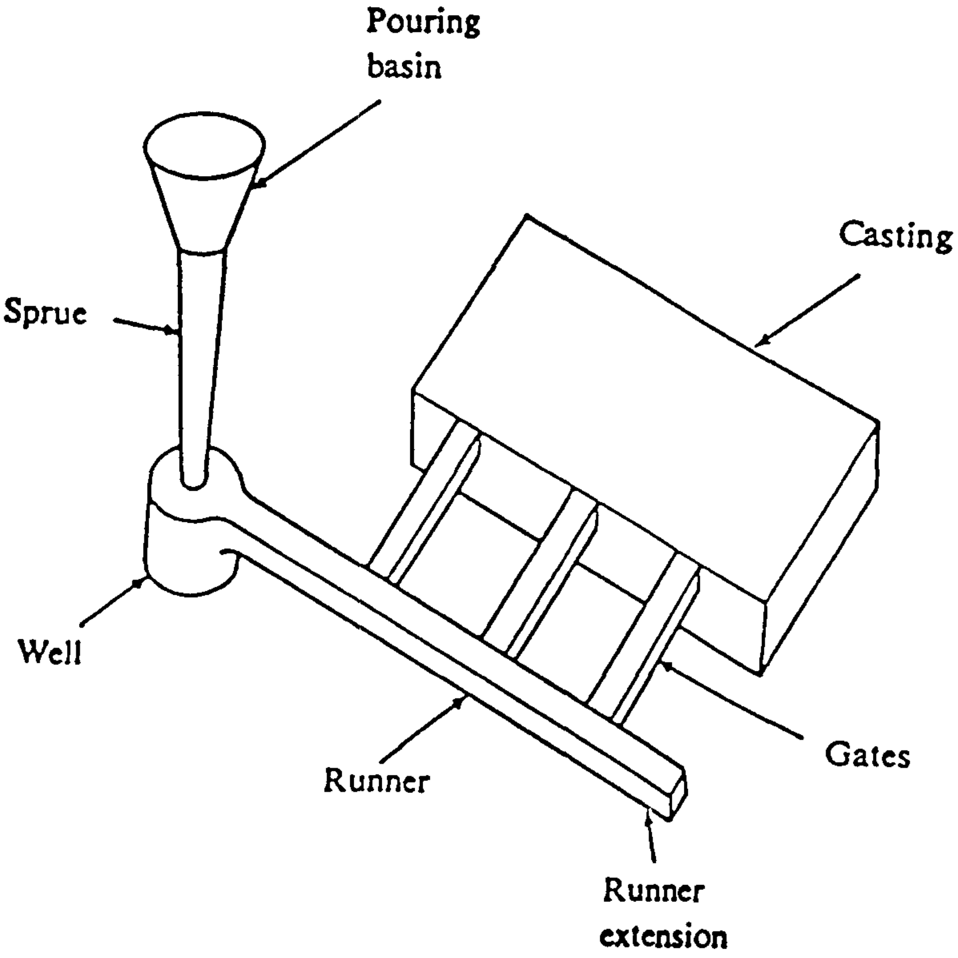
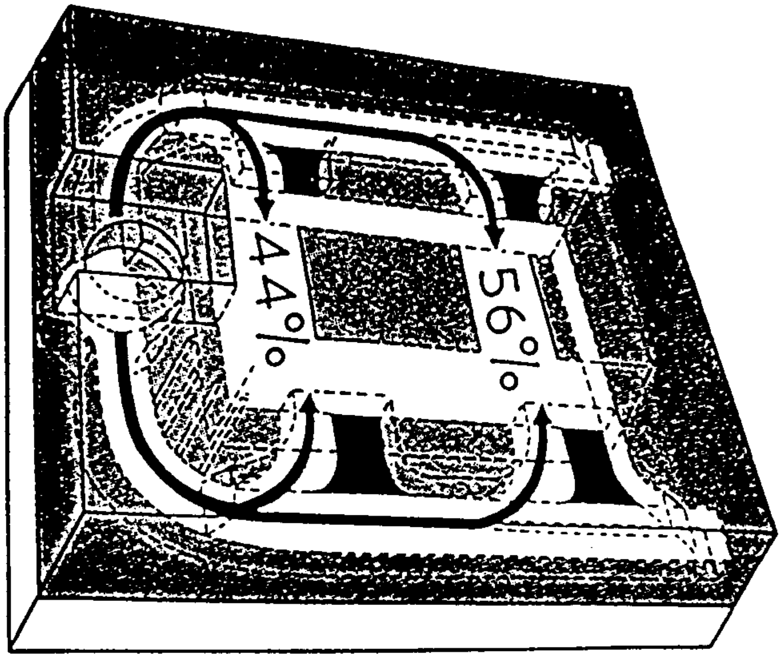


FIGURE 5

A simple parting-line gating system with three gates. (FIGURE 5)



Improved flow conditions obtained by changing sprue-to-runner-to-gate cross sectional area ratio

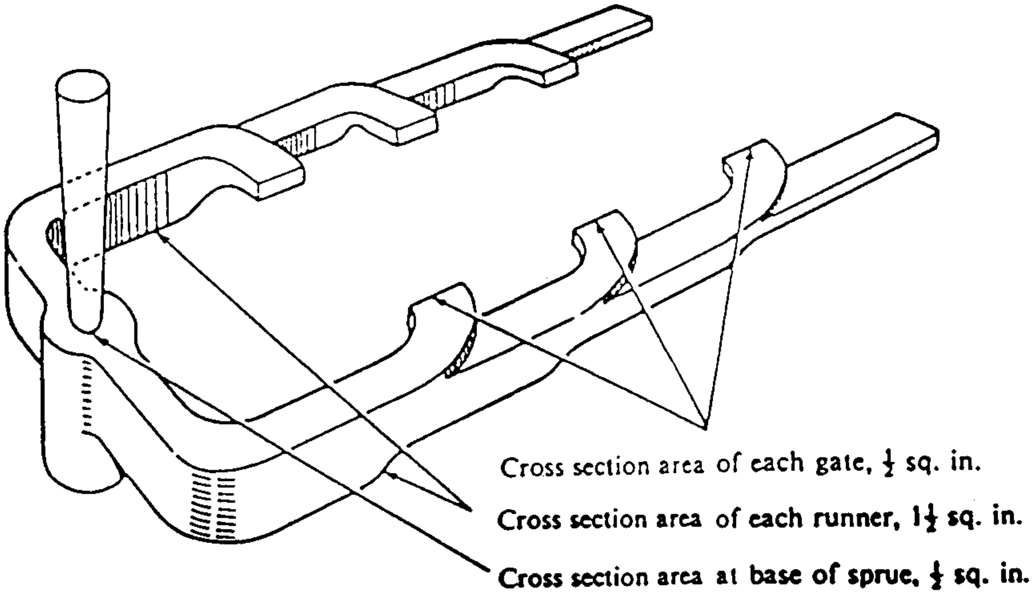


FIGURE 6

Gating system designed to give equal flow from all the gates. Gating ratio is 1:4:4 or 1:6:6 to reduce runner velocities. Cope gates make it possible for the runner to be completely filled before the metal begins to flow through the gates. (FIGURE 6)

This take home exercise is used to reinforce the students' use of library resources and prepare for tour of foundry by learning common terms.

Explain the use and the reason of each of the following terms in foundry work. Do NOT use the same word to define the term!

In Design, what is the use or importance of:

Fillets ______________________________

radii ______________________________

The Parting line ______________________________

In Pattern Making, what is the use or importance of:

Shrink rules ______________________________

draft ______________________________

runners ______________________________

gates ______________________________

core prints ______________________________

core boxes ______________________________

match plates ______________________________

loose pieces ______________________________

shell molds ______________________________

the mold cavity ______________________________

patterns ______________________________

In The Casting Process, what is the use or importance of:

The Cope ______________________________

The Drag ______________________________

The Sprue ______________________________

risers ______________________________

permeability ____________________

green strength ____________________

durometer ____________________

The Muller ____________________

binders ____________________

green sand cores ____________________

baked sand cores ____________________

chaplets ____________________

The follow board ____________________

The bottom board ____________________

chills ____________________

The pouring basin ____________________

parting compound ____________________

vents ____________________

PHASE II - Class tour of local foundry (Auburn Foundry Inc., Auburn, IN) to observe current technology application of high density green sand molding and the foundry's application of just-in-time manufacturing concepts in foundry operation.

Gray iron foundry technology had its beginning in antiquity. Records indicate the Chinese were making crude iron castings as early as the sixth century before Christ. The combination of properties which make gray iron unique and one of the most useful basic construction materials are its strength, high modulus of elasticity (stiffness), resistance to corrosion and heat, easy castibility into intricate shapes, good machinability, and relative cost economy. Gray iron is the material used for countless familiar items such as automotive cylinder blocks, cylinder heads, piston rings, manifolds, brake drums, kitchen and bathroom sinks, bath tubs, machine tools, refrigeration and air compressors, soil pipe, water mains, pipe fittings, electric motor end bells, gear housings for trucks, tractors and farm machinery, etc.

The nature of the foundry process is such that environmental control both inside and outside the plant is a major management concern. In order to attract and retain good employees the foundry must constantly seek to reduce noise, heat, dust, and improve ventilation inside the plant. In order to be considered good community neighbors in this modern day it must find ways to eliminate any air or water pollution. Auburn Foundry has in the past and intends in the future to place as much emphasis on solutions to these problems as to those of customer service.

The tour sequence follows the making and shipping of part number 197C296C flywheel ring for Dana-Spicer Clutch Division.

1. Core Room - Overview of core making operations.

2. Cupola Melt - Includes the charge yard and material composition for specification control. Explanation of cupola furnace controls and use of SPC charts.

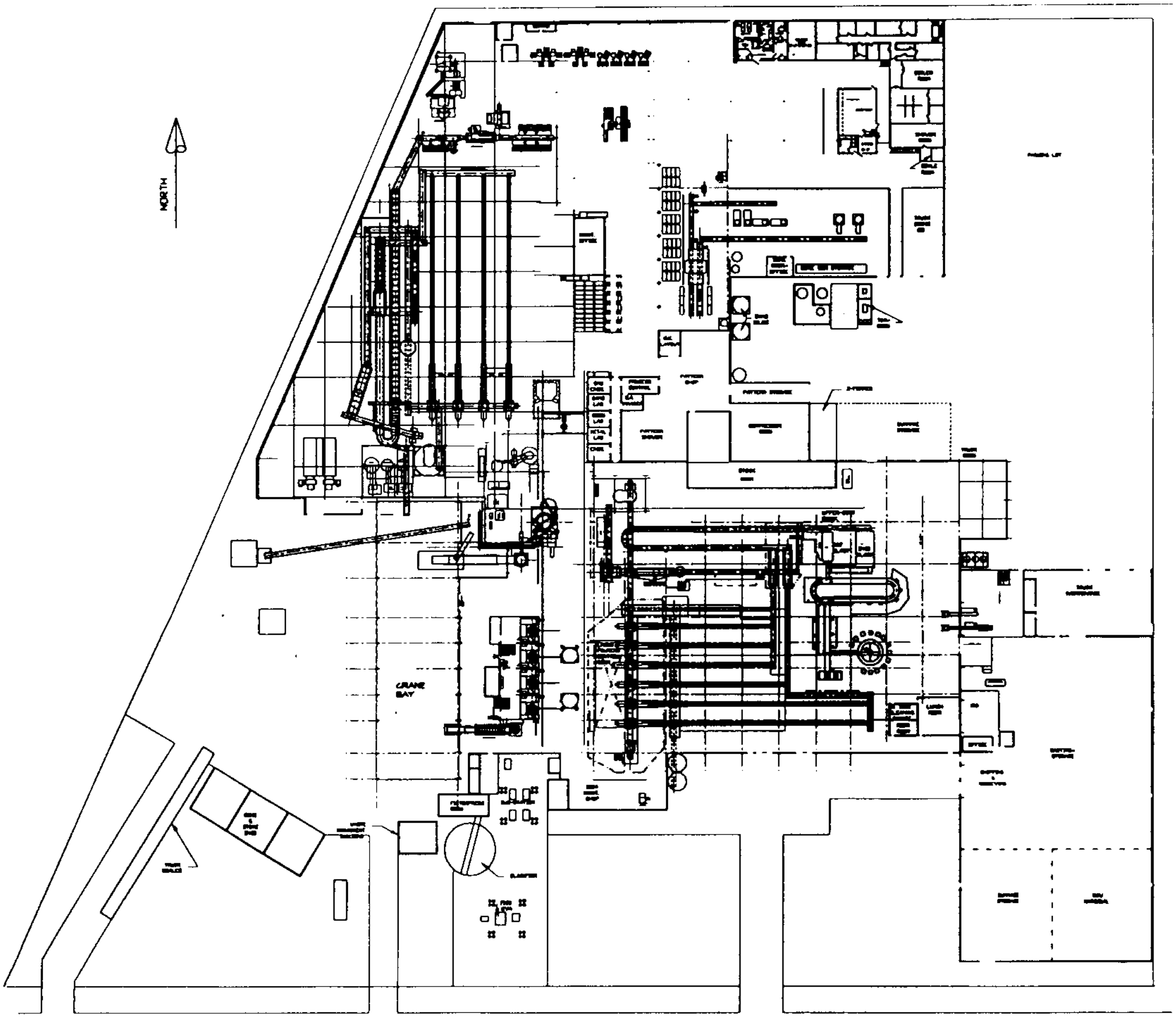
3. Molding Area - High density green sand molding (flaskless molding) will see the total line operation including pattern, sandmold, core setting, and pouring of mold.

4. Quality Lab - Will tour Q.C. Lab and observe sand testing procedures and iron testing procedures. Will review inspector's instruction sheet, process file, microstructure analysis of cast part, and sand data report.

auburn foundry, inc.

SINCE 1911

635-639 WEST ELEVENTH STREET ■ AUBURN, INDIANA 46706 ■ TELEPHONE (219) 925-0900
FAX DAYS—925-5137 EXT. 259
FAX NIGHTS—925-5137
TELEX II—910 2405 797



5. Shake-Out - In this area all molding sand and gating systems are removed.

6. Cleaning - All castings are shot blasted.

7. Grind - Hand grind operation to remove all the excess flashing and gates and runners.

8. Prepare to Ship - In this area the casting is weighed and the quality control tags are completed and attached to the loads. (A load is a skid or woodflat of casting ready for shipment.)

9. Warehouse - All loads must pass through the warehouse for accounting and control.

10. Ship - Final processing of shipping documents and load onto carrier for shipment.

PHASE III - Class tour of a local manufacturing facility to observe machining operations of castings utilizing current technology workcells and just-in-time manufacturing concepts.

Dana - Spicer Clutch Division has set up their plant to operate utilizing workcells. This is the tour sequence to follow the part machining and assembly of part number 197C295 flywheel ring (made from 197C296 flywheel ring casting from Auburn Foundry). The 45 lb. casting is received into Dana operations and 3 lbs of material is removed in metal chips and recycled back to the foundry to become part of their charge yard scrap material.

Sequence of Tour Events

Introduction - 10 minute video presentation on Dana-Spicer Clutch Division

Tour of Manufacturing Area

Most equipment is of the "dedicated" type as opposed to "flexible".

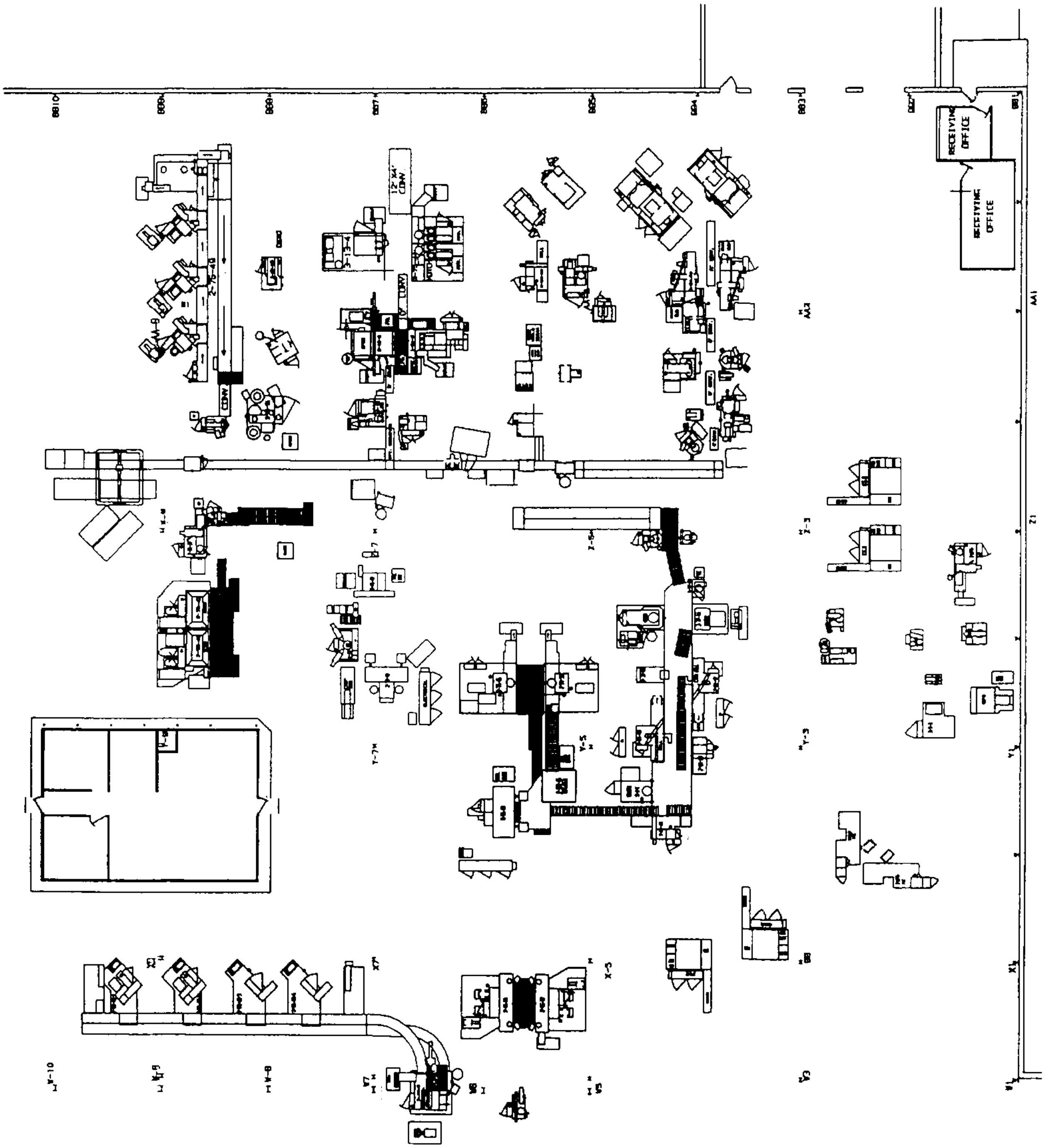
Heavy Duty Assembly is fed (components) like a fishbone (refers to plant flow layout).

1. Receiving

2. "Cell 930"

    A. Lathe OP.5 Face, Turn, Bore, Chamfer (Run W/OP.7)
    Threader Op.7 Thread 11 1/2 - 12 R.H. (Run W/OP.5)

DANA SPICER CLUTCH DIVISION



SPICER CLUTCH DIVISION, DANA CORPORATION
201 BRANDON ST., AUBURN, IN 46706, TEL: (219) 925-3800, FAX: (219) 925-2474

B. Mill OP.10 Mill (4) Slots
(Run W/OP.15)
Drill OP.15 Hollow Mill (6) Embosses
(Run W/OP.10)

C. Mill OP.20 Mill (4) Slots
(Run W/OP.25)
Drill OP.25 Drill (4) 0/32" Holds
(Run W/OP.20)

D. Drill OP.30 Drill (14) Holes
(Run W/OP.35)
Drill OP.35 Spotface (8) & (2) Holes
(Run W/OP.30)

E. Drill OP.40 Tap (2) Holes
(Run W/OP.45)
Balancer OP.45 Balance Flywheel Ring
(Run W/OP.40)

F. Inspection OP.50 Inspection Sell Out Stations

3. Flywheel Cover Assembly

4. Flywheel Cover Testing

5. Final Inspection

6. Packaging and Shipping

REFERENCES:

1. Doyle, Lawrence E., *Manufacturing Processes and Materials for Engineers,* 3rd Ed., Prentice-Hall, Inc., 1985.

2. Hamilton, Ed, *Patternmaking Guide,* 2nd Ed., American Foundrymen's Society, Inc., 1990.

3. *Sand Technology Manual,* Collection of papers from: International Minerals & Chemical Corporation Industrial Group, Industrial Minerals Division, Mundelein, IL 60060. (312)566-2600.

4. Dansk Industri Syndikat, *DISA Foundry Technology,* Printed in Denmark, 1989.

THE ANISOTROPY OF TOUGHNESS IN HOT-ROLLED MILD STEEL

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Kalamazoo, Michigan 49008-5065

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THE ANISOTROPY OF TOUGHNESS IN HOT-ROLLED MILD STEEL

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Western Michigan University, Kalamazoo, MI 49008

KEY WORDS: hot-rolled mild steel, toughness, brittle fracture, anisotropy

PREREQUISITE KNOWLEDGE: Students should be familiar with the basic theory of metallic chip formation and its application in the milling process. They should also have been exposed to steelmaking and they should know that rolling direction strength properties differ from transverse direction strength properties and that inclusion shape has a major effect on experimental toughness measurements. Finally, they should understand why mechanical reliability is improved when processes such as forging are used to manage anisotropy through manipulation of grain flow.

OBJECTIVES: Observe the difference in the toughness of mild steel between the rolling direction and the transverse direction after making test bars on a simple milling machine.

EQUIPMENT AND SUPPLIES:

1. 4" x 1/2" x 18" hot rolled AISI 1018 bar stock
2. Horizontal bandsaw, or equivalent.
3. Metal identification stamps or permanent marking pens.
4. Bridgeport milling machine, or equivalent.
5. Charpy impact test machine, or equivalent.
6. Dial Calipers, 0 to 50 mm span.

INTRODUCTION: A milling machine exercise is described which reinforces important mechanical engineering concepts such as anisotropy, the effect of steelmaking processes on mechanical properties and design against brittle failure.

As preparation for this exercise, classroom lectures should have covered the basic theory of metallic chip formation, the effect of varying feeds and speeds on tool wear, metal removal rates and surface finish and the difference between up-milling and climb-milling. Several textbooks provide an adequate base in this regard. Students should also have been exposed to steelmaking, either in a manufacturing process course or in a materials course. They should know that rolling direction strength properties differ from transverse direction strength properties and that inclusion shape has a major effect on experimental toughness measurements. Finally, they should understand why mechanical reliability is improved when processes such as forging are used to manage anisotropy through manipulation of grain flow.

PROCEDURE: The laboratory tasks are uncomplicated. Finished Charpy V-notch impact test specimens have outer dimensions of 10mm x 10mm x 50mm. Since we wish to machine specimens that are either parallel or transverse to the rolling direction and are from the same piece of steel, it is convenient to purchase 4" x 1/2" hot rolled AISI 1018 bar stock in a convenient length. The instructor must first teach the safe operation of the machine tools to be used in the exercise. Then the next task requires cutting the bar stock into 1/2" x 4" strips, some perpendicular and some parallel to the rolling direction of the steel. This is easily done in a horizontal bandsaw but any metal cutting saw can be used since dimensional control is not critical. Stamp the samples on their ends according to their orientation in the original bar stock. This can alternatively be done with permanent ink, using a different color for each orientation. In the third task, one of the two saw-cut sides of the strip is milled flat. Any available milling machine will be satisfactory. We have used horizontal spindle Kearney & Treckers and vertical spindle Bridgeports with excellent results. After the first surface is flat, remove the specimen, turn it over and clamp it in the vise again after clearing away loose chips. Then mill the sample to a thickness of 5 mm. Repeat these steps for the other two sides, finish one surface to flatness and then finish the opposite side to flatness and the specified 5 mm width. The 1.5 mm deep V-notch is made in the final operation, using a milling cutter which has a 60° included angle on its periphery. A horizontal spindle milling machine is best suited for this operation. If your laboratory is only equipped with the ubiquitous Bridgeport, you can make a non-standard impact bar by tipping the head at a 45° angle and then milling a 90° included angle notch with a conventional cutter. Use a new cutter to insure a sharp radius at the bottom of the notch. In either situation, cut the notch 25 mm from the end of the impact bar that has the identification stamp and cut the notch to the same depth for all bars. After the notch has been cut, it may be necessary to saw away any excess length from the other end. Machine at least three bars for each orientation, ideally six.

Test the bars in a swinging pendulum or drop-weight impact test machine. For best dramatic effect, pick the bars in random order and read the identifying mark after the sample has been broken. Students will quickly learn to correlate lower values of impact strength with samples cut parallel to the transverse direction of the bar stock and higher values with samples cut parallel to the longitudinal direction.

SAMPLE DATA SHEET:

Specimen Number	Specimen Orientation	Impact Energy, Joules

INSTRUCTOR NOTES: Student report formats are usually dictated by instructors. For this exercise, require that three considerations be discussed. Students should write a clear explanation for the gross difference between the two groups of impact strength data. We typically measure impact strengths of about 130 J for samples cut parallel to the rolling direction of the original bar stock and 60 J for samples cut transverse to the rolling direction. A complete answer will discuss the presence of nonmetallic inclusions, their shape and orientation and the resultant effect on crack propagation. A simple statement that the steel has anisotropic properties is not an adequate answer. For the second discussion requirement, students should write a clear explanation for the variance among the impact strength data within each group. We find that the data vary over a total range of 30% to 40% of the mean value. The cause is multi-faceted. Inclusion contents differ; the location of inclusions in the piece may not be the same and the machined dimensions of each sample almost always are different. This is particularly true for the notch depth and bottom radius. A few students may claim that the sample temperatures were not constant and therefore the variation reflects testing within the ductile-brittle transition range. This observation is astute only if your laboratory tends to be quite cold. The observation that the data vary due to experimental technique is not acceptable. Finally, ask students to write a paragraph that discusses the application of these results to the design of structural parts. They should by now have sufficient insight to consider that the rolling direction of parts made from rolled steel bar stock must be considered when specifying section sizes. They should also recall the classroom discussion of how mechanical reliability is improved when processes such as forging are used to manage anisotropy through manipulation of grain flow.

REFERENCES:

1. Guichelaar, P. and M. Kenig, "Nurturing The Mechanical Skills Of Pre-Engineering Students," Pro. 1989 ASEE Annual Conference, pp. 163-166.
2. Kalpakjian, S., Manufacturing Engineering and technology, Addison-Wesley, 1989.
3. Schey, J., Introduction to Manufacturing Processes, McGraw-Hill, 2nd Edition, 1987.

SOURCES OF SUPPLY: Hot-rolled steel is available from most industrial supply houses or steel supply centers.

DEVELOPMENTS IN CARBON MATERIALS

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Telephone 615-576-8595

DEVELOPMENTS IN CARBON MATERIALS

Timothy D. Burchell
Leader, Carbon Materials Technology Group
Oak Ridge National Laboratory

DEVELOPMENTS IN CARBON MATERIALS

Overview of Presentation

The following carbon-based materials will be reviewed and their applications discussed:

- The Fullerenes
- Graphite (synthetic or manufactured)
- Activated carbon fibers
- Carbon-carbon composites

Carbon R&D activities at ORNL will be emphasized.

DEFINITION OF TERMS

SYNTHETIC GRAPHITE

A MATERIAL CONSISTING OF GRAPHITIC CARBON WHICH HAS BEEN OBTAINED BY GRAPHITIZING NON-GRAPHITIC CARBON.

CARBON-CARBON COMPOSITES

A CARBON FIBER REINFORCED CARBON MATRIX MATERIAL IN WHICH THE FIBER AND/OR MATRIX MAY BE GRAPHITIC (GRAPHITE-GRAPHITE COMPOSITE)

FULLERENES

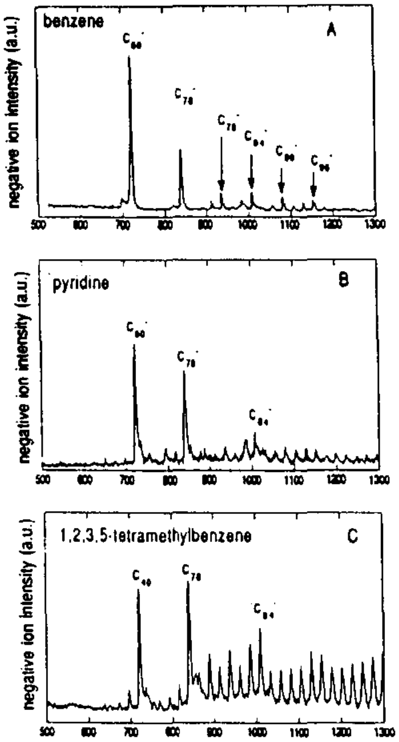
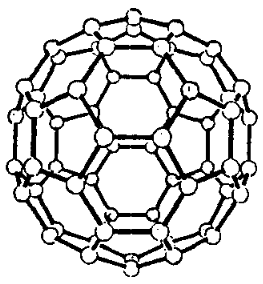
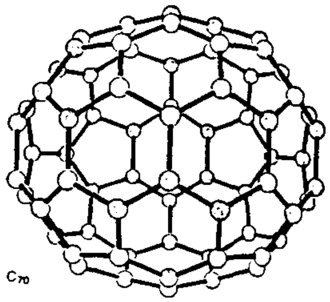


Fig. 4. Laser desorption TOF mass spectra of soot extracts resulting from sequential extractions with (A) benzene, (B) pyridine, and (C) 1,2,3,5-tetramethylbenzene. The spectra are plotted on the same mass scale for comparison.

THE FULLERENES

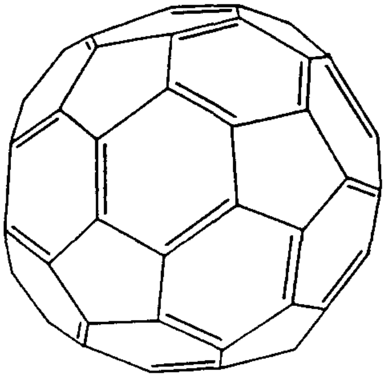


C_{60}

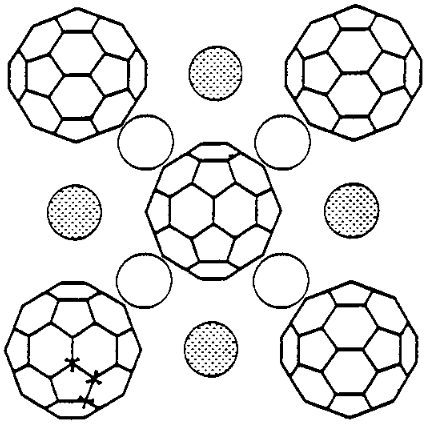


C_{70}

THE FULLERENES



Lowest energy Kekulé structure of soccer ball C_{60}.
Reprinted by permission for *Nature* vol. 329, pp. 529–531;
copyright © 1987 MacMillan Magazines Limited.



Structure of the A_3C_{60} superconductors. The open and hatched spheres represent the alkali atom at tetrahedral and octahedral sites, respectively. Note the two possible orientations of the C_{60} molecules and the three inequivalent carbon sites (marked by arrows on the molecule in the lower left corner) as described in the text. After P. Stephens *et al.*[22]

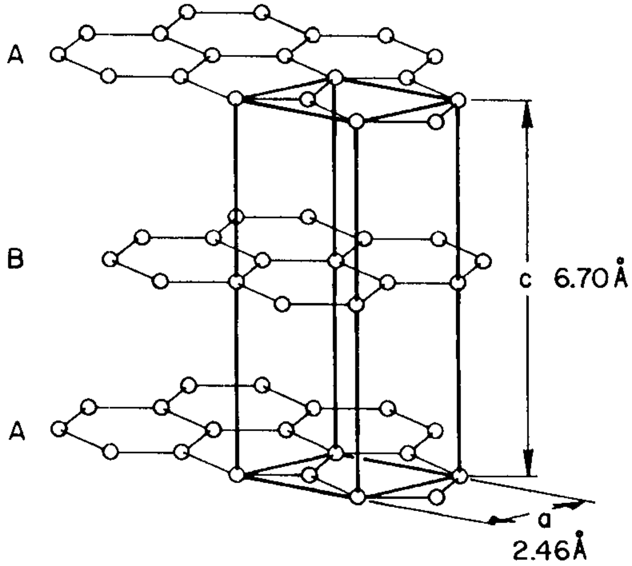
Potential Applications of the Fullerenes

- Pharmaceutical
- Tribological (lubricant)
- Super conductor
- Thermal insulator

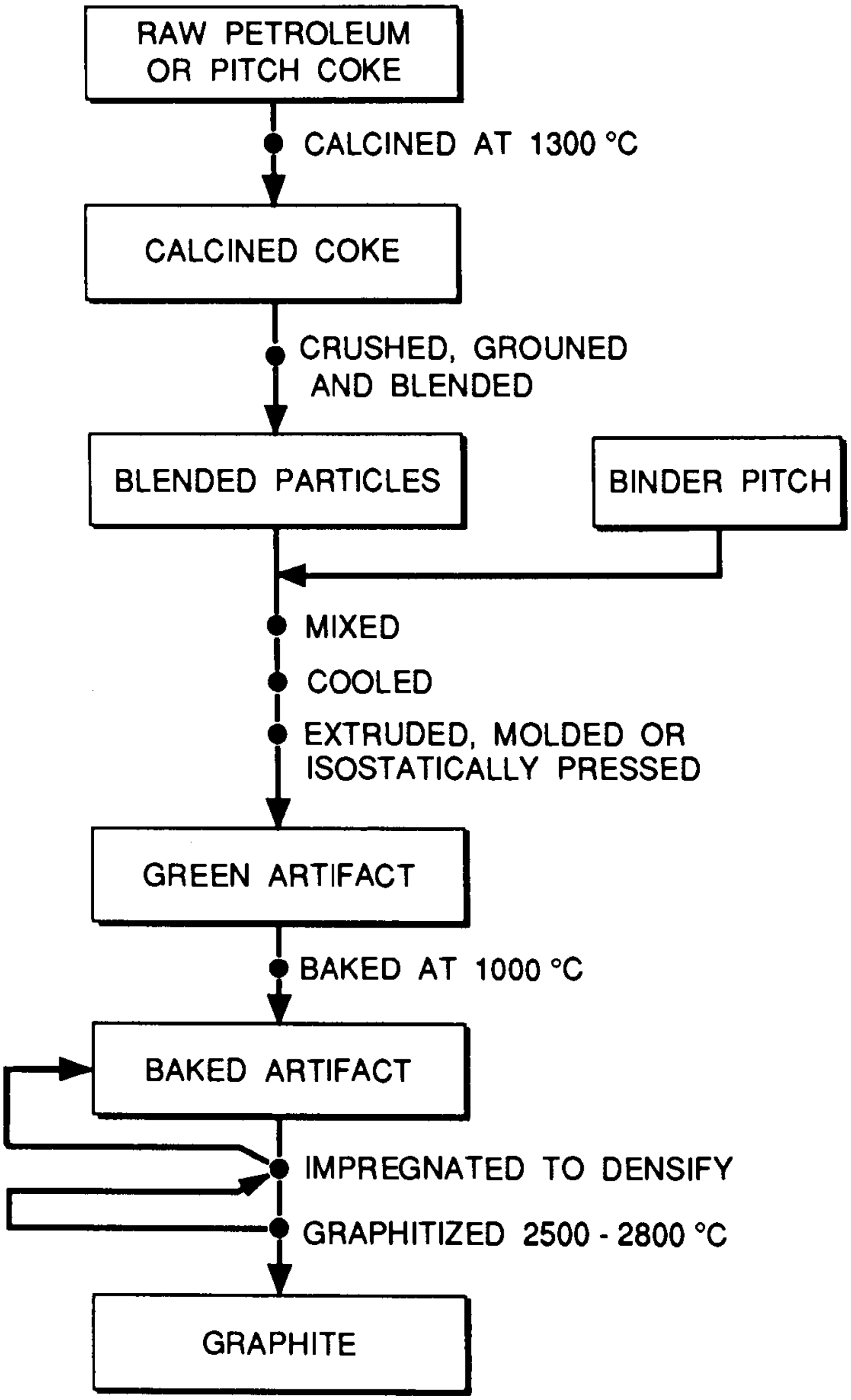
SYNTHETIC GRAPHITE

What is Graphite?

- An allotropic form of carbon
- Hexagonal 2D crystal structure
- Layered crystal planes
- Highly anisotropic
- Conductor in plane, insulator across planes
- A fun material to work with!



THE MAJOR PROCESSING STAGES IN THE MANUFACTURE OF CONVENTIONAL ARTIFICIAL GRAPHITES



Graphite Possesses Unique Properties

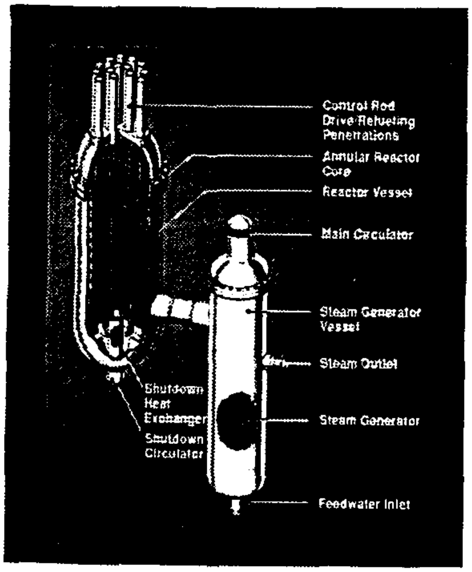
- High temperature stability
- High electrical and thermal conductivity
- Chemically inert
- Strength increases with temperature
- Processible and machinable to all forms and sizes

Applications of Graphite

- Arc furnace electrodes
- Resistance heating elements
- Crucibles and boats (semiconductor industry)
- Rocket motor throats, missile nose tips
- EDM electrodes
- Seals and bearings
- Electric motor brushes
- Metal refining and casting
- Nuclear moderator

The CMT Group Is Developing A Design Data Base For the Graphite Core Of The MHTGR

Modular High-Temperature Gas-Cooled Reactor With Steam Cycle Power Conversion



Graphite Data Base Development

- Effects of reactor environment on graphite properties
  - Neutron irradiation
  - He coolant chemistry and oxidation
  - Temperature and pressure
- Strength, strain to failure
  - Elastic constants
  - Fatigue behavior
  - Fracture mechanics data
  - Thermal physical properties
  - Oxidation behavior and mechanisms
- C/C composite control rod development

ACTIVATED CARBON FIBERS

Activated Carbons are a Broad Family of Carbon Materials

- Coal-derived carbons
- Almond shells
- Olive pits, peach pits
- Charcoal
- AC from waste paper
- Activated carbon fibers

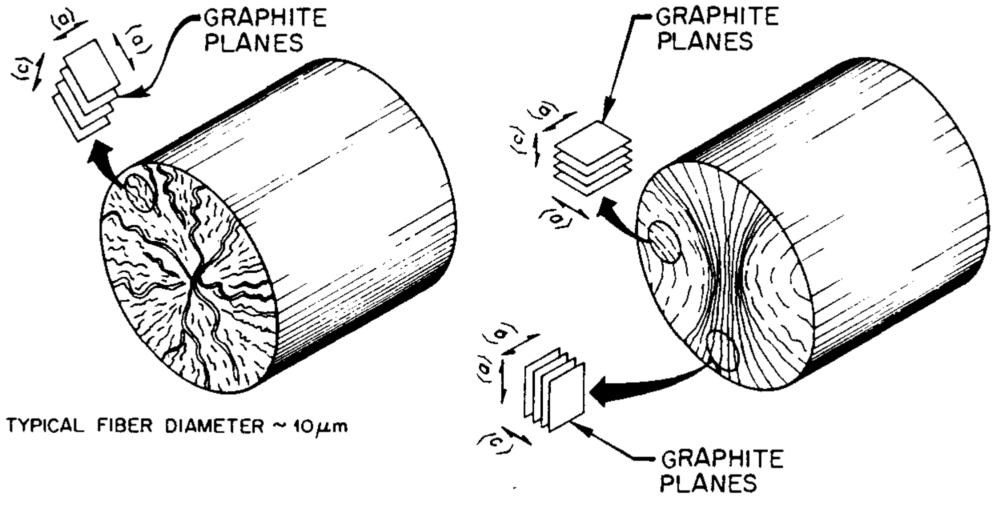
Applications of Activated Carbons

- Gas separation
- Air filtration
- Water filtration
- Solvent recovery
- Military suiting
- Molecular sieves

Activated Carbon Fibers (ACF)

- ACF can be derived from pitch (coal tar or petroleum), polyacrylonitrile (PAN), or rayon precursors
- Fibers are loosely consolidated into a felt, mat or woven cloth, or rigid porous monolith
- Product is thermally activated at temperatures in the range 700-1000°C in steam

TYPICAL CARBON FIBER STRUCTURES



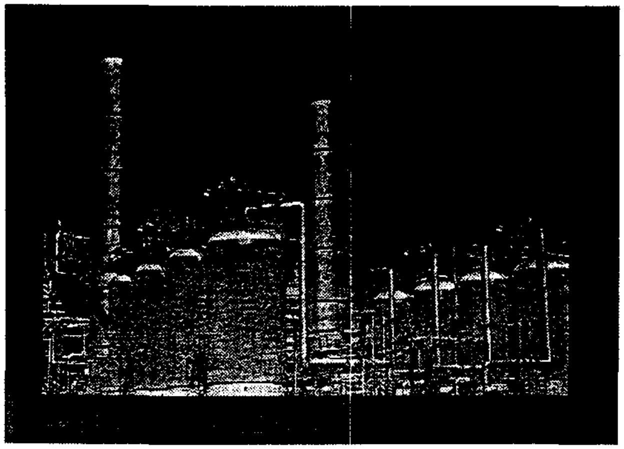
RADIAL HOURGLASS

G.M. Pennock et al.
CARBON, Vol.31,
p.596,597 1993

Carbon Fiber Composite Molecular Sieve

At ORNL we have developed a rigid, monolithic-activated carbon material named a carbon fiber composite molecular sieve. The material retains the advantages of ACF and, additionally, overcomes problems associated with granular activated carbons.

Hydrogen Separation Is Widely Achieved Using Pressure Swing Adsorption Systems



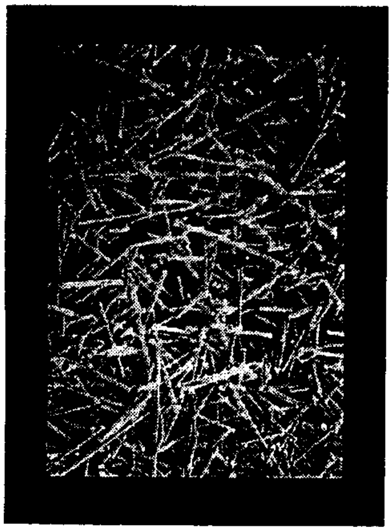
UOP Polybed PSA Unit treats gas streams from reformer hydrogen plant to produce 66 million SCFD of hydrogen.

Gas Separation Is Widely Achieved Using Pressure Swing Adsorption Systems

"Although there is some room for improvements in the pressure swing adsorption process, the potential gains in process economics in the future are likely to come from the development of new and improved adsorbents."

– R. V. Jasra, N. V. Choudary, and S. G. T. Bhat
Research Center, Indian Petrochemicals Corp.
In their review "Separation of Gases by Pressure Swing Adsorption", Separation Science and Technology, Vol. 26, pp. 885-930, 1991.

CMT Group Has Developed A Novel Adsorbent/Molecular Sieve Based On Carbon Fibers



Carbon Fiber Composite Molecular Sieve

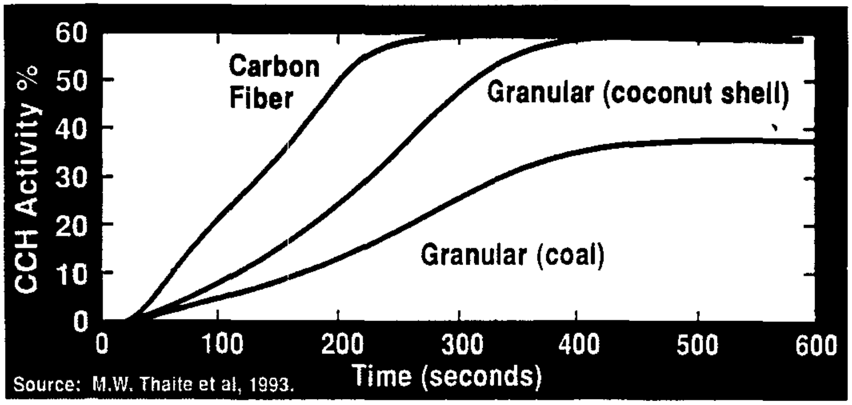
- **High adsorption/desorption rates**
- **High active surface area**
- **Microporous [5-10 Å] fibers**

Applications: Gas separation, gas storage, air filtration, pressure swing adsorption systems

Activated Carbon Fibers Adsorb And Desorb More Rapidly Than Granular Activated Carbon

- CCl_4 adsorption rate is twice that of granular carbon
- >10 times higher adsorption/desorption rates reported for tolulene and methane

Carbon Tetrachloride Adsorption Rates



Source: M.W. Thaite et al, 1993.

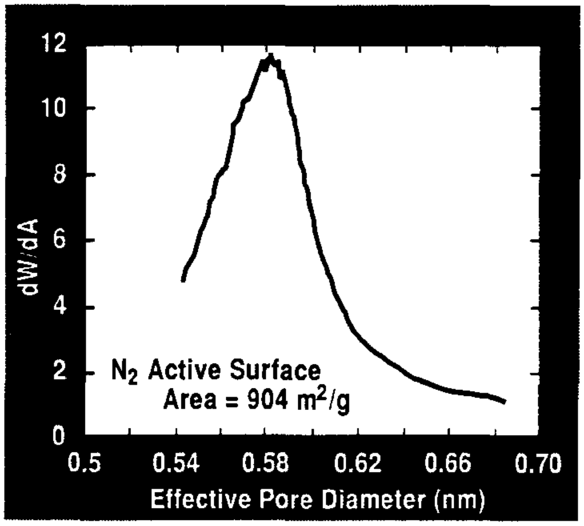
ORNL Activated Carbon Fiber Composites Have Very High Active Surface Areas

N_2 BET Surface Areas Of Materials That Have Well-known Molecular Sieve Properties

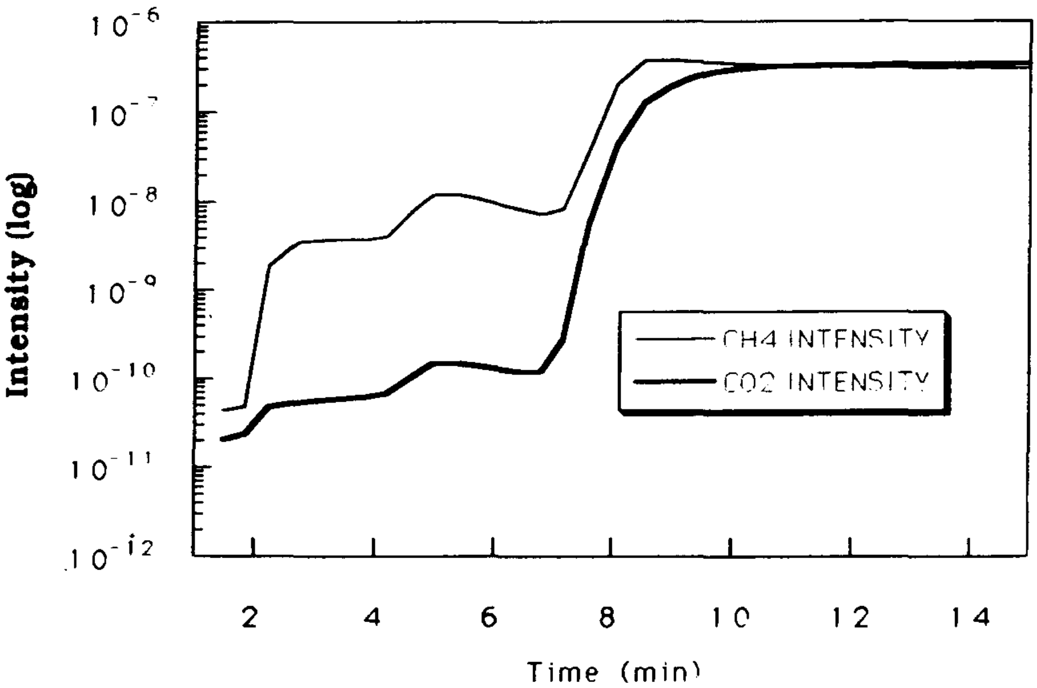
Molecular Sieve	$N_2(m^2/g)$
Linde 5 Å zeolite	316
Linde 4 Å zeolite	35
Linde 3 Å zeolite	11.6
Carbosieve	1040
Saranchar	900
Pitch fiber CFCMS (non-optimal condition)	>1,650

- Active surface areas up to 1670 m^2/g have been achieved with ORNL CFCs
- Carbon fiber active surface areas > 2000 m^2/g and 10 Å pore width have been reported (OSAKA Gas)

ORNL Carbon Fiber Composites Have High Active Surface Area And Are Very Microporous


ORNL - UK2 CFCMS Material

- **Narrow pore size distribution**
- **Pore size centered at ~ 6 Å pore width**
- **High active surface area (904 m^2/g)**
- **Active surface areas >2000 m^2/g possible**


SEPARATION OF CO_2 & CH_4 By CFCMS (STP)
(Initial composition 50vol% CO_2 and CH_4)

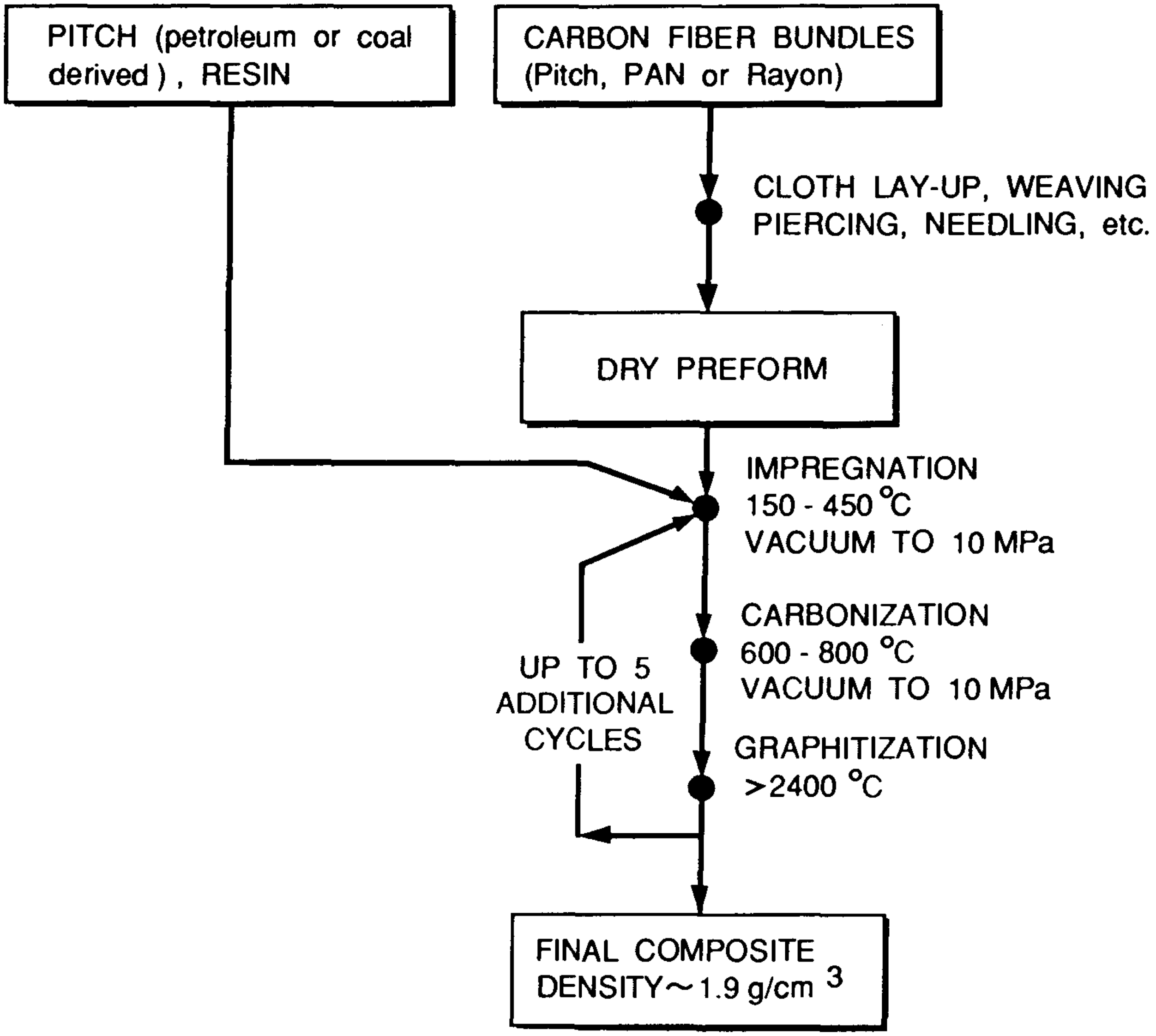
Carbon: CFCMS-48; BET=822m^{2}g^{-1}, L=0.74 nm

Summary And Conclusions

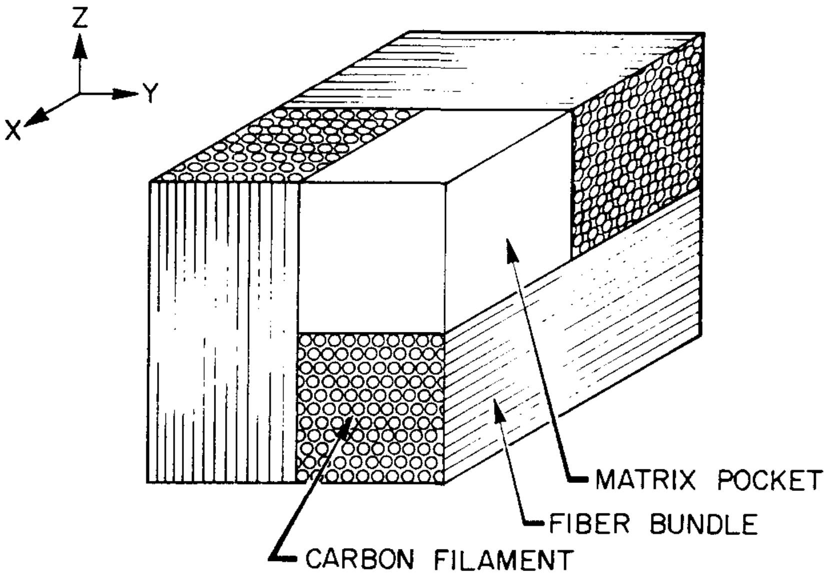
- **ORNL/UKCAER have developed a monolithic adsorbent carbon based on carbon fibers**
- **Gross macroporosity and microporosity can be controlled through processing variables**
- **Activated carbon fibers have adsorption rates greater than twice that of conventional activated carbons**
- Very high active surface areas can be developed in carbon fibers (OSAKA gas report >2000 m^2/g (N_2) ASA, 10 Å pore width)
- Narrow micropore size distributions can be achieved in the range 5-10 Å
- A monolithic activated carbon material has significant advantages over granular activated materials for use in pressure swing adsorption equipment, such as the elimination of channelling, by-pass flows and pressure drops

CARBON-CARBON COMPOSITES

FLOW DIAGRAM FOR A TYPICAL CARBON / CARBON COMPOSITE PRODUCTION PROCESS



THE STRUCTURE OF AN ORTHOGONAL CARBON - CARBON COMPOSITE SHOWING THE THREE MUTUALLY PERPENDICULAR FIBER REINFORCEMENT DIRECTIONS (x, y, and z)



Applications of Carbon-Carbon Composites

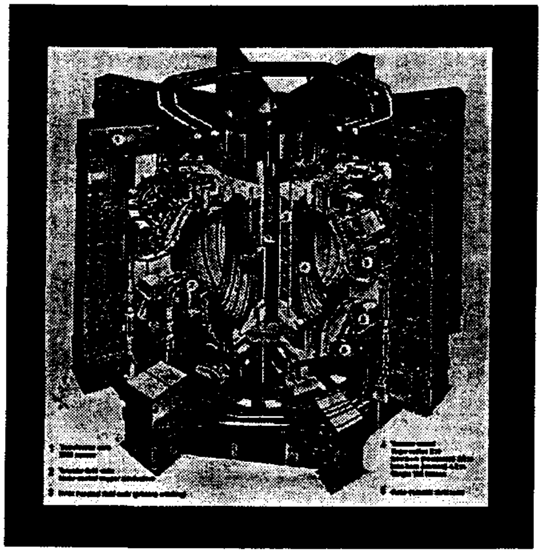
- Reentry vehicle nose tips
- Rocket motor throats and nozzles
- Exit cones
- Aircraft brakes
- Thermal protection
- Thermal management systems
- Fusion reactor first wall armor

Carbon-Carbon Composites for Fusion Energy Devices

ORNL has an active program to develop high-thermal conductivity, carbon-carbon composites for plasma facing components in tokamak fusion reactors. Moreover, we are investigating the effects of neutron damage on the structure and properties of carbon-carbon composites.

CMT Group Is Leading The Development Of Neutron Irradiation Tolerant C/C Composite Materials For Fusion Energy First Wall Applications

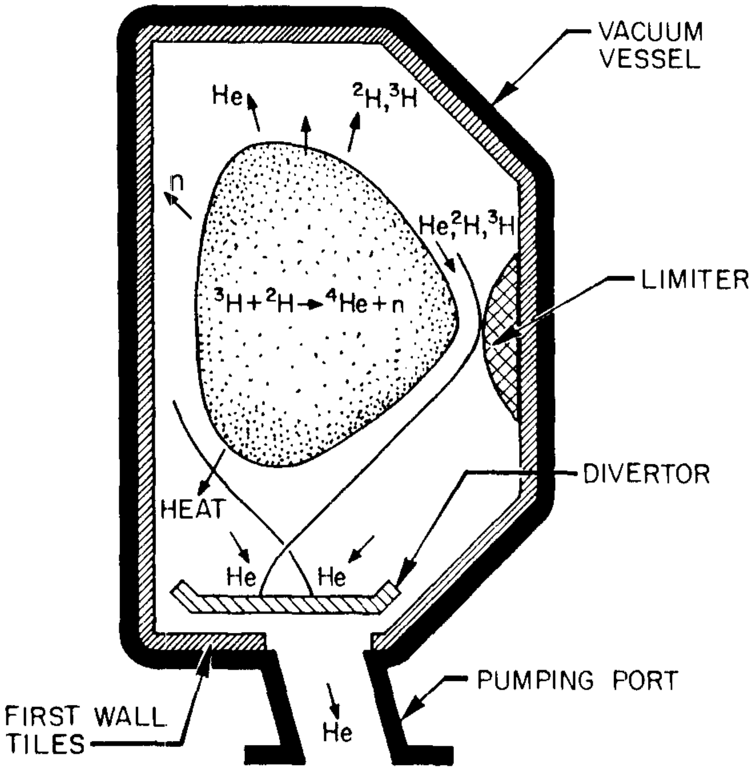
Joint European Torus



First Wall C/C Composites

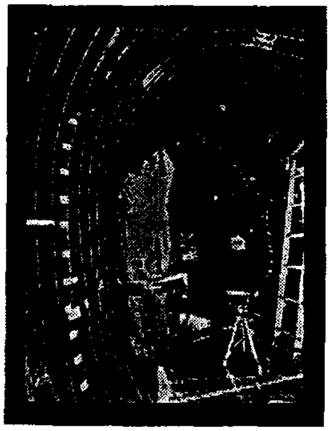
- **Neutron damage**
  - **Dimensional stability**
  - **Effects on thermal conductivity**
- **Development of high thermal conductivity uni-directional C/C composite**

THE MAJOR PLASMA FACING COMPONENTS OF A TOKOMAK FUSION DEVICE

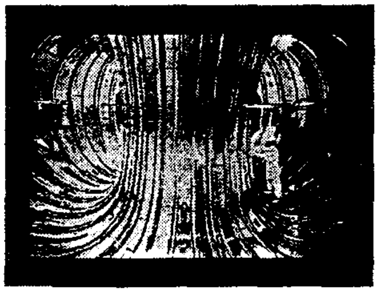


Carbon Materials Are Widely Used For Tokomak First Wall Armor

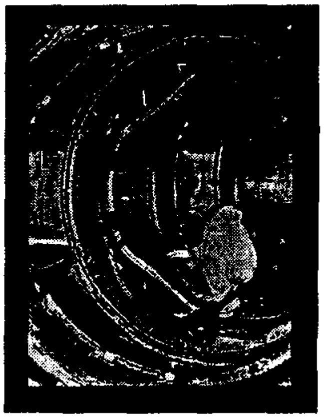
JT-60U (Japan)



JET (England)



TFTR (USA)

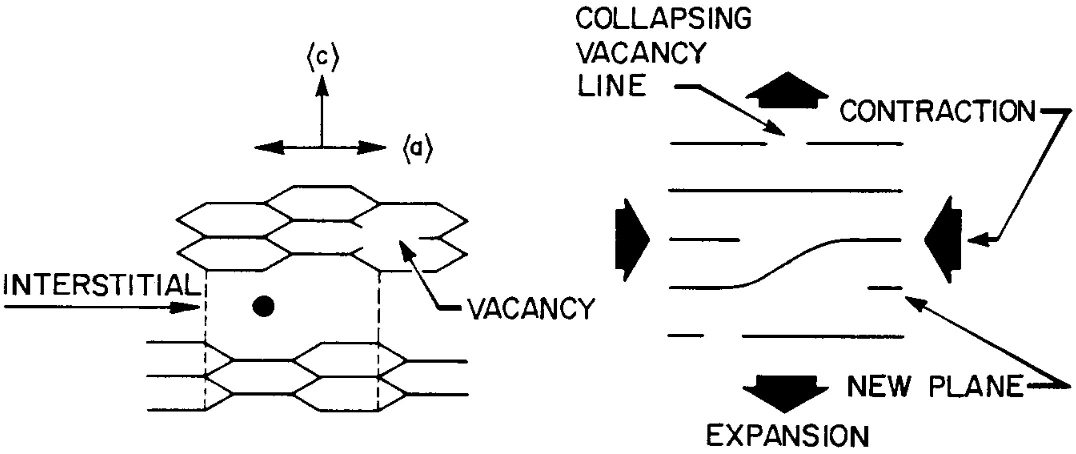


PLASMA FACING COMPONENTS

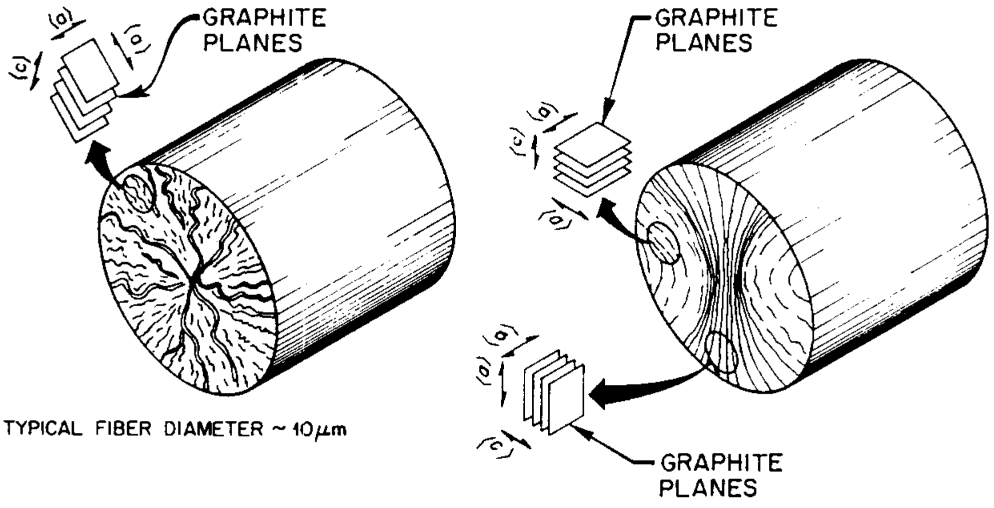
MATERIALS REQUIREMENTS

- LOW ATOMIC NUMBER
- HIGH THERMAL CONDUCTIVITY
- GOOD THERMAL SHOCK RESISTANCE
- EROSION RESISTANT (PLASMA SPUTTERING)
- ADEQUATE STRENGTH AND STIFFNESS TO WITHSTAND ELECTROMAGNETIC FORCES DURING DISRUPTIONS
- RESISTANT TO IRRADIATION DAMAGE

RADIATION DAMAGE IN GRAPHITE



TYPICAL CARBON FIBER STRUCTURES

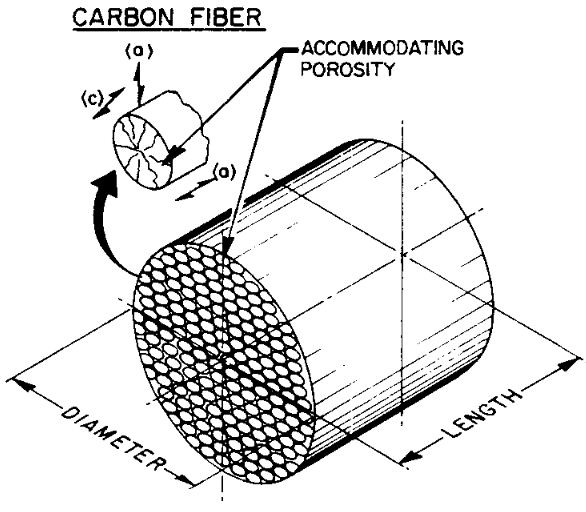


RADIAL

HOURGLASS

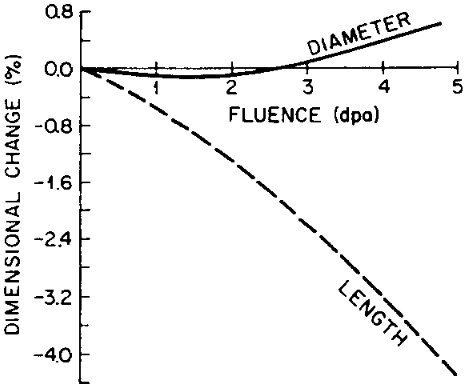
G.M. Pennock et al.
CARBON, Vol.31,
p.596,597 1993

MICROSTRUCTURAL INTERPRETATIONS OF IRRADIATION INDUCED DIMENSIONAL CHANGES IN 1D C/C COMPOSITES

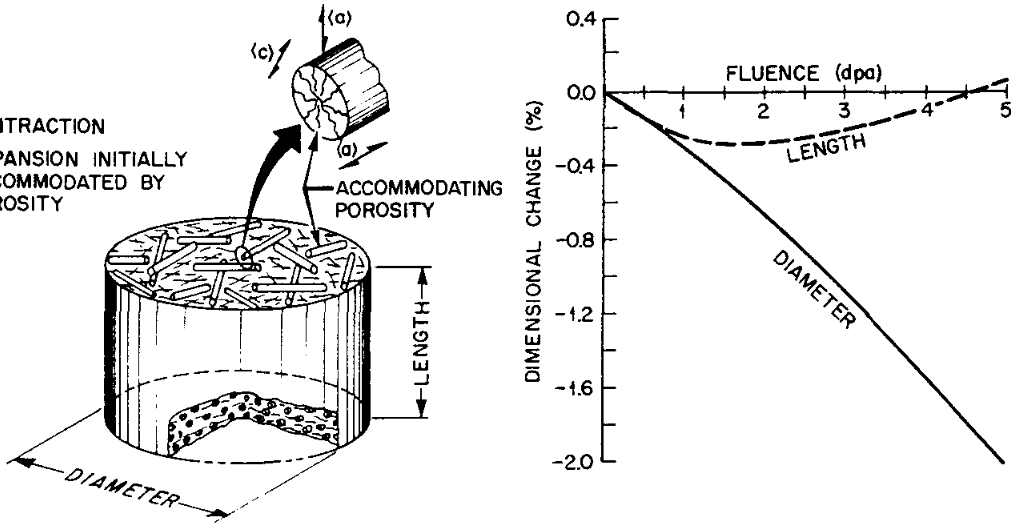


1D C/C COMPOSITE

(a) CONTRACTION
(c) EXPANSION INITIALLY
ACCOMMODATED BY POROSITY



MICROSTRUCTURAL INTERPRETATIONS OF IRRADIATION INDUCED DIMENSIONAL CHANGES IN 2D C/C COMPOSITES



Developments in Carbon Materials Recommended Texts for Carbon Materials

Introduction to Carbon Science

Harry Marsh
Published Butterworth & Co., Ltd., 1989
ISBN 0-408-03837-3

Carbon Fibers

J. B. Donnett and R. P. Bansal
2nd Edition, Published Marcel Dekker
New York, 1992

The Industrial Graphite Engineering Handbook

UCAR Carbon Company, 1991
(39 Old Ridgebury Road, Danbury, CT 06817-0001)

More Advanced Text:

Physics of Graphite

B. T. Kelly
Published Applied Science, 1981
ISBN 0-85334-960-6

Adsorption, Surface Area, and Porosity

S. J. Gregg and K. S. W. Sing
2nd Edition, Published Academic Press, 1982

LIQUIDS THAT TAKE ONLY MILLISECONDS TO TURN INTO SOLIDS

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Liquids That Take Only Milliseconds to Turn into Solids

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Greenville, North Carolina 27858

KEY WORDS:

Electrorheology, electrorheological fluid, fluid power transmission, hydraulics.

PREREQUISITE KNOWLEDGE:

The student should understand the elementary concepts of magnetism. A basic understanding of fluid flow would be helpful.

OBJECTIVE:

To observe the unusual characteristics of electrorheological fluids. To understand how these fluids change from a liquid to a solid. To understand the potential power transmission advantages of these fluids.

EQUIPMENT AND SUPPLIES:

1) Plastic bottle containing electrorheological fluid.
2) Energizing probe
3) Personal safety equipment
4) Paper towels
5) Pen

INTRODUCTION:

Electrorheological fluids change their flow characteristics when subject to an electrical field. Response, which takes only milliseconds, is in the form of a progressive gelling that is proportional to field strength. With no field present, the fluid flows as freely as hydraulic oil (Korane, 1991).

Electrorheological fluids represent a maturing technology that has the potential to widen the performance range of automated electromechanical and electrohydraulic equipment. Research and ongoing developments are refining this technology and experts predict an important future for these fluids.

Importance of Electrorheological Fluids:

Current automation capabilities are not advanced enough to build a robot that could play tennis. Even though cameras and computers could direct the robot towards a ball, robots move in an awkward, lumbering fashion because conventional hydraulic valves cannot keep pace with the commands of the computerized controllers.

With electrorheological fluid technology, this type of response time is possible. This technology will allow devices that can operate instantly and without mechanical valves. Increased productivity and better product quality through more dependable and responsive automated equipment is just a small part of what this maturing technology can deliver.

How Electrorheological Fluid Functions:

Electrorheological fluids are composed of two primary components. They are the carrier fluid and the suspended particles. The carrier fluid needs to be a good insulator, compatible with the materials they contact. Typical particle materials include polymers, minerals, and ceramics (Scott, 1984).

When an electrical field is applied across the fluid, positive and negative charges on the particles respond by separating, so each particle then has a positive end and a negative end. Particles of the electrorheological fluid then link together in the same manner that the north pole of one magnet is attracted to the south pole of another magnet (Duclos, 1988).

When the electrical field is removed, the particle attraction is no longer present. The fluid then begins flowing freely. Please see Figures 1 and 2.

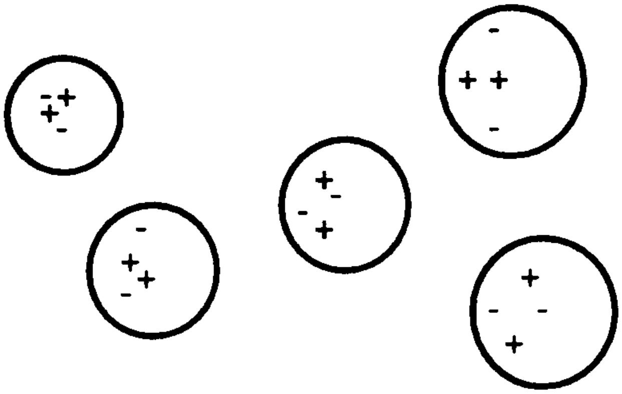

Figure 1 - No electrical field present, charges are random, no particle attraction.

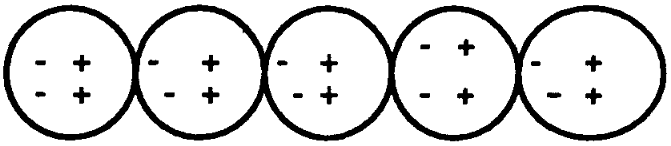

Figure 2 - Probe energizing the fluid, particles linked together.

Potential Applications:

Electrorheological fluids can change from solids to liquids so fast they will work well with fast-acting computers. These characteristics suggest a number of unusual engineering applications such as fluid clutches and vibration isolators (Duclos, 1988).

According to Hans Conrad, professor of materials science and engineering at North Carolina State University, electrorheological fluids will lead to a whole new generation of brakes, automatic transmissions, actuator devices, hydraulic valves, pump parts, and motors (Conrad, 1992).

PROCEDURE:

Safety Considerations:

1. Be very careful to avoid being shocked by the energizing probe.
2. Protective eye wear is mandatory for all those in the lab area.
3. Do not allow the electrorheological fluid to touch anyone's skin or clothing.
4. Obtain a "Material Data Safety Sheet" on the fluid from the supplier. Read the sheet completely and ask questions relating to any information you do not understand.

Electrorheological Fluid Preparation:

1. Check the cap on the plastic bottle containing the fluid and make sure it is securely tightened.
2. Shake the bottle to distribute the stratified particles evenly within the fluid.
3. Set the bottle down on the lab bench and carefully remove the top.

Observing Electrorheological Fluid as a Liquid:

1. Slowly insert the probe into the container of fluid until the metal rods are submerged approximately two inches below the surface of the fluid.

2. Slowly withdraw the probe from the fluid and hold it slightly above the bottle. Observe what happens to the electrorheological fluid on the metal rods of the probe.

3. Set the probe on a paper towel and record you observations.

Observing Electrorheological Fluid as a Gelled Solid:

1. Slowly insert the probe into the container of fluid until the metal rods are submerged approximately two inches below the surface of the fluid.

2. Press and hold down the button on the top of the probe.

3. Slowly withdraw the probe from the fluid and hold it slightly above the bottle. Observe the status of the electrorheological fluid on the metal rods of the probe.

4. With the probe slightly above the fluid level in the bottle, release the button (de-energizing the probe) and observe what happens to the electrorheological fluid on the metal rods of the probe.

5. Set the probe down on a paper towel and record your observations.

SAMPLE DATE SHEET:

Record below the characteristics of the fluid when the probe was not energized:

Record below the characteristics of the fluid when the probe was energized:

INSTRUCTOR NOTES:

1. When the probe was removed from the fluid without being energized, students should have observed the fluid flowing off the metal rods and back into bottle. The fluid will flow as easily as oil.
2. When the probe was removed from the fluid while being energized, students should have observed a gelling of the fluid around the rods of the probe.
3. De-energizing the probe will cause the gelled electrorheological fluid to liquify and drip into the bottle.

REFERENCES:

Korane, K.: Putting ER Fluids To Work. Machine Design, May 9,1991, pp. 52-57.

Scott, D.: Amazing Hardening Fluids Open a New World of Hydraulic Drives. Popular Science, April 1984, pp. 42-46.

Duclos, T.G.: Electrorheological Fluids and Devices. Automotive Engineering, December 1988, pp.45-48.

Conrad, H.: The Impact of ER Fluids. Compressed Air Magazine, March 1992, pp. 14-17.

MECHANICAL PROPERTIES OF CROSSLINKED POLYMER COATINGS

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MECHANICAL PROPERTIES OF CROSSLINKED POLYMER COATINGS

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Keywords: polymer, crosslinking, hardness, impact resistance

Prerequisite knowledge: Some familiarity with polymers and polymer structures would be beneficial, although not required. Also, students familiar with basic mechanical properties and their measurement for metals, especially hardness and fracture toughness, will be able to apply that knowledge to the material presented here.

Objectives

- Fabricate and test thin films to explore relations between a polymer's structure and its mechanical properties.
- Expose students to testing methods for hardness and impact energy that are simple to perform and which have results that are easy to comprehend.
- Show importance of polymer properties in materials that students frequently encounter.
- Illustrate a system which displays a tradeoff between strength and impact resistance, the combination of which would need to be optimized for a particular application.
- Expose students to coatings technology and testing.

Equipment and Supplies

- 2-mm diameter pencil leads and lead holders, to cover a range of hardnesses. The available pencil hardness range is 6B (softest) to 9H. To test the materials described here, pencils from 2B to 5H (2B, B, HB, F, H, 2H, 3H, 4H, 5H) should be sufficient.
- Steel test panels—7.5 x 15 cm, 0.8 mm thick, with dull matte finish (from Q-Panel Co., Cleveland Ohio).
- Drawdown bar (film applicator)—see Appendix.
- Micrometer.
- Drop weight impact tester—see Appendix .
- Oven(s), capable of temperature control in the range 70-190 ^{o}C.
- Large glass plate, or other hard flat surface.
- Raw materials: see Procedure for specific examples and suppliers.
- Tukon® or other microhardness tester (optional).

INTRODUCTION

Organic coatings, the paints and finishes we use for protection and decoration, are polymeric materials—they consist of high molecular weight chain-like molecules, which contain many atoms linked together by covalent bonds. Although the characteristics of a particular paint or finish can be modified somewhat by additives, the basic chemical and molecular structure of the polymer (or polymers) from which a coating is made is primarily responsible for many of its important properties, such as hardness, durability, and chemical resistance.

Many common coatings are composed of polymers with a crosslinked molecular structure. After application as a liquid, these finishes do not merely 'dry' in the conventional sense. They form hard solid films by undergoing a chemical reaction, called crosslinking (or "curing"), after they have been applied to a surface.

Crosslinking is a process by which linear polymer chains ("primary chains") are joined together at common junction points. These junction points can be provided by low molecular weight compounds, which can chemically attach themselves to the ends of at least three different primary chains (see Figure 1). Such compounds are referred to as "crosslinking agents" because they provide the means of converting a collection of linear molecules into a crosslinked polymer. The structure of a crosslinked polymer can be depicted a net (Figure 1), and these materials are often referred to as network polymers.

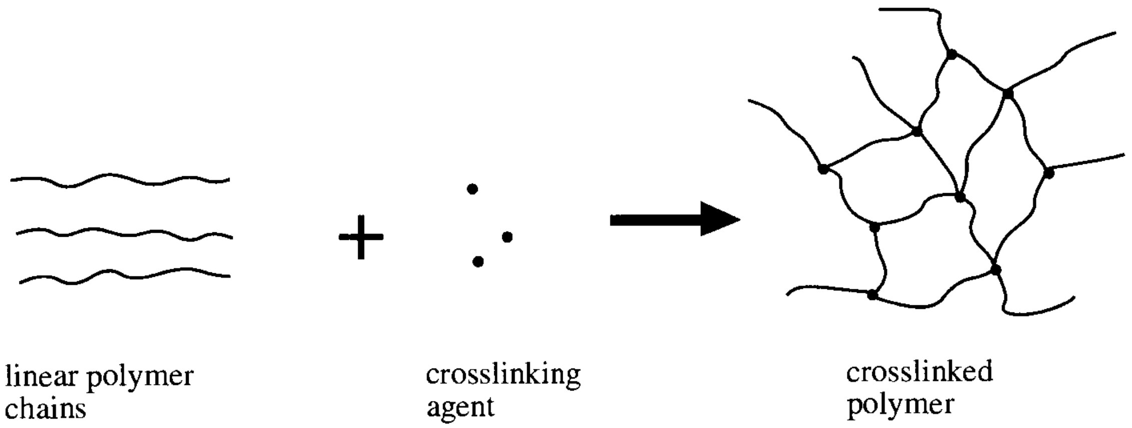
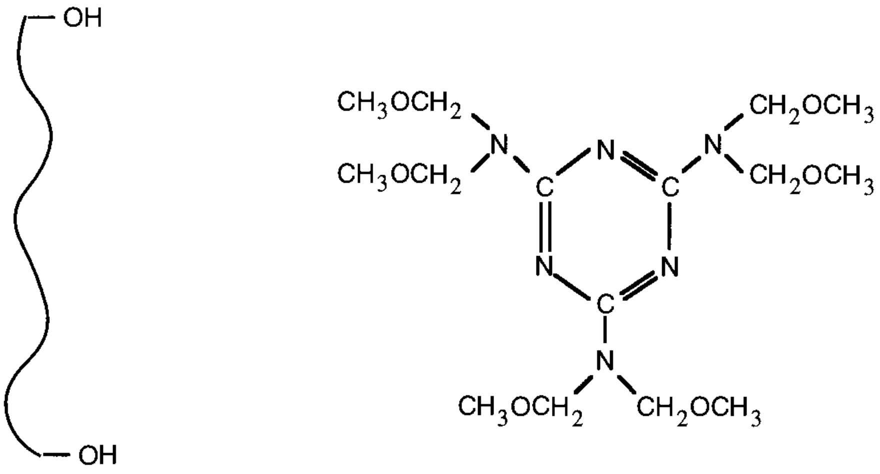


Figure 1. Formation of a Crosslinked Polymer

The properties of a network polymer depend strongly on its degree of crosslinking. To use the net analogy, a network with extensive crosslinking (many junction points, or many chains emanating from each one) would be classified as a "tight" network, while one with more limited crosslinking would be called a "loose" network. A higher degree of crosslinking in a polymer coating (tighter network) usually leads to increased hardness and solvent resistance, but may also result in increased brittleness. Other mechanical and chemical properties can be affected as well, and usually an optimum degree of crosslinking is sought which gives an attractive combination of properties for a particular application.

In this laboratory, you will prepare a series of crosslinked coatings, and will determine how the degree of crosslinking in these materials affects their hardness and impact resistance.

Like the schematic of Figure 1, you will fabricate crosslinked polymer networks by combining a primary resin with a crosslinking agent. (The word "resin" is used to describe a polymeric, or high molecular weight, compound). The primary resin consists of linear polymer chains which contain hydroxyl groups (-OH) at the chain ends (see Figure 2). Acrylic and polyester resins of this type are commonly used for automobile finishes and industrial coatings. The hydroxyl groups are the reactive sites at which these primary chains will attach to the crosslinking agent .



primary resin chain

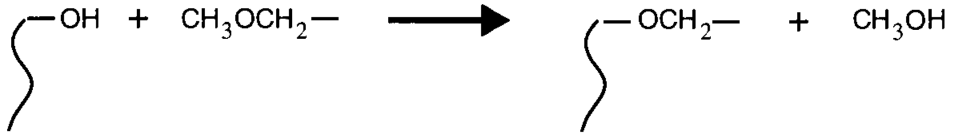
crosslinking agent
(hexamethoxymethyl melamine)

Figure 2. Components for Polymer Network

Several different types of compounds can serve as crosslinking agents for the primary chains. The one you will use is a substance called hexamethoxymethyl melamine (HMMM). Its chemical structure is shown in Figure 2.

The HMMM chemical structure may appear complex, but the important thing to note is that it contains six (CH_3OCH_2-) groups. These are the chemical groups that can react with the (-OH) groups of the primary resin chains. Such a reaction will result in a connection between the HMMM and the primary chain. Because it contains six (CH_3OCH_2-) groups, the HMMM can attach itself to as many as six different primary chains, and can therefore serve as junction points for the network (see Figure 1). It should be noted that, despite the relative sizes of the molecular drawings in Figure 2, the primary resin chains will typically be much larger than the crosslinking agent molecules.

Chemically, the crosslinking occurs by a condensation reaction which produces methanol (CH_3OH) as a by-product:



This curing reaction requires the presence of a small amount of acid catalyst. You will use *para*-toluene sulfonic acid (*p*-TSA).

Also, elevated temperatures are needed for the reaction to take place. After the components (primary resin, crosslinking agent, catalyst, and some solvent) are applied to a surface as a liquid film, the coated part will be heated in an oven for a specified time. The crosslinking can therefore be controlled and delayed until an appropriate time after application. You may have heard of automobile finishes being "baked" in this fashion.

The degree of crosslinking, and therefore the final properties, of the cured films can be controlled by a number of factors. Two that you will explore are the curing temperature, and the ratio of crosslinking agent to primary resin in the liquid mixture. The degree of crosslinking will generally increase as the cure temperature is increased, because this speeds up the reaction. Also, adding more crosslinking agent to the mixture will, to a point, result in more extensive crosslinking.

Two important coating properties you will measure for these cured films are hardness and impact resistance.

Although there are a number of methods available to measure the hardness of polymeric materials, one of the most fundamental involves attempting to scratch the material with objects of differing hardnesses. This is in fact what you will do in this laboratory, using a series of pencil leads to evaluate the hardness of your coating. This type of hardness measurement is particularly appropriate for these materials, because coatings in service may have to resist scratching or gouging by hard objects.

You will expose the cured materials to impact by dropping a weight onto coated steel panels. Although other impact tests (Charpy, etc.) are available which can provide controlled rapid deformation of bulk specimens, subjecting the polymers to impact as thin films on substrates better simulates service conditions for these materials, and does not require machining of special sample geometries.

PROCEDURE

Note to Instructor: Many commercially available products can serve as components for the coating mixtures. The procedure as described here, as well as the results shown in the next section, are for a specific experiment which uses the following raw materials (different materials may require slight modifications, as specified by the supplier):

- primary resin—hydroxyl-functional polyester, #57-5803, Cargill, Inc., Minneapolis, MN.
- crosslinking agent (HMMM)—Resimene® 747, Monsanto Co., St. Louis, MO.
- acid catalyst (*p*-TSA)—K-Cure® 1040, King Industries, Norwalk, CT.

1. **CAUTION** This step should be conducted in the fume hood!!

   Prepare three different coatings formulations in small jars. The ratio (by weight) of primary resin to crosslinking agent should be varied as follows:

   Formulation A- 85:15
   Formulation B- 70:30
   Formulation C- 55:45

   These ratios should be based on the "solids" portion of the components. That is, if the components as supplied contain an added solvent, the solvent fraction is not included in the ratios.

   Add solvent (xylene, methyl isoamyl ketone, or other as recommended by resin supplier) to the formulations such that each contains 30% solvent overall. Remember to include in this 30% any solvent that may have been originally contained with the components. At 1% by weight, add to each mixture the K-Cure® acid catalyst product. Mix each formulation thoroughly with a mixing stick or spatula.

   Note to Instructor: In the interest of time, formulations can be completely or partially mixed for the students in advance, although determining appropriate amounts for each component can provide a good exercise, especially if the compounds originally contain a solvent fraction. When mixed in advance, the formulations may have a limited shelf-life (several weeks). Also to save time, a shorter experiment can be conducted which examines only one formulation instead of three.

2. **CAUTION** This step should be conducted in the fume hood!!

   Prepare samples by applying thin films of the coating mixtures to steel panels. Because the measured mechanical properties of a film can be affected by its thickness, it is important that uniform, constant thickness films be prepared for all samples. For each sample preparation:

   Place a panel on the glass plate (it can be secured with a small piece of masking tape). Deposit an amount (approximately 1 teaspoon) of the coating mixture near the far end of the panel, in the center of its width. Place the drawdown bar with the 0.15 mm clearance (see Appendix) at the far end of the plate and, in a continuous motion, pull it toward you across the liquid so that it leaves behind a film. It may take a trial or two for practice, but you should soon be able to quickly prepare film samples that appear smooth and uniform. The bar should be cleaned with a quickly evaporating solvent such as acetone between each sample preparation. Make three replicate samples for each combination of formulation type (A, B, or C) and curing temperature you intend to test (remember to label the panels with a marker!). Let each panel sit horizontally for at least ten minutes before curing (step 3) to allow for solvent evaporation.

3. For each test temperature, cure the samples by placing them (3 for each formulation) into the appropriate preheated oven. Panels can be placed horizontally or hung vertically in the oven. Curing temperatures in the range 70 - 190 ^{o}C should be investigated—see instructor for specific temperatures and ovens to be used. For each cure temperature, use a baking time of 30 minutes.

   Note to Instructor: If time and the number of available ovens make it difficult to examine several different cure temperatures, try instead using a single temperature and varying the amount of time samples remain in the oven. This will also produce films with varying degrees of crosslinking.

4. After the panels are removed from the oven and have cooled, lightly touch the films with your finger. Have they become hard and rigid, or are they still sticky and tacky? Using the micrometer, measure the thickness of the cured films. You can do this by obtaining

the difference in thickness between coated and uncoated portions of the panel. Check for uniformity between samples and within the same sample. Is the final film thickness the same as the clearance of the drawdown bar?

Note to Instructor: The thickness of the cured films will be considerably less than the drawdown bar clearance. The bar will leave behind a wet film that is typically about half as thick as the bar's clearance. This film will then contract upon solvent evaporation and curing. Using a bar with a 0.15 mm clearance depth, we obtained cured films that had thicknesses in the range 0.04 - 0.05 mm.

5. Measure the hardness of the cured films by comparing their hardnesses to those of the pencils. Pencil leads are ranked in order of <u>increasing hardness</u> as follows:

   6B, 5B, 4B, 3B, 2B, B, HB, F, H, 2H, 3H, 4H, 5H, 6H, 7H, 8H, 9H

   In a controlled fashion (see below), you will try to cut into each coating with these pencils. Leads harder than the coating will be able to gouge the film, while those softer than the coating will crumble and will leave the film uncut. For each trial:

   Adjust the lead so that approximately 3 mm extends from the lead holder. With the lead holder perpendicular to a piece of abrasive paper, prepare the lead by grinding it against the paper so that a flat surface, normal to the lead's length axis, is obtained. Place the test panel horizontally on a flat surface. With the pencil pointing away from you, place the lead against the film at a 45^{o} angle, and push the lead down and away from you in an attempt to cut the film. An alternative method is to grip the pencil in your fist, and hold the lead against the surface such that it is pointing toward you (45^{o} to the surface), and then pull rather than push the lead. In either case, you must exert enough downward pressure so that either the paint film is gouged (preferably down to the steel panel surface), or the lead crumbles. Make at least three trials with each lead tested, and note whether that lead cuts the film or not. The hardness of the film is reported as that of the hardest lead which <u>does not</u> gouge the film.

   Note to Instructor: This procedure can be supplemented by having students read ASTM D3363-74: Film Hardness by Pencil Test.

6. **CAUTION** Be certain to wear safety glasses while using this apparatus!!

   Using the drop weight tester, investigate the films' resistance to impact. This will provide a measure of their brittleness—a lower impact resistance indicates a more brittle (less tough) material. For each test:

   Place the coated panel horizontally on the support ring with the film facing upward. Raise the weighted indenter to a specified height inside the drop tube, and allow it to fall onto the panel. The energy of the blow is given by the load (indenter weight) multiplied by the height from which it was dropped. Joules or inch-pounds are appropriate units for impact energy. Remove the panel, and examine the film in the impact area. A failure is indicated by cracks caused by the blow. A magnifying glass may be useful, although failures are usually detected easily by eye. Note the energy of each impact, and whether a failure occurred.

   You are trying to determine the highest impact energy each film can sustain without failure. Perform tests from different heights and try to determine the impact energy at which the film exhibits a transition from passing to failing (i.e., if it passes the first test, you would increase the drop height). You can perform several tests on the same panel as long as a flat, coated area can be placed over the entire support ring. Beginning with large variations, you should be able to locate the transition point to within a drop height of 25 cm. If possible, try to narrow it down to within a 10 cm height, and perform multiple tests from several heights in this region. Report the "impact energy" of the sample as the highest energy at which the sample consistently <u>does not</u> fail.

   NOTE: For all samples, use the 0.9 kg indenter initially. If no failure is achieved even from the greatest possible height, switch to the 1.8 kg indenter.

7. (optional) Measure the hardness of the test films using a Knoop indenter on the Tukon® Microhardness Tester. Use an applied load of 50 g. Report hardnesses as Knoop Hardness Numbers (KHN).

EXPERIMENTAL RESULTS

All samples baked at 90 °C and above became hard to the touch after removal from the oven. Samples exposed to a lower curing temperature (70 °C) remained sticky and fluidlike. It can be concluded that little crosslinking occurred at this low temperature, and these films were not subject to the hardness and impact tests.

Figure 3 shows the pencil hardness for all films cured at 90 °C and above. The 55:45 and 70:30 mixtures produced films whose hardness increased from HB to 3H as the curing temperature was raised from 90 to 130 °C. The increase in crosslink density caused by curing at higher temperatures resulted in greater film hardnesses. For these two formulations, the pencil test did not detect any further changes in hardness at cure temperatures above 130 °C.

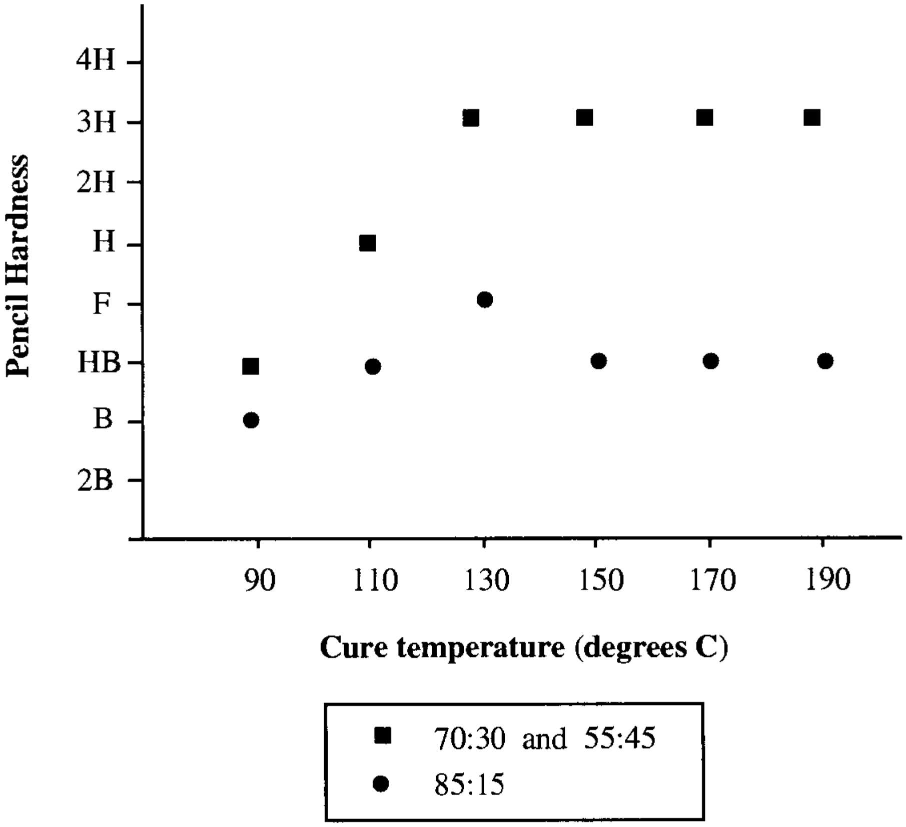


Figure 3. Pencil Hardness Values for Cured Films

Although the pencil hardness results for the 55:45 and 70:30 formulations were indistinguishable, films made from the 85:15 mixture (the one containing the least amount of crosslinking agent) exhibited significantly lower hardnesses at all curing temperatures. While also showing some increase in hardness with temperature, this formulation achieved a maximum hardness of only F. The behavior of the 85:15 samples demonstrates that reducing the amount of crosslinking agent in the mixture can result in significantly softer films.

It is interesting to compare pencil hardness results with Knoop Hardness Numbers (KHN) for these films. KHN values for the series of films from the 70:30 mixture appear in Figure 4. Indentations were measured at least thirty seconds after the Knoop indenter was removed; no changes in the measured indentation were noted at increased times (up to 10 minutes). The general trend of hardness increasing with cure temperature, seen in the pencil hardness results of Figure 3, is confirmed. Because the KHN analysis provides a less discretized measure of hardness, the curve in Figure 4 shows a somewhat smoother increase and leveling of hardness compared to the pencil test. Also note that the Knoop test identifies significant changes in hardness above the 130 °C cure temperature that were not detected in the pencil test. (*Note to Instructor:* Have students comment on the relative merits and drawbacks of the two types of hardness tests.)

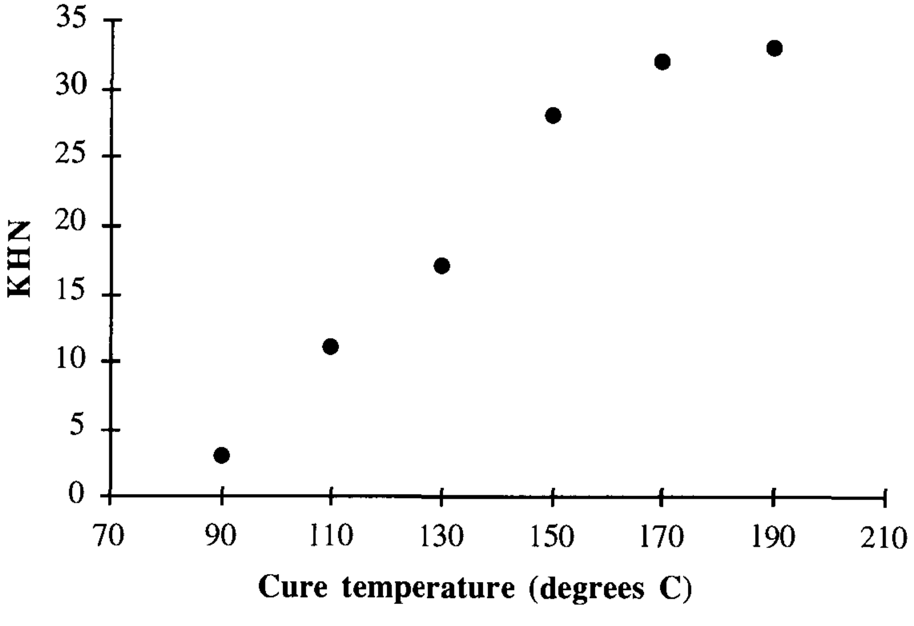


Figure 4. Knoop Hardness Numbers for Cured Films from the 70:30 Formulation

Figure 5 contains impact energy results from the drop weight test. Note that 20 Joules was the highest impact energy that could be achieved experimentally. Samples with a listed impact energy of 20 J did not exhibit any failure through the entire measurement range.

Films from all formulations showed considerable toughness and flexibility at the lowest cure temperatures, showing no failure even under an impact of 20 J. For films made from the 55:45 mixture (the highest crosslinking agent percentage) detectable failures appeared at a cure temperature of 130 °C (impact energy of 16 J). Further increases in the cure temperature for this formulation resulted in increased brittleness—impact energies fell to a final value of 3 J for the 190 °C cure. Similar behavior is seen for films from the 70:30 mixture. The changes these materials exhibit as a function of cure temperature indicate a strong inverse relationship between impact energy and degree of crosslinking.

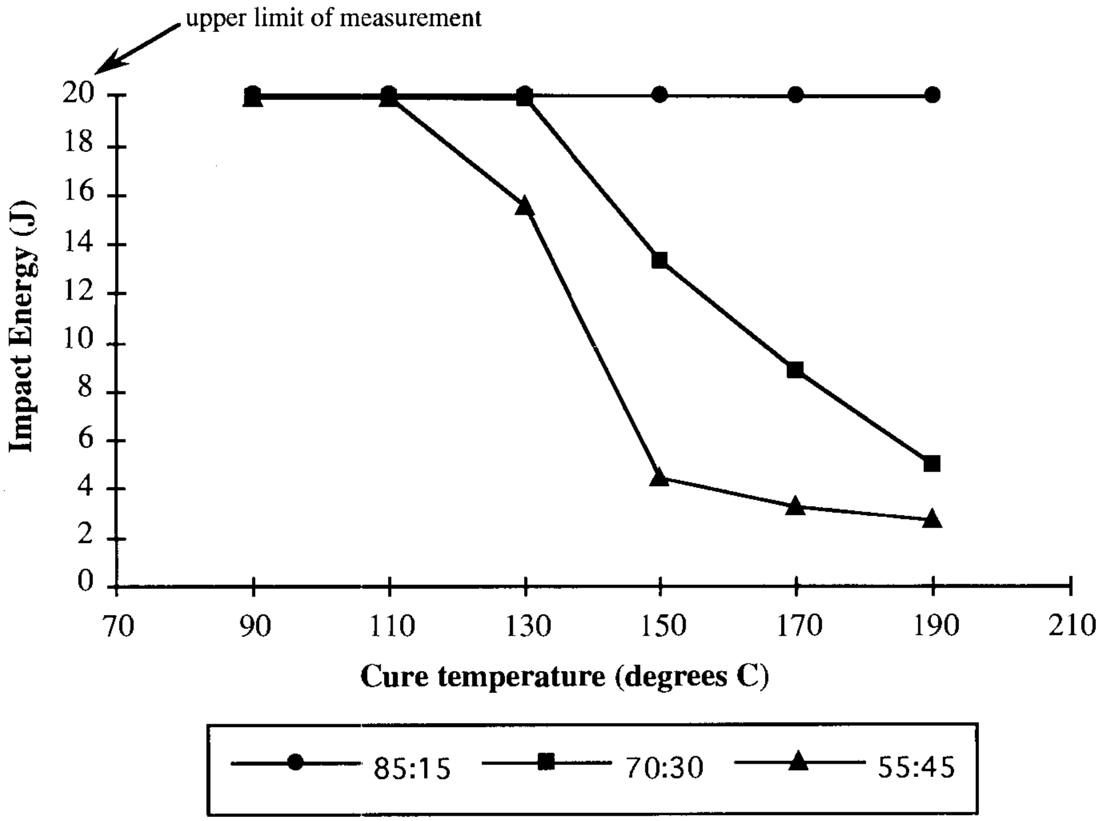


Figure 5. Impact Energies for Cured Films (a value of 20 J indicates no observable failure throughout the entire measurement range)

For cure temperatures where some samples exhibited detectable impact failures, the effect of crosslinking agent ratio on film toughness is apparent. The formulation with the largest percentage of crosslinking agent, 55:45, produced the most brittle films. The 70:30 samples had somewhat higher impact energies, and the 85:15 mixture produced the least brittle films, which did not show impact failures even when cured at 190 °C. This supports the earlier conclusion that more extensive crosslinking in these materials leads to a reduction in impact resistance. Even though samples from the 85:15 mixture are relatively tough, recall that they also exhibited considerably lower hardnesses than those from the other two formulations.

In summary, the hardness and impact energy of these organic coatings are directly related to their internal network polymer structure, in particular, the degree of crosslinking, which can be adjusted by changing the crosslinking agent percentage in the formulation, as well as the film's cure temperature. These materials display a tradeoff between hardness and toughness, with harder films generally exhibiting a lower resistance to impact. For a given application, changes in the formulation and cure temperature can be used to prepare coatings with an optimum combination of these mechanical properties.

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Appendix—Equipment Notes to Instructors

The *drawdown bar* is a steel bar, 3 to 4 inches in length, with a flat side that rests squarely on the test panel. Along this side, a 2-inch long section has been machined away such that it provides a clearance, which will leave behind a film when the bar is drawn along the surface of a panel and over the deposited liquid mixture, as described in the Procedure. We used a bar with a clearance depth of 0.15 mm (6 mil). Draw down bars are available commercially from Paul N. Gardner Co., Inc., Pompano Beach, FL.

In the *drop weight impact test*, the test panel sits horizontally on a circular steel support ring. A 4-foot long vertical tube, with a slot cut along its length, is positioned directly above the center of the support ring. A cylindrical steel weight, tapered at the impact end to a hemispherical tip, fits inside the tube. A bolt protruding from the side of the indenter weight fits through the slot in the tube, and is used to lift the weight to the desired drop height. A unit conforming to ASTM guidelines can be made by referring to ASTM D-2794-90: Resistance of Organic Coatings to the Effects of Rapid Deformation, although alternate designs would be suitable for the purposes of this experiment. A commercial device of this type is available from Paul N. Gardner Co., Inc. (see above).

EXPERIMENTS IN DIAMOND FILM FABRICATION IN TABLE-TOP PLASMA APPARATUS

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EXPERIMENTS IN DIAMOND FILM FABRICATION IN TABLE-TOP PLASMA APPARATUS

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KEY WORDS: Diamonds, plasma, heterojunctions, photoconductors.

PREREQUISITE KNOWLEDGE: The student should be familiar with the basics of materials science and chemistry. Levels at which these experiments are performed are second semester junior and either semester senior year. The students are first given lectures on band structure in solids, film formation via chemical vapor deposition, semiconductor junction formation, and photoconductivity. Though a scanning electron microscope would be helpful, film morphology can be seen with reasonable optical microscopy. A video filmed by the instructor showing the plasma deposition onto silicon and sapphire gives the student an idea of what is expected.

OBJECTIVES: To illustrate the process of plasma assisted chemical vapor deposition and to show devices which can be made simply in the laboratory. These devices illustrate clearly the concepts of bandgap, junctions, and photoelectronic processes. Films and devices are measured electrically, optically, and thermally.

EQUIPMENT AND SUPPLIES:

(1) Silicon wafers, sapphire wafers, and microscope slides (one per student)
(2) U.S.P. ethyl alcohol
(3) Stoppered flask with tubing fittings
(4) Controlled air leak
(5) High voltage (>2000 volts) d.c. supply
(6) Microwave oven (500 watts), small (optional)
(7) Polycarbonate belljar (25 cm. diameter)
(8) Silver paint (DuPont 4817)
(9) Diode curve tracer
(10) Microscope (optical, 400x) or electron microscope

INTRODUCTION: Using simple chemicals and materials, such as ethyl alcohol, sapphire, and silicon wafers, students can heteroepitaxially deposit diamond films onto silicon and sapphire wafers in a direct current plasma and/or microwave assisted plasma. By placing 1 part in 10^6 of either boron trichloride or phosphorus trichloride into the ethyl alcohol, the diamond films can be doped p- or n-type respectively. Undoped films serve as good photoconductor films. Small polycarbonate belljars purchased from Cole-Parmer along with mini vacuum pumps serve as the reaction chamber, minimizing danger of accident. The systems may be placed into a small microwave oven (500 watts, 2.45 GHz). The reaction with partial pressures of oxygen combined with the ethyl alcohol yields diamond films whose properties are dependent on deposition variables heretofore unknown to the student (and sometimes to the professor).

The three laboratory sessions spent provide the student with experience in:

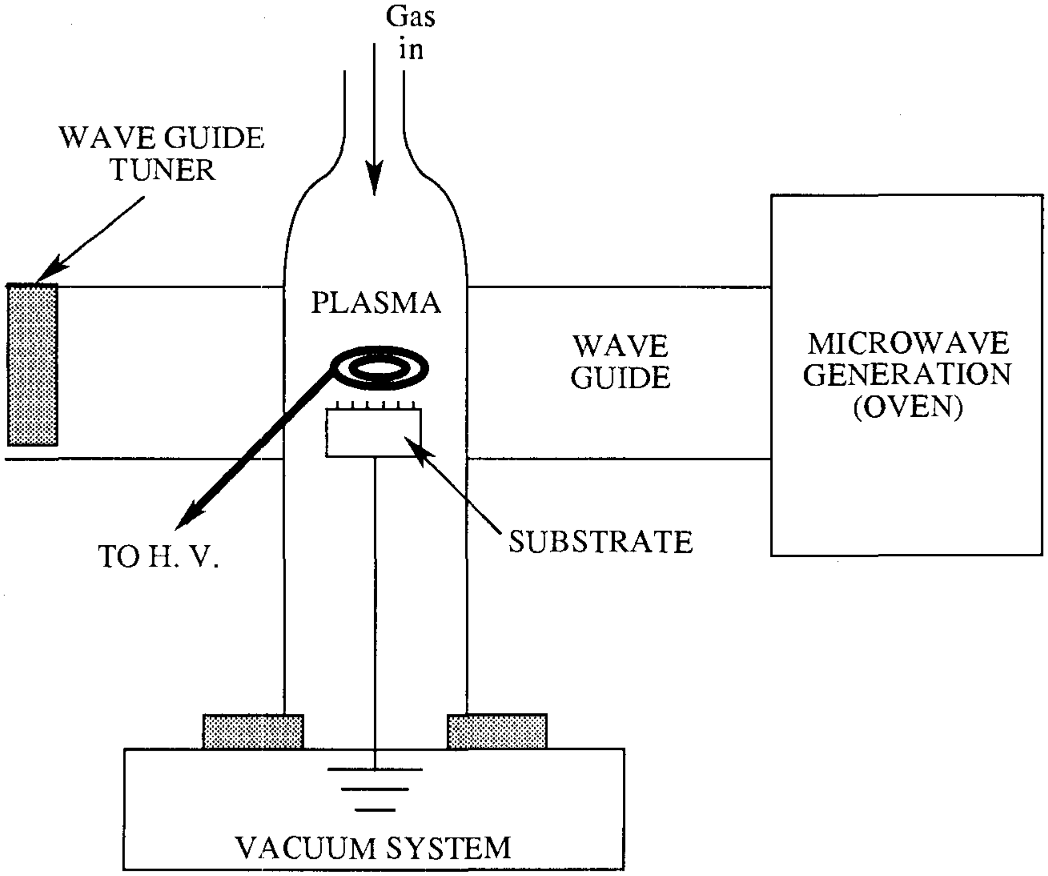
1. Chemical vapor deposition;
2. Plasma assisted reactions;
3. Heterojunctions and band structures;
4. Photoconductivity and spectrophotometric identification of films; and
5. Microscopy of films.

These experiments were developed by the professor and seniors in the process of the senior design project capstone course at Western New England College.

The Reaction: Although the literature is replete with carbon sources made up of a carbon gas source (such as methane in argon), little is said regarding safer methods of laboratory use for students. For the sake of safety, availability, and cost, alcohols were chosen as sources. A typical reaction for the ethyl alcohol used is shown below.

$$CH_3CH_2OH + O_2 \text{ -----> } 2C + 3H_2O$$

System Design/Procedure: The apparatus shown in Figure 1 was the functional system used, with a modification of high voltage plasma assist. This system is a modification of that described by R. C. DeVries. The microwave source was a conventional 2.45GHz, 500 watt Toshiba microwave oven. The chamber was a polycarbonate belljar, supplied by Cole-Parmer. The substrates were not heated from an external source, since, in the presence of the microwave oven ground, arcing occurred.

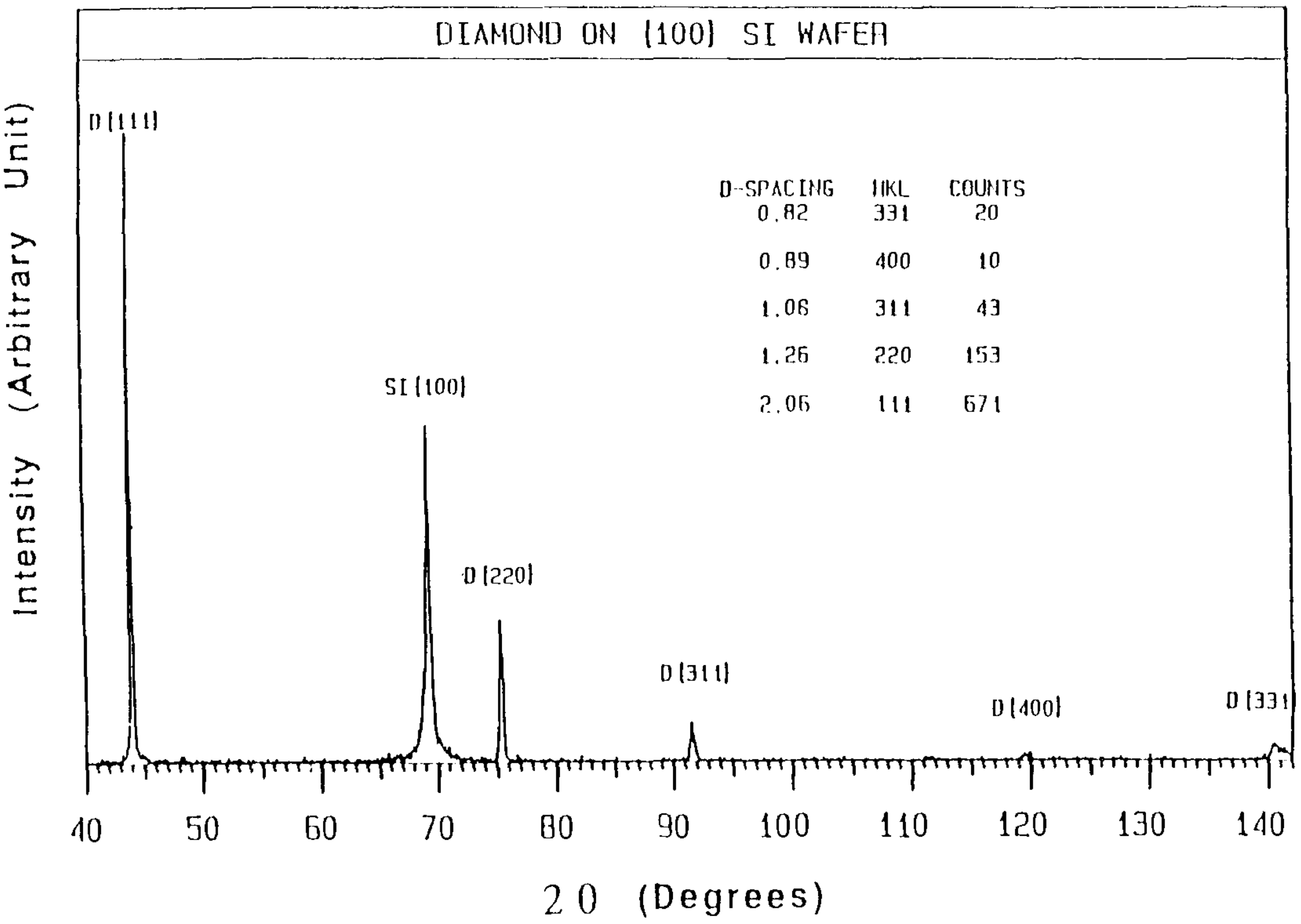


The vacuum pump is run until the system reaches 80 microns or lower pressure. The pump is allowed to run while a mixture of 1 part air and 1 part argon is bubbled through the ethyl alcohol and "leaked" into the vacuum system until the pressure rises to 3-30 Torr. The microwave power and/or the high voltage was turned on and the plasma (blue-red column) decomposition/oxidation of the ethyl alcohol proceeded. It should be noted that the morphology of the films obtained was quite different, dependent on the plasma source used. Though the process works better with the semiconductive substrates, with microwave assist the sapphire and glass substrates also provide suitable substrates. All films are polycrystalline with reasonable densities; best results and densities were obtained on single crystal silicon substrates. Typical yellowing or browning of the film indicates the presence of carbon (or graphite) included in the film. Each system appears to have somewhat different characteristics and the experimenter should note these. Typical growth rates of these films are of the order of 0.01 to 0.1 micrometer per minute. An x-ray diffraction scan of a film on (100) Silicon is shown in Figure 2. This film was grown at approximately 0.06 micrometers per minute.

Typical dopants for the diamond film are introduced via dissolution of boron trichloride (acceptor) and ammonium chloride (donor) into the ethyl alcohol in the 1-100 ppm ratio to carbon in the alcohol. Care should be taken to clean the system well between each type change. The number of ionized acceptors and donors is only expected to be from 1-10% in the diamond film at room temperature. Though time usually doesn't permit, all four varieties of n/p, p/n, n/n, and p/p heterojunctions prove to be interesting in demonstrating isotype and heterotype heterojunctions and their characteristics.

Safety Note: Although the alcohol is safer than methane in the laboratory, a safety mesh or Plexiglas sheet should be placed between the student and the apparatus. Good practice indicates the wearing of safety masks during the deposition.

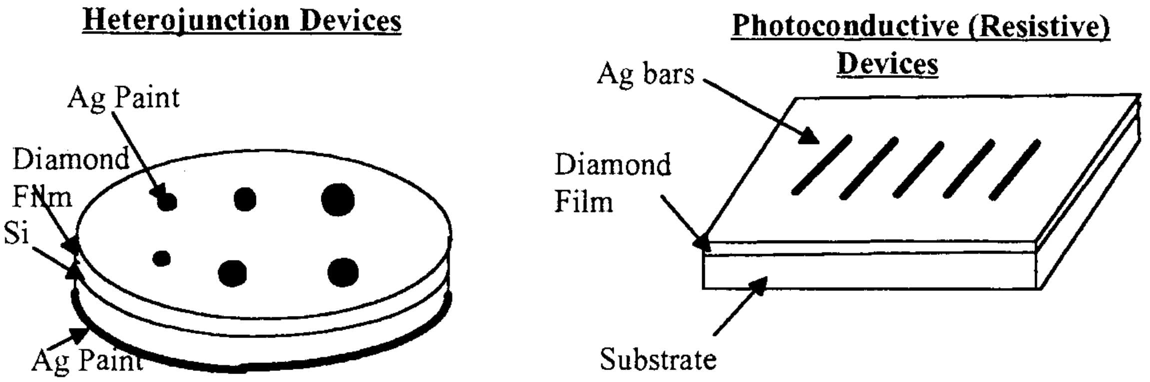
Device Preparation: Devices of the heterojunction type are post-deposition prepared by roughing the back side of the silicon wafer with 00 grit silicon carbide paper. Silver paint (DuPont #4817) is applied to the back of the silicon wafer and is allowed 15 minutes to dry. Dots of silver paint may be applied to the front of the wafer over the diamond. Fine aluminum wires may be attached for testing of the devices. Sufficient light enters the device to cause both photovoltaic and photoconductive (reversed biased) responses in the junctions. Devices of the non-junction photoconductive types on either sapphire or glass work well with silver paint electrodes applied juxtaposed in parallel bar formation.Both devices are shown in Figure 3. Carbon or gold semi-transparent electrodes function better for photo devices and can be easily deposited as replacements for the silver on diamond top electrodes. Attachment to these electrodes by means of fine aluminum wires serves to ease testing of the devices in the absence of probes.



D-SPACING	HKL	COUNTS
0.82	331	20
0.89	400	10
1.06	311	43
1.26	220	153
2.06	111	671

Figure 2
X-ray Diffraction of Diamond Film on Silicon (100)

Figure 3


Heterojunction Devices

Photoconductive (Resistive) Devices

Typical Measurements: Measurements made on devices will vary according to doping levels, type mixes, resistivities of the silicon, and conditions of deposition. Typical diode characteristics in the dark and light are shown in Figures 4 and 5. Typical photoconductor characteristics are shown for light and dark conditions in Figures 6 and 7. All characteristics are taken on a Tektronix curve tracer. The light sources used were tungsten-halogen "white" and 253.7 nm ultraviolet.

A typical spectrophotometric transmission curve vs wavelength for diamond is shown in Figure 8. The cutoff (bandgap) wavelength as well as impurity and other absorption energy levels are easily calculated (as shown in the appendix) from

$$E = hc/\lambda.$$

Spectral response characteristics can optionally be made with an added expenditure of time and effort.

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SOURCES OF SUPPLY: The microwave oven was an $88 one obtained from a local appliance store. The laboratory supplies and chemicals were obtained from Cole-Parmer and Fisher Scientific. The high voltage supply is a $35 surplus TV. 4000 volt d.c. supply. The silicon wafers and sapphire disks were obtained through Ms. Shannon John, Wafernet, San Jose, CA. The silver paint was obtained from Bert Bricker, Wilmington, DE. All other supplies are easily obtained from local university supply houses.

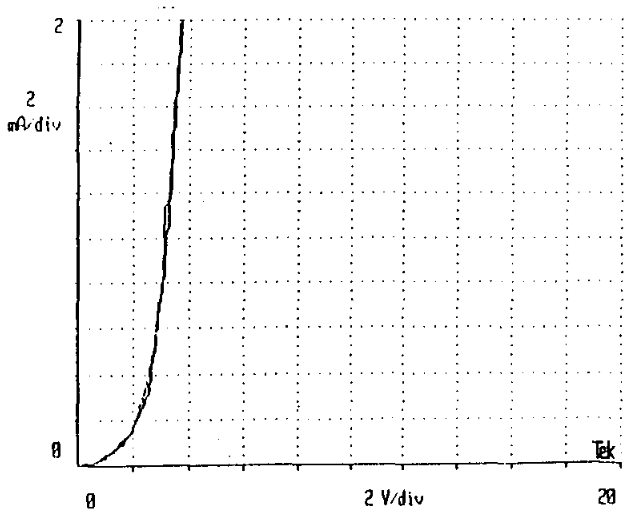


Figure 4

Forward Biased p/n Diamond/Si Heterojunction Diode

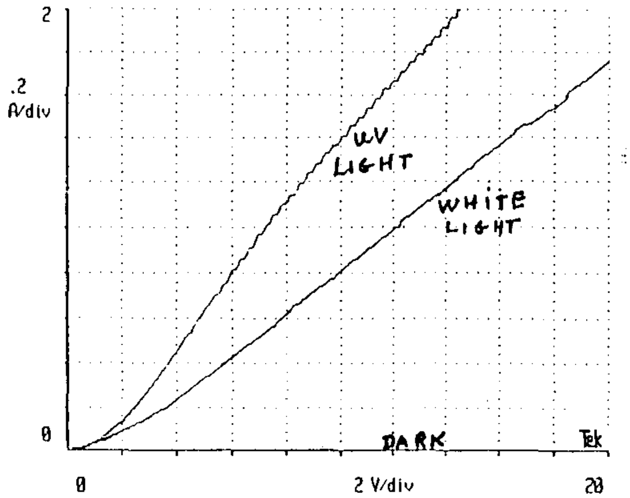


Figure 5

Reverse Biased p/n Diamond/Si Heterojunction Diode

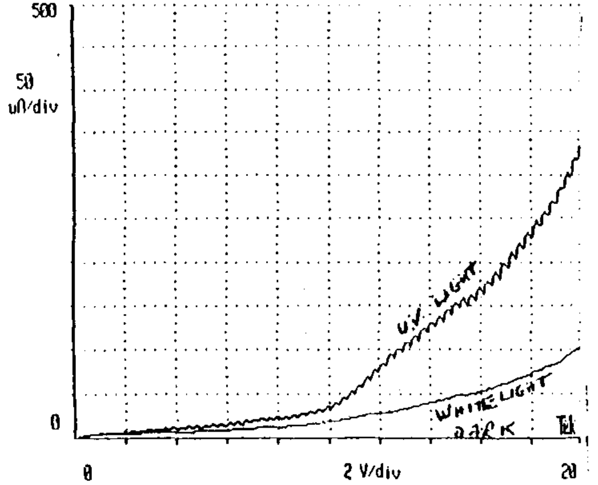


Figure 6

Low Sensitivity Diamond Photoconductor on Glass

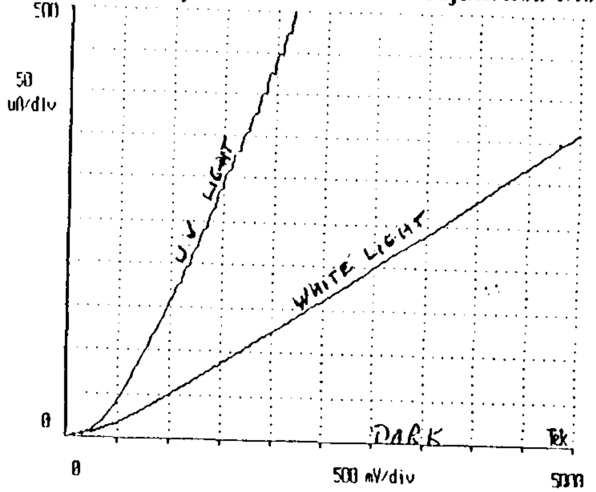


Figure 7

High Sensitivity Diamond Photoconductor on Sapphire

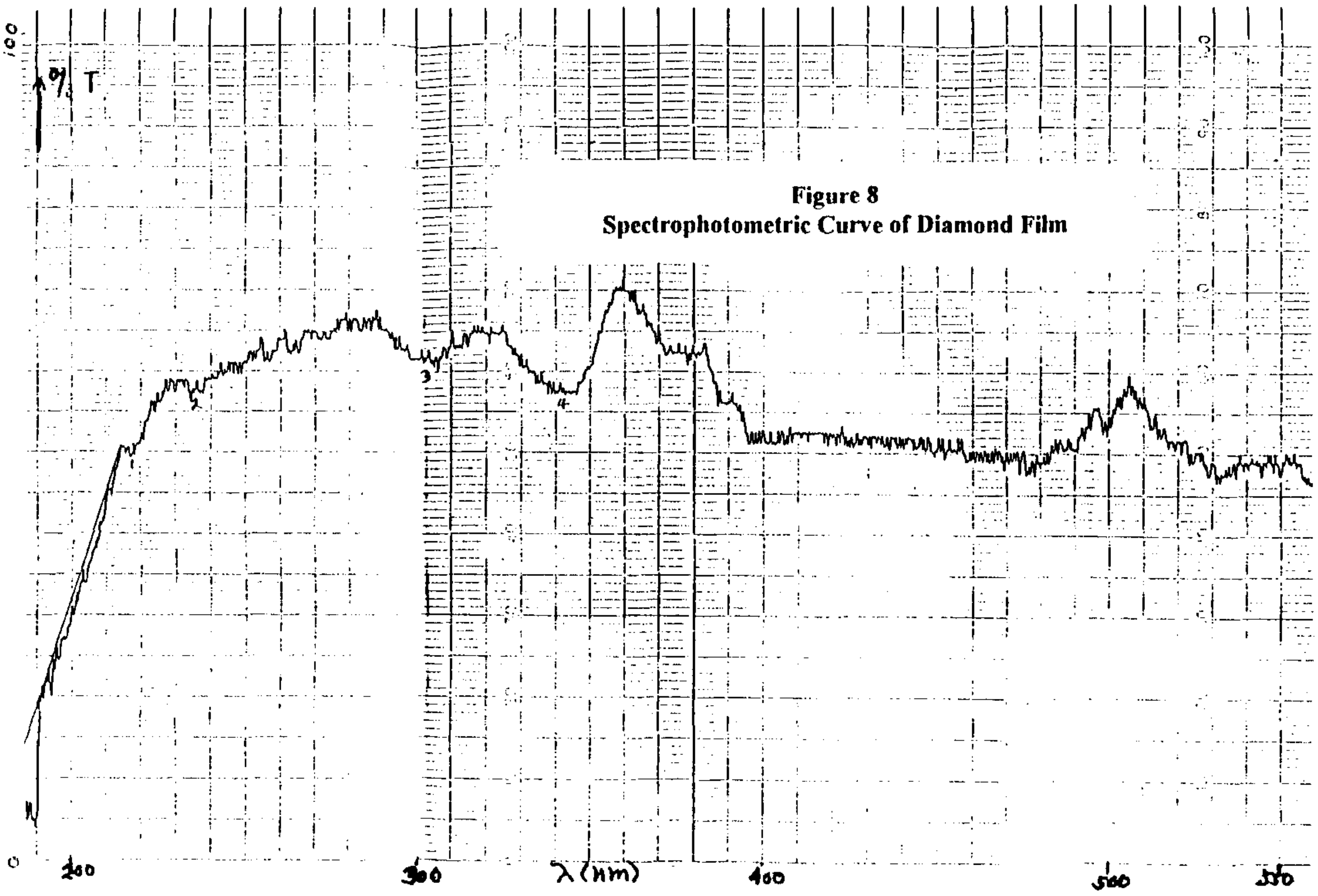
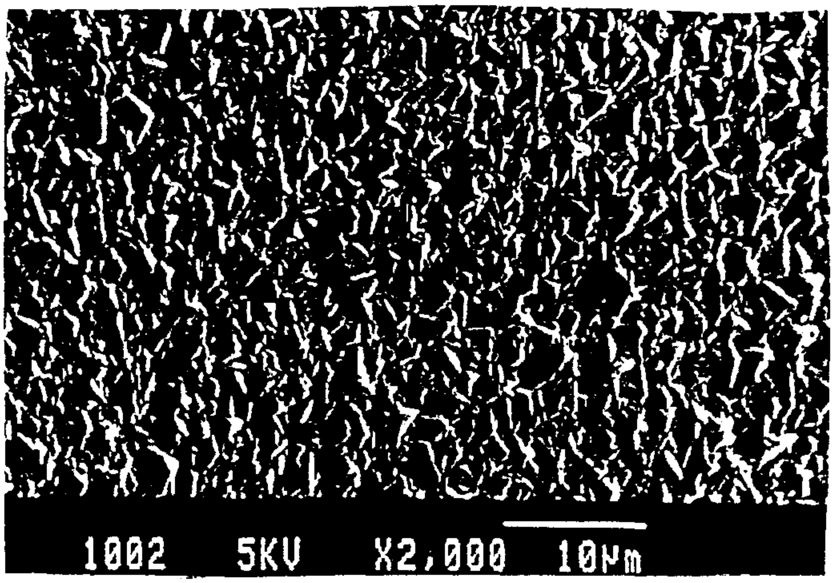


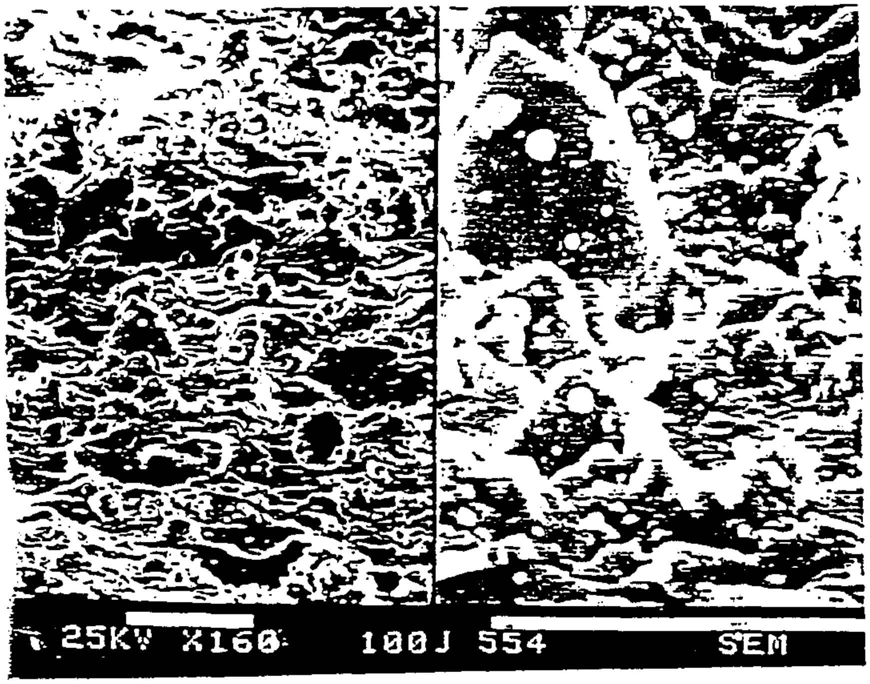
Figure 8
Spectrophotometric Curve of Diamond Film

Some original figures were unavailable at time of publication.

APPENDIX



Diamond Film #3



Diamond Film at 160x and 320x Magnification and 30° Angle

APPENDIX:

Heterojunction Theory: Heterojunctions are semiconductor junctions which are composed of two or more materials of differing band gaps in metallurgical contact with one another. The Anderson Model (in the reference by Milnes and Feucht) was the model chosen for diamond on silicon, n/n, p/p, p/n, and n/p structures. The energy band diagram for isolated and in-contact n/p (Si/diamond) is shown in Figures A1 and A2.

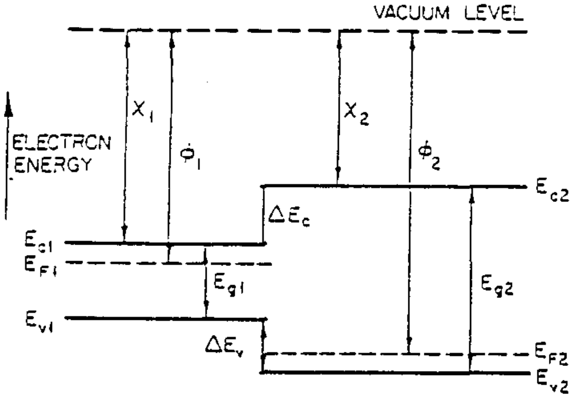


Figure A1
Energy Band Diagram for Two Isolated Semiconductors

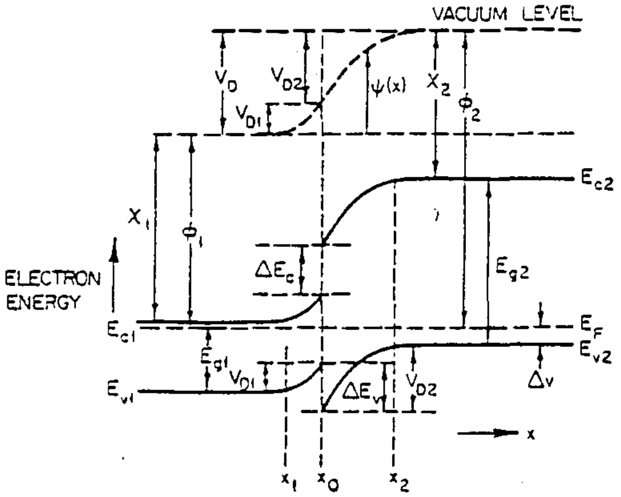


Figure A2
Energy Band Diagram for n-p Heterojunction (V = 0)

The energy barrier of a junction for non degenerate materials, materials whose electron affinity and bandgap are not affected by doping, can be calculated using the equation:

$$E_b = E_{g1} + \Delta E_V - \delta_n - \delta_p - qK_2V_a \qquad \text{Eq. 1-1}$$

Where:

E_{G1} = Bandgap of Material One
ΔE_V = Difference In the Energy of the Valance Bands
δ_n = Difference Between the Fermi Level and the Conduction Band
δ_p = Difference Between the Fermi Level and the Valence Band
q = Charge on an Electron (1.6×10^{-19}C)
V_a = Applied Voltage

The expression for K_2 is for low forward bias conditions and neglects the effects of injection carriers:

$$K_2 = 1 \Big/ \left(1 + N_{D2}\varepsilon_2 / N_{D1}\varepsilon_1\right) \qquad \text{Eq. 1-2}$$

Where:

N_{D2} = Number of Donor Atoms in Material Two
N_{D1} = Number of Donor Atoms in Material One
ε_1 = Permeativity of Material One
ε_2 = Permeativity of Material Two

The built-in voltage of the junction, V_D, is the sum of the partial built-in voltages, $V_{D1} + V_{D2}$, where V_{D1} and V_{D2} are the electrostatic potentials of the semiconductors 1 and 2 at equilibrium. The equation for the length of the transition or depletion region of an n-p junction can be found using the derivation:

$$(X_0 - X_1) = \left[\frac{2}{q}\frac{N_{A2}\varepsilon_1\varepsilon_2(V_D - V_a)}{N_{D1}(\varepsilon_1 N_{D1} + \varepsilon_2 N_{A2})}\right]^{1/2} \qquad \text{Eq. 1-3}$$

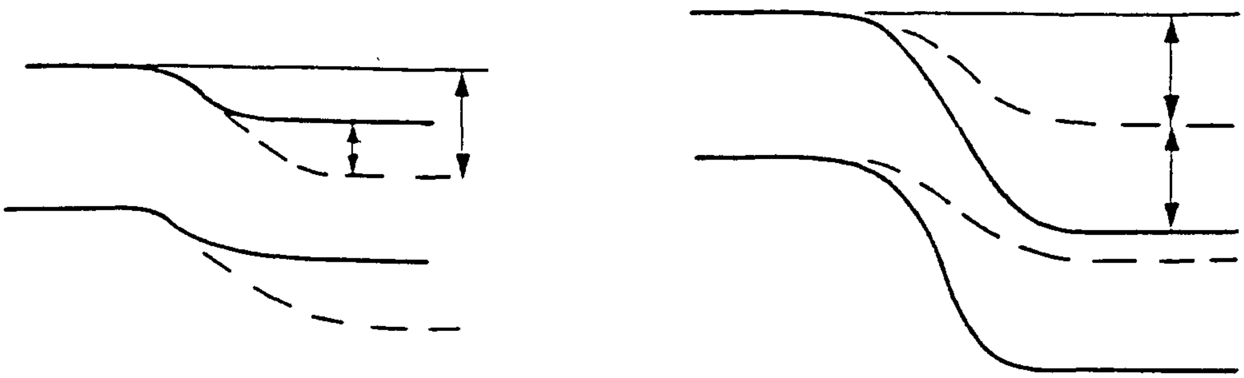
$$(X_2 - X_0) = \left[\frac{2}{q}\frac{N_{D1}\varepsilon_1\varepsilon_2(V_D - V_a)}{N_{A2}(\varepsilon_1 N_{D1} + \varepsilon_2 N_{A2})}\right]^{1/2} \qquad \text{Eq. 1-4}$$

The total width of the depletion region is therefore equal to:

$$W = (X_2 - X_0) + (X_0 - X_1) = \left[\frac{2\varepsilon_1\varepsilon_2(V_D - V_a)(N_{A2} + N_{D1})^2}{q(\varepsilon_1 N_{D1} + \varepsilon_2 N_{A2})N_{D1}N_{A2}}\right]^{1/2} \qquad \text{Eq. 1-5}$$

Where:

N_{A2} = Number of Acceptor Atoms in Material Two
V_D = Built-In Voltage



Energy Diagram of a p-n Junction for (a)Forward Bias (b)Reverse Bias

The above diagrams demonstrate the principle of rectification, which states that the total current flowing for a voltage V_a is not the same for a voltage of $-V_a$. The total current flowing left to right through the junction using the equation:

$$I = I_0[\exp(qV_a / \eta kT) - 1] \qquad \text{Eq. 1-6}$$

Where:

I_0 = Reverse Saturation Current
η = Quality Factor
k = Boltzman's Constant (1.38×10^{-23} J/K)
T = Temperature (K)

Dividing by the area of the junction the current density can be calculated.

$$J = J_0[\exp(qVa / \eta kT) - 1] \qquad \text{Eq. 1-7}$$

APPENDIX

COMPARISON BETWEEN Si, GaAs, β-SiC AND DIAMOND (300 k)

	Si	GaAs	β-SiC	Diamond
Bandgap (eV)	1.12 (indirect)	1.42 (direct)	2.35 (indirect)	5.48 (indirect)
Mobility ($cm^2 V^{-1} s^{-1}$) μe μn	 1500 450	 8500 400	 900 50	 1800 1600
Electron Sat Drift Vel' V_{sat} ($cm\ s^{-1}$	1.0×10^7	2×10^7	2.5×10^7	2.7×10^7
Breakdown Field E_B ($V\ cm^{-1}$)	3×10^5	4×10^5	4×10^6	1×10^7
Dielectric Constant ε	11.9	12.9	9.7	5.6
Thermal Conductivity (K) ($W\ cm^{-1} K^{-1}$)	1.5	0.5	5	20
Johnson Fig. of Merit[1] $M_J = \left(\frac{E_B V_{sat}}{2\pi}\right)^2$ $(V\ s^{-1})^2$	2.3×10^{23}	16.2×10^{23}	2533×10^{23}	18465×10^{23}
Ration Relative to Silicon	1	7.0	1101	8028
Keys Fig. of Merit[2] $M_K = K \left(\frac{V_{sat}}{\varepsilon}\right)^{1/2}$ ($W.cm^{-1/2}\ s^{-1/2}\ K^{-1}$)	13.8×10^2	6.2×10^2	80.3×10^2	439.2×10^2
Ratio Relative to Silicon	1	0.45	5.8	31.8

[1] Related to the power and frequency limits of transistors

[2] Describes high frequency limits set by thermal problems

PLASTIC PART DESIGN ANALYSIS USING POLARIZED FILTERS AND BIREFRINGENCE

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Plastic Part Design Analysis Using Polarized Filters and Birefringence

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KEY WORDS: birefringence, stress/strain, transparent/translucent plastic materials, index of refraction, optically heterogeneous, photoelastic fringe patterns, polarizing filters, ASTM D-4093.

PREREQUISITE KNOWLEDGE: The student should understand the fundamentals of plastics processing, materials characterization, and plastic part design.

OBJECTIVES: To understand the application of ASTM D-4093, "Standard Method for Photoelastic Measurements of Birefringence and Residual Strains in Transparent or Translucent Plastic Materials" to plastics part design and processing. To be able to observe and understand the impact of using a polariscope in plastics manufacturing in an effort to improve product quality. To understand how critical part design is to possible future failure analysis.

EQUIPMENT AND SUPPLIES:

(1)Glare-stop polarizing filters (12" X 12")
(2)Plate glass (4 pieces - 0.125" X 12" X 12") to protect filters
(3)Light source (overhead projector)
(4)Part Loading Fixture (optional)
(5)Transparent or translucent plastic parts
(6)Polarizing unit

INTRODUCTION: As the demand for engineered plastic products increases, greater emphasis must be placed on material, processing and part analysis. ASTM D-4093, "Standard "Method for Photoelastic Measurements of Birefringence and Residual Strains in Transparent or Translucent Plastic Materials" has some very real, cost effective applications for plastics product design, materials selection and testing, processing and failure analysis.

Internal stress in a molded plastic product can cause the part to warp, twist or bend, craze/fracture and/or eventually fail. The cause of this stress is residual physical pulling that is present when the plastic is pliable, but becomes captured upon solidification of the plastic. These stresses cannot be seen by the naked eye, but with the use of a polarized filter and a light source through a transparent or translucent part, these residual internal stresses are identifiable.

This laboratory will investigate the possible causes of internal residual stresses in a plastic part as a result of poor part design. Processing parameters and material selection can also have an impact on internal stresses but that is beyond the scope of this experiment.

Light waves travel through a vacuum in a straight line from its source, vibrating in a direction perpendicular to its axis of transmission (the direction in which it is traveling). When the light wave strikes a heterogenous material, the vibration that was perpendicular to its axis of transmission becomes distorted. In fact, the vibration splits into two separate waves vibrating in separate planes. The two waves have different indexes of refraction, and the difference between these is called the birefringence. When these two light waves emerge from the heterogeneous medium, they are out of phase, and appear to the human eye as different color bands when passed through an optical filter. These color bands in conjunction with injection molding design will be the focus of this experiment. Large concentrations of different color bands indicate a high level of internal, residual stress. See ASTM D-4093.

When a two dimensional model is subjected to loads, the resulting stresses exist generally over the entire surface of the model. The stresses in the model produce proportional optical effects which appear as isochromatic fringes. Starting with the unloaded model, and applying the load in increments, fringes will appear first at the most highly stressed points. As the load is increased and new fringes appear, the earlier fringes are pushed towards the areas of lower stress. Under white light, the photoelastic fringe pattern appears as a series of successive and contiguous different colored bands in which each band represents a different degree of birefringence corresponding to the underlying stress in the part. The color of each band identifies the birefringence, or fringe order (and stress level), everywhere along the band. White light is composed of all wavelengths in the visible spectrum. Table 1 will give the complete color sequence, the fringe order and the corresponding stress level possibly found in a part.

Color	Relative Retardation	Fringe Order	Stress	
			psi	MN/m^2
Black	0	0	0	0
Gray	160	0.28	90	0.62
White	260	0.45	144	0.99
Pale-Yellow	345	0.60	192	1.32
Orange	460	0.80	256	1.76
Dull Red	520	0.90	288	1.99
Purple (Tint of Passage)	575	1.00	320	2.21
Deep Blue	620	1.08	346	2.38
Blue-Green	700	1.22	390	2.69
Green-Yellow	800	1.39	445	3.07
Orange	935	1.63	522	3.60
Rose-Red	1050	1.82	582	4.02
Purple (Tint of Passage)	1150	2.00	640	4.41
Green	1350	2.35	752	5.19

Green-Yellow	1440	2.50	800	5.52
Red	1520	2.65	848	5.85
Red Green Transition	1730	3.00	960	6.62
Green	1800	3.10	992	6.84
Pink	2100	3.65	1168	8.05
Pink Green Transition	2300	4.00	1280	8.86
Green	2400	4.15	1328	9.16

Table 1. Isochromatic Fringe Characteristics using photoelastic plastic. Experimental Stress Analysis by Dally and Riley.

The basic polariscope utilizing the two polarizing filters will show full field interpretation of fringe patterns in an effort to obtain an overall assessment of nominal stress magnitudes and gradients. A more sophisticated polarizer and analyzer with two insertable quarter-wave retardation plates that are mechanically coupled for synchronous rotation will provide additional analysis. The more sophisticated unit provides quantitative measurements for:

- determination of principal stresses
- magnitude and sign of the tangential stress along free boundaries and in regions where the state of stress is uniaxial
- magnitude of the difference in principal stresses in a bi-axial stress state.

The importance of proper plastics part design can be shown using ASTM D-4093. Part failure can be minimized through attention to inside sharp corners, varying wall thickness, sink marks, molded in metal parts, bosses, ribs, weld lines, gate size and location, blind holes, and functional surface characteristics. The specific specifications for each part is a function of the particular material selected. Even though there are approximately 15,000 different plastics available, specific design guidelines have been developed for generic plastic classifications. The generic guidelines are available from the material manufacturer.

Molded in stresses will be concentrated around gates, openings, inside sharp corners, sharp transitions in cross section, and weld lines. The stressed areas can detract from various strength properties by as much as 20 percent. Loads, heat, pressure, light, and chemicals should be used with caution in the affected stressed areas.

PROCEDURE:

1. Obtain a transparent/translucent injection molded plastic part with engineering applications. The part should fit between the two polarizing filters and should be easily rotated about the X, Y, and Z axis. Use the polarizing filters to identify any and all residual stresses. If there are no noticeable fringe patterns, find another plastic part. Be sure to rotate your part thoroughly in an effort to seek out all fringe patterns.

Each of the filters is unidirectional. Set up the polarizing unit on the overhead so that the axes are crossed and the polariscope is in the dark-field. If the transmission axes coincide the polariscope is in the light-field. Dark-field means that with no stress in the model, all light is extinguished and the model appears uniformly black. As the load is applied, the most highly stressed regions begin to take on color, first grey, then white, violet and so on. The purple fringe is referred to as the tint of passage and is very sensitive to a small change in stress level. The purple tint of passage is selected to mark the increment in relative retardation equal to a fringe order of unity (N = 1). The tint of passage is a sharp dividing zone occurring between red and blue in the first order fringe, red and green in the second order fringe, and pink and green in the third, fourth, and fifth order fringes. Beyond five fringes, white light analysis is not adequate. See Table 1.

The part can be loaded either in tension or compression using the part loading fixture. This analysis will begin to show how the part reacts under various forces and can also be used in failure analysis. Fringe patterns will change in proportion to the force applied.

2. Locate where all gate(s), ejection pins, core pins, parting lines, etc. may have been in the molding tool. Generate an isometric drawing of the part, include all bosses, ribs, bolt holes, inserts, tapers, sink marks, blind holes, and functional surface characteristics. Assume your part represents the typical/average part produced.

3. Determine the specific design criteria for the material that may have been used in your particular part. Using available calipers, micrometers, and other measurement tools as necessary determine if your part falls within the design criteria as specified by the manufacturer. Cite all deviations from the manufacturers specifications. Several assumptions need to be made 1) you know what specific material is used to process the part and 2) the part specifications available to you are identical to those used by the original designer.

4. Identify at least 10 critical engineering properties (as best as possible) that you would design into the part. Consider the following and all assumed subheadings as a minimum:

Mechanical
Chemical
Physical
Dimensional

5. Map (using CAD) the fringe patterns of each critical area of the part that you have selected. Record as accurately as possible the fringe patterns, include shape, width, height, and the colors associated with each pattern. Associate color, fringe order, and stress present in various areas of your part.

6. Associate the fringe patterns with your part as designed: i.e., identify what design characteristics may have caused the fringe patterns. Recall material selection and processing may impact the development of the fringe patterns. For the sake of this experiment, assume that the impact of processing and material selection is negligible.

7. Identify where failure may occur in the part in normal use. Identify the conditions and environment under the heading normal use.

8. Identify what design changes need to be implemented to avoid future part failure.

INSTRUCTOR NOTES:

1. Due to the variety of transparent/translucent plastic parts available it is important that the students be encouraged to critique each part and material as a team. The use of the overhead will bring the image to the screen for enhanced group participation.

2. Photographing with the more spectacular fringe patterns can be beneficial. 400 color print film works well with an 80A (blue) filter to compensate for the projector halogen bulb. A close up lens with the room lights out produces the best quality prints.

3. Processing parameters, material changes and chemical resistance tests can be used in conjunction with the polarizing filters to further expand on the study of materials.

4. It is wise to keep a variety of parts and test samples at the polarizer to demonstrate the effectiveness of the apparatus.

5. Pressure transducers can be used in conjunction with the part loading fixture to develop a numerical index of forces.

6. Depending on equipment availability slow speed video photography of the part being loaded can contribute to design/failure analysis.

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SOURCES OF SUPPLY: The polarizing filters can be obtained at a local camera store. The overhead projector should be readily available. All other parts for the polarizing unit and part loading fixture can be obtained from a well stocked hardware store. Commercial polarizing units available from Measurements Group - Photoelastic Division, P.O. Box 2777 Raleigh, North Carolina. A basic polariscope can be produced for less than $60.00.

MICROWAVE SINTERING OF MACHINING INSERTS

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KEYWORDS: microwave, dielectric loss factor, sintering, structural ceramics, machining insert, design of experiments, bulk density.

PREREQUISITE KNOWLEDGE: The student should be aware of the normal procedure for producing high quality ceramic parts from both dry powders and wet plastic forming, and an introduction to the dielectric properties of materials is also necessary. Both of these can be found in most texts for introductory materials courses. Some knowledge of metalcutting tools for a lathe is beneficial, and this may require a demonstration or observation in the university shop. A thorough concern for safety with microwave equipment is necessary.

OBJECTIVES: To use a designed experiment to determine the process control parameters for microwave sintering in the production of ceramic cutting tool inserts. Parameters may include compaction load, heating rate, sintering temperature and time, and use of a preliminary degassing step.

EQUIPMENT AND SUPPLIES:

1. Prepared ceramic powder blend
2. Die set for triangular machining inserts with either 0 or 11 deg rake
3. Universal testing machine set up for compression
4. Scale which reads to hundreths of a gram
5. Microwave oven equipped with optical pyrometer or thermocouple
6. Mechanical vacuum pump
7. Dewaxing cover with thermocouple
8. Vacuum chamber with insulated sample holder
9. Jolly balance

INTRODUCTION: Microwave heating is also known as UHF dielectric heating. A dielectric material (an insulator) becomes polarized when placed in an electric field, and an alternating current thereby causes cyclic distortion (dipolar rotation) of molecules and heating (1,2). Higher frequencies of the field cause a more intense internal friction effect, but penetration of the field into the interior of a workpiece is less. Radio frequencies (10 to 300 MHz) can be used, but a household microwave oven operates at 2,450 MHz (2.45 GHz) and an industrial oven at 28 GHz has been developed (1).

The dielectric loss tangent and the relative dielectric constant are material properties that determine heating efficiency and penetration. When these are low, the material tends to be opaque to microwaves, so higher values are necessary for adequate energy absorption. Some plastics such as PVC have high values in the radio range and can be "ultrasonically welded." Some ceramics such as alumina are opaque at room temperature but become absorbers exponentially as temperature increases, so preheating them can trigger a response. Metals are not dielectric materials and reflect microwaves, which allows them to be used to contain the radiation in the oven.

Ceramic and cemented carbide cutting tools are made from a wide variety of combinations of tungsten carbide, titanium carbide, alumina, silicon nitride, boron nitride, and other ceramics using powder pressing and sintering technology (3). Many of these materials have properties that allow them to be sintered with microwaves to greatly decrease production times and costs. With the advent of surface coatings of TiCN, low cost and high strength of the substrate are more important than wear resistance.

PROCEDURE:

1. The instructor will give the sintering cycle in a standard oven for your ceramic mix. (For a cemented tungsten carbide insert, this would include dewaxing at 433 C for one hour, degassing at 1175 C for 45 minutes, and final sintering at 1475 C for two hours.) Determine which variables you want to study and design a two factor, three level experiment with bulk density as the response (4). Discuss your plan with the instructor first.

2. Determine the weight of powder necessary to fill the die cavity, allowing for 30% porosity after pressing. Weigh out that amount and assemble the die set for pressing. Load the first sample, called a "compact," to 3200 kg. Unload the die, and measure the thickness of the compact and observe the condition of the edges. If the thickness is not between 3.75 to 3.83 mm, adjust the weight of the powder for the next compacts.

3. To start the sintering process, load up to three compacts in the sample holder in the oven. If your powder was coated with a wax plasticizer, that must be vaporized off first, so cover the samples with the dewaxing hood and connect it to the vacuum pump. Be sure that the thermocouple in the hood is touching one of the compacts. Turn on the oven with medium power and bring the temperature up to about 400 C. Switch to low power and try to keep the sample at that temperature for ten minutes or until no more wax vapors are present.

Turn off the oven, open the door and replace the dewaxing hood with the vacuum hood. SAFETY PRECAUTION: the samples are hot enough to cause severe burns, and other parts of the system may be hot. Wear safety glasses while working in the oven. Pump out the chamber, backfill with nitrogen, and evacuate again. Start the oven at high power and monitor the temperature with the optical pyrometer. When it approaches your planned cycle limit, reduce power and try to keep temperature within ten degrees of optimum. At the end of your cycle, turn off the oven and wait for the inserts to cool before turning off the vacuum.

4. Use the Jolly balance to measure bulk density. Plot results and make recommendations for optimum processing. Densities approaching 99% need to be achieved for maximum tool life.

INSTRUCTOR NOTES: This project demonstrates an emerging energy conserving technology, results in producing a usable product, relates a familiar device to the study of materials, requires knowledge of electrical properties, and allows many avenues for a designed experiment with minimal time consumption. It also demonstrates that real life is not as simple as it first appears, because there is a high probability of nonuniform fields, arcing, hotspots, and broken or porous parts. In fact, working on troubleshooting skills may be one of the main hidden objectives of this experiment.

An interesting addendum is to use the inserts on a lathe and measure wear rate while cutting a material such as cast iron or 390 aluminum. This requires knowledge of optimum feeds and speeds for the material to avoid biasing and may take far too much time for a materials class. But it can provide continuity to a later course on processes. In our manufacturing processes course, the students also make the tool holder for the insert, so they have firsthand knowledge of the complete system.

Finding a source of small amounts of raw materials is one problem with this experiment. We were lucky to find a manufacturer that would sell us a small container of sized and blended powder at cost. Another problem is avoiding reactions between the insert and the atmosphere or the container, which is well documented and easily avoided. Unfortunately this is compounded by the need to use an insulating container with a low dielectric loss. Some graphite boats used in traditional sintering proved to be better absorbers than our samples and therefore useless. A boat made from board insulation also heated faster than the sample, so we started using a wool insulation. Casting plaster also seems to work well for the outer structure.

The cost of an optical pyrometer for high temperatures, with a small focal point and appropriate focal length, is about $2,000. A platinum thermocouple with molybdenum sheath is about $350, but it will read about 100 C too cold because of heat transfer down the sheath. A ceramic sheath will tend to heat from microwave absorption and is even more unreliable. A controller specifically for microwave ovens can be purchased for about $10,000, but trial and error for finding appropriate power settings for each stage is acceptable. I am working on utilizing a standard furnace controller with a platinum thermocouple and may find that to be the best way to provide acceptable control of temperature.

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KNOTTY KNOTS

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KEYWORDS: String, Knots, Load reduction factor, Stress concentration

PREREQUISITE KNOWLEDGE: The student should be familiar with different types of ropes, strings, and threads and may be aware that these materials can be weakened by tying a knot in the line. At the high school and college levels this concept may be found in physics programs, but is familiar to the student studying mechanics of materials in terms of the stress concentration factor. A background in the tying of different styles of knots may be helpful.

OBJECTIVES: Tension tests are performed to determine the effect of different discontinuities on the behavior of a material. String is first tension tested and then different types of knots are tied in the string to determine the impact of the type of knot on the load capacity of the string.

EQUIPMENT AND SUPPLIES:

1. String samples of different types: Fish line, thread, suture thread, string to tie cake boxes. An effort should be made to secure string that will carry a maximum of 9 Kg (20 pounds) so the loads will not exceed this amount.
2. Model IN-50 Chatillon tubular scale to 25 Kg (50 lbs.) with two 's' hooks and a 'homemade T' handle attached to one of the 'S' hooks and the scale, and a slider on the barrel of the scale for maximum load value indication.
3. Two load block holders for string ends (see Appendix Figures A1 and A2 for details). Needed are two standard size wooden spools 20 mm dia. by 30 mm long (3/4 in. dia. by 1 1/8 in. long), two carriage bolts m5x1 by 50 mm (1/4 20-UNC-2 by 2 in.) long each with one washer and one nut, two 150 mm (six inch) long pieces of 35 mm x 60 mm (1/2 in. by 2 1/4 in.) flex angle or equivalent with an 'S' hook attached to the moveable holder.
4. Meter (Yard)stick
5. Scissors for cutting string
6. Vise attached to workbench to hold one holder
7. Data Sheet (see below for the format)
8. Heavy gloves (the kind a welder uses)
9. Safety glasses (always use eye protection)

PROCEDURE: Tension tests are to be performed to determine the effect of different discontinuities on the behavior of a material. The initial step is to select a type of string to be tested. First is the tension test for a single strand of string. This is the baseline for all comparisons. In each condition four trials are run: samples are to be made, loaded to failure and the failure loads for each trial recorded and the average failure load tallied.

The subsequent conditions are to select a knot style to tie in the center of the string sample to be load tested. Again, four samples with failure loads and an average failure are evaluated. The ratio of the average load carried with a knotted string to the average load carried by an unknotted string is calculated. Reduction in knotted string load capacity changes as a function of knot type. The test procedure should be applied to four different type knots to show the range of impact the type of knot presents and the variations between the knot types. The test procedure is presented in detail below.

The initial step is the selection of a variety of string to test. Twenty samples, each three feet long, are required. The first condition is a tensile evaluation for a single strand of string. This is the baseline for all comparisons. Four samples are to be made, loaded to failure and the failure load for each sample recorded in the table shown below in the second column, and then the average failure load computed and tallied for future use.

To test the single strand secure a 0.8 meter (30 inch) length of string, tie a loop on each end, fasten one loop to each threaded extension of the binding post of the load application apparatus (see Figure A1), and wind the string one and one-half times around the spool. The spool winds are needed to reduce the stress concentration in the string mounting area. The string under test is loaded by pulling on the spring scale that you read as the load is applied. If a maximum load indicator slider is available, it will help recording the maximum load. The experimenter should wear a pair of heavy gloves, and in one hand support the load application device and with the other hand, apply load. One load applicator is to be firmly held in the vise. The hand-held load application device is to be held level with the vise mounted load application device to assure valid results. **BE SURE YOU or your partner CAN READ THE SCALE just before the line breaks. (The load may be close to 9 Kilograms (20 pounds) -- Please be very careful,** wear safety glasses and avoid injury to yourself or the scale.)

Table of String Failure Loads in Kilograms (Pounds)

Unknotted String		Knot 1	2	3	4
Trial	Load	Load	Load	Load	Load
1					
2					
3					
4					
Total					
Average					
Load ratio: Knot style to Unknotted					

Just below this table is a good place to describe the knot type used for each number (a sketch can be made to show how to make the knot, too). For example, single loop, figure of eight, figure of eight plus an half-loop, stevedore's.

The second condition is to select a knot style such as suggested in the notes below the table shown above, and tie the knot in the center of the string to be load tested. Again, four samples with failure loads and an average failure load are to be evaluated. Please add the data to the table, and compute the average load carried by the knotted samples and the ratio of the average load carried by a knotted string to the average load carried by unknotted string. One hundred times this ratio represents the percentage of load carried by the knotted string.

The third, fourth, and fifth conditions are the second condition repeated for three more knot types. Ashley (1), Boy Scouts of America (2), and The Encyclopedia of Sailing (3) provide good sources for different knots which serve the same purpose.

COMMENTS :

There are some interesting things to consider after performing the first experiment.

1. Some knots tend to cut into and destroy string, and this varies between string types.
2. A test with Number 6, Black non-capillary braided suture silk found an average load of 4.367 Kilograms (9.625 lbs.) in an unknotted string with reduction factors of 54.0%, 51.3%, 65.1%, and 65.1% for a single loop knot, figure-of-eight knot, figure-of-eight plus an extra half loop knot, and stevedore's knot, respectively.
3. A different string will react differently, and a yellow covered plastic line 025-6-7013-66 available in the Mechanical Laboratory took an average load of 7.656 Kilograms (16.875 lbs.) in an unknotted string with reduction factors of 54.1%, 62.2%, 49.0%, and 40.4% for a single loop knot, figure-of-eight knot, figure-of-eight plus an extra half loop knot, and stevedore's knot, respectively.
4. Instead of tying a knot in the center of a string, cut the string and tie selected knot to hold the ends together.
5. Some lines when cut and the cut ends are tied as suggested in 4 above have new conditions to consider. The surface finish on the string may be such that the tied knot slips and pulls out in contrast to holding. A second single loop knot on each free cut end may provide enough resistance to minimize knot(s) slipping/pulling out.
6. In the case of college students the connection between the load reduction factor and a stress concentration factor (4) can be made. As an example consider that a given string carries 5 Kilograms via a test and a knot is placed in the string which limits the load to 3 Kilograms. The load reduction factor is 0.60 or 60% while the reciprocal of 0.60 is 1.67 and, this is often referred to as a stress concentration factor. The meaning of the stress concentration factor is that when an engineer makes a design calculation which involves a stress concentration factor, the actual material selected for a member in a design must have the computed load increased by the factor. In this example, when a calculated load of 10 Kilograms is made for the material, the selected material for the member must be modified by 1.67 so 1.67 times 10 Kilograms or 16.7 Kilograms is the load the tensile member must withstand (without the stress concentrator present).
7. Strings can be conditioned by changing humidity, temperature, soaking in water, alcohol, oil, etc., and cooking in a microwave to simulate artificial aging.

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APPENDIX 1 - LOAD APPLICATION APPARATUS

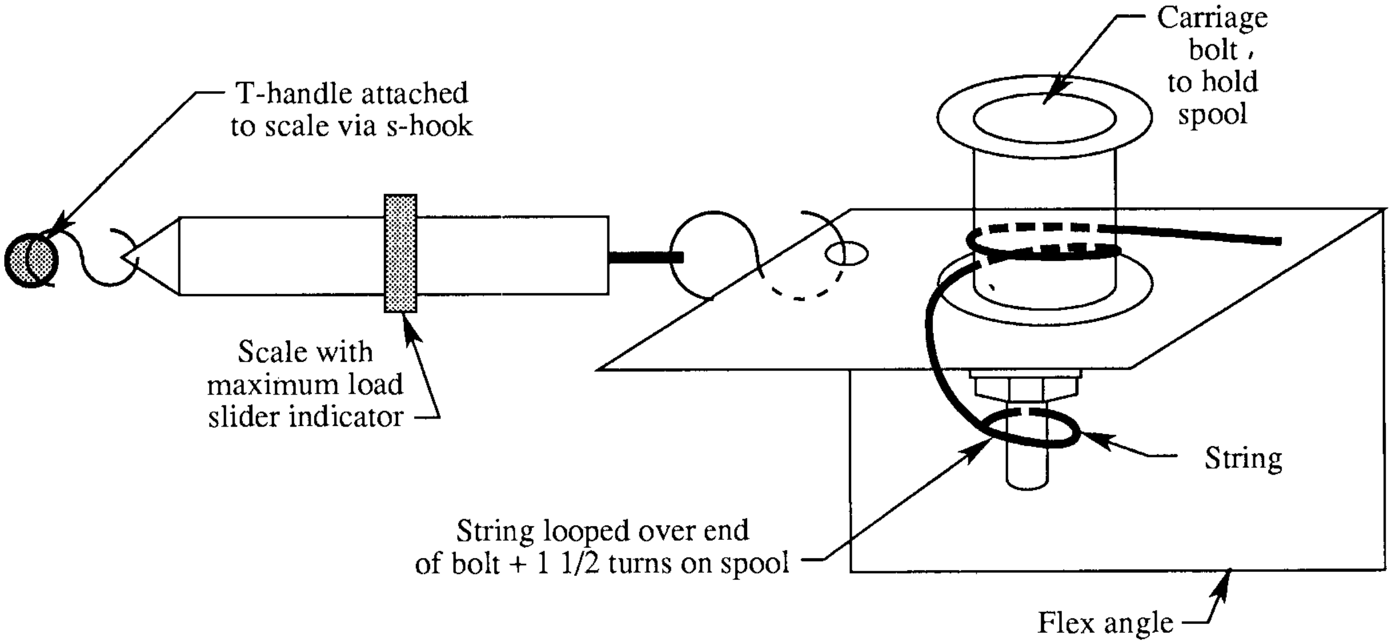

Figure A1 - Load block (2 needed).

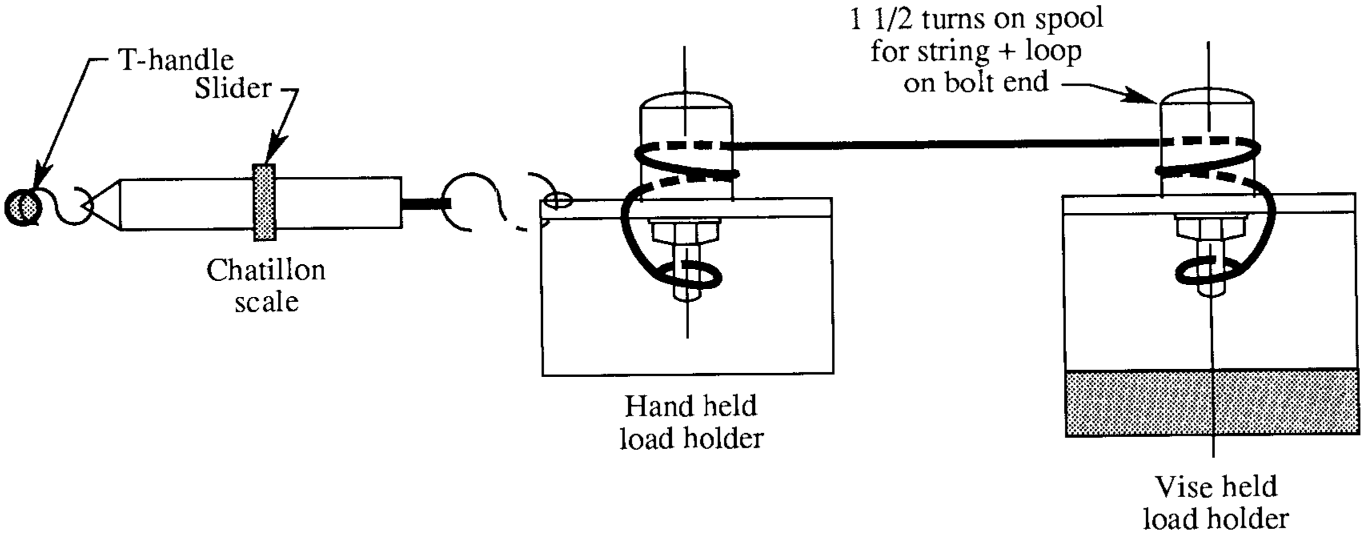

Figure A2 - Load application set-up.

THREE-POINT BEND TESTING OF POLY (METHYL METHACRYLATE) AND BALSA WOOD

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THREE-POINT BEND TESTING OF POLY(METHYL METHACRYLATE) AND BALSA WOOD

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ABSTRACT: A procedure for determining the Young's modulus for poly(methyl methacrylate) and balsa wood in three-point bending is described. A linear variable differential transformer (LVDT) is used to measure displacement as a function of a series of discretely applied loads. Emphasis is given to experimental methodology that includes a verification of the operation/calibration of the LVDT using gage blocks. In addition, the displacement of the test apparatus itself is measured and removed from the total displacement data, allowing just the response of the materials to be obtained. The problem statement for balsa wood is posed to require that an experimental procedure be devised both to determine the Young's modulus and to address a specific design requirement for maximum center deflection of road bed planking in a laboratory bridge truss.

KEY WORDS: three-point bend testing, LVDT, Young's modulus, poly(methyl methacrylate), balsa wood.

PREREQUISITE KNOWLEDGE: first-semester sophomore laboratory experiment requiring training in the use of a digital multimeter and the assessment of measurement uncertainty; flexural testing concepts, including the obtainment of Young's modulus, as described in an introductory materials science or mechanics of materials course.

OBJECTIVES:

(a) Experimental Goals:

1. to measure the deflection of a flat plate of poly(methyl methacrylate), or PMMA, in three-point bending in order to determine its Young's modulus and
2. to design and implement an experimental procedure to determine the Young's modulus for a balsa wood beam in three-point bending and to assess the maximum center deflection allowance specified for the construction of a laboratory bridge.

(b) Learning Goals:

1. to become familiar with the use of a linear variable differential transformer (LVDT), a widely used transducer for making linear displacement measurements;
2. to become familiar with performing a laboratory calibration procedure using National Institute of Standards and Technology (or equivalent) traceable standards in characterizing the operation and accuracy of a transducer provided with a factory calibration;
3. to become familiar with an experimental procedure for determining the Young's modulus of a material tested in three-point bending by accounting for any displacement in the test apparatus itself; and
4. to become familiar with designing and then implementing a measurement procedure to obtain desired experimentally-determined material parameters.

EQUIPMENT AND SUPPLIES: (1) LVDT (direct current (DC)-operated and spring-loaded) -- Lucas Schaevitz GCD-121-125 with mounting fixture; (2) Lab jack with stabilizing weights; (3) Gage blocks: Jansson 0.020" and Mitutoya 0.050", 0.1001", and 0.200"; (4) Protoboard (Global Specialties Model 203A), supplying ±15 V DC or alternative power supply with the same DC voltage capability; (5) Digital multimeter (Fluke model 45); (6) Three-point bend test apparatus (American Instrument Co. cat. no. 4-3900); (7) Set of 8 brass masses (Central Scientific Co. cat no. 09600); (8) Steel beam (16 mm wide X 10 mm thick X 115 mm long); (9) PMMA plate (36.9 mm wide X 2.17 mm thick X 94.9 mm long); (10) Balsa wood beam (19.0 mm wide X 6.27 mm thick X 254 mm long); (11) Metric micrometer; and (12) Metric vernier caliper.

PROCEDURE:

Introduction

The modulus of elasticity, or Young's modulus, is a fundamental material parameter defined using Hooke's law. For pure elastic deformation, stress, σ, is related to strain, e, by $\sigma = Ee$, where E is Young's modulus. This modulus provides a measure of material stiffness and is widely used in machine and structure design analyses. For example, often structures have a requirement of undergoing no more than a specified deflection for a given applied load. Computations yielding the necessary size of structural members to achieve the specification can be performed knowing Young's modulus for the material employed.

An LVDT [1,2] is a widely used electromechanical transducer that converts linear displacement, sensed by the movement of the device's core (Figure 4-54, Holman [3]), into a DC signal that can be detected, for example, by a digital multimeter (DMM) set to measure DC voltages.

This work consists of performing two displacement experiments involving the use of an LVDT. The first experiment investigates the response of a flat plate of PMMA to loading in a three-point bend test apparatus allowing determination of Young's modulus. The second experiment involves designing a measurement procedure in order to characterize the load-deformation behavior of a balsa wood beam proposed to be used in a laboratory bridge design.

Prior to performing the first displacement experiment, the LVDT that is employed must be calibrated using a series of gage blocks [4] to determine if the device is operating as expected. HOWEVER, THE PERFORMANCE DATA PROVIDED BY THE MANUFACTURER WAS OBTAINED UNDER MORE CONTROLLED LABORATORY CONDITIONS AND SHOULD BE USED IN THE ANALYSIS OF DATA MEASURED FOR THE TWO DISPLACEMENT EXPERIMENTS.

A schematic of the three-point bend test apparatus is given in Figure 1. Originally, the test apparatus incorporated a dial indicator to measure sample deflection. This is now accomplished using an LVDT. However, the dial indicator still is an integral part of the apparatus, providing vertical positioning and alignment of the loading nose (Figure 1, Reference [5]). Once a sample has been positioned in the apparatus, various applied masses are placed on a circular load platform so as to leave enough room for the LVDT to contact the platform near its circumference.

A. DEFLECTION OF A FLAT PLATE OF PMMA (Instructor Note 1)

1. Using the 0.020, 0.050, 0.1001, and 0.200" gage blocks, perform a calibration of the LVDT to be used with the three-point bend test apparatus (Instructor Note 2). To gain an additional gage block length, combine the 0.020 and 0.050" gage blocks by rubbing them together until they stick. Likewise, combine the 0.020 and 0.1001" gage blocks; then the 0.050 and 0.1001" gage blocks; and finally the 0.020, 0.050, and 0.1001" gage blocks. Before beginning the calibration procedure, adjust the height of the LVDT against a smooth rigid surface (e.g., steel beam placed on laboratory bench top) until the DMM reads 0.0 ± 0.2 V DC.

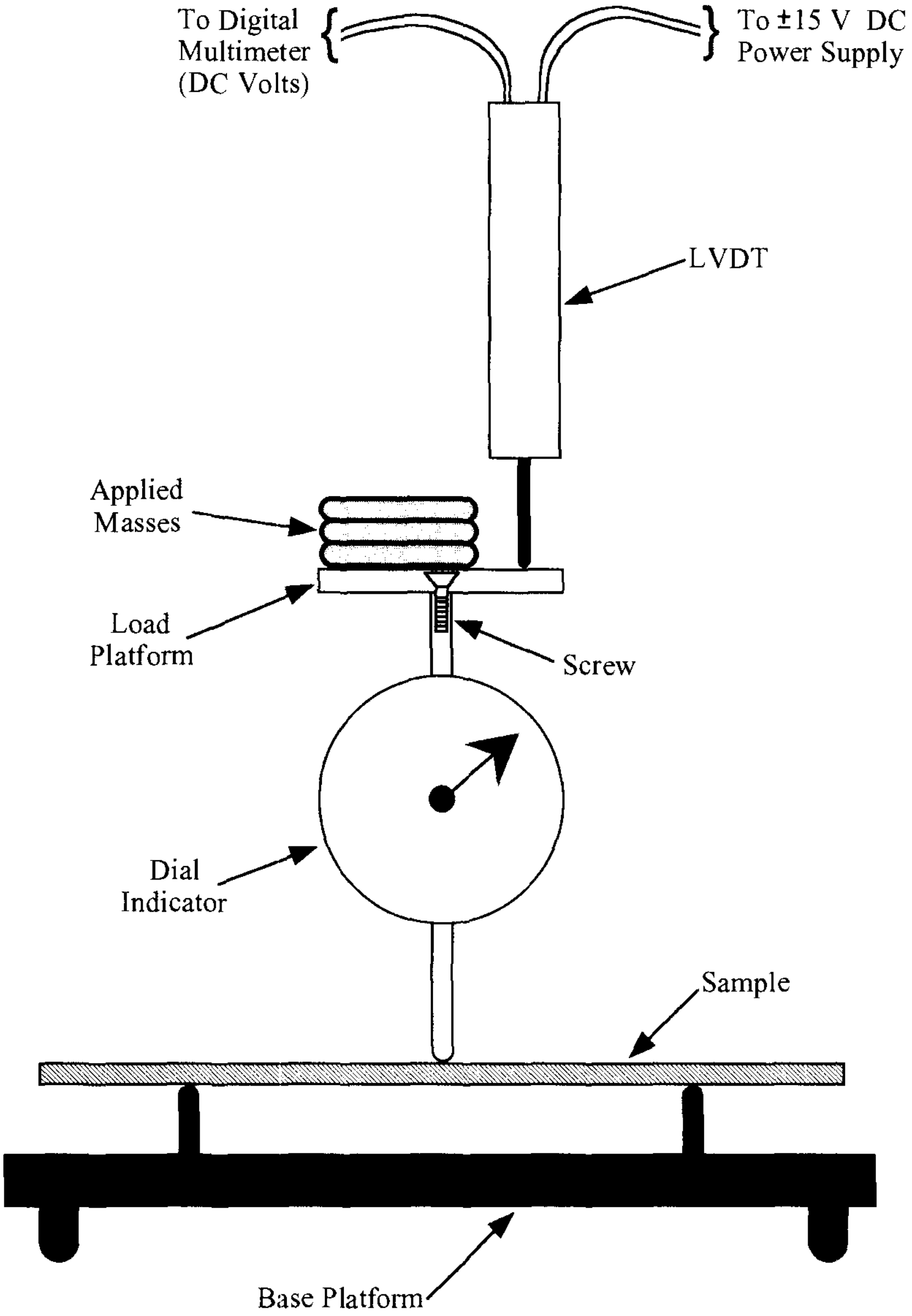

Figure 1. Schematic of Three - Point Bend Test Apparatus Instrumented with LVDT

2. Determine the displacement of the apparatus by testing the steel beam, having a 3 in. span support [5] length (Instructor Note 3). With the LVDT contacting the load platform of the three-point bend test apparatus, adjust the initial height of the LVDT until the DMM reads between +8 and +9 V DC. Load the beam with masses of 50, 100, 150, 250, 350, and 550 g. Record the output voltage for each loading.

3. Determine the deflection of the PMMA plate for a span support length of 3 in. Adjust the LVDT to yield a DMM reading between +8 and +9 V DC. Load with the same masses used previously, except for 550 g (Instructor Note 4). Record the output voltage for each loading. Repeat the process.

4. Measure [4] the thickness of the PMMA plate with a micrometer and its width with a vernier caliper. The span support length can be read directly off of the three-point bend test apparatus.

Data Analysis

1. Adjust the gage block data so that zero displacement equals zero volts. Plot LVDT output voltage, V, versus gage block thickness, in, for all of the gage blocks used. Perform a computer least-squares fit analysis on the data that is appropriate (Instructor Note 5) to determine the slope.

2. Adjust the steel loading data so that zero displacement equals zero volts. The value generated for each mass should be tabulated for possible use in correcting (Instructor Note 6) the deflection data of the materials (PMMA and balsa wood (Part B)) being tested.

3. Ignore the first set of PMMA loading data because of complications caused by a sample seating problem. Adjust the second set of PMMA loading data so that zero displacement equals zero volts. This data possibly has two components. The major portion is the actual PMMA deflection. There may be a minor contribution to the measured deflection data coming from the displacement of the apparatus itself. This can be evaluated quantitatively from the steel loading data since the steel bar that was used is a rigid body (i.e., undergoes no deflection) to a very good approximation for the masses used to load it. The apparatus displacement needs to be removed from the total deflection data for each mass to yield only the material response. This is accomplished using the relation

$$V_s = V_m - V_a \, , \qquad (1)$$

where
V_s is sample deflection voltage;
V_m is measured deflection voltage; and
V_a is apparatus displacement voltage.

Plot applied force, N, versus deflection, mm, of the PMMA during loading. Although this graph will probably exhibit some scatter, perform a computer least-squares fit analysis on the plotted values to determine the slope.

4. Compute E, GPa, for PMMA using the relation [6]:

$$E = \frac{L^3 m}{4bd^3} \, , \qquad (2)$$

where
L is span support length;
m is slope of the force-deflection plot;
b is beam width; and
d is beam thickness.

Uncertainty Analysis

Compare the current experimental calibration data generated for the LVDT using gage blocks with that supplied by the manufacturer.

B. DEFLECTION OF A BALSA WOOD BEAM

As part of an engineering assignment, it is of interest to design a bridge truss with road bed planking of balsa wood beams. First, it is necessary to determine experimentally the Young's modulus for balsa wood so that subsequent design analysis can be undertaken. Second, one of the design specifications requires that a center deflection of no more than 0.38 mm occurs when the bed planking experiences a load of 5.4 N applied at the center. The plank span support length is specified to be 8 in.

After examining a sample of the balsa wood planking, follow the guidelines that are detailed in Table 2-7 of Holman [7] and implement a test procedure that will obtain the requested material parameters. INCLUDE IN THE LAB WRITE-UP AN

EVALUATION AS TO WHETHER THE BALSA BEAM PLANKING THAT WAS TESTED WILL MEET THE DEFLECTION SPECIFICATION.

As part of the uncertainty analysis, determine the uncertainty in an LVDT displacement measurement on the balsa wood beam resulting when loaded with 5.4 N (Instructor Note 7). BE SURE TO FACTOR THIS CALCULATION INTO THE EVALUATION OF THE BEAM'S ABILITY TO SATISFY THE DESIGN SPECIFICATION.

SAMPLE DATA SHEETS AND PLOTS: All of the experimental steps were performed several times to verify that the results were reasonably reproducible. The data sets appearing in this section are considered to be representative.

LVDT Operation/Calibration Verification: The measured voltages obtained from the DMM as the LVDT was exercised using gage blocks with thicknesses ranging from 0.020 to 0.200 in. appear in Table I. The zero-adjusted output voltages are also listed and appear in a plot (Figure 2) versus gage block thickness. The least-squares fit analysis yielded a slope of 83.8 ± 0.2 V/in, comparing favorably with the scale factor = 83.513 V/in provided by the manufacturer.

Test Apparatus Displacement: Appearing in Table II are the measured and zero-adjusted output voltages obtained as the LVDT monitored the displacement of the three-point bend test apparatus as it was used to load a rigid steel beam for applied masses ranging from 0 to 550 g (Instructor Note 8).

PMMA Plate Deflection: The measured and zero-adjusted LVDT output voltages corresponding to the combined PMMA plate and test apparatus displacements are given in Table III. The zero-adjusted voltages are corrected (Table IV) to account for the apparatus displacement yielding voltages corresponding to the deflection of the PMMA sample alone. The applied masses are then converted to applied forces, N, using g = 9.81 m/s^2, while the corrected sample deflection voltages are converted (dropping the minus signs) to deflections, mm, using the manufacturer's scale factor = 83.513 V/in. The variation of applied force versus deflection is given in Figure 3. A least-squares fit analysis yielded a slope of 13.0 ± 0.6 N/mm (E = 3.81 ± 0.18 GPa), which compares favorably with a value of 12 N/mm corresponding to E = 3.4 GPa listed by Ashby and Jones [8].

Table I. Measured and Zero-Adjusted LVDT Output Voltages for Various Gage Block Thicknesses

Gage Block Thickness, in	Measured Voltage, V	Zero-Adjusted Voltage, V
0.000 (No Block)	-0.02	0.00
0.020	+1.67	1.69
0.050	+4.24	4.26
0.070	+5.90	5.92
0.1001	+8.42	8.44
0.1201	+10.08	10.10
0.1501	+12.55/6	12.57/8
0.1701	+13.73	13.75
0.200	+13.73	13.75

Table II. Measured and Zero-Adjusted LVDT Output Voltages Assessing Three-Point Bend Test Apparatus Displacement

Applied Mass, g	Measured Voltage, V	Zero-Adjusted Voltage, V
0	+8.58	0.00
50	+8.59	+0.01
100	+8.60	+0.02
150	+8.62	+0.04
250	+8.62/3	+0.04/5
350	+8.68	+0.10
550	+8.72	+0.14
0 (Unloading)	+8.58/9	+0.00/1

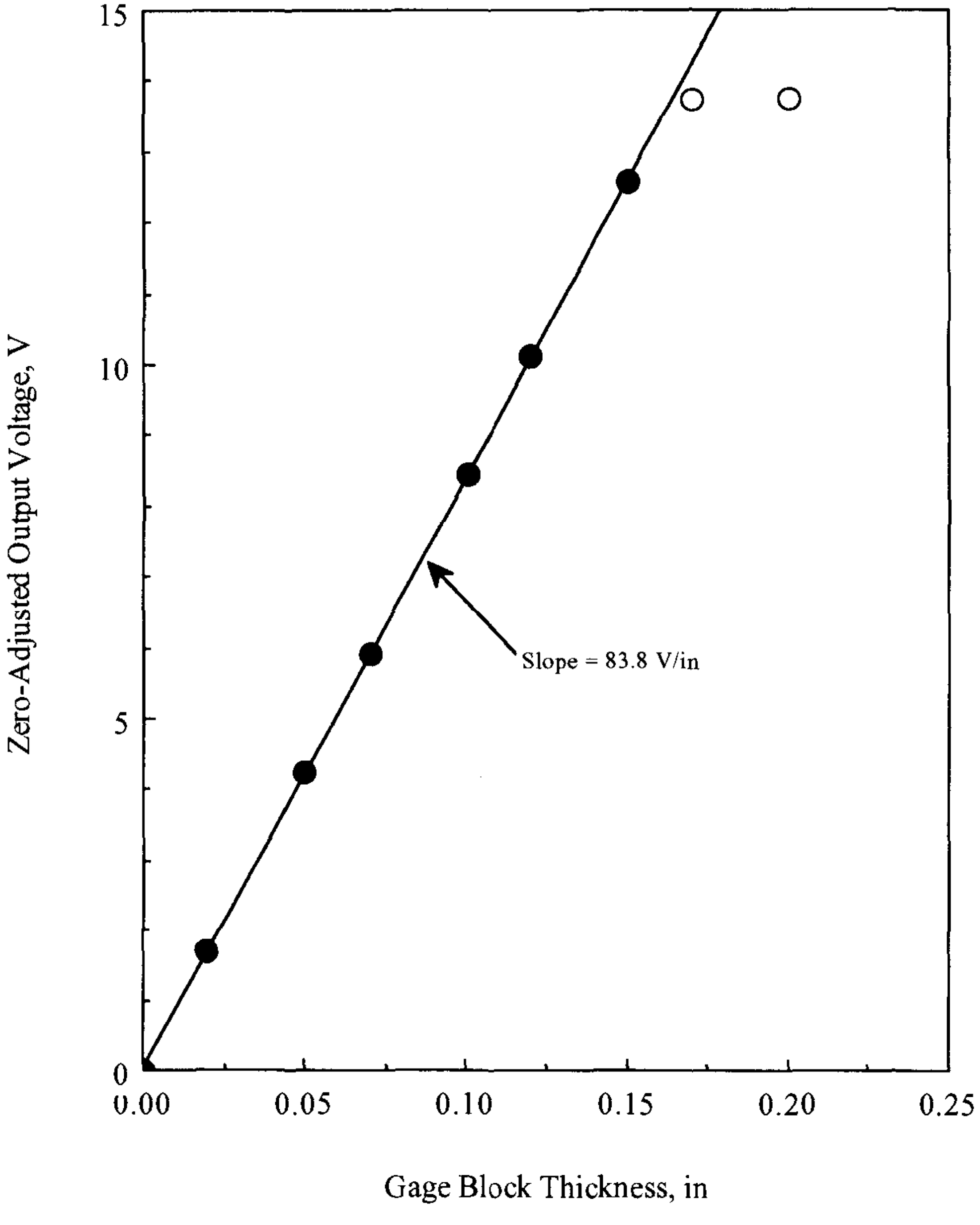

Figure 2. Zero-adjusted output voltage versus gage block thickness providing verification on LVDT operation/calibration.

Table III. Measured and Zero-Adjusted LVDT Output Voltages Assessing Total Displacement for PMMA Plate Loaded in Three-Point Bend Test Apparatus

Applied Mass, g	Measured Voltage, V	Zero-Adjusted Voltage, V
0	+8.54	0.00
50	+8.45	-0.09
100	+8.36/7	-0.18/7
150	+8.19	-0.35
250	+8.03/1	-0.51/3
350	+7.78	-0.76
0 (Unloading)	+8.53/4	-0.01/0.00

Table IV. Zero-Adjusted LVDT Output Voltages Assessing Total Displacement in Three-Point Bend Testing of PMMA Corrected to Account for Apparatus Displacement

Applied Mass, g	Zero-Adjusted Voltage, V	Apparatus Displacement Voltage, V	Corrected Sample Deflection Voltage*, V
0	0.00	0.00	0.00
50	-0.09	+0.01	-0.10
100	-0.18/7	+0.02	-0.20/19
150	-0.35	+0.04	-0.39
250	-0.53/1	+0.04/5	-0.58/5
350	-0.76	+0.10	-0.86

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*Corrected Sample Deflection Voltage = Zero-Adjusted Voltage - Apparatus Displacement Voltage

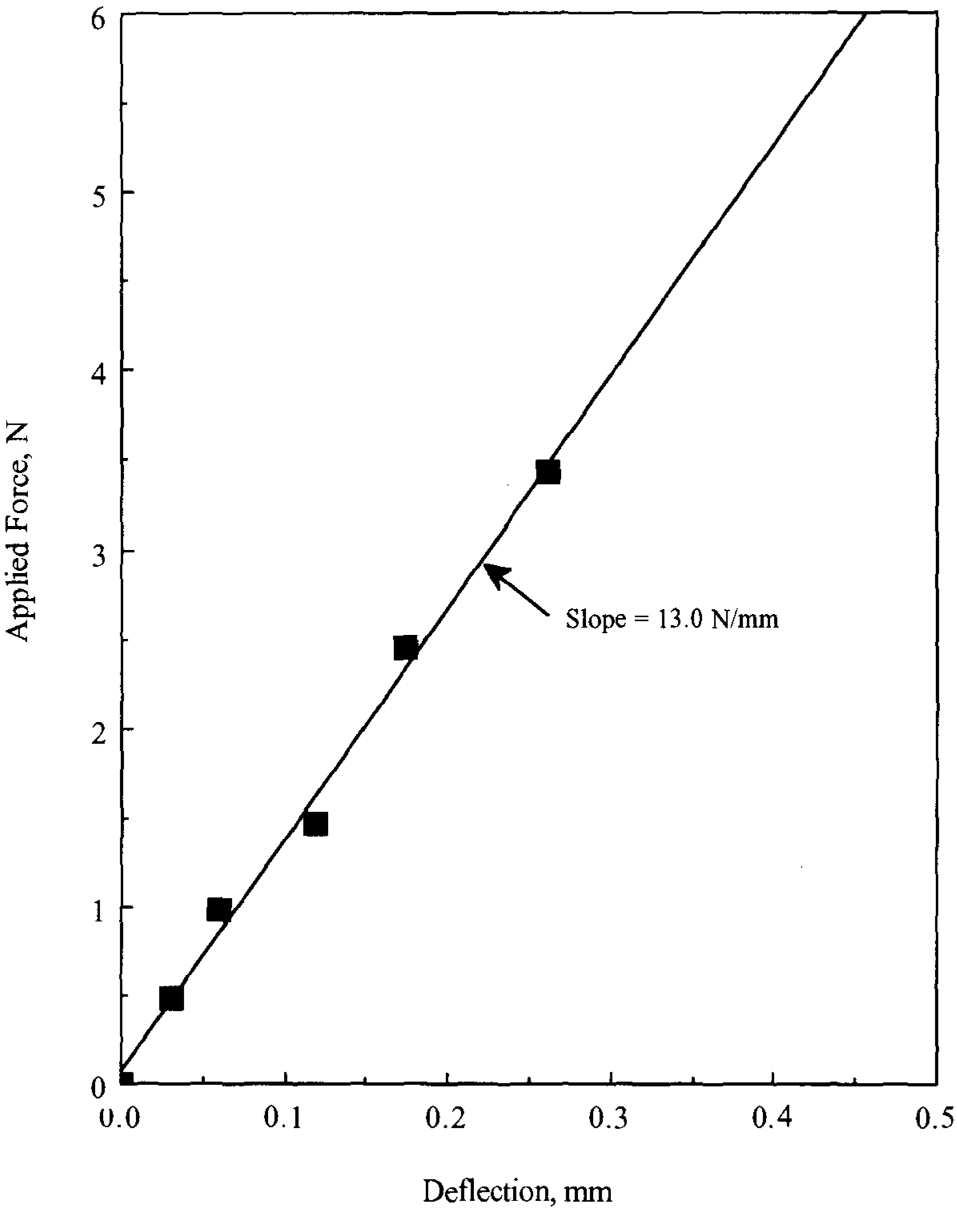

Figure 3. Elastic response of PMMA plate in three-point bend tester.

Balsa Wood Beam Deflection: Tables V and VI contain the various voltages for balsa wood beam testing that are analogous to those appearing in Tables III and IV, respectively, for PMMA plate testing. Figure 4 shows the variation of applied force versus deflection of the balsa wood beam. A slope of 12.9 ± 0.4 N/mm (E = 5.78 ± 0.18 GPa) was obtained by least-squares fit analysis to be compared with a value of 8.9 ± 1.8 N/mm corresponding to E = 4.0 GPa ± 20% (parallel to wood grain) given by Ashby and Jones [9]. While the agreement is not as good as that obtained for PMMA, the current balsa wood determination is still reasonable given the large variability [9] in the properties of wood. The measured center deflection is 0.405 mm, slightly exceeding the stated design specification requirement even when measurement uncertainty is considered (Instructor Note 7).

INSTRUCTOR NOTES:

1. This particular PMMA sample was chosen because it was readily available without incurring additional machining cost. True beam dimensions are preferable rather than the plate-like geometry used. American Society for Testing and Materials (ASTM) [5] specifies a 10 mm (versus 19.0 mm in this work) wide sample when the thickness is less than 3 mm. Additionally, the sample overhang must be greater than or equal to 10% of the span length for each end.

2. The LVDT calibration provided by the manufacturer involves displacements reported in inches. To connect with this calibration, the first part of the experiment uses gage blocks with thicknesses given in inches. Posing the experiment with mixed units is beneficial, however, because it allows students to gain facility with units conversion.

3. A 3 in. span support length for the steel beam was chosen to relate to the subsequent PMMA plate testing. However, this length is also valid for the balsa wood beam testing because the steel beam is rigid.

4. On unloading the PMMA sample that had been loaded to 550 g, the LVDT output voltage did not return essentially to the initial value. This suggests that the elastic limit of the PMMA plate may have been exceeded, necessitating that deflection data be obtained with the smaller applied masses. It is also possible that loading the sample to 550 g caused the sample to shift on unloading, making the LVDT reading unreliable.

5. The last two gage block thicknesses cause the linear range of the LVDT to be exceeded. This is readily seen in Figure 2 which the student must obtain before properly

Table V. Measured and Zero-Adjusted LVDT Output Voltages Assessing Total Displacement of Balsa Wood Beam Loaded in Three-Point Bend Test Apparatus

Applied Mass, g	Measured Voltage, V	Zero-Adjusted Voltage, V
0	+8.70	0.00
50	+8.60	-0.10
100	+8.55/6	-0.15/4
150	+8.41/2	-0.29/8
250	+8.13/4	-0.57/6
350	+7.94	-0.76
550	+7.51	-1.19
0 (Unloading)	+8.73/4	+0.03/4

Table VI. Zero-Adjusted LVDT Output Voltages Assessing Total Displacement in Three-Point Bend Testing of Balsa Wood Corrected to Account for Apparatus Displacement

Applied Mass, g	Zero-Adjusted Voltage, V	Apparatus Displacement Voltage, V	Corrected Sample Deflection Voltage*, V
0	0.00	0.00	0.00
50	-0.10	+0.01	-0.11
100	-0.15/4	+0.02	-0.17/6
150	-0.29/8	+0.04	-0.33/2
250	-0.57/6	+0.04/5	-0.62/0
350	-0.76	+0.10	-0.86
550	-1.19	+0.14	-1.33

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*Corrected Sample Deflection Voltage = Zero-Adjusted Voltage - Apparatus Displacement Voltage

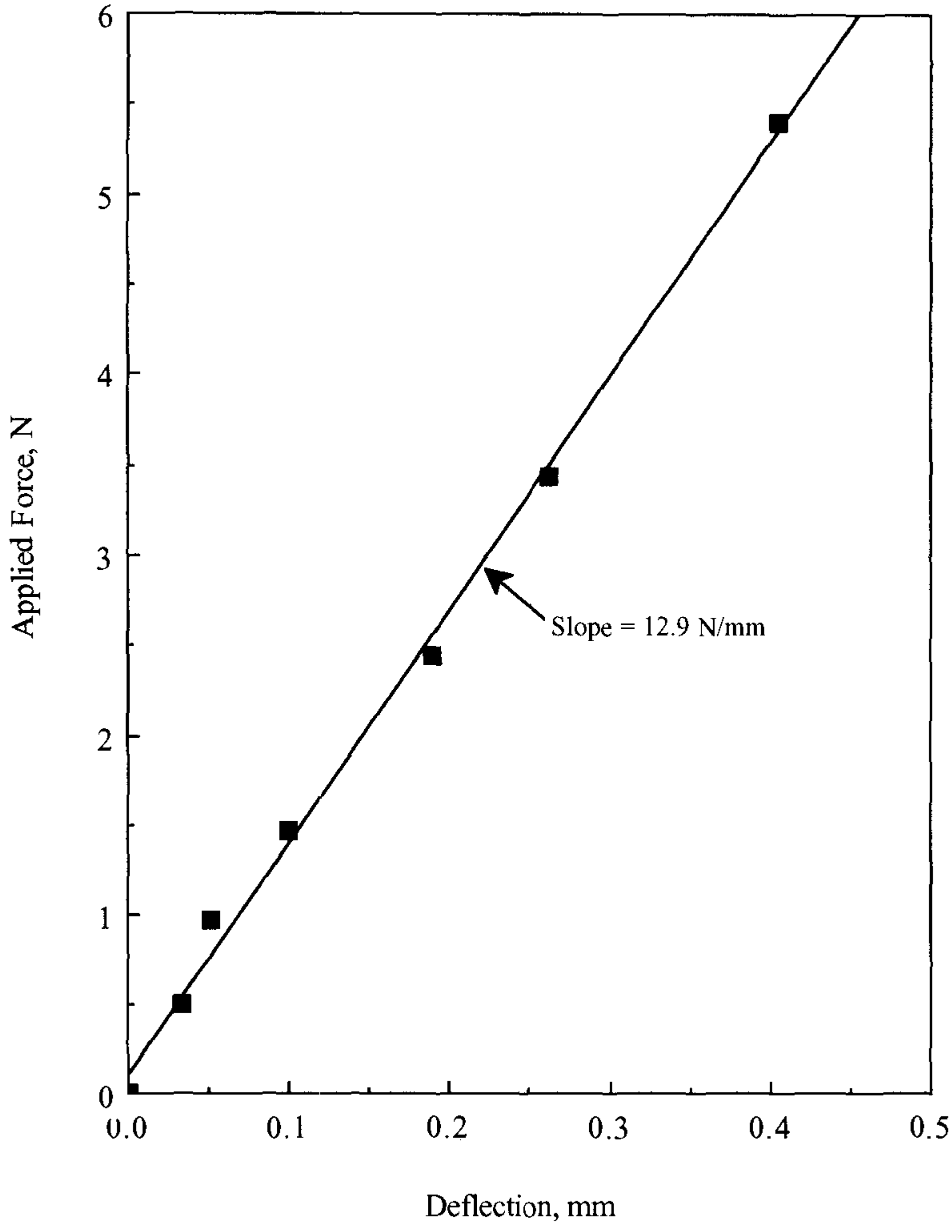

Figure 4. Elastic response of balsa wood beam in three-point bend tester.

performing the least-squares fit analysis, having discarded those data which are outside the linear range.

6. Tegart [10] provides an excellent treatment of the characteristics of materials testing machines. A sample pulled in tension is considered assuming linear elastic behavior for both sample and machine. The basic principles developed have application for materials tested in other stress states, such as compressive or flexural.

7. The calculation of the uncertainty in an LVDT displacement measurement depends on the uncertainty specified by the DMM manufacturer for the DC voltage scale used. The Fluke model 45 has a stated uncertainty of ±(0.025%(reading) + 2 least count digits). From the least-squares fit analysis, a force of 5.4 N causes a deflection of 0.409 mm, corresponding to a reading of 1.35 V with an uncertainty of ±0.02 V. This translates into a displacement uncertainty of ±0.006 mm.

8. Referring to Table II, the zero-adjusted voltages are positive indicating that the LVDT core is experiencing an upward displacement. This initially surprising result occurs because the load platform (Figure 1) is mounted to the vertical post by a butt joint with a top fastening screw. When the screw is slightly loose, the load platform pivots on the vertical post causing the LVDT to sense a small upward displacement as the masses are applied. At the same time, the side of the platform supporting the masses is undergoing a downward displacement. As an aside, an assignment could be given to redesign the load platform, seeking to correct the off-axis positioning of the applied masses and improve on the butt-joint mount.

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SOURCES OF SUPPLIES: The LVDT is available from Lucas Schaevitz, 7905 N. Route 130, Pennsauken, NJ 08110 for $545.00 (1993 price). The three-point bend test apparatus was originally manufactured by the American Instrument Co. which is no longer in business. It is not known whether this particular apparatus is still being made by another company. However, an apparatus could be machined and use a dial indicator (model 262J) purchased for $99.50 (1993 price) from B.C. Ames Co., 131 Lexington Street, P.O. Box 70, Waltham, MA 02254. The type of back on the indicator must be specified depending on the apparatus design. Alternatively, the dial indicator could be replaced with a newly-designed and machined fixture for the load platform/loading nose assembly.

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DIELECTRIC MEASUREMENTS OF SELECTED CERAMICS AT MICROWAVE FREQUENCIES

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Dielectric Measurements of Selected Ceramics at Microwave Frequencies

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KEY WORDS

Dielectric relaxation, ceramics, phase change, perturbation, frequency shift, Q-change.

OBJECTIVES

1. To understand the basic concepts of dielectric relaxation.
2. To study the process of dielectric measurement of strontium titanate and lead titanate zirconate ceramics at microwave frequencies.

SUMMARY

Dielectric measurements of strontium titanate and lead titanate zirconate ceramics are conducted at microwave frequencies using a cylindrical resonant cavity in the TE_{011} mode. The perturbations of the electric field are recorded in terms of the frequency shift and Q-changes of the cavity signal. Slater's perturbation equations are used to calculate ε' and ε'' of the dielectric constant as a function of temperature and frequency.

INTRODUCTION

A microwave resonant cavity has become a standard technique to study dielectric relaxation in a compound of interest. It is a definite improvement over the waveguide techniques in which the sample under investigation was placed in a hole in the waveguide itself to study dielectric behavior. In a resonant cavity the perturbations of the electric and magnetic fields are well defined depending on the structure of the cavity.

For this experiment a cylindrical resonant cavity in the TE_{011} mode was designed to study the dielectric behavior of strontium titanate ceramic at microwave frequency of 9.7 GHz at different temperatures. The dielectric behavior of lead titanate zirconate was studied as a function of frequency from 8.8 to 9.7 GHz. The strong perturbations of the electric field were recorded in terms of the frequency shifts and Q-changes at different temperatures. Using Slater's perturbation equations (Ref. 4), the real and imaginary parts of the complex permittivity were calculated at different temperatures. The relaxation times for these compounds were calculated using Debye's theory for polar molecules. Dielectric behavior of a mixture of barium and strontium titanates and lead titanate zirconate has been studied at different frequencies (Ref. 1-3). The resonant cavity technique has been very successfully used to study dielectric relaxation in a number of compounds of interest (Ref. 4-7).

EXPERIMENTAL PROCEDURE, THEORY AND RESULTS

The details of the microwave spectrometer used in this investigation operating in the x-band of frequencies were given in a number of papers (Ref. 4 and 7). The block diagram of such a spectrometer is shown in Figure 1. The signal was produced by a tunable klystron and transmitted through the waveguides to the resonant cavity where the material under investigation was placed into the cavity. The modulated signal as displayed on the oscilloscope was of the form of a butterfly. A part of the signal from the directional coupler was mixed with a standard frequency taken from an oscillator. A radio receiver was used to detect the change in the frequency of these signals in the form of two markers also displayed on the oscilloscope. These markers were used to find the positions of frequency shifts and Q-changes as the sample under investigation goes through a dielectric change. A true copy of the signal as taken from the oscilloscope is shown in Figure 2.

When a dipole is placed in an electric field $\vec{E}$, it experiences a torque given by

$$\vec{\tau} = \vec{p} \times \vec{E} \tag{1}$$

where **p** is the dipole moment vector. The dipole rotates in the electric field and stores potential energy given by

$$U = -\vec{p} \cdot \vec{E} \tag{2}$$

At lower frequencies, the dipole can follow the orientations of the applied field without any phase difference. At higher frequencies, there is a phase lag between the orientation of the field and the dipole and the dielectric constant becomes a function of frequency. As a matter of fact, the dielectric constant becomes a complex quantity

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \tag{3}$$

With ε' and ε" being the real and imaginary parts of the dielectric constant and ε" being necessarily the loss term for the dielectric constant. The value of the dielectric constant basically depends upon the number of dipoles that can follow the orientations of the applied field. At higher frequencies the number of dipoles following the orientations of the field goes down and hence a lower value for the dielectric constant. The ratio of ε' and ε" is expressed as a loss tangent as given by

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'} \tag{4}$$

ε' and ε" are further related to the frequency shifts and Q-changes as expressed in the Slater's perturbation equations (Ref. 4).

$$\frac{\Delta f}{f_o} = -\frac{\varepsilon'-1}{2} \frac{\int \vec{E}_s \cdot \vec{E}\, dv}{\int \vec{E} \cdot \vec{E}_a\, dV} \tag{5}$$

and

$$\Delta\left(\frac{1}{Q}\right) = \varepsilon'' \frac{\int \vec{E}_s \cdot \vec{E}\, dv}{\int \vec{E} \cdot \vec{E}_a\, dV} \tag{6}$$

where E is the field of the unperturbed cavity, E_a is the microwave field as applied to the cavity and E_s is the field of the sample itself, v and V are the volumes of the sample and cavity respectively.

The perturbations of the sample under investigation were studied by experimentally recording the frequency shifts and the width changes of the microwave resonant signal by placing a fixed length of the sample into the resonant cavity. The width change of the signal is directly related to the Q-change of the cavity. These changes in width and frequency of the signal were recorded at different temperatures as the substance under investigation goes through a phase change. The frequency of the signal was kept constant for one set of data taken from one temperature to the other for strontium titanate. Dielectric relaxation in lead titanate zirconate was studied as a function of frequency. Dry nitrogen gas at the liquid nitrogen temperature from a heat exchanger was allowed to circulate around the cavity containing the sample under investigation. A thermal equilibrium was maintained by controlling the amount of cold air circulating around the resonant cavity and this method is very useful in maintaining a certain temperature while the material under investigation goes through a phase change.

Figures 3 and 4 show the dielectric behavior of strontium titanate as a function of temperature at a microwave frequency of 9.7 GHz. Figure 3 shows the behavior of Q-change as a function of temperature. From Slater's equations, Q-change corresponds to the dissipation term (ε'') in the dielectric constant. This curve shows a peak around 20°C and after that there is a sharp drop in the dissipation factor. The frequency shift vs temperature behavior as displayed in Figure 4 shows a dramatic change in the real part of the dielectric constant around -57°C. The strontium titanate ceramic undergoes a phase change at this temperature and polarization of the material follows the orientation of the field. The relaxation times using Debye's theory are of the order of 10^{-12}- 10^{-14} sec.

The dielectric behavior of lead titanate zirconate as a function of frequency is displayed in Figures 5 and 6 with Figure 5 showing the Q-change vs applied frequency and Figure 6 shows the behavior of $\Delta f/f_o$ at different frequencies. The dissipation factor (ε'') as displayed in Figure 5 shows an increase as the frequency increases up to around 9.6 GHz and after that has a decreasing trend. The real part of the dielectric constant decreases as the frequency increases and then levels off around 9.6 GHz. This behavior is shown in Figure 6. The dielectric behavior of strontium titanate and lead titanate zirconate ceramics has been studied very successfully by using the microwave resonant cavity as a probe. This method will also be applied to study the mass dependence of these materials at different microwave frequencies and the free energy of activation calculated at these frequencies.

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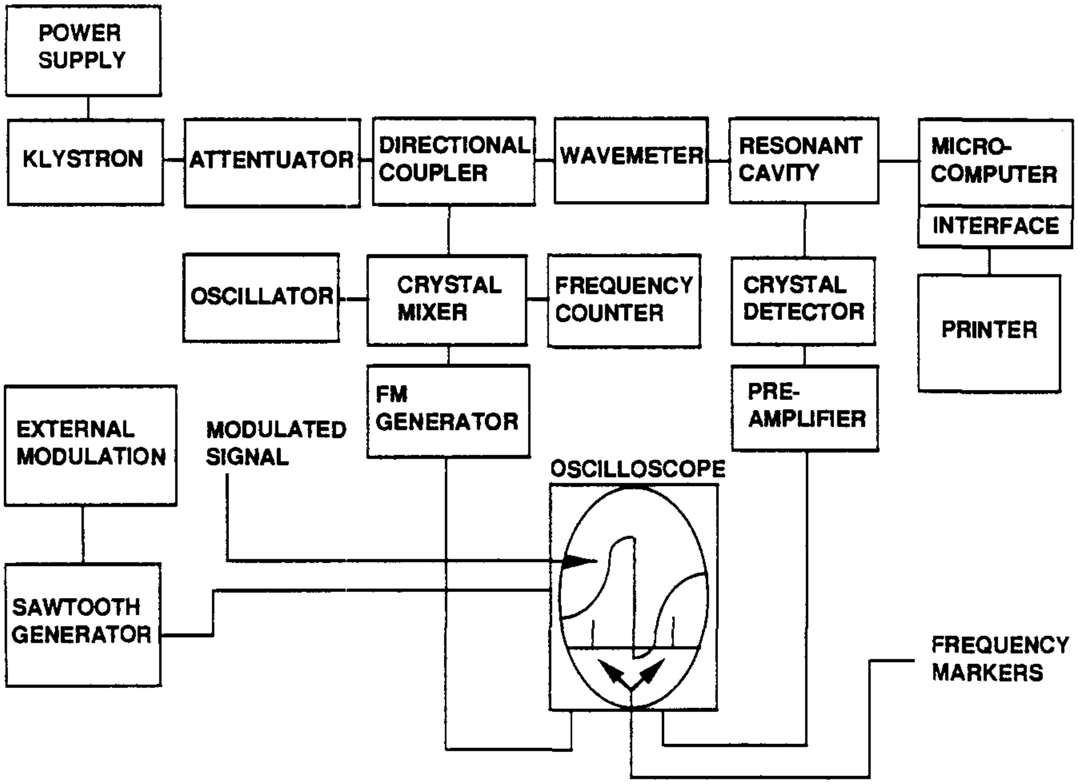
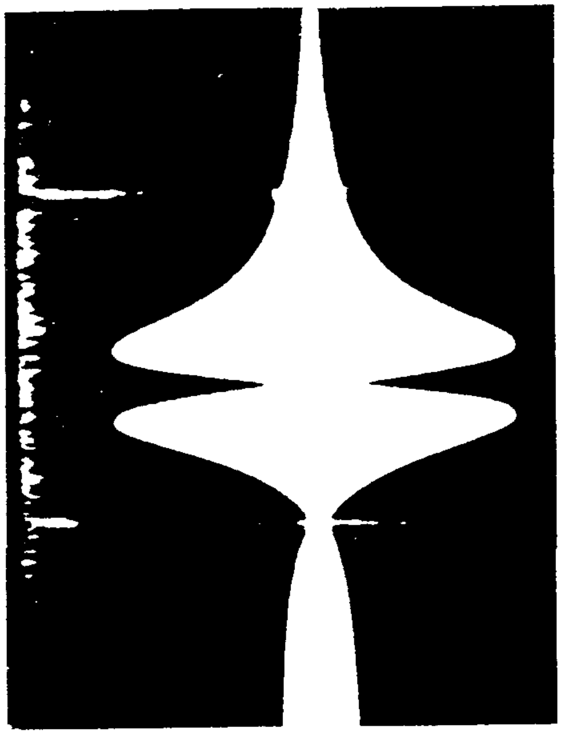
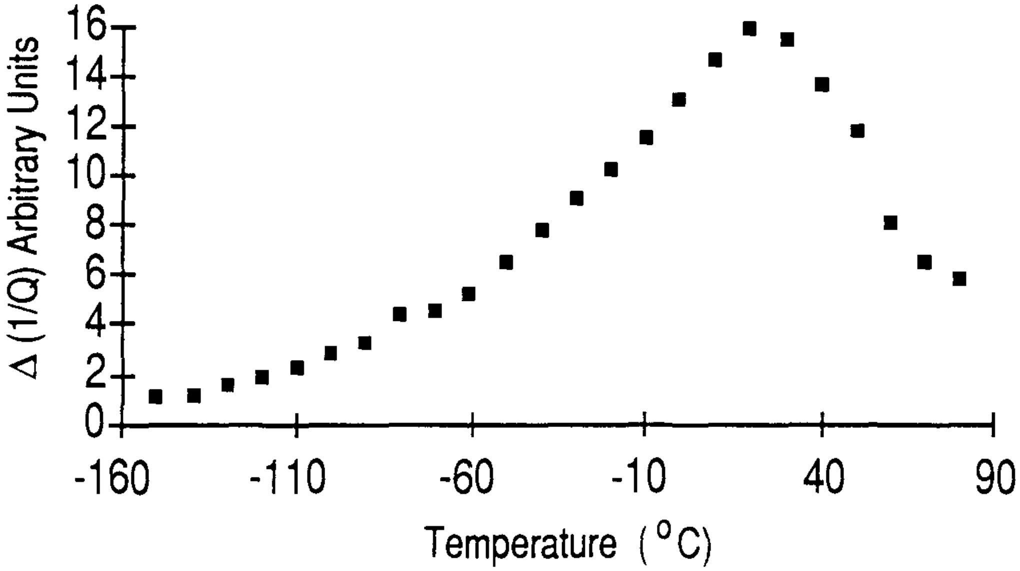


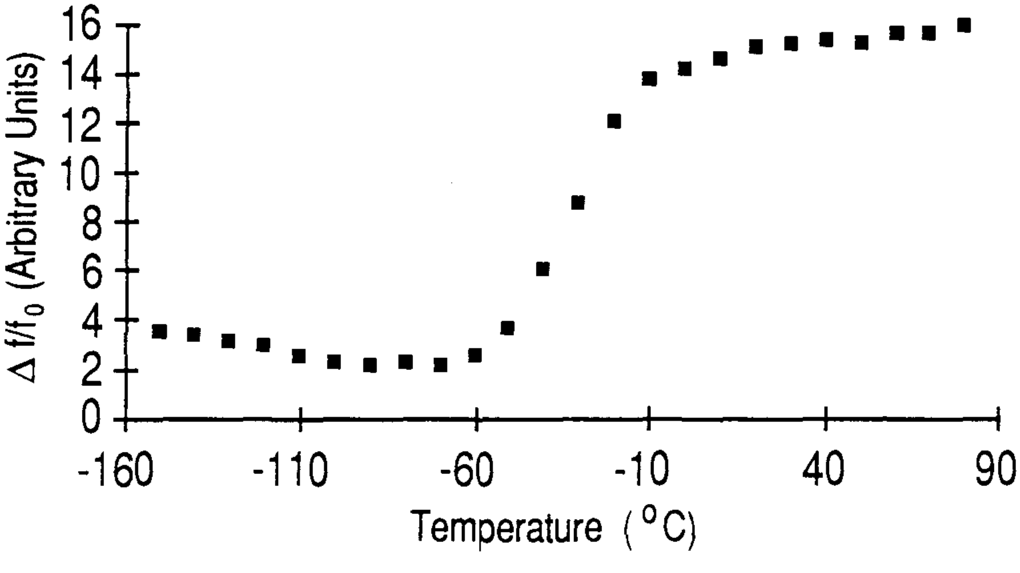
1. Block diagram of the microwave spectrometer used in this investigation.



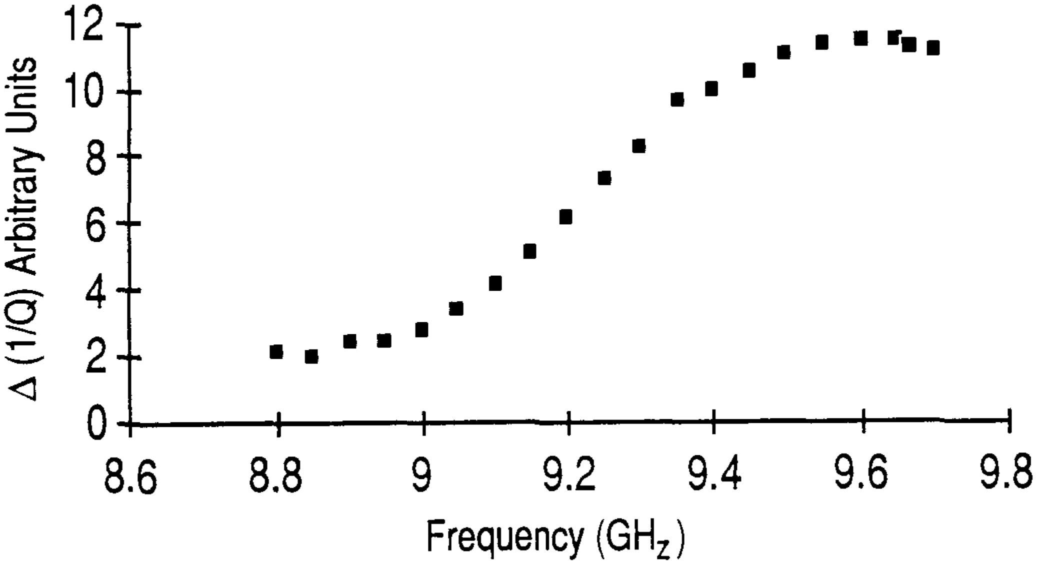
2. Dual trace oscilloscope display for frequency markers and cavity derivative profile for a typical scan. (The marker interval is always twice the radio receiver reading and in this scan the spacing is 8 MHz, with center frequency at 9.7 GHz.)



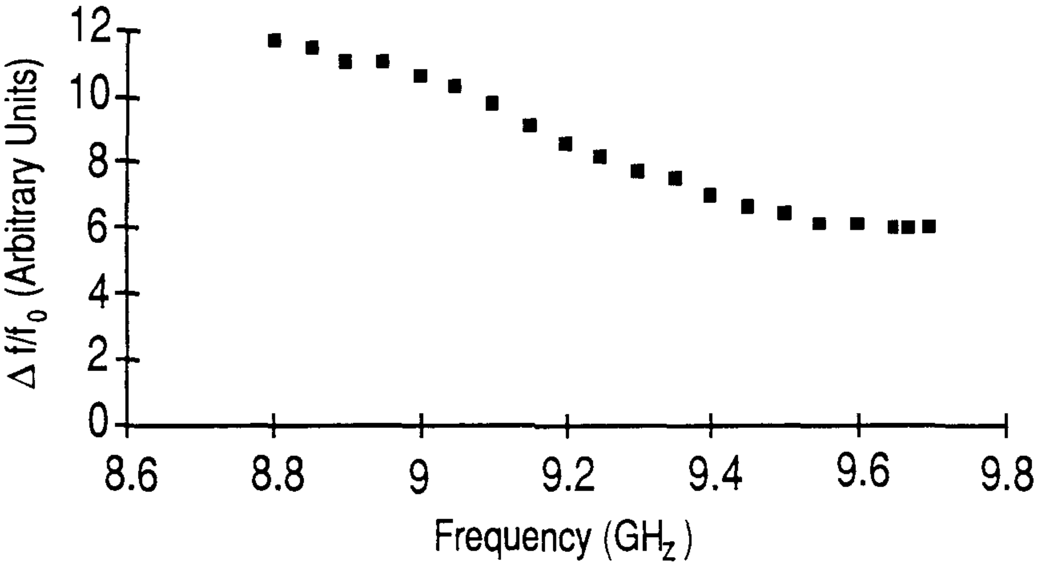
3. Microwave loss Δ(1/Q) as a function of temperature for a sample of strontium titanate.



4. Frequency shift $\Delta f/f_0$ as a function of temperature for a sample of strontium titanate.



5. Microwave loss Δ(1/Q) as a function of frequency for a sample of lead titanate zirconate.



6. Frequency shift $\Delta f/f_0$ as a function of frequency for a sample of lead titanate zirconate.

UNIVERSITY OUTREACH FOCUSED DISCUSSION: WHAT DO EDUCATORS WANT FROM ASM INTERNATIONAL

Thomas Passek

Assistant Director Chapter Relations
ASM International
Materials Park, Ohio 44073

Telephone 216-338-5151

Brainstorming

17 November 1993

Dear National Educators Workshop '93 Attendees:

Subject: **Summary of information gathered during our Focused Discussion on** ***"How ASM could better serve educators?"***

At the end of the day on Thursday I conducted a focused discussion on "How ASM International could better serve the Educational Community." I was asked at the end of this session if I would share with the participants the results of this discussion, and so I am.

With only 30 minutes to discuss several issues the group provided me with 10 flip-chart pages of ideas and comments which can be seen on the attachment. No editing was done to the comments so the power of the scribe prevails. If there is anything on here that your group provided that you feel needs further clarification, please call me at 1-800-336-5152. This information is being entered into our "Voice of Member Database" which will be used by ASM to be more responsive to our member's needs. If you have any additional suggestions, comments or complaints please use the enclosed form to let us know how we could improve our service to you.

ASM has a wealth of information to share with you and your students. Please let us know how we could make it more useful, convenient and customized for your needs.

Sincerely,

ASM International®

Thomas S Passek

Thomas S. Passek
Assistant Director,
Chapter Relations & Development

xc: Edward L. Langer , Managing Director
Melinda Carter, Student Chapter Relations

Enclosure

NATIONAL EDUCATORS WORKSHOP FOCUS GROUP

Background information:

- 4 November 1993, Norfolk ,VA.
- Group size (40-50)
- Make-up (primarily educators from universities/colleges and technical schools)
- Meeting format (30 minutes in duration)
  - (broke them up into 4 groups)
  - (each group had their own scribe)
    - NO editing of comments
- Conducted by Thom Passek

Question : What does ASM provide that is of value to educators and how could it be improved?

* Mat. DB - make it less expensive
  Mac version
* Education Yearbook - good
* Metals Handbook- is a classic
* Coupons/discounts are good
  - overall pricing for education could be improved and better publicized
* Joint TMS/ASM membership - good
* Home study courses - good

Question : What should ASM be providing for educators that it is not?

* Demonstration materials - at no charge
* Videotapes - reasonable cost to college/Univ./CC/TI
  lower price on tapes
* More information on "Careers in field of materials"
*ANSI-ISO-ASTM : Cross reference; cost too high
*Materials Dictionary
*Student access to DB - via videotapes at the chapter level.
* resource sharing among technical societies
* Recognition of students accomplishments in publications. - TEAM AWARDS
* Local combined meetings between societies (joint, local, regional)
* Regional student conferences
* Charge less money for student stuff
* An inexpensive journal for ONLY student papers
* Description of Mat'ls experiments
* Database of Materials prices
* Provide some FREE workshops
* Get programs sponsored by companies (or agencies)
* Answer to "How to GET information on the kits for K-12 & up -
  called and NO follow-up

Question : I have heard a lot of interest in ASM providing video for use by educators. What type of videos would you like to see - what processes, how long, what type of pricing, support materials? In what form should they be offered ?

* Tutorial videos
* Video plant tours (40-50 minutes)
* Processing with close-ups (20-25 minutes)
* Basic principles
*Testing (with -in industrial setting , and/or not)
* Maximum price - $ 35
* Instructor guides to accompany
* Length 5-10 minute segments with stopping points for discussion. ACTION ORIENTED (from industry)
* 3 dimensional models
    frenel lenses type pictures
    ??? Notebook of crystals 2D to 3D ??? (I'm not sure this was video related vs. a visual tool)
* Demonstrations - experiments
* Library - nominal fees
* Provide text

Question : What 3 changes would you like to see made at ASM ?

* More responsive to requests
* More affordable
* Articulate mission - (Re-examine)
    - relation to other societies
* More hands-on demo kits at ALL levels
* Reduce mailings, i.e., redundancy, junk mailing
* Outreach to elementary
    - Interactive video programs
    - Public Relations
* Clean, low cost videotapes for recruitment to materials sciences
    - non-imposing
    - show relationships to industry

ASM INTERNATIONAL
The Materials Information Society

FACT SHEET

ASM STUDENT OUTREACH PROGRAM (SOP)

SOP FACTS:
Since 1952 , ASM Foundation for Education and Research and ASM Chapters have initiated and maintained outreach activities. ASM's Student Outreach Program mission is to encourage the development of capable well-educated materials specialists. Today, the program plans to address the decreasing interest and literacy among pre-college students in science and mathematics. The aim is also to improve student academic preparation. Programming has been developed to meet the challenges in three levels : k-12th grade, undergraduate and graduate. Currently, the Student Outreach Program provides career awareness products/services, resources, scholarship, awards competitions, school partnership assistance and classroom visitation support.

ASM SOP serves as a resource to groups and individuals interested in
1. Supporting and improving science and math education
2. Increasing career awareness opportunities (engineering and materials science)
3. Improving academic preparations for students to enter the work world (from K-12th grade through college levels)

ASM MISSION - To encourage the development of capable, well-educated materials specialists.

CURRENT FOCUS
To address decreasing interest and literacy
in science and math among pre-college students

WHAT ASM OFFERS MEMBERS AND EDUCATION ADVOCATES...

Partnership Support
- Structure
- Training
- Tools
- Facilitation

Program Development Support
- Scholarships
- Student and young member nights
- Plant tours
- Career talks
- Technical programs (clinics/science workshops)
- International Student Exchange

NOTE: Local chapters have initiated and maintained these and other programs and will share their information

Resource Materials
- LEADS list (List of Educational Aids and Direct Sources)
- Career brochures, kits, videos and experiments
- Partnership Guide
- Partnership training materials

Communication Networks
- ASM News (article space)
- Chapter Network News (announcements)
- Mailing lists (partnership coordinating organizations, peer societies, schools)
- Contacts for national education movements, curriculum development, forming partnerships
- Referrals/contacts for targeted programs (merit badge clinics, science fair judging, technical fairs)

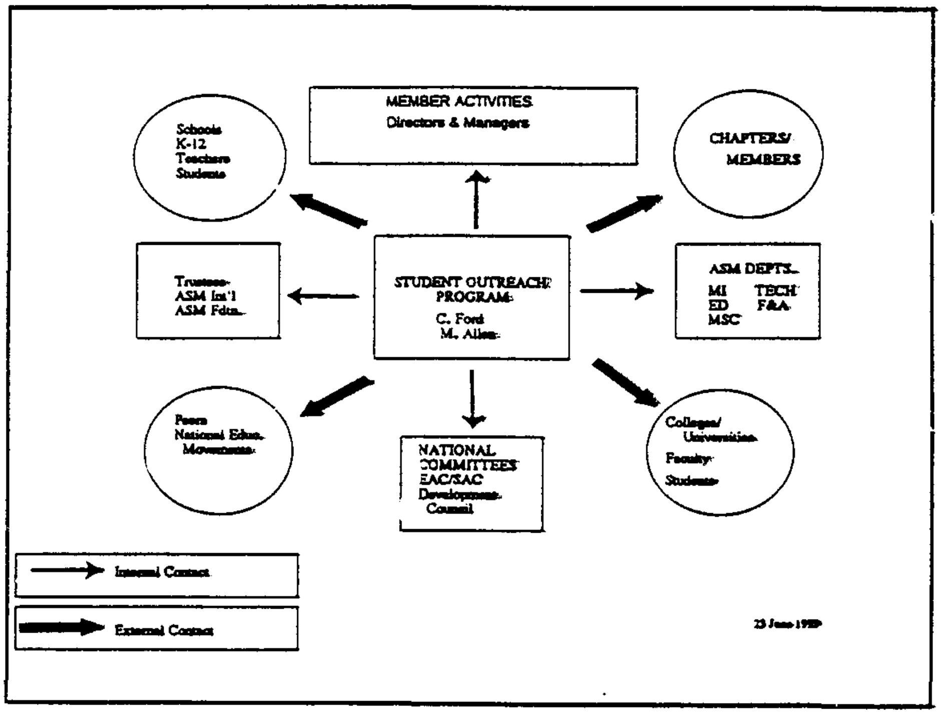
National Committee Involvement
- Scholarship award administration
- Student Chapter award administration
- New program research and development to better serve students

STUDENT OUTREACH PROGRAM WILL BENEFIT YOU BY...
1. Increasing your access to resources
2. Broadening your outlook and expose you to opportunities
3. Providing contacts for new program ideas and implementation

"For the first time, we are being treated as professionals. It's great!"
Brentwood Elementary School Teachers
Los Angeles, California

This is not to help me, it is to help society, today and tomorrow. If we don't do it now, we may never have the opportunity..
Mat Wisbrock, Chapter Volunteer

Student Outreach Program will serve as an Information Exchange Network



To learn more about chapter activities nearest you, contact
ASM INTERNATIONAL
STUDENT OUTREACH PROGRAM
MATERIALS PARK, OHIO 44073-0002
(216) 338-5151

Mr. Farrell,
Thank you for coming and showing us your hydraulic demonstration. When I get older I want to be an engineer or an architect. I think I'll probably be an engineer after I saw how much fun it is.
Your friend,
Robbie
Robbie Kaspa
2nd Period

Thank you for all the hard work you've done to help me (and my colleagues) rise to the challenges of public education. Knowing that other professions are willing and eager to help us meet the needs of our students renews my enthusiasm for teaching!
Sincerely,
Martha Rogers
Martha Rogers and
John White
Thanks!
John White

Mr. Farrell,
Thanks for coming and speaking to our class. It was very interesting and a great experience to lift three hundred pounds! I really enjoyed your hydraulics demonstration. Thanks again for showing us simple things that can affect our everyday life...
Sincerely,
Abby Henry
Abby Henry
8th Period

ASM Books for Class Instruction

ASM provides a 50% discount to University/College Professors/Instructors and to University/College Bookstores for books adopted as reference or course texts. The following purchase options are available:

> * Professors /Instructors may order directly from ASM at the 50% discount rate and pass on this savings directly to their students.
>
> * Students may purchase directly from ASM; their order must provide verification of the university course, instructor's name, date of course.
>
> * University /College Bookstores will be allowed a discount of 50% off list price on ASM publications. These books are for resale to University/College students. (In order to ensure that the student discount is applied to the bookstore orders, it would be helpful if the Professor/Instructor includes the student discount at the time of order)

If you have any questions please contact Joan Bellian at ASM International - 216-338-5151 (460).

A partial listing of universities and colleges that have adopted ASM books for class instruction :

Institution	**Book**
Univ. of California -Davis	Vol. 11, F/A Vol. 12, Fractography
Wright State Univ. (OH)	Vol. 11, F/A
U.S. Naval Academy	" "
Lafayette College	" "
Duke Univ.	" "
Oakland Univ.	Vol. 18, Friction, Lubrication ...
Montclam Community College	Plastic Part Technology
W. Carolina Univ.	" "
Univ. of Wisconsin	Vol. 4, Heat Treating
San Jose State	Vol.1, Packaging and Vol. 17, NDE
Wayne State Univ.	Steels
Univ. of New Haven	Steels
Colorado School of Mines	" "
Columbia Univ.	" "
Washington State Univ.	Engrg. Plastics, Vol.2
California State Univ. - Long Beach	Practical Heat Treating
Lehigh Univ.	Metals Handbook; Desk Edition
Univ. of Michigan	Metals Engineering: A Tech. Guide
Univ. of Michigan - Dearborn	" "
Univ. of Alabama - Huntsville	Vol. 17, NDE
Univ. of Nebraska - Lincoln	Materials Science & Engineering
	Ceramics & Glasses, Vol. 4
DeAnza College	" "
Truckee Metal Community College	Inspection of Metals

The Organization

MATERIALS INFORMATION (MI) is a service operated jointly by the Institute of Materials (incorporating the Institute of Materials, The Institute of Ceramics and The Plastics and Rubber Institute) and by ASM International® (formerly the American Society for Metals). The core of MI's work is the monitoring, collecting and indexing of the world's materials literature to produce abstracts journals and bibliographic databases. MI also offers a document deliver service, a translations service and a range of other publications surveying and analyzing the international literature in engineering materials.

Much of MI's activity centers around the world's technical journals: more than 2,500 journal titles are acquired by purchase, donation or exchange. Coverage of conferences, patents, books, reports and other types of documents is also part of the service. MI casts its net worldwide, sourcing materials in more than thirty languages.

Abstracts Journals

Metals Abstracts (together with **Metals Abstracts Index** and **Alloys Index**) is still MI's largest publication, covering all aspects of metals science and technology through from ore preparation, extraction and smelting to the finished products, their properties and applications. Steelmaking and nonferrous alloy production are major areas of coverage and the abstracts journal also includes all aspects of foundry technology and heat treatment, working, machining, joining and finishing. Powder technology and metal matrix composites are also covered and the scope extends to related areas of physics and chemistry. Each monthly issue contains some 4,500 items, giving full bibliographic details (titles, dates, authors, journal references, conference information, publishers, etc.) in addition to the abstract itself which summarizes the contents of the paper.

Engineered Materials Abstracts, Metals Abstracts' sister publication, covers the technical literature on polymers, ceramics and composites in engineering applications. Most areas of light and heavy engineering are covered, including such applications as sports equipment and domestic appliances. Coverage focuses on structural materials (in the very broadest sense of the word), but important non-structural engineered materials are also included - for example optical fibers, nuclear fuel rods, piezoelectric materials and superconducting ceramics.

Bibliographic Databases

Fast access to all of the above information can be obtained through MI's bibliographic databases. These are, in effect, vast electronic libraries and they now contain information on well over a million documents. **METADEX** is the database equivalent of Metals Abstracts and while **EMA** is the equivalent of Engineered Materials Abstracts. A bibliographically-based numeric properties databank **Metals Datafile** - is also available. The databases can be accessed in several different ways:

1. MI's **Search Service** carries out custom searches on demand and also provides a regular updating service. For ASM student members, we charge only $20 for this service - as opposed to $130 for regular members.

2. The databases can be interrogated *online* from almost any home or office computer; all that is required is a modem and software to make the connection via the phone lines to a remote host computer. Powerful retrieval capabilities mean that it only takes a few seconds to search through nearly thirty years of international literature for appropriate subject keywords or other data such as authors' names and journal or conference references.

3. All of the published material collected and indexed by MI since 1985 (with the exception of the numeric information in Metals Datafile) is now also searchable on *compact disc*. This is the **DIALOG OnDisc METADEX/Materials Collection**, produced in collaboration with DIALOG Information Services, Inc. The discs are updated quarterly and the easy-to-use search software offers even more power and flexibility than online searching. The CD-ROM has been very popular with the academic commmunity, particularly in Europe. Universities subscribing in the United States include: Lehigh University, Massachusetts Institute of Technology, Carnegie Mellon University, the University of Cincinnati, and others. Materials Information encourages students at these institutions to make use of this resource through on-site training seminars.

The education of tomorrow's materials scientists and metallurgists in the area of information resources is a key component of Materials Information's vision for the future. We welcome any comments or suggestions you may have in this area.

Instructional Videos for Educational Institutions and Students

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Prices subsized
exclusively for the
educational community!

Videos can:

- **show students materials processes occurring within industry.**
- **can be a flexible way to teach; customize your own course based on individual tapes.**
- **maximize effectiveness of educational programming with video support.**

All Video Tapes $39.95 each.
Corresponding Lesson Material (where available) $3.00 each.

HEAT TREATING — 39 TAPES AVAILABLE

Practical Heat Treating

- **Designed for furnace operators as well as heat treaters and supervisors.**
- **Extensive on-location footage of actual heat treating operations.**
- **The most comprehensive video training series available today aimed at all heat treating operations personnel.**
- **Troubleshooting information describes typical problems, their causes and ways to avoid costly errors and rework.**
- **Addresses major quality control and testing issues as they affect daily heat treating operations.**
- **Provides extensive coverage of all major metals categories, and heat treating equipment considerations.**

Topics covered include:

Tape 1 — Heat Treatment of Carbon Steels: Iron-cementite diagrams □ introduction to structure of metals □ amount of carbon present in steel □ austenite formation

Tape 2 — Heat Treatment of Alloy Steels: Amount and effects of alloying elements present □ effect of alloying on time at hardening temperatures □ effect of alloying on quenching time

Tape 3 — Heat Treatment of Tool Steels: Definition □ carbon contents and major alloying elements □ AISI classification □ water-, air-, oil-hardening types, as well as shock-resisting and high-speed steels austenitizing temperatures

Tape 4 — Heat Treatment of Cast Irons: Definition □ major classes □ graphite and carbon comparisons □ cast iron treatments

Tape 5 — Heat Treatment of Stainless Steels: Definition □ annealing and hardening □ protection during heating □ quenching, tempering and stress relief

Tape 6 — Heat Treatment of Aluminum: Alloy designations include: wrought and cast alloys and tempers □ strengthening mechanisms □ heat treating processes include - annealing, stress relief, solution treating, quenching, straightening, aging and testing

Tape 7 — Heat Treatment of Other Nonferrous Alloys: Titanium alloys includes - commerical alloys: hardening mechanisms; heat treating times and temperatures related to stress relief, annealing, solution treating and aging; cleaning and atmospheres: potential problems

Tape 8 — Types of Furnaces and Associated Equipment — Classification of furnaces includes - energy source, temperature ranges, operation methods and application of heat □ types of ovens and furnaces includes - box, pit, car bottom, elevator, integral quench, bell, gantry, rotary, roller hearth, shaker, salt bath, belt conveyor, vacuum, fluidized bed, pusher, plasma and continuous

Tape 9 — Furnace Atmospheres: Definition □ use of furnace atmospheres includes - protection from detrimental elements, heat transfer and supply of alloy elements

Tape 10 — Instrumentation and Controls: Temperature sensing variables include - mechanical, visual, thermocouples, optical comparators and radiation □ atmosphere sensing variables include - visual, chemical, physical and electrical

Tape 11 — Equipment Operation and Maintenance: Equipment design □ manual operation categories include - specifications, start-up instructions, use, shut-down and emergency procedures

Tape 12 — Surface Treatments: Reasons for surface treatments □ work hardening includes - shot peening, coining and burnishing □ metal/alloy build-up on the surface includes - braze or weld build-up and plating

Tape 13 — Testing and Quality Control of Metals and Alloys: Properties affected by heat treating includes - physical and mechanical properties, microstructure and chemistry changes, and hardness testing, tensile testing and other mechanical tests

Tape 14 — Problems Associated with Heat Treated Parts: Major causes of properties not being achieved includes - design, material selection, improper manufacturing steps and improper heat treating process

Tape 15 — Vacuum Heat Treating Processes: Vacuum heat treating processes □ vacuum fundamentals □ partial pressure □ pumping systems

Narrator Presentation, 2D & 3D Animated Graphics, Photos, Parts Demonstrations, Video Clips, Graphs & Word Charts, VISUALS: 85-90%

Induction Heating

- **Specific applications where induction melting offers unique advantages**
- **How induction simplifies or improves dozens of processes — from high tech semiconductor processing to sealing toothpaste tubes**
- **How the over-all cost of using induction equipment can be lower than with competitive methods**

Basic principles of induction heating are presented, along with methods of selecting frequency, equipment and power. Typical applications are also described, including heat treating, preheating, melting and metal joining. Topics covered include:

Tape 1 — Introduction: Induction heating basics - eddy currents, reference depth, power needs, circuits and load matching

Tape 2 — Power Supplies/Equipment Selection: Different types of fixed and variat frequency power sources that include line frequency, frequency multipliers, motor-generator sets, solid-state, and radio frequency

Tape 3 — Coil Design and Selection — Part I: Equations for calculating voltage, power, and other important parameters

Tape 4 — Coil Design and Selection — Part II: High-frequency coil design covers coil materials and geometries □ load matching □ important parameters

Tape 5 — Preheating Prior to Hot Working: Static, multistage, and in-line heating □ steady-state and transient heat transfer □ calculations for billet heat, power density, heat and cycle times, power loss from radiation, and coil line

Tape 6 — Induction Heat Treatment: Using induction to harden, temper, normalize, anneal, and stress relieve □ selective heat treating applications

Tape 7 — Induction Brazing and Soldering: How coil parameters influence heating pattern □ tubular and soft copper inductors □ coils for conveyorized operations

Tape 8 — Induction and High Frequency Welding: Speed is a major advantage □ skin, proximity, and contact effects in high frequency welding □ high frequency induction versus high frequency resistance welding

Tape 9 — Induction Melting: History and advantages □ differences between and advantages of coreless and channel furnaces □ refractory wear (erosion) and buildup

Tape 10 — Part Handling Equipment: Factors affecting the design of induction heating systems □ handling systems for slab, bar, billet, and bar end heating

Tape 11 — System Control and Factory Automation: Programmable logic controller □ solid-state power supplies □ energy monitors for critical applications

Tape 12 — Special Applications: Magnetic particle welding □ food and drug packaging □ semiconductor processing and assembly

Tape 13 — Safety and Economics: Advantages over competitive equipments and techniques □ energy costs □ efficiency □ frequency □ power □ cooling water

Lecture, RP Kodalith Full Screen Words, RP Graphs and Line Drawings, Several Photos, VISUALS: 90%

Heat Treatment: Metallurgy & Applications

- **How to efficiently heat treat steels — carbon, alloy, stainless and tool**
- **Properties of carbon and alloy steels produced by solid state changes**
- **How to accurately predict the effects of temperature, time and composition on structure and properties when heat treating**

A comprehensive, authoritative course that emphasizes the fundamentals of heat treating cast irons, and carbon, alloy, stainless and tool steels. It examines difficult procedures such as carburizing, carbonitriding and nitriding. Topics covered include:

Tape 1 — Physical Metallurgy: Structure and Properties: Physical makeup and dissimilarities between metals and nonmetals □ Versatility of metals in strength, hardness, ductility, toughness and malleability, plus magnetic and electrical properties

Tape 2 — Microstructures of Steels: Ferrite, Pearlite and Bainite: Structure-property relationship in steel □ Carbon and alloying elements in heat treatment □ Development of iron-iron carbide diagram

Tape 3 — Isothermal and Continuous Cooling Transformation Diagrams: Use of isothermal and continuous cooling transformation diagrams to set up heat treating procedures □ Formation of martensite - plate and lath

Tape 4 — Annealing and Normalizing of Steel: Uses of heat treatment in controlling size, shape and distribution of carbide phase in ferrite to produce pearlite or spheroidite

Tape 5 — Hardening and Hardenability: Development of the Jominy End Quench Hardenability Test and its use in predicting heat treating hardening response of various section sizes □ Study of various quenching media

Tape 6 — Tempering of Steel: Methods of obtaining a combination of strength, hardness, ductility and toughness in steel to ensure product reliability □ Effects of controlling time and temperature on various alloys

Tape 7 — Surface Hardening: Surface heating without changes in surface chemistry □ Induction heating □ Flame hardening transformation to austenite and quenching to martensite

Tape 8 — Heat Treatment of Cast Iron: Similarities and differences between steel and cast iron □ Cast iron classifications - gray, white, malleable and ductile

Tape 9 — Heat Treatment of Tool Steels: AISI classifications of tool steel - water ha ing, cold work, shock resisting, air hardening, hot work, high speed and special pu.

Tape 10 — Constitution and Heat Treatment of Stainless Steels: Effect of chron on stainless behavior □ Differences between martensitic, ferritic and austenitic

Tape 11 — Limitations of Heat Treatment: Designs to minimize cracking and distortion □ Types of cracking during heat treatment, including causes and prevention

Lecture, Line Drawings and Diagrams from Text, Several Photos, Microstructures from Text, VISUALS: 80%

METALLURGY/SUPERALLOYS

Metallurgy for the Non-Metallurgist

15 TAPE SERIES

- **What metals are and how they are put into usable form**
- **Which factors influence the materials selection process**
- **How tensile, hardness, impact, and other tests are performed and what their results can tell us**

To answer the question, "What is a metal?", there are sections devoted to the crystal structure of metals and why most metals are "hardenable"; to material properties and their relationship to microstructure; and to the reasons for alloying of metals to achieve specific properties. Topics covered include:

Tape 1 — Metals: A History. Defines metals and alloys □ discusses metallic properties, metal crystals, isotropy, ores □ early use and discover of metals

Tape 2 — Materials Characterization and the Selection Process. Factors that influence materials selection, including - structure, mechanical properties, elasticity, grain size, and the effects of temperature □ economic, functional and production requirements

Tape 3 — Mechanical Properties and Their Measurement. The stress-strain curve □ tensile tests, hardness tests, impact tests □ understanding test results

Tape 4 — Solidification of Metals. Cooling curves and phase equilibrium diagrams □ dendritic segregation, solidification and purification methods, eutectic compositions, the Lever Rule, advances in solidification

Tape 5 — Steels and Cast Irons: Applications and Metallurgy. History of steelmaking □ the role of carbon and oxygen □ furnaces, melting, alloying, and casting techniques

Tape 6 — Heat Treatment of Steel. Transformations in response to changing temperature □ quenching and tempering □ the TTT diagram □ Jominy hardenability

Tape 7 — Case Hardening of Steel. Mechanical, thermal, and chemical treatments used to increase hardness □ the effects of time, temperature, structures, grain size, and quenching □ depth of penetration

Tape 8 — Nonferrous Metals: Industrial Applications and Properties. How nonferrous metals such as aluminum, beryllium, magnesium, titanium, copper, lead, tin, zinc are extracted and made into a usable form □ material properties □ refining techniques

Tape 9 — Extractive Metallurgy. Techniques used to win metals from ores □ flotation, drying, roasting, matte smelting, converting, fire and electrolytic refining □ high-melting-temperature metals, like tungsten and molybdenum

Tape 10 — Getting Metals into Usable Form: Hot Working. Amorphous casting □ continuous casting □ casting into ingot molds □ sand casting □ lost wax method

Tape 11 — Getting Metals into Usable Form: Cold Working. How cold working increases a materal's strength □ role and behavior of grains (crystals) in cold working □ cold rolling, cold forming, extrusion, drawing, swaging, and machining

Tape 12 — Strengthening Mechanisms: Nonferrous Metals. Techniques used to harden nonferrous metals, including age hardening and strain hardening □ diffusion mechanism theories

Tape 13 — Joining. Soldering, brazing and fusion welding techniques, and equipment □ what makes a good weld □ effects of heating and cooling

Tape 14 — Corrosion and Corrosion Prevention. Non-corroding materials □ corrosion mechanisms and requirements □ types of corrosion - wet, dry, and galvanic

Tape 15 — Quality Control and Failure Analysis. Statistical sampling and evaluation □ dye-penetration □ radiography □ magnetic-particle inspection □ accoustic emission

Lecture, Full Screen Words with Pointer, Line Drawings on Poster, VISUALS: Less than 5%

Superalloys

4 TAPE SERIES

- **Understand why nickel, iron-nickel, and cobalt-base superalloys have high strength at high temperatures**
- **Understand specific goals of heat treating applicable to superalloys and how the goals are achieved**
- **Be familiar with modern processing techniques that impart desirable properties to superalloys**

The advent of the aircraft turbine engine gave great impetus to high-temperature materials. The concept of the jet engine dictated the need for new engineered materials that would combine high-temperature and strength with resistance to elevated temperature corrosion. The use of superalloys has spread beyond aircraft engines as other industries recognized their high-temperature advantages. The turbine engine remains the cornerstone of superalloy use.

Tape 1A — Physical Metallurgy, Heat Treatment and Processing. (Part 1) Focusing on physical metallurgy of nickel, iron-nickel, and cobalt-base superalloys. Gamma prime and gamma double prime precipitates, coherence, and lattice mismatch are examined as they relate to superalloy mechanical properties.

Tape 1B — Physical Metallurgy, Heat Treatment and Processing. (Part 2) Covers goals of heat treating and how they are achieved with superalloys, directional solidification, single-crystal solidification, powder metallurgy, and redundant working of ingots.

Tape 2 — Deformation and Creep. Discusses relationships between superalloy structures, strength and creep; mismatch, fault energy, precipitate size, volume fraction, flow stress, antiphase bounding energy, looping, shearing, temperature, diffusion, rupture, orientation, and morphology.

Tape 3 — Fatigue. Discusses low cycle fatigue and fatigue crack propagation; the economic implications of fatigue and fracture; relationship between cyclic life and plastic strain range; roles of slip bands; grain size, and temperature; the interative fatigue law; and parameters affecting rate of fatigue crack propagation.

Lecture, Full Screen Words CG, Photos from Texts, Kodalith Line Drawings (overhead), Microstructures (overhead), Chalkboard, VISUALS: 50-60%. This series does not have corresponding lesson material available.

METALLOGRAPHY

Principles of Metallography

14 TAPE SERIES

- **How to evaluate microstructures through demonstrations using graphics and over 200 actual micrographs**
- **Basic principles of metallography; history, definitions, preparation steps and equipment**
- **Helpful techniques for preparing specific metals and alloys for metallographic inspection supported by excellent step-by-step demenstrations**

Learn "how to" prepare specimens for viewing with a metallograph, and making photomicrographs for permanent records. Incorporated in the tapes are step-by-step demonstrations in the processing of a specimen. Starting with selection and cutting of the specimen and continuing with mounting, grinding, polishing and etching. Topics covered include:

Tape 1 — Metallography: Its History and Aims. Defines basic terminology used in metallography; reviews Henry Clifton Sorbys' pioneering work in metallography; examines meaningful features of meteorite and Samurai sword microstructures

Tape 2 — The States of Matter. Attractive forces between atoms, types of bonding, properties of metallic bonding, unit cells, lattices, valence numbers, grains, and grain boundaries

Tape 3 — Introduction to Phase Diagrams. Defines terminology; discusses important details of phase diagrams, the lever rule and inverse lever rule; relates numerous specific phase diagrams with specific microstructures

Tape 4 — Microstructure and Classifications of Steels. Details single phase constituents and their microstructures; two-phase constituents and their microstructures; isothermal transformation diagrams; and continuous cooling transformation

Tape 5a — Metallographic Specimen Preparation I. Covers cutoff and sectioning techniques and equipment; mounting techniques and equipment; and the effect of particle size (grit) and pressure on metal removal rate

Tape 5b — Metallographic Specimen Preparation II. Grinding steps, cooling fluids, and patterns; rough and fine polishing; polishing cloths, abrasives, and fluids; diamond and other polishing compounds; electropolishing and electroetching; chemical etching

Tape 6 — Fundamentals of Metallographs and Photomicrography. Components of light microscopes; image contrast, resolution, and depth of field; numerical aperture; wavelength of light source; examination modes; image recording; and requirements of successful photomicrography

Tape 7a — Quantitative Metallography I. Metrology and stereology; what to consider before starting tests; guidelines for measuring structural gradients; the importance of randomness; how other tests can complement visual tests; statistical analysis; and definition of standard symbols and calculations

Tape 7b — Quantitative Metallography II. Quantitative techniques of analyzing microstructures; grain size measurement parameters; methods of measuring grain size; and measuring particle spacing and shape

Tape 8a — Auxiliary and Special Metallographic Techniques I. Brinell, Vickers and Rockwell hardess testing; Knoop and Vickers microhardness testing; relationship between error and indentation size; applications; hot stage microscopy

Tape 8b — Auxiliary and Special Metallographic Techniques II. General characteristics of electron microscopy equipment; specific characteristics, features, and capabilities of scanning, transmission, and scanning transmission electron microscopes and electron microprobe analysis; energy dispersive spectroscopy; wavelength dispersive spectroscopy

Tape 9a — Plastic Deformation and Annealing of Metals I. Effects of plastic deformation on microstructure; alignment of grains and inclusions; annealing; recrystallization; definition of cold working and hot working

Tape 9b — Plastic Deformation and Annealing of Metals II. How dislocations effect mechanical properties; viewing dislocations through the transmission electron microscope; strengthening through cold work; recystallization and alloying

Tape 10 — Metallographic Interpretation. Individual preparation techniques for cast irons, steels, aluminum, cobalt, copper, lead, nickel, titanium, refractory metals, precious metals, and others. Effects of heating, cooling, alloying, etching, different lighting techniques, and phase transformation on microstructure

Lecture, RP Micrographs, Video Clips Equipment, RP Photos, Micrographs from Text, Demo Samples, VISUALS: 45-55%

CORROSION

Corrosion

15 TAPE SERIES

- **How to solve corrosion problems and prevent them**
- **The important characteristics of metals and solutions that influence corrosion**
- **How a corrosion reaction takes place and some of the variables that affect corrosion**

Topics covered include:

Tape 1 — Introduction to Corrosion. Definitions; four ways of classifying corrosion; costs associated with corrosion; ways to solve corrosion problems; ways to prevent corrosion
Tape 2 — Basic Concepts in Corrosion. Active, immune, and passive corrosion behavior; how corrosion is affected by a metal's metallurgy, inherent reactivity, and tendency to form insoluble corrosion products; how a solution's characteristics affect corrosion
Tape 3 — Thermodynamics: Potential pH Diagrams. Changes in a metal's equilibrium with its environment; the Nernst equation; the electromotive force series; potential-pH (Pourbaix) diagrams, how they're used and what they can tell us; three states of passivity; several real-world applications of mixed potential theory
Tape 4 — Kinetics of Corrosion: Polarization. Kinetics of the corrosion reaction; electrochemical reactions; important anodic and cathodic reactions; mixed potential theory; some applications of mixed potential theory
Tape 5 — Eight Forms of Corrosion: Uniform, Pitting Concentration Cell. Introduction to the eight forms of corrosion - uniform, pitting, concentration cell, galvanic, stress corrosion cracking, erosion-corrosion, intergranular, dealloying; detailed discussion covers causes of, ways of identifying and methods to prevent the first three types
Tape 6 — Eight Forms of Corrosion: Galvanic and Stress Corrosion Cracking. Two of the eight forms of corrosion - galvanic and stress corrosion cracking; hydrogen damage, a form of stress corrosion cracking; formation mechanisms, real-world examples and methods for control
Tape 7 — Eight Forms of Corrosion: Erosion Corrosion, Intergranular and Dealloying. Erosion, intergranular and dealloying corrosion are studied; how to identify each type; causes; successful methods to control or prevent; examples
Tape 8 — Corrosion Testing and Monitoring. Classification, purposes, and steps in conducting corrosion tests; surface preparation; standards; practices; planned interval tests; accelerated testing; monitoring; direct and indirect measurements; numerous real-world examples
Tape 9 — Electrochemical Test Methods. General classes of electrochemical measurements; potential current, resistivity, polarization curves, linear polarization, frequency response; methods and equipment for these measurements; galvanic series; pitting potential; protection potential; corrosion inhibitors
Tape 10 — General Material Considerations and Applications to Ferrous Alloys. Multiple requirements of materials for engineering applications; relative costs and properties of metals and nonmetals; general corrosion behavior, typical applications, and common corrosion modes of ferrous alloys
Tape 11 — Nonferrous and Nonmetallic Materials. Properties, corrosion behavior and prevention and applications of nonferrous metals and alloys and nonmetallic materials: nickel, copper, aluminum, titanium, zinc, lead, cobalt and their alloys, refractory, noble metals, ceramics, polymers
Tape 12 — Corrosive Environments. Important characteristics of corrosive environments; pH, oxidizing/reducing potential, temperature, degree of ionization, detrimental/beneficial species; how these relate to the different forms of corrosive environments; how to protect metal from a corrosive environment
Tape 13 — Economics and Failure Analysis. Corrosion costs; corrosion protection and its economic importance; opportunities and motivations to reduce corrosion costs; corrosion economics; definition, purposes, and steps of failure analysis; causes of corrosion failures; failure analysis techniques, equipment, and applications
Tape 14 — Methods of Control-Design, Material Selection, Environment Modification. Design methods and materials selection to control corrosion; changing environment to control corrosion; definition, bonding, types, and application of inhibitors
Tape 15 — Methods of Control-Coatings and Potential Modification. Anodic and cathodic protection systems; impressed-current protection; measuring and verifying protection; barrier, inhibitive, and sacrificial coatings; organic, inorganic, and metallic coatings; desireable coating features; elements of the coating process, curing mechanisms

Lecture, Full Screen Words CG, Line Drawings on CG, RP Photos, Parts Demonstrations, VISUALS: 55-65%

CERTIFICATION

Understanding ISO 9000

3 TAPE SERIES

- **Gain an understanding of what ISO 9000 is and what it does**
- **Learn about the internal and external implications of ISO 9000 for your organization**
- **Understand the process for achieving ISO 9000 registration and/or compliance**

As ISO 9000 continues to grow in popularity, more and more people are looking for answers to questions they have about the benefits and implications of the process. The purpose of this videocourse is to provide clarification to this important quality and certification issue.

Tape 1 — What is ISO 9000? Understand why QUALITY has become the code word to success in today's industrial and technical world. Understand the philosophy behind the ISO 9000 series of standards, and what this means in the context of your business. The 20 basic elements of the ISO 9000 quality system are explained in detail: management responsibility ☐ quality systems ☐ contract review ☐ design control ☐ document control ☐ purchasing ☐ purchase supplied product, etc.

Tape 2 — How Does My Company Achieve ISO 9000 Certification? The process for achieving ISO 9000 registration is explained...what you are committing to and how to prepare for the assessment. Determine if and when you need the help of a consultant. How to plan a certification strategy. How detailed does this plan have to be? What if there are deficiencies? How do you fix the deficiencies? What if there is nothing wrong? Know what guidelines to follow explicitly, what you "absolutely have to do", what you "may have to do", and what you "don't have to do". The internal audit...what is it? Who performs the external audit? What does an auditor look for?

Tape 3 — What Can I Expect During ISO 9000 Assessment? What Will I Gain From Certification? This tape answers these questions and more without complicated jargon. What is the approximate cost of being audited? What are the internal and external implications of the audit? Who within the company will be interviewed? What documentation will be needed? How registration expenditures can be minimized through proper planning.

Question and Answer Discussion, Key Point Word CG over Video, VISUALS: Key Words 75%. This series does not have corresponding lesson material available.

ADVANCED COMPOSITES

Composites II: Material Selection and Applications

10 TAPE SERIES

- **The key role of fibers, their placement and matrices in influencing physical properties of composites**
- **How properties of the composite are affected by fiber and matrix variables**
- **Become familiar with the stress-strain behavior of composites. Understand some of the failure modes of composites**

Topics covered include:

Tape 1 — Introduction: Discusses what composites are; their advantages; physical properties, and history; effects of fiber material, orientation, density, and distribution on physical properties; size effect

Tape 2 — Strength and Stiffness of Fiber-Reinforced Composites: Factors affecting properties of unidirectional composites; fibers and matrix materials, stiffness, fiber length and orientation, temperature, and humidity. Several widely used fiber/matrix combinations are studied to illustrate principles discussed in the lesson

Tape 3 — Fatigue, Thermal, and Environmental Properties of Fiber Composites: Covers fatigue and how it is influenced, thermal stresses, impact damage and how it is influenced, damage from environmental effects, and electrical properties of composites

Tape 4 — Failure Modes and Laminate Analysis: Discusses axial tensile and compressive strengths, fiber and matrix failures, transverse and through-thickness properties, anisotropic nature of composites, elastic constants, and laminate analysis computer programs

Tape 5 — Design Approaches: The composite structure development process. Manufacturing, quality assurance, nondestructive evaluation, maintainability and repair considerations. How composites design differs from metals design. Cost considerations, material selection process, laminate design, parts consolidation, failure criteria and sandwich construction. Discusses the importance of through-thickness stresses

Tape 6 — Joints and Finishing Techniques: The importance of good joint design. Mechanically fastened joints and adhesively bonded joints. Joints combining mechanical fasteners and adhesive bonding. Machining considerations and techniques

Tape 7 — Destructive Testing of Composites: Introduces destructive test methods most commonly performed on composites, including tensile, compression, inplane shear, three-point loading flexure, and interlaminar shear. Advantages, procedures, problems and limitations of each, and how to interpret results

Tape 8 — Materials Testing for Quality Control: Incoming materials quality assurance: fibers, resins and prepregs. Process control. Types of defects. Nondestructive evaluation methods. Visual, sonic, ultrasonic, acoustic emission, radiography, thermography, holography, and computer-assisted tomography

Tape 9 — Commercial Applications of Composites: Land transportation. Marine. [illegible]tainers. Electrical and electronic. Recreational. Miscellaneous

Tape 10 — Composites in Aerospace and the Future: Fighter aircraft, commercial aircraft, vertical takeoff rotorcraft, general aviation, gas turbine (jet) engines, spacecraft, space shuttle orbiter, missiles, special aircraft. Metal matrix composite applications.

Lecture, RP Kodaliths Word Charts, Line Drawings and Graphs, Full Screen Words CG, VISUALS: 30%

Welding Inspection and Quality Control

19 TAPE SERIES

- **Learn about welding metallurgy so you'll understand which areas of different metals are critical and why — and know what potential problems to look for when using different metals**
- **Learn correct joint preparation procedures; the importance of temperature control — and the mechanics of it**
- **Learn the more common — and the more specialized — welding and cutting processes**

Prepares personnel to become Certified Welding Inspectors, is of practical use as a fundamental course for new personnel and as a state-of-the-art refresher for experienced personnel. Helps management understand the responsibilities of people they supervise. Topics covered include:

Tape 1 — Welding Inspector: Who is the welding inspector ☐ qualifications ☐ code of ethics ☐ communications ☐ responsibilities

Tapes 2 and 3 — Metric Systems, Welding Terms and Symbols: SI International system of units ☐ basic metric units ☐ welding terms and definitions ☐ welding positions ☐ elements of and construction of welding symbols

Tapes 4 and 5 — Welding Metallurgy: Iron carbon system ☐ iron-iron carbide diagram ☐ diffusion ☐ effect of cooling rates and alloy addition on transformation

Tape 6 — Weld Joint Preparation and Temperature Control: Checks prior to weld joint preparation ☐ joint preparation check ☐ preheating and interpass heating

Tape 7 — Welding and Cutting Processes I: Choosing the welding process ☐ imperfections and discontinuities ☐ oxyacetylene welding ☐ brazing

Tapes 8 and 9 — Welding and Cutting Processes II: Electrogas welding ☐ plasma arc welding ☐ resistance welding

Tape 10 — Welding Procedure and Welding Operator Qualifications: Welding procedure specifications ☐ joint designs ☐ welding positions ☐ heat input

Tape 11 — Codes Governing Welding Inspection: Structural welding code ☐ ASME boiler and pressure vessel code ☐ spot examination of welded joints

Tape 12 — Weldment Imperfections and Discontinuities: Classification of discontinuities ☐ base metal discontinuities ☐ methods of testing

Tapes 13 and 14 — Chemical, Metallurgical and Mechanical Testing of Welds: Comparison of destructive and nondestructive tests ☐ chemical tests ☐ forms of corrosion ☐ testing for corrosion resistance ☐ metallographic tests

Tape 15 — Visual and Liquid Penetrant Inspection: Selection of NDT method ☐ relationship of welding processes, discontinuities and inspection methods ☐ visual inspection prior to, during, and after welding

Tapes 16 and 17 — Magnetic Particle and Radiographic Inspection: Magnetic particle inspection ☐ type of magnetizing current ☐ demagnetization

Tape 18 — Ultrasonic Inspection: Advantages ☐ limitations ☐ criteriea for successful inspection ☐ test equipment ☐ techniques

Tape 19 — Eddy Current Inspection, Acoustic Emissions, Proof Tests and Leak Tests: Eddy Current inspection ☐ fundamentals ☐ inspecting welded pipe and tubing ☐ applications ☐ acoustic emissions

Lecture, Line Drawings and Pages from Text. VISUALS: 10%

Fundamentals of Nondestructive Testing

10 TAPE SERIES

- **The technology of NDT and the basic groups of tests based on their responses to various mechanical, electrical or metallurgical changes**
- **The steps necessary to establish proper inspection approaches depending upon the nature, shape and size of test objects, plus techniques to optimize the discontinuity image**
- **Guidelines of inspection approaches for basic products, including the parameters necessary for conducting routine ultrasonic examinations**

s covered include:

Tape 1 — Introduction to Nondestructive Testing: Introduction to the basic concepts of NDT ☐ History and development ☐ Conditions necessary to perform meaningful NDT

Tape 2 — Interpretation and Classification of Discontinuities: Definition of terms - indication, discontinuity, defect ☐ Overview of steelmaking ☐ Metals processing - ingot processing, primary process (rolling, extruding, drawing, forging, casting), secondary processes (machining, heat treating, grinding, plating), in-service effects and welding processes

Tape 3 — Liquid Penetrant Testing: History and development of penetrants ☐ Basic principles ☐ Characteristics and nature of penetrant material ☐ Methods of processing

Tape 4 — Magnetic Particle Testing: History and development of magnetic particle testing ☐ Principle of magnetism and basic definition ☐ Magnetic particle equipment and materials

Tape 5 — Ultrasonic Testing Fundamentals: History and development ☐ Principles of wave propagation ☐ Transmission and reflection of ultrasound ☐ Characteristics of ultrasound as they apply to inspection of materials

Tape 6 — Ultrasonic Testing Applications: Calibration standards ☐ Factors to consider when selecting the correct method ☐ Basic test approaches - compressional for discontinuities and thickness measurement, angle beam and immersion

Tape 7 — Fundamentals of Radiography I: History and development ☐ Generation of x-rays ☐ Technique variables using x-rays ☐ Gamma-radiography

Tape 8 — Fundamentals of Radiography II — with Applications: Radiographic film characteristics ☐ Steps involved in the processing of radiographic film ☐ Quality levels

Tape 9 — Eddy Current Testing: History and development ☐ Basic eddy current principles ☐ Equipment and the various probes and coils ☐ Test variables

Tape 10 — Specialized Nondestructive Testing Methods: Overview including principles, equipment, procedures, variables, interpretation of test results, advantages and limitations for the following methods - visual, thermal, acoustical imaging, acoustic emission and others

Lecture, Title Cards (Cartoon), Pages from Text, Photos from Text, RP Photos, Full Screen Word Poster, VISUALS: 35-40%

Solving Quality Problems with Statistical Process Control

3 TAPE SERIES

- **How to recognize when a process is being overcontrolled**
- **How to tell whether a process needs to be adjusted or should be left alone**
- **How to interpret data points that fall outside of the control limits**

Statistical process control is a valuable, analytical tool in process and product quality improvement programs. Overviews of basic statistical concepts, detailed instruction in constructing and interpreting control charts, analyzing data for variance, statistical significance, cause and effect relationships, and designing programs to locate and correct non-conforming product and process characteristics are presented. Topics covered include:

Tape 1 — Introduction to SPC. Benefits of SPC; in-process sampling and control systems; pitfalls of overcontrol; chance variabilities; the process average, $\bar{x}$; standard deviation, σ; 3σ; control limits; comparing discrete, batch, and continuous processes; $\bar{x}$ and R charts; calculating upper and lower control limits for discrete processes; proper sampling intervals

Tape 2 — Control Charts for Measurement. Control chart decision rules; how to tell when process changes are needed; 3σ-limits for individual data points, process capability index, Cp; calculating control limits for batch and continuous processes via the standard deviation of averages and average moving range techniques

Tape 3 — Control Charts for Attributes. Process variables and process attributes; the p-chart, c-chart, and other process attribute charts; when to use a process attribute chart; proper sample size for attribute charting; guidelines for setting up an in-plant SPC system; role of management in SPC; employee training requirements

Lecture, RP Kodalith Words, Graphs and Charts. VISUALS: 28%. This series does not have corresponding lesson material available.

Principles of Failure Analysis

15 TAPE SERIES

- **Extensive coverage of all major types of failures encountered throughout the metalworking industry**
- **The causes of failures from material and manufacturing considerations to in-service conditions and environments**
- **Major categories of part and part configuration failures**
- **A thorough treatment of proper procedures used in failure analysis**
- **Extensive use of on-location footage, photos, graphs, films, demonstrations and animation to "bring the technical material to life"**

Provides extensive coverage of general failure analysis, procedures, types of failures and stress, ductile and brittle fractures, fatigue failures, wear failures, corrosion failures and elevated temperature failures. Also covered are failures of cast and wrought ferrous metals, joining failures, failures of tools and dies, failures of shafts and bearings, gear failures, failures of mechanical fasteners, boiler and heat exchanger failures, and failures of pressure vessels.

Tape 1 — General Procedures for Failure Analysis. Collection of data and samples □ preliminary examination □ non-destructive inspection □ mechanical testing □ selection and preservation of fracture surfaces □ macroscopic and microscopic examination

Tape 2 — Types of Failure and Stress. Major failure types □ combinations and interactions of failure types □ failure definitions □ distortion □ applied stresses □ types of loading □ stress systems □ visualizing residual stress □ directions of residual stresses

Tape 3 — Ductile and Brittle Fractures. Definitions and comparisons □ ductile-fracture modes and causes □ ductile-to-brittle transition □ causes of brittle fracture □ cleavage □ intergranular fracture □ thermally induced brittle-behavior problems

Tape 4 — Fatigue Failures. Factors effecting fatigue life □ laboratory tests □ S-N curves and prediction of fatigue life □ stages of crack initiation, propagation and fatigue fracture □ fatigue cracking □ effects of variables □ overstress □ frequency of loading

Tape 5 — Wear Failures. Abrasive wear □ adhesive wear □ contact-stress fatigue □ corrosive wear □ lubrication modes □ lubrication surface effects □ types of lubricants □ lubricant failures □ non-lubricated wear □ procedures for wear failure analysis

Tape 6 — Corrosion Failures. Electro-chemical reactions □ uniform corrosion □ pitting □ selective leaching □ intergranular corrosion □ selective attack on inclusions □ concentration cell corrosion □ galvanic corrosion □ velocity-affected corrosion in water

Tape 7 — Elevated-Temperature Failures. Types of elevated-temperature failures □ metallurgical instabilities □ environmental affects □ failures in industrial applications to include steam-turbine components, gas-turbine components, contact with liquid metals, furnace parts and fixtures, cement mills and incinerators, engine exhaust valves, petroleum refinery components, and components of steam reformers

Tape 8 — Failures of Cast and Wrought Ferrous Metals. Casting defects and classifications, failures of Class B, C and G cast metal □ effect of composition on microstructure

Tape 9 — Failures of Welded, Brazed and Soldered Joints. Welded joint surface and subsurface failures □ failures due to design, fit-up and workmanship □ preweld and postweld heating problems □ discontinuities in arc welds

Tape 10 — Failures of Tools and Dies. Characteristics and analysis of tool and die failures □ influence of design on failure □ failures of cold working tools and dies/fracture vs. wear □ failures caused by heat treatment

Tape 11 — Failures of Shafts and Bearings. Fracture origins in shafts □ fatigue and wear failures of shafts □ ductile and brittle failures in shafts □ shaft stress-raisers □ failure mechanisms of slide bearings

Tape 12 — Failures of Gears. Gear-tooth contact as it relates to wear pattern and tooth ratio □ operating loads □ causes of gear failure □ classification of gear failures

Tape 13 — Failures of Mechanical Fasteners. Threaded fasteners: failure origins, causes of failures, fretting failures, corrosion, hydrogen damage, damage due to elevated temperatures □ rivets □ blind fasteners: types and causes of failures

Tape 14 — Failures of Boilers and Heat Exchangers. Failure of boilers and related equipment: rupture from overheating, rupture from embrittlement, corrosion and scaling, fatigue and erosion, stress corrosion cracking, multiple mode failures □ failure of heat exchangers: operating conditioners, sources of failures, corrosion, stress corrosion cracking, corrosion fatigue, weld joints, effect of elevated temperatures, failure analysis procedures

Tape 15 — Failures of Pressure Vessels. Causes of failures and procedures for analysis □ metallurgical discontinuities □ fabrication practices □ pressure vessels constructed of composite materials □ service-related failures □ brittle and ductile fractures

Narrator Presentation, 2D and 3D Animated Graphics, Photos, Parts Demonstrations, Video Clips, Graphs and Word Charts. VISUALS: 85-90%

Mechanical Testing of Metals

12 TAPE SERIES

- **How strain gages, load cells, and extensometers are used to acquire mechanical test data**
- **How to interpret engineering stress-strain curves**
- **Which tensile properties are structure sensitive and which are structure insensitive**

A comprehensive overview of mechanical testing methods currently used in industry, with strong emphasis on applications and techniques. Includes testing of the major analytical procedures (i.e., hardness, tension, compression, fatigue, creep, ductility), and specialized areas: sheet metal grid analysis and computer applications. Topics covered include:

Tape 1 — Introduction to Mechanical Testing. Definitions of basic terms; benefits and applications of testing; calculating standard deviation, probable error, precision, and maximum error; writing test reports and setting up table and graphs; features of the certified testing laboratory

Tape 2 — Instrumentation & Calibration of Mechanical Testing Equipment. Calibrating universal testing machines, hardness testers, and impact and torsion testing machines; applicable ASTM standards; principles of strain gages and strain gage load cells; using extensometers to measure strain; test specimen preparation

Tape 3a — Hardness Testing, Part I. Rockwell and Rockwell superficial testing; Rockwell testing machines; Brinell hardness testing; Brinell testers; scale selection in Rockwell testing; load selection in Rockwell testing; load selection in Brinell testing; surface preparation; special testing machines; applications. *Includes equipment demonstrations*

Tape 3b — Hardness Testing, Part II. Microhardness testing with Knoop and Vickers indenters; surface preparation; applications of indentation microhardness testers; ultrasonic microhardness testing; Scleroscopes, durometers, Mohs' hardness, and file hardness. *Includes equipment demonstrations*

Tape 4 — Fundamentals of Tension and Compression Testing. The tensile test and the stress-strain curve; structure-insensitive properties such as Young's modulus; structure-sensitive properties such as yield strength and tensile strength; application of compressive properties; testing machines, specimen grips, extensometers, and instrumentation

Tape 5 — Special Applications of Tension and Compression Testing — An in-depth look at the stress-strain curve; precision elastic and microelastic limits; effects of temperature, strain rate, and grain size; the true stress-true strain curve; strain hardening exponenet, n; r-value and plastic anisotropy in sheet products; notch sensitivity; comparing tensile testers

Tape 6 — Shear, Torsion, Creep and Creep-Rupture Testing. Single and double-shear tests; torsion testing; the shear modulus, G, shear and torsion testing machines; creep curves and the three stages of creep; creep testers; the Larson-Miller and other parameters; creep equations; isochronous stress-strain curves

Tape 7a — Ductility and Formability Testing. Using tensile test data to evaluate springback tendency, stretch formability, and other forming characteristics; determining r-value, strain rate sensitivity, and the work hardening exponent; hydraulic bulge, hole expansion, and stretch bend tests; the Erichsen, Olsen, and limiting dome height tests; the Swift, Englehardt, and Fukui tests

Tape 7b — Circle Grid Analysis. The forming limit diagram; shape analysis via the stretch-draw chart; distinguishing between tooling and design problems; making a material edge condition analysis; selecting the optimum lubricant by using coefficient of friction test data; the formability data sheet; applications of circle grid analysis

Tape 8 — Fracture Testing. Charpy impact test; three ways to indicate resistance to impact; effects of specimen orientation; instrumenting the Charpy test; deriving dynamic fracture toughness from Charpy data; fracture toughness testing; using test data to find KIc. *Includes equipment demonstrations*

Tape 10 — Application of Computers to Mechanical Testing. Benefits of computerized data acquisition and control; levels of computerization; analog-to-digital (A/D) converters and their selection; I/O (input-output) devices and their selection; designing a computerized mechanical testing system; software development

Lecture, RP Kodalith Line Drawings and Word Charts, Word Posters (Paste-up), Chalkboard, VISUALS: 40-50%

THERMAL SPRAY COATINGS TECHNOLOGY

Thermal Spray Technology

8 TAPE SERIES

- **Understand thermal spray processing science as well as applications and practice**
- **Learn how thermal spray processing interacts with the materials it is designed to protect**
- **Consider testing, characterization and standards used to evaluate coatings**

This course provides an understanding of thermal spray processing science as well as applications and practice. You will learn how thermal spray processing interacts with the materials it is designed to protect.

Tape 1 — Surface Science. Wear, corrosion, hardening, carburizing, nitriding, electroplating, electroless plating, phosphating, vapor deposition, hardfacing.

Tape 2 — Equipment & Theory: Combustion and Electric Wire-Arc. Thermal spray history, basic theory of coating processes, porosity, bonding, application, combustion spray processing and equipment, wire-arc spray processing and equipment, material feed, deposit characteristics, HVOF.

Tape 3 — Equipment and Theory: Plasma Spray. Theory of 4th state of matter, APS Airplasma spray, powder feed, particle distribution trajectory, LPPS plasma spray variations, material feed systems, controls, consoles and power supplies, ancillary equipment, safety and hygiene.

Tape 4 — Processing and Design. Bonding, cleaning, processing, masking, temperature control, spray pattern, process variation, automation, fusing, densification, finishing, stripping.

Tape 5 — Materials. Material production methods, particle classification methods, quality control, material specification, standards.

Tape 6 — Applications. Aerospace, automotive, biomedical, ceramic and glass, marine, non-skid, electronics, printing, processing industries, textiles.

Tape 7 — Testing and Characterization: Methods and Mounting. Variations in test equipment, metallurgy and materials, grain size, bonding, coating buildup, response generators and variations, metallographic procedures, sectioning, vacuum impregnation, mounting materials and techniques.

Tape 8 — Testing and Characterization: Preparation and Procedures. Specimen preparation, grinding, equipment, abrasive types, material reaction, deformation and smearing, polishing concepts and procedures, etching, lubricants, hardness and tensile testing.

Narrator Presentation, 2D Animated Graphics, Photos, Video Clips, VISUALS: 90%

MACHINING

Principles of Machining

12 TAPE SERIES

- **Rigidity and heat — why these characteristics influence most machine tool and cutting tool decisions**
- **How 50° temperature reduction can give 5 times more tool life**
- **How the carbon contents affect the heat treatment and machinability of steel**

This videocourse presents the principles and practices of machining, tool material selection, cutting speeds, lubricants, and related topics. Primarily concerned with mechanical chip making machining processes, it examines everything from basic machining processes to microstructural aspects of metals and hardness of microconstituents and the effects on machinability. Topics covered include:

Tape 1. The Machine Tool — Principles and Operations. Basic components in a machining process □ machine tool structure □ cutting tools □ workpiece □ heat and rigidity □ turning and boring □ drilling □ planing □ shaping □ chip formation • BUE

Tape 2. Cutting Tools — Design and Effects. Chip removal □ work hardening □ coolants □ free machining additives □ cratering □ flank wear □ rake angles and inclination

Tape 3. Tool Life — Measurement and Control. Power measurements □ tool life □ wear patterns □ tool life curve □ thermal overloading □ machinability □ cutting speed □ grindability

Tape 4. Machinability. Machinability vs. microstructure □ phase diagrams □ microstructures of iron and steel □ economics □ tool changing cost □ optimum machining conditions □ reducing machining costs □ cutting fluids □ gases, oils and water-base fluids □ cutting fluid application □ surface quality □ causes of poor finish □ residual stress

Tapes 5 and 6. Turning I and II. Characteristics of turning □ enginge, turret, bench lathes □ speed □ feed □ depth of cut □ tracer attachments □ taper attachments □ single point tools □ classes of tools □ counter balanced fixtures

Tapes 7 and 8. Milling I and II. Milling characteristics □ knee and column milling machines □ special milling machines □ milling cutters

Tapes 9 and 10. Drilling I and II. Major drill points □ Web thinning of twist drills □ characteristics of drilling □ use of bushings □ principles of holemaking □ angles and point tolerances

Tapes 11 and 12. Grinding I and II. Characteristics of grinding □ polishing □ buffing □ honing □ lapping □ precision grinding machines □ grinding wheels □ Abrasives

Lecture, Line Drawings and Graphs from Text, RP Kodalith Charts, VISUALS: 5%

Although the format of these videos is setup to instruct workers in the industry, they can easily be cued to the process portion of the video to be used to augment your lessons. **NOTE:** Estimated percentage of lecture vs visuals. RP refers to Rear Projection whereby 35mm slides, overheads, etc. are projected on a screen from behind so that a pointer can be used to identify specific words or areas. Kodalith is an acetate or overhead in clear words or drawings on black background.

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Metallurgy / Materials Education Yearbook, 32nd Edition, July 1993-94

A 215-page reference book listing colleges and universities which offer four year and advanced degree programs in metallurgy/materials, ceramics and polymers (U.S. and International Schools). Includes Department Chairmen, addresses, phone numbers, faculty and their specialties. The yearbook is produced and distributed through the support of Dr. Kali Mukherjee, Sr. Editor and The ASM Foundation for Education and Research.

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NEW DEVELOPMENTS IN ALUMINUM FOR AIRCRAFT AND AUTOMOBILES

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Alloy Technology Division
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Telephone 412-337-5922

Common bond for aircraft and automobiles is need for cost-efficient, lightweight structure.

Aluminum base materials

New Developments in Aluminum for Aircraft and Automobiles

- Automotive
  - Needs
  - Developments
  - Directions

- Aircraft
  - Needs
  - Developments
  - Directions

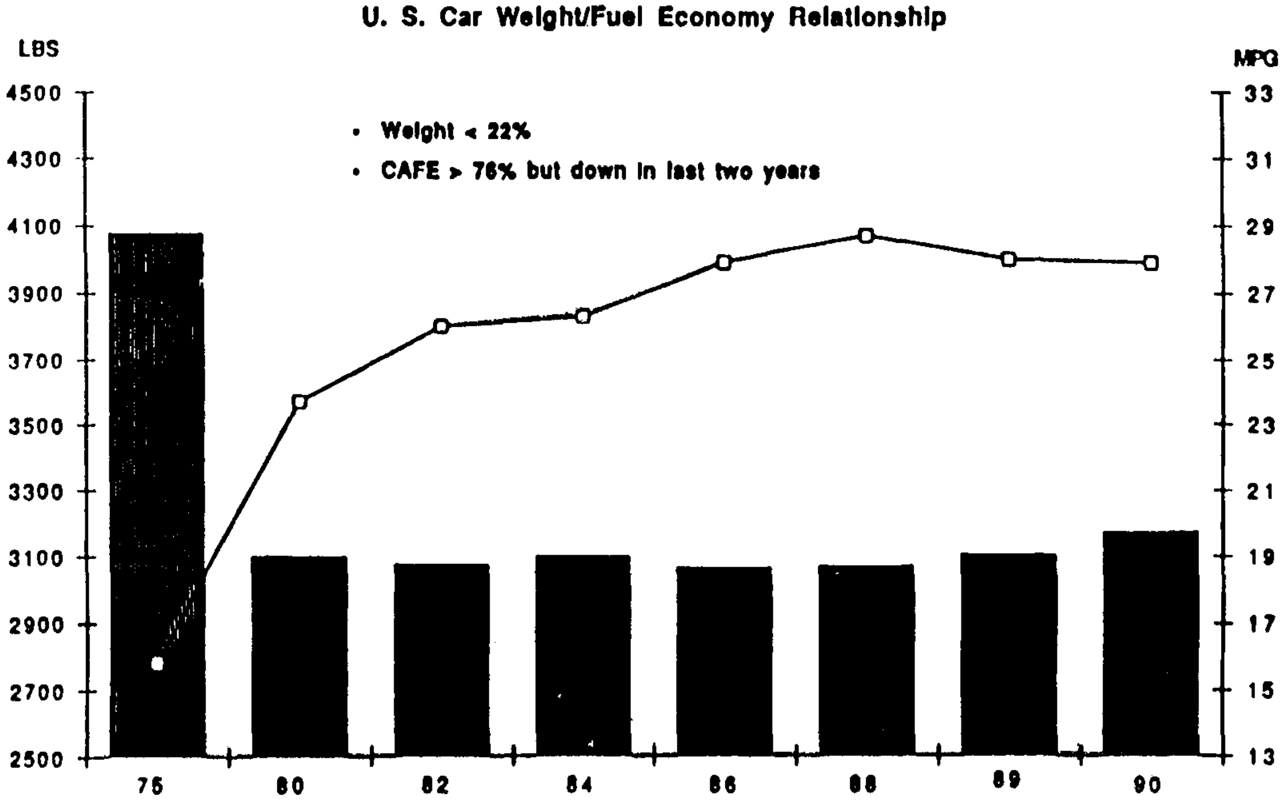
Forces Shaping Future Automotive Materials Needs

- Need for fuel efficiency
- Changing consumer preferences
- Growing environmental awareness
- Globalization of market

BACKGROUND, AUTOMOTIVE

1975 TO 1991 - SOURCES OF REDUCTION IN FUEL CONSUMPTION

TIRES	22.4
WEIGHT	32.2
AERODRAG	34.7
POWER TRAIN	10.7
	100%



Source: U. S. Environmental Protection Agency

1046jsb10624

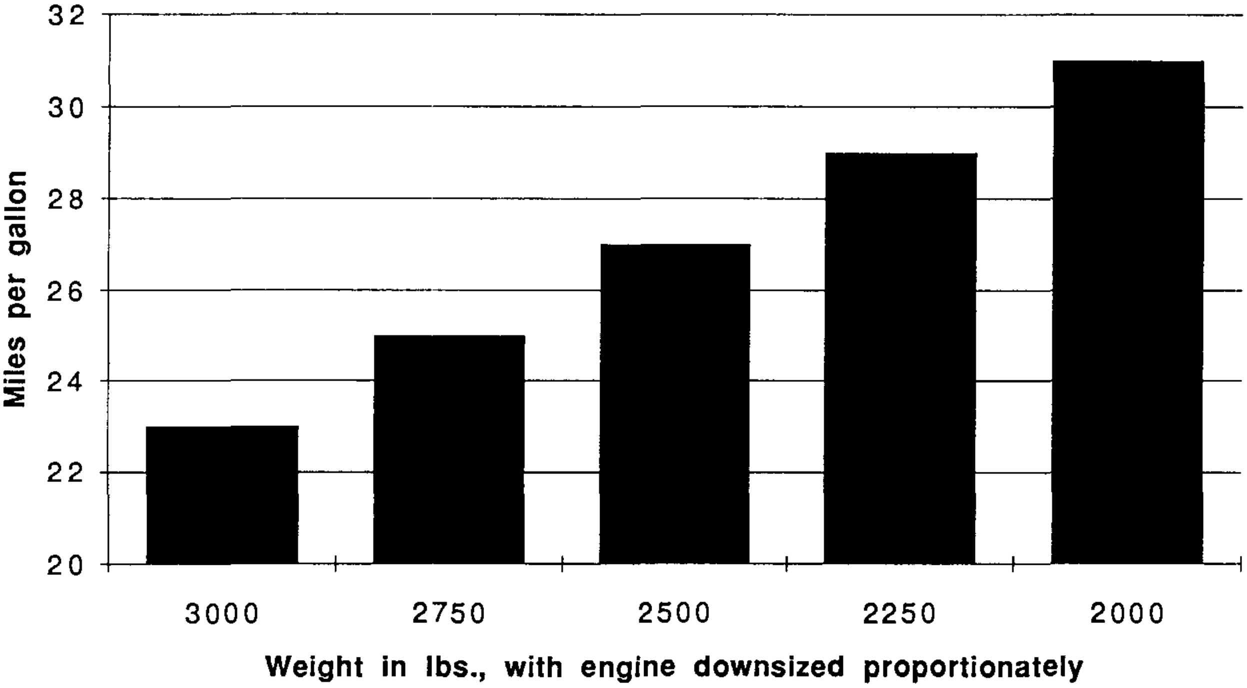
Automotive

Why use aluminum?

- Weight reduction
  - Increased fuel economy
  - Decreased emissions
  - Increased performance
  - Increased cargo capacity
- Longer vehicle life
- Recycling capacity

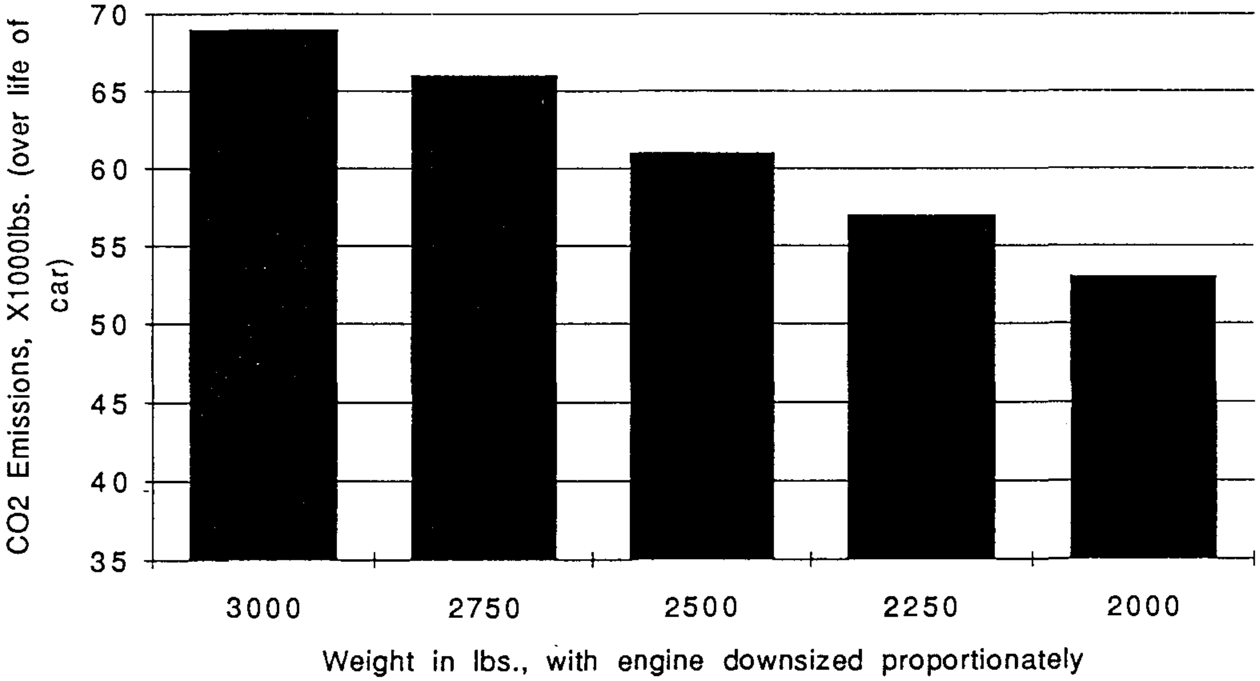
Energy Cycle

LOWER WEIGHT = HIGHER MPG



CAR WEIGHT/EMISSIONS

LESS WEIGHT = LOWER EMISSIONS



Aluminum Strength/Weakness versus Competitive Materials

Al Strength vs Steel

- Lightweight effectiveness
- Corrosion Resistance

Al Strength vs Plastic

- Lightweight effectiveness
- Stiffness
- Recyclable
- Ease of repair

Al Weakness vs Steel

- Stiffness
- Ease of manufacturing
- Cost

Al Weakness vs Plastic

- Design options
- Corrosion resistance
- Dent resistance

Automotive

- **Hang-on components**
- Bumper components
- Space Frame components

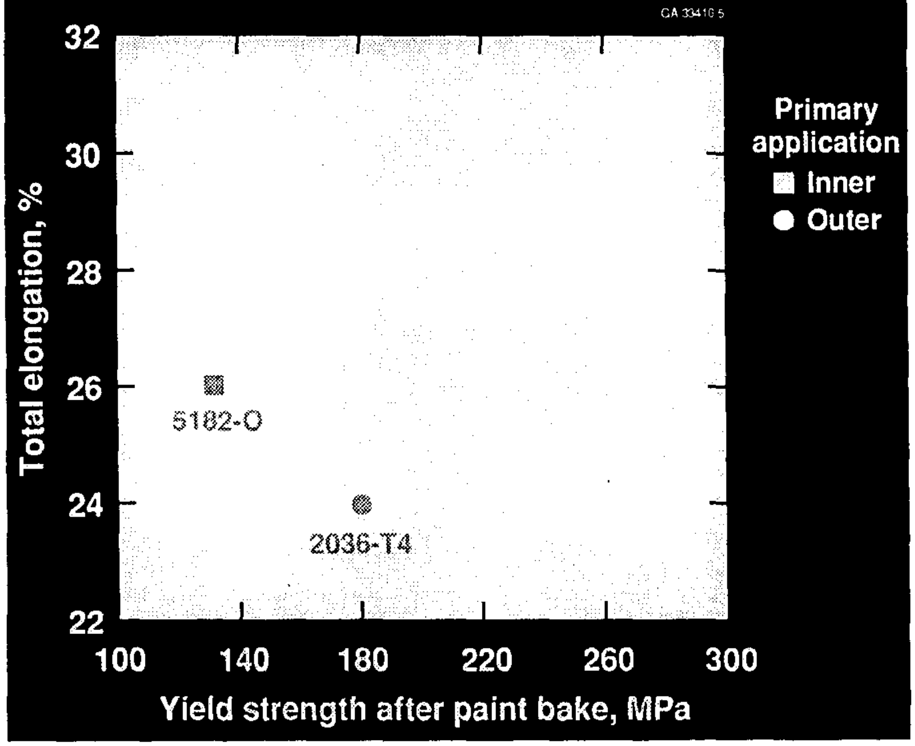
Automotive

Hang-on components
- Outer panels
  - Class A surface
  - Corrosion resistant
  - Y.S.
  - U.S. and Europe: > 207 MPa
  - Japan: 138 MPa < Y.S. < 172 MPa
  - Formable
    - Stretchable
    - Drawable
    - Hemmable
  - Alloys
    - 2XXX
    - 6XXX

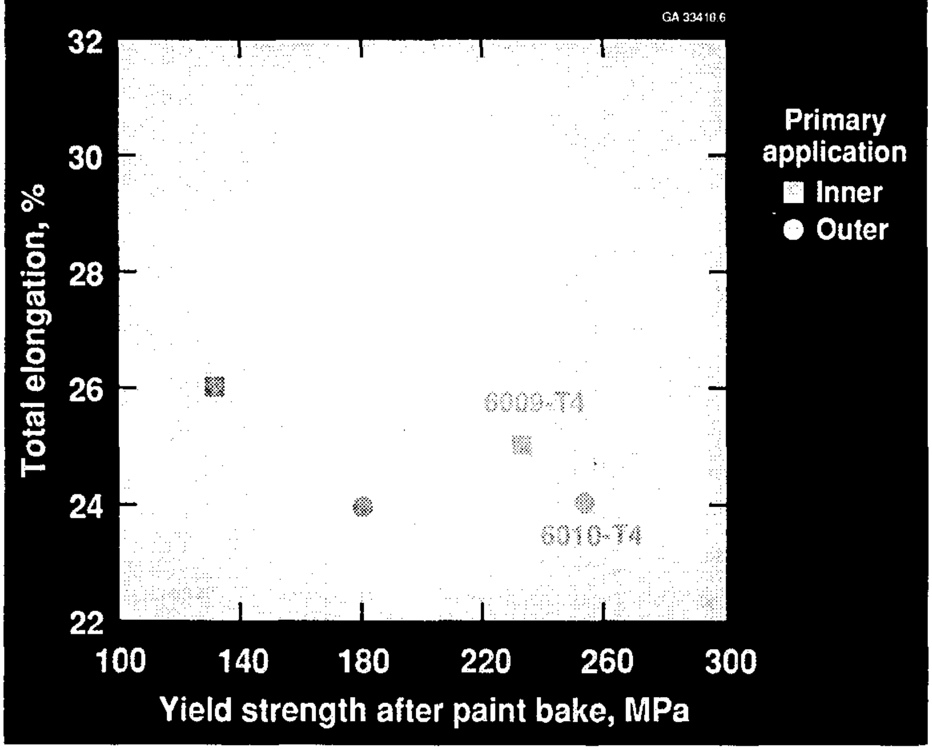
Automotive

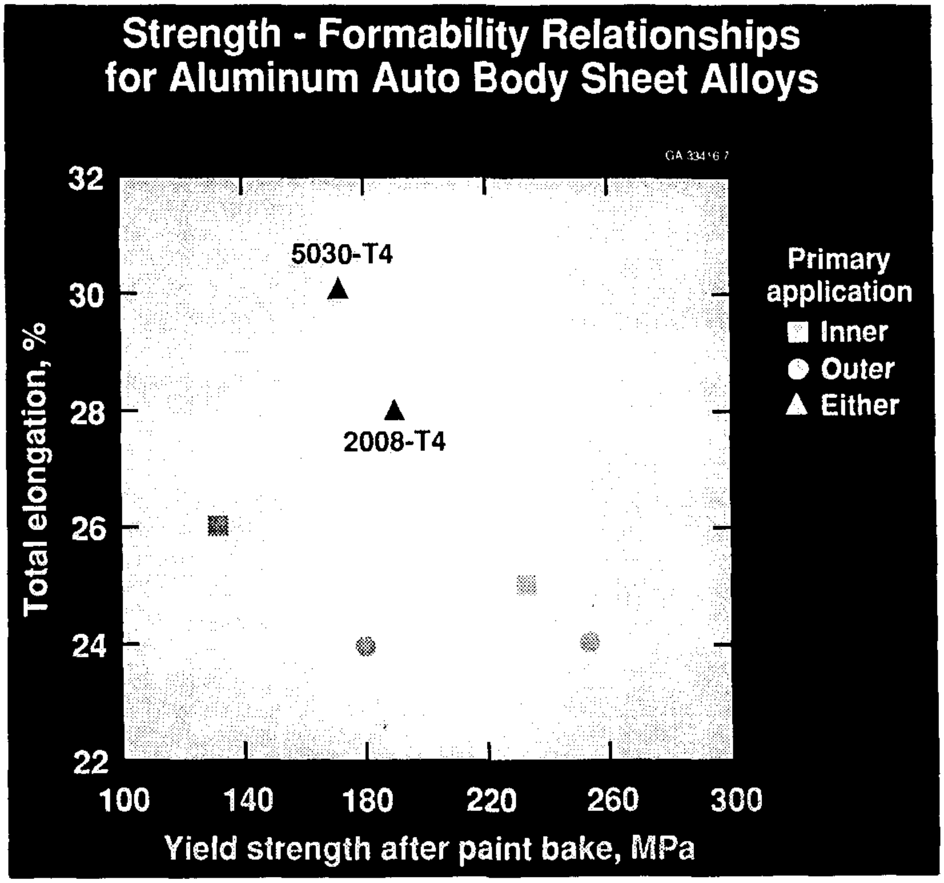
- **Hang-on components**
  - Outer panels
    - Class A surface
    - Dent resistance
    - TYS
      [illegible]
      Japan: 130 MPa < TYS < [illegible]
    - Formable
      - Stretchable
      - Drawable
      - Hemmable
    - Alloys
      - [illegible]
      - 6XXX
  - Inner panels
    - Formable
      - Stretchable
      - Drawable
      - Hemmable
    - Alloys
      - 5XXX
      - 6XXX

Strength - Formability Relationships for Aluminum Auto Body Sheet Alloys



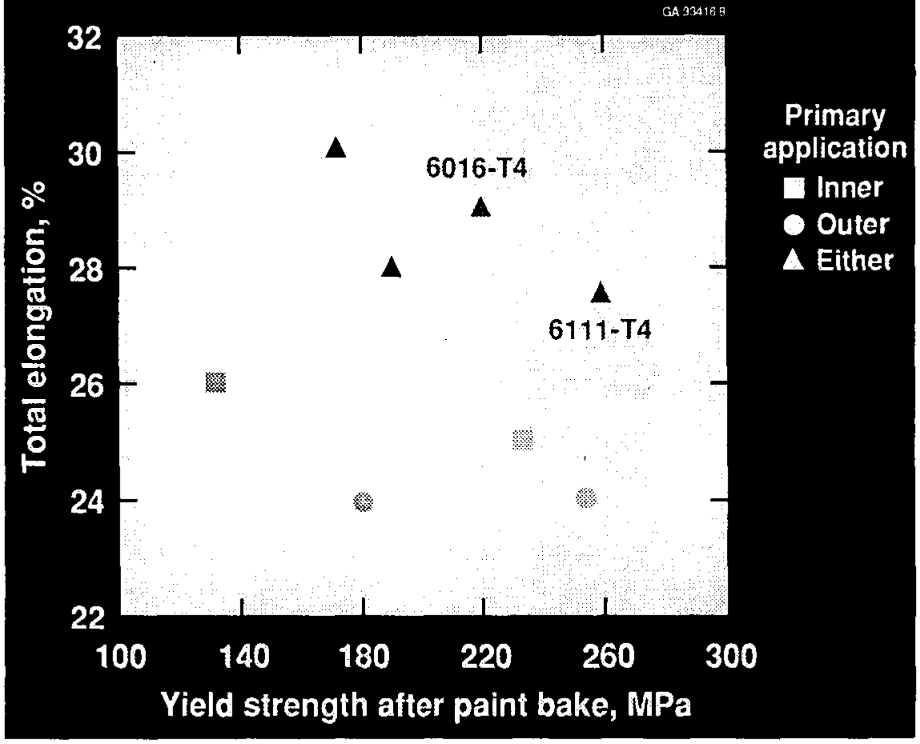
Strength - Formability Relationships for Aluminum Auto Body Sheet Alloys





Strength - Formability Relationships for Aluminum Auto Body Sheet Alloys

Strength - Formability Relationships for Aluminum Auto Body Sheet Alloys



Automotive

- Emerging materials for hang-on components
  - Near term
    - 2XXX and 6XXX low bake temperature
    - 5XXX Luder-free

Automotive

Emerging materials for hang-on components

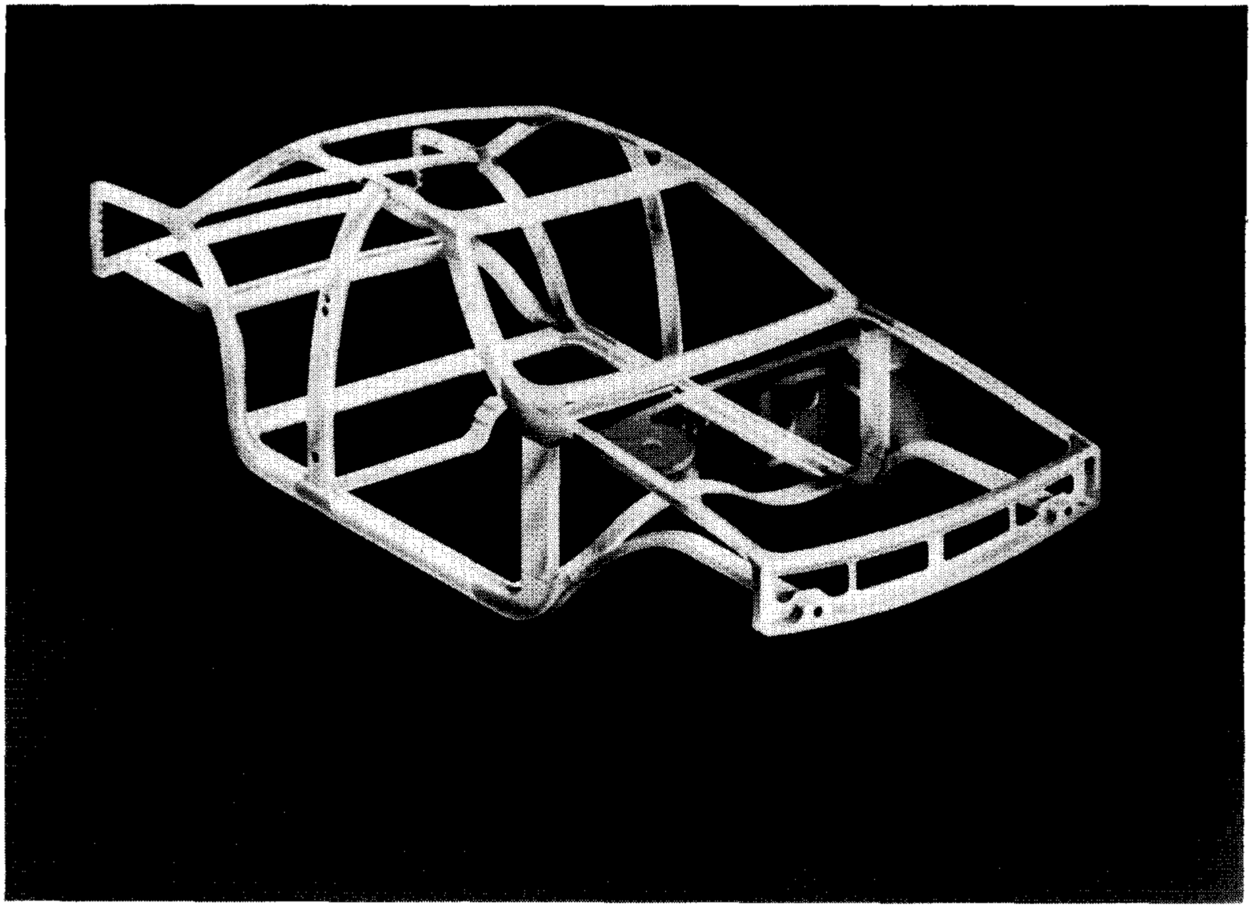
- [illegible]
  - [illegible]
  - [illegible]
- Long term
  - Low cost
  - Formability, strength, weldability, and finish of best DQ steel
  - Corrosion resistance of best Al sheet

Automotive

- Hang-on components

Bumper components

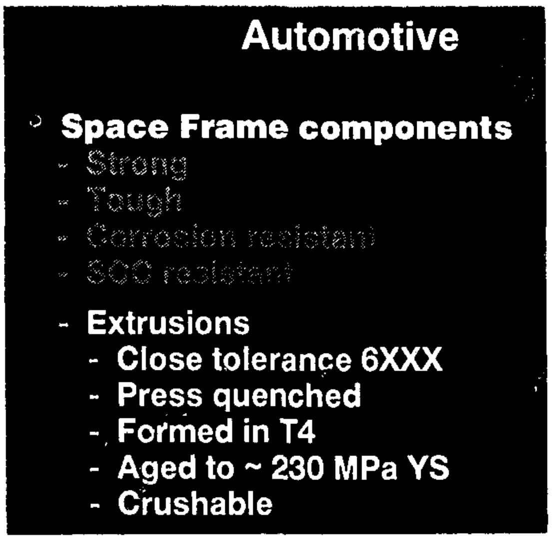
- [illegible] components



This rendering of a generic spaceframe illustrates the use of less than 100 aluminum extrusions and interconnecting aluminum die cast nodes which are robotically welded to form the car body. A limited number of aluminum sheet components (i.e. inner fenders, floor pan) are then attached to complete the body.

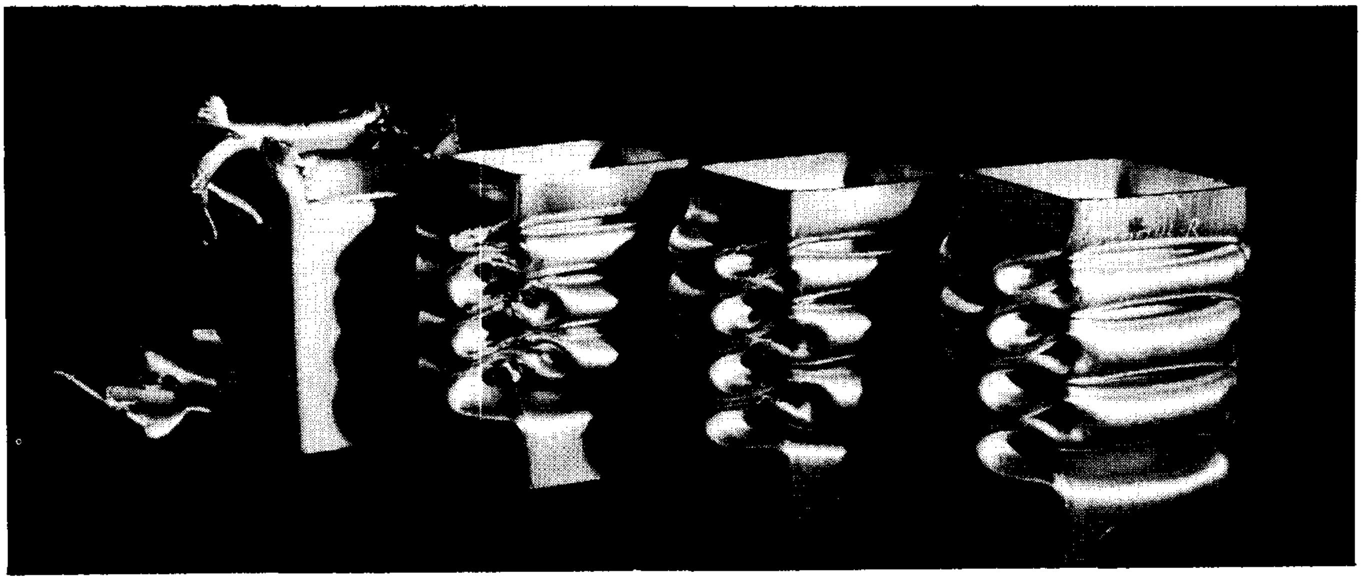
Automotive

- **Space Frame components**
  - Strong
  - Tough
  - Corrosion resistant
  - SCC resistant



Automotive

- **Space Frame components**
  - Strong
  - Tough
  - Corrosion resistant
  - SCC resistant
  - Extrusions
    - Close tolerance 6XXX
    - Press quenched
    - Formed in T4
    - Aged to ~ 230 MPa YS
    - Crushable



Automotive

Space Frame components

- Strong
- Tough
- Corrosion resistant
- SCC resistant

- Extrusions
  - Close tolerance 6XXX
  - Press quenched
  - Formed in T4
  - Aged to ~ 230 MPa YS
  - Crushable

- Die castings
  - Proprietary vacuum casting
    - < 5 ml gas/100g metal
    - Low porosity
  - High Si, low Mg
  - Fe to reduce die erosion and welding
  - SHT aged to T6
    - YS 115 to 140 MPa
    - 18 to 22% elongation
    - Crushable

Evolution of Aluminum Aerospace Alloys

New aluminum base alloys continue to be introduced

- 1920's - 2017, 2014
- 1930's - 2024
- 1940's - 7075
- 1950's - 7178, 7079, X2020
- 1960's - 7175, 7475, 2124
- 1970's - 7050, 7150, 2324
- 1980's - 2034, 2090, 8090, 2091
- 1990's - 7055, C188, ???
- 2000's - ???

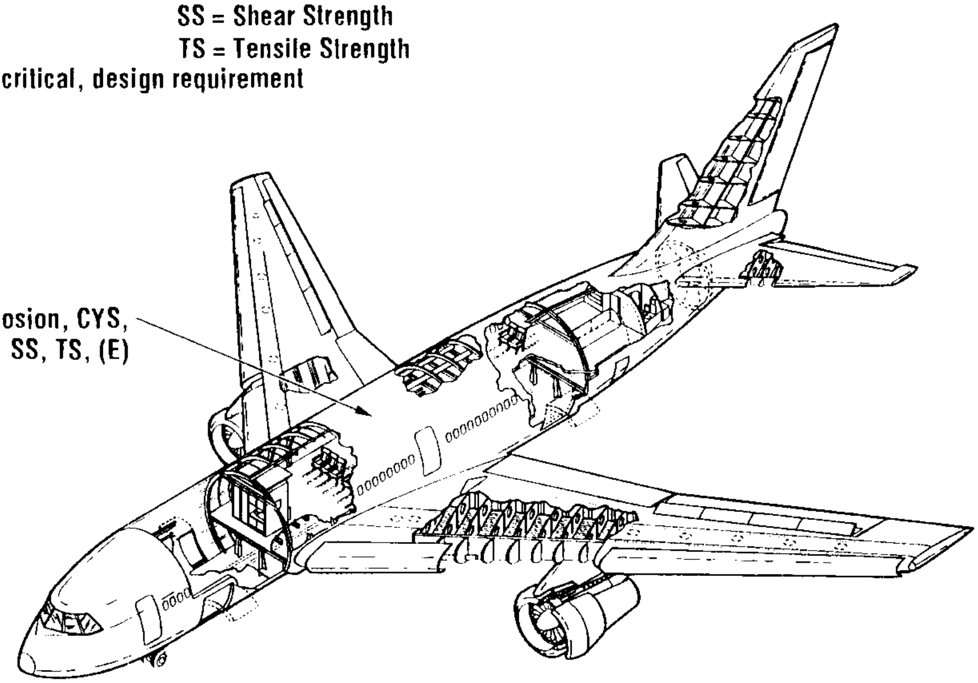
Forces Shaping Future Aircraft Materials Needs

Many factors are driving change in 1990's:

- Aging commercial fleet
  - ➪ *fatigue, corrosion*
- Attention to cost effectiveness
  - ➪ *procurement, inventory, manufacturing, operating*
- Fuel prices ???
  - ➪ *incremental weight savings*
  - ➪ *radical design/material changes*
- Future supersonic commercial aircraft
  - ➪ *radical design change, high temperature*
- New competition

Property Requirements for Jetliner and Military Transport Applications

Material properties:
Corrosion
CYS = Compressive Yield Strength
E = Modulus
FAT = Fatigue
FCG = Fatigue Crack Growth
FT = Fracture Toughness
SS = Shear Strength
TS = Tensile Strength
( ) = Important, but not critical, design requirement



Fuselage skin: Corrosion, CYS, FAT, FCG, FT, SS, TS, (E)

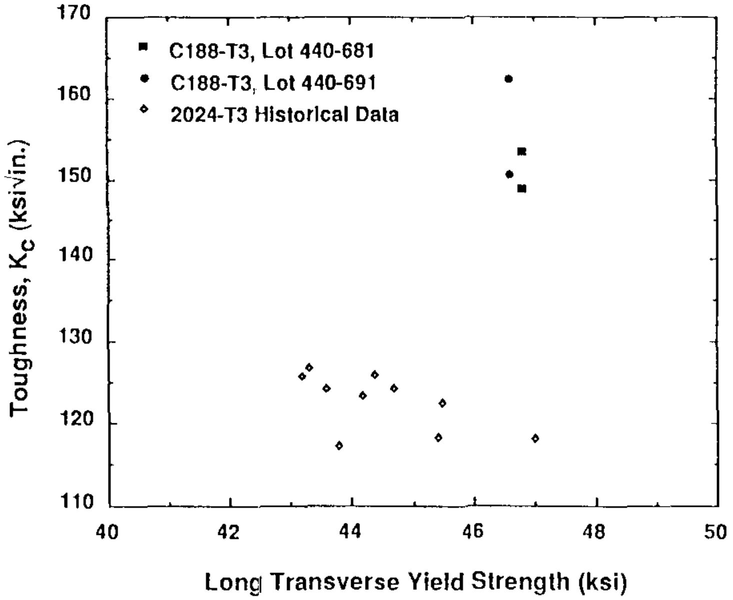
Fuselage

Skin

	Commercial and Transport	High Performance
Standard:	2024-T3 7475-T76 (thick)	7475-T76
Newly used:	2XXX-T3	
Being evaluated:	6013-T6 2091 8090 GLARE®	

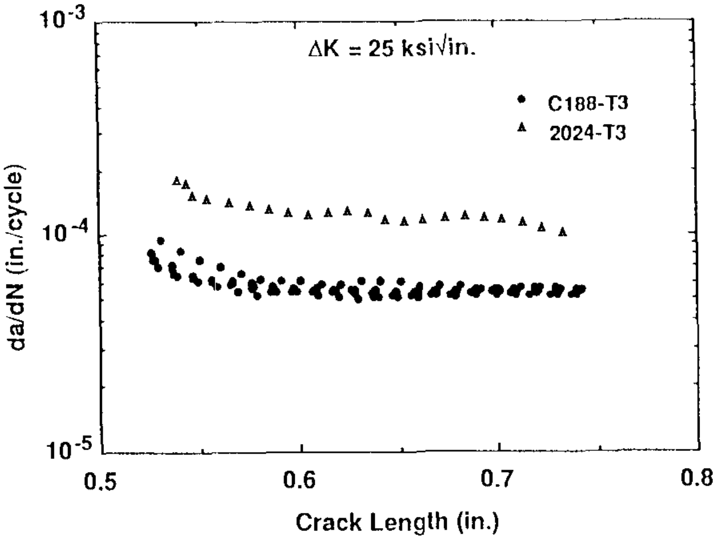
Toughness vs. Yield Strength:

Strength/toughness relationship for C188-T3 and 2024-T3 alclad sheet, 0.100 in. thick, T-L orientation. Toughness measured using 16 in. wide M(T) specimens.

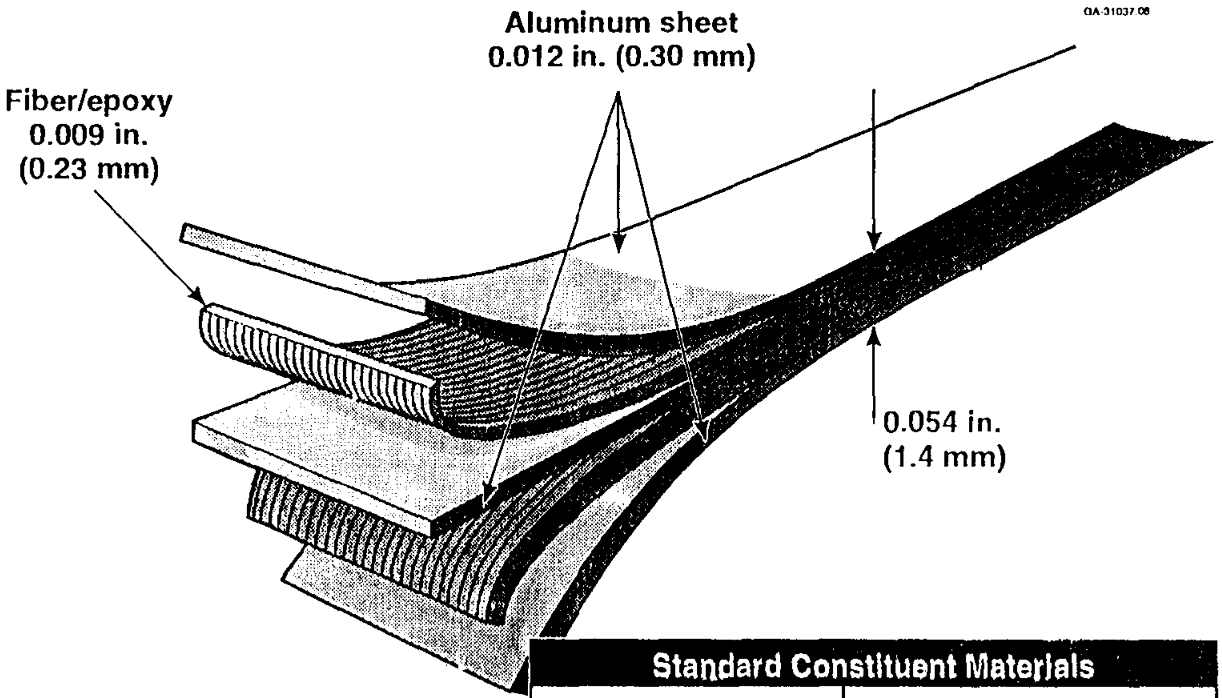


Constant ΔK Test:

Fatigue crack growth rate vs. crack length for C188-T3 and 2024-T3 alclad sheet tested at constant ΔK=25 ksi√in., R=0.1, T-L, high humidity (R.H.>90%) air.

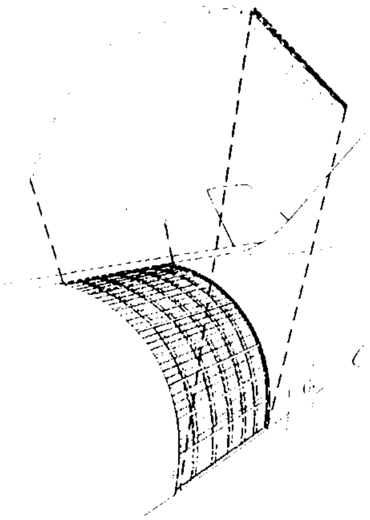


Fiber/Metal Structural Laminates
(Typical 3/2 Lay-Up Shown)



Standard Constituent Materials	
Aluminum sheet alloy	2024 and 7475
Fiber	Aramid and glass
Prepreg	Unidirectional and cross-ply

Fiber-Metal Laminates



Benefit: Weight Reduction
Application: Fuselage Skin
Target: 20 - 25%

Weight Reduction Because of:

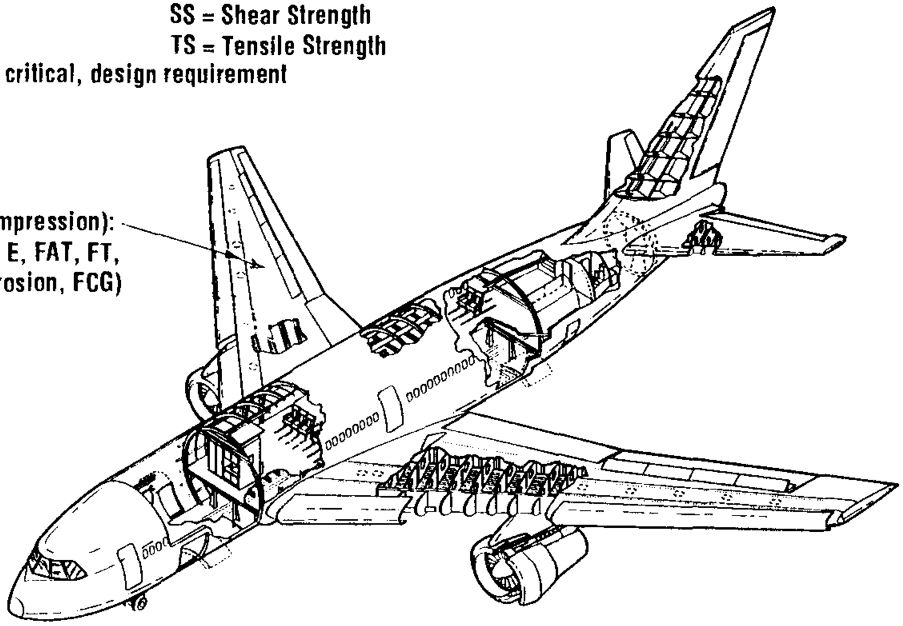
- Density Reduction (10 - 15%)
- Downgaging Sheet Thickness (10%)
- Part Elimination (Doublers, Tear Straps)

Downgaging Possible Because of:

- Superior Fatigue Properties
- Excellent Damage Tolerance
  (Residual Strength, Fracture Toughness)

Property Requirements for Jetliner and Military Transport Applications

Material properties:
Corrosion
CYS = Compressive Yield Strength
E = Modulus
FAT = Fatigue
( ) = Important, but not critical, design requirement
FCG = Fatigue Crack Growth
FT = Fracture Toughness
SS = Shear Strength
TS = Tensile Strength

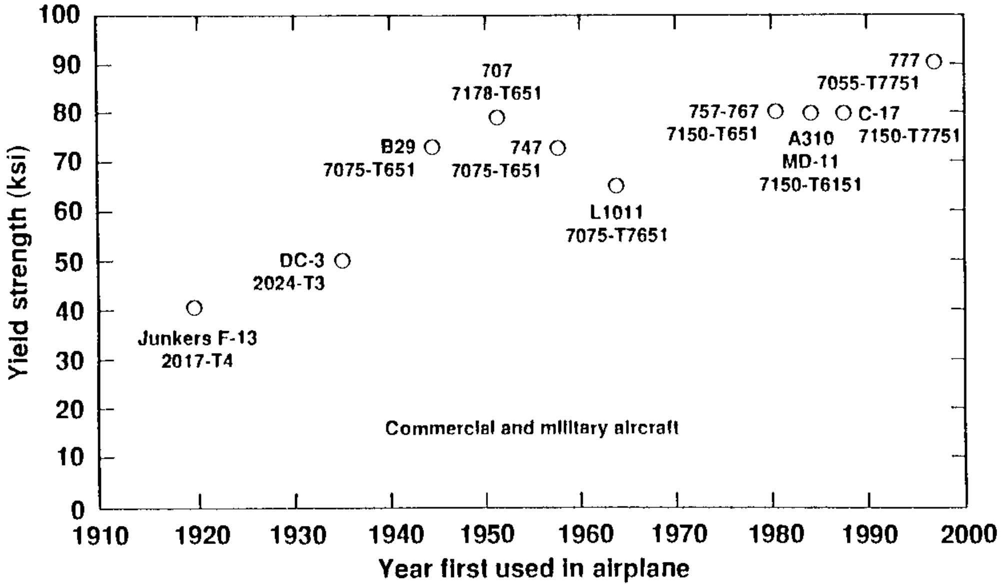


Upper wing (Compression):
Skins: CYS, E, FAT, FT,
(Corrosion, FCG)

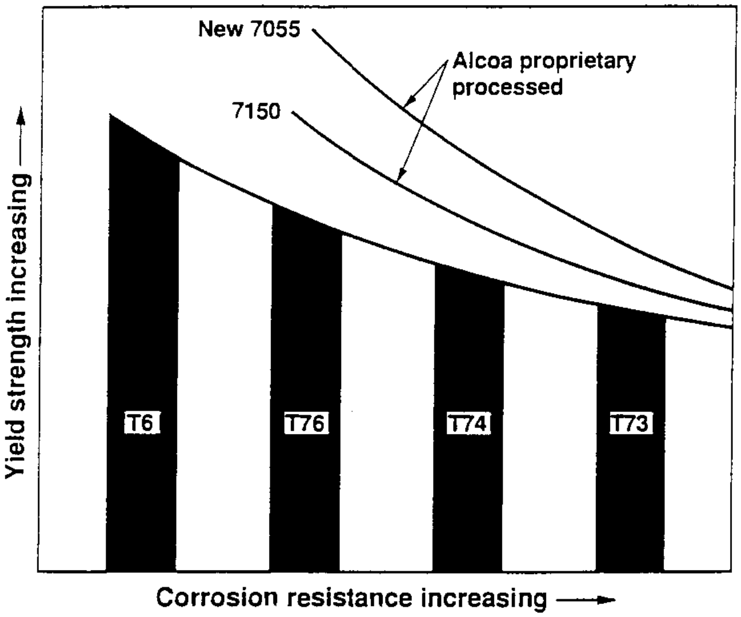
Wing

Upper Cover

	Commercial and Transport	High Performance
Standard:	7150-T6	7475-T73
	7150-T61	7050-T76
		2124-T8
Newly used:	7150-T77	
	7055-T77	
Candidates for development:	DRA	DRA
	Al-Gr	Al-Gr
	Al-Be	Al-Be
		CRA



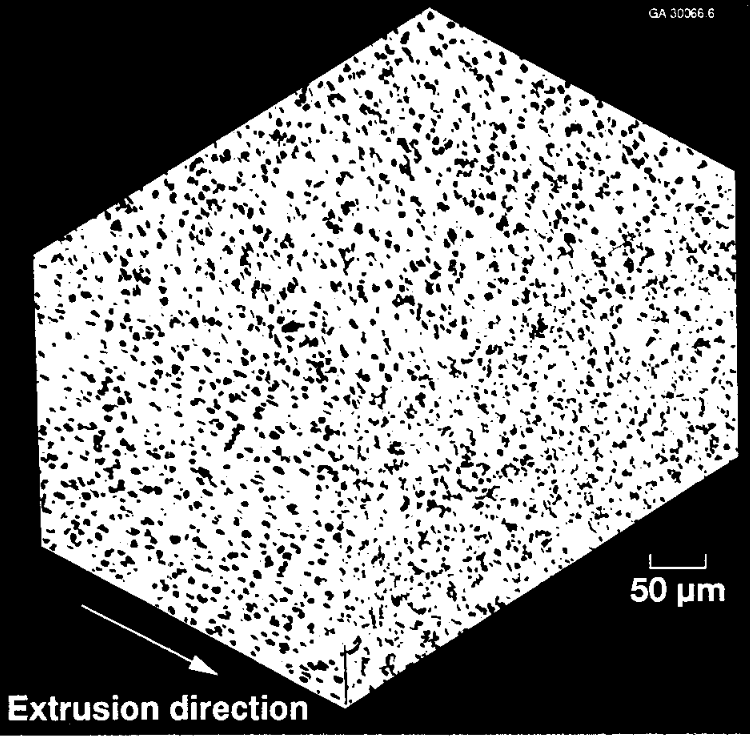
Upper Wing Skin Plate Alloy/Temper Chronology



Schematic Illustration of Strength/Corrosion Resistance Improvements of the New Alcoa Aluminum Alloy 7055 Compared to Aluminum Alloys 7150 and 7050

The DMMC Material Microstructure:

The P/M processing route can produce a reinforcement that is well-distributed in the aluminum matrix



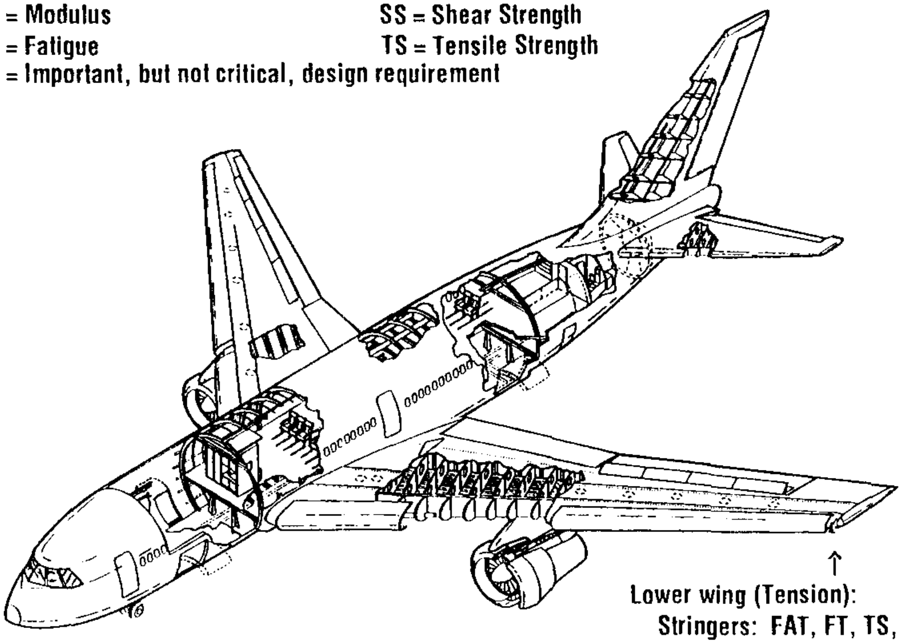
Wing

Lower Cover

	Commercial and Transport	High Performance
Standard:	2024-T3	7475-T73
	2324-T39	2419-T8
	2224-T3	
Being evaluated:	8090-T8	
	7475-T76	
Possible candidates:	ARALL	X7093-T73
		Al-Li

Property Requirements for Jetliner and Military Transport Applications

Material properties:
Corrosion
CYS = Compressive Yield Strength
E = Modulus
FAT = Fatigue
( ) = Important, but not critical, design requirement
FCG = Fatigue Crack Growth
FT = Fracture Toughness
SS = Shear Strength
TS = Tensile Strength



Lower wing (Tension):
Stringers: FAT, FT, TS, (Corrosion, FCG)

SCLEROSCOPE HARDNESS TESTING

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Scleroscope Hardness Testing

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KEY WORDS: brinell, durometer, elastic/plastic, equotip, hardness, rockwell, scleroscope

PREREQUISITE KNOWLEDGE: Elastic and plastic responses of materials; basic definitions of mechanical properties, such as ultimate strength, yield point, stiffness, resilience, toughness and restitution.

OBJECTIVES: To observe and understand the principles of elastic and plastic hardness, via scleroscope tests.

EQUIPMENT AND SUPPLIES:

1. Tube (15 mm OD x 30 cm tall; transparent and rigid)
2. Ball bearing (10 mm diameter; steel)
3. Solid bench or block (anvil)
4. Metric ruler
5. Metal samples (minimum 5 mm thick)
6. Brinell scope (20x magnification)
7. Equotip portable hardness tester (scleroscope)

INTRODUCTION: Hardness is commonly defined as "resistance of metals to plastic penetration". Labs routinely report plastic penetration by Mohs (scratch), Brinell (dent), and Rockwell (depth) tests of metals. However, non-metal products (paper rolls, plastic foams) are tested quickly and cheaply for elastic penetration by durometers. Depth is indicated on a spring loaded dial-gage.[1,2]

Shore Scleroscopes (rebound) should not be confused with Shore Durometers, nor neglected in the Materials Lab. A scleroscope measures the rebound height for a diamond tipped tup, or drop weight.[3,4] Remember how "bouncing a quarter" off a taut army cot or "kicking the tires" on a used car views rebound as a measure of hardness.

BASIC PROCEDURE: A simple scleroscope can readily demonstrate both "elastic" and "plastic" hardness. Place a clear tube of glass or plastic (15 mm dia. x 30 cm tall) vertically on a sample. The sample should be thick enough (minimum 5 mm) to avoid anvil effects, from a solid backup-block or workbench. A steel ball bearing or Brinell ball (10 mm diameter) is dropped down the 30 cm tube. A metric ruler is held alongside the clear tube for measuring the rebound height.[5]

Students enjoy participating in this crude test. Accuracy is a challenge. We learn to discard initial readings (unfocused eye); to question low rebounds (tube friction) and high rebounds (work hardening); to avoid parallax (scale to ball); to read the scale quickly and consistently (bottom of ball bearing). Such tedious data gathering prepares students to appreciate a commercial scleroscope.

One measure of plastic hardness is the average diameter of dents produced by the falling ball-bearing. A standard Brinell scope (20x) works well for tiny dents, typically (0.1-0.3 mm). Students soon see that data matches intuition; harder material gives higher rebounds and smaller dents. Several common materials with various hardness values are tested, such as steel, copper, brass and aluminum. Students are challenged to compare test data with published stress/strain curves for typical metals. Bored veterans are challenged to develop a "hardness index", combining both elastic and plastic components, e.g., the coefficient of restitution and the impact energy/area.[6]

COMMERCIAL TEST: Purdue's MET department recently acquired an "Equotip" hardness tester, with electronic readout. The operation of the Equotip is similar to our lab simulation of a scleroscope. A spherical tungsten-carbide tip impacts the sample being tested. Impact and rebound velocities are magnetically sensed for an Equotip hardness indication. Again, harder materials have faster rebound velocities and higher Equotip hardness values. Ball-bearing measurements for aluminum are compared with Equotip hardness values, in Table 1 and Figure 1. Before testing, all samples were sanded with an 80 grit, to remove surface oxides and raised areas. As expected, Equotip hardness values correlate somewhat with rebound heights.

A significant advantage of the Equotip tester is its portability. When field testing is required, this Equotip unit can test a wide range of thickness (minimum 3mm), unlike most portable testers. This discussion leads to a demonstration of other portable testers, such as Webster pliers and Ames units. Thus, students understand the need for various testers, their advantages and limitations. The scleroscope provides an opportunity to observe elastic resilience and plastic toughness of many materials.

Table 1

Aluminum Sample	Rebound ht., cm Average*	Range	Dent dia., mm Average*	Range	Equotip Hardness Average*	Range
1	4.8	2.0	1.18	0.5	231.8	50
2	3.8	0.7	1.08	0.2	342.6	48
3	4.3	1.1	1.10	0.2	316.0	13
4	4.4	0.5	1.18	0.3	321.8	67
5	3.4	0.4	1.08	0.3	326.6	37
6	3.7	0.6	1.12	0.2	297.2	41
7	3.5	0.5	1.06	0.1	303.4	43
8	4.1	0.6	1.08	0.4	322.2	25
9	3.7	0.7	1.16	0.1	314.4	67
10	3.7	0.9	1.16	0.1	316.2	66

*5 observations per sample

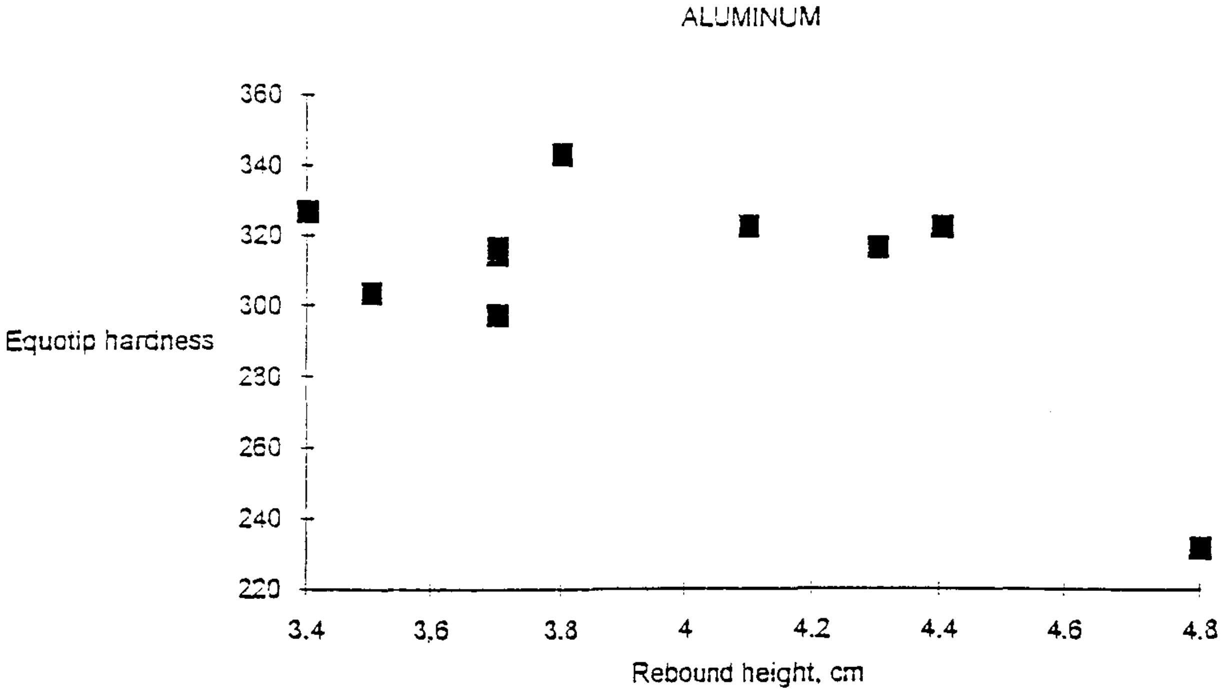

Figure 1

REFERENCES:

1. Boyer, H., Hardness Testing, Materials Park, OH: ASM Publication, 1987
2. Landau, D., Hardness, New York: The Nitralloy Corporation, 1943
3. Gilbert, B., Shore Scleroscope Hardness Tests Made on Mohs' Scale Minerals from Talc to Quartz, Inclusive, Urbana, IL: Department of Mining and Metallurgical Engineering, 1954
4. ASTM E 448-82, "Standard Practice for Scleroscope Hardness Testing of Metallic Materials", p 560-563
5. Widener, E., "It's Hard to Test Hardness", 1991 ASEE Annual Conference Proceedings, p 577-580
6. Baumeister, T., et al. Mark's Standard Handbook for Mechanical Engineers, McGraw-Hill Book Company, 1978

GRAPHING TECHNIQUES FOR MATERIALS LABORATORY USING EXCEL

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Graphing Techniques for Materials Laboratory Using Excel

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Elkhart, Indiana

KEY WORDS: Spring rate, stress concentration, endurance limit, column buckling, linear regression, slope-intercept, logarithmic graphing.

PREREQUISITE KNOWLEDGE: Students should have a course in computer literacy and some knowledge in strength of materials including stress-strain, cyclic loading, and column buckling.

OBJECTIVES: To introduce the use of Microsoft Excel spreadsheet for data reduction and evaluation of experimental results. This paper is intended to serve as a tutorial with a number of sample experiments on a strength of materials course.

INTRODUCTION

Engineering technology curricula stress hands on training and laboratory practices in most of the technical courses. Laboratory reports should include analytical as well as graphical evaluation of experimental data. Experience shows that many students neither have the mathematical background nor the expertise for graphing.

This paper briefly describes the procedure and data obtained from a number of experiments such as spring rate, stress concentration, endurance limit, and column buckling for a variety of materials. Then with a brief introduction to Microsoft Excel the author explains the techniques used for linear regression and logarithmic graphing.

GENERAL GRAPHING PROCEDURE

The concepts one needs to know on Microsoft Excel worksheet are shown in figure 1. Arrange the data such that all the X values are in the first column of the data area to be selected for graphing. The first row of data is usually column headings and is displayed in the legend. The second column is the Y values for the first curve and the third column is the Y values for the second curve and so on.

Highlight the data to be graphed and select the type of graph by clicking on FILE _ NEW _ CHART _ OK. A bar graph is displayed as default. Now GALLERY shows up in the menu bar. Select GALLERY _ X (select an appropriate number for the desired type of graph for X)

To add legend highlight the data table including the legend and click on the CHART menu when a dialog box is displayed. Click on ADD LEGEND.

To add text for title and axes click on
CHART _ ATTACH TEXT _ CHART TITLE _ OK. A set of squares appear on the graph. Type the title,

ENTER, and then click the mouse with the arrow on the graph. Go back to chart menu and click on ATTACH TEXT and repeat the same procedure for CATEGORY AXIS and VALUE AXIS

Entering data, graphing techniques and printing of your work are explained step by step with the help of experimental data in the following. Experiments are selected to give readers a general overview of mostly used graphing assignments.

EXPERIMENTS

Spring Rate:

Spring rate is the amount of load that is required to compress the spring per unit of deflection. The data table shows the force(lb) used to compress a spring causing a reduction in length(in) recorded as deflection.

This is an example of a set of curves including legend, title, and axes. This graph also demonstrates the linear regression technique using the LINEST function.

Worksheet 1. Spring Rate:
Click the mouse on FILE _ NEW - WORKSHEET _ OK
Enter title at the top, pick cell C3 and highlight C3:D3 with the mouse, change the font style, font type, and size as follows:
FORMAT _ FONT _ BOLD _ SIZE 10 _ OK

Create column headings
Select cell and type text using appropriate font settings.

Enter data:
For the graph start column with zeros.Type the load data in A6 to A11 and spring A deflection data in B6 to B11.

To change the precision of the displayed value highlight the column, click FORMAT _ NUMBER, position the cursor at the CODE bar and type in 0.000 _ OK.

Slope-Intercept Formula / Linear Regression:

Assign names to columns
Select the cell and highlight the selected column (do not include zeros), click
Click on FORMULA _ DEFINE NAME _ type in name (example: A for column A) _ OK

Create title for slope-intercept in the cells as shown:
To enter the slope-intercept function LINEST for spring A highlight the two adjacent cells below the slope intercept titles, type the formula for spring A exactly as
=LINEST(B,A) where B is the name of the second column and A is the name of the first column as

defined earlier. Press and hold CONTROL + SHIFT and press ENTER. The slope and intercept values are now displayed in the selected cells.

Linear Regression:
The linear regression (L.R.) column for spring A is created by entering the slope - intercept formula in cell C6 as =$A$14*A6+$B$14 _ ENTER, once entered click EDIT _COPY
highlight cells C7:C11 _ ENTER. (C7:C11 means cell C7 through cell C11)

Repeat for spring B using column E for load, column F for deflection, and column G for slope-intercept formula.

Graphing Spring Rate (figure 2):
Highlight A5:C11, press and hold CONTROL and highlight F5:G11
Select FILE _ NEW _ CHART _ OK, bar graph is displayed, click on GALLERY, pick #5 _ OK.

at CHART menu click on ADD LEGEND
at CHART menu click on ATTACH TEXT _ CHART TITLE _ OK
type title SPRING RATE _ ENTER, click mouse
click on CHART _ ATTACH TEXT _ CATEGORY AXIS _ OK
type LOAD(lb), click mouse
click on CHART _ ATTACH TEXT _ VALUE AXIS _ OK
type DEFLECTION(in) _ ENTER, click mouse
click on CHART _ ADD ARROWS, position mouse at the arrow ends, press and hold and move to the desired location
type SPRING A for the first spring
repeat for spring B

Stress Concentration
The presence of shoulders, grooves, keyways, threads, or any type of discontinuities in machine elements results in modification of simple stress distribution.

In this experiment force is applied at the end of a cantilever beam with a hole creating stress proportional to the deflection. Purpose of this experiment is to measure stress level at various distances from the hole. Strain gages are installed at the desired locations on the beam for stress readings. The experimental data is displayed in the given table.

Worksheet 2. Stress Concentration:
Type STRESS CONCENTRATION in B2 _ ENTER
type Gage #1, Gage #2, Gage #3, and Gage #4 in B4, C4, D4, and E4
type data table as given in A5:E9

Graphing Stress Concentration (figure 3):

highlight A4:E9
click on FILE _ NEW _ CHART _ OK
click on GALLERY _ LINE _ 5 _ OK
click on CHART _ ADD LEGEND _ OK

click on CHART _ ATTACH TEXT _ CHART TITLE _ OK, click
type STRESS CONCENTRATION _ ENTER, click
click on CHART _ ATTACH TEXT _ CATEGORY AXIS _ OK
type DEFLECTION(in) _ ENTER, click
click on CHART _ ATTACH TEXT _ VALUE AXIS _ OK
type STRESS(psi) _ ENTER, click

Printing :
click on FILE _ PRINTER SET UP
click on SET UP _ LANDSCAPE _ NLQ _ OK _ OK
click on FILE _ PRINT _ PREVIEW _ OK
click on ZOOM for close observation
click on PRINT

Endurance Limit:

Endurance limit is a material property expressed in terms of stress(psi) that can be endured during cyclic loading regardless of the number of applied cycles. This stress value is also called the fatigue limit.

In this experiment a 1/4" diameter grooved shaft is rotated under load till failure. The data table shows the groove distances from the loading point X, Groove diameter d, Load plus the weight of the fixture P, Accumulated number of cycles N, and the stress S.

Worksheet 3. Endurance Limit:
Type in the worksheet as shown, calculate the stress values using P, d, X, and Kt (=1).

Select cell E24
type formula for stress S = 32*P*X/(3.14*d*d*d) in the worksheet as
=32*C24*A24/(3.14*B24*B24*B24) and click on E24,
stress value is displayed in E24
select E24 _ COPY, highlight E25:E29 _ ENTER, stress values are displayed in column E.

Graphing Endurance Limit (figure 4):
highlight D23:E28
FILE _ NEW _ CHART _ OK
GALLERY _ SCATTER _ 5 _ OK
Enter necessary texts for the graph and print.

Column Buckling

Long columns and the more slender short columns usually fail by buckling when critical load is reached. In this experiment the critical load for 1/4" diameter and 12", 16", 20", and 24" long rods is determined.

Worksheet 4. Column Buckling:
Type the title COLUMN BUCKLING DATA SHEET in B1
click on FORMAT _ FONT _ HELV BOLD _ OK
type EXPERIMENTAL in B2
type the data table exactly as shown.

If you highlight B3:E6 column B is considered as the category axis and if you highlight A3:E6 column A serves as legend with row 3 as category axis.

highlight A3:E3
click on FORMAT _ BORDER _ SHADE _ OUTLINE _ OK, click

Graphing Column Buckling (figure 5):

highlight A3:E6
click on FILE _ NEW _ CHART _ OK
click on GALLERY _ LINE _ 6 _ OK
click on CHART _ ADD LEGEND _ OK
click on CHART _ ATTACH TEXT _ CHART TITLE _ OK
type COLUMN BUCKLING _ ENTER, click
click on CHART _ ATTACH TEXT _ CATEGORY AXIS _ OK
type LENGTH(in) _ ENTER, click
click on CHART _ ATTACH TEXT _ VALUE AXIS _ OK
type LOAD(lb) _ ENTER, click

Follow the previous instruction for printing.

REFERENCES:

1. Eliason, A. L., *Microsoft Excel Student Edition*, Prentice Hall, 1993.
2. Denton, Hillsman, Roach, and Thelen, *Strength of Materials-Laboratory Manual*, Purdue University, Mechanical Engineering Technology, 1993.

Concepts You Need to Know

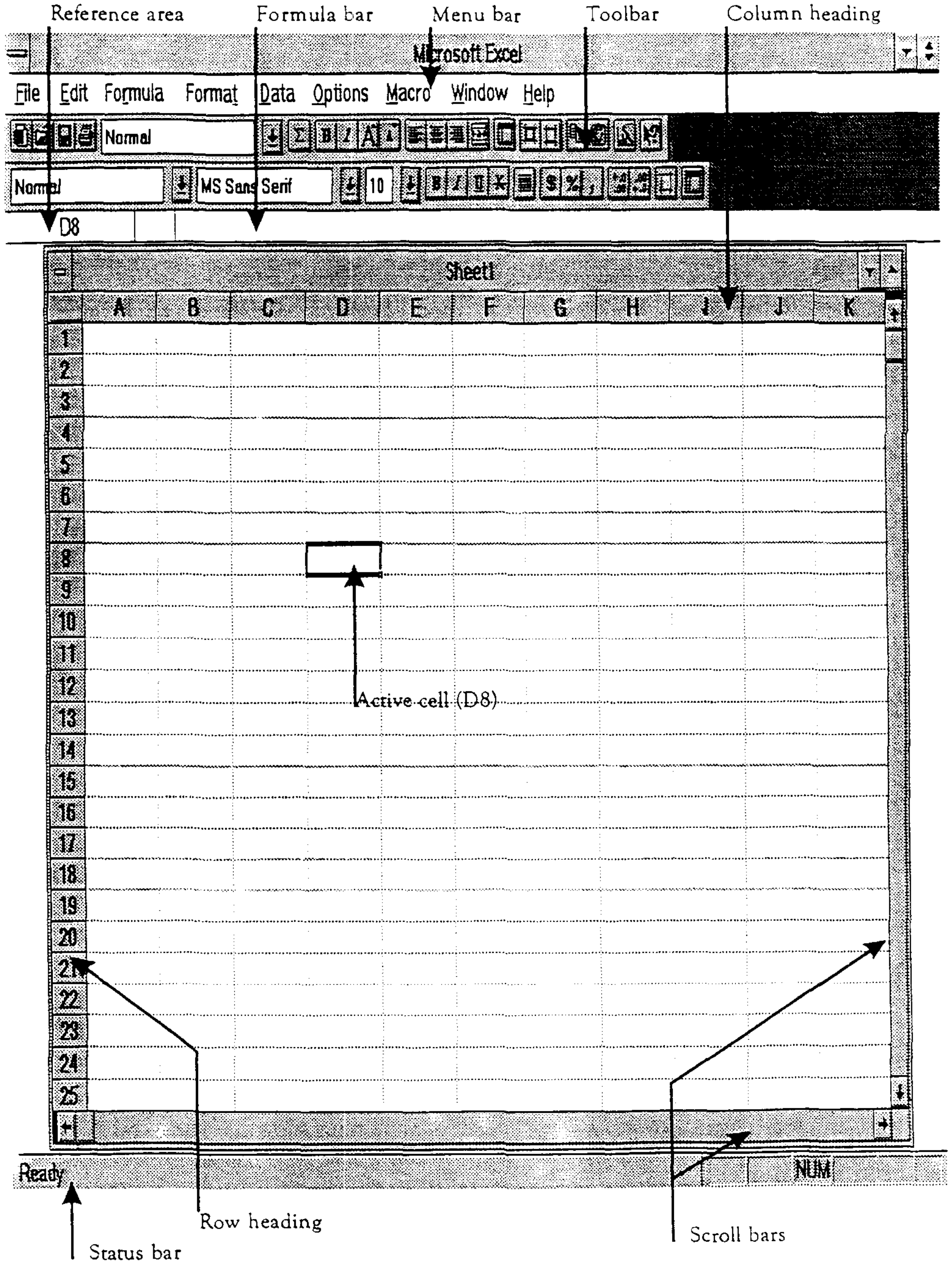
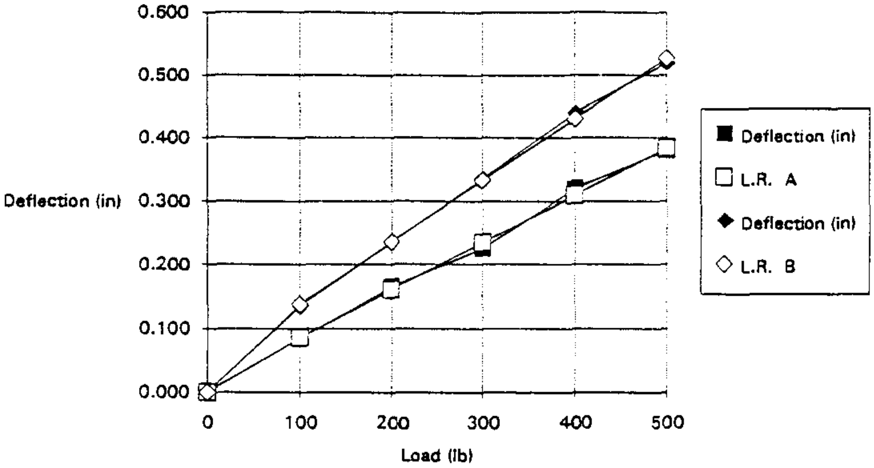


Figure 1

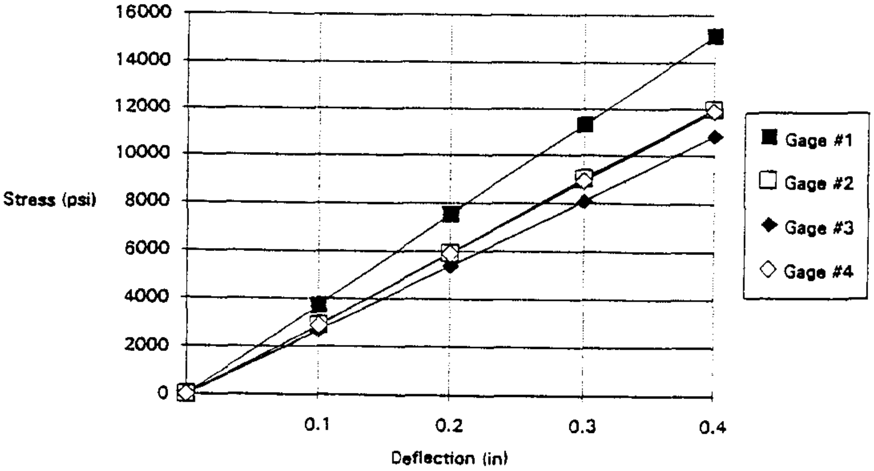
	A	B	C	D	E	F	G
1	Worksheet 1. Spring Rate:						
2							
3			SPRING RATE				
4							
5		Deflection (in)	L.R. A			Deflection (in)	L.R. B
6	0	0.000	0.000		0	0.000	0.000
7	100	0.085	0.086		100	0.135	0.138
8	200	0.165	0.161		200	0.235	0.236
9	300	0.225	0.235		300	0.335	0.333
10	400	0.320	0.310		400	0.440	0.431
11	500	0.380	0.384		500	0.520	0.528
12							
13	Slope	Intercept			Slope	Intercept	
14	0.000745	0.0115			0.000975	0.0405	

Figure 2. Spring Rate



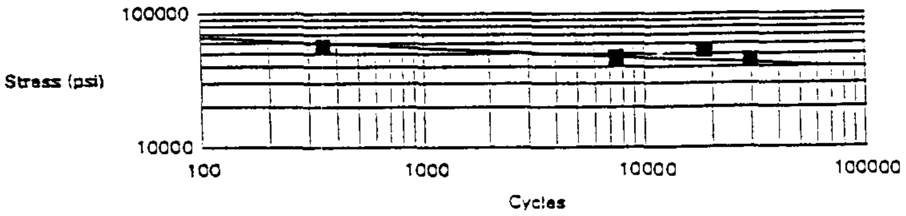
	A	B	C	D	E
1	Worksheet 2. Stress Concentration				
2					
3			Stress Concentration		
4		Gage #1	Gage #2	Gage #3	Gage #4
5		0	0	0	0
6	0.1	3730	2900	2660	2870
7	0.2	7520	5930	5380	5900
8	0.3	11320	9030	8070	8920
9	0.4	15120	12010	10840	11900

Figure 3. Stress Concentration

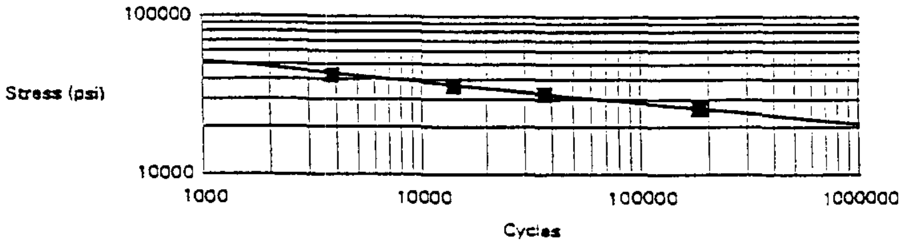


	A	B	C	D	E
1	Worksheet 3. Endurance Limit:				
2					
3			ALUMINUM		
4	X	d	P	N	S
5					
6	20.6875	0.294	6.80916	346	56491.08
7	18.1875	0.3	6.80916	7406	46743.7
8	15.6875	0.274	6.80916	18175	52919.52
9	13.1875	0.274	6.80916	29404	44486.13
10	10.6875	0.274	6.80916		
11					
12			BRASS		
13	X	d	P	N	S
14					
15	20.6875	0.298	5.3021	3748	42240.35
16	18.1875	0.3	5.3021	13947	36398
17	15.6875	0.298	5.3021	36871	32031.2
18	13.1875	0.301	5.3021	179778	26129.51
19	10.6875	0.299	5.3021		
20					
21			STEEL		
22	X	d	P	N	S
23					
24	19.25	0.279	13.2614	1725	119792
25	16.75	0.279	13.2614	3878	104234.6
26	14.18	0.279	13.2614	7481	88241.59
27	11.75	0.279	13.2614	16259	73119.79
28	9.28	0.279	13.2614	53230	57749.08

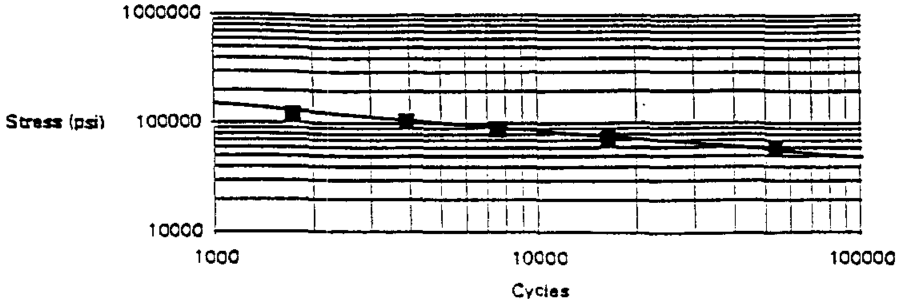
Figure 4. Endurance Limit for Aluminum

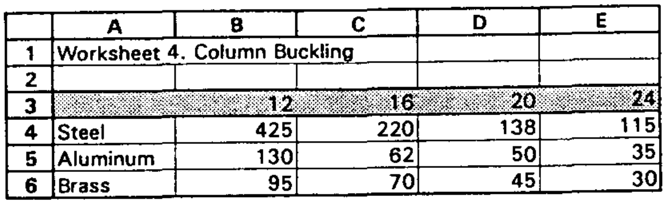


Endurance Limit for Brass



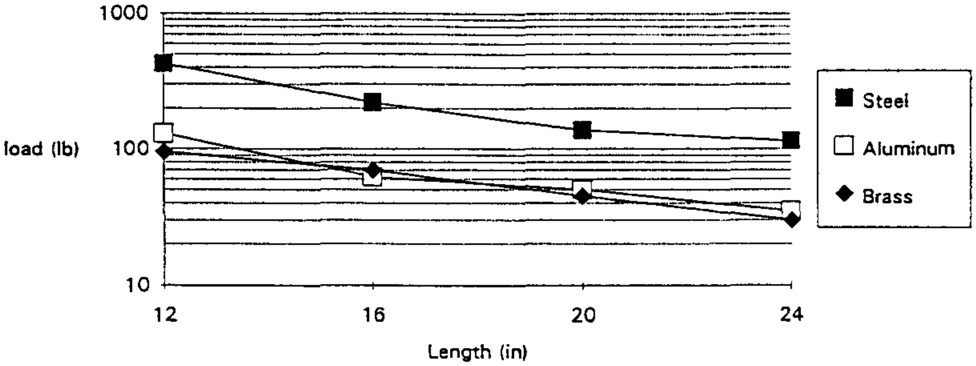
Endurance Limit for Steel





	A	B	C	D	E
1	Worksheet 4. Column Buckling				
2					
3		12	16	20	24
4	Steel	425	220	138	115
5	Aluminum	130	62	50	35
6	Brass	95	70	45	30

Figure 5. Column Buckling



AN AUTOMATED DIGITAL DATA COLLECTION AND ANALYSIS SYSTEM FOR THE CHARPY IMPACT TESTER

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and

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An Automated Digital Data Collection and Analysis System for the Charpy Impact Tester

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Abstract: The standard Charpy Impact Tester has been modified by the addition of a system of hardware and software to improve the accuracy and consistency of measurements made during specimen fracturing experiments. An optical disc, light source and detector generate signals that indicate the pendulum position as a function of time. These signals are used by a computer to calculate the velocity and kinetic energy of the pendulum as a function of its position.

Key Words: Charpy, fracture energy, impact testing, automated data collection, experimental errors.

Prerequisite knowledge required:

To meet the first objective, the student should be familiar with fracture testing and very basic use of computers. To meet the second and third objectives, the student should be familiar with basic analog and digital electronics, computer interfacing, experimental error analysis and simple BASIC programming.

Objectives:

This work was undertaken to explore three areas of materials experimentation. The most obvious first objective was to add instrumentation to significantly improve the accuracy of fracture measurements made with a Charpy impact tester. Measurements made with the uninstrumented Charpy are performed by visually observing the maximum height achieved in the upswing of the pendulum after the fracture is completed. Depending on the energy required for fracture, the final position of the pendulum may be anywhere on the 26 inch long scale inscribed on the machine's circumference. Since the approximate final point where the visual observation is to be made is unknown, it is difficult to make an accurate determination of its final position. The uncertainty of carefully made visual measurements is approximately 1 to 2 in-lb[1] (.113-.226 J).

The second objective was to provide a platform for students to discover and study problems inherent in all instrumentation and automated data collection systems. This system has three modular sections: the sensor and its analog conditioning electronics; the digital circuitry and digital input-output interface to the computer; and the computer program (algorithmic technique) which accumulates, evaluates and displays the experimental data. Each of these modular areas (and problems which must be addressed within them) can be studied in isolation.

The third objective was to provide a mechanism to allow students to redesign part of the instrumentation to study different aspects of the fracturing experiment and to minimize certain errors in making and evaluating measurements. The initial configuration provides a measurement of the pendulum's kinetic energy prior to and subsequent to the fracture of the sample. The three modules were designed to optimize these two measurements. If one wanted to study the process of the fracture as it occurred, changes to each of the three modules would be necessary. This system was designed to make it easy for students to experiment with various alternatives to each of the three modules.

Equipment and supplies:

Needed supplies include: a Charpy impact tester, an Intel based personal computer, a digital input-output interface card; and various discreet physical and electronic components (described in detail below).

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[1] The units of in-lb are used because the Charpy is labeled in in-lb. SI units are included parenthetically.

Procedure:

Modification of the Charpy - An optical position detection system was constructed and attached to the Charpy. This system consists of three sections: the optical position disk, the light sensor, and the signal conditioning circuitry. The optical position disk is an aluminum disk mounted on the pendulum axis and rigidly attached to the pendulum arm. Physical details of this disk are given in Appendix 1. The light sensor section consists of a light emitting diode and photosensitive transistor situated such that the optical position disk can allow or interfere with the light link between them. The signal conditioning circuitry serves to drive the light sensor section and to generate a digital TTL signal suitable for input to the computer input-output interface card. Physical and electrical details of the light sensor and signal conditioning circuitry are given in Appendix 2.

The signal generated by the experimental apparatus is coupled to an Intel based personal computer through a generic interface card. The computer program which controls the experiment directs the operator in the setup of the Charpy, acquires the experimental data, measures the times between signal pulses, computes and displays physical parameters including velocity and kinetic energy of the pendulum just before the fracturing impact and just after the fracture.

With an unmodified Charpy, an experiment is conducted by raising the pendulum to a standard position to the right of center and then releasing it. The pendulum swings down, breaks the sample and continues up to the left of center. The observer watches and notes the value on the inscribed scale at the position where the maximum height is reached by the pendulum to the left of center.

With the modified Charpy, the energy used to break the sample is determined by measuring the velocity of the pendulum's mass immediately prior to and subsequent to the fracturing of the sample and then computing the loss in total energy.

Theory - The work done in fracturing the sample can be found by measuring the total energy lost by the mass affecting the fracture. The total energy in the pendulum's mass is:

$$1/2mv^2 + mgy \qquad (1)$$

where m is the pendulum's mass, v is its velocity, and y is its height above its minimum altitude. The measurement of initial velocity and final velocity were made very near the pendulum's minimum altitude and equally displaced from that position. y_f and y_i are very small and are equal. Therefore, the total energy used to break the sample is:

$$1/2m\left(v_f^2 - v_i^2\right). \quad (2)$$

The signal generated by the optical circuit is of the form below:

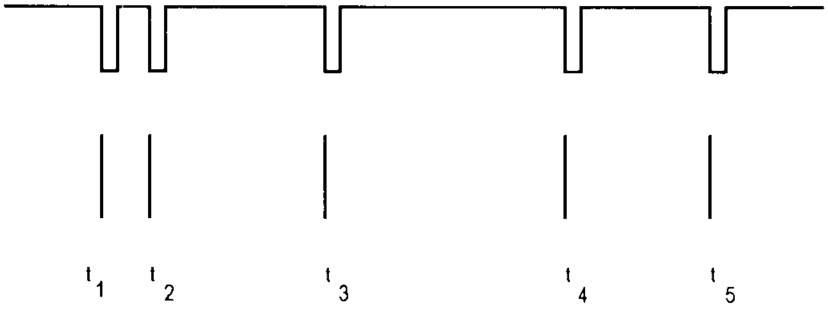


Figure 1

The computer looks for falling edges in the incoming signal. The first edge at t_1 starts the computer's timing function. The computer measures the time between t_2 and t_3 which are generated by two slits just prior to the pendulum contacting the sample. The breakage occurs during the interval between t_3 and t_4. The computer measures the time between t_4 and t_5 which is generated by two slits just subsequent to the break. Since the position of the pendulum's mass is accurately known when each of the slits generates its signal, the velocities of the mass can be accurately computed.

Analysis of data collection errors - The principal source of measurement error in this experiment is the quantitizing error in measuring time. The computer must count time in discreet steps. If an event occurs between count 5 and 6, it is not possible to know whether the event occurred just after the counter became 5 or just before the counter became 6. The impact of this quantitizing error depends on the size of the time steps being counted. If the counter increments every 0.1ms, the error would be an order of magnitude greater than if the counter increments every 0.01ms.

In our case, the counter is incremented every 0.021 ms. Physical dimensions of the modified Charpy are such that 676 counts occur in the time between t_2 and t_3. Since the actual time of t_2 could be almost 1 count from the time it is detected and the actual time of t_3 could be almost 1 count from the time it is detected, the interval t_2-t_3 as measured by the computer could differ from reality by as much as 2 counts or 0.3%.

Several interesting student projects could be designed around the determination of the effect of other errors introduced by physical aspects of the machine such as: accuracy of the position of the mass at t_2, t_3, t_4 and t_5; accuracy of measurement of the pendulum's mass.

***Modifications for equipment improvement* -** The next logical step for this experiment is to add more optical slits and to make them thinner and closer together. A velocity measurement could be made as each slit passes thereby giving a graph of velocity (energy) as a function of position as the pendulum mass breaks the sample.

***Conduct of fracture experiments* -** Experiments which have been successfully completed include measurement of the fracture energy of wood samples both with and against the grain; the fracture energy of wood samples as a function of water content; and the fracture energy of wooden samples which have been fractured and then glued together with various types of glue. Wood samples were used because they are readily available, easily worked and inexpensive. Polymers, ceramics and composites could also be tested by this method.

Sample data sheet:

Calibration Check: (Run with no sample in place)

Initial speed:	3.534 m/s
Initial kinetic energy:	5.666 J
Final speed:	3.534 m/s
Change in KE	$-1.02\ (10^{-3})$ J

Sample Type: Pine (white)
Orientation: with the grain
Dimensions: 1.35 x 1.25 x 5.5 cm

Sample #	Fracture Energy (J)
1	2.00
2	2.57
3	2.28
4	1.54
5	2.00
6	3.43
7	2.28
8	1.99
9	2.25
10	1.99

Instructor notes:

Conducting experiments on the unmodified Charpy was frustrating to most students. Two students were required, one to accurately position the sample and to release the pendulum. The second student would observe the highest position of the pendulum after it fractured the sample. Typically, several trial fractures were necessary to determine the approximate location of the maximum position of the pendulum after the fracture.

With the addition of the automated data collection system to the Charpy, experiments can be conducted by a single student in less than half of the time previously required. The accuracy of the measurements achievable has been improved ten-fold.

References:

Weigman, Bernard J. and Spiegel, F. Xavier: "An Automated Data Collection System for a Charpy Impact Tester ", NASA CP-3201, 1992.

Eggebrecht, L.C.: *Interfacing to the IBM Personal Computer*, Howard W. Sams & Co.

Thompson and Kuckes: *IBM-PC in the Laboratory*, Cambridge University Press.

Krutz, Ronald L.: *Interfacing Techniques in Digital Design*, Wiley.

Stone, Harold S.: *Microcomputer Interfacing*, Addison Wesley.

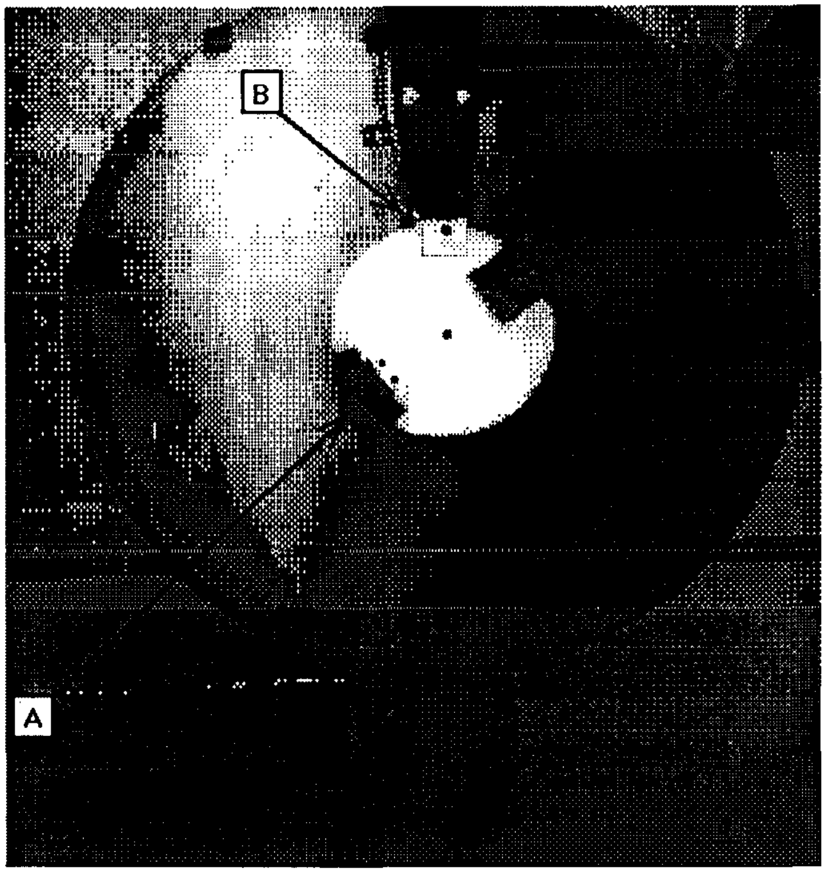
Cripps, Martin: *Computer Interfacing - Connection to the Real World*, Edward Arnold Publisher (Hodder & Stoughton).

Spiegel, F. Xavier and Weigman, Bernard J.: " An Automated System for Creep Testing" , NASA CP-3151, 1991.

Fellers, William O.: *Materials Science, Testing and Properties for Technicians*, Prentice Hall, 1990.

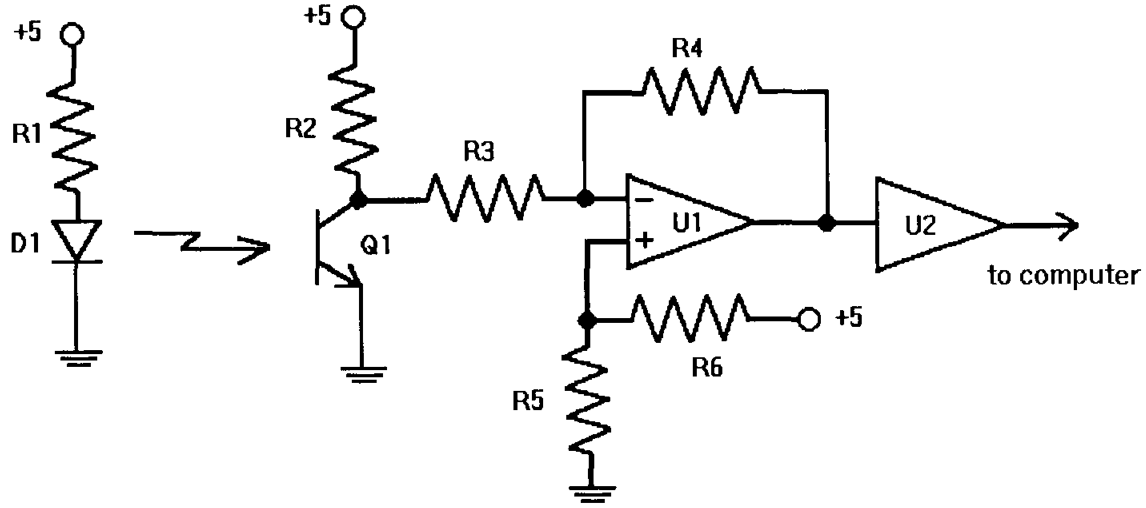
Sources of Supplies: The resistors and integrated circuits used here should be common stock to any electrical engineering/ computer science department. The IR emitter/receiver pair is available through Radio Shack. The mounting saddle for the light link is tooled from scrap wood. The optical disk is made from ordinary 0.125" sheet aluminum.

Appendix 1



The optical system consists of the white metal disk centered on the pendulum's axle and rigidly attached to the pendulum, the light source/sensor which straddles the disk (B), and the interchangeable slit plate (A). When the pendulum is near the bottom of its swing, the slit plate will be passing through the light source/sensor. The pulse train issued by the light sensor is routed to conditioning circuitry (Appendix 2) and then to the digital input board in the PC.

Appendix 2 - Light Sensor and Signal Conditioning Circuitry



R1 = 270 Ω
R2 = 2000 Ω
R3 = 3000 Ω
R4 = 30000 Ω
R5 = 3000 Ω
R6 = 3000 Ω
U1 = LM324 Quad OpAmp
U2 = SN7404 Hex Inverter
D1/Q1 = Generic IR source/receiver pair (Radio Shack)

PEEL PROPERTIES OF A PRESSURE SENSITIVE ADHESIVE

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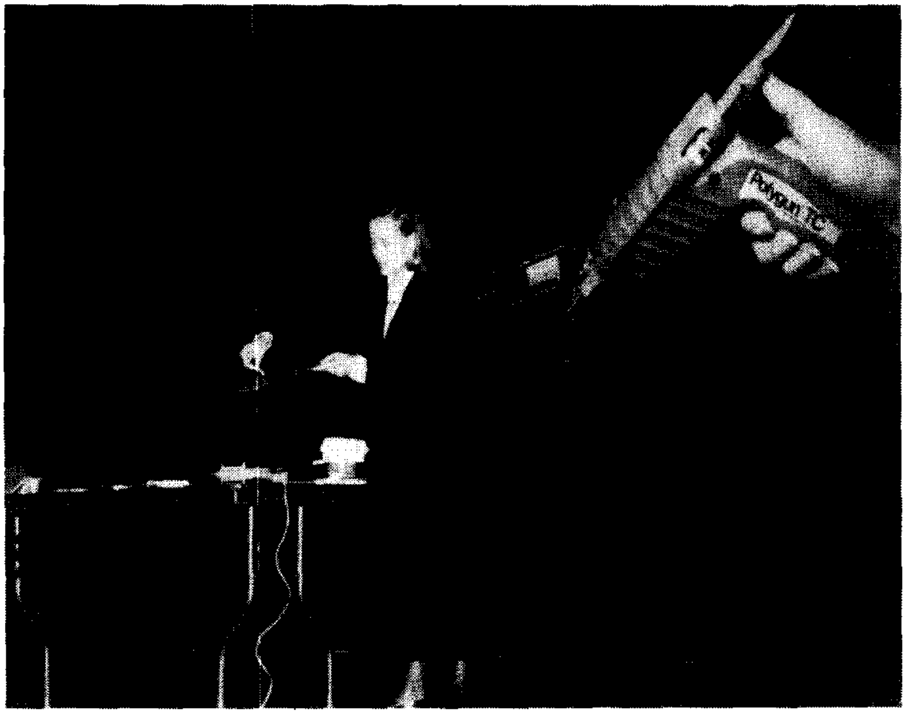

Elizabeth Edblom
3M Center

Peel Properties of a Pressure Sensitive Adhesive

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St. Paul, Minnesota

Key Words: Pressure Sensitive Adhesive (PSA), peel

Prerequisite Knowledge: Knowledge of basic adhesive terminology is helpful but not necessary.

Objective: To observe some of the factors affecting peel force on adhesive samples prepared by the student such as adhesive composition, adhesive coating weight, peel rate, and dwell time . To measure the peel rate/peel force response for a standard adhesive tape.

Equipment:

1. solution of synthetic rubber
2. solution of tackifier resin
3. coating rods
4. drying oven approved for organic solvents
5. 1 or 2 mil polyester film - #PE-1 or PE-2
6. one inch tape specimen cutter - #SC-100
7. 4.5 lb rubber roller - #HR-101
8. 2 x 6" stainless steel test panels - #TP-26
9. analytical balance
10. paper binder clip
11. plastic beaker
12. string
13. steel BB balls
14. stop watch
15. ruler
16. Magic tape
17. organic solvents; toluene, heptane, isopropyl alcohol, acetone, and ethyl acetate
18. computer and computer graphing program or graphing paper

Introduction:

Peel resistance is a function of many factors, including adhesive composition, strength of the interfacial bond, adhesive thickness, backing stiffness, peel angle, peel rate, wet out or dwell time, temperature, and application pressure.

Resistance to peel is defined as the force required to peel a strip of adhesive tape from a rigid surface. Peel force, however is not equivalent to the forces of adhesion at the interface, rather it is a combination of adhesion forces and the work done on the adhesive. It is not possible to separate these two forces. None-the-less, of the frequently used pressure sensitive adhesive tests, resistance to peel produces more information about expected performance than do tests such as tack and creep.

Observe a piece of masking tape on a steel panel as it is slowly peeled from the panel. The adhesive releases by forming individual strands. These strands are stretched and drawn until sufficient elastic energy is stored and the adhesive separates from the interface. Adhesion forces typically are weak Van der Waals attractions. Essentially all the work done in peeling the tape goes into stretching the adhesive.

Behavior of an adhesive can be separated into elastic and viscous behavior, represented in Figure 1 by a spring and a dash pot filled with a viscous material, respectively.

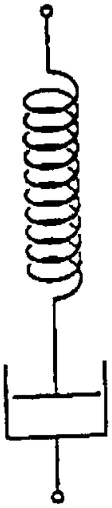

Figure 1. Spring and Dash Pot Model.

Viscoelastic properties are dependent upon the speed at which the adhesive is deformed. Since this experiment focuses on the effect of peel rate on peel force let's consider what happens to the model as it is pulled (peeled) at different rates. When pulled slowly the adhesive exhibits liquid-like behavior; the dash pot moves but the spring does not deform. Since no energy is stored in the spring, the adhesive splits when exposed to these slow peel forces. If the system is pulled rapidly, the spring stretches. When the movement is very fast, the viscous material does not have time to deform. This solid-like behavior results in a tape pop off. At intermediate peel rates both components play a role. It is at these rates that the highest peel forces are attained. In this experiment the students will control peel force and measure the resulting peel speed.

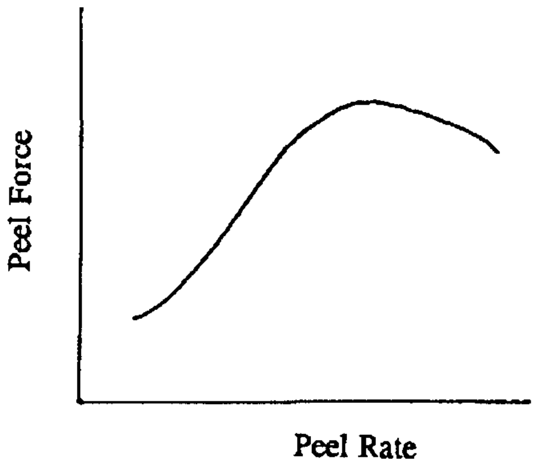

Figure 2. Generalized Peel Force vs. Peel Rate for a PSA.

There are numerous American Society of Testing and Materials (ASTM) for peel adhesion. This experiment will focus on the 180° peel as it is easy to maintain a 180° peel angle without a special fixture.

Procedure:

Compounding and Coating:

Simple rubber/resin pressure sensitive adhesives can be compounded by blending a rubber solution with a tackifier resin solution. Use the solutions of Kraton®-1107 elastomer and Wingtack Plus resin prepared by your instructor to compound your adhesives. Note the percentage solids of each solution (weight in grams of material/100 grams solvent). Compound 100 gm of the following three adhesives: 40/60, 50/50, and 60/40 parts rubber/resin based on percentage solids.

To coat the adhesive pour a small amount of solution onto the film and at a slow and even rate spread the adhesive by rolling the coating rod the length of the film pushing the adhesive into a thin flat layer. The coating step is critical as ridges and variations in adhesive thickness will affect peel results. After coating the handspread the rod should be cleaned with heptane. At this stage the adhesive contains solvent. To dry the adhesive, place the coated polyester sheets in an oven approved for organic solvents at 150°F for 20 minutes. Taping the handspreads to a stiff board will prevent them from wrinkling during the drying step. After removing samples from the oven let the tape cool to room temperature.

Now you are ready to measure your adhesive coating weight. Cut a piece of backing film and tape of equal size, approximately 0.10 m^2. The difference in weight of these two samples is your coating weight in gm/m^2. A coating weight of about 25 gm/m^2 is a good place to begin.

Peeling the Tape Sample:

Peel properties of PSA tapes are influenced by how the tape sample is cut and applied to the substrate. To minimize cut variability on a cardboard surface, use a one inch tape specimen cutter to cut strips of tape about 6" in length. Use extreme caution when cutting tape samples with the one inch cutter. Lay the tape adhesive side down on a clean steel panel. Try to place the tape strip straight on the panel as angling of the sample will affect your results. Acetone or isopropyl alcohol can be used to remove residue from the metal surface. Use the 4.5 lb roller with a minimum of pressure to evenly smooth the tape to the panel. Allow the tape to wet out on the panel for 30 seconds.

Fasten a paper binder clip to one end of the tape sample and hook the string suspending the plastic beaker loaded with BB's to the clip. A 200 gm load of BB's is a good place to start. At the start of the test the beaker should hang below the panel, see Figure 3. Peel the tape at a 180° angle off of the steel panel and note the time it takes for the tape to peel a convenient distance. Marking a distance on the steel panel before beginning the peeling experiment will help improve the accuracy of the peel speed determination. Record force in grams (weight of BB's + beaker) and peel speed (peel distance/time).

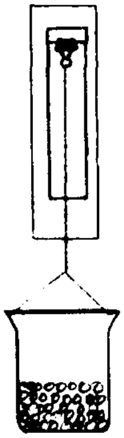

Figure 3. Arrangement for Peel Test.

Choose a different coating rod and prepare a second handspread of your adhesive with a higher coating weight and repeat the peel resistance test. What happens to the peel force? Try a lower coating weight. Next try coating the other adhesive formulations. What happens to the peel force as the percentage of tackifier in the formulation increases? Finally, increase and decrease the dwell time between tape application and peeling. Is the effect the same for all coating weights of adhesive and all formulations? Enter your data into a table similar to the one shown below. Include a section to describe any observations you made during the peeling of each tape sample.

Adhesive Composition	Coating Weight g/m^2	Roll Down Pressure lb	Dwell Time sec	Peel Force g	Peel Rate cm/sec
40/60 K-1107/W+		4.5	30		
50/50 K-1107/W+		4.5	30		
60/40 K-1107/W+		4.5	30		

Effect of Peel Rate on Peel Force:

Preparing reproducible handspreads is difficult. For this reason commercial rolls of Magic Tape will be used in the determination of the effect of peel rate on peel force. Coating weight must be determined for the tape. Cut a sample of tape and record its weight. To remove the adhesive layer, place the tape in a jar of ethyl acetate for 20-30 minutes. Remove the film from the solvent and scrape any remaining adhesive off the backing using a razor blade. Dry the film in the drying oven for 10 minutes. Weigh the film and calculate the coating weight.

Make at least ten different peel rate measurements by varying the load of BB's in the plastic beaker. Measure each force point three times. Use the data table to record your results. Calculate both average and standard deviation peel rate and use those values in a plot peel force vs. speed. Does it look like the plot shown in Figure 2?

Comment on any differences in the manner in which the tape samples peeled as peel rate increased. Were the peels smooth at all speeds? Were any adhesive residues left on the panels? What would you expect to happen if the tape were allowed to peel at an infinitely slow rate?

Notes to the Instructor:

1. Kraton® polymers from Shell Chemical Company are especially suitable for solvent based compositions. With gentle agitation it may take several hours for a Kraton® rubber to completely go into solution. Solutions should be made up ahead of time at 40-50% solids using toluene or a 40/60 blend of toluene/heptane. Kraton®-1107 has been chosen as the

elastomeric material for this experiment. Information for more elaborate adhesive formulation can be found in Shell's adhesive bulletins SC68: Selector Guide and SC198: Adhesive Formulating Guide. To request samples of literature from Shell call 800-323-3405. Tackify the Kraton®-1107 with the Wingtack Plus resin from Goodyear Chemicals. The resin solution should also be prepared ahead of time at a concentration of 40-50% solids in either solvent listed above. Contact Mike Martin at Goodyear at 216-796-7482 for a resin sample.

2. RDS coating rods, equipment item 3, are available from R.D. Specialties (560 Salt Road P.O. Box 206, Webster, NY 14580, 716-265-0220) in a variety of rod and wire sizes. A starter set of six 1/4" diameter rods is available for $65.00. This set would give students the capability of coating a broad range of thicknesses.

3. Equipment items 5-8 can be ordered from Chemsultants International Network, 9349 Hamilton Drive, P.O. Box 1118, Mentor, Ohio 44061-1108, 216-352-0218.

4. Use extreme caution with the one inch cutters. Sharp razor blades produce a cleaner sample edge, but may also be responsible for deep cuts in the students fingers.

5. Organic solvents must be used in a hood or well vented area.

References:

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SYMMETRY AND STRUCTURE THROUGH OPTICAL DIFFRACTION

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Symmetry and Structure Through Optical Diffraction

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KEY WORDS: diffraction, symmetry, laser, optics

PREREQUISITE KNOWLEDGE: Basic diffraction from introductory physics and exposure to symmetry concepts.

OBJECTIVES: To understand diffraction and how it relates to symmetry and structure through direct observation. To provide an exploratory environment in which students can readily pose and answer "what-if?" type questions concerning diffraction. To provide an introduction to some useful computational tools. Other offshoots for further investigation would include optics and principles of laser operation.

EQUIPMENT AND SUPPLIES:

(1) computer and laser printer with
(2) drawing software such as CrystalPaint or Claris Cad for creating patterns
(3) optical bench including:
    He-Ne laser
    beam expander
    slide holder
    two lenses
    image screen
(4) instant slide film (such as Polaroid PolaBlue 35mm)
(5) instant slide processor (Polaroid 35mm AutoProcessor)
(6) 35mm camera and tripod

INTRODUCTION:

In this paper, we describe an activity which was created as part of a new introductory materials science course at the University of Washington. This course differs significantly from traditional versions in both content and pedagogy. Our new course was developed within the framework of the ECSEL program at the University of Washington. ECSEL, the Engineering Coalition of Schools for Excellence and Leadership, is a coalition of seven universities that has been funded by the National Science Foundation to undertake a twofold mission:

(1) to dramatically improve the quality of the engineering undergraduate experience

(2) to significantly enhance the participation of women and under-represented minorities.

The other schools in the ECSEL coalition are Howard University, Morgan State University, City College of New York, Massachusetts Institute of Technology, Pennsylvania State University, and the University of Maryland.

In our introductory materials science subject, the changes have been major [1]. The format of the course moved from the traditional engineering emphasis on lectures, problem sets and exams, to a very heavy emphasis on student-initiated projects and research, undertaken both individually and in large and small groups. The course also places a heavy emphasis on developing experience in oral and written presentation skills. In order to allow such an innovative exploratory classroom environment to flourish, we found it necessary to dramatically change our grading strategies as well. We developed a journal-based assessment technique, which simultaneously offers many educational and social advantages [2]. The traditional curriculum has been replaced by a series of nine modules. These modules, though largely independent, share a number of powerful unifying conceptual themes, in particular the closely related ones of symmetry and thermodynamic driving forces. The modules are entitled "Symmetry and Strength", "Ferroelectrics", "Solidification", "Diffraction and the Determination of Structure", "Mechanical Properties of Materials", "Glass", Biological Materials", "Semiconductivity and Superconductivity" and "What's in it and Why?".

Essential to the philosophy of our course is that the ratio of structured activity to free-form student-initiated project-based activity is tailorable to an individual student's needs and aesthetics. There is a common structure to each of the modules listed above, however. The common components of each module include an "idea sheet", which can act as a launching pad for students who have a hard time getting started on projects on their own, a reference sheet, some curricular offshoots which we call "illuminating sidetracks" and an optional problem set, for those who find that medium useful. For each module, we have also prepared a structured laboratory activity. This flexible structure allows those students who feel more comfortable with the traditional format of homework, quizzes and exams to rely heavily on those activities, while those who prefer to explore their own ideas in depth can do so. The activity described in this paper is the structured lab activity portion of the module on "Diffraction and the Determination of Structure".

This activity focuses on the understanding of diffraction and its relationship to symmetry and atomic structure. During the lab, students design and create two-dimensional patterns exhibiting various symmetries using a variety of computational tools. These patterns are then made into slides and analyzed using optical diffraction. Patterns representing some two dimensional slices of common crystallographic structures are also analyzed in the lab. This lab activity allows students to use their creativity in the design of the patterns, as well as to experiment with different computer drawing/painting software. A major benefit to this lab is the student-directed activity it encourages, in contrast to a traditional lab, in which students follow an directive set by the instructor. Once the optical bench is set up, it is relatively easy for the students to use it on their own, exploring a wide variety of relatively sophisticated questions. Another advantage to the optical diffraction activity is that, unlike in the case of X-ray diffraction, students can actually <u>see</u> the pattern from which they are diffracting, and observe directly how changes in their patterns affect diffracted intensity. We believe that a more concrete understanding of diffraction, a topic difficult to visualize and to explain in lecture, is achieved with this approach.

Diffraction in its various manifestations is clearly one of the most important tools in materials science. In our optical diffraction activity, the patterns generated by the students can be arbitrarily complex, due to the variety of computational tools available, and are often very pleasing aesthetically. While most students choose relatively simple patterns, ambitious ones can experiment with multi-component structures, with variations in density, and with crystalline defects, opening up a far wider realm of materials science phenomena to their exploration. It is the relatively quick and painless turn-around time between making changes in structure and observing changes in diffracted intensity that makes this a valuable activity educationally.

The basic process occurring in optical diffraction by a laser source can be described in terms of Fraunhofer diffraction and interference.[3, 4] The light emitted from a laser is assumed to be a beam of parallel, monochromatic light that is coherent. When this light interacts with an object, the diffraction pattern produced is the Fourier transform of the object. The diagram below illustrates different diffraction patterns produced with variation in distance between scattering centers, as well as with variation of the scattering efficiency of the objects. Students find the mathematical concepts associated with diffraction difficult to visualize. Furthermore, as patterns become more and more complex, the mathematics becomes more and more unwieldy. Yet with our optical set-up, a qualitative appreciation for aspects of diffraction can be attained, an appreciation that can serve as a motivation as well as a sound basis for more advanced study.

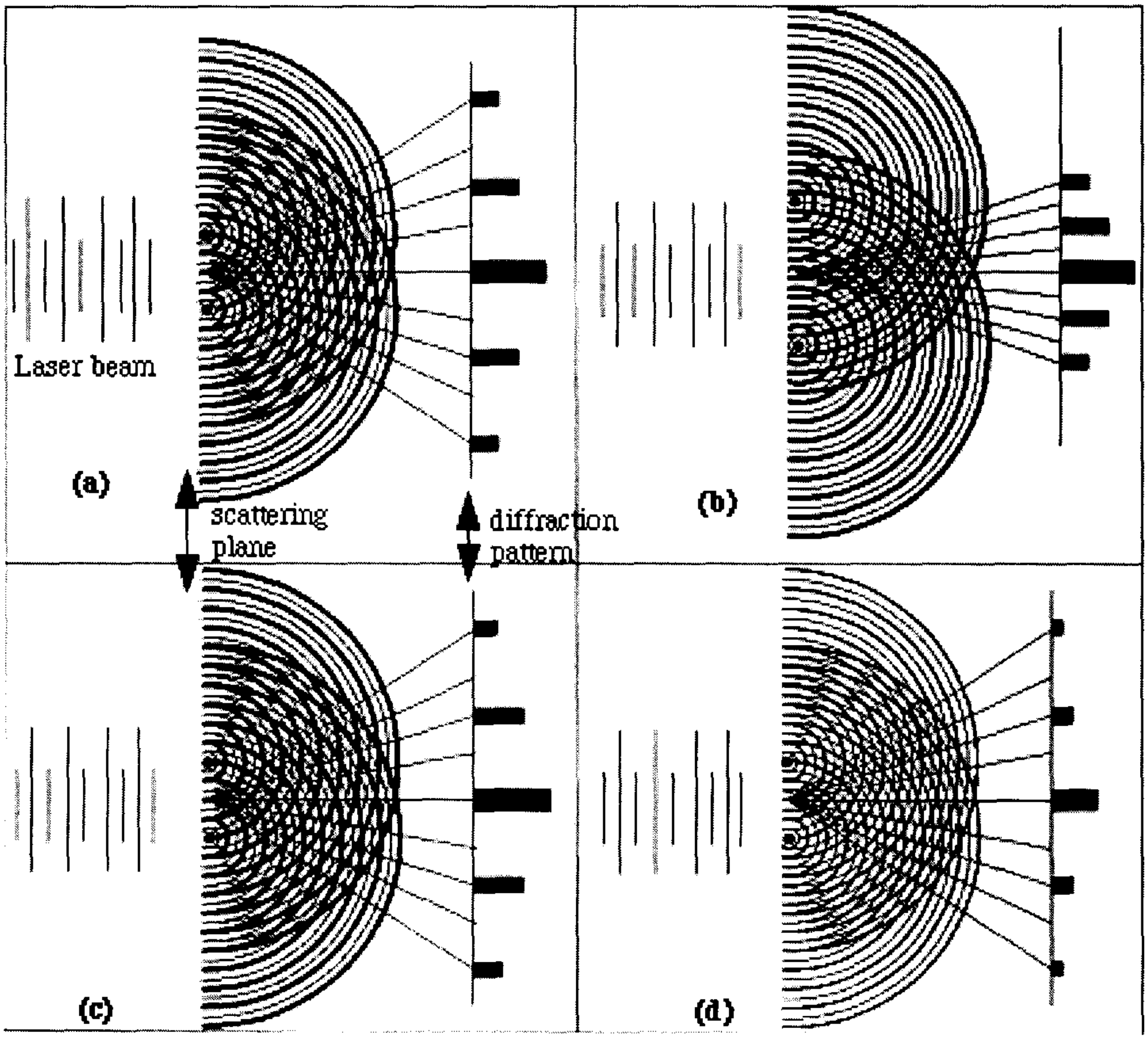

Figure 1. (a) and (b) Comparison of diffraction pattern formation with different distances between scattering centers. As the distance becomes larger, the diffraction pattern becomes smaller. (c) and (d) Different scattering efficiencies of objects. The relative intensities within each pattern do not change, but absolute intensity does.

PROCEDURE:

Safety considerations: It's rather hard to injure oneself in this lab, but students should not look directly into the laser.

(1) Setting up optical bench :

Refer to Figure 2 below or any introductory physics textbook for positioning the lenses. Once lens (f1) is fixed, move lens (f2) back and forth to focus on either the back focal plane, for the diffraction pattern, or the image plane, for the image. Use a slide with a sample pattern, such as a grid, to focus the images onto a screen or the wall. Mark the positions of lens (f2) corresponding to each plane.

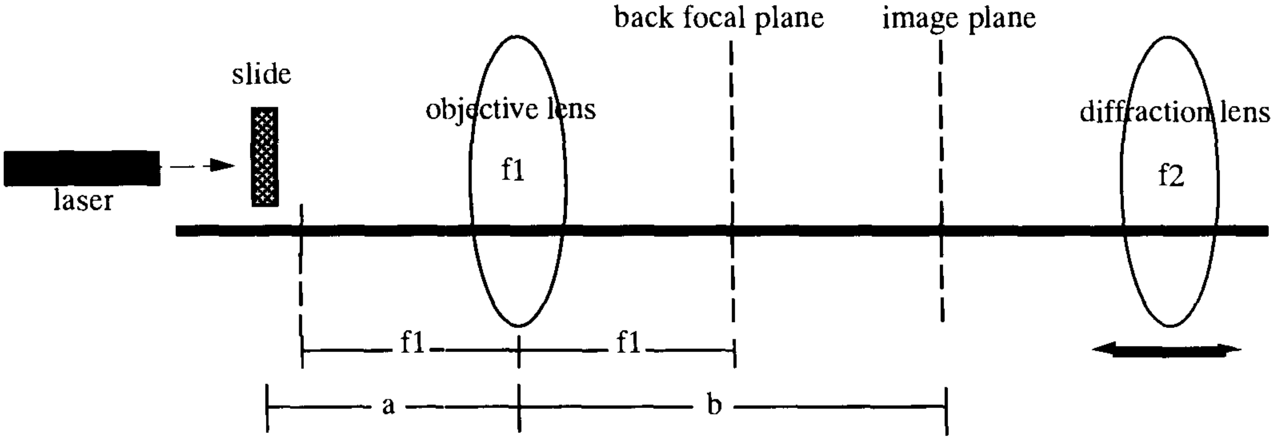

Figure 2. Diagram for optical bench set up, where f1 and f2 are the focal lengths of the lenses.

The position of the image plane can be determined by the equation,[5]

$$\frac{1}{a}+\frac{1}{b}=\frac{1}{f1} \tag{1}$$

(2) Generating the patterns:

There are a wide variety of software packages that can be used to generate two-dimensional periodic patterns. Perhaps the most fun is CrystalPaint, which is also the most educational in terms of playing around with symmetry groups. The software allows the Macintosh user to choose one of the 17 planar crystallographic groups and to decorate it with a motif generated through a MacPaint-like palette. As an alternative, students can experiment with various draw, paint, or CAD programs to create their patterns. Periodic greyscale changes can mimic the creation of ordered, multicomponent solids. When designing these patterns, keep in mind that the features should be as small in size as possible, so that when reduced into a slide they will be effective in diffracting radiation on the order of the wavelength of light. Sample patterns from various software are shown below in Figure 3.

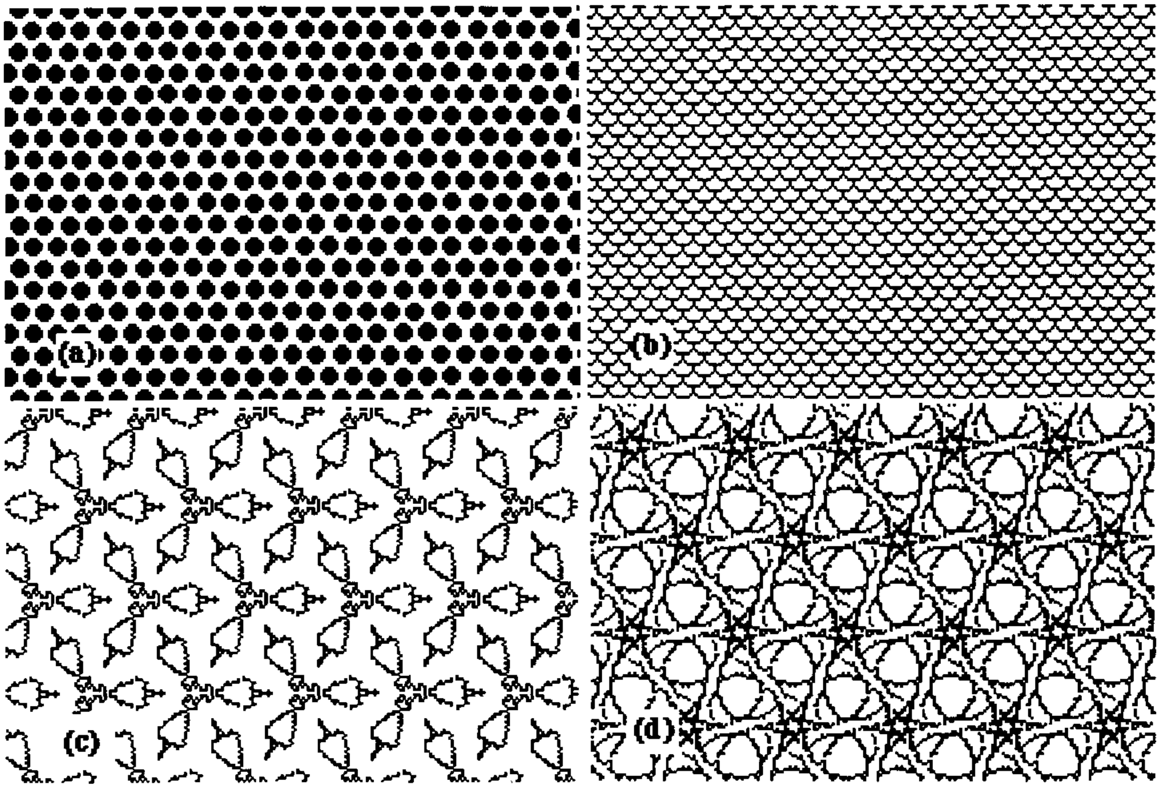

Figure 3. (a) BCC (111) plane made in Claris CAD. (b) MacPaint pattern. (c) CrystalPaint pattern with P31M symmetry. (d) CrystalPaint pattern with P6 symmetry.

(3) Making the slides:

Using the camera and tripod, take pictures of the patterns with the PolaBlue film, keeping in mind the correct feature size needed to interact with the laser light. This might take some experimenting. Take pictures at a few different camera distances making the pattern effectively larger/smaller. This will show the effect of changing distance between scattering objects on the diffraction pattern as shown in Figure 1. Rewind the film and follow the instructions on the AutoProcessor. (The processing of the slides only takes about five minutes, so students can quickly see the diffraction results from their patterns.)

(4) Getting diffraction patterns and images:

Once the slides are made, insert one in the slide holder in front of the laser. Knowing the correct positions for the intermediate lens, look at both the diffraction pattern and image. Try to find relationships between the two and record observations. Look at the diffraction pattern from another slide. Have other students try to guess what the image of the pattern might look like, then show the image. Pictures of the diffraction patterns may be taken and included in a report along with the original pattern. Figure 4 shows pictures of diffraction patterns and images obtained by using optical diffraction in the lab.

(5) Optional Group Activity:

Students can break into groups to create "mystery patterns". The challenge from one group to another is then to determine as much information as possible concerning the real space pattern another group has generated, given only its diffracted image.

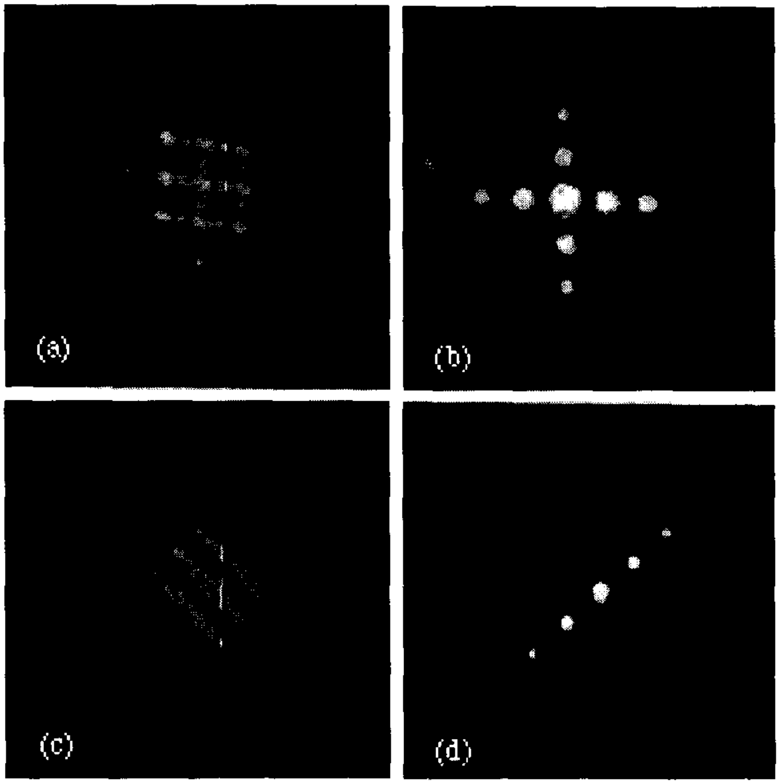

Figure 4. (a) Image of square grid pattern and (b) corresponding diffraction pattern produced in lab experiment. (c) Image of rectangular grid pattern and (d) corresponding diffraction pattern.

INSTRUCTOR NOTES:

Be sure to provide as much time and independent access to the equipment as possible, so the students have the opportunity to develop their insight and understanding.

SUMMARY:

We have described one of the structured lab activities that is a component of a new introductory materials science course developed within the ECSEL program at the University of Washington. The activity has the qualitative feature that it provides students with a relatively simple mechanism for exploring concepts that are relatively complex, and at the heart of modern experimental materials science. It is an activity which is quite inexpensive in terms of equipment requirements but quite rich in its potential applicability. It could well be used as a supplement to a wide variety of other classroom or laboratory activities having to do with diffraction.

ACKNOWLEDGMENTS:

The authors would like to thank Dr. G. H. Kim for his help in this experiment.

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2. Kalonji, Gretchen: Alternative Assessment in Engineering Education: The Use of Journals in a Core Materials Science Subject. in *Engineering Education: Curriculum Integration and Innovation*, p. 215. Proceedings of the Engineering Foundation Conference, Santa Barbara, CA, January 1992.

3. Taylor, Charles: *Diffraction.* IOP Publishing Limited, Bristol, 1987.

4. Lipson, S. G. and Lipson, H.: *Optical Physics.* Cambridge University Press, Cambridge, 1981.

5. Halliday, D. and Resnick, R.: *Physics, Part II.* John Wiley & Sons, Inc., New York, 1978.

SOURCES OF SUPPLIES:

The optical bench including laser and lenses can be obtained from many companies selling general scientific lab equipment. Camera and slide film supplies are available at stores selling photography equipment. CrystalPaint software is no longer available.

TESTING RIGIDITY, YIELDPOINT, AND HARDENABILITY BY TORQUE WRENCH

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TESTING RIGIDITY, YIELDPOINT, AND HARDENABILIITY BY TORQUE WRENCH

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KEY WORDS: force, elastic-plastic, resilience, rigidity, stiffness, stress-strain, torsion, toughness.

PREREQUISITES: Brittle and ductile materials; elastic or plastic deformation of metals; work-hardening of ductile or malleable metals.

OBJECTIVES: To demonstrate the phenomenon of work-hardening in ductile metals; to estimate a yield point and measure the shear strength; to use a torque-wrench and raise the material-stiffness.

EQUIPMENT/SUPPLIES:
Assorted metal bars, 3-mm sq x 5-cm.
Small bench-vise, or C-clamp.
End-wrench, or crescent-wrench, for 3-mm bar-stock.
Torque-wrench, "Craftsman" or equivalent, 45-cm (18" lg), 150 ft-lb capacity (+ or -).

INTRODUCTION: Tensile tests and stress-strain curves provide mechanical properties, such as Young's elastic-modulus (E). Literally, based on Hooke's Law, this familiar design-parameter represents material stiffness of a homogeneous metal sample, loaded axially and uniformly to its elastic limit; however, such stiffness typically is the slope of a straight line, from origin to proportional limit, on a stress-strain curve. Often, such limits are almost identical to the material's yield point; then the triangular area under this line (stress times strain over two) defines elastic energy in the sample (resilience modulus). Consider the axial Moment-of-Inertia (I) to be shape-stiffness of a cross-section, so your overall stiffness of design is E times I (Ref. J.P.Den Hartog, "Strength of Materials", Dover Inc, 1961).

Rigidity (G) is a similar modulus, representing a direct linear relationship between shear-stress/strain-angle, but for torsional loads and maximum strains at the outside surface of a twisted sample. Theoretically, to resist torque, the optimal design is a right-circular prism, shaped as a thin-wall tube not a solid rod. The ultimate distribution of material, limited by zero thickness and infinite radius, would be a mono-molecular layer at maximum distance from the neutral axis. Hence, our overall torsion-design stiffness is G (rigidity) times J (polar moment-of-inertia).

PROCEDURE:

A. For a start, 1) clamp the large-loop of a paper-clip in a small vise; 2) hook a fish-scale (spring-loaded) over the small inside-loop; 3) pull until that small loop bends about 90-degrees; 4) reverse the pull to restore the loop and straighten the clip; 5) have a student read both pulls (forces); 6) the restoring force should exceed the original one, if the leverage is constant. (Note: Weights could be hung on a horizontal clip to bend and harden the wire. However, avoid bending at new places on the wire, as work-hardening occurs in the old.)

B. For practical tests of ductile metals: 1) clamp a 5cm length of 3mm square rod (say 3/4" by 3/4" by 2") in a vise; 2) fit an end-wrench onto the rod; 3) twist 90-degrees, then 180; 4) reverse twist to 90-degrees, then back to zero; 5) feel increasing twist-resistance from work-hardening, until fatigue and fracture occur. (Ref: M.A. Sarapin, "United Association Apprentice Training Program", Metals Course #38, 1979-89).

C. For quantitative tests, use a torque-wrench: 1) braze a square socket-fitting to the wrench end; 2) mount a new rod in the vise; 3) twist the rod slightly, then release and observe its elastic return to zero-degrees; 4) record the twist-angle at yield-point, via protractor, just after the rod has a permanent set; 5) twist an extra 45, 90, 135, 180 degrees, recording the corresponding torques; 6) reverse the twist, then record torques which restore the rod to 135, 90, 45, and zero degrees; 7) test small rods (3-mm square) of steel, cast-iron, aluminum, copper, brass; 8) plot torques vs. angles for all samples; 9) compare results with published moduli of E and G (Ref: ASM Metals Handbook, ed.9, v 1 & 2).

Ed Widener has taught Mechanical Engineering Technology at Purdue University since 1978, concentrating on statics, fluid mechanics, materials, and technical writing. Member of ASME, ASEE, ASM, TAPPI. Registered PE in New York and Indiana. ABET visitor from 1983-1990. BS(Physics) and BS(ME) from Purdue University; MS(EM) from University of Kansas... Taught nights at IUPU-Indianapolis and Danville-JC,IL. Mechanical Development Engineer for "Coria" collagen meat-casing with Teepak Co., Continental Group, IL (1976-78). Process Group Leader for mechanical contractor, Baker-McHenry-Welch,Indianapolis (1974-76). Project Leader for creped-wadding and secondary-fiber with Kimberly-Clark Co., Memphis, TN (1968-74). Process Engineer for "Nomex" nylon with E.I.duPont Co. Richmond, VA (1963-68). Associate Engineer for electric-furnace alloys with Union Carbide Co., Niagara Falls, NY (1952-60). Fuel Engineer for open-hearths and rail-mills with U.S.Steel Co., Gary, In (1951-52).

ADHESIVES AND ADHESION
THE STATE OF THE INDUSTRY

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ASTM Definition

An adhesive is a substance capable of holding material together by surface attachment.

Practical Adhesion

The ability to form a bond that can carry a load.

Practical Adhesion Classifications

Autohesion: When adherend and adhesive have similar chemical compositions and rheological states

Heterohesion: When adherend and adhesive are different in composition and mechanical state

Why Use An Adhesive?

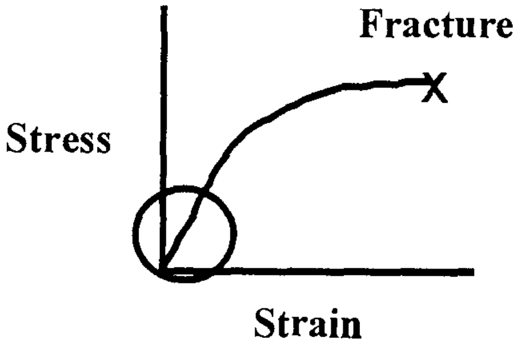
1. Uniformly distribute stress over the entire bonded areas.
2. Effectively bond dissimilar materials.
3. Maintain integrity of the bonded material.
4. Provide maximum fatigue resistance.
5. Provide continuous contact between mating surfaces to effectively bond and seal against the environment.
6. Cut costs; increase and simplify production.

Adhesive Classifications

1. Structural Adhesives
2. Hot Melt Coatable Adhesives
3. Pressure Sensitive Adhesives
4. Reactive Adhesives
5. Solvent Activated Adhesives

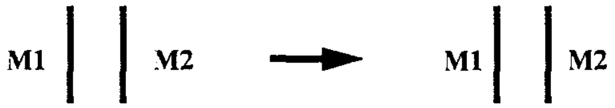
Testing Adhesive Bond Strength

Adhesives are tested to fracture but used in a region of small deformation.

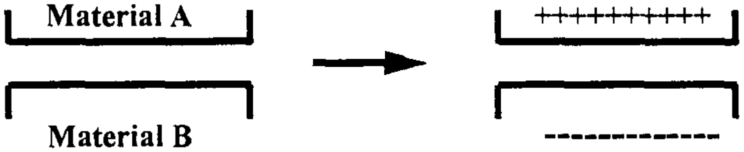


Theories of Adhesion

- Diffusion Theory



- Electrostatic Theory



Theories of Adhesion

- Mechanical Interlocking Theory
- Surface Energetics and Wettability Theory

Physical Forces of Adhesion

- Dipole-Dipole
- Dipole-Induced
- Electrostatic
- Dispersion

What Makes an Adhesive Tacky?

Wetting

and

Resistance to Debonding

Bond Making

Linear Properties

Longer Times

- Surface Properties
  - Conditions for Wetting and Spreading
- Bulk Properties
  - Time to Achieve Contact

Bond Making

For Wetting

- Substrate must not have a weak boundary layer, i.e. oil, water, dust, rust, processing aids.
- Adhesive's critical surface tension for wetting, γ_c must be lower than the surface energy of the substrate.

Bond Making

For Contact

- Adhesive's shear modulus at time and temperature of contact must be below

  1×10^6 dynes/cm^2 (14.5 psi).

- Elasticity (spring-back) must be less than 40% of the first 10 sec. deformation.
- Viscosity (non-recoverable flow) must be at least 30% of deformation.

Bond Breaking

Non-Linear

Fracture Properties

Shorter Times

- Surface Properties
  - –20-10%
- Bulk Properties
  - –Adhesive strength
  - –80-90%

Bond Breaking

For Strength

- Adhesive must store elastic energy to exhibit strength.
- Relates to adhesive's energy to fracture.
- Relates to adhesive's stress relaxation rate versus the testing rate.

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- Dr. Gregg Dahlke
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- Dr. Albert Martin
- Dr. Spencer Silver
- Dr. David Yarusso

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USE OF BELLS TO ILLUSTRATE CERAMIC FIRING EFFECTS

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11 5'93

9:46

USE OF BELLS TO ILLUSTRATE CERAMIC FIRING EFFECTS

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KEY WORDS: Porcelain Ceramics, Firing, Densification, Slip Casting

PREREQUISITE KNOWLEDGE: This demonstration is designed for high school students in Materials Science, and demonstrates the effects of densification and glassy bond formation in a porcelain composition on the ringing quality and tone of a simple bell. Understanding is largely intuitive, so there is no special prerequisite knowledge necessary.

OBJECTIVE: Students will develop an understanding of the effect of glass formation on properties of a ceramic object, and will observe the glass content to increase with firing temperature.

EQUIPMENT AND SUPPLIES:
1. At least six simple slip-cast[1] bells, purchased from a local hobby ceramics supplier and with an original weight constant to within 5%; the bells should be made from a low-firing clay to permit densification within laboratory furnace capabilities.
2. Laboratory furnace capable of 1200 C and preferably programmable for unattended operation.
3. Common steel drill, 1/16-1/8 in., to make holes for hanging, plus suitable string to suspend bells.
4. (Optional) Special high-temperature marking crayon, for noting firing temperature on sides of bells. An alternative would be to scratch the information with a sharp tool.

PROCEDURE: Using a wet sponge, remove all flash (excess clay at the joint between the mold halves) from the bells. Using an electric drill, make the hole for hanging the bells. Number all the bells, using a special crayon capable of leaving a mark that will withstand firing, or scratch reference numbers in the bells. Weigh all of the bells after cleaning and drying at 100 C, to be certain that all are within 5% of the same weight, and record the weight of each bell. Using the inside-marking blades of a dial-gauge caliper, make two scratches on each bell, 1" apart at the open lip at the bottom. These will be used later to calculate relative density.

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[1] Slip casting is a technique for making hollow ceramics, in which a suspension of clay (the slip) is poured into a mold made from plaster of Paris. The porous mold absorbs water from the slip next to it, creating a hollow clay shell when the remaining slip is poured out.

Consult your local ceramic supplier to find the proper temperature for bisque firing[2] and for firing to full maturity. If these temperatures are given as pyrometric cone numbers, the ceramic shop will have a conversion table that can be used to obtain corresponding temperatures. For instance, say that bisque firing temperature is 700C and full firing is carried out at 1100 C. One of the bells should be left unfired, one should be fully fired, and three bells should be fired to temperatures evenly spaced between the highest firing temperature and the bisque firing temperature. For our example, one bell would be fired at 700, one at 800, one at 900, one at 1000, and one at 1100 C. Mark the firing temperatures on the bells before firing them. Bells should be fired one at a time, each time starting with the furnace at room temperature and firing to the set temperature, holding one hour, and cooling in the furnace with door closed. A programmable controller would be preferable, and should be set to heat at about 100 C/h to the set temperature, hold one hour, and turn off to allow the bells to furnace cool. Weigh the bells after firing, recording data in the journal. After firing, measure the distance between scratch marks, using the same dial-gauge caliper used to make the marks. Weigh the bells, recording the weights and reference distances in the journal. Calculate relative density by the formula

$$\text{Relative Density} = \frac{\text{Weight Before Firing/Weight After Firing}}{(\text{Reference Distance})3}$$

This relative density is relative to the starting density of the bells before firing. While not an actual density, the relative density is easy to determine on the irregular bell geometry and gives an indication of temperatures at which bells are densifying.

If desired, evaluate the fired bells to determine how much open porosity remains, using the methods outlined in any standard ceramics text; see, for example, ref. 1. For a more rough-and-ready approach, try an old ceramist's trick; apply your tongue to the fired pieces, and see whether the moisture is removed by capillary action of fine surface-connected pores. If this happens, the tongue will stick to the bell. As the glassy bond develops and the pores are eliminated, the sticking tendency will lessen noticeably and may disappear altogether.

Finally, attach string to the bells, using the holes drilled previously. Lift each bell in order of firing temperature and strike it very gently, using an available

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[2] Bisque firing is a term used by ceramic artists, meaning a firing temperature sufficient to drive hydration water from the clay, but not high enough to develop significant glassy phase. At the bisque stage, the ceramic is quite durable and may be handled more readily than in the slip-cast condition.

metal piece such as a machine screw 1/4 in diameter and 4" long. The nonfired bell will emit only a very dull thud and will be quite fragile, as it is only bonded together by the clay structure. The bisque-fired bell will be thoroughly dehydrated but should not contain any bonding glass. It may ring, but the frequency will probably be quite low and the ring duration will be shorter than higher-fired bells. The four remaining bells will ring for a longer time and at a frequency that increases with firing temperature, because the high frequencies resulting from the striking process will propagate better in the denser, better-bonded ceramic material. The highest-fired bell will ring best of all. The amount of glassy material in each bell can be visualized by holding them close to a light bulb. The glassy phase in the high-fired bell transmits light better than the porous material fired at the lower temperature. Firing at excessively high temperature, or holding for a long time at peak temperature, can result in slumping, caused by the combination of excessive glass and low glass viscosity at low temperature. An example of such an overfired condition could also be made, to illustrate this effect.

SAMPLE DATA SHEETS: Not applicable, except if optional porosity measurements are made as part of the experiment.

INSTRUCTOR'S NOTES: The explanation above should be sufficient to make a set of bells. Once made, the bells should last for years, given reasonable care and if they are not struck too hard. (It might be prudent to buy extra bells left unfired, as this condition will be the most fragile). This demonstration would make a good lead-in to a more in-depth study of the effects of firing temperature on the structure of a porcelain composition. The composition could be located approximately on a ternary phase diagram and its structure inferred. As a follow-on, the time dependence of glass formation could be easily studied by firing bells at a temperature one increment less than the highest temperature--1100 C in our example--for several different times, say 1 h, 2h, 4h, and 8h, and the effect on ringing determined. A microphone and oscilloscope might even be used to analyze the frequency response of the bells. The slip casting process itself could be included in this experiment, by simply buying a plaster of Paris bell mold from the ceramic supply house, along with a supply of suitable slip. Slip is a water suspension of fine clay, with an agent added to neutralize the surface charge causing clay particles to clump together. The solids content of such a material can be quite high, as may be demonstrated by measuring the density of the liquid. The plaster mold draws water away from the slip next to it, causing a hard casting to build up. If you elect to cast your own bells, there should be a consistent overnight drying period between castings, to allow the water to leave the mold.

The reason for the original tolerance on bell weight is to minimize effects of thickness on the ringing qualities of the bells.

REFERENCES

1. Van Vlack, L. H., Physical Ceramics for Engineers, Addison-Wesley, 1964.

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This document contains a collection of experiments presented and demonstrated at the National Educators' Workshop: Update 93 held at the NASA Langley Research Center in Hampton, Virginia, on November 3–5, 1993. The experiments related to the nature and properties of engineering materials and provided information to assist in teaching about materials in the education community.

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