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NIST Special Publication 822

National Educators Workshop: Update '90

Standard Experiments in Engineering Materials Science and Technology

**Proceedings of a workshop held in Gaithersburg, Maryland
November 13–15, 1990**

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Standard Experiments in Engineering Materials Science and Technology

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PREFACE

NEW:Update '90 was held November 13-15, 1990, on the campus of the National Institute of Standards and Technology (NIST) in Gaithersburg, Maryland. 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 toured state-of-the-art NIST laboratories. Faculty in attendance represented 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 '90 marked the fifth annual National Educators Workshop:Update. Seventy-four participants including first-time attendees and those who attended previous NEW:Updates aided in evaluating nearly 30 experiments that were presented before the group. Additional updating information relating to materials science, engineering, and technology was also presented.

Material in this publication can serve as a valuable guide to faculty who are interested in useful experiments 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 was a blend of experiments on new materials and traditional materials. Uses of computers in MS&E, experimental design and an approach to systematic materials selection were among the demonstrations presented.

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 an international panel through the cooperation of the Materials Education Council. Authors received comments from the panel prior to NEW:Update 90, allowing them to make necessary adjustments prior to demonstrating their experiments.

Participants from the United States and Canada, who attended NEW:Update 90, observed demonstrations of the experiments and provided critiques for the authors to make further modifications prior to this publication.

Videotapes were made of the workshop by Westinghouse Materials Company of Ohio. As with previous NEW:Updates, critiques were made of the workshop to provide continuing improvement of this activity.

The Materials Education Council of the United States was represented again and will publish selected experiments in the ***Journal of Materials Education.***

NEW:Update '90, and the **'86, '87, '88** and **'89** 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 MS&E, we have included exemplary pre-university activities such as Adventure in Science, ASM International Education Foundation's Career Outreach Program, and several programs run through high schools.

Through the workshops we have learned about the Materials Science Technology Project at Richland High School (Richland, Washington) that has received support from Battelle Pacific Northwest Laboratory (PNL). An experiment was presented from the National Science Foundation and AT&T supported program at Science School in Newark, New Jersey. One high school science fair experiment, presented by its student author, had value for MS&E education at the college level.

NEW:Update '90, with its diversity of faculty, industry, and government MS&E participants, served as a forum for both formal and informal issues facing MS&E education that ranged from the challenges of keeping faculty and students abreast of new technology to ideas to ensure that materials scientists, engineers, and technicians maintain the proper respect for the environment in the pursuit of their objectives.

NEW:Update '90 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 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, VA 23504.

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

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November 13-15, 1990
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MATERIALS PROCESSING LABORATORY INSTRUCTION: STRUCTURE-PROPERTY-PROCESSING RELATIONSHIPS

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KEY WORDS: Solder Alloys; Solidification Rate; Creep/Stress-Relaxation; Recovery and Recrystallization

PREREQUISITE KNOWLEDGE REQUIRED: Ability to analyze simple phase diagrams; Ability to use reflected light microscope and determine ASTM grain size.

OBJECTIVES:

- Prepare metallographic samples;
- Determine thermal process conditions from phase diagrams;
- Distinguish differences in cast microstructures due to variations in freezing range and solidification rate;
- Determine effects of thermomechanical treatments on microstructure and properties;
  a. Recognize creep and stress relaxation behavior in stressed materials;
  b. Determine recrystallization conditions at various $\mathbf{T/T_m}$;
- Measure hardness and microhardness;
- Perform quantitative image analysis microstructures to determine grain size and phase fractions;

EQUIPMENT:

a. Two Thermolyne muffle furnaces (maximum temperature of 1000C with microprocessor programmable operating control);
b. One Olympus SZ-TR Stereo Zoom Microscope with magnification range of 7X to 40X;
c. Two Olympus BHM reflected light microscopes with magnification ranges of 50X to 500X;
d. One LECO hardness tester (Rockwell scales);
e. One LECO microhardness tester (Vickers indenter);
f. One Nikon Epiphot inverted metallographic microscope with magnification range of 50X to 1000X in bright field and differential interference contrast and attachments for:
    i. closed circuit television viewing of images;
    ii. photographic image recording with automatic exposure control (35mm and Polaroid film);
    iii. Nikon MircoPlan II image analysis system includes: digitizing tablet with preprogrammed image measurement/analysis menu; an alphanumeric printer; a multi-function cross-hair cursor.

g. One Lab Sciences Universal Materials Testing Machine operated in compression mode.

MATERIALS: Series of solder alloys: 1) 75Pb-25In; 2) 60Pb-40In; 3) 50Pb-50iN; 4. 40Pb-60In; **or,**
1) 70Pb-30Sn; 2) 60Pb-40Sn; 3) 50Pb-50In; 4) 40Pb-60Sn;
5) 37Pb-63Sn.

PROCEDURE: 1. Organize into working groups (one group assigned to study a given alloy):

The following tasks are to be completed by each group:

2. Melting and solidification of each alloy to show the effect of freezing range and cooling rate on as-cast microstructure and hardness:

   a. melt approximately 25 grams of each alloy in porcelain crucibles at constant superheat (to be determined by Group*);
   b. solidify each alloy in two ways:
      i. slow cool by casting into heated plaster molds;
      ii. cast into a "chill" mold (an aluminium block at room temperature.

(Molds are either, plaster of paris, or aluminum blocks which measure approximately 5 cm x 5 cm x 5 cm, with four holes of 1 cm diam. x 1 cm deep spaced uniformly on one face. Plaster molds must be thoroughly dry and are heated in the furnace with crucible during the melting of the charge. <u>Measurement of cooling rates can be approximated by determining the time from pouring until no liquid motion is seen on the casting surface</u>.)

3. Measurement of microstructure (grain size; phase fraction and phase distribution), hardness and microhardness of the <u>as-received</u> and <u>as-cast</u> samples of each alloy which will include:

   a. metallographic specimen preparation;
   b. photographic recording of microstructure (magnification determined by Group*);
   c. image analysis of microstructure.

NOTE: <u>Each</u> member of <u>each</u> group must carry out the full range of processing specimen preparation and analysis for <u>at least</u> one set of the group's specimens. The results obtained by the individual are to be noted in the final report.

4. Heat treatment of as-cast and as-received alloy samples (temperatures and times of heat treatments to be determined by Group*).

5. Repeat Step 3 after doing heat treatments.

6. Compression deform each alloy (samples from each of the as-cast and as-received conditions) to at least two levels of % height reduction, e.g., 25% and 50%. During deformation use the dial gauge in contact with the sample grip fixture to follow deformation vs. time at fixed load **and** use the pressure gauge to follow the load drop vs. time after load increase. (**NOTE: Loads are not to exceed 900 pounds.**)

7. Measure microstructure, hardness and microhardness of each cold-worked sample as in Step 3.

8. Carry out a Recrystallization heat treatment of each sample from Step 6 attempting to obtain the minimum as-recrystallized grain size (choice of Recrystallization Temperature must be justified).

9. Repeat Task 7.

10. Submit all raw data to the Central Data File (Room A3) as soon as it is acquired (including prints of photographs).

**11. Each Group is to submit a typed Summary Report (addressed to, Supervisor, Materials Laboratory) in the following format:

a. using 8½" x 11" paper with one inch margins.
b. text report is not to exceed 10 pages, exclusive of data tables, figures and references.
c. all data from all Groups is to be included in the report of each Group and to be discussed in the Discussion of Results section.

(**NOTE:** A typical report includes the following sections:
Introduction
Experimental Plan/Procedures
Results (Data, Tables, Figures)
Discussion of Results (theory vs. experiment)
Summary/Conclusions
References

Further details are given in DEPARTMENTAL STANDARDS FOR FORMAL WRITTEN REPORTS.

12. Each individual is to keep a LABORATORY NOTEBOOK as a permanent, up-to-date record of their activities. (**Laboratory Notebook requirements and procedures are described in DEPARTMENTAL STANDARDS FOR LABORATORY NOTEBOOKS**). (NOTE: The notebooks may be collected for inspection at any time upon request by the Instructor. Keep them available.)

SUPPLEMENTAL INFORMATION: 1. No use of furnaces, microscopes polishing equipment or testing equipment is permitted without permission of Instructor.
2. Safety goggles are required at all times.
3. Use of breathing masks and gloves is required during melting, metallographic specimen preparation, heat treating and deformation processing operations.

SAMPLE DATA: Attached are examples of: 1. a sample preparation flow sheet; 2. microhardness measurements; 3. grain size measurements;

There are no "standard" data sheets used. Each group constructs their own form of data presentation. As noted, however, the Final group reports must include the data from the other groups.

INSTRUCTOR NOTES: This Experiment is one of only two conducted in our Junior year Materials Processing Lab. (See attached ME 395B , Materials Lab, course handout.) It occupies about 80% of the semester long laboratory. The first Experiment is an individually performed and reported procedure in which each student learns to observe, record and analyze the microstructure of a set of stainless steel samples. It serves as an important preliminary experiment to the lengthy group Experiment II described here.

This Lab course is closely tied to a lecture course on materials and manufacturing processing (M/MP). The two courses incorporate, uniquely, we believe, a pedagogical feature which we call **peer assisted learning** (pal). In pal we involve the more proficient students in a tutorial role to provide assistance to their less proficient peers. This use of students teaching students, or cooperative learning, or **peer assisted learning** is necessitated in both the lecture and the laboratory course.

In the lecture course we need the assistance of those students who have had a previous materials engineering/science course to help us carry out a series of extraordinary make-up tutorials. Those sessions are required because of the fact that approximately 67% of incoming Junior transfer students have not had the prerequisite introductory Materials course. To overcome this deficiency we: 1) conduct a materials engineering/science diagnostic survey at the start of the lecture course to identify the extent of the deficiencies; 2) conduct a set of 30 minute tutorial lectures, on an optional attendance basis, prior to the regularly scheduled class period; 3) use the pal groups to provide supplementary help to the students with deficiency. In the lab course the pal process is used to leverage our instruction in the performance of experimental procedures. That is, in each lab group one person is identified as the "lead experimentalist" for each separate procedure, e.g., melting, deformation processing, hardness testing, microscopy, image analysis and metallographic specimen preparation. That person then serves as the instructor for others in her/his group as each one of them goes through that part of the Experiment. (NOTE: Each person in each group must do at least one completely cycle of the entire Experiment from melting to image analysis.) The use of pal in the lab course frees the official Instructors to supervise critical operations for safety and to assist with such operations as interpretation of microstructural observations, etc..

There have been several useful outcomes from this intensive group Experiment. One has been the success of the pal process. It has made for more efficient use of the lab facilities since essentially no time is lost in waiting for the Instructor to provide operating information. Additionally, the "lead experimentalists" develop an apparent pride in their role and become quite proactive as instructors.

An equally important outcome from these Experiment II labs has been the compilation of the results into summary reports which have been published and/or presented at technical society meetings, e.g., "Observations of the Effect of Alloy Composition and Processing on Ambient Recrystallization Behavior of Solders", 2nd Electronic Materials and Processing Conference, ASM International, Phila., PA, 1989. A second summary presentation of work on the Pb-In alloys is scheduled for the 4th Electronic Materials and Processing conference to be held in Montreal in August, 1991.

The development of student observational and reporting skills is another feature of this course which we emphasize. Of particular interest to us is that the students become proficient in keeping a laboratory notebook. As noted in the Experiment II descripiton their notebooks must conform to a specific format and they are collected for evaluation at least four times during the semester. That evaluation includes a critique on both content and presentation. Report writing also gets attention through the individual reports for Experiment I as well as the group report from Experiment II. In fact in the case of the individual reports each of those is read and edited by two classmates before being graded by the Instructor. The student editors are frequently harsher critics than the Instructor.

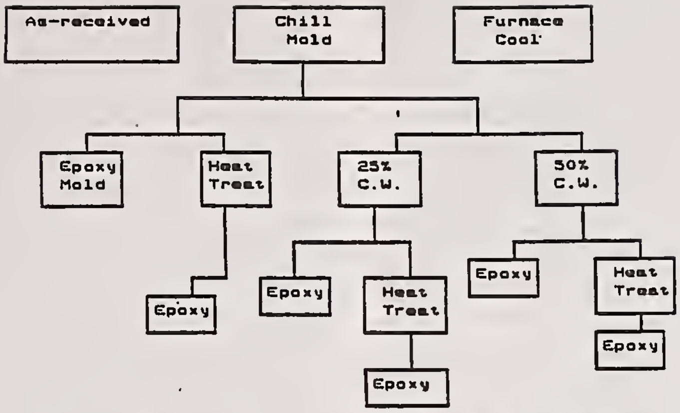
Perhaps of greatest value has been the recognition on the part of many of the students of the relation between structure and properties of materials. Especially notable from the work with the solder alloys is the demonstration of the dynamic nature of materials microstructure and the relation to property changes. Additional recognition of the effect of composition on both microstructure and properties is gained when the groups compare data between groups for the final reports. Significant differences in sample preparation, observed phase fractions and phase distributions, e.g., primary vs. eutectic Pb in Pb-Sn alloys, and microhardness are noted and remarked on by the groups.

The **metallographic sample preparation technique for these soft alloys** deserves some special mention. In general higher Pb content alloys are somewhat easier to prepare than higher Sn or higher In alloys. In all cases, however, the emphasis is on using the minimum amount of time with each abrasive step and using the minimum amount of pressure. Such variations of technique as "single hand, one direction" vs. "two hands, both directions" have been reported to be successful. We recommend the former technique for each novice and we also encourage them to actually keep count of the number of strokes on each paper, and the number of revolutions of each wheel in the polishing steps. Since each student's self-adjusting pressure application, or feel, cannot be duplicated in another student we only can use such data as guidelines. Nevertheless, the true novice finds such guidelines helpful; along with frequent trips to the stereo-zoom microscope as grinding and polishing proceeds.

SOURCES OF SUPPLIES: The most expensive items in this Experiment II are the diamond pastes used in the metallographic specimen preparation. We use a procedure which uses 9, 3, and 1 micron pastes for polishing. Each alloy group has separate wheels and cloths (also a potentially large expense, but separate cloths are really all that is necessary). The Pb-Sn solder alloys are quite inexpensive and can be purchased from many industrial suppply houses. The indium bearing alloys are more expensive and we have relied on alloy manufacturers for donations. A final obvious cost item is the photographic expenses. We use a combination of 35 mm and Polaroid film. As the proportion of the latter increases so do the costs. Mounting of the samples for metallographic preparation requires epoxy mounting materials which do not have a high curing temperature. We have used a resin and hardener combination manufactured by the Hysol Corporation for use in the microelectronics industry. The resin is #RE-2038 and the hardener is #HD-3416. We have used a ratio of R:H = 10:1, by weight with success. However, the system is very sensitive to moisture and in humid weather a vacuum bell jar is recommended to extract dissolved gasses from the mix.

FIGURES:

1. Copy of Evaluation Sheet used for Lab Notebook Inspection.
2. Copy of Processing Flow Sheet for One Set of Samples/One Alloy.
3. Copy of "Table 1." and "Table 2." from Publication of Summary of Lab Experiments on Pb-Sn Alloys.(ASM 2nd EM&P Conf. 1989).
4. Copy of a Summary Table from a Group Rpt. on Pb-In Alloys.
5. a. (**upper left**) 70Pb-30Sn, Slow Cool/50% Def./Rm. Temp.. Note beginning recrystallization in lighter areas. 400X.
   b. (**upper right**) 70Pb-30Sn, Slow Cool/50% Def./Annealed ($0.6T_E$), 30 min. Note recrystallization in two-phase regions. 400X.
   c. (**lower left**) 75Pb-25In, Slow Cool/50% Def./Rm. Temp. 200X. Note recrystallization colonies at deformed grain boundaries.
   d. (**lower right**) 75Pb-25In, Slow Cool/50% Def./Annealed ($0.75T_s$), 30 min. 200X. Note grain growth of recrystallized regions.
6. Copy of Course Syllabus, **ME 395B**.



The process for both the As-Received and the Furnace Cool is the same. After each sample is put in the epoxy it is polished according to the procedure described in the report then etched and observed under the microscope.

FIGURE 2. SAMPLE EXPT.II FLOWSHEET

ME 395B
Spring 1989
Laboratory Notebook Inspection Report

____________. Your Laboratory Notebook was inspected _____

The rating is given below.

Content
S___ U___

Presentation
S___ U___

If there are any questions regarding these ratings please see me.

J. A. Clum

FIGURE 1. LAB NOTEBOOK EVALUATION SHEET

Table 1 - Hardness(VHN) of 37Pb-63Sn

Condition***	(25/50)*	(Pb/Sn)**
AR/--/--	11.6/17.5	14.5/17/6
AR/25/--	12.3/14.1	13.0/15.3
AR/50/--	11.4/15.4	14.2/13.0
SL/--/--	15.8/18.9	16.0/16.1
SL/25/--	----	11.7/11.9
SL/50/--	12.9/17.9	12.7/11.4
FA/--/--	15.3/9.52	14.1/16.0
FA/25/--	14.1/14.3	11.4/10.5
FA/50/--	14.2/17.1	8.58/9.80

* Refers to applied load being either 25gm or 50gm. Time was 20sec. for either load.
** Refers to indentation being centered in either a Pb-rich, or, a Sn-rich grain. (These data were measured with a load of 10gm applied for a period of 10sec.).
*** Symbols used are as follows:
(starting condition)/(% compression)/(anneal)
i) AR=as-received; SL=slow cool; FA=fast cool
ii) 25=25% compression; 50=50% compression
iii) (--)=no furnace anneal; HT=0.9Tm, 1hr.

Table 2 - Hardness(VHN) of 50Pb-50Sn

Condition	(25/50)	(Pb/Sn)**
AR/--/--	9.81/9.09	8.58/9.06
AR/25/--	8.82/8.36	8.49/9.06
AR/50/--	10.6/9.50	(9.53)
SL/--/--	15.7/14.3	9.16/10.1
SL/25/--	14.8/14.9	(13.3)
SL/50/--	9.12/12.8	10.3/9.69
FA/--/--	13.1/15.4	(11.8)
FA/25/--	13.2/12.9	10.3/9.80
FA/50/--	9.46/8.61	(11.3)

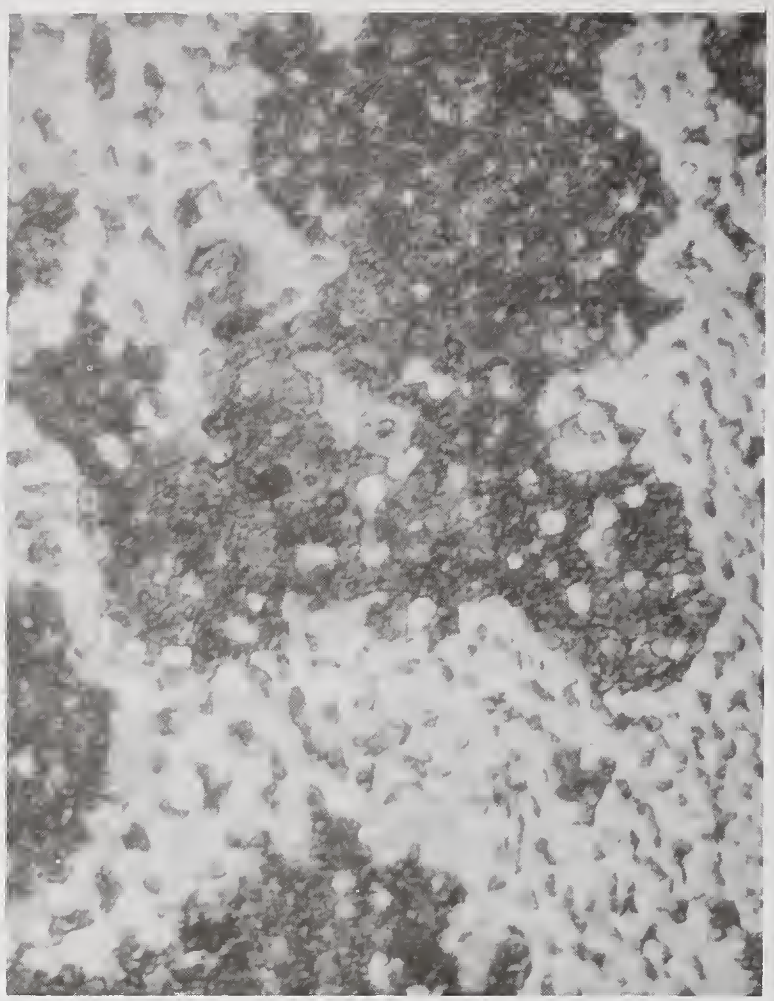
** Load of 10gm applied for 5sec.
Bracketed values are for a mixed structure.

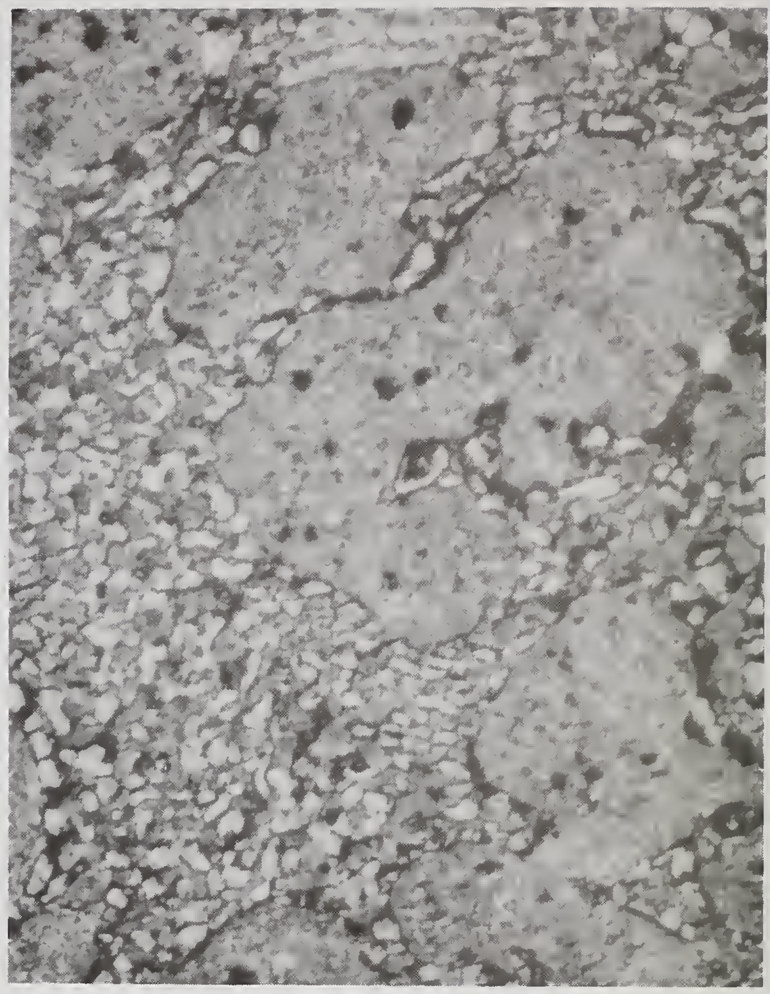
	GROUP 1		GROUP 2		GROUP 3	
SAMPLE	G.S. #	VIC. #	G.S. #	VIC. #	G.S. #	VIC. #
O00N01	11-12	11.60	15	12.70	12	9.03
O00H02	11-12	10.90	13	10.70	10-11	9.24
O25H03	8-9	13.28	13	12.20	13-14	9.96
O25N04	7-8	13.75	12	12.90	13-14	9.36
O50N05	11-12	11.80	9	10.20	13-14	9.51
O50H06	N/A	12.10	15	10.60	12-13	8.85
F00N07	1	11.40	6	11.40	13	8.62
F00H08	1	12.50	5	12.30	13-14	9.17
F25N09	1	13.39	5	11.60	12-13	8.59
F25H10	1-2	13.55	6	11.00	13-14	10.10
F50N11	10-11	12.63	4	13.50	14	10.20
F50H12	N/A	14.30	5	13.70	11-12	8.64
C00N13	5-6	13.05	12	17.70	13-14	8.42
C00H14	10-11	13.60	9	12.40	12-13	9.22
C25N15	11-12	14.95	14	12.50	12-13	8.55
C25H16	N/A	14.75	11	14.30	13-14	9.50
C50N17	11-12	11.55	14	11.10	13-14	9.25
C50H18	N/A	12.05	15	11.20	13	8.75

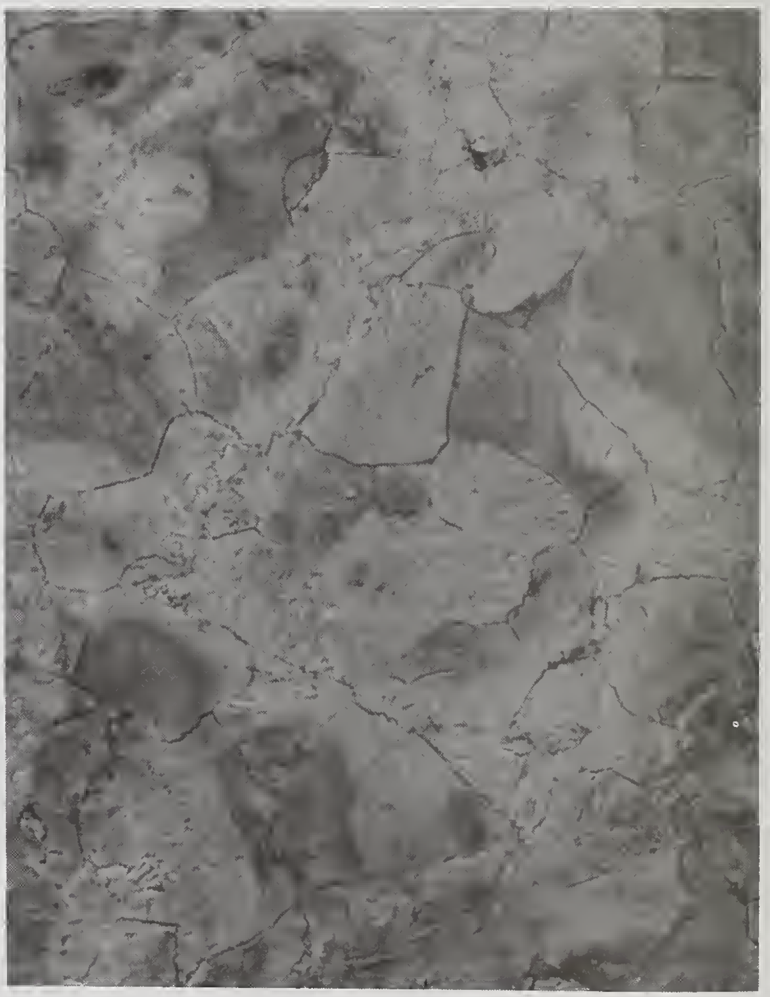
CODE KEY

TYPE OF CAST	%COLD WORK	SAMPLE NUMBER
O: ORIGINAL	00	01
F: FURNACE COOLED	25	
C: CHILLED	50	18

Figure 4a. Comparison of Vickers microhardness and ASTM grain size for three Pb-In alloys. Group 1, 2, 3 are 75Pb25In, 60Pb40In, and 40Pb60In, respectively.







COURSE:
ME 395B
Materials Lab

WHEN:
Spring 1990
T,R, 1:15-4:15

INSTRUCTOR:
J. A. Clum, M&IE Department
Office: EB S-18, Phone 777-4860

TEXT:
"The Role of Microstructure in Metals", A. R. Bailey, Metallurgical Services Lab. Ltd., 1982

SUPPLEMENTAL:
Metals Handbook, ASM International
8th Ed. v. 7 "Atlas of Microstructures"
v. 8 "Metallography, Structures and Phase Diagrams"
9th Ed. v. 9 "Metallography and Microstructures

COURSE DESCRIPTION:

A laboratory course designed to introduce students to the methods of materials characterization (microstructures and properties). Experiments involve specimen preparation and microscopic structure examination. The specimens are chosen to illustrate the role of materials processing and history on microstructure and properties. Materials processing includes: casting, mechanical deformation and heat treating of metal alloys.

COURSE SCHEDULE:

I. Introduction to laboratory facilities, notebook use and laboratory safety procedures and regulations.

II. Safety Review and Experiment I (individual assignment using metallurgical microscopes).

III. Experiment II (group project on materials processing and microstructure/properties measurement)

COURSE GRADING:

Topic	% of grade
Safety Quiz	5
Experiment I Report	25
Experiment II Report	35
Lab Notebook*	35
	100

*Lab Notebooks are to be available for collection and examination at all times. All entries should be permanent and up-to-date. (See Dept. Stds. for Lab Notebooks.)

NOTE:

Each student should bring to lab
1. A notebook; permanently bound, 10 1/8 x 7 1/8 inch, 5x5 quad ruled pages.

2. A pair of safety glasses with side shields or safety goggles.

1990/09/19

Paper for the 4th Annual National Educators' Workshop on Standard Experiments in Engineering Materials, Science & Technology, November 13-15, National Institute of Standards & Technology, Gaithersburg, MD

INSTRUCTIONAL SOFTWARE FOR TEACHING POLYMER SCIENCE AND ENGINEERING

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Denton, TX 76203-5308

and **Michael R. Kozak**
Department of Industrial Technology
University of North Texas
Denton, TX 76203

1. Formulation of the problem

Teaching a comprehensive course in polymer science and engineering (PSE) is a complex task. Let us consider teaching processing as an example. The student has to acquire, at least to some extent, skills of a processing machine operator. Moreover, he has to be able to modify the process parameters in function of the nature of the material and property specifications imposed upon the product. Further, contemporary processing machines have computerized decision-making units where process parameters are changed automatically. However, one cannot trust blindly those decision units; they work according to certain universal algorithms - not necessarily best suited to a specific resin or a specific product. Other constituting elements of PSE such as synthesis, characterization, mechanical testing or viscoelasticity pose similar problems in teaching.

2. Approach taken

We have solved the above complex problem by using a variety of concomitant tools and approaches to teach PSE: a) lectures; b) video presentations; c) computer software for personal computers with schemes, computations and animations - used by the students individually on their own time; and d) hands-on experience with processing equipment (computer-controlled closed loop; injection molder; temperature controller and liquid chiller; pneumatic loader and dryer; robot). At the University of North Texas we are working on a project called CIMSE, or Computer Instruction in Materials

Science and Engineering. The software we are developing for instruction in polymers forms a part of CIMSE. Already in 1981 a distinguished national panel [1] has concluded that there is a "... need for programs and actions that will augment the present representation of polymer studies in the universities and enhance their quality. The intellectual challenges and practical dividends ... and the relevance of polymers to national needs ... warrant attention by the academic community and interest on the part of funding agencies." We believe that our CIMSE effort related to polymers at least one possible response to the call of that national panel.

3. Software developed

Previously [2] we have adapted a utility called TK!Solver for solving numerical problems in materials science and engineering. Now we have amplified the TK!Solver software (and also converted it from black/white to color). We have taken advantage of capabilities of Hyper Card Stacks [3,4] and more recently of Super Card with color capabilities.

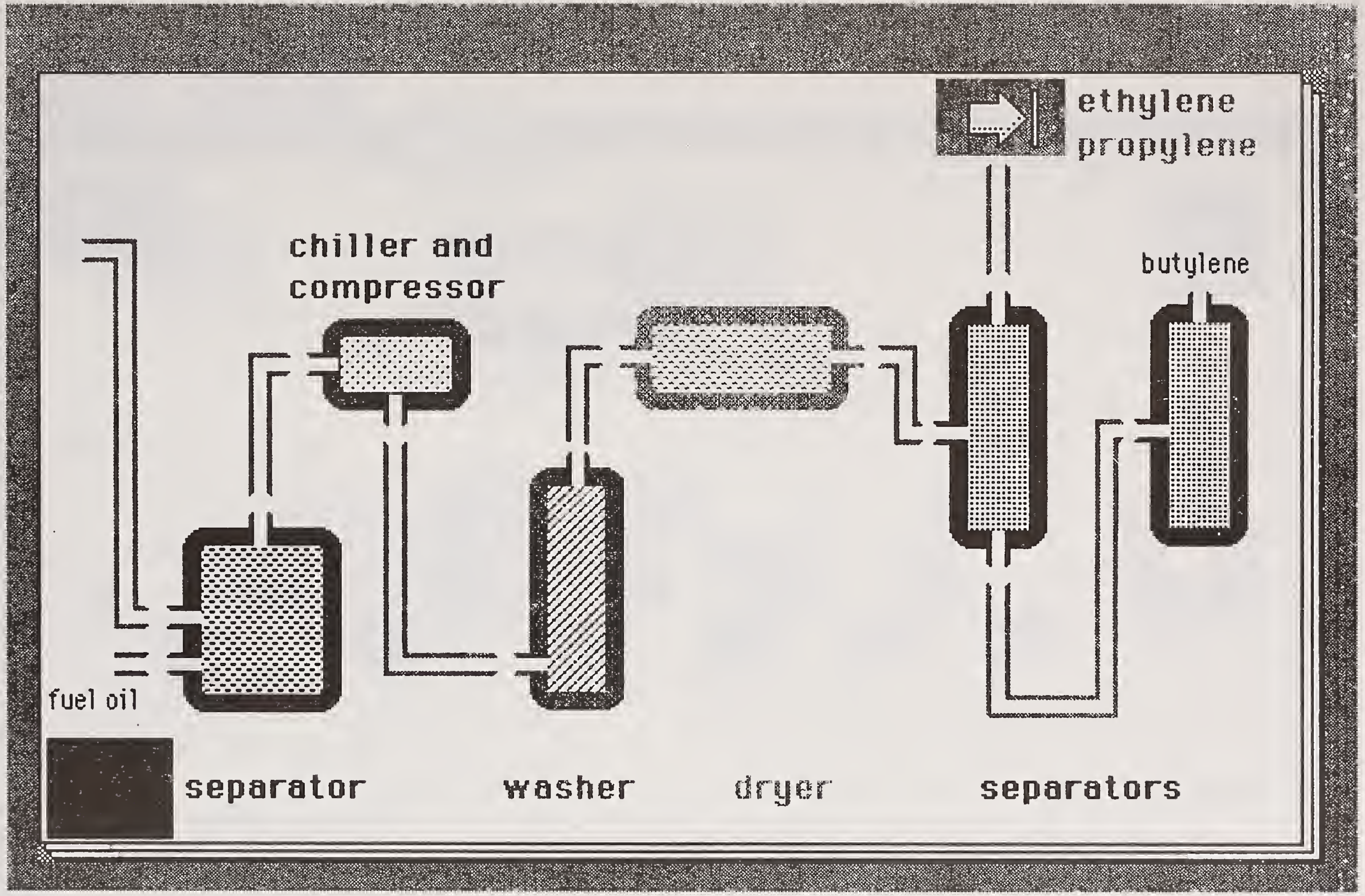
During the workshop we shall show our instruction scheme and demonstrate the software we have developed for Macintosh computers. We hope to develop analogous software for IBM PCs at a later time. Since "One picture is worth a thousand words" we show some of the screens that the student can call from a disc - to study from and work with. The following pages of this paper are black/white copies made from color screens/printouts.

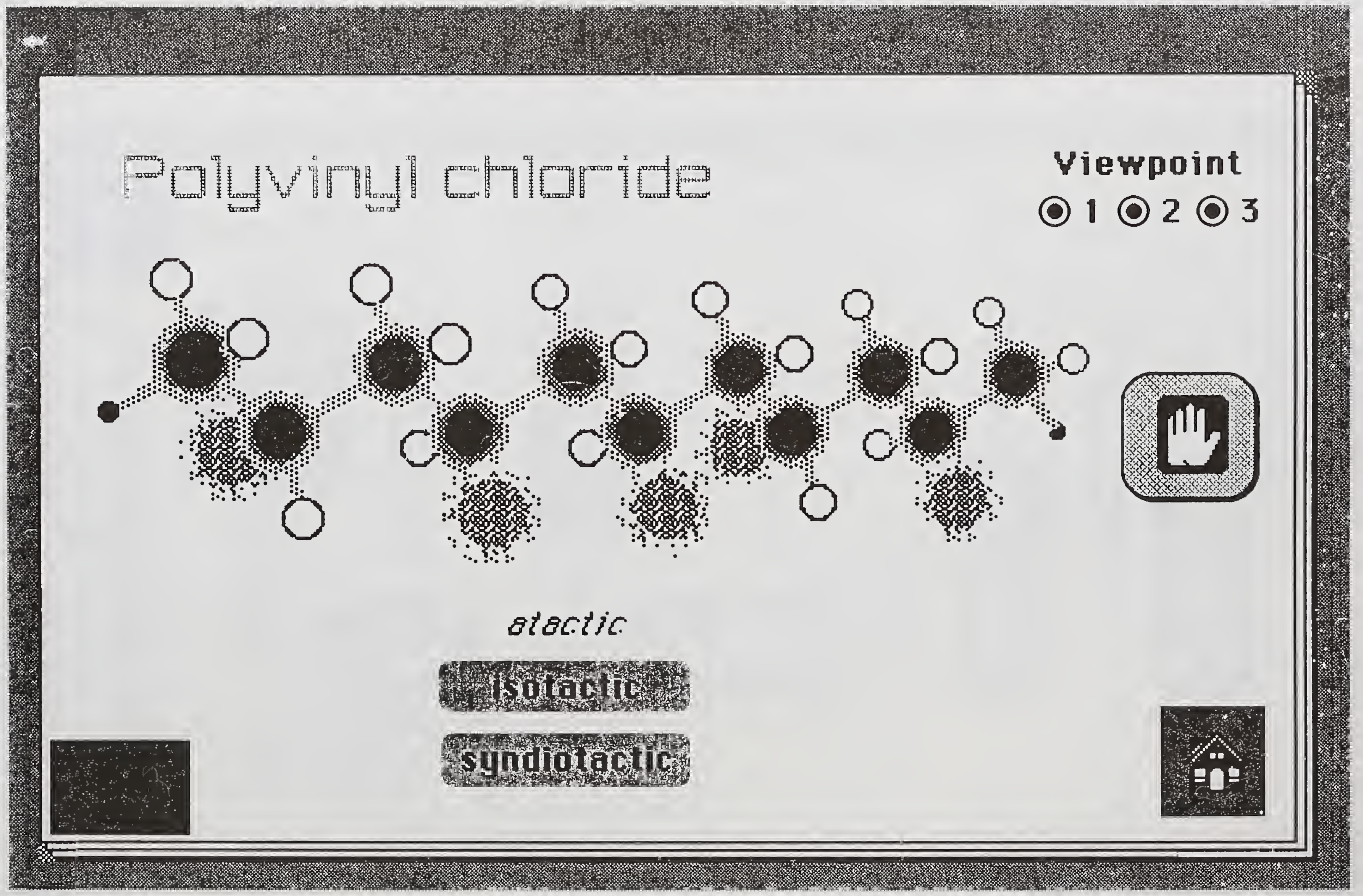
References

1. Polymer Science and Engineering , Report of a Panel of the National Academy of Sciences and National Academy of Engineering, C.G. Overberger and R. Pariser, co-chairmen, National Academy Press, Washington, DC 1981.
2. W. Brostow and D.P. Turner, J. Mater. Educ. 1988, 10, 119.
3. D. Goodman, HyperCard Developer's Guide , Bantam, New York 1988.
4. D. Goodman, The Complete HyperCard Handbook , Bantam, New York 1988.

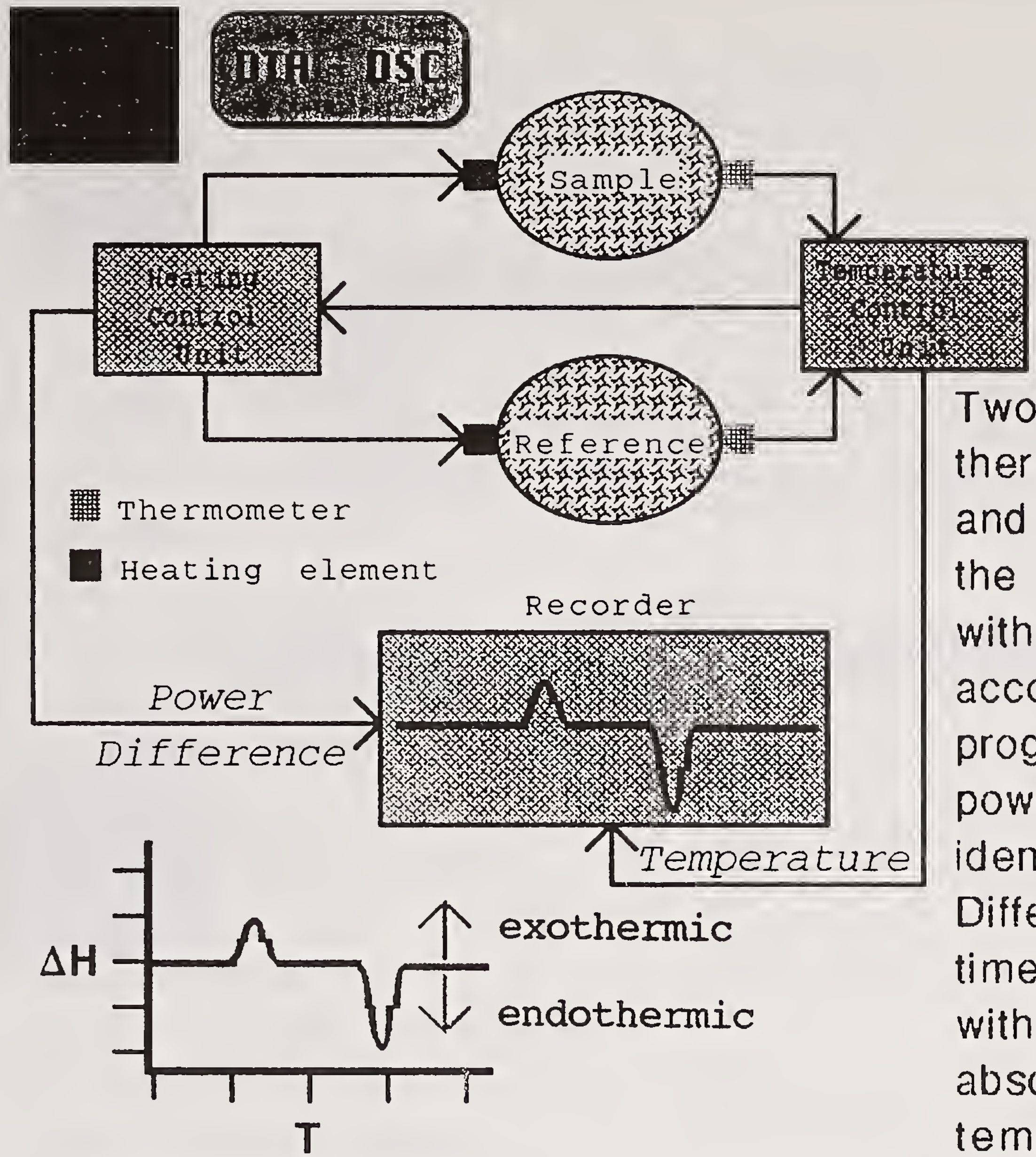
Acknowledgments

We would like to acknowledge: participation of Mr. Miguel A. Bilbao, Mr. Slawomir Blonski, Mr. Miroslaw Latka, Dr. Nikolai Medvedev, Miss M.-H. Tai and Mr. David P. Turner in software development; CIMSE use and evaluation by our students in various courses; partial financial support of the E.I. du Pont de Nemours & Co., Wilimington, DE, and the University of North Texas; and also guidance from Mr. John T. Lund and his Committee on Educational Aid of du Pont.


ethylene
propylene
chiller and
compressor
butylene
fuel oil
separator
washer
dryer
separators


Polyvinyl chloride
Viewpoint
◉ 1 ◉ 2 ◉ 3
atactic
isotactic
syndiotactic

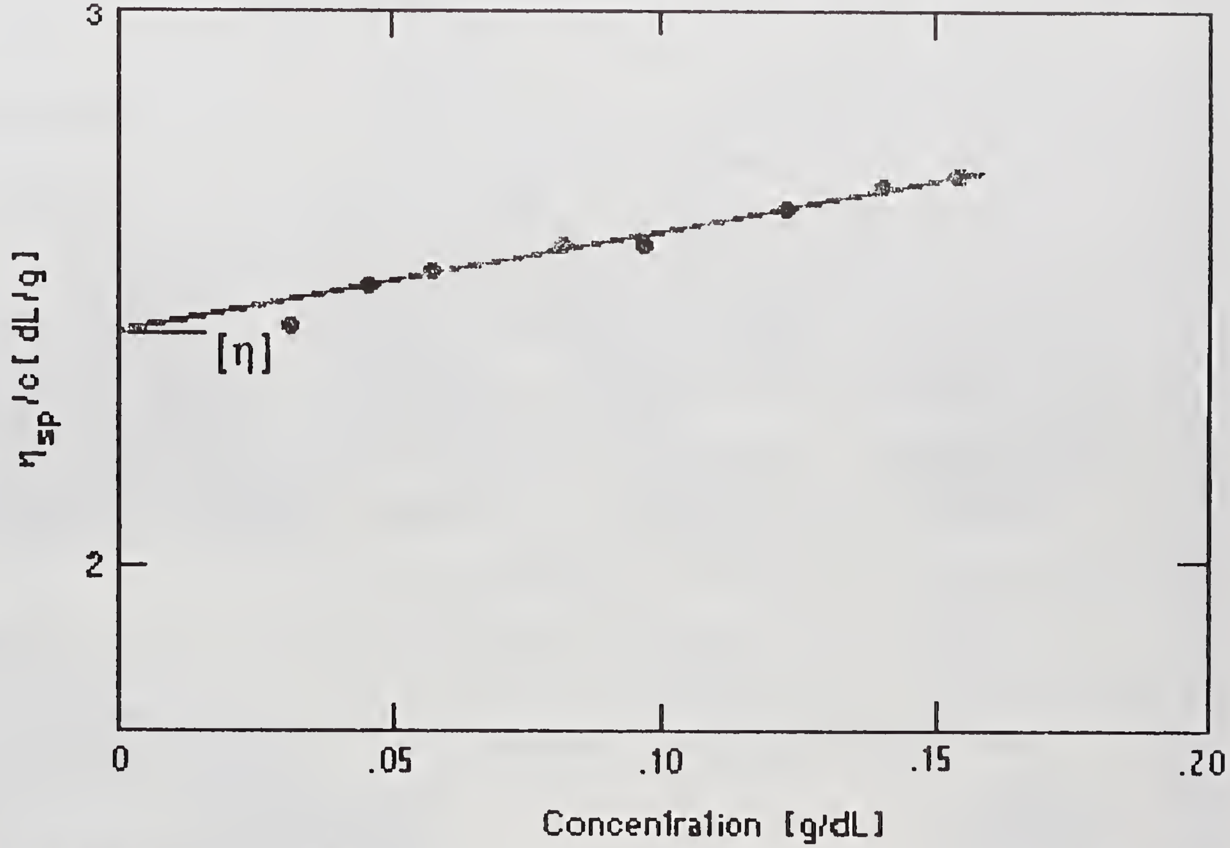
DSC



Two sample holders which are thermally isolated from one another and which contain the sample and the reference substance are heated with a high degree of accuracy according to a linear temperature programme. Temperature and heating power are monitored and supplied by identical thermometers and heaters. Differential power as a function of time is transmitted to a recorder with power as ordinate and the time abscissa transformed to temperature.

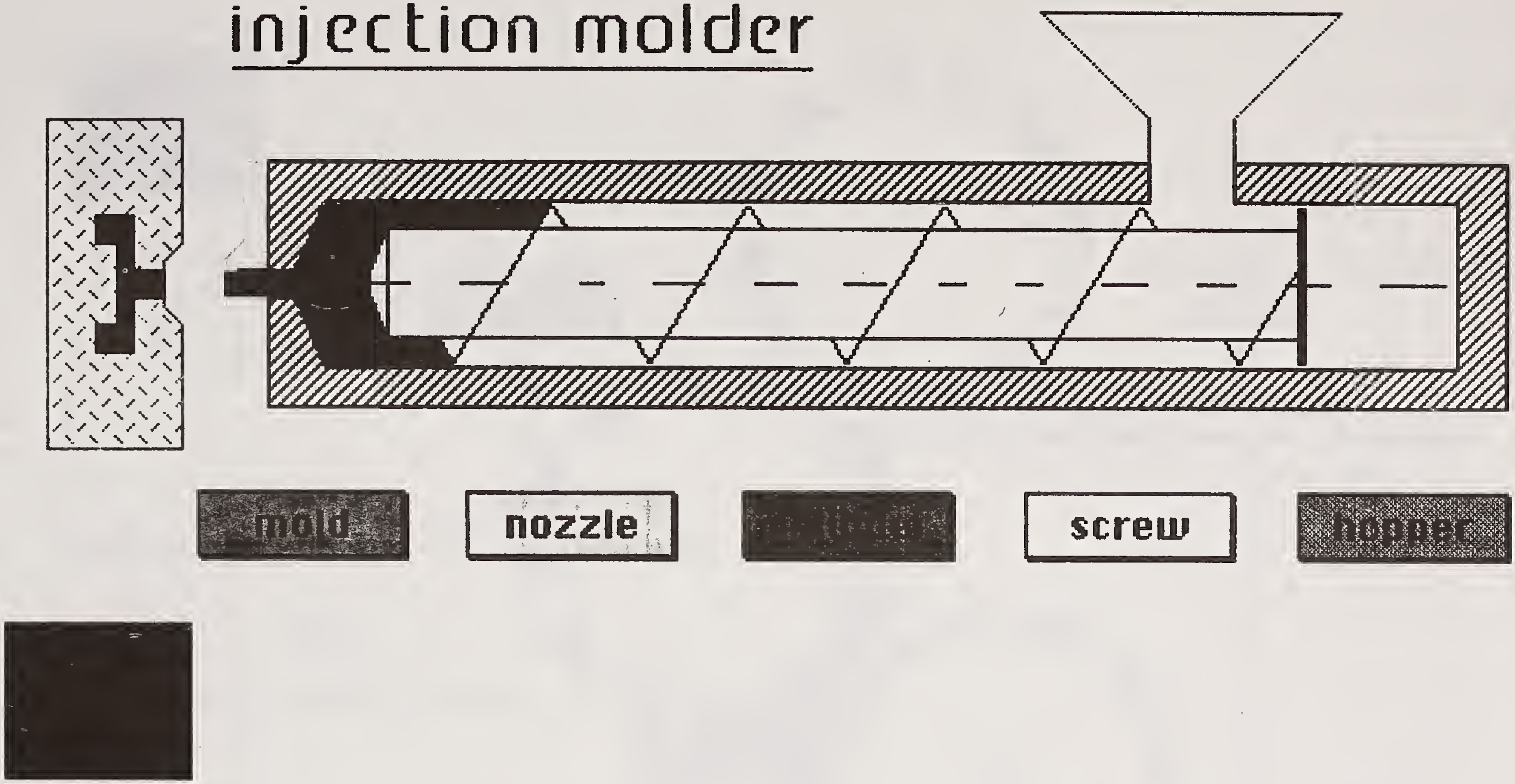
Intrinsic viscosity

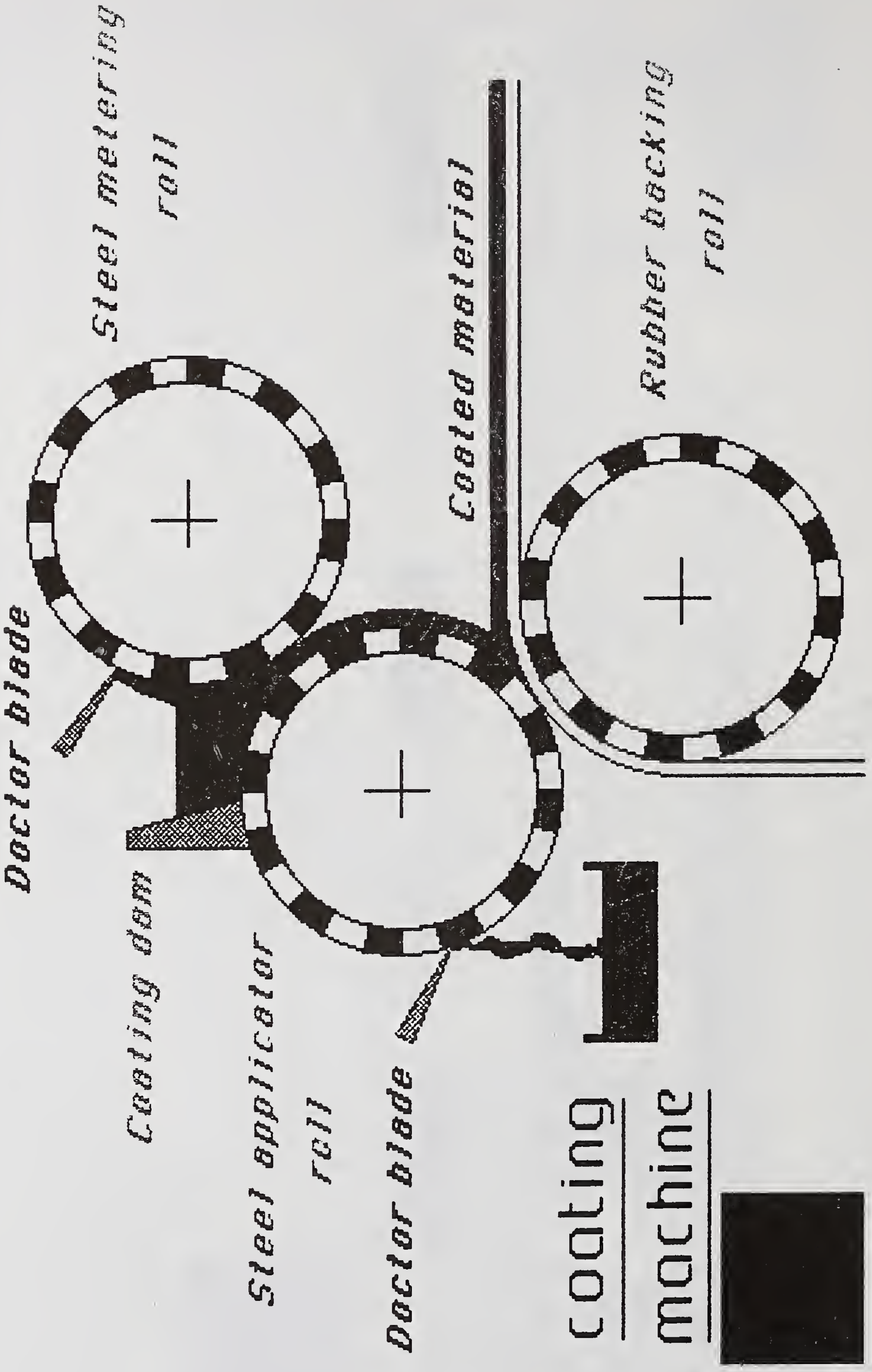
Viscosity measurements are made more accurate by extrapolating results to low polymer concentration.
If the specific viscosity divided by the concentration is extrapolated to infinite dilution, the resulting value is called the intrinsic viscosity or the limiting viscosity number.

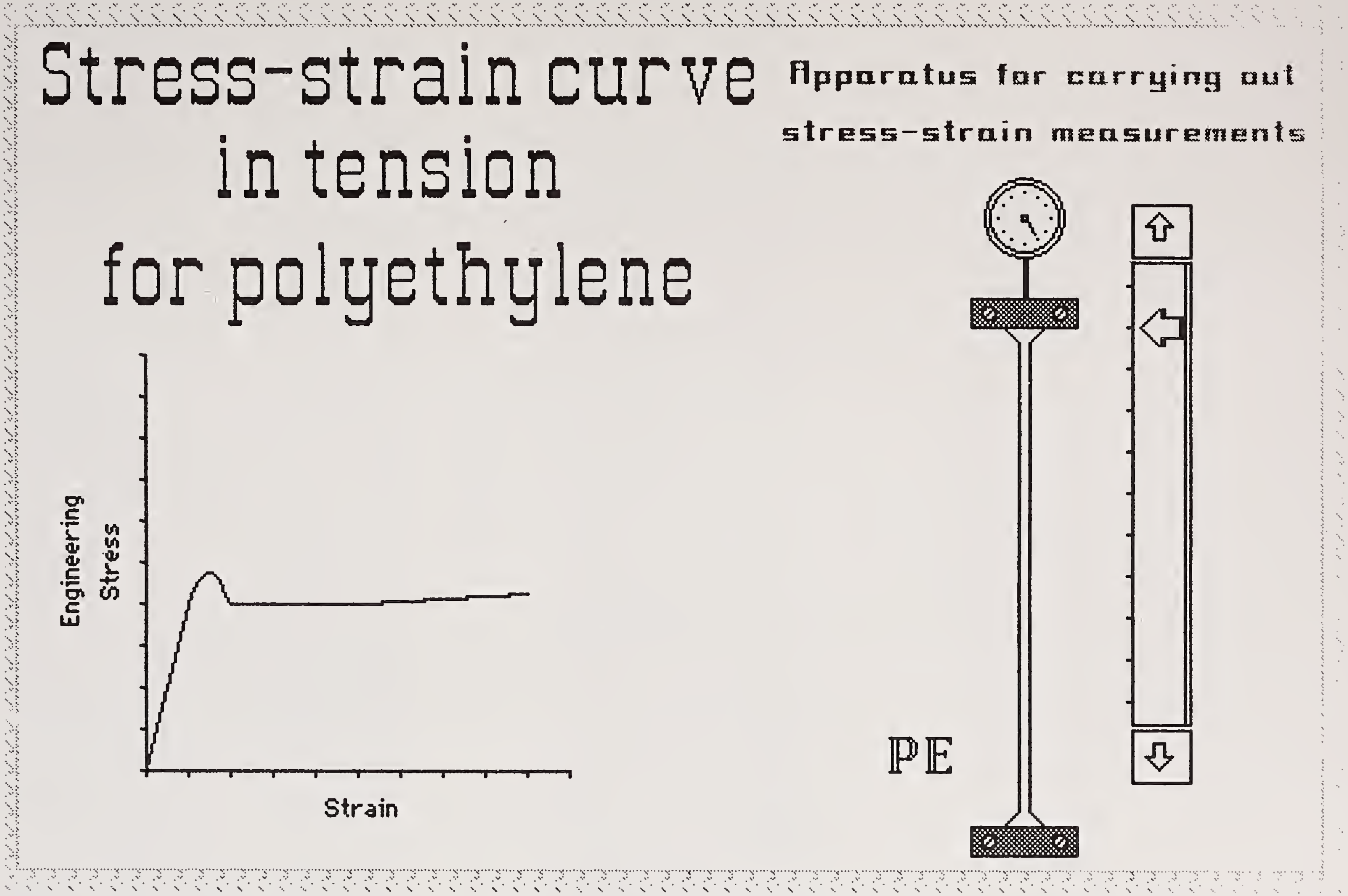


$$[\eta] = \lim_{c \to 0} \eta_{sp}/c$$

cross-section of typical injection molder




coating machine
Steel metering roll
Doctor blade
Coating dam
Steel applicator roll
Doctor blade
Coated material
Rubber backing roll



Stress-strain curve in tension for polyethylene

Apparatus for carrying out stress-strain measurements

DESIGN PROJECT FOR THE MATERIALS COURSE:

TO PICK THE BEST MATERIAL FOR A COOKING POT

(Including two laboratory experiments and an oral presentation)

Kopl Halperin
Division of Science, Engineering, and Technology
The Pennsylvania State University at Erie
The Behrend College

SUMMARY

We have developed a sequence of laboratories and an oral presentation which comprise a design project in materials. We have chosen a common household object, a saucepan. The first laboratory of the term concerns itself with non-steady-state heat conduction through the pan; the last laboratory of the term tests corrosion of the pan. The students are then asked to make a presentation as if they were competing for the job of materials supplier to a firm considering making saucepans. Each lab group picks the material it feels to be the best, based on the measured properties of thermal diffusivity and corrosion potential, as well as on everything else they have learned during the term and during library research into other properties which may be important. The students are graded on how well they defend their choice of material. The feedback from the students has been good; they seem to have learned something and enjoyed doing so.

INTRODUCTION

Many engineering students take a single course in the properties of materials, which typically includes a lab. This course is a good place to teach some of the fundamentals of design, as the choice of a material is one of the main components of any design project. Typically there is more than one material under consideration for any project. The designer, or the student designer, must trade-off among the better properties of each when attempting to find the optimal material.

We have chosen a simple household object, a saucepan, to illustrate the design process. Saucepans are available made from a variety of materials; this effectively proves that no one material is best for all of the relevant materials properties. The material which is optimal for one property or one application of saucepans may not be appropriate for another property or application. The manufacturer must decide what to market, and the consumer must decide what to buy, and in the case of saucepans, both are design decisions. Table 1 shows the available saucepan materials. Instructors will note that the materials span the range of polymers, ferrous and non-ferrous metals, glasses, and ceramics, and that most of the pots on the market are composites.

We have turned the choice of material for a saucepan into a design project for the students in our Properties of Materials course. At the end of the term, the students, working in groups of two or three, must choose the best material for a saucepan based on everything they have learned in the course, and must defend that choice to their classmates. The class gets a chance to rate how well they feel each choice was defended.

LABORATORY WORK

The students performed two laboratory experiments relevant to this design project. In these, they measured non-steady-state heat transfer and non-steady-state corrosion potential. These two experiments illustrate two of the most important properties of cooking pots; their ability to rapidly and efficiently heat what is in them, and their ability to withstand corrosive environments such as the foods being cooked in them. This latter property is one of the important ones for determining how long the pot will last, and it is important for ease of cleaning, and for toxicity. Both experiments are non-destructive.

The two experiments have been described elsewhere (1,2). Briefly, in both of them each student brings in a small saucepan. The instructor at this point owns a fairly impressive array of pots, and generally also contributes one or two to the experiment. In the heat transfer experiment it is important to rule out the effects of the geometry of the cooking surface, and so pots of similar geometries are compared to one another. It turns out that material dominates geometry (1).

In the heat transfer experiment, 500 ml of water is poured into the pot and the pot is placed on a pre-heated electric burner or over a flame. We have found the flame to work better; the electric burner makes uneven contact with some of the pots. Of course, ability to work on an electric burner is a criterion in the design of a saucepan. The temperature of the water is monitored constantly and recorded every 15 seconds. The experiment is terminated when boiling is reached or when a couple of minutes of constant temperature readings occur. The experiment is repeated with each of the other pots. The results are compared to tabulated thermal conductivities and diffusivities, and it is generally obvious that the material with the highest heat transfer coefficients heats water the fastest, as expected.

The heat transfer experiment is performed first in the term as a simple way to introduce the students to what is expected of them on a lab report, and in order to introduce the concepts of design early on. In addition, the students have already had an introduction to heat transfer, and are thus familiar with the properties involved. We have found this to work out well.

Figure 1 shows a student plot of data from the heat transfer experiment.

The corrosion experiment is performed at the end of the term. Corrosion is a property studied in the properties of a materials course, and thus is familiar to them by the end of the term. And since this experiment is once again one involving design, it comes at the end as a wrap-up, and a way to lead into the final oral presentation, the design project.

In the corrosion experiment, 500 ml of 3 wt % table salt solution or of vinegar is placed in each pot. A reference electrode is inserted into the solution, and another lead attached to the pot wall. The potential is measured on a multimeter. The property measured is non-steady-state corrosion potential of the pot. Non-steady-state is, of course, the way saucepans find use. We have had the students heat the pot with the corrosive solution in it. This is particularly useful with stainless steel, which is attacked by boiling organic acids such as vinegar, but not by room temperature ones (3).

The results are plotted as corrosion potential against time. The results are compared to published corrosion rates, and follow as expected. The only pot material in which surprising results are gotten is stainless steel; for stainless, the non-steady state corrosion we measure may not be the same as the steady state corrosion tabulated in corrosion rates tables, as the protective oxide layer apparently needs time to form.

Figure 2 shows a student plot of data from the corrosion experiment.

THE DESIGN PROJECT

The students give two short oral presentations on lab work during the course of the semester. At the end of the term, they are asked to give a more major presentation, a design project involving their data from the two saucepan experiments described above.

Each student group is asked to pick the material they prefer of the ones saucepans are made from. Table 1 lists the options. They then are asked to present a talk as if they were trying to convince a manufacturing firm to buy their material to use for making saucepans.

Each group has to list the important properties that a saucepan should have. Some of these properties are shown in Table 2. The students must do library research on at least one property that they did not test. In their presentations, they show data from their two experiments and from their library research to back up their argument as to why their material has the optimal combination of properties.

There is no right or wrong optimal material for making a saucepan. The grading is on the effectiveness of the presentation, the depth of the research, and on whether the group made a convincing case for their material as the best one for the job. The students get a chance to evaluate the effectiveness of their peers, in writing.

CONCLUSIONS

The range of pots that different groups have chosen for their design was great. Some chose aluminum, because of its heat transfer advantage, some chose stainless, because of a combination of middling heat transfer, good corrosion resistance, and good mechanical properties. Some chose vitreous enamel on steel because of its next-to-top heat transfer properties and excellent corrosion resistance. Some chose cast iron, because of its good thermal conductivity and its nutritional advantages (4). We have learned that students will enjoy and do well at a design project which involves materials which are familiar to the students, and which they can relate to their own lives.

REFERENCES

1. Negrin, Barry; Landry, Adam; Halperin, K.: Heat Conduction in Pots and Pans. J. Mater. Educ., vol. 11, 1989, pp. 397-406.

2. Meehan, Kevin; Halperin, K.: Corrosion Potential of Common Cooking Pots Under Conditions of Simulated Use. J. Mater. Educ., vol. 11, 1989, pp. 407-415.

3. Schweitzer, P.A.: Corrosion Resistance Tables: Metals, Plastics, Nonmetallics, and Rubbers. 2nd ed., Marcel Dekker, Inc., New York and Basel, 1986.

4. Brittin, Helen C.; Nussaman, Cheryl: Iron Content of Food Cooked in Iron Utensils. Journal of the American Dietetic Association, vol. 86, 1986, pp. 897-901.

TABLE 1. SAUCEPAN MATERIALS AVAILABLE TO THE CONSUMER IN THE UNITED STATES, FALL 1990.

I. Simple Materials

Material	Class
Stamped aluminum alloy	Non-ferrous metal
Anodized aluminum alloy	Non-ferrous metal
Cast iron	Ferrous metal
Borosilicate glass	Glassy ceramic
White ceramic	Polycrystalline ceramic

II. Composites

Materials	Classes
Cast aluminum alloy, PTFE lined	Non-ferrous metal, thermoplastic
Wrought aluminum alloy, PTFE lined	Non-ferrous metal, thermoplastic
Anodized aluminum alloy, PTFE lined	Non-ferrous metal, thermoplastic
Cast iron, PTFE lined	Ferrous metal, thermoplastic
Vitreous enamel on mild steel	Glassy ceramic, ferrous metal
Stainless steel, aluminum core	Ferrous metal, non-ferrous metal
Stainless steel, copper core	Ferrous metal, non-ferrous metal
Stainless steel, aluminum bottom	Ferrous metal, non-ferrous metal
Stainless steel, copper bottom	Ferrous metal, non-ferrous metal
Copper alloy, tin lined	Two non-ferrous metals
Copper alloy, nickel lined	Two non-ferrous metals

TABLE 2. SOME IMPORTANT CRITERIA OF MATERIALS FOR SAUCEPANS

I. Criteria Measured by the Students in the Lab

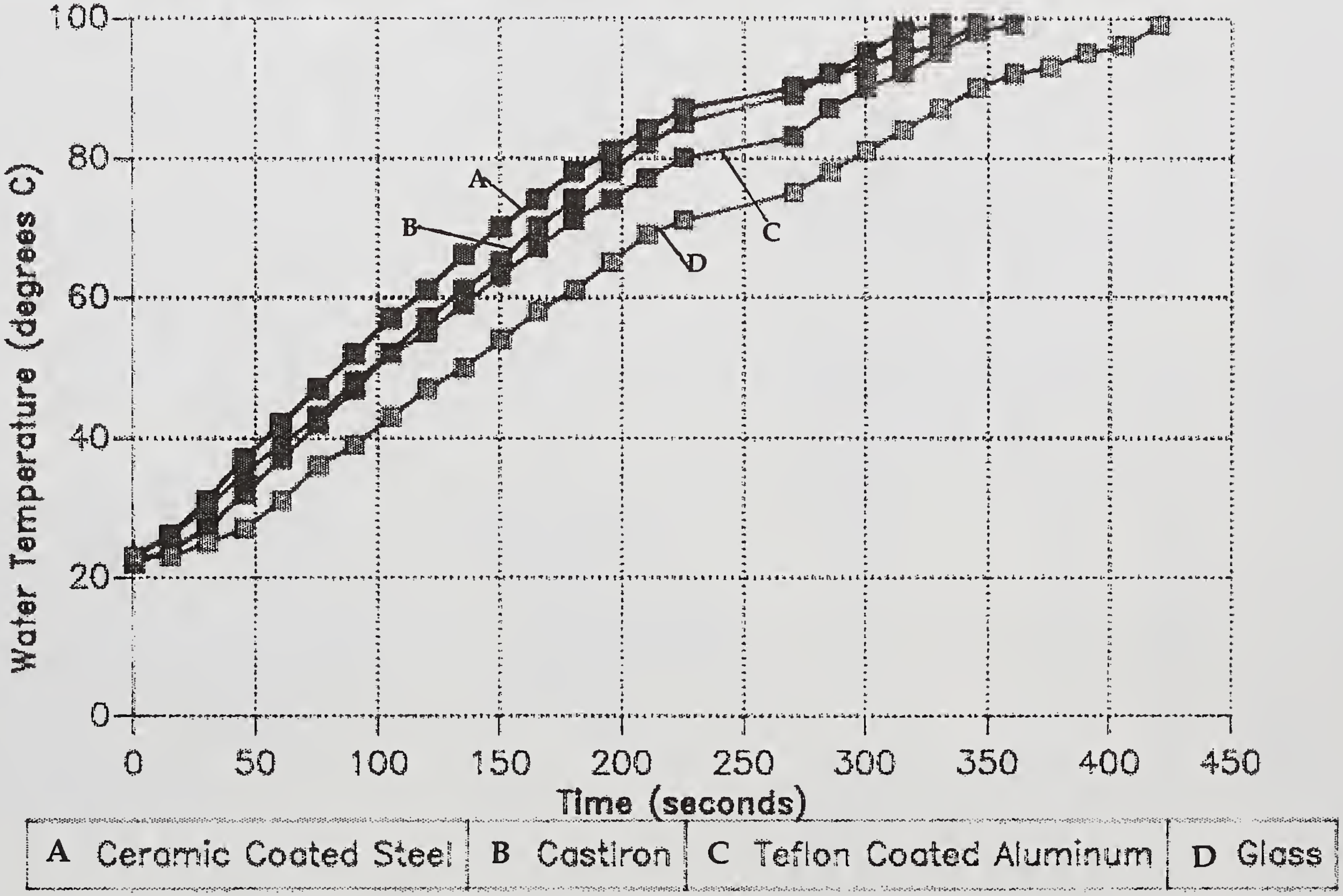
Criterion	Physical Property
Fast-heating	Thermal conductivity and diffusivity
Non-corrosible	Corrosion potential

II. Other Criteria

Criterion	Physical Property
Non-breakable	Toughness
Non-dentable	Strength, ductility
Non-meltable	Melting point and softening point
Non-stick	Friction coefficients
Non-abrasible	Abrasion resistance
Microwavable	Electrical conductivity
Inexpensive	Extraction and forming costs, marketing
Nutritious	
Non-toxic	Bio-chemical properties

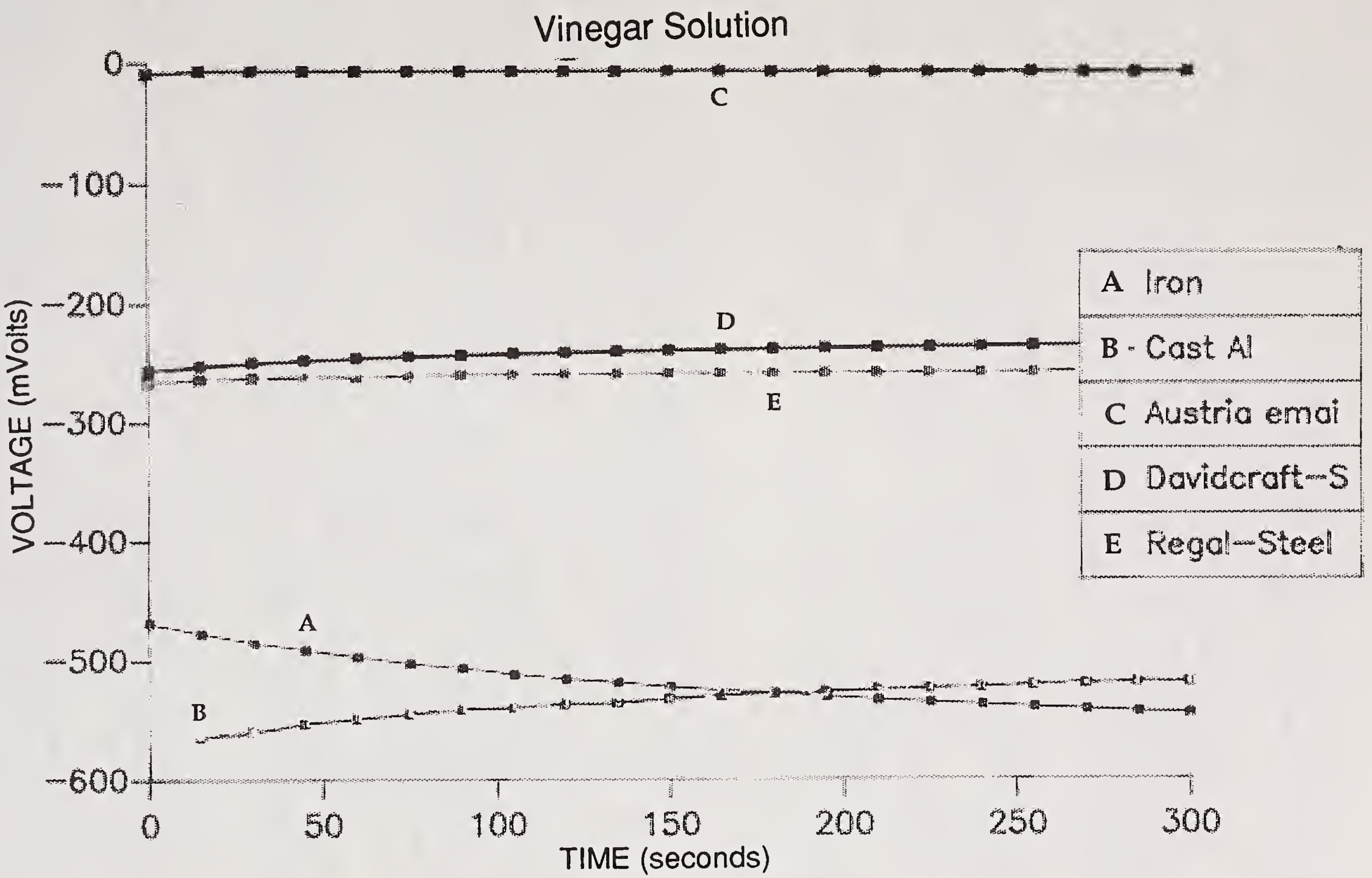
Figure 1

Water Boiling Times for Various Pots



from: Albert Byers, "Testing of Cookware for Comparison of
Thermal Conductivity Values," Lab Report, Fall 1990.

FIGURE 2
VOLTAGE vs. TIME



from: Donald Dagen, Jennifer Pell & Vicki Roberts, “The Best Cooking Pot for Your Family”, Lab Report, Spring MO.

Appendix 1: Lab handout on heat transfer experiment.

Heat Transfer through Common Pots

Object: To determine the heat transfer behavior of various common pot and pan materials under simulated cooking conditions. To compare those non-equilibrium conditions to the tabulated values of relevant materials properties: thermal conductivity k, or thermal diffusivity. To determine which pot heats water the fastest, and decide if that result is based more on materials properties or on pot geometry.

Specimens: Various pots and pans, depending on what is brought in by different students in different sections. Possibly including: American-made stainless steel, Far Eastern stainless steel, ceramic coated steel, aluminum, PTFE-lined aluminum, cast iron, and glass or ceramic, with some more exotic materials possible.

Special apparatus: Gas burners, thermometer, watch with second hand. Or possibly computer A-to-D board with thermocouples.

Procedure:

1. Light the burners.
2. Choose a pot.
3. Pour 500 ml of water into the pot.
4. Take a reading of the initial water temperature.
5. Place the pot on the burner and simultaneously lower the thermometer into the pot.
6. Take a temperature reading immediately, and again every 15 seconds.
7. Continue taking readings until the water temperature reaches boiling (100 degrees celsius) or until the temperature levels off, meaning the reading is the same for four consecutive times.
8. Dump out the water and allow the pot to cool, run cold water over the thermometer.
9. Select another pot, and repeat.

Plot: temperature versus time for four to six pots on the same plot. Be sure to label the overall plot, the x-axis including units, the y-axis including units. Make sure all data plotted together is from the same burner. Make a second plot for data from the other burner.

Report: The report will be a formal written report, following the standard format I have given you. Discuss the correlation of the data with published values of k. Discuss what you feel is the best pot material for heat transfer.

Note to NEW90 participants: sometimes I have given the students a heat transfer equation to fool with, and sometimes not. Sometimes I have given them a calculation of energy wasted by using the slowest pot, and sometimes not. I own a number of the cheaper pots available; it is an inexpensive lab to run.

Appendix 2: Lab Handout on Corrosion experiment and final project.

Corrosion of Common Pots

Object: To determine the corrosion behavior of various common pot and pan materials under simulated cooking conditions. To compare those non-equilibrium conditions to the conditions of more typical corrosion results. To compare the corrosion results obtained on the pot and pan experiments to the more typical galvanic series, as in Table 21.1 from M.F. Ashby and D.R.H. Jones, **Engineering Materials**, Pergamon Press, Oxford, 1980. To combine the corrosion data with other data on pots and pans, to pick the best possible pot material.

Specimens: Various pots and pans, depending on what is brought in by different students in different sections. Possibly including: American-made stainless steel, Far Eastern stainless steel, ceramic coated steel, aluminum, PTFE-lined aluminum, cast iron, and glass or ceramic, with some more exotic materials possible.

Special apparatus: Reference electrode, voltmeter or computer A-to-D. Or one can use a strip of low-potential material for the reference electrode, for example copper.

Procedure:

1. Make a solution of salt water. Our solution will be 10 g NaCl in one liter water. You must calculate what that is by weight % and by mole %.

1A. Make an acidic solution of 1/2 vinegar and 1/2 water. The acidity of this is nominally 2.5%.

2. Choose a pot, pour 500 ml of the salt solution into the pot.

3. Lower the reference electrode into the salt water. Attach one lead to the reference electrode, the other to the wall of the pot. The two leads go to a voltmeter.

4. Measure the initial voltage, and the voltage subsequently at 15 second intervals, until 5 minutes. In most pots, these voltages will be negative.

5. Vigorously scrape the bottom of the pot (unless it is Teflon lined aluminum) with a glass rod. Glass is chosen so that it adds nothing to the water, being harder and more inert than pot materials. A steel spoon might give spurious readings by corroding itself.

6. Then make the same measurements as in the previous step.

7. Repeat the above steps with the remaining pots, and with acidic solution.

Plot: voltage (remember it is usually negative) versus time for each pot, scratched and unscratched, separately for salt solution and acidic solution.

Report: I´ll want the following plots: One for each pot, scratched and unscratched on the same plot. Plot one for all the pots unscratched, on the same plot. In your discussion, compare this kind of experiment to the equilibrium data of the charts you have. And discuss what the scratching does. Discuss what you feel is the best pot material for corrosion, and then discuss the best pot material in general, taking into account your pot heat transfer data.

The report will be the last oral presentation, and will include the results from this lab and from the heat transfer one at the beginning. Taking into account corrosion, heat transfer, and any other variables you feel are important (with bonuses given for good discussion of extra variables), answer the question (and defend your answer) What is the best cooking pot material? 8 minutes per group.

CRYSTAL MODELS FOR THE BEGINNING STUDENT

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Baltimore, MD 21210

SUMMARY

A set of hard sphere models for the B.C.C., F.C.C., Hexagonal, and H.C.P. crystal structures has been produced to help materials science students to understand the relationships of the atom positions in these and related structures. The models consist of sets of arrayed vertical rods and uniform size spheres containing oversized diametral holes for easy placement onto the rods. Many unit cells result that can easily be assembled and disassembled one sphere (atom) at a time. The crystal models are constructed so that nearest neighbors touch and the important planar as well as directional linear arrays are easily visualized and identified. The effect of stacking sequences, vacancies, and impurity atoms can also be shown quite dramatically.

INTRODUCTION

Ball and stick models of the crystal systems have been available for quite some time[1]. Experience has shown that these models have been very useful but do not show many unit cells, and consequently can be misleading as to nearest neighbors, size and position of interstices. Models which are glued together invariably show only certain planes and relationships and consequently can also be misunderstood because important crystallographic information is rendered either partially or totally invisible. Ruoff pictured some very interesting models which seem to alleviate many of these difficulties[2]. These models represented many unit cells and were constructed in such a way that the nearest neighbors touched and the individual atoms could be removed one at a time to show various planes, interstitial sites etc.. Models for B.C.C., F.C.C., and the hexagonal system were designed and constructed based on Ruoff's pictures.

DESIGN

The calculations for the spacings and placement of atoms in most crystal systems are straightforward once the size of the atom is known. Hard wooden spheres, nominally one inch (2.54 cm) in diameter, with a 1/8 inch (0.3175 cm) diametral hole were selected based on the desired size of the models and ease of visualization. Various colors were selected for ease of demonstrating ordered structures and stacking sequences. The spheres were each measured for uniformity of size and placement of the diametral hole. Upon inspection only a few spheres were rejected, none for size, although since the spheres were painted the diameter was 1.025 inches (2.604 cm). The spheres which were rejected had holes which were off center. The eccentricity was checked by placing the sphere on a 3/32 inch (0.238 cm) rod and rotating the sphere. Visual observation was sufficient to determine eccentricity.

The number of unit cells to be shown for each crystal system to allow for clarity of observation was determined to be at least twenty seven unit cells. Twenty seven might seem to be a large number of unit cells but experience with smaller models met with mixed success. With the above restrictions, the design concluded with the selection of 3/32 inch diameter Type 303 stainless steel for the rods and Micarta for the base material. The Type 303 stainless steel was selected for durability and rigidity, while Micarta was selected because there was a scrap supply made available. Any hard wood will probably work as well. The base layout patterns for the three models are shown in Figures 1-3. These patterns show the placement of the stainless steel rods. The holes are 1/2 inch (1.27 cm) deep and the rods extend approximately six inches (15.24 cm) above the base. Note that in the case of the hexagonal system (Figure 3) there are no rods to support all of the possible stacking sequences. Placement of such rods would not allow the spheres to stack at all. Merely dropping a sphere into the system once the first layer is placed will give one of the other two stacking sequences and the third sequence can be demonstrated by dropping spheres on the second layer. Photographs A-E show some of these models assembled to demonstrate the B.C.C., ordered B.C.C, disordered B.C.C., the (111) plane of the F.C.C., and the ABC stacking of closest packed planes (F.C.C.). Table 1 lists the supplies and cost information.

THE EXPERIMENT

The student is given the bases with rods imbedded and a supply of spheres, and instructed to place the spheres on the appropriate rods or interstices to construct the basic models. The bases are not labeled in any way in order that the student put some thought into the lattice position of each layer and the appropriate placement of subsequent layers. Once the model is constructed with identical spheres, the student is advised to find various planes, directions and interstices by disassembling the model. The effect of a vacancy, impurity, or interstitial can also be investigated by merely leaving a position vacant or introducing a smaller or larger sphere at a regular lattice site or at an interstitial site. Stacking sequences can readily be studied using the hexagonal model and the relationship between the F.C.C. and hexagonal structures is dramatically seen. Ordered structures can be demonstrated by using different colored spheres in the appropriate positions. Disordered structures can be demonstrated by randomly selecting different colored spheres and dropping them in place. The relationship between primitive and conventional unit cells can also be demonstrated.

EXPERIENCE

The students have received these models with enthusiasm. Quite often the students refer to these models in subsequent laboratory sessions to better understand a particular feature of a crystal or to explain a concept to a classmate. Colleagues have frequently used these models and remarked how useful and straightforward the models are. These models have been used for over 500 hours and have shown no appreciable wear or damage.

CONCLUSIONS

For a modest investment, a set of crystal models for three of the most important crystal systems can be constructed. These models can be assembled, disassembled, and modified to show the fundamental relationships between atoms, directions, planes, and interstices as well as vacancies, impurities, interstitials and stacking sequences. Ordered and disordered structures can be easily demonstrated. More detailed drawings suitable for machining are available upon request.

ACKNOWLEDGEMENTS

The author wishes to express his gratitude to Mr. John Ross, friend and Research Associate at The Johns Hopkins University, for his continued encouragement and advice on this project, and to Mr. Walter Krug and Mr. Michael F. Franckowiak for their assistance and the machining of the bases. I also wish to thank my daughter, Stephanie, for typing this manuscript. A special thanks to Mr. Howard Moore, Technical Assistant, Loyola College Physics Department, for his speedy production of the photographs.

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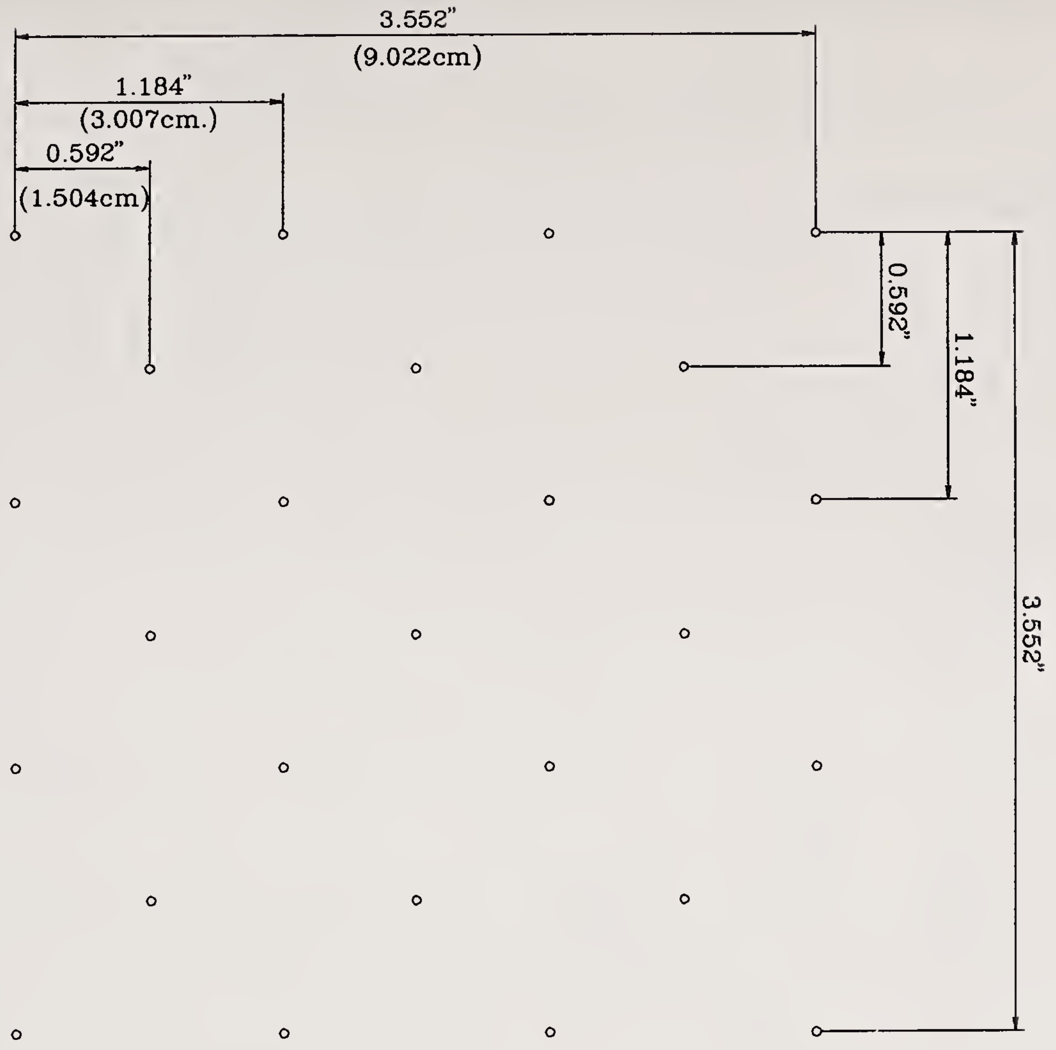


Figure 1

Base layout for the placement of the holes for the B.C.C. model.

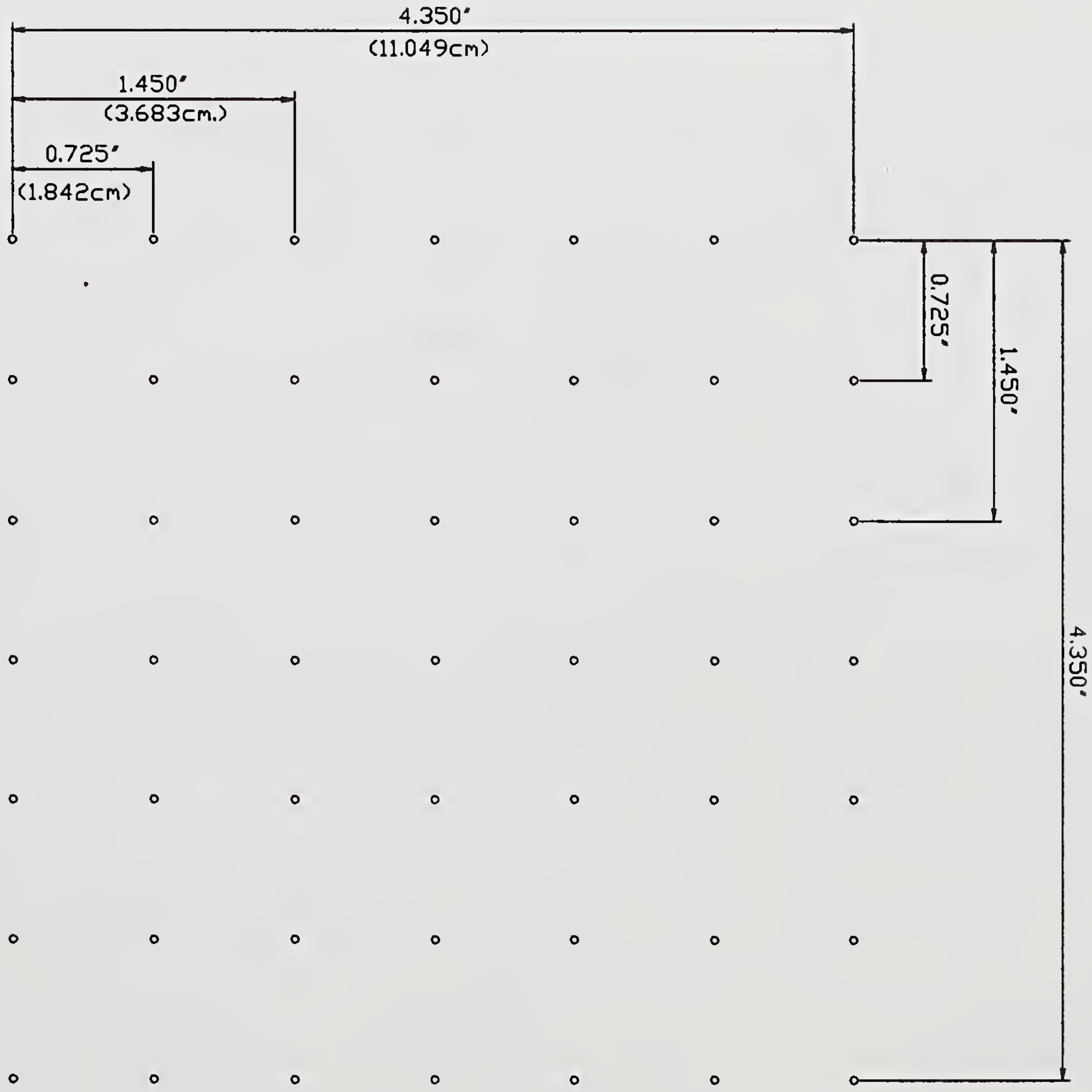


Figure 2

Base layout for the placement of the holes for the F.C.C. model.

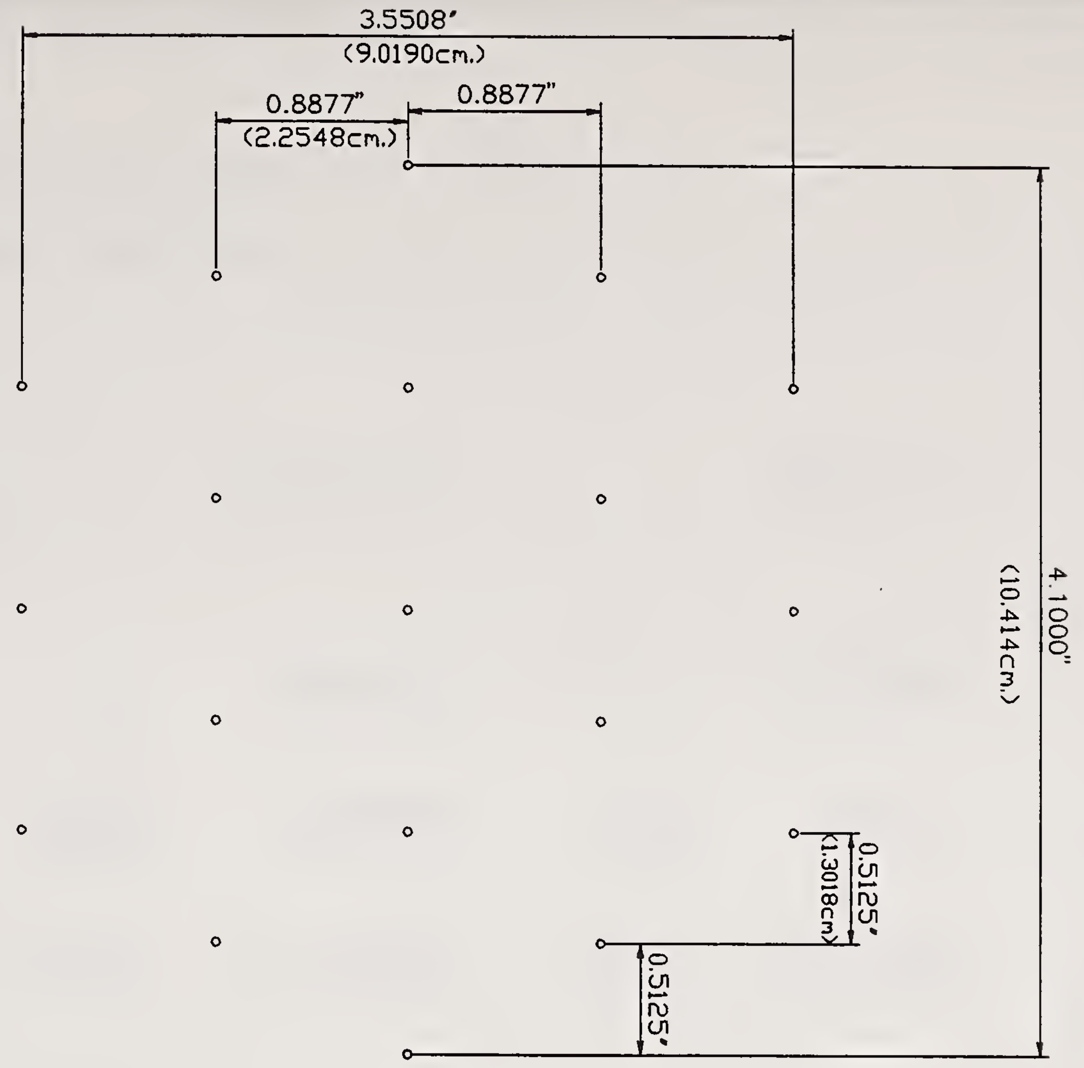


Figure 3

Base layout for the placement of the holes for the hexagonal model.

Table 1

SUPPLIES AND COSTS

		B.C.C.		F.C.C.		Hexagonal
Rods	(25)	$ 4.00	(49)	$ 8.00	(19)	$ 3.00
Spheres*	(92)	38.00	(180)	68.00	(120)	45.00
Machining		50.00		90.00		100.00
Total		$ 92.00		$166.40		$148.60

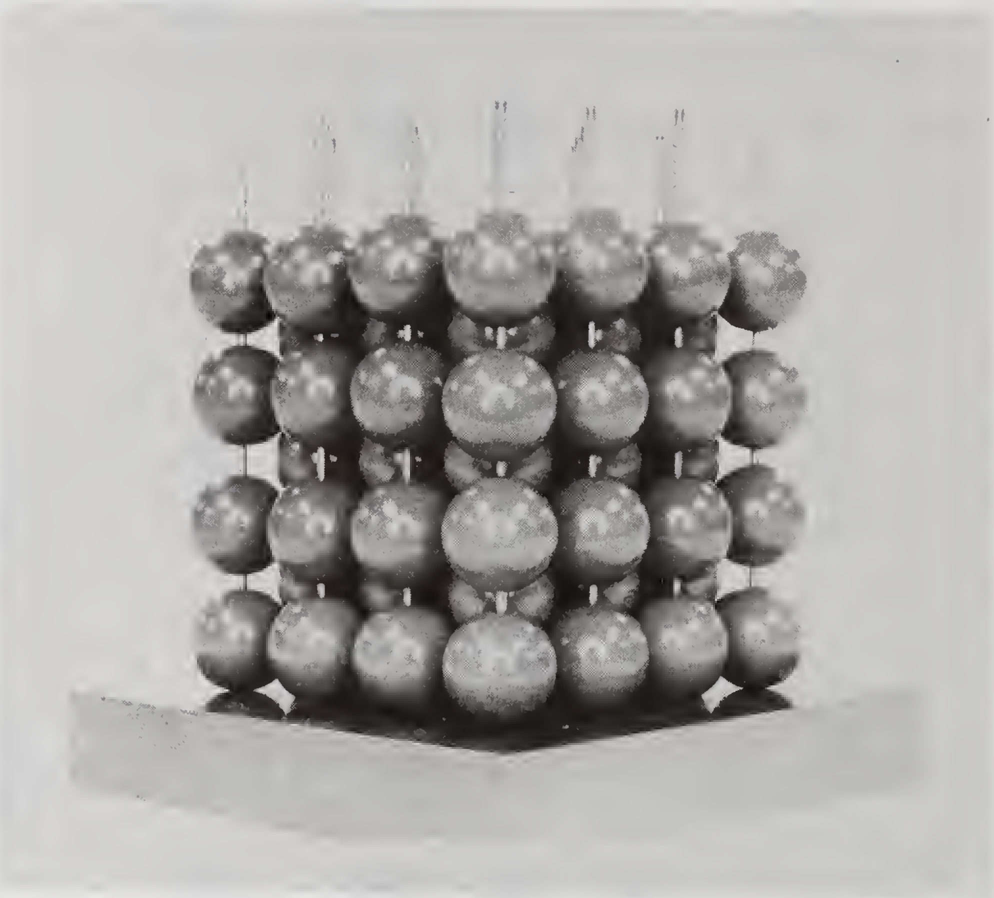
*Available at: Klinger Educational Products Corp.
112-19 14th Road
College Point, New York 11356

Table 1

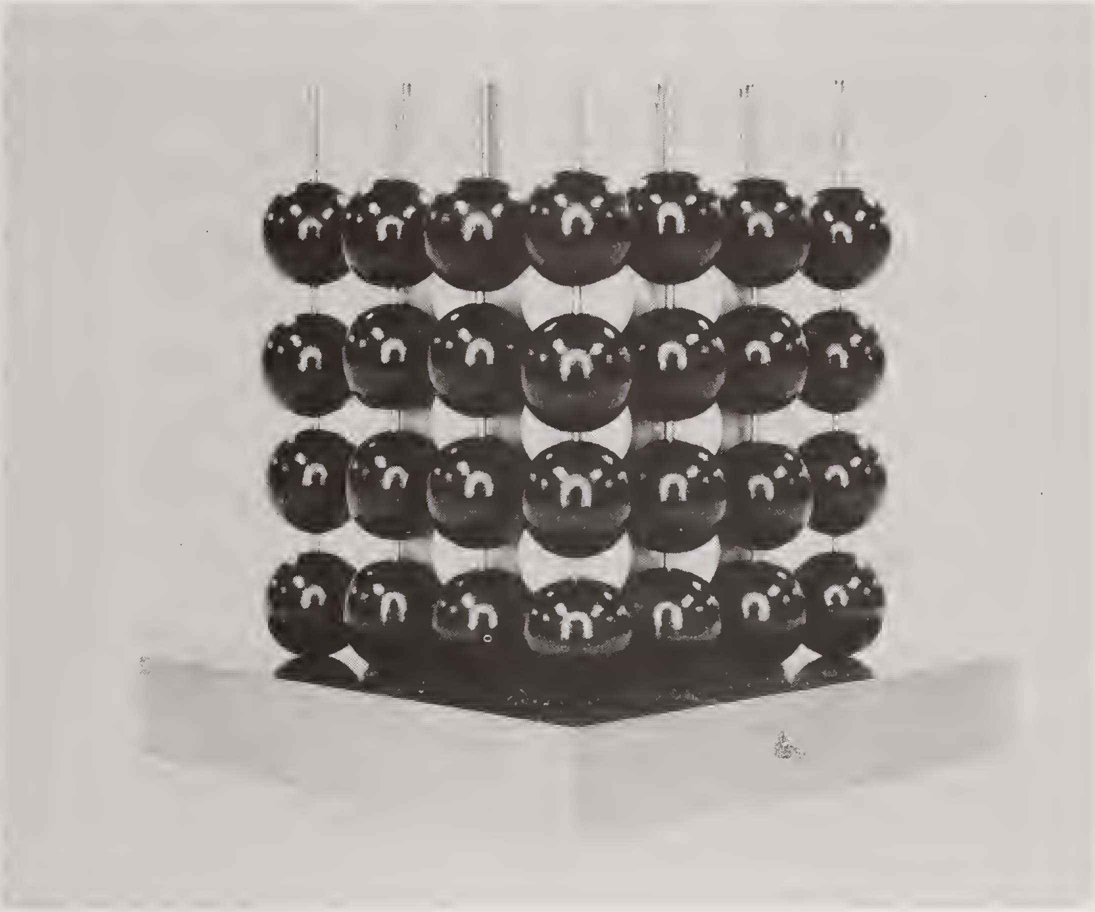
SUPPLIES AND COSTS

	B.C.C.		F.C.C.		Hexagonal	
Rods	(25)	$ 4.00	(49)	$ 8.00	(19)	$ 3.00
Spheres*	(92)	38.00	(180)	68.00	(120)	45.00
Machining		50.00		90.00		100.00
Total		$ 92.00		$166.40		$148.60

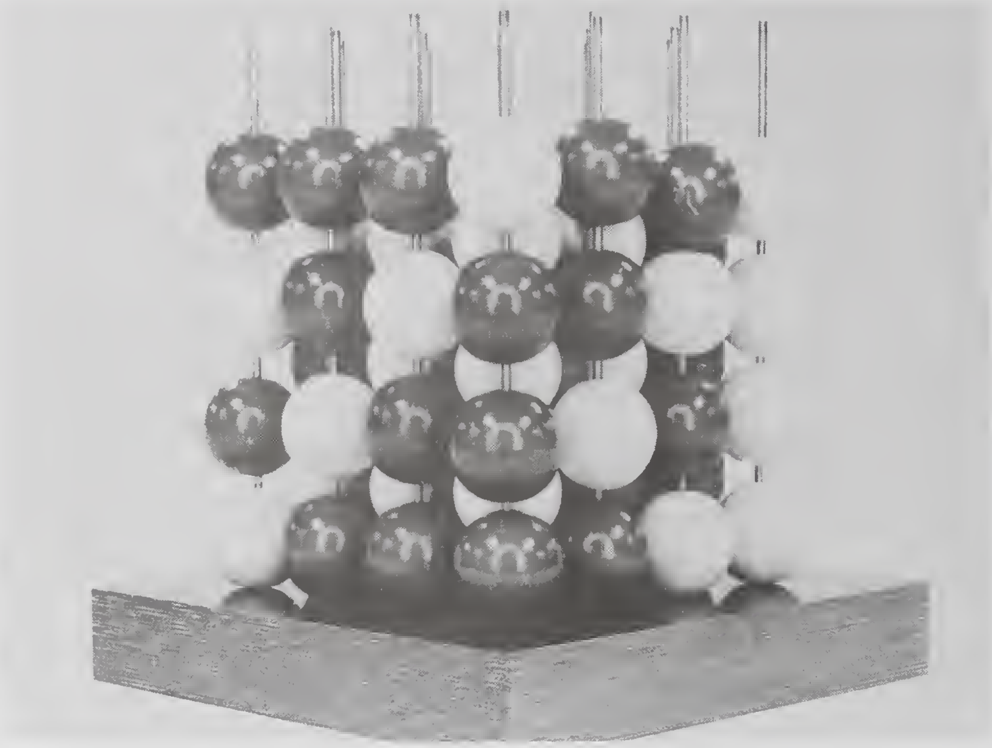
*Available at: Klinger Educational Products Corp.
112-19 14th Road
College Point, New York 11356



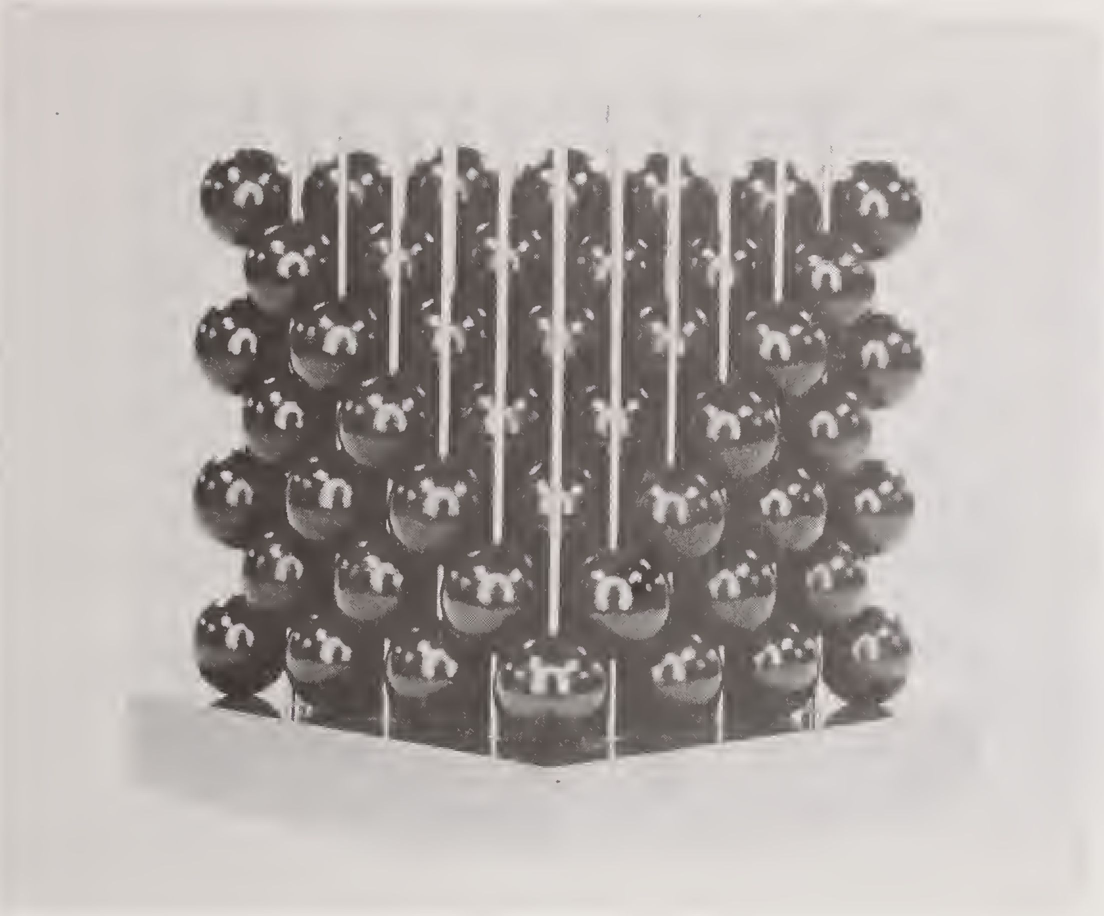
Photograph A
The Body Centered Cubic Structure



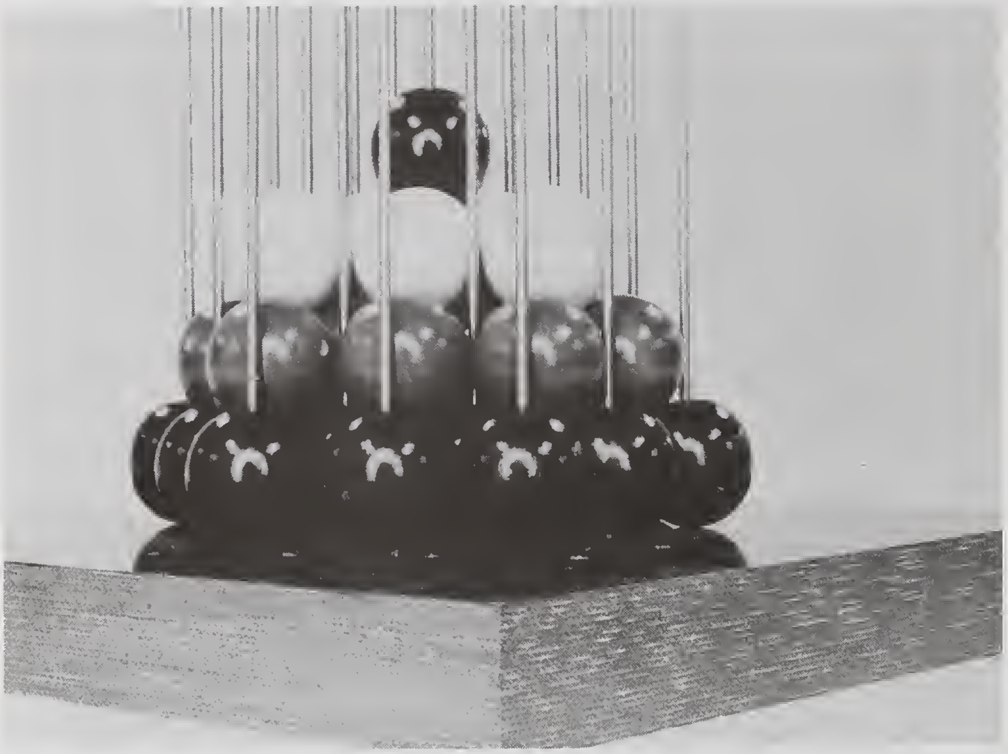
Photograph B
An Ordered Body Centered Cubic Structure



Photograph C
A Disordered Body Centered Cubic Structure



Photograph D
The Face Centered Cubic Structure with the Closest
Packed Plane (111) clearly visible



Photograph E
ABCA Stacking of Closest Packed Planes Demonstrating
the Face Centered Cubic Structure

MICROSTRUCTURE ANALYSIS
(UNIVERSITY AND INDUSTRY PARTNERSHIP)

by Donald H. Martin - Assistant Professor IPFW
and
Gary L. Dawson - Metallurgical Analyst Dana Corporation

SUMMARY:

This paper describes a laboratory experiment using an industrial partnership, utilizing the University and Industry strengths. In Phase I the student will tour the Plant Research and Product Development Laboratories. He will follow all the necessary steps to prepare a material sample and analyze the nodule count and roundness measurements in ductile iron, using the Leco 2001 Image Analysis System. In Phase II the student will be given a known sample and will be required to develop a written laboratory report which will include: title, objectives, procedures, library research, conclusion, and bibliography.

INTRODUCTION:

Industries today are forming partnerships with each other and utilizing each others particular strengths to form the strong, long lasting business directions that are necessary to survive. Education needs to be a part of that long term partnership. Purdue (IPFW) and Dana Corporation are working together to provide the needed real world working exposure to students when it comes to material testing and analysis.

OBJECTIVES:

1. To understand the methods of preparing and evaluating metallographic specimens, and to familiarize the student with the fundamentals of metallography.

2. To visit an Industrial Metallography Laboratory and follow the process of obtaining, preparing, examining, interpreting, and photographing representative metallic specimens.

3. To provide for the identification and evaluation of known material samples (material analysis and reporting) including lab report & research.

EQUIPMENT:

Dana Corporation
1. Abrasive wheel cut-off saw (12")
2. Sample Mounting Press
3. Roll Grinder Sample Polisher
4. Hardness Testers
5. Image Analysis System (Leco 2001)

Purdue

6. Set of Known Material Samples
7. Specimen Mounting Equipment
8. Polishing Equipment (250, 400 & 600 grit)
9. Acid Etch (2% NITAL)
10. Metallurgical Microscope
11. Camera for Photomicrography

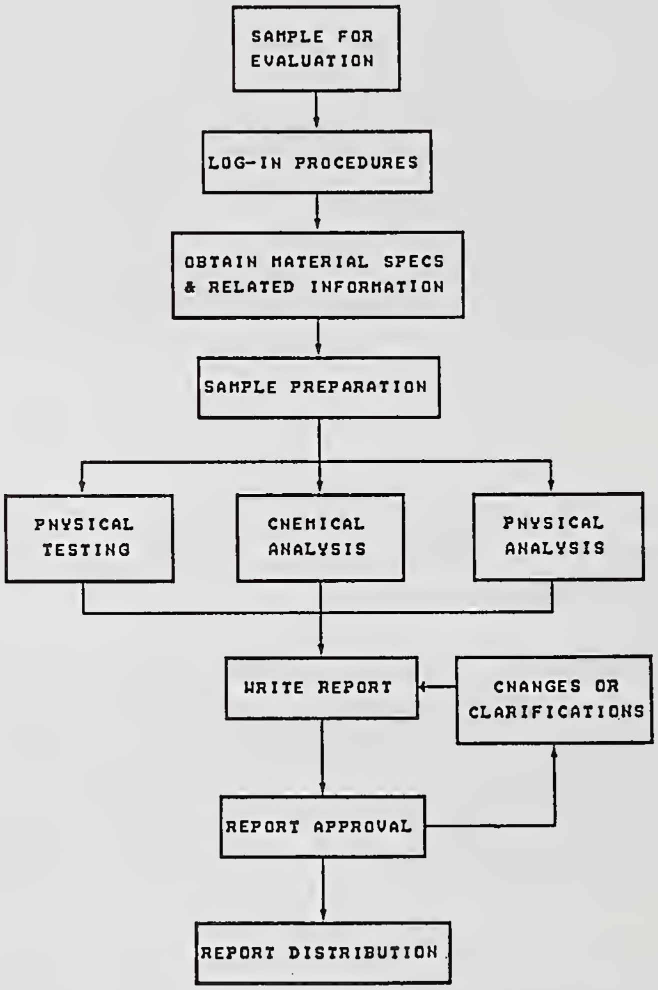
PROCEDURE:

PHASE I: DANA CORPORATION LIGHT AXLE DIVISION TOUR

Dana Corporation - Spicer Light Axle Division, Materials Engineering, Fort Wayne, Indiana recently developed a Laboratory Manual for their complete operation. Included in the Manual is the following analysis flow chart.

MATERIALS ENGINEERING LAB

ANALYSIS FLOW CHART



This pictorially shows all the necessary steps used to process a sample through their Materials Engineering Laboratory.

This is the actual sequence that is followed for testing and reporting material samples in the Dana Corporation - Materials Engineering Laboratory.

During the tour of the Dana Corporation - World Axle Development Center students will observe the following:

1. Axle Design and Development
2. Axle Testing (Machine Endurance Testing/Fatigue Testing)
3. Metal Sample Preparation
4. Metal Sample Testing
5. Leco 2001 Image Analysis System Operation
6. Analysis and Reporting of Data

The Leco 2001 Image Analysis System is a powerful, fast, flexible tool that is used to perform very detailed examination of metal samples. The system is easy to operate and does not require in depth knowledge of computer operating systems (it is menu driven). The operator is able to measure the grain size in steel, nodule count and roundness measurements in ductile iron, length and width measurements of inclusions in steel and particle size distribution of carbides in tool steel. Simply prepare the sample, place it under the microscope and use the Image Analysis System to evaluate the sample for necessary characteristics.

Student response sheet must be turned in the following class period. (Attached)

PHASE II: LABORATORY AT INDIANA-PURDUE UNIVERSITY

MATERIAL: Two (2) mounted and prepared known samples.

DISCUSSION: This lab provides a brief introduction into the topics of metallography and metallic microstructures. Metallography can be defined as the study of the structure and physical properties of metals and alloys, especially by the use of the microscope and X-rays (taken from Webster's New World Dictionary). Microstructures can be thought of as the structure of a metal or alloy as seen under a microscope.

PROCEDURE: (The following is a brief procedure for preparing a sample for microscopic examination).

1. If the specimen is smaller than 1/2 inch in diameter or in length, it must be mounted in bakelite in order to provide a sample large enough to be easily handled.
2. The specimen should be ground down to a flat surface on a belt sander.
3. The coarse scratches are removed by using progressively finer grades of emery paper. The direction of sanding should be 90° or perpendicular to the direction of the scratches. As the different grades of emery paper are used, the specimen will be rotated 90°.
4. When changing emery paper grades, i.e. going to a finer grade, the specimen should be thoroughly washed and dried to remove any remaining course grains of emery which could accidentally scratch the surface as sanding and polishing continues.

5. When a highly polished surface is achieved, free of any scratches, the specimen should be washed in an alcohol bath and dried in a warm air blast to prevent oxidation of the polished surface.
6. Normally a specimen would be etched. Etching removes parts of the polished surface, causing the microstructure to stand out or develop more clearly. The etching solution to be used depends upon the type of specimen and the test results sought.

   (Since the samples were already mounted and polished, experiment began with Step #7.)
7. Finish polish the metal specimen if necessary to remove any heavy scratches.
8. Examine each specimen using a metallurgical microscope at high power. Sketch the general outline or shape of the sample as well as the observed microstructure, noting any significant features.
9. Make a Rockwell hardness reading of each sample, recording the reading and the cross-referenced Brinell hardness reading.
10. Look up the microstructure of each sample, and using the hardness data, attempt to identify each structure or sample.
11. Write Laboratory Report following the format and instructions for laboratory reports (must include research and references).

LABORATORY REPORT FORMAT AND INSTRUCTIONS

TITLE: State name of experiment. If possible, briefly state primary and overall--range--objectives.

OBJECTIVES: List step-by-step sequence of making experiments, if possible to predict.

EQUIPMENT AND ACCESSORIES: Name and sketch; describe operating principles of equipment. Itemize any standard accessories to adapt equipment for test.

TOOLS AND SUPPLIES: Hand tools--standard or special, expendable items, handbooks or tables, special grips, clamps, scales, gauges, measuring devices or others, sandpaper, crayons or special marking devices, clay, magnets, mirrors, rags, towels, drip pans.

SKETCHES: Block diagrams, flow charts, system diagrams, pictorial, cross section, assembly, detail, mechanism, plumbing, electrical, structural, method sketches to illustrate complete operation details. (Frequently, a complete knowledge of small details will point out errors or fallacies in experimental methods and erroneous results that lead to wrong conclusions.)

DATA: Take standard type data over wide enough range to include any pertinent changes, or change in trends.

Record all variables that could have slightest effect on the test. Re-evaluate later during analysis and write-up for further check on their effect. List data to be taken including initial, periodic, and final readings.

GRAPHS: List requirements with x/y/z coordinate titles and units. List required graphs based on both direct readings and calculated data.

WRITE-UP: Include graphs, sketches, drawings, applicable equations with sources and units, calculated results, comparisons with data of other researchers (including other teams and library references), reasons for variances between tests. Discuss improvements you might make in a repeated experiment, such as sample selection, method, technique, equipment change, etc. In your write-up include description of any special or new skills or techniques you learned and include sequence of making the test or experiment.

CONCLUSIONS: Summarize knowledge that has been verified or expanded and give possible utilization in industry. Briefly list any possible directions or goals to expand knowledge for more efficient or better usage or processes or materials.

REFERENCES: List complete bibliography.

DANA CORPORATION LIGHT AXLE DIVISION TOUR
STUDENT RESPONSE

The following are three questions that will be answered during your tour at Dana. The three areas you will be touring are the Engineering Test Lab, MET Lab, and Technical Service Department. You may find it interesting how all three areas are related. Please answer the questions and return to your instructor per his instruction.

1.) What is the importance of a wet cut vs. a dry cut as far as a metallurgical analysis and how does each effect the physical properties?

Answer: Dry - It can heat the part and cause microstructural transformation and alter hardness values

2.) Using an IA (Image Analysis) system, how can you ascertain the density of PM (Powder Metal) components?

Answer: (100% - % porosity) x density of steel

3.) Name two types of actuators that are used in fatigue testing and why are both types required?

Answer: To simulate road or running conditions, the component must be exposed to the same type of loading experienced in the field. (E.I. axle shafts = rotary: Spindles = linear)

NOTES FOR THE LABORATORY INSTRUCTOR
AT IPFW

MACROGRAPHIC (Qualitative) - Samples evaluated by eye, or by magnifier (to 30 x). Sample preparation is simpler, inspected areas are larger, and interpretation is generally descriptive. Some specimens may be smoothed and etched, but unprocessed pieces may be adequate. For example, a tensile or bending sample exhibits ductile necking and cup-cone fracture, whereas another sample shows a brittle glassy-smooth fracture and no necking.

MICROGRAPHIC (Quantitative) - Samples evaluated by metallurgical microscope (reflected light, 30 x to 2000 x). Sample preparation is much more critical, smaller areas inspected and measured, with statistical interpretation of data. Therefore, specimens are carefully polished (with aluminum-oxide abrasive or diamond paste) and then lightly etched (with acids or bases).

SAMPLE PREPARATION:

MACROSCOPIC - Sample cutout by torch or hacksaw. Specimen sectioned by "Abrasimet" water-cooled cutter. Burrs ground off. Surface planed by belt-sander and smoothed on table-grinders (water-cooled). Polishing by No. 00 or 000 (size) emery paper may suffice. Avoid frictional heating, structural distortion, and obliteration of features. Then, sample is alcohol-wiped (degreased), water-dipped (heated), and completely wetted by reagent (etched). A 10% solution of nitric acid ($HNO3$) is used for steel structures; forged-steel flowlines and heat-treating quench cracks are revealed by 50% water solution, of hydrochloric acid (HCl) or 25% solution of nitric. After deep-etching is complete, the sample is scrubbed in hot water, dipped in benzene or acetone, and dried in warm air-blast. When a dry surface is coated with printer's ink, an impression may be transferred to art-paper. Also, a simple macrograph may be obtained by pencil-rubbing or photography.

MICROSCOPIC - Samples are obtained and smoothed as before. However-flame-cutting and frictional over-heating should be avoided, because <u>all</u> surface and sub-surface distortion must be removed by fine polishing and light etching. Then, a thin section is cutoff (water-cooled) and mounted in thermoset plastic (<u>Bakelite</u> or <u>Lucite</u>). Remove plastic "flash", lightly round edges (top and bottom), carefully flatten the sample surface (by wet-belt sander) and leave only parallel scratches. After thorough washing (hands and sample) in soap and water, start the complex process of Grinding and Polishing. (Instructions posted at the machines). Examine polished samples by microscope to detect defects, which require repolishing. Ideally, a <u>mirror-surface</u> (scratch-free at 1000 x) is achieved before etching. Handbooks are used to selectreagents, strengths, and applications. For high magnifications (above 100 x) only a <u>lightly-etched</u> surface is wanted. Additional etching is easy - <u>over etching</u> requires considerable repolishing.

Finally, rinse etchant off (warm, running water), flush with alcohol, and dry with warm air-blast. The sample is ready for final microscopic study.

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A NONDESTRUCTIVE TESTING METHOD TO DETECT DEFECTS IN STEEL PLATES

BY

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KEY WORDS: Nondestructive Testing, Modal Analysis, Frequency Response Measurement

ABSTRACT

The vibrational response of a structural system to random excitation contains a unique characteristic signal for that structure. Using proper signal analysis techniques, this signal can be retrieved from the random response. A study of this characteristic signal, which contains information of system behavior, can be used to diagnose system performance. This study examines the dynamic response of two similar metal plates and evaluates the changes in dynamic properties due to changes in mass, stiffness and damping. This dynamic response method will use frequency and modal domain analysis for identifying the dynamic parameters of the system. The test objects will be tested before and after introducing simulated defects. A metal rod will be tested similarly to determine the stiffness co-efficient of the material.

OBJECTIVES

The objectives of this experiment are as follows:

i) To relate changes in stiffness, mass, and damping to dynamic parameters of a structure.

ii) To compute the stiffness of a steel rod by evaluating the dynamic response function.

THEORY

A structure when vibrating freely, no external load acting upon the system, tends to assume a vibrational pattern which is a function of the material and the boundary conditions of the structure. A freely vibrating system contains information which is unique to its type and can be studied for extracting the structural behavior of the system. Dynamic information of such vibration can be measured and expressed by different parameters such as frequency, damping, and modes of vibration. Experimentally these parameters can be measured by using a dynamic analyzer. An analysis of the dynamic properties obtained from such a nondestructive testing procedure can then be used to identify the structural strength, integrity or possible defects.

An experimentally measured dynamic response function can also be used to compute the stiffness of a structural member. For a known value of fundamental frequency (ω_n), the stiffness coefficient, E, of a structural member can be determined from the standard equations for natural frequencies, as found in the literature [1]. For a free-free rod the formula for determining the stiffness coefficient E, equation (2), is obtained from the standard equations for natural frequencies of a free beam as shown in equation (1).

$$\omega_n = C_n\sqrt{\frac{EI}{mL^4}} \qquad \cdots\cdots(1)$$

where ω_n is the natural frequency for the nth mode,
C_n is a constant,
E is the modulus of elasticity,
I is the moment of inertia,
m is the mass per unit length,
and L is the length of the rod.

$$\therefore E = \frac{(\omega_n^2)mL^4}{C_n^2 I} \qquad \cdots\cdots(2)$$

The dynamic properties of a structure are functions of its mass, stiffness and damping. A change of these properties, which may result due to many reasons, such as degradation, or material fatigue, will have a direct influence on the dynamic behavior of the structure. Conversely, therefore, changes in the dynamic properties can be used to study the changes in the structural behavior of a system. The trend of such variation in the dynamic behavior will be used to diagnose the structural performance as well.

MATERIAL SET-UP AND EQUIPMENT

A steel rod of length 25.35" (64.4 cm.) and mass 1.416 lbs (642.3 gm) was used to collect the data for the stiffness measurement. The diameter of the rod (ϕ) is 0.50394" (1.28 cm). To determine the dynamic response of the rod for a near ideal free-free condition, the rod was suspended at both ends by rubber bands. The lower natural frequencies of the rubber band do not affect the frequencies of the steel rod, and so this method was taken as being a very close approximate to the free-free condition. The rod was divided into 10 equal segments consisting of 11 nodal points. The rubber bands were placed at nodal points 3 and 9.

Two metal plates, designated as "A" and "B", are used to collect the data for the structural performance experiment. Both plates are same in size and shape, except that the plate B has a three-eighth inch tapped hole in it (see Figure 2). Plate A is tested for two conditions, with and without a cut. This test is performed to examine the variation in the dynamic properties between the defective and undefective cases, due to a change in the stiffness about the stronger axis (a cut perpendicular to the longitudinal axis). A stiffness change is simulated by a one and three-fourth inch cut (a one sixteenth inch band saw blade is used to cut the plate) into its side. Figure 1 shows the location and dimension of the cut in the plate A.

Two experiments are performed on Plate B. One test is conducted to determine the effects of an external mass to the dynamic behavior of the plate. In this case a steel block (2"x2.75"x1") is mounted on the plate with a threaded bolt. Figure 2 shows the mounting location of the extra mass on plate B. In the last test two weatherstripping dampers are added to plate B, as shown in Figure 3, to observe their influence. The self adhesive on the back of the weatherstrip is used to bond the damper to the plate.

Both of the carbon tool steel plates tested have the dimensions of 9"x12"x5/8". Plate A weighs 19.18 lbs. and Plate B weighs 18.82 lbs. The weight of the additional block is 1.54 lbs. A neoprene sponge rubber (3/16"x3/8"x 9") made by Maico Inc, Cleveland, Ohio is used for adding dampers on plate B. Each plate is divided into a grid with 25 excitation points as shown as in Figures 1-3. For all test cases, the plates are simply supported over one and a half inch wide plastic foams. These supports are placed along nodal lines 6-10 and 16-20.

A mid-size impact hammer (PCB model no. 086B03), a 0-500 lbf range, with a steel tip (hard tip) and a mini-size impact hammer (PCB model no. 086C80, 0-50 lbf), with a plastic tip are used to excite the plates and rod respectively. An accelerometer PCB model 303A03 is used for all experiments.

Data Acquisition

A portable Compaq III computer is used for collecting and quantifying the dynamic information of the plates and rod. In the experiment the test objects are struck by a known input force (by the use of the hammer) and the response of the objects are collected by an accelerometer. It has also been demonstrated by the author that a microphone instead of an accelerometer can be used to collect the output response for the test object [2]. A Fast Fourier Transform (FFT) dual channel analyzer (WAVEPAK) is used to digitize the analog responses (the input force and output acceleration) of the test object. This digitized information is processed in the computer by the use of a STARSTRUCT program (a modal analysis program) in order to obtain the basic dynamic information of the plates. This software package also extracts modal information of the test objects.

A maximum cutoff frequency of 8000 Hz for the plates is used to collect the data. This corresponds to a 5 to 10 Hz frequency resolution. A cut-off frequency of 4000 Hz was used for the steel rod.The resolution was adjusted depending upon the range of the well-defined peaks found in the FRF functions.

PROCEDURE

The response of a test object, under any of the above conditions, due to an excitation such as a hammer, when measured by a response measuring device such as an accelerometer, can provide the dynamic information of the system. The following procedure shows the basic technique used to collect the data for all cases.

i) The plate (or rod) is excited at each of the nodal points--the intersections of the grid lines that divide the surface area of the plates into a number of segments--and corresponding response noted. Figures 1-3 show the grid pattern for the plates.

ii) For a fixed response point (keeping the output point constant) the input and output data for the system are collected by exciting the structure at different points one at a time.

iii) An average of at least three hammer strikes are made at each point. The hammer spectrum and coherence plots are monitored for each strike. This ensures a reliable frequency response function (FRF).

iv) The transfer function of the system measured as a frequency response function (FRF) is obtained by simultaneously measuring the input impulsive force and the output response acceleration of the system. The FRF function is the ratio of the fourier transform of the response signal to the fourier transform of the known transient input force, and is computed by the Fast-Fourier Transform (FFT) analyzer.

v) Once the data are collected for all grid points, the saved data are used by the STARSTRUCT program to compute and isolate the mode shapes of the structure. These mode shapes provide the corresponding modal information of the system. The modal analysis program also provides dynamic information such as resonant frequency, damping, and mode shapes for the test object. A mode shape is a well defined shape (waveform) assumed by a vibrating system in which the motion of every particle is simple harmonic with the same frequency. A few of the mode shapes for a plate are shown in Figure 4.

In the modal domain measurement, the deflection pattern of the system, the mode shape, corresponding to each resonant frequency is clearly identified. This information reflects the effects of the actual boundary conditions the structure is experiencing. By applying a curve-fitting technique, modal parameters are derived from the measured transfer function matrix. A complete detailed description of this method of analysis can be found in literature [3].

vi) The modal parameters (resonant frequency, damping, and associated mode shape) are used to determine the stiffness of the steel rod as described in the theory portion of this paper. The information can also be used to diagnose the performance of the plates.

RESULTS AND FINDINGS

Stiffness Effects

The plate A is tested for two conditions, with and without a cut, as shown in Figure 1, to determine the effects of stiffness change. The frequency and damping for these two conditions are compared in Figures 5a,b. In these figures, the dynamic parameters, such as frequency, are plotted for each of the corresponding modes of vibration. Only frequencies of the comparable modes (found in both cases) are plotted and compared. From this plot, it is found that except for the modes whose vibration patterns remain unaffected by the cut, such as longitudinal bending (2L in Figure 5b), and 1st torsion (lT), the remaining modes show a lower frequency for the plate with a cut. This suggests that the dynamic behavior clearly reflects the condition of the structure. The longitudinal bending resistance (bending about the longitudinal axis) of the plate is not decreased by the cut as it is perpendicular to this axis of vibration, and therefore no variation of the dynamic frequency along this axis of bending.

A frequency change due to a change in the stiffness of the plate, however, does not indicate the location of defects since frequency is the global property of a system. Mannan and Richardson [4] have used the mode shape information to compute the system matrices, to locate the faults in an aluminum plate. Modal damping obtained from such a test is not the actual damping of the system. The damping variations are shown in Figure 5a.

Mass Effects

Plate B is tested with and without the addition of a weight attached at node point 24. The damping variation for these two cases are compared in Figure 6. In both cases damping increases with the addition of the mass. The modal frequency for the plate without the added mass is found to be greater than that of the plate with an attached mass. This means that frequency decreases with the addition of a mass to the system.

Damping Effects

Figure 7 shows the variation of damping and frequency for each mode of vibration for plate B with and without a damper. An increase in the modal damping is observed for the plate when the damper is added to the plate. However, except for the 2nd anti-torsional mode (AT2), no change is found between the corresponding modal frequencies.

Estimation of Member Stiffness

An estimation of the member stiffness can be determined from the measured modal frequency (ω) as presented in equation (1). Equation (2) is obtained by keeping all other variables, such as area, length, mass, constant. The FRF for the rod is collected using the above mentioned procedure. Figure 8 shows the frequency response function of the steel rod when the accelerometer was placed at the fourth node, and the rod was excited at the seventh node by the hammer.

The peaks in the graph correspond to each of the modes of vibration, first mode, second mode, etc.. As shown in figure 8, the first bending mode has a frequency of 140 Hz. This mode shape was confirmed by observing the mode shapes as computed by the STARSTRUCT program. The appropriate values are substituted into the Equation (2) and E is calculated for the first bending mode. C_n (equation (2)), for the first bending, has the constant value of 22.3733. E was found to equal $29.145x10^6$ psi which closely agrees with the standard value of modulus of elasticity for steel.

CONCLUSIONS

The experimental results presented here show the application of a nondestructive testing procedure for system diagnosis. A measurement of the dynamic behavior and their variations with different structural and boundary conditions can demonstrate the capability of this method in identifying the performance of a structural system. The study shows a noticeable shifting in the frequency as a function of the mass and stiffness changes of steel plates. This technique can easily be used in the classroom to calculate the stiffness of a structural member.

REFERENCES

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[2] Bissinger, G., and Chowdhury, M. R., (1990), "Comparison of Modal Analysis Measurements with Microphone and Accelerometer on Hammer-Impacted Structures," The Eighth International Modal Analysis Conference, Orlando, FL, Jan. 29 - Feb. 1.

[3] "Practical Aspects of Modal Analysis" (1988), reference materials, Structural Measurement Systems, Inc. San Jose, CA.

[4] Mannan, M. A., and Richardson, M.H., (1990), "Detection and Location of Structural Cracks using FRF Measurements", Proceedings of the Eighth International Modal Analysis Conference & Exhibit, v. 1, pp. 652-7.

ACKNOWLEDGEMENTS

The author is grateful to the North Carolina Board of Science and Technology for its partial support in funding the purchase of Modal Analysis System.

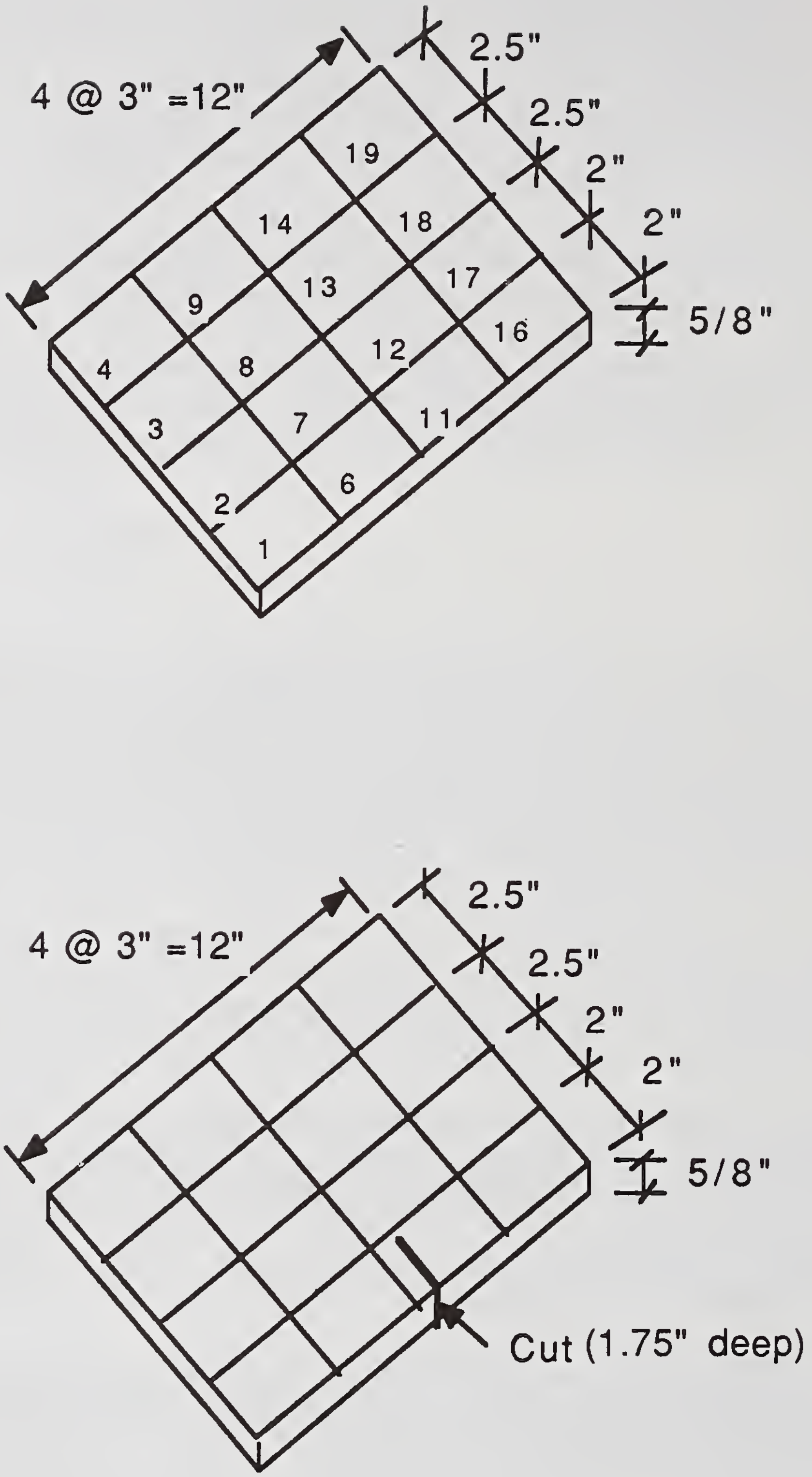

Figure 1 : Plate A with and without a cut

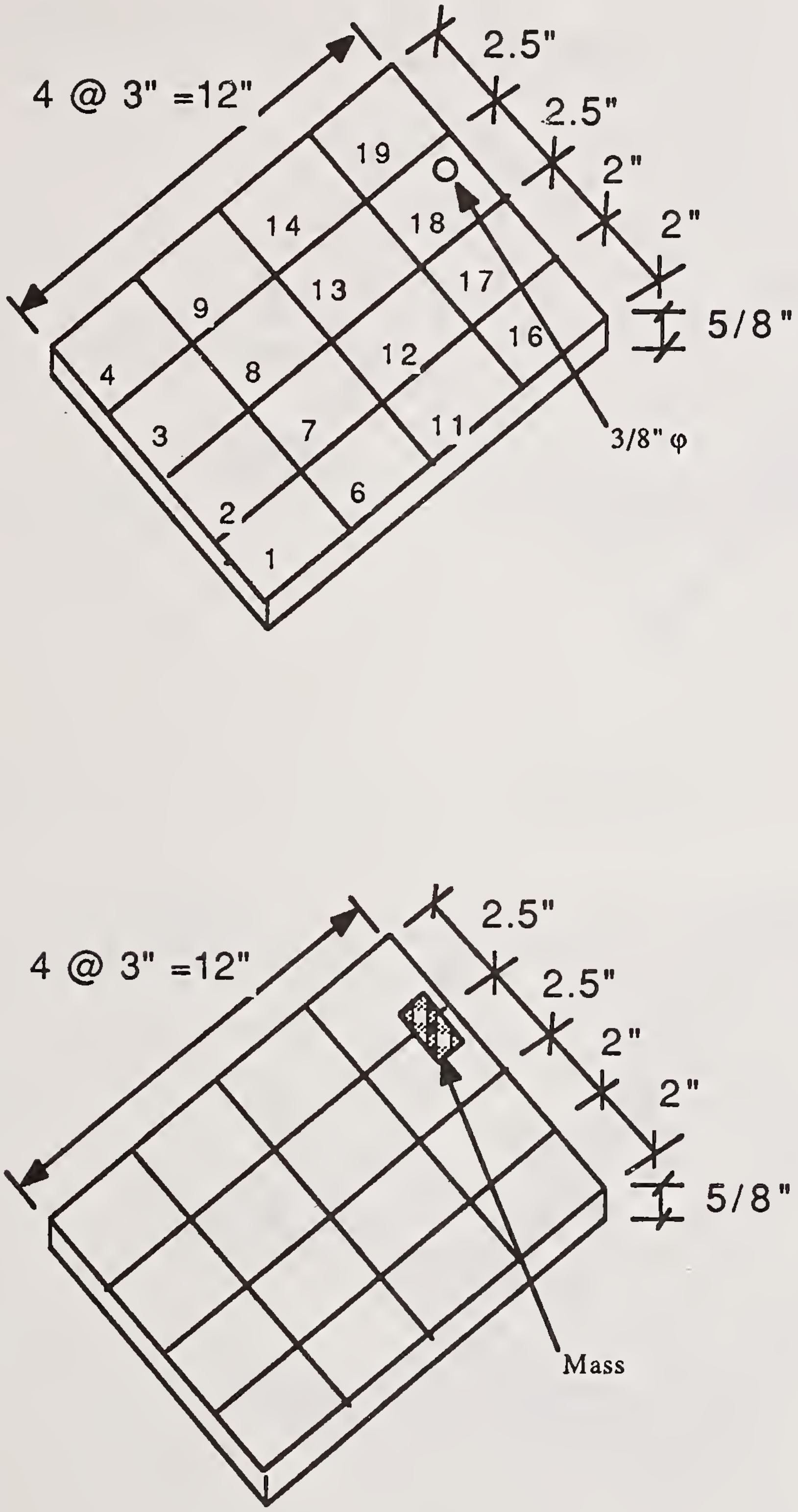

Figure 2 : Plate B with and without an added Mass

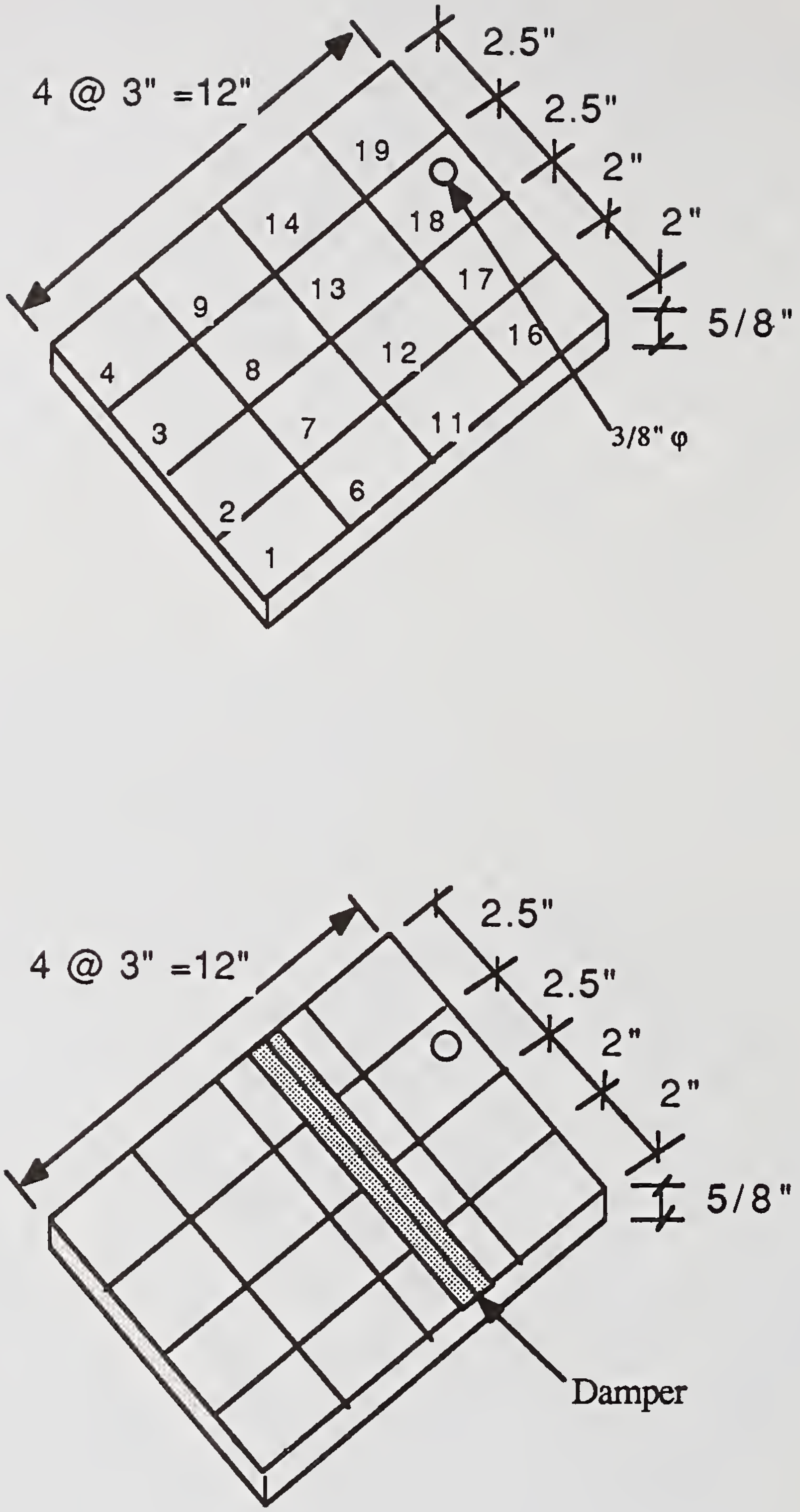

Figure 3 : Plate B with and without Dampers

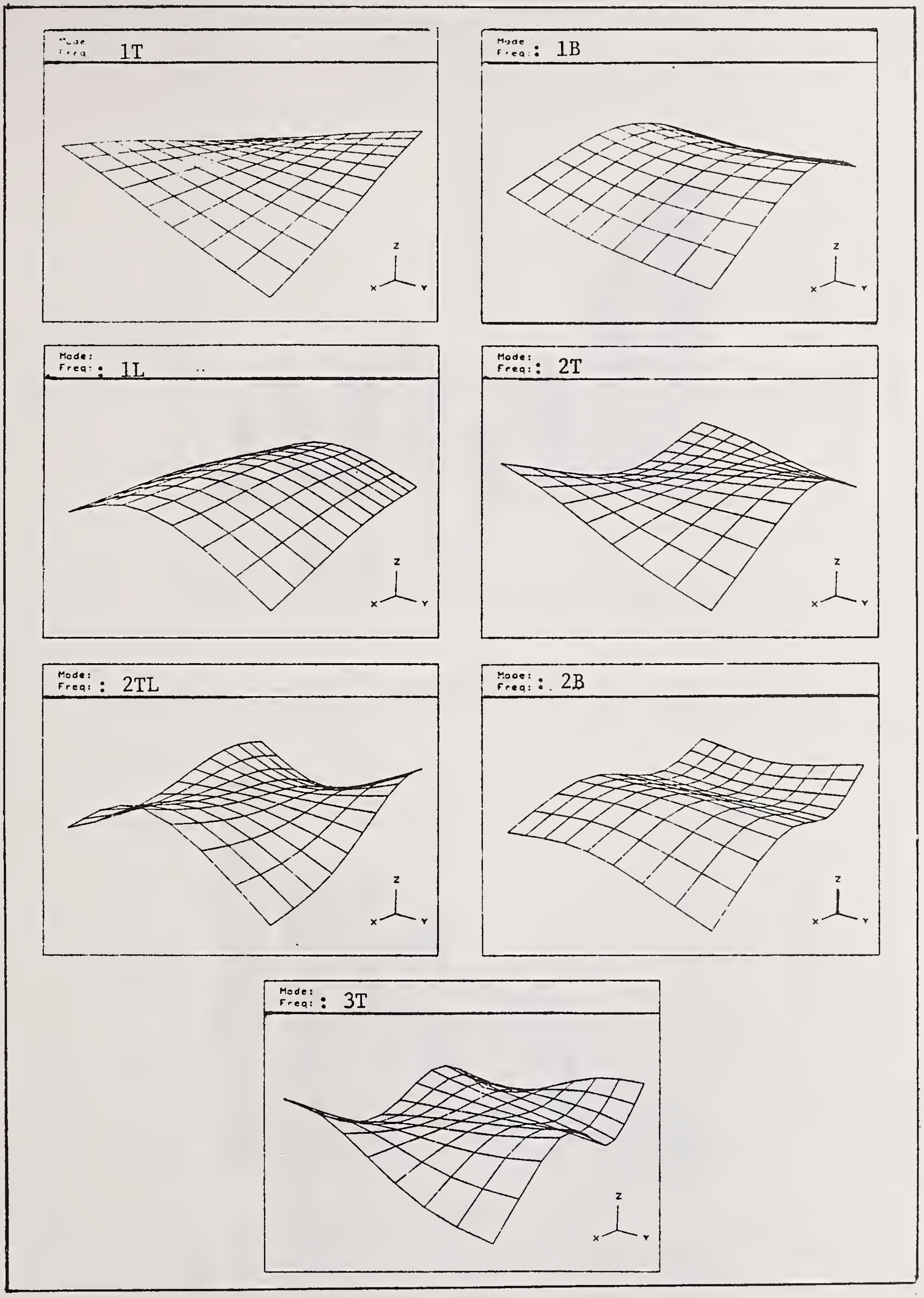

Figure 4: Mode Shapes of a Plate.

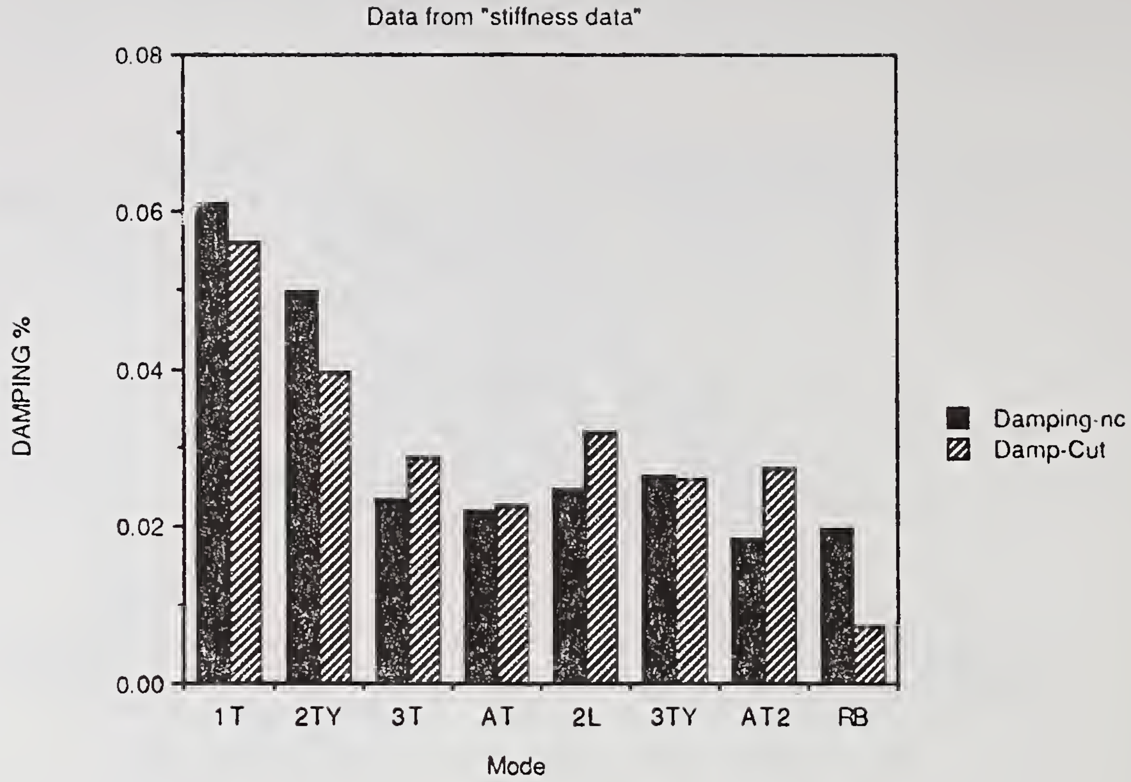

Figure 5a.: MODE VS. DAMPING FOR PLATE WITH AND WITHOUT CUT

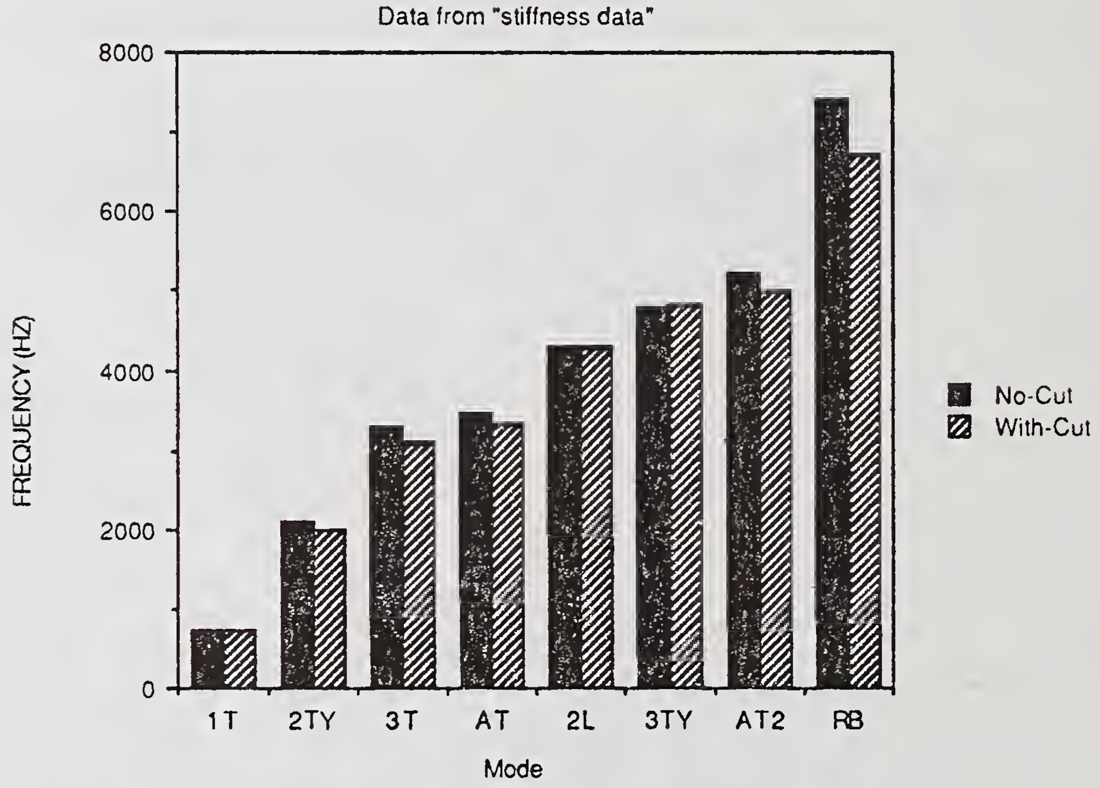

Figure 5b.: MODE VS. FREQUENCY FOR PLATE WITH AND WITHOUT CUT

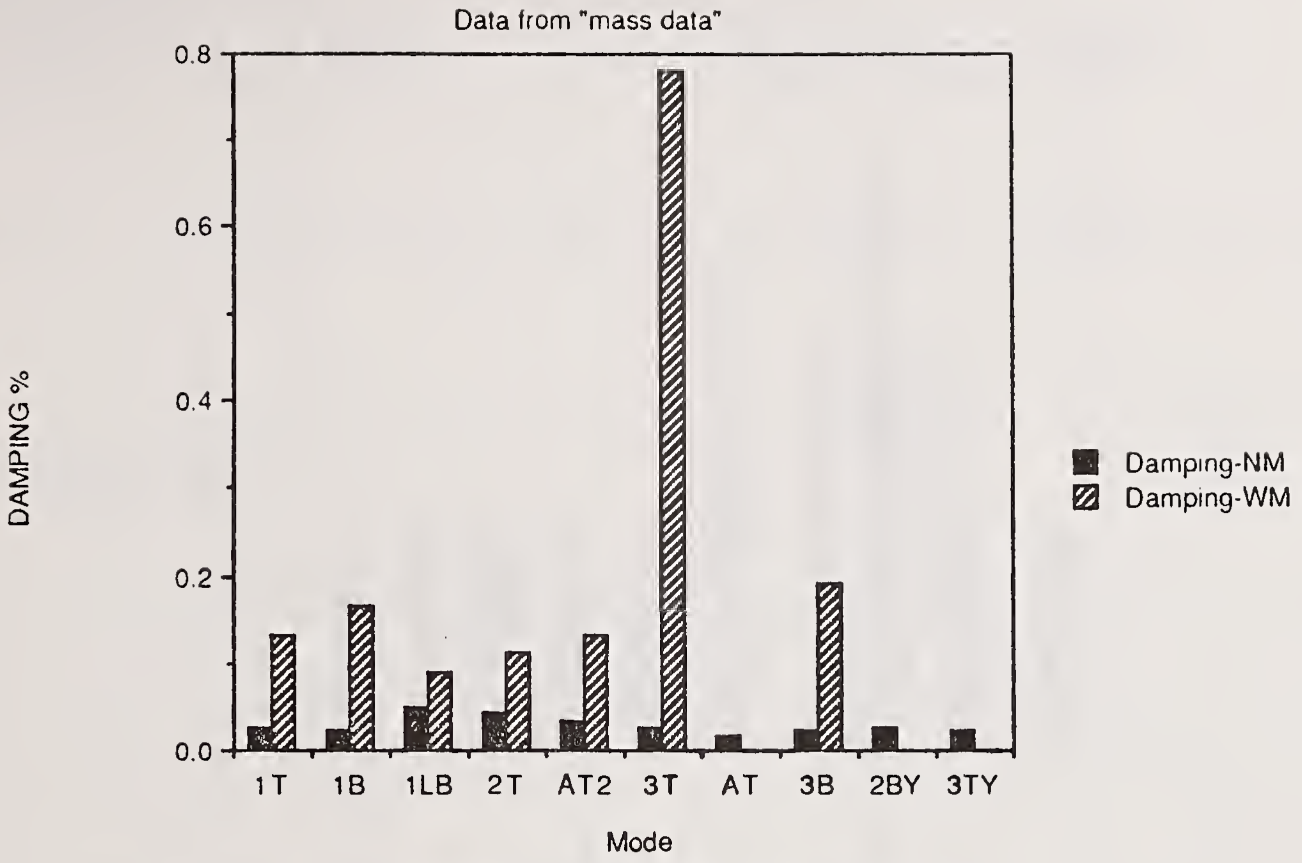

Figure 6a.: Mode vs. Damping for Plate With and Without Mass

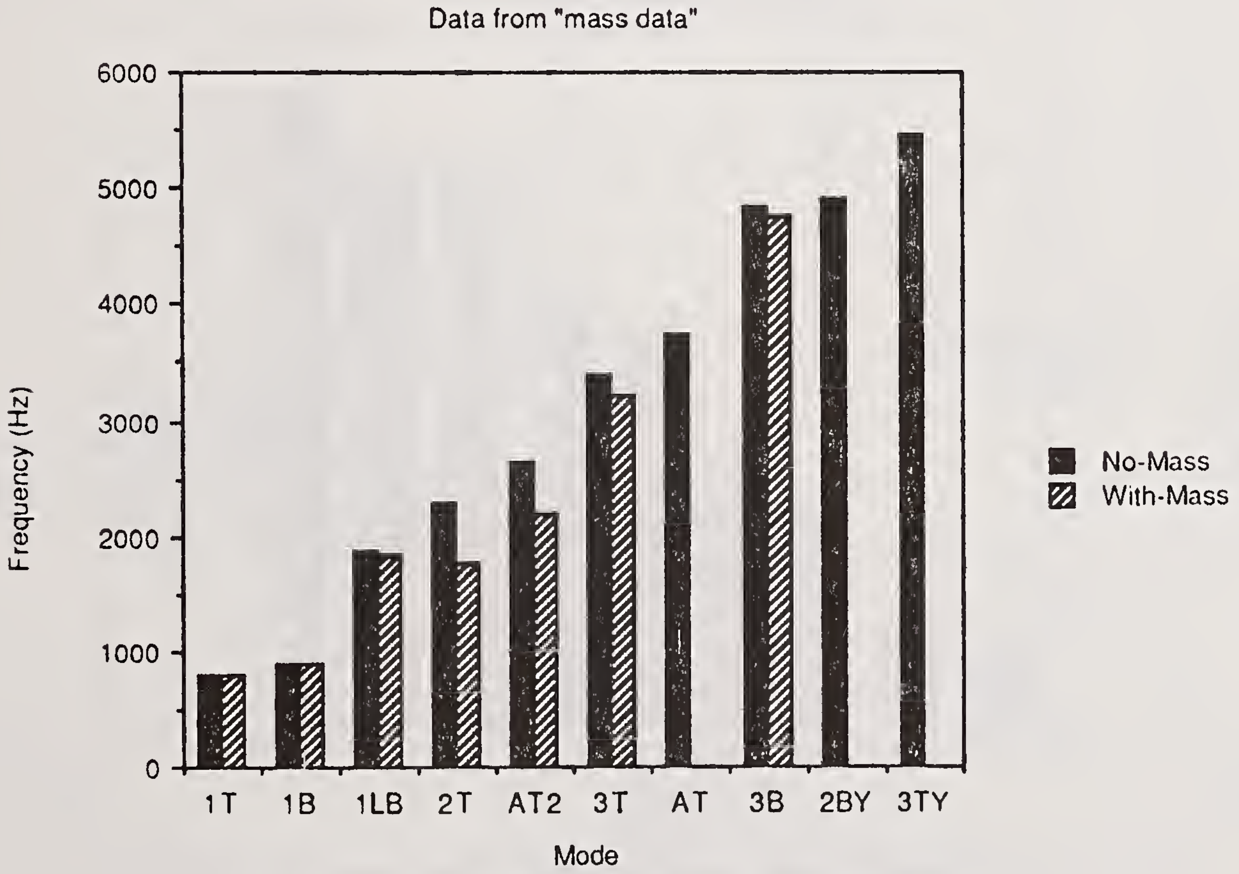

Figure 6b: Mode vs. Frequency of Plate With and Without Mass

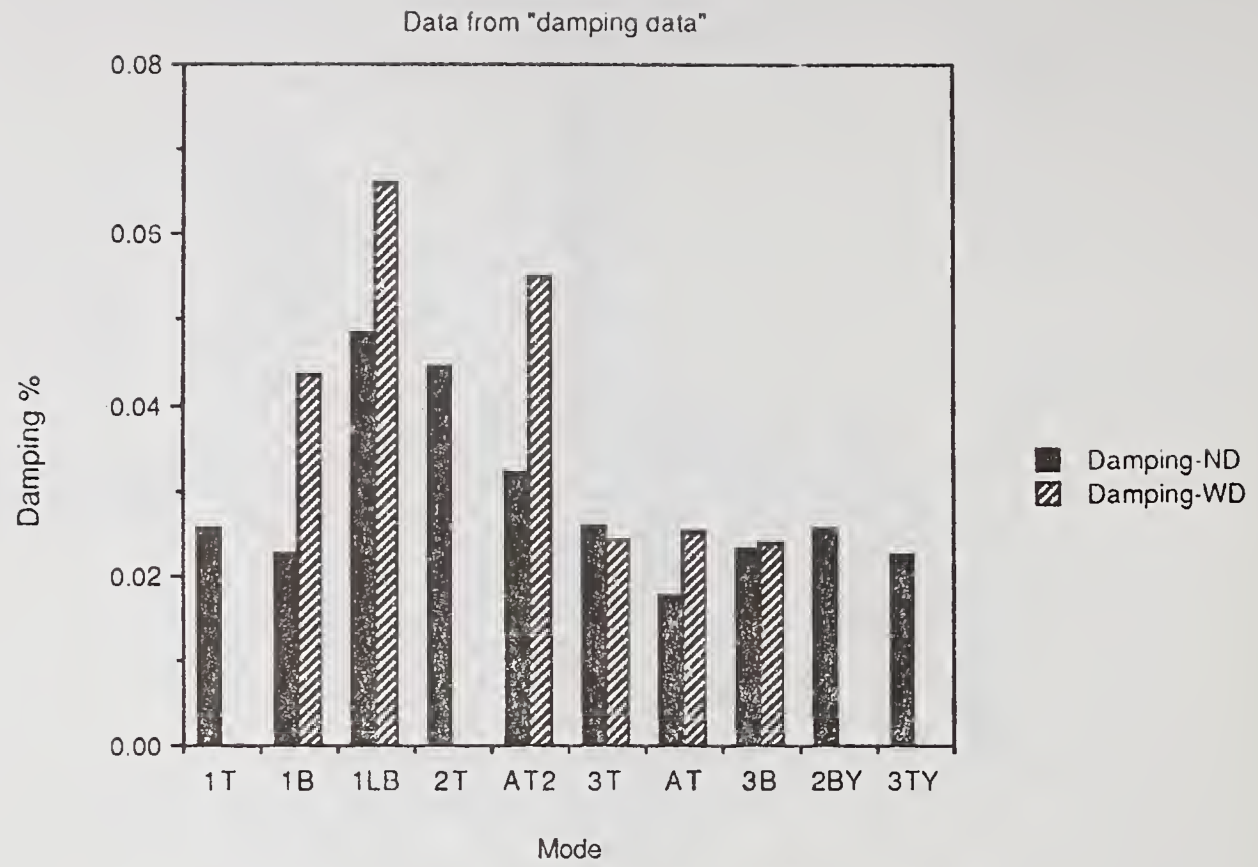

Figure 7a.: Mode vs. Damping for Plate With and Without Damper

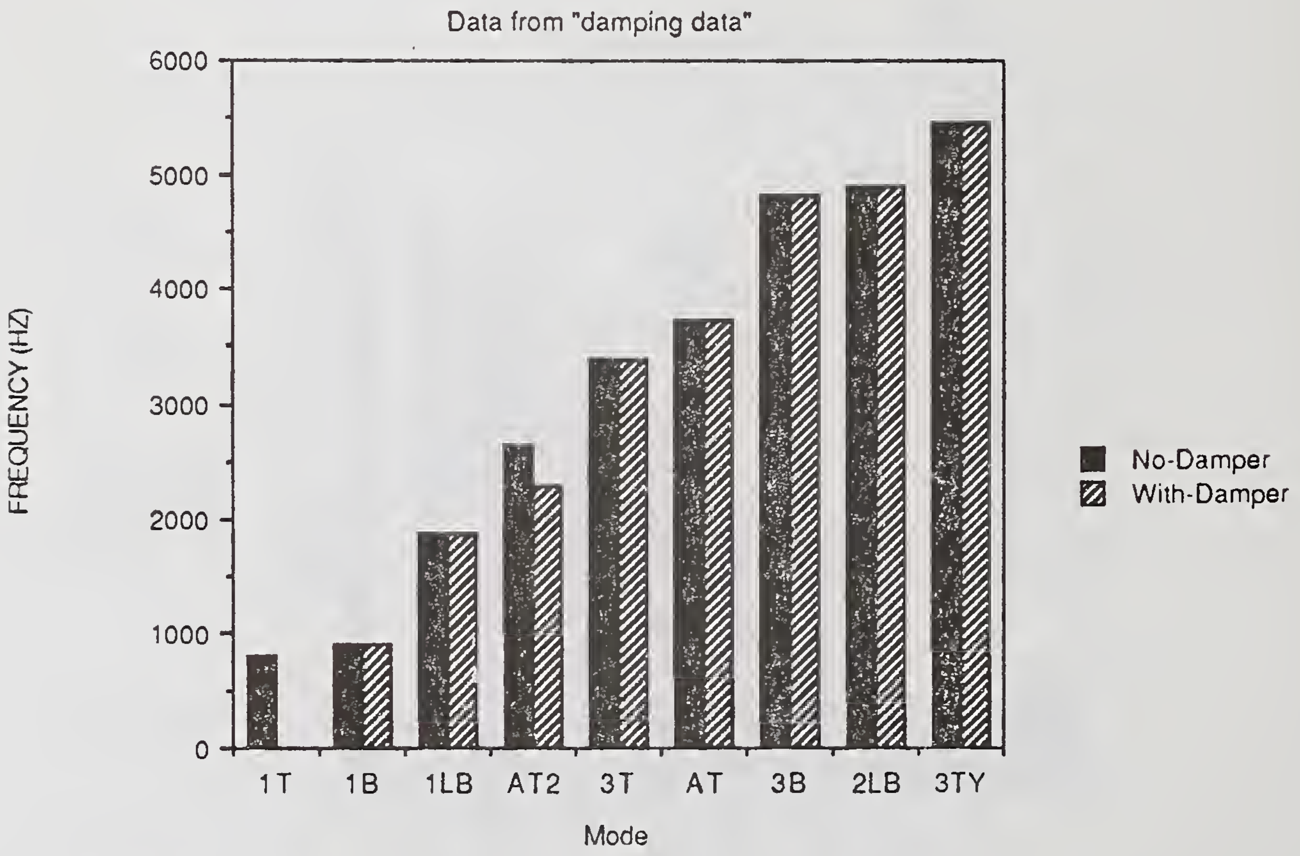

Figure 7b.: MODE VS. FREQUENCY FOR PLATE WITH AND WITHOUT DAMPER

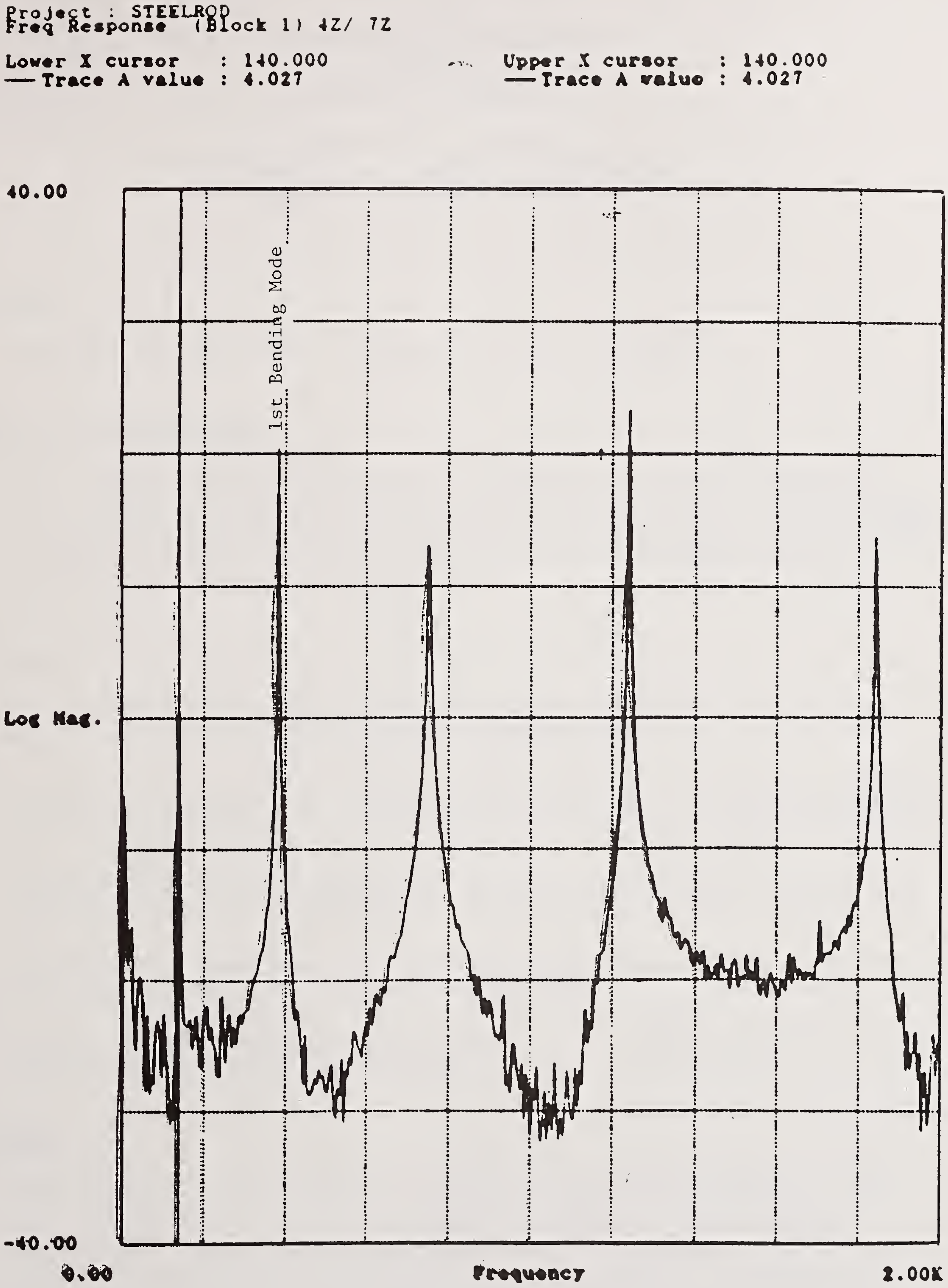

Figure 8: A Frequency Response Function for the Steelrod
(output response at node 4 and input force at node 7)

COOLING FIN MATERIALS
and
CONVECTIVE COOLING

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KEY WORDS

Thermal Conductivity, Convective Heat Transfer

PREREQUISITE KNOWLEDGE

The effect of the choice of material for use as a cooling fin, and the contrast between free and forced convection can be demonstrated without prerequisite knowledge. A knowledge of heat transfer theory is needed to derive the equations used for numerical analysis.

OBJECTIVES

Depending on the level of the class, any of the following may be stated as experimental objectives:

Demonstrate that the thermal conductivity of a metal is an important property in determining the metal's effectiveness as a cooling fin.

Demonstrate the enhanced cooling obtainable with forced, as opposed to free, convection.

Numerically determine the convective heat transfer coefficient for free and forced convection.

Determine the thermal conductivity of an unknown metal, using known metals as standards.

EQUIPMENT

Test Fixture: Six rods of common engineering metals, 3/8 inch in diameter and 5.0 inches in length, were mounted vertically by pressing them into holes drilled into a 3/16 inch thick aluminum baseplate. The materials selected were 1020 cold rolled steel, 308 stainless steel, commercially pure copper, 70-30 cartridge brass, 1024 aluminum, and a titanium alloy of unknown composition. Chromel-Alumel thermocouples were epoxied to the free ends, and three thermocouples were mounted to the baseplate.

Thermocouple Measuring Device: We are currently using a computer controlled automated data taker. This is desirable as there are several pieces of data to collect (nine temperature readings plus the time of observation), and also the experiment requires up to an hour to insure that thermal equilibrium has been obtained.

Laboratory Hotplate

Small fan: A muffin fan mounted to stand for positioning works well.

PROCEDURE

Before turning on the fan or the hotplate, record all temperatures.

Place the test fixture on the hotplate and adjust the hotplate setting to approximately half power.

Record temperatures every five minutes until thermal equilibrium is obtained. For free convection, this will be typically thirty to sixty minutes.

Turn on the fan and again record temperatures every five minutes until the new thermal equilibrium is obtained. For forced convection, this will be typically twenty to forty minutes.

Note: With an automated data taker, students can start the experiment, go off to attend other things, and return later to inspect the data to judge if thermal equilibrium has been achieved.

ANALYSIS OF DATA

The initial temperature readings are averaged together and taken as the ambient room temperature. This temperature is then subtracted from the equilibrium free end temperatures for both the free and forced convection experiments. Typical numerical results are shown in Table 1. The effect of the choice of material, as well as the enhanced cooling obtained with forced convection can be demonstrated by constructing Figure (1).

The equilibrium end temperatures correlate with the thermal conductivities of the metals. The values used here for subsequent analysis, in SI units (watts/meter-kelvin) are:

Copper	400
Aluminum	240
70-30 Brass	140
1020 Steel	50
Stainless Steel	25

Another illustration of the effect of material choice on cooling fin performance is obtained by calculating the total power dissipated by each rod. This is determined from the relationship:

$$Q = \frac{\pi}{2}\sqrt{h\kappa D^3}\left[\frac{(h/\kappa\lambda)\cosh(\lambda L)+\sinh(\lambda L)}{(h/\kappa\lambda)\sinh(\lambda L)+\cosh(\lambda L)}\right]\Theta_0 \tag{1}$$

with

h -- *Convective Heat Transfer Coefficient*
κ -- *Thermal Conductivity*
D -- *Rod Diameter*
L -- *Rod Length*

and

$$\Theta = (T - T_A) \tag{2}$$

The subscript A refers to the ambient temperature, and the subscript zero refers the baseplate temperature difference. For a round cooling rod:

$$\lambda = 2\sqrt{\frac{h}{\kappa D}} \tag{3}$$

For a class in which the materials are emphasized, students are given typical values for the convective heat transfer coefficient, for example, h=20 watts/meter^2-kelvin for free convection and h=100 watts/meter^2-kelvin for forced convection, and instructed to compute Equation (1) for their temperature data and the values of thermal conductivity given above. Since the calculation involves several steps, computer spreadsheets or programmable calculators are recommended. The results of the data of Table 1 with the values of h determined below are shown graphically in Figure (2).

For a class in which heat transfer is emphasized, the calculated free end temperature difference is derived to be:

$$\left(\frac{\Theta_L}{\Theta_0}\right) = \left[\frac{1}{(h/\kappa L)\sinh(\lambda L) + \cosh(\lambda L)}\right] \tag{4}$$

The subscript L refers to the free end of the cooling rod. This is a standard solution found in heat transfer textbooks and assumes that heat flow through the rod in by one dimensional thermal conduction. The heated end is taken to be at constant temperature, and all other surfaces are considered to lose heat by convection. The experimental value of the convective heat transfer coefficient is determined by plotting Equation (4) with thermal conductivity as the independent variable and the convective heat transfer coefficient as an adjustable parameter. Figure (3) shows the experimental data and two computed curves for the values of h indicated. This curve fitting is most easily done using an electronic spreadsheet.

As a final piece of experimental information, the thermal conductivity of the unknown titanium alloy can now be determined. Using the value of h=15 watts/meter^2-kelvin for free convection, Equation (4) computes the thermal conductivity of the titanium alloy to be 18 watts/meter-kelvin to agree with the measured equilibrium temperature difference. In general, the thermal conductivities of poor conductors can be determined in this manner. Since the computed curves of Figure (3) flatten out with increasing thermal conductivity, highly conductive unknown materials can be measured less accurately.

The manufacturer of the data taking device states an accuracy of ±0.15% on voltage measurements. From the variation of temperature readings at the beginning of the experiment, when all thermocouples should read the same, an accuracy of ±0.5% is assigned to temperature readings. From the curve fitting sensitivity to test parameters, an accuracy of ±10% is assigned to the measured values of convection heat transfer coefficient and power dissipation.

REFERENCES

A detailed derivation of the heat transfer formulae can be found in J. P. Holman, *Heat Transfer* (McGraw-Hill, 1990), 7th edition.

NOTES ON EQUIPMENT

Common shop metals were used for the test fixture construction. The apparatus is not sensitive to dimensions, so long as the rods are long and thin.

Omega Engineering, Inc., P.O. Box 4047, Stamford, CT 06907-0047 (1-800-826-6342) is an excellent source of thermocouple supplies, as well as many other instrumentation transducers and measuring devices.

Our automated Data Taker was purchased from Zi-Tech Instrument Corporation, P.O. Box 391567, Mountain View, CA 94039 (1-415-966-8484). It accepts 23 channels of input, which can be configured as analog or digital signals. It has an internal battery with storage for approximately 11,000 data points so that it can be programmed from a PC, disconnected and transported to a laboratory setting, and subsequently uploaded to a PC for data analysis.

Table 1

FREE END TEMPERATURE
MINUS AMBIENT TEMPERATURE

	Free Convection (deg C)	Forced Convection (deg C)
Copper	37.5	13.3
Aluminum	34.3	6.3
70-30 Brass	29.2	6.3
1020 Steel	22.1	2.6
Stainless Steel	11.6	0.6
Titanium	7.9	0.4

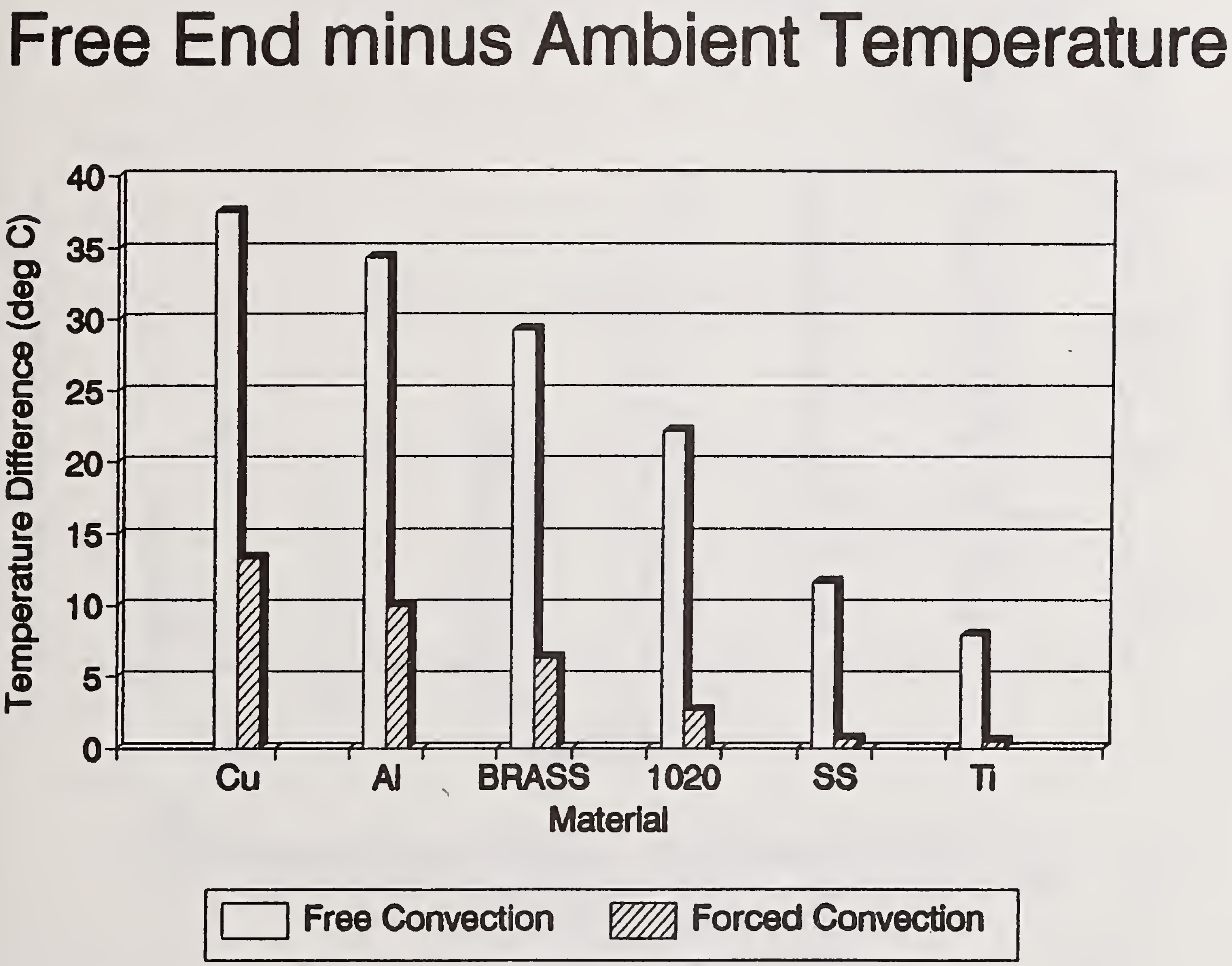

Figure (1): Experimentally Measured temperature differences.

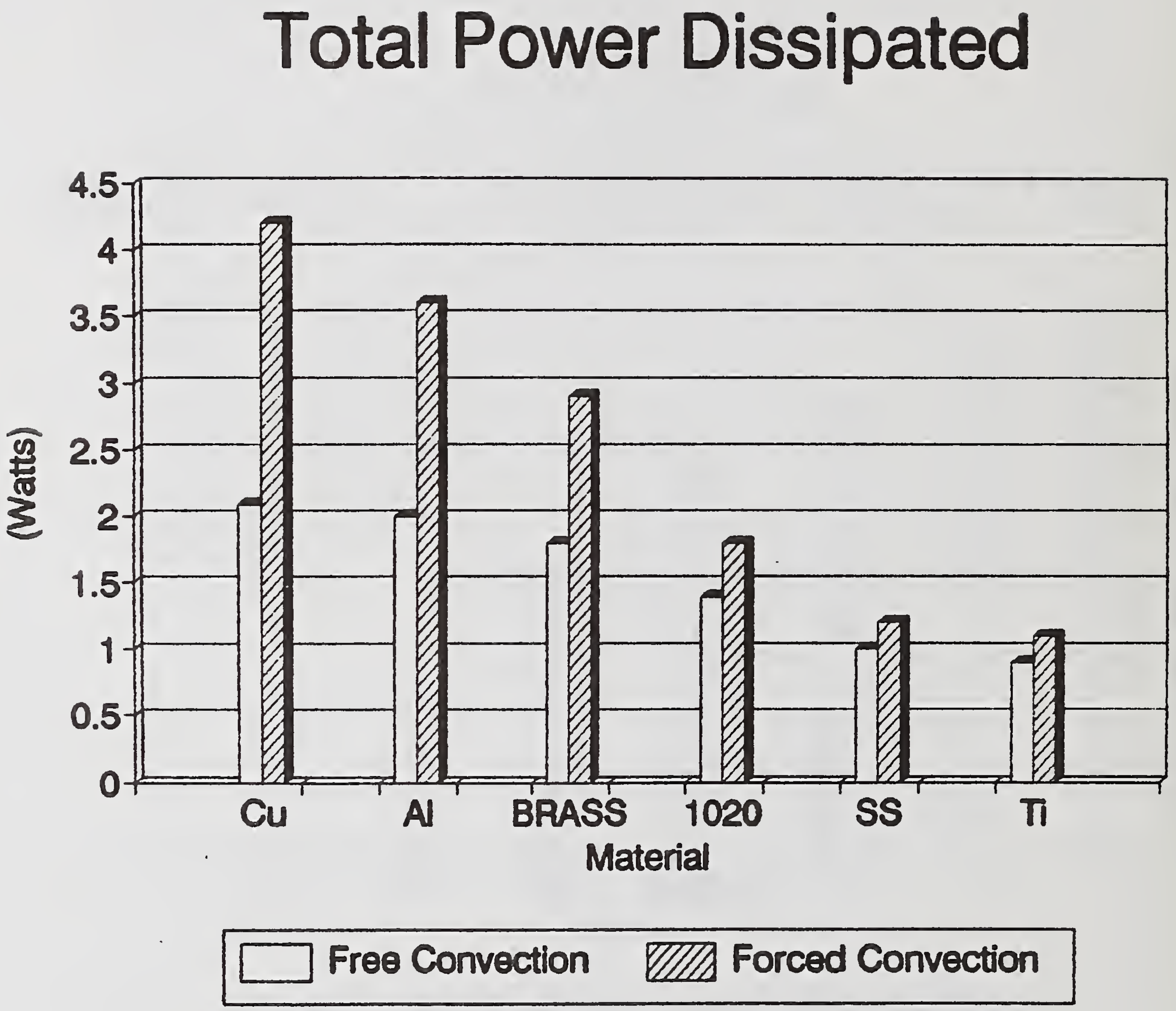

Figure (2): Computed power dissipation of each material with free and forced convection.

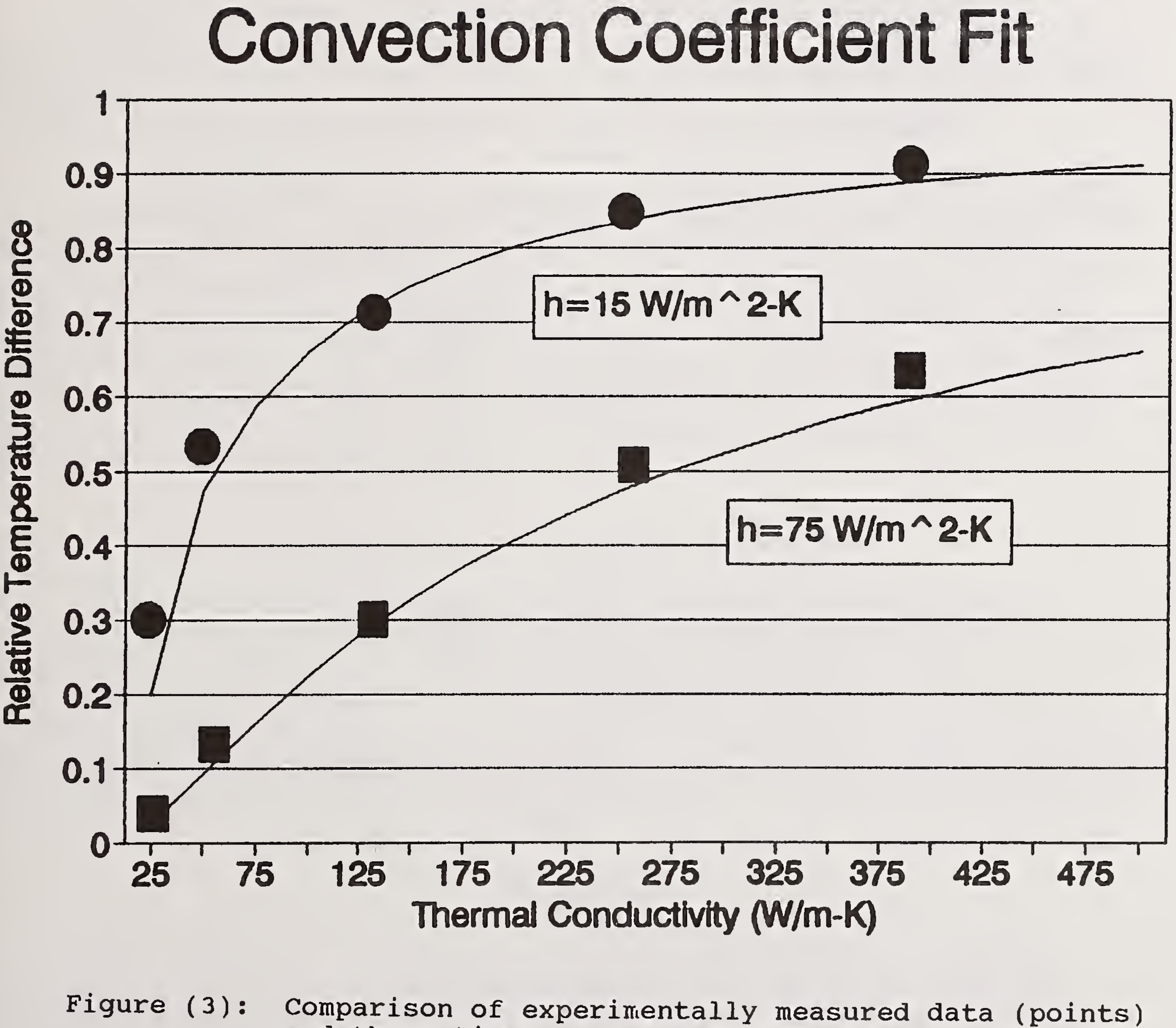

Figure (3): Comparison of experimentally measured data (points) and theoretically computed values (smooth lines).

FABRICATION AND EVALUATION OF A SIMPLE COMPOSITE STRUCTURAL BEAM

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OVERVIEW

This experiment involves the team design, fabrication, and evaluation of composite structural materials. These composite materials, in the form of small structural beams, are fabricated from organic fibers or particles selected by the student team and bonded with an adhesive system also chosen by the team.

This experiment was created in response to a national call for more hands-on, open-ended design experiences for engineering students. The experiment has been repeated over a period of three years at Miami University. The first time the experiment was run, the results suggested that viable, cost competitive substitutes for lumber and reinforced concrete might be developed using cellulose as a primary ingredient. Subsequent iterations of the experiment have been directed towards the use of recycled cellulose and polymers in the development of innovative, cost-effective building materials.

PREREQUISITE KNOWLEDGE REQUIRED

This experiment is part of the laboratory work done in introductory material science course for bachelor-level engineering students. Some knowledge of physics and chemistry is desirable but extensive knowledge regarding the mechanical properties of materials is not required. At Miami, the students are expected to pursue the project to some depth, utilize library resources, make industrial contacts, and to utilize a systematic engineering approach to problem solving.

OBJECTIVES

The overall objective is to create an artificial wood composite to compete with real lumber for home construction applications such as framing and flooring. Specifically, the assigned objectives are as follow:

1. To design a composite structural material using organic fibers or particles that are bonded with one type of adhesive. The particulate material has to be an

agricultural product or by-product. Chopped or pulped recycled paper would, by this requirement, be very acceptable. No particle can exceed 0.02 cc in volume before bonding. Materials selected need to be safe for student use, reasonable in cost, and available to complete the experiment on time.

2. To devise a beam fabrication scheme capable of yielding at least one test beam per student per team. The fabrication method selected has to be inherently safe, cannot require painstaking labor, and should likely employ fabrication resources close at hand.
3. To iteratively refine the material choices and beam fabrication method experimentally. In particular, the students are urged to investigate adhesive wetting and bonding reactions and to fabricate and break prototype beams.
4. To produce a final set of composite beams and evaluate these composites vis-a-vis real lumber. This evaluation includes consideration of a number of factors including bending strength, stiffness, cost, and more qualitative factors such as potential environmental impact.

EQUIPMENT AND SUPPLIES

Some of the adhesives employed, to be considered, include the following:

- two-part epoxies and polyesters.
- polyvinyl acetates.
- starch and related adhesives.
- lignin-based adhesives.
- urea-formaldehyde.
- phenol-formaldehyde.
- portland and other inorganic cements.
- protein-based glues such as casein.
- flour pastes.

Some of the particulates, that can be used for composite fabrication, include the following:

- softwood and hardwood paper pulp.
- jute, cotton, and other commercial organic fibers.
- recycled paper, cardboard, and polymers.
- sawdust and shredded bark.
- wheat straw and other plant stalk materials.
- husk materials such as peanut and walnut shells.

Examples of successful composite beams include the following:

- melamine laminated paper.
- paper pulp bonded with wood glue.
- strips of grocery bags bonded with resorcinol-type resin.

PROCEDURE: BEAM FABRICATION

The nominal dimensions for a bend test beam are 15 X 30 X 250 mm. The beams can be cut from larger sheets or bars or they can be fabricated one beam at a time. Fabrication methods that pose safety and environmental hazards, require tedious labor, or require the use of highly-specialized pressing and curing apparatus should be avoided.

Consider simple fabrication schemes such as those illustrated in Figure 1. As shown in Figure 1A, one popular approach is to bend up a sheet metal tray, fill the tray with the freshly mixed composite, and press the mix under dead weight until the adhesive cures. Figure 1B illustrates an approach often favored by paper science students. As in paper-making, the composite is prepared as a water-based slurry or mix which is subsequently de-watered by pressing on a screen and air drying. After drying, the thick sheet of composite is oven cured to set the adhesive. Both of the fabrication methods described above usually result in relatively weak composites with high void fractions unless an effort is made to minimize bubbles in the adhesive.

A three-step approach to fabrication can produce superior products. In the first step, prepare thin, prepreg sheets of uncured composite. In the second step, as shown in Figure 1C, laminate 50 to 100 of these sheets in a stack or coil. In the third step, set the adhesive by heat curing.

Attempts to manufacture test beams with structurally efficient cross-sections (e.g., I-beam, corrugated box, and honeycomb) usually fail. The added complexity of such section configurations typically results in serious problems with beam quality and missed deadlines.

PROCEDURE: BEAM TESTING

The simple test apparatus illustrated in Figure 2 is used to provide tensile strength and stiffness data. This apparatus can be fabricated using building block components from a Model 9014 Materials Testing Kit produced by the Engineering Science Division of Scott Aviation Corporation and an Ono Sokki electronic linear gage sensor with digital readout.

Basically, the tester is a three-point bending rig where the beam is simply supported at each end and loaded in the center using an hand-pumped hydraulic cylinder acting through a yoke which fits around the beam. Displacement of the yoke, and hence the beam, is monitored using the linear gage sensor. Load applied to the center of the beam is obtained by monitoring a gage indicating the applied hydraulic pressure. Before using the rig to test fabricated composite beams, it is a good idea to use the rig to slowly bend and break wood practice beams. This helps develop a feel for the proper use of the rig and to establish a consistent data-taking routine.

Wearing of safety glasses is mandatory. Fracture of both wood and composite materials is usually gradual with the fracture propagating slowly from the region of highest stress. Nonetheless, it is wise to anticipate the possibility of sudden fracture and the risk of flying debris.

As may be seen in Figure 3, plotting the beam deflection versus applied load reveals the elastic-plastic nature of the beam prior to failure. The slope of this curve in the elastic region, the stiffness, is subsequently used to determine the modulus of elasticity. As may also be seen in Figure 3, pressure gage readings are converted to units of applied force using a calibration procedure. The calibration procedure requires the use of a force gage to obtain a relationship between pressure gage readings and units of applied force.

The actual cross-sectional dimensions of the test beams can be determined using a micrometer or vernier caliper. The section measurements are needed plus the distance between the beam supports for the subsequent calculation of bending strength and elastic modulus. Both calculations are based on elastic beam theory. Use of elastic beam theory for estimating bending strength is reasonable when the beams fail by fracture without significant plastic deformation.

The estimation of bending strength (the maximum tensile stress in the outer fibers at fracture) is illustrated in Figure 4. The calculation of the modulus of elasticity is illustrated in Figure 5. Basically, the calculation of the bending strength and the modulus of elasticity, is done to normalize test results to facilitate the fair comparison of beams of different dimensions. Often the beams produced will vary considerably in cross-section from beam-to-beam and from team-to-team.

Measurement of beam weight permits the calculation of the strength and elastic modulus on a specific basis (per unit density). Determination of these properties on a specific basis emphasizes the importance of using low density materials in most structural applications.

Another test requirement is the examination of the resulting fracture surfaces. Almost always, the fracture will be seen to initiate in a poorly bonded region in the most highly stressed portion of the beam. of the beam.

PROCEDURE: EVALUATION OF RESULTS

Since this is a multi-objective experiment, the weighted ranking scheme illustrated in Figure 6 can be employed in the evaluation of results. In this scheme, there are several performance categories. Based on test results, calculations, and/or engineering judgement, the performance of each composite beam in each category is ranked from 0 to 10 relative to an arbitrary performance rating of 5 for a standard wood beam. For example, if a composite beam outperformed the wood standard in a specified category, it would receive a rating (category rank) from 6 up to 10 depending upon its relative superiority to wood. Vice versa, for example, if the cost of materials to make the composite beam exceeded the cost of an equivalent wood beam, the ranking for the composite would be less than the arbitrary rank of 5 for wood.

Assuming that the beams are ranked relative to wood in each category, it is also necessary to weight each category with a numerical value representing the relative importance of that particular category in the overall expectations of composite beam performance. The rank within each category is then multiplied by the assigned category weight and the resulting weighted products for each beam are summed for an overall performance rating.

A final project report is required. This report should include the final test results but must also include a clearly written description of the systematic, iterative approach employed in designing, fabricating, and evaluating the specific composite material that was developed.

INSTRUCTOR NOTES

The weighted ranking scheme is relatively easy to understand and it is a systematic approach for combining both quantitative and qualitative performance data. Often young engineers are prone to dismiss qualitative factors such as environmental concerns as unimportant when they cannot find appropriate definitions and equations for such factors in their engineering references.

Typically students respond enthusiastically to the open-ended challenge to select their own component materials and to develop a safe and cost-effective method of beam fabrication.

Carefully steering student choices, however, will improve results and reduce potential hazards. For example, when students were encouraged to consider the use of water-base adhesives, the consistency of the results improved with a corresponding decrease in beam fabrication difficulties.

The Manufacturing Engineering Department at Miami University has a well-equipped model shop for student use. In addition, there are materials science and paper science laboratory facilities suited to the safe production of simple composites. Facilities include mixing areas under exhaust hoods, basic mixing machinery, hand-operated presses, ovens, and a full complement of safety gear. Student efforts are carefully monitored. One-on-one instruction is provided for students involved with unfamiliar tools and procedures.

The first time the experiment was assigned, there was probably too much emphasis placed on designing a minimal cost composite and on the development of a novel fabrication method. Lately, emphasis has been placed having the students choose their adhesive intelligently to promote wetting and facilitate component mixing. In the future, the use of fire retardant adhesives will be promoted and emphasis will be placed on discovering simple methods to reduce void formation to improve composite integrity. To maintain the open-ended nature of the experiment, bonus points are usually awarded for to students who elect to pursue lines of investigation that are deemed, a priori, to be desirable.

REFERENCES

This experiment was inspired by several stories regarding interesting structural engineering and architecture projects where the students were expected to design, build, and test load-bearing structures based on ordinary materials such string, cardboard and toothpicks. This experiment is quite similar except that the students are now encouraged to work with recycled waste materials, renewable-resource agricultural materials, and today's rapidly advancing adhesives technology.

Two references that the students should find valuable are:

- Vance Setterholm, "Recycling Wood Fiber in Municipal Solid Waste: Opportunity for Government-Industry Partnership", TAPPI Proceedings, 1990 Pulping Conference, pp2-5. (Note: Mr. Setterholm is Assistant Director, USDA Forest Service, Forest Products Laboratory, Madison Wisconsin.)

- J.E. Gordon, The New Science of Strong Materials or Why You Don't Fall Through the Floor, Second Edition, 1984, Princeton University Press, Princeton, New Jersey.

SOURCES OF SUPPLY

Initially, most of the composite component materials were obtained from local suppliers such as hardware stores and lumber yards. When emphasis was directed towards finding more effective adhesives, the students focus shifted towards adhesives and coatings in common use in the forest products industry. Many suppliers to the forest products (paper and wood furniture) industries are listed in the Thomas Register and maintain offices in Georgia, Ohio, and Wisconsin.

Material expenses for a team of four students are estimated to be less than $20.00 or $5.00 per student if all materials were purchased. The most costly component material is typically the adhesive which usually comprises 10 to 20% of a test beam by volume. In seeking information on adhesives by contacting industrial suppliers, many students have received generous technical assistance including free samples of adhesives.

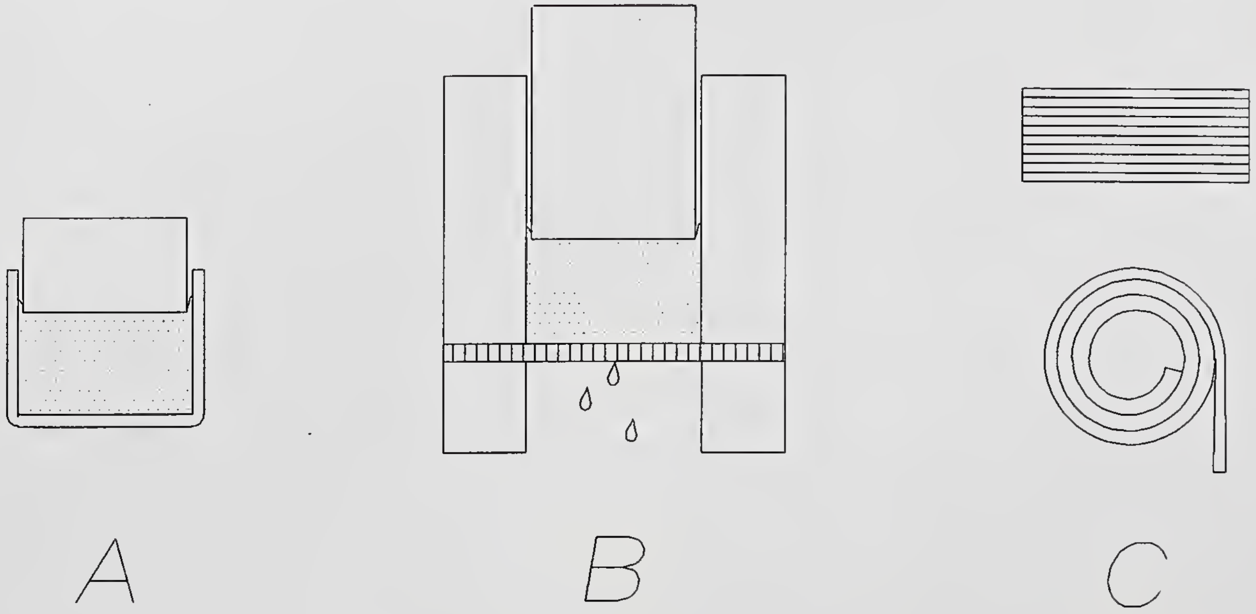


FIGURE 1.

CROSS-SECTIONAL VIEW OF TYPICAL FABRICATIONS

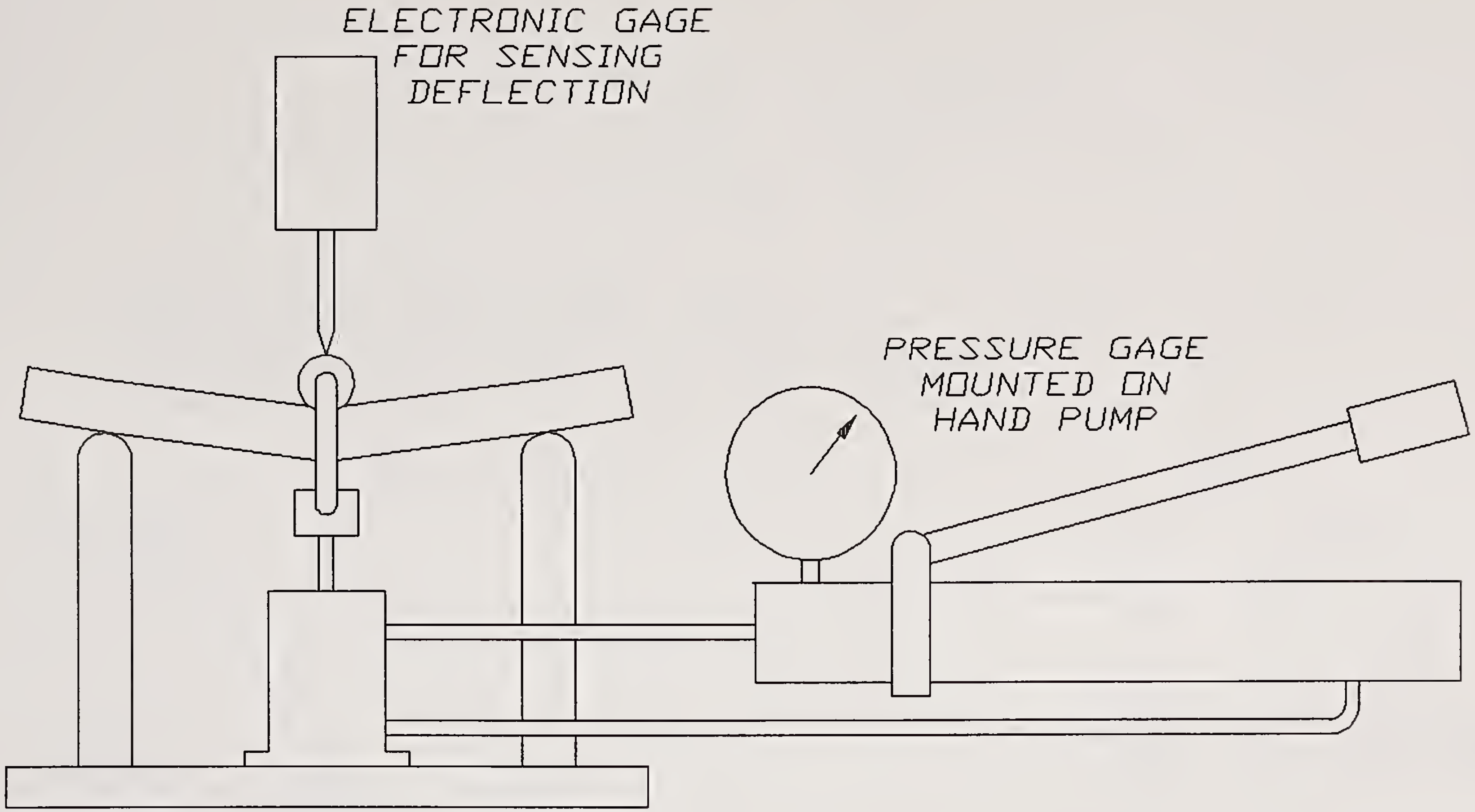
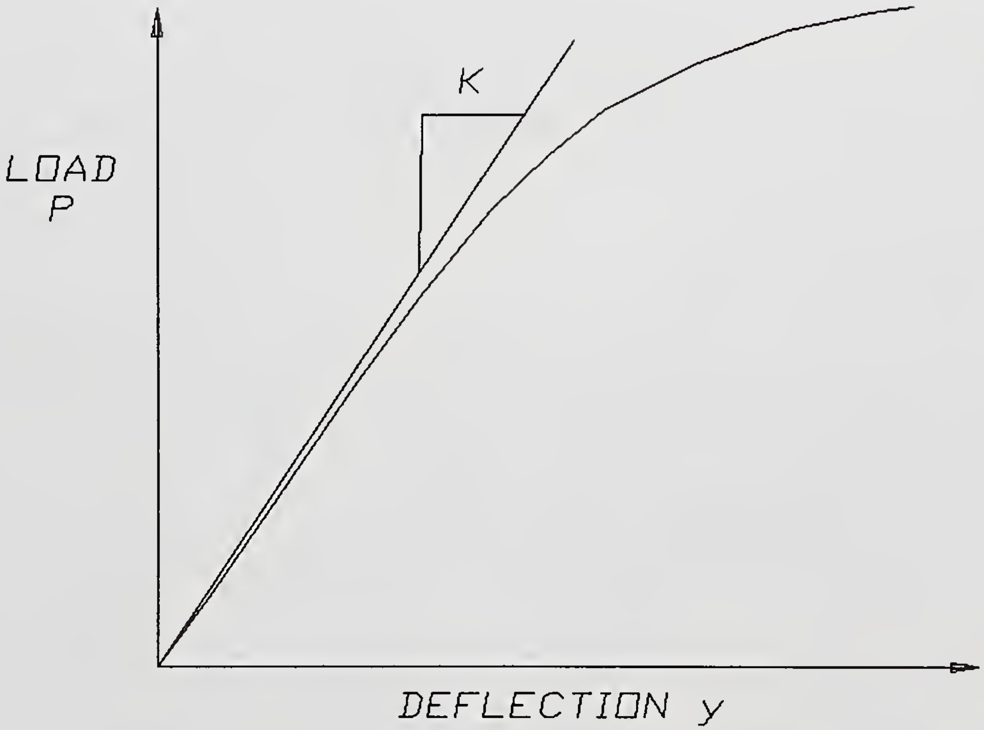
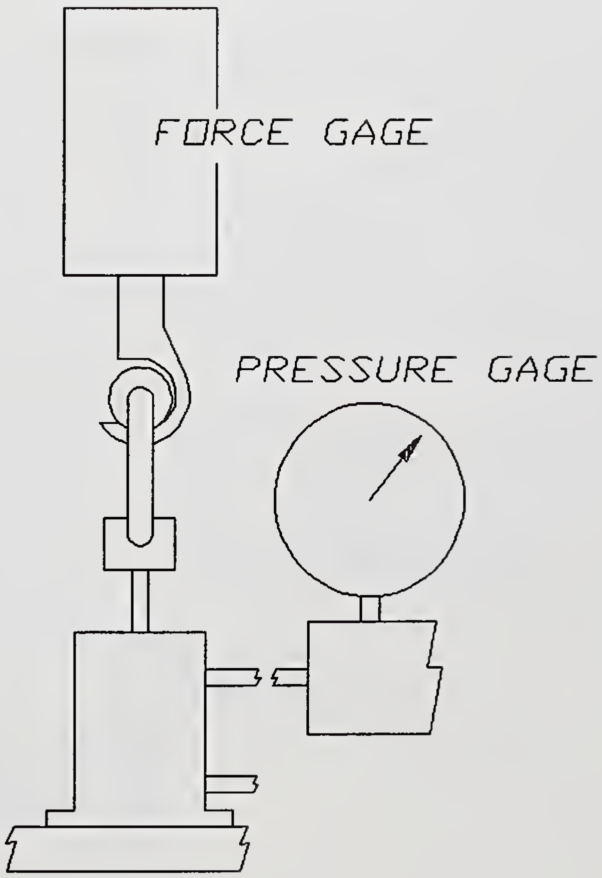


FIGURE 2.

BEAM TESTING APPARATUS
FOR STRENGTH AND STIFFNESS

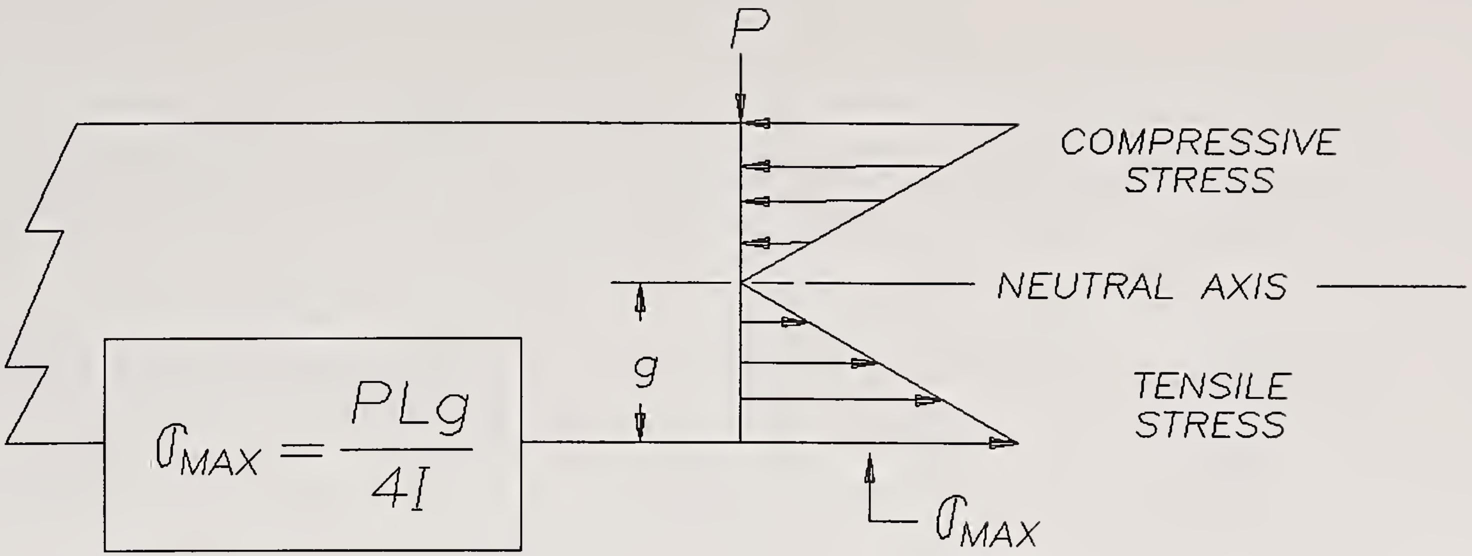




NOTE: THE LOAD, P, IS OBTAINED FRON A CALIBRATION CURVE OF LOAD VERSUS PRESSURE. THE CALIBRATION SETUP IS ILLUSTRATED TO THE RIGHT.

FIGURE 3.

LOAD VERSUS DEFLECTION
AT CENTER OF BEAM



σ_{MAX} IS THE MAXIMUN TENSILE STRESS AT OUTER FIBERS OF BEAM.

P IS LOAD APPLIED AT CENTER OF SIMPLY SUPPORTED BEAM.

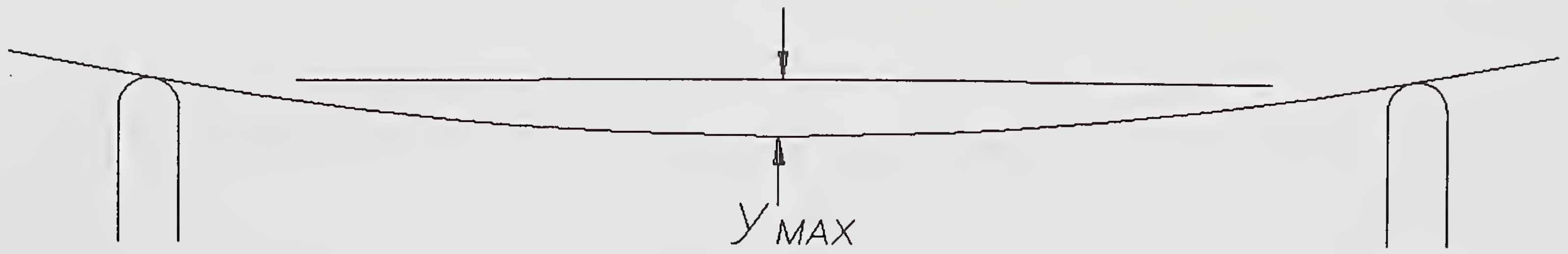
L IS DISTANCE BETWEEN BEAM SUPPORTS.

g IS DISTANCE FROM NEUTRAL AXIS TO OUTER FIBERS.

I IS MOMENT OF INERTIA.

FIGURE 4.

CALCULATION OF MAXIMUM TENSILE STRESS



THE MODULUS OF ELASTICITY, E, IS A MATERIAL PROPERTY. IT CAN BE RELATED TO THE BEAM DEFLECTION AS FOLLOWS:

FROM ELASTIC BEAM THEORY,

$$y_{MAX} = \frac{PL^3}{48EI}$$

WHERE P, L, AND I ARE AS DEFINED IN FIGURE 4.

SOLVING FOR THE MODULUS OF ELASTICITY

$$E = \left[\frac{P}{y_{MAX}}\right]\left[\frac{L^3}{48I}\right] = \left[\ K\ \right]\left[\frac{L^3}{48I}\right]$$

WHERE K, THE STIFFNESS, IS MEASURED AS SHOWN IN FIGURE 3.

FIGURE 5.
CALCULATION OF ELASTIC MODULUS

PERFORMANCE CATEGORY	PERF' WEIGHT	RANK WOOD	TOTAL WOOD	RANK TEAM A	TOTAL TEAM A
SPECIFIC TENSILE STRENGTH - mm	6	5	30	6	36
SPECIFIC ELASTIC MODULUS - mm	10	5	50	3	30
MATERIAL COST - $/kg	6	5	30	10	60
EASE OF FABRICATION	4	5	20	2	8
ENVIRONMENTAL OR SAFETY HAZARD	8	5	40	6	48
			170		182

FIGURE 6.
WEIGHTED RANKING SCHEME FOR EVALUATION OF RESULTS

MEASUREMENT OF STRAIN RATE SENSITIVITY IN METALS

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Seattle, WA 98195

Prerequisites:

1. Knowledge of the use of a universal testing machine for tensile testing.
2. The ability to reduce load-elongation data to produce engineering stress-strain curves.
3. Familiarity with the true stress-strain concept and conversion of engineering stress-strain data to true stress-strain data.

Equipment and Supplies:

1. Tensile testing machine with crosshead speed control over several orders of magnitude.
2. 500Kg full scale load cell.
3. Grip assembly for wire or rope samples.
4. Measuring equipment for gauge length and diameter measurement.
5. 1/8 inch diameter 60wt% Sn-40wt% Pb solid solder.

The Department of Materials Science and Engineering at The University of Washington is fortunate to have an Instron 4505 Universal Testing Machine to use for work of this type. This machine is a computer controlled testing machine with a computer system for data acquisition. Figure 1 shows the complete testing machine setup and figure 2 the grip assemply used in this work. These experiments could also be performed using an analog system and a micro computer or hand calculator for data reduction.

Objective:

This experiment demonstrates the high temperature stress-strain behavior of metallic materials. Eutectic lead-tin solder melts at 183°C, thus room temperature deformation represents deformation at $0.63T_m$, where T_m is the melting temperature in degrees kelvin. Under these conditions the shape of the stress-strain curve is very dependent on the applied strain rate. Thus the flow stress, defined as the stress to produce plastic flow at a given strain, is also sensitive to the strain rate imposed on the material under these conditions.

Experimental:

60wt%Sn-40wt%Pb solder was purchased locally in coiled form. Samples were carefully straightened and fit in the wire grips. The grips were positioned so that the gage length was 75 mm. The sample was allowed to relax before the test was started.

Results:

The data reported here was collected using a computer controlled universal testing machine equipped with an IBM-PC to record the data. This data was then imported to a spread sheet which was programmed to calculate engineering stress and strain and true stress and strain. These data were then used to plot the engineering and true stress strain curves in figures 3,4 and 5. The duration of the tests reported here varies from a few minutes to one week.

The flow stress-strain rate relationship can be expressed through the strain rate sensitivity equation:

$$\sigma = K \dot{\varepsilon}^m$$

where

σ=flow stress
K=constant
$\dot{\varepsilon}$= strain rate
m= Strain rate sensitivity coefficient, which is constant

The strain rate sensitivity coefficient can be determined by plotting log flow stress vs strain rate, fitting the data to a straight line. The strain rate sensitivity is equal to the slope of this line. The true stress-strain data should be used in making this plot. In this case the curves shown in figure 5 were used to determine the flow stresses. The results are shown in figure 6.

Eutectic lead tin solder exhibits superplastic behavior under some conditions of strain rate and temperature. This can be illustrated by plotting fracture strain vs strain rate. This data is shown in figure 7. At the lowest strain rates, superplastic behavior is being approached.

Acknowledgment:

The authors wish to thank Mr. D.F. Lii for help in setting up the Instron Machine, Drs Wendell Jones and John Stevens, Sandia National Laboratory, Albuquerque, NM and the attendees at the NEW-90 UPDATE Conference who supplied helpful comments.

References:

1. George E. Dieter, Mechanical Metallurgy, 3rd ed, McGraw-Hill, New York (1986) pp. 295-300.

2. Thomas H. Courtney, Mechanical Behavior of Materials, McGraw-Hill, New York (1990) pp295-309.

3. M. A. Meyers and K. K. Chawla, Mechanical Metallurgy, Principles and Applications, Prentice-Hall, Englewood Cliffs, N.J. (1984) pp559-574.

STUDENT HANDOUT

MEASUREMENT OF STRAIN RATE SENSITIVITY IN 60-40 SN-PB SOLDER

Objective: The objective of this experiment is to examine the effect of strain rate on the stress strain behavior of 60-40 Sn-Pb solder.

Procedure:

1. Five samples, each with the same gauge length, are to be pulled at five different crosshead speeds. There should be at least one order of magnitude difference between the crosshead speeds.

2. Set up the testing machine to pull each sample at the correct crosshead speed. Record the sample diameter, gauge length and test temperature before initiating the test. After fracturing the sample convert the raw data to engineering stress-engineering strain and true stress-true strain data.

3. Plot the engineering stress-engineering strain curves on the same axes. Label the strain rate for each curve. Repeat for true stress-true strain data.

4. Determine the flow stress for several selected strains for each strain rate used. Plot the log flow stress vs log strain rate. Fit the data to a straight line for each selected strain and determine the strain rate sensitivity coefficient.

5. Examine the effect of strain rate on the strain to fracture. Do any correlations exist? Explain.

Figure 1. A view of The Universal Testing Machine used in this study, showing the crosshead with grips holding a sample, the machine control tower, and a PC to communicate with the tower and to acquire data.

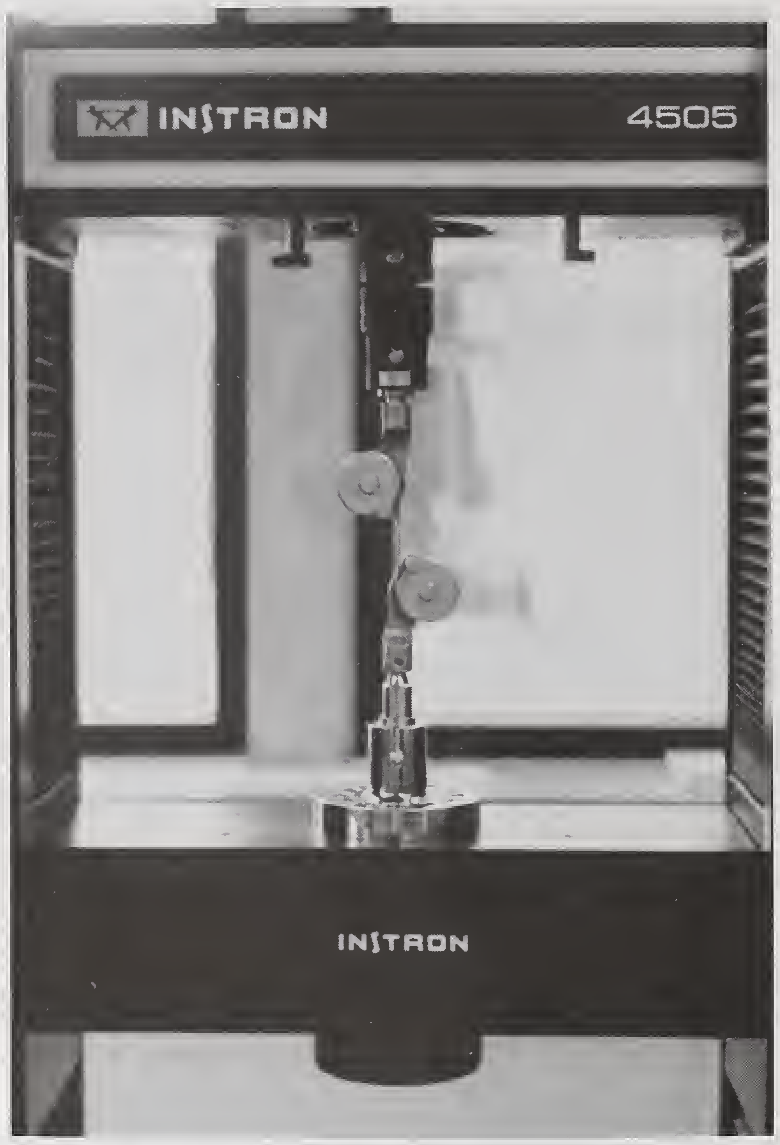

Figure 2. A close-up view of the sample and grip assembly.

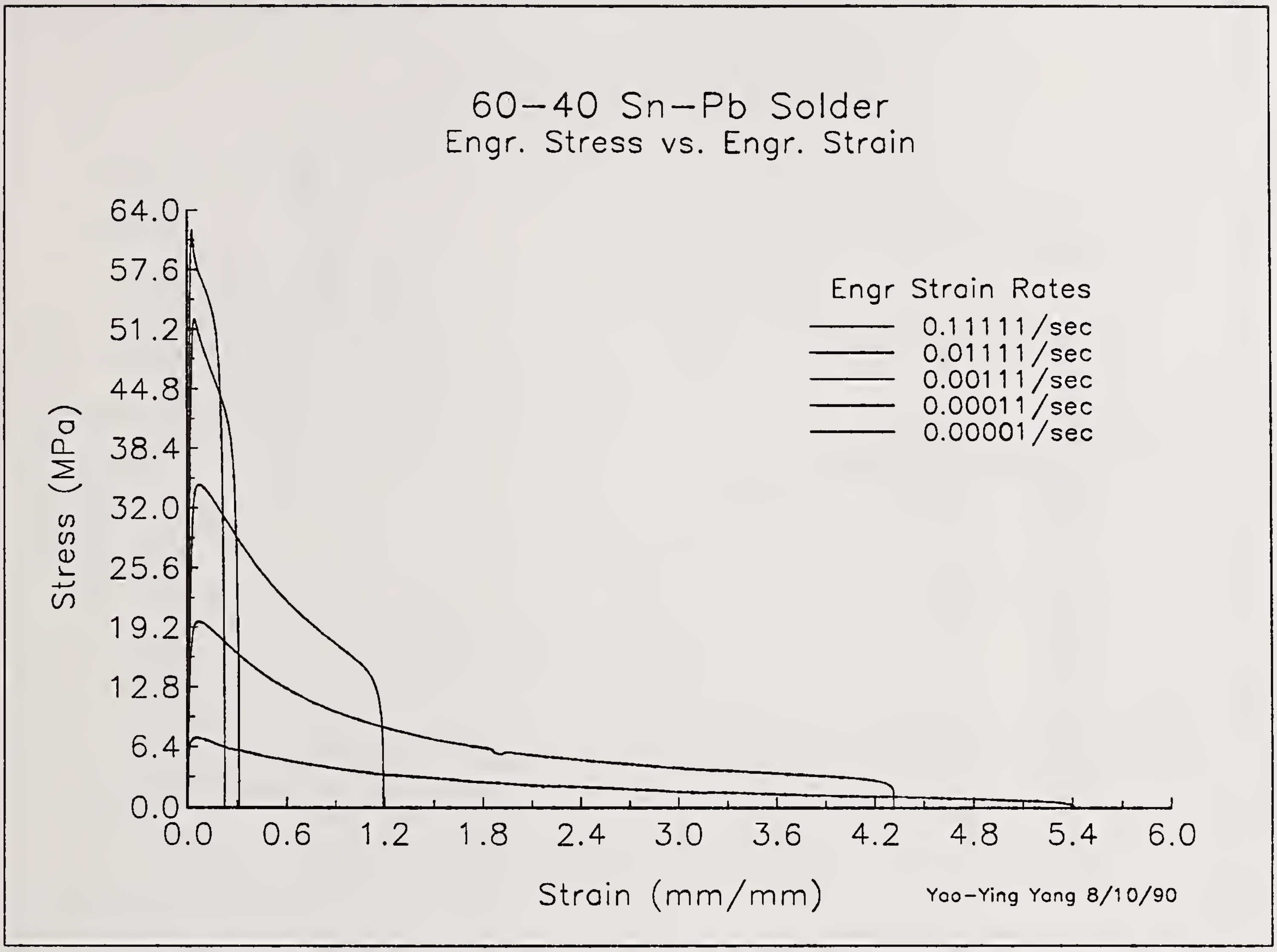

Figure 3. Engineering stress-engineering strain curves for 60-40 Sn-Pb solder deformed to failure.

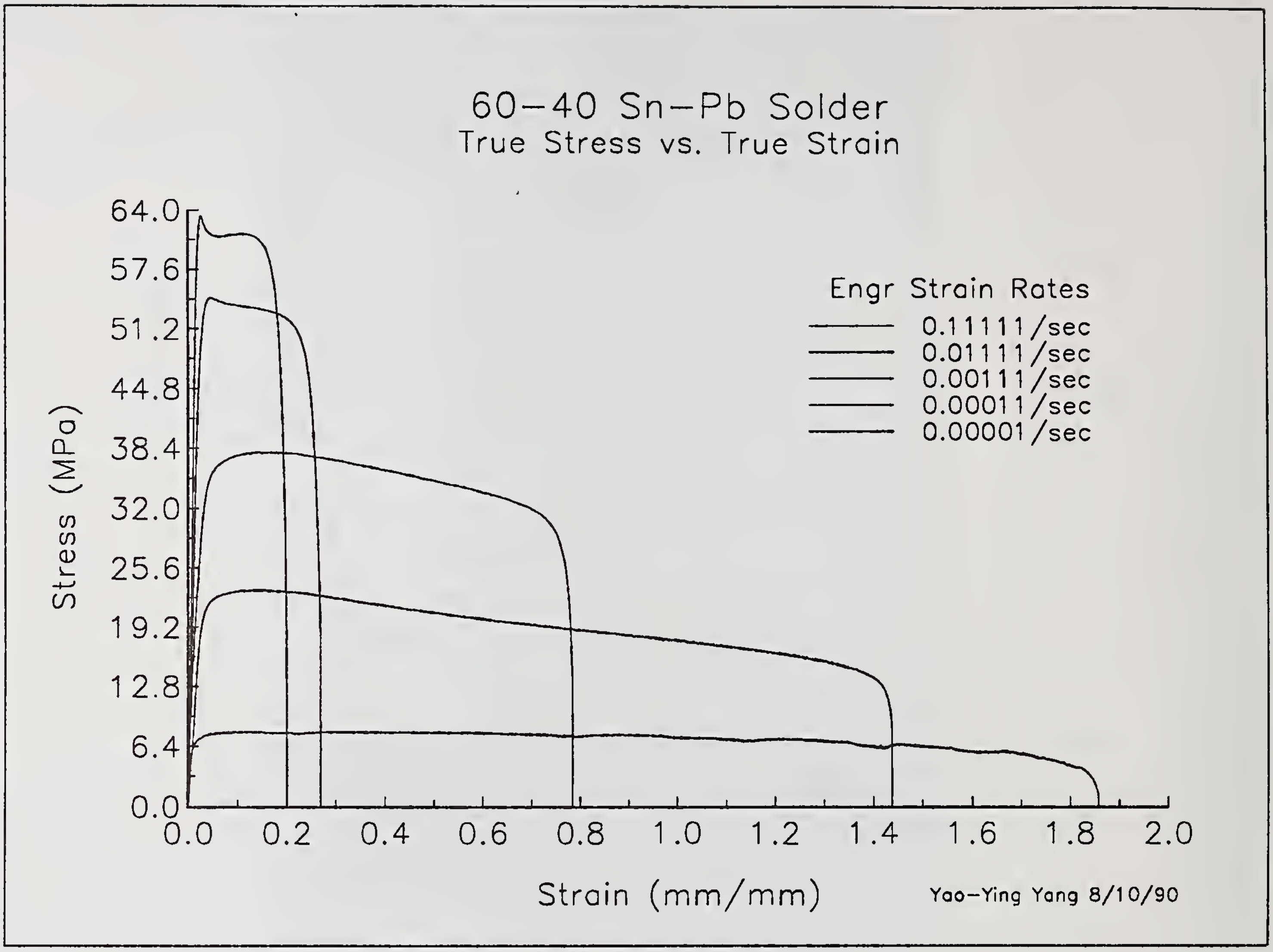

Figure 4. True stress-true strain curves for 60-40 Sn-Pb solder deformed to failure.

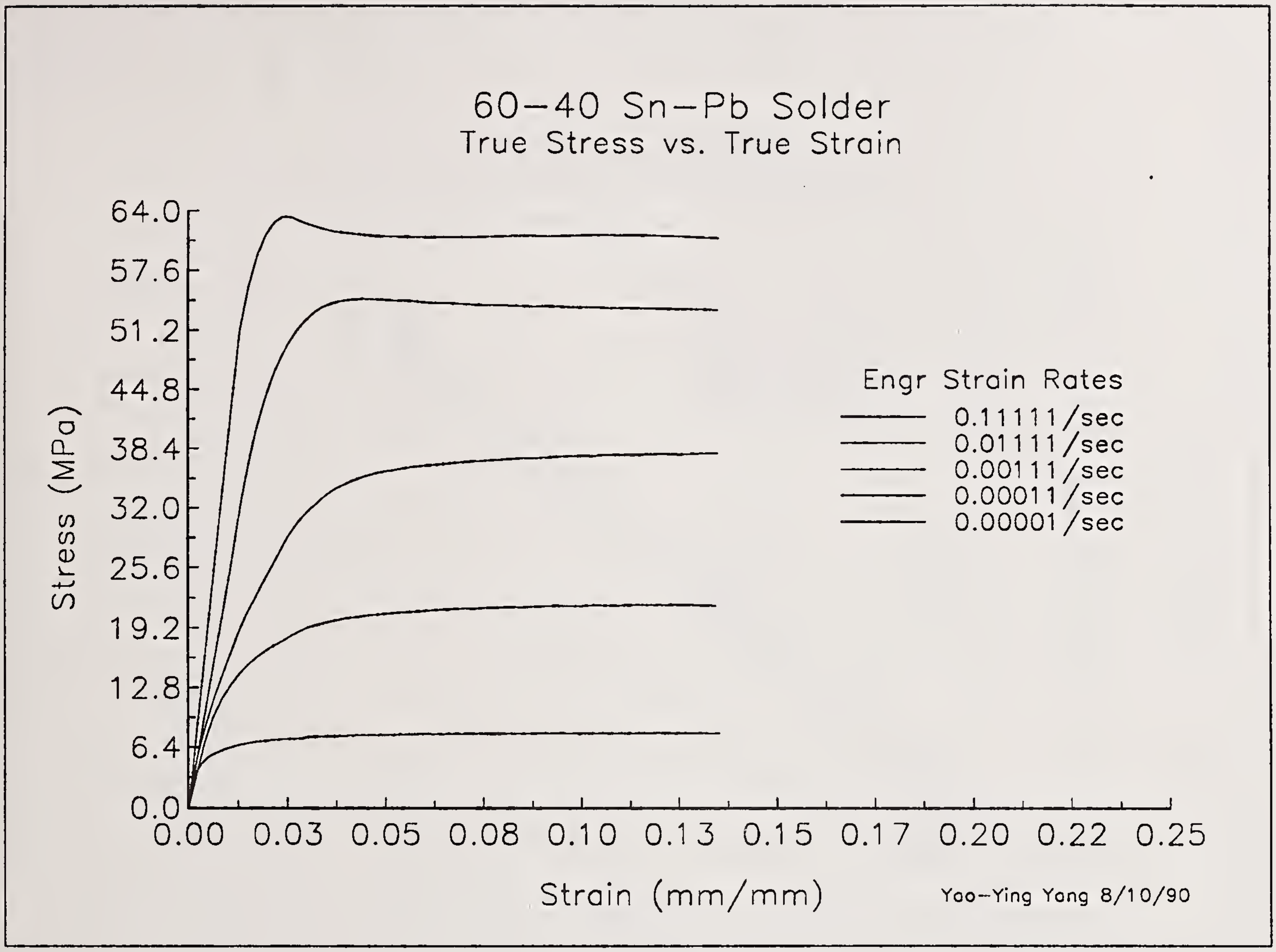

Figure 5. True stress-true strain curves for 60-40 Sn-Pb showing the low strain region. These curves were used to prepare figure 6.

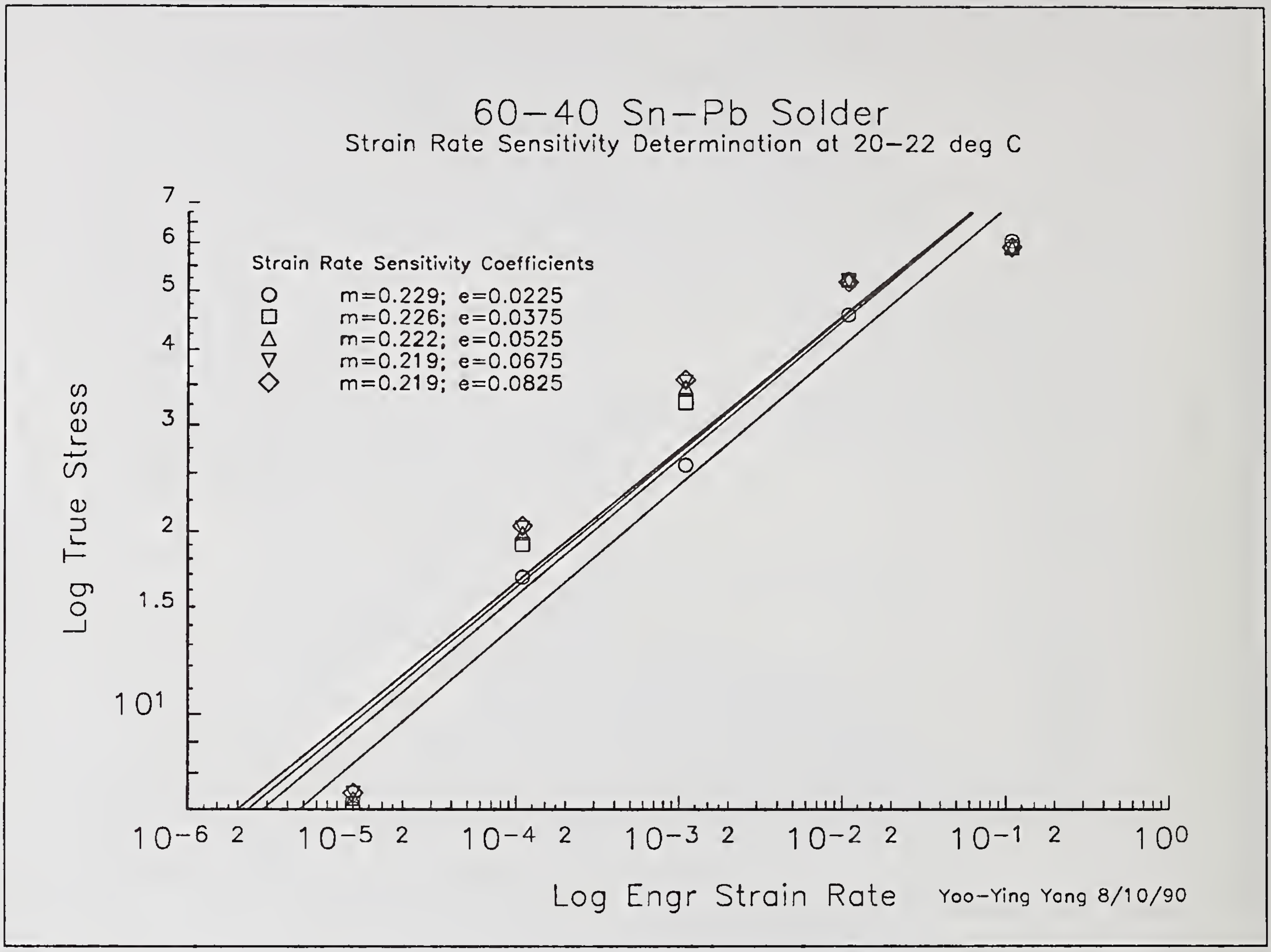

Figure 6. Log flow stress vs log strain rate for 60-40 Sn-Pb. The strain rate sensitivity coefficients are determined by measuring the slopes of the straight lines shown.

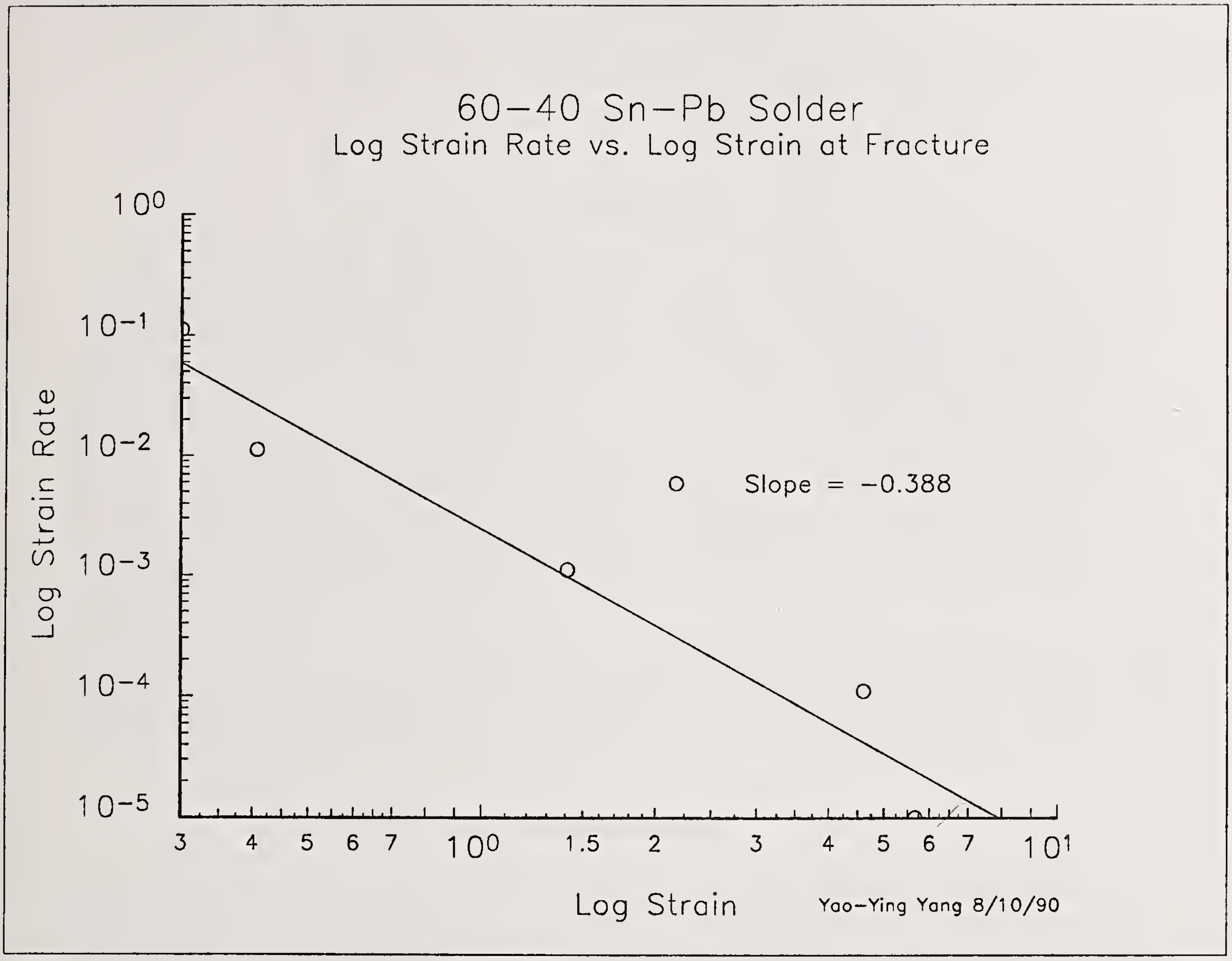

Figure 7. Log strain rate vs log fracture strain.

POST HEAT TREATMENT IN LIQUID PHASE SINTERED TUNGSTEN-NICKEL-IRON ALLOYS (1)

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Dept. of Manufacturing Systems
NC A&T State University

SUMMARY

At sintering temperature, the tungsten-nickel-iron alloy undergo grain growth and pore coarsening which degrade mechanical properties. An attempt was made to show that a post heat treatment can control the grain boundary condition toward promotion of mechanical properties of the alloy. Upon a scanning electron microscopy examination of such a heat-treated specimen, a formation of precipitation reactions at tungsten-tungsten grain boundary was traced and the type of the morphology was characterized. Energy dispersive x-ray analysis was performed to determine the composition of the precipitate and a quantitative analysis was used to measure the rate of the precipitation reaction. Also a transmission electron microscopy examination was made to determine the crystal structure of the precipitate, using the electron diffraction analysis. An instrumented Charpy test was demonstrated to show the effect of the interfacial precipitation reaction on mechanical properties of the alloy.

Key Words: Tungsten heavy alloys, liquid phase sintered W-Ni-Fe, post heat treatment in tungsten alloys, precipitation at W/W interfacial grain boundary, SEM fracture surface analysis of tungsten heavy alloys.

INTRODUCTION

The tungsten heavy alloys can find application wherever a high density material is needed. In some applications not only adequate hardness but also high strength coupled with fracture toughness are desirable. Typical application include: 1) static and dynamic balances and counterbalances, 2) rotating inertia members and gyroscopes, 3) penetration mechanics and projectiles, and 4) Radiation shielding. Cobalt as the binder metal in these alloys has escalated in cost by many times in last years. Nickel has been used as a substitute for cobalt and interestingly, klc values for nickel bonded materials can exceed those for cobalt alloys (ref. 1).

Tungsten (W)-nickel (Ni)-iron (Fe) alloys are manufactured using the standard powder metallurgical technique of hydrostatically cold pressing elemental powders followed by a liquid phase sintering. During the sintering process, the liquid phase of nickel and iron dissolve some of the tungsten, while very effectively wetting the remaining solid tungsten. At the sintering temperature, most materials undergo grain growth and pore coarsening, which degrade mechanical properties (ref. 2). Property improvements are related to processing variables such as heat treatment after sintering to control grain boundary conditions. The primary outcome being higher mechanical properties and a better combination of properties such as strength, toughness and hardness. This paper investigates a post heat treatment leading to possible formation of an interfacial precipitation reaction in a commercially produced tungsten heavy alloy (90W-7Ni-3Fe). An attempt is made to show that this precipitation reaction, resulting in changes to the microstructure, can possibly enhance the mechanical properties of the alloy.

(1) The work has been done under a contract with Battelle columbus

EQUIPMENT AND SUPPLIES

90W-7Ni-3Fe alloy specimens, vacuum furnace, instrumented Charpy tester, scanning electron microscope, energy dispersive x-ray system, transmission electron microscope, specimen preparation stations for optical and transmission electron microscopes, optical microscope with camera.

EXPERIMENTAL TECHNIQUES & PROCEDURES

Material Processing of Commercial Alloys

The production of tungsten-nickel-iron (90W-7Ni-3Fe) begins by mixing controlled amounts of the appropriate elemental powders [90 weight% (wt%), 7 wt% nickel, 3 wt% iron] and then hydrostatically compacting the blend to produce a green billet. The sintering process starts by heating the billet to 900 °C for 2 hours in hydrogen atmosphere. The temperature is then raised to 1310 °C with an 80 °C/hr heating rate and then held for 1 hr. This is followed by a temperature increase to 16 °C above liquid phase temperature with a 40 °C/hr heating rate and holding at that temperature for 1 hour. At the end of this hold time the temperature is reduced to 1200 °C. Then hydrogen is switched to helium and further cooling proceeds to ambient temperature. During the sintering process, the liquid phase of nickel and iron dissolve some of the tungsten, while very effectively wetting the remaining solid tungsten. Figure 1 shows the resulting microstructure in an optical microscope from the polished surface of the produced as-sintered billet . Spheroidal tungsten-rich grains are embedded in a Ni-Fe-W matrix. The bulk chemical analysis results for the produced as-sintered billet is presented in table 1.

Fracture Surface of the As-Sintered Specimen

Many sub-size notched specimens were cut from the as-sintered billets (commercial 90W-7Ni-3Fe), for the study of fracture surface by scanning electron microscopy (SEM). These specimens were broken with a sharp blow to create fracture surfaces. Photomicrographs were taken on various areas of a fracture surface by SEM. Figure 2 shows such a fracture surface with cleavage failure of the tungsten and ductile dimple failure of the matrix. An important common feature in these micrographs is the presence of the many smooth separation facets at the w-w grain boundaries, while a few facets with precipitation were observed. It will be subsequently shown that the microstructure of this interface region can be altered by appropriate heat treatments. A counting analysis was made on different shots to measure the average percentage of occurrence of the precipitation formation at the tungsten/tungsten interface. The percentage of precipitation occurrence (P.P.O) is defined as follows:

$$\text{P.P.O} = (N_{\text{ppt}} / N_{\text{total}}) \times 100$$

where, N_{ppt} referred to the number of facets with precipitation and N_{total} is the total number of w-w interfacial facets. To establish a comparison basis for the P.P.O values from different micrographs, all the shots are taken with the same magnification. Figure 3 illustrates the w-w smooth and precipitated separation facets from the resulting micrograph in a shot. The P.P.O for this shot is calculated as follows:

$$N_{\text{total}} = 41, \quad N_{\text{ppt}} = 15$$

P.P.O = (15 / 41) x 100 = 37 %

Similarly, the P.P.O. values for different shots (all at X400 mag.) from the micrographs of the as-sintered specimen were calculated and listed in table 2. The weighted P.P.O for as-sintered specimen is 49 %, as indicated in the table.

Fracture Surface of the Post Heat-Treated Specimen

A range of different post heat treatments, see table 3, on many as-sintered specimens were performed to locate the optimal heat-treatment process for producing the precipitation reaction at w-w grain boundary. All these heat treatments were done in vacuum furnace. The rates of heating and cooling were 50 °C/min. The post heat-treated specimens were broken by a sharp blow to create fracture surfaces. SEM examination of these fracture surfaces showed that a precipitation reaction occurs at the interfacial w-w grain boundary. However, a significant rate of formation of this precipitation is related to the post heat treatments numbers 1, 2, 10, 11, and 15 from table 3. SEM photomicrographs for different shots (all at X400 mag.) of these fracture surfaces are taken. The P.P.O values from the micrographs related to the post heat treatments with significant rate of precipitation formation are listed in table 2. The weighted values of these P.P.O's for different post heat treatments are illustrated in figure 4. From figure 4, the optimal post heat treatment is concluded to be heat treating process number 10 (solution treatment at 1000 °C for 4 hrs) which has an average P.P.O of 88 %. Figure 5 shows the SEM photomicrograph of the fracture surface of such a post heat-treated specimen with many precipitated facets. The type and identification of these precipitates will be considered in a latter section.

Energy Dispersive Spectroscopy

The SEM photomicrograph of the polished surface of the optimal post heat treated specimen (solution treatment at 1000 °C for 4 hrs) is shown in figure 6. Three points A, B, and C are marked on this micrograph, as shown in the figure, for chemical analysis purpose. These three points correspond to tungsten grain, Ni-Fe-W matrix, and W-W grain boundary in the alloy microstructure. The energy dispersive spectroscopy (EDS) are performed to determine the chemical composition of the alloy at these points. The results are reflected in figures 7,8, and 9. This analysis is discussed later.

Charpy Test

Charpy V-notched specimens were machined from commercially produced alloy to the dimensions shown in figure 10. Instrumented impact tests on both, as-sintered, and the optimal post heat-treated specimen (solution treatment at 1000 °C for 4 hrs) were conducted and compared. The results of these instrumented impact tests are shown in figure 11. These results are discussed in a latter section.

Transmission Electron Microscopy (TEM)

The precipitate structure was analyzed by TEM. Specimen sections were taken from the Charpy test specimens at locations near and far from the fracture surface. Thin sections of approximately 300 microns thick were sliced using a slow speed diamond saw. These sections were then thinned on a Minimet grinder/polisher. Further thinning to 50 microns was accomplished by using a dimple grinder after which ion milling was used to thin the specimen to final electron transparency. The TEM micrograph of the precipitate phase at the w-w grain boundary and the diffraction pattern corresponding to the selected area are shown in figures 12 and 13 respectively. The results are discussed later.

DISCUSSION OF RESULTS

It was shown that the as-sintered commercial alloys (90W-7Ni-3Fe) has a low weighted P.P.O. An examination of the SEM micrographs of the fracture surfaces of the as-sintered specimens compared to the post heat-treated ones shows that the rate of the precipitation formation in these alloys increases for successive post heat treatments. The average percentage of precipitation formation at the w-w interfaces reaches to a maximum of 88 % corresponding to the solution treatment at 1000 ^{o}C for 4 hours. At post heat treatments above 1000 ^{o}C temperature, the precipitation appear to be much coarser and less homogenous and have a lower P.P.O.

The EDS compositional analysis of the tungsten grain and of the matrix as well as of the grain boundary in a microstructure of the post heat-treated specimen were shown in figures 7, 8, and 9. As indicated in these figures, the elemental analysis for the matrix is as follows:

Element	Weight %
W	16.70
Fe	23.96
Ni	59.33

Also from these figures, the presence of large nickel and iron concentrations in the precipitates at the w-w grain boundary was verified. Possible electron beam spreading could have allowed the surrounding tungsten grain to contribute to the tungsten peaks.

The precipitations morphologies are of different types, nodular and lamellar, on the same specimen. Secondary and Backscattered modes are used to show the nodular type morphology as shown in SEM photomicrograph of the fracture surface, figure 14.

Charpy tests utilized notched specimens to determine the fracture toughness and impact strength. A comparison of the computer results, figure 11, shows a slight improvement in toughness and impact strength, calculated as follows:

% Increase in Impact Strength = [(39833-37489) psi / 39822 psi] x 100 = 6 %

% Increase in toughness = [(7-6) ft.lb / 7 ft.lb] x 100 = 14 %

TEM micrograph for the thin foil shows the precipitate phase at the w-w grain boundary, figure 12. Diffraction pattern corresponding to the selected area of figure 12, obtained at 240 Kev, is shown in figure 13. By applying the Bragg's Law formula and using the wavelength of the incident electron beam (0.033 A^{o}), the interplanar spacing could be calculated. Having the interplanar spacing, the lattice parameter of the diffracted spots may be determined. Such a simple calculation shows that all diffraction spots from many different precipitate have a lattice parameter of 3.60 A^{o} consistent with a fcc structure, equivalent to the fcc matrix phase. Higher accelerating voltage (1 Mev) is required to obtain a more complete pattern to determine the crystallographic orientation relationship between the fcc precipitate and the adjacent bcc tungsten grain, (ref. 3).

MECHANISM OF PRECIPITATION

Different theories has been proposed to explain the mechanism of precipitation enrichment at the tungsten grain boundaries. The following theory, as put forth by Posthill, (ref.4), is considered here. Posthill concluded that a grain boundary having rational orientation relationships with both adjacent grains would be expected to minimize the surface free energy, thus facilitating nucleation by lowering the critical free energy. This would also increase the probability of precipitate nucleation on that boundary. He tentatively concluded that precipitation on w-w grain boundaries is due to nickel and iron coming out of supersaturated w-phase solution. While grain boundary diffusion is obviously essential for the nucleation and growth of the precipitation, it also leads to some transport of nickel and iron to the matrix region. Figure 15 illustrates this mechanism.

CONCLUSION

The present examination of the post heat-treated 90W-7Ni-3Fe alloy confirms that a precipitation of matrix phase type does occur at the tungsten-tungsten interfacial grain boundary. The post heat treatment at 1000 ^{o}C temperature for 4 hrs showed the maximum percentage of precipitation occurrence at the w-w grain boundary. The types of morphologies are identified as nodular as well as lamellar with a fcc structure. In this study it is believed that this reactions occurs by precipitation of nickel and iron from the supersaturated tungsten grains to the boundaries. These reactions are expected to promote the impact resistance and toughness of the heavy alloy since they improve the cohesion at interfacial grain boundaries (ref. 5). However, the Charpy tests results showed that the fracture toughness and the impact strength of the optimal post heat-treated specimen have improved slightly. One reason that might account for this, could be the irregular formation of harmful precipitation hardening in w-phase and matrix region of the heavy alloy. This kind of precipitation is expected to increase the brittleness of the alloy since they reduce the interphase boundary cohesion (ref. 6). Research should be conducted toward locating a post heat treatment that accelerates the precipitation occurrence at the w-w interface boundary and at the same time lowers the harmful precipitation formation in the microstructure of the tungsten heavy alloy.

TABLE I. BULK CHEMICAL ANALYSIS RESULTS (WT%) FOR THE COMMERCIAL ALLOY 90W-7NI-3FE

AG	< 1E-4
CR	< 4E-4
MG	< 2E-4
SB	< 0.002
V	< 0.001
AL	5E-4
BI	< 0.001
MN	< 4E-4
CU	< 2E-4
MO	< 0.002
N	5E-4
0	< 0.0071
W	90.74
TH	< 0.01
CA	< 0.001
NA	< 0.002
PB	< 0.001
SI	5E-4
B	< E-4
SN	< 4E-4
ZR	0.002
CO	< 6E-4
U	< 0.01
H	<0.0091
NI	6.08
FE	2.68

TABLE II. CALCULATED P.P.O AT W-W GRAIN BOUNDARY IN COMMERCIAL ALLOY 90W-7NI-3FE FOR AS-SINTERED AND POST HEAT-TREATED SPECIMENS

H.T.	shot #	N_{ppt}	N_{total}	P.P.O
as-sintered	1, 2, 3, 4	23, 18, 20,23	44, 47, 35,38	52, 38, 57,61
# 1	1, 2	23, 20	41, 37	56, 54
#2	1, 2	43, 31	63, 48	68, 65
#10	1, 2, 3	56, 44, 46	62, 49, 55	90, 90, 84
#11	1, 2	31, 20	46, 31	67, 65
#15	1, 2	26, 22	40, 34	65, 65

TABLE III. POST HEAT TREATMENTS OF 90W-7Ni-3Fe ALLOY

H.T. #	1st h.t. (°C)	Time (hrs)	2nd h.t.(C)	Time (hrs)
1	700	1		
2	800	1		
3	900	1		
4	1000	1		
5	1100	1		
6	1200	1		
7	700	4		
8	800	4		
9	900	4		
10	1000	4		
11	1100	4		
12	1200	4		
13	1000	1	500	2
14	1000	1	600	2
15	1000	1	700	2

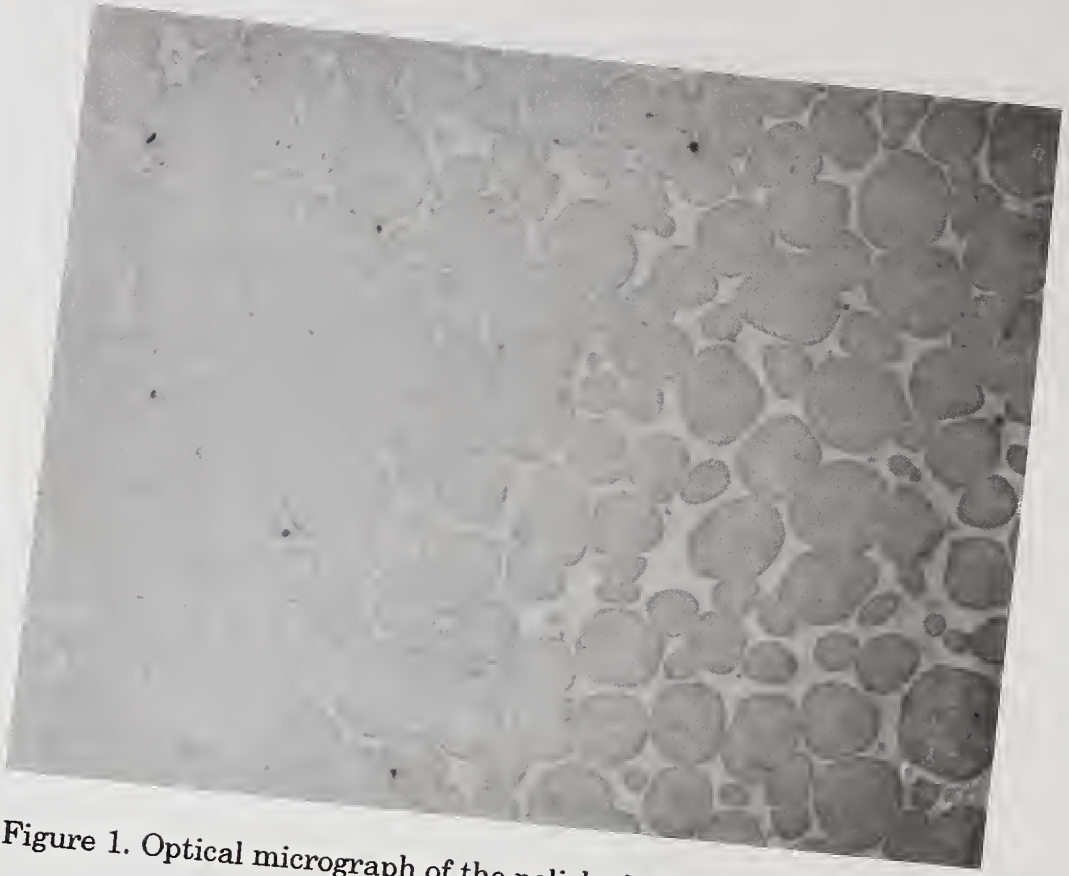


Figure 1. Optical micrograph of the polished surface of commercially supplied 90W-7Ni-3Fe heavy alloy, X200.

Figure 2. SEM micrograph of the fracture surface of the as-sintered 90W-7Ni-3Fe heavy alloy.

Figure 3. Separated and precipitated facets in the microstructure of the fracture surface for the as-sintered 90W-7Ni-3Fe heavy alloy

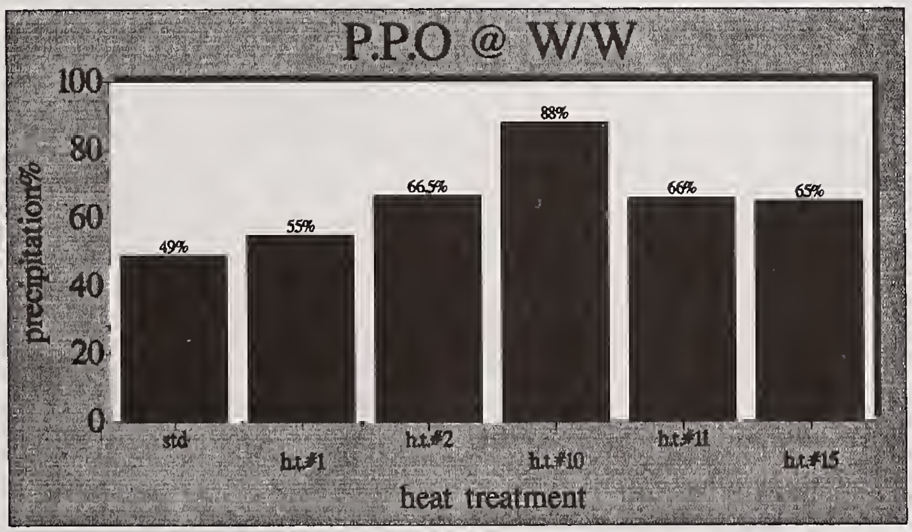


Figure 4. Percentage of precipitation occurrence at W-W grain boundary for as-sintered and post heat-treated 90W-7Ni-3Fe heavy alloys.

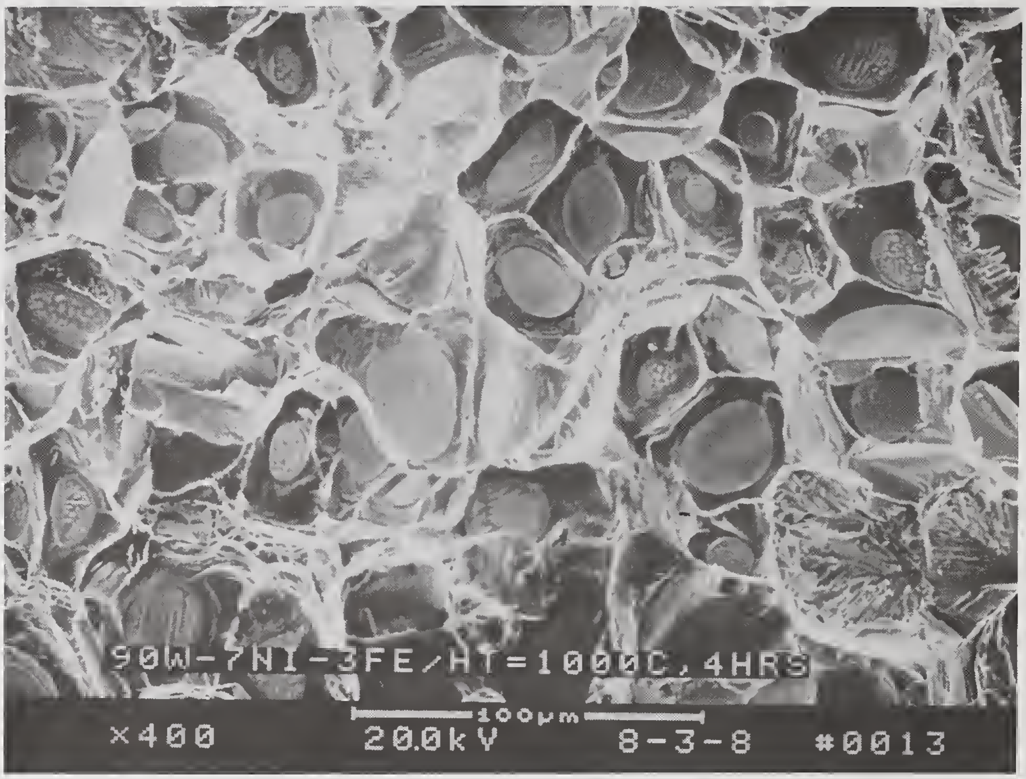


Figure 5. SEM micrograph of the fracture surface of the commercial tungsten 90W-7Ni-3Fe heavy alloys, post heat treated at 1000°C for 4 hrs.

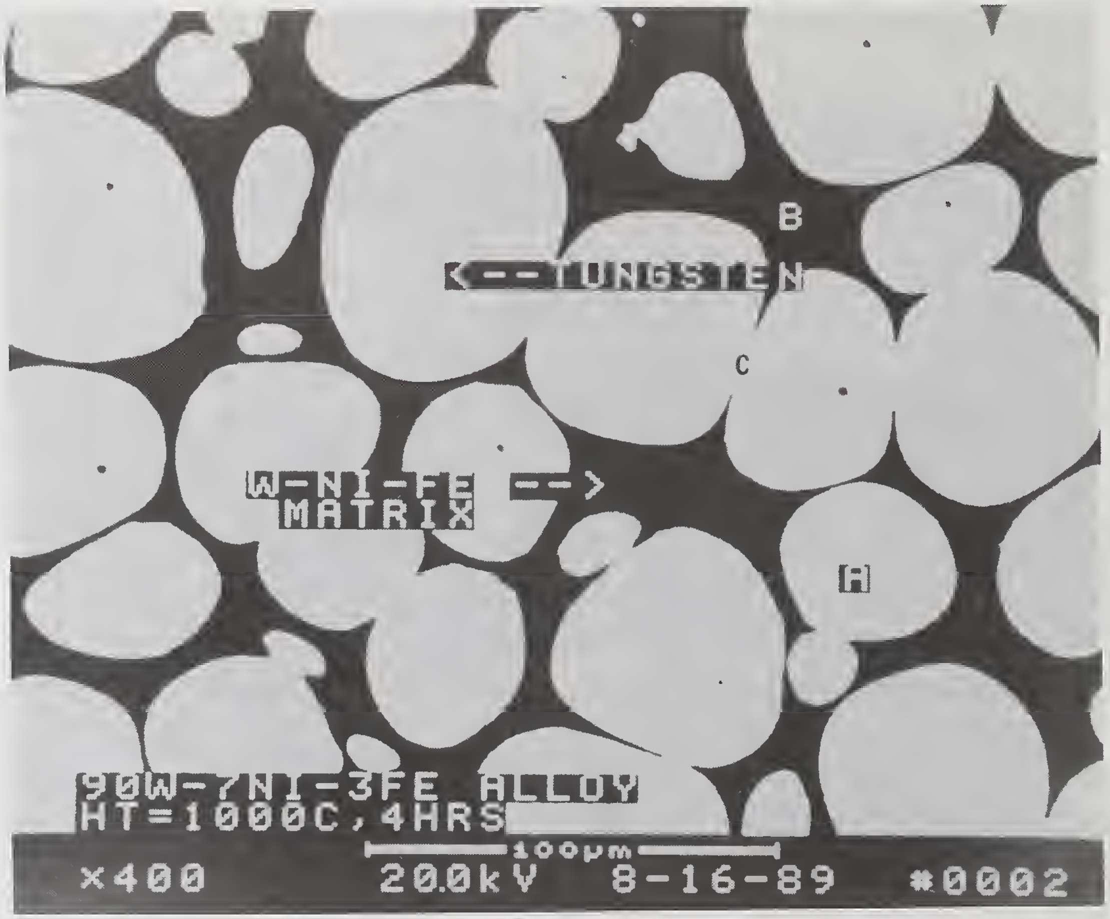
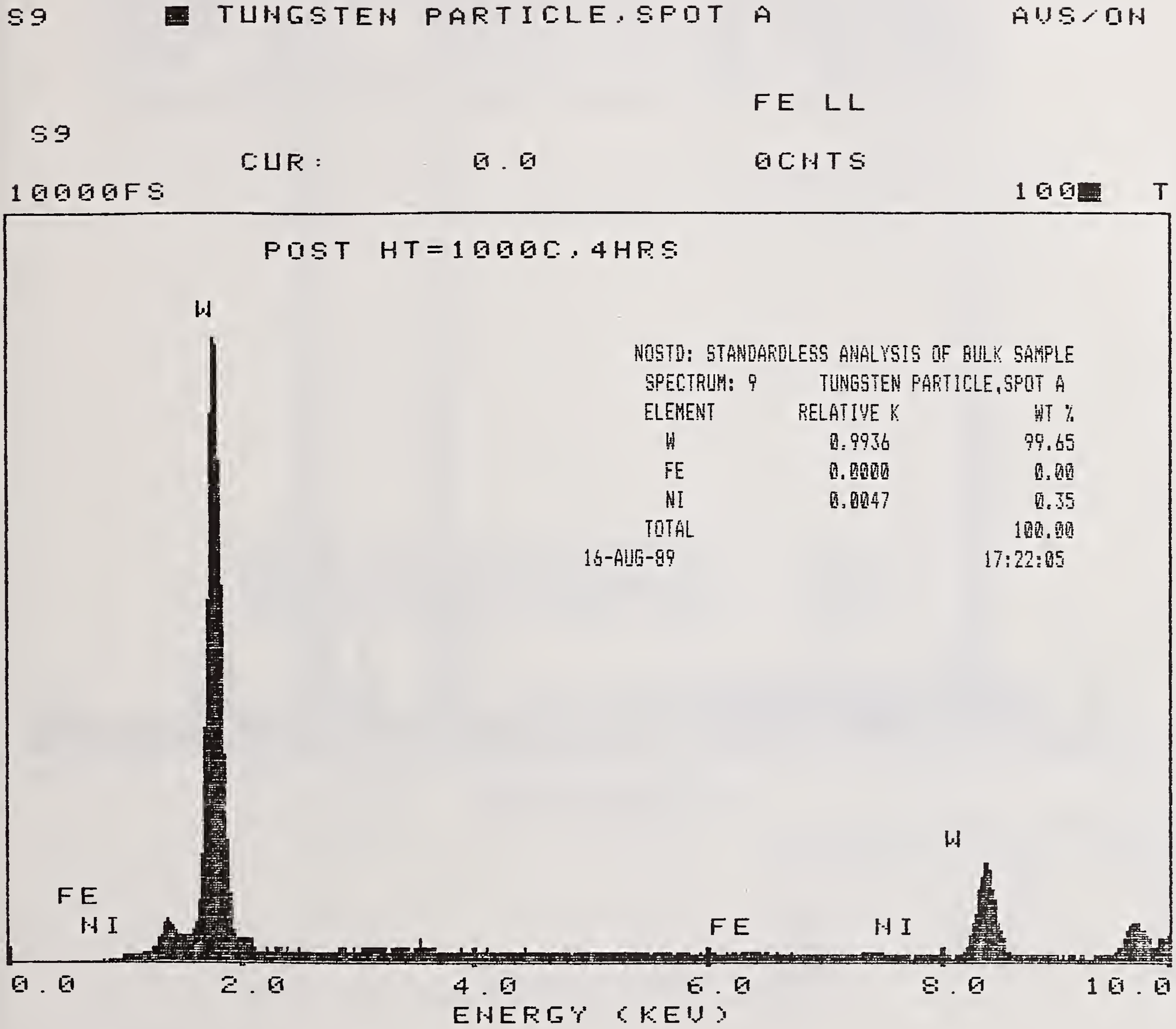


Figure 6. SEM micrograph of the polished surface of the commercial tungsten 90W-7Ni-3Fe heavy alloy, post heat treated at 1000°C for 4 hrs.



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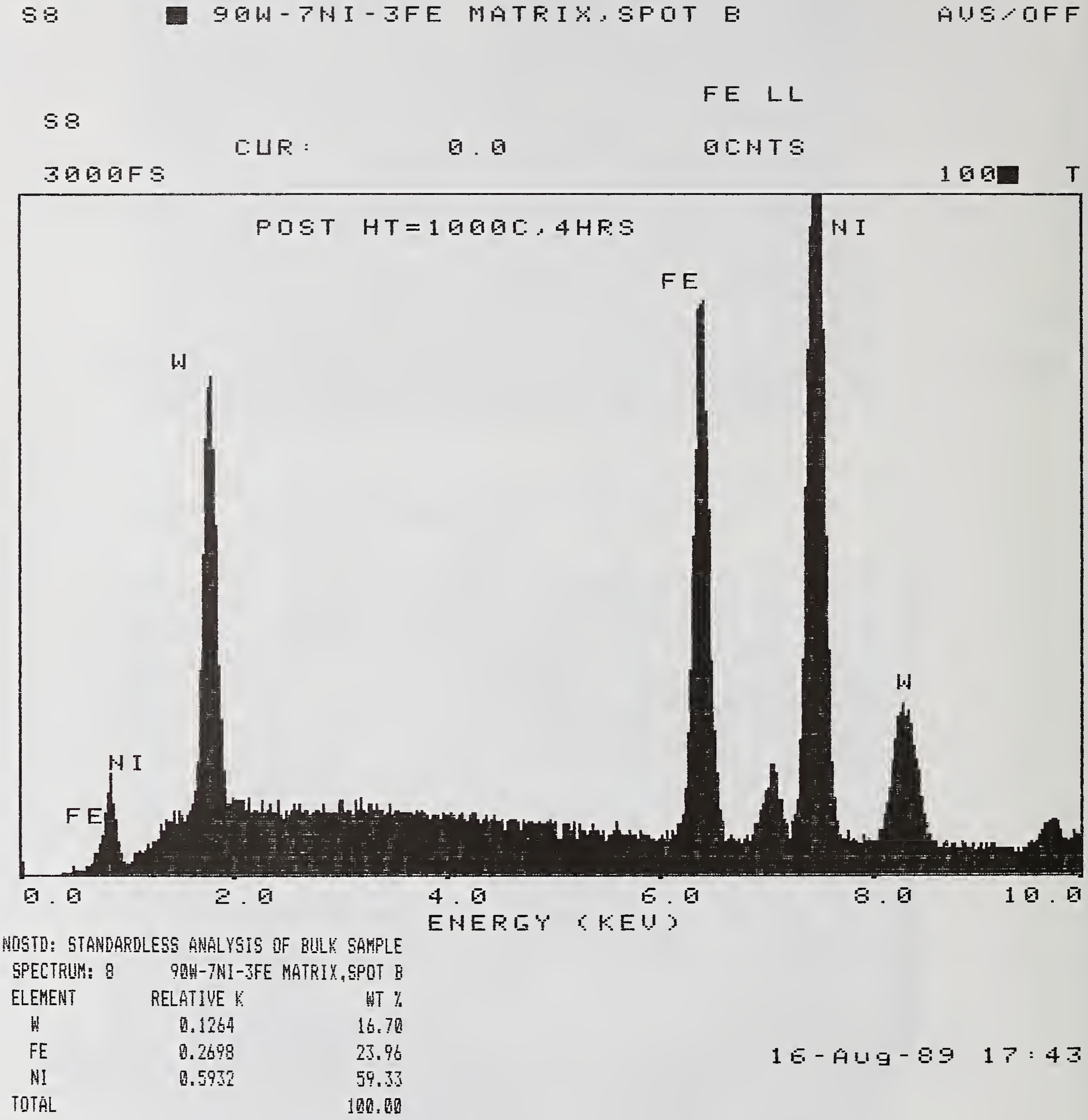
NOSTD: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: 9      TUNGSTEN PARTICLE,SPOT A
 ELEMENT        RELATIVE K              WT %
    W             0.9936               99.65
    FE            0.0000                0.00
    NI            0.0047                0.35
 TOTAL                                100.00
16-AUG-89                           17:22:05
```

Figure 7. Tungsten alloy (90W-7Ni-3Fe), post heat treated at 1000°C for 4 hrs.
EDS chemical analysis at spot A of the microstructure shown in figure 8.



```
NOSTD: STANDARDLESS ANALYSIS OF BULK SAMPLE
 SPECTRUM: 8      90W-7NI-3FE MATRIX,SPOT B
 ELEMENT         RELATIVE K            WT %
   W              0.1264              16.70
   FE             0.2698              23.96
   NI             0.5932              59.33
 TOTAL                               100.00
```

Figure 8. Tungsten alloy (90W-7Ni-3Fe), post heat treated at 1000°C for 4 hrs. EDS chemical analysis at spot B of the microstructure shown in figure 8.

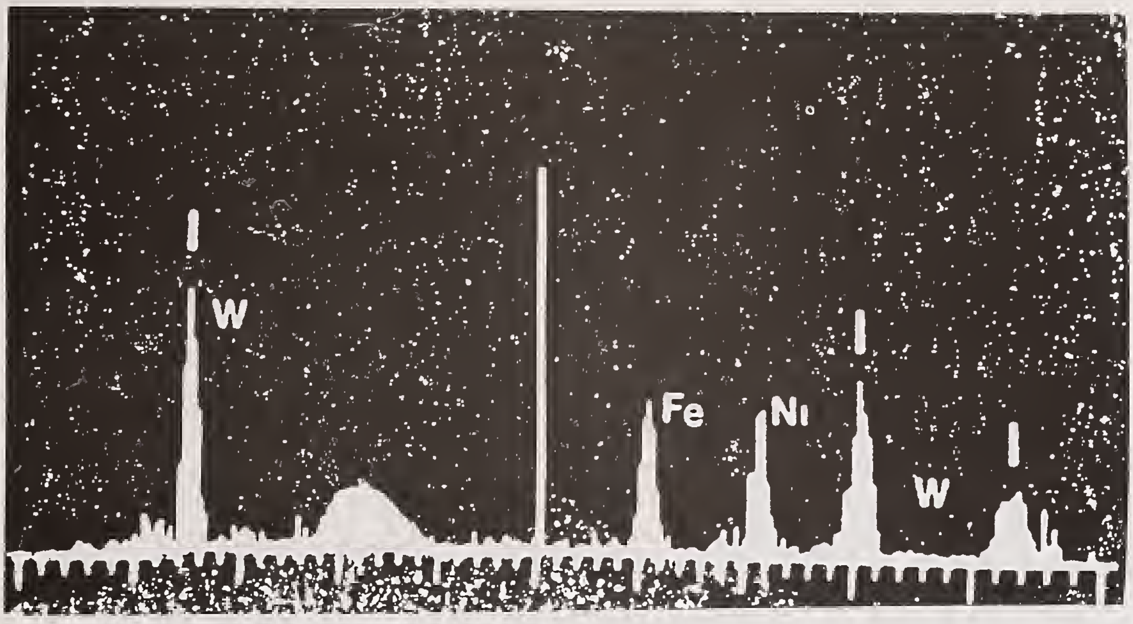


Figure 9. EDS spectra of the w-w boundary precipitate, showing substantial nickel and iron, post heat-treated tungsten 90W-7Ni-3Fe alloy.

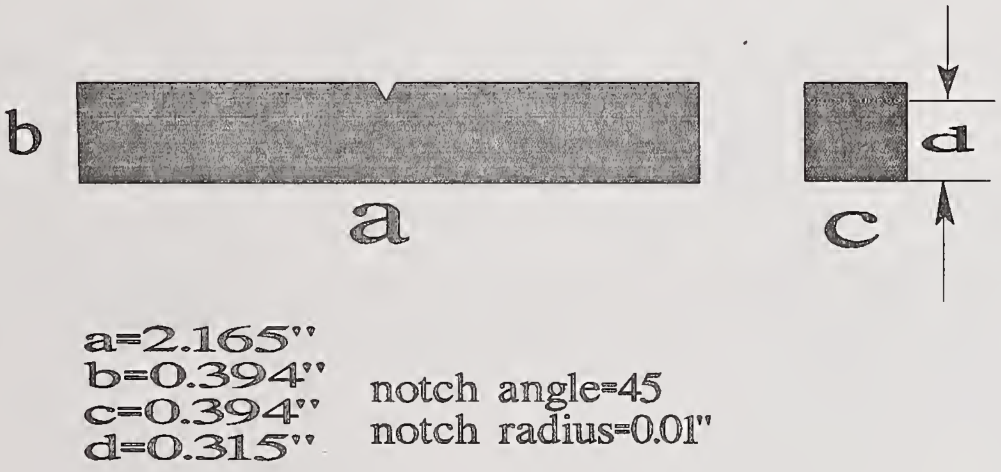


Figure 10. As-sintered Charpy V-notched specimen

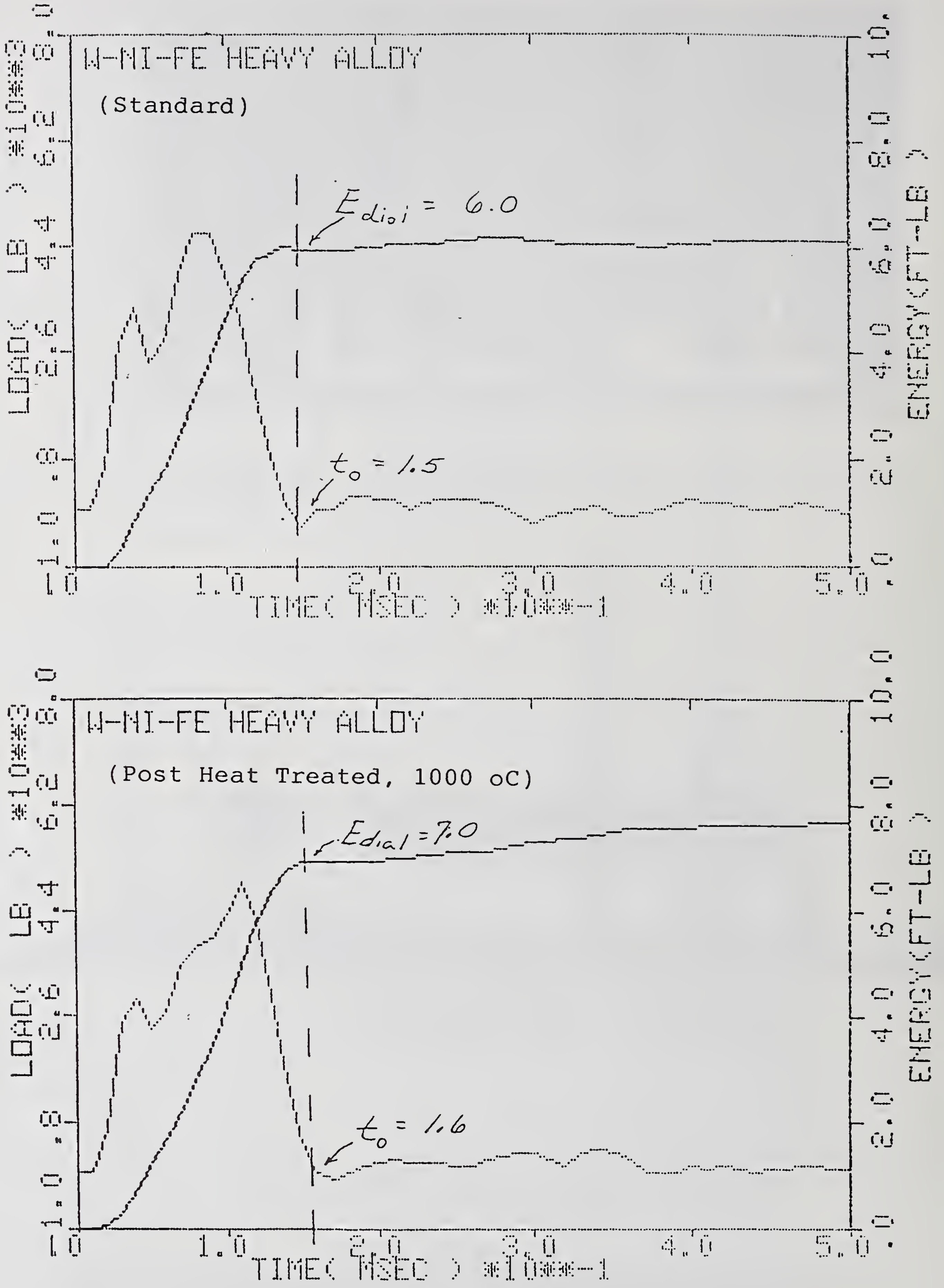


Figure 11. Graphical presentation of the instrumented impact results for the V-notched specimen, tungsten 90W-7Ni-3Fe heavy alloys.

Figure 12. TEM micrograph, showing precipitation at w-w grain boundary in tungsten90W-7Ni-3Fe heavy alloy, post heat treated at 1000°C for 4 hrs.

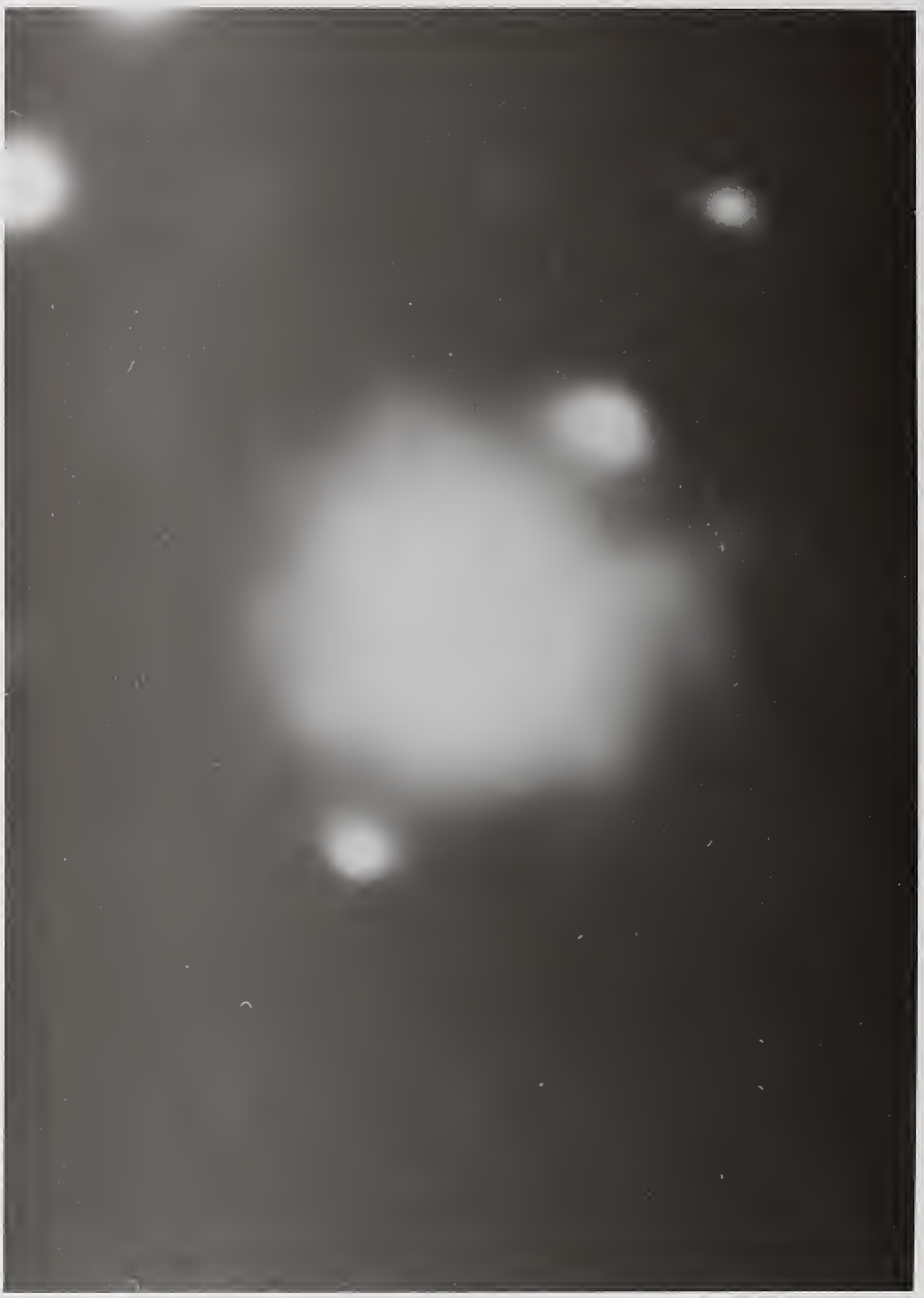


Figure 13. Electron diffraction pattern corresponding to the TEM micrograph shown in fig 11.

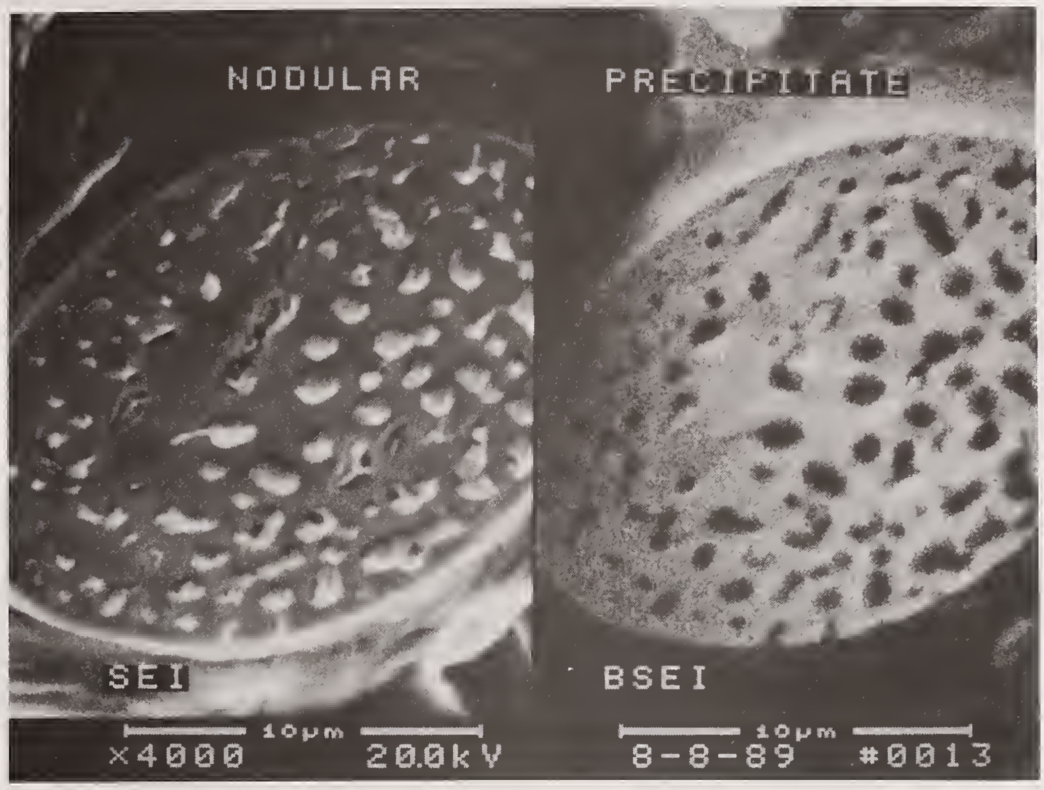


Figure 14. SEM secondary and backscattered electron imaging of nodular morphology of precipitation at w-w grain boundary in 90W-7Ni-3Fe alloy.

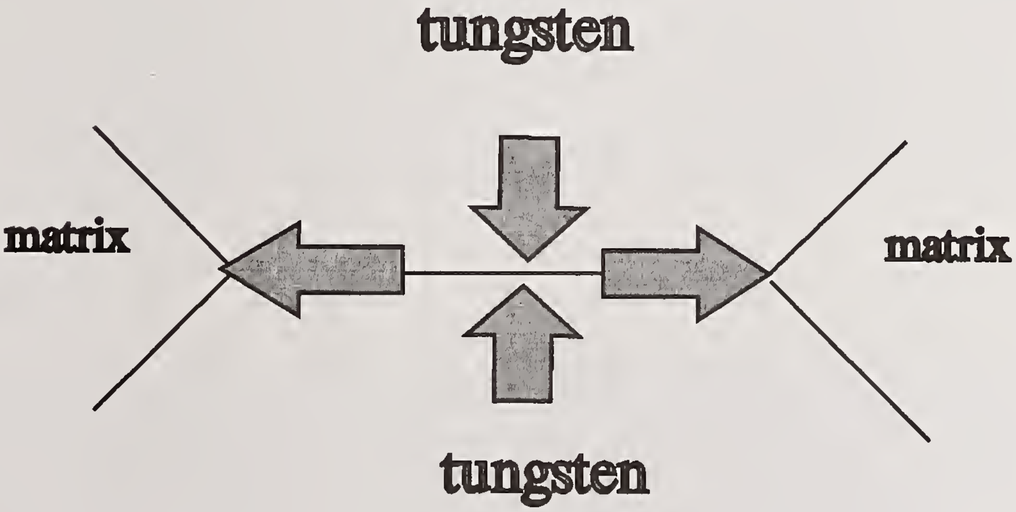


Figure 15. Mechanism of precipitation enrichment at w-w grain boundary in tungsten 90W-7Ni-3Fe heavy alloy.

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IMPROVED TECHNIQUE FOR MEASURING COEFFICIENTS OF THERMAL EXTENSION FOR POLYMER FILMS

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SUMMARY

The coefficient of thermal extension (CTE) is an important material property that describes the change in length of a material as a function of the change in temperature. More specifically, $\alpha = (1/L_o)[(L_f - L_i)/(T_f - T_i)]$, where α is the CTE of the material, L_o is the length of the specimen at room temperature, and L_i and L_f are the lengths of the material at temperatures T_i and T_f, respectively. CTE values can be determined through the use of thermomechanical analysis (TMA). A commercial TMA system has been modified to improve the accuracy of the measurement by increasing the length of the specimen. CTE values for Teflon* unexposed and exposed to thermal extremes have been determined using the modified TMA system.

INTRODUCTION

Thermal expansion and contraction take place in materials due to changes in temperature. The way a material expands is dependent upon its chemical structure. When a material expands upon heating, the free space between atoms expands. A rise in temperature causes atoms to vibrate at higher frequencies; and, in turn, the average separation between atoms increases. Therefore, materials with chemical structures that have strong covalent bonds will have lower CTE values than those of chemical structures that have weak interatomic bonds. Because of their weak interchain bonds, polymers have relatively high CTE values. Quartz (SiO_2), on the other hand, has very strong molecular bonds and, therefore, has a low expansion coefficient. CTE values of some common materials (as found in References 1, 2, and 3) are given in Table 1.

The coefficient of thermal extension is determined by measuring the change in length of a specimen through a predetermined temperature range. The equation used for calculating CTE values is

$$\alpha = (1/L_o)[(L_f - L_i)/(T_f - T_i)] \qquad (1)$$

where α is the CTE of the material, L_o is the length of the specimen at room temperature, and L_i and L_f are the lengths of the specimen at temperatures T_i and T_f, respectively. When the length of the specimen is plotted as a function of the temperature, it can be seen that the CTE is proportional to the slope of the extension curve.

TECHNIQUE

Thermomechanical analysis (TMA) was the method used to measure CTE values for this discussion. TMA is a technique in which changes in specimen length due to a controlled rate of temperature change are electronically recorded. The TMA system used was a DuPont 940 (Reference 4). It consisted of a specimen holder, probe, linear variable differential transformer (LVDT), thermocouple, heating unit, and a DuPont 1090 Thermal Analyzer (Figure 1).

The specimen is clamped on both ends (Figure 2). The upper clamp is held by a stationary support, while the lower clamp is hooked to and supports the probe. As heat is applied to the sample region, the dimensions of the sample, sample holder, probe, and clamps change. The LVDT, positioned outside the heater cavity, sends an electrical signal to the analyzer which corresponds to the net movement of the probe. The quartz specimen holder and probe and the steel clamps have CTE values much smaller than most samples, and thus, do not contribute significantly to the signal.

Originally, the TMA system was designed for specimens with length 0.2 inches (5 mm) and width 0.1 inches (2.54 mm). The quartz probe has been lengthened so that specimens now have a length of approximately 1.5 inches (38 mm) and a width of 0.1 inches (2.54 mm). The increased specimen length greatly improves the accuracy of the thermal extension measurement. As given in Reference 1, the precision of the test can be determined using the equation,

$$\delta\alpha/\alpha = \pm(\delta\Delta L/\Delta L + \delta L/L + \delta\Delta T/\Delta T) \qquad (2)$$

where:

ΔL = the change in length of the material, mm,
$\delta\Delta L$ = the imprecision in measuring ΔL, mm,
L = the length of the material at room temperature, mm,
δL = the imprecision in measuring the length of the material, mm,
ΔT = the temperature range of the test, °C,
$\delta\Delta T$ = the imprecision in measuring ΔT, °C,
α = the coefficient of thermal extension of the material, /°C, and
$\delta\alpha$ = the imprecision in the measurement of α, /°C.

It is noted in Reference 1 that CTE measurements should be made under conditions such that the imprecision in measuring the length, δL, is ±0.025 mm; the imprecision in measuring the change in length, $\delta\Delta L$, is $\pm 10^{-3}$ mm; and the imprecision in measuring the change in temperature of the test, $\delta\Delta T$, is $\pm 0.5^{o}C$.

As L becomes larger, the percent error, $\delta L/L$, becomes smaller. A larger initial length of the specimen also causes a larger change in length over the same temperature region, causing $\delta\Delta L/\Delta L$ to become smaller as well. The smaller percent errors in measurement of L and ΔL cause the percent error in calculating α to decrease. For example, the percent error in calculating α for aluminum with initial length 5 mm, and a ΔL of 11.5 μm (calculated from $\alpha_{Al} = 2.3 \times 10^{-5}/^{o}C$ over a temperature range of $0^{o}C$ to $100^{o}C$) was calculated to be approximately ±9.7%. The error in calculation with an initial length of 38 mm and a ΔL of 84.7 μm, was approximately ±1.7%.

EXPERIMENTAL

Before measurements can be made, the LVDT signal must be calibrated and the expansion of the quartz holder and probe with the steel clamps must be measured. The LVDT is calibrated by measuring the distance between the hooks with a micrometer and comparing this measurement to the LVDT signal on the recorder. Calipers were used to initiate movement of the hooks and successive measurements were made at small increments of length. The measurements from the micrometer were plotted as a function of the readings from the analyzer. The slope of this curve is the multiplicative LVDT calibration factor, which is in units of mm/mV. The LVDT calibration factor for this test was 0.9860 mm/mV.

As a sample is heated, the displacement of the probe is recorded as voltage output by the LVDT. Corresponding temperature readings are measured by a thermocouple and are also recorded. The LVDT factor is entered into the DuPont Thermal Analyzer; during analysis, each displacement data point is multiplied by the LVDT calibration factor. This multiplicative factor converts the electronic readings from millivolts (mV) to millimeters (mm). A temperature region is selected from the generated curve, as in Figure 3, and an initial CTE, α_{TA}, is calculated by the thermal analyzer using Equation 1.

The CTE value calculated by the thermal analyzer must be corrected to account for the contributions of the quartz and steel. The correction factor for the quartz and steel, α_{corr}, has been determined to be $0.065 \times 10^{-5}/^{o}C$. Thus, the final equation for the coefficient of thermal extension of the sample is

$$\alpha_F = \alpha_{TA} - \alpha_{corr} \qquad (3)$$

where α_F is the final CTE of the sample corrected for the quartz holder and probe and the steel clamps.

The TMA tests were run at a heating rate of $2^{o}C/min$. Each test began at room temperature and ended at approximately $120^{o}C$.

SPECIMENS

Specimens of various thicknesses were cut to dimensions 38 mm x 2.5 mm (1.5 in x 0.1 in). The specimen holder is designed to test films; it is necessary to keep the films in tension in order to get accurate extension readings. Weights must be added to the weight tray to keep the probe and film in tension (Figure 1). The range of weights that can be added to this system is from 1 gram to 10 grams. Thin films of thicknesses up to 2 mils cannot support even the smallest applied load; specimens of 0.5 and 1 mil thicknesses could not even withstand the load of the probe alone. In this study, it was found that the applied load caused the thinner films to expand at an artificially high rate; the weight caused the films to elongate so that the effects of temperature were obscured by the effects of the applied load. It is for this reason that thicker specimens and smaller loads are recommended. The specimens in this discussion had a thickness of 5 mils (0.127 mm) and the applied load was 1 gram; therefore, the applied stress to each film was 30.4 kPa.

MATERIAL

The film studied was a copolymer of polytetrafluoroethylene (PTFE) and fluoroethylenepropylene (FEP). Its tradename is FEP Teflon, and it is manufactured by the DuPont Chemical Company. The chemical stucture of FEP Teflon is given in Figure 4. The specific grade of FEP Teflon used was FEP Teflon-140; as discussed in Reference 3, the 140 indicates an intermediate melt viscosity. FEP Teflon-140 is specifically designed for applications involving high current loads or repeated thermal cycling. FEP Teflon is also used on the Space Shuttle and is being studied for its Space Station possibilities. In space, FEP Teflon is exposed to a wide range of temperatures. It is for this reason that the National Aeronautics and Space Administration (NASA) at Langley Research Center in Hampton, Virginia is studying the effects of extreme temperatures on FEP Teflon. FEP Teflon was exposed to temperatures between $-150^{\circ}F$ and $+150^{\circ}F$ in a thermal cycling chamber for one thousand cycles. The effect of exposure to thermal extremes on the coefficient of thermal extension of 5-mil FEP Teflon was studied. There were three test specimens for the baseline FEP Teflon and three for the thermal cycled FEP Teflon.

ANALYSIS

A sample extension curve for baseline FEP Teflon-140 is given in Figure 3. In Reference 3, the value of α for FEP Teflon-140 is cited as $13.9 \times 10^{-5}/^{\circ}C$ for the temperature region between $0^{\circ}C$ and $100^{\circ}C$. The average α value for the three baseline Teflon specimens was $(18.2 \pm 0.3) \times 10^{-5}/^{\circ}C$. For the thermal cycled specimens, it was $(23.1 \pm 1.6) \times 10^{-5}/^{\circ}C$. The temperature range used for both the baseline and thermal cycled films was from $26^{\circ}C$ to $100^{\circ}C$. This temperature range was chosen because all the CTE curves were approximately linear in this region, and

because the region coincided well with the regions used for the reference value for Teflon and the α value for the quartz and steel. The raw data are given in Table 2.

CONCLUSIONS

Two things can be concluded from these results. The first is that the closeness of the baseline data to the cited value gives confidence to the method used for this test. It can also be concluded that thermal cycling does affect the coefficient of thermal extension of Teflon. After exposure to thermal extremes, such as those during cycling, Teflon showed a higher coefficient of thermal extension. This demonstrates that FEP Teflon is affected by thermal cycling.

ACKNOWLEDGMENTS

The authors of this paper would like to acknowledge Patricia D. Gillespie, of the College of William and Mary, for her research on thermomechanical analysis which contributed to the discussion presented here.

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[*] The use of trademarks or manufacturers' names in this paper does not constitute endorsement, either expressed or implied, by the National Aeronautics and Space Administration.

TABLE 1. Coefficients of Thermal Extension.

MATERIAL	α (/°C)	TEMPERATURE RANGE (°C)	REFERENCE
Quartz	0.56×10^{-5}	20 - 700	1
Steel	1.1×10^{-5}	0 - 100	2
Aluminum	2.3×10^{-5}	0 - 100	2
Copper	1.7×10^{-5}	0 - 100	2
Glass (ordinary)	0.32×10^{-5}	0 - 100	2
FEP Teflon-140	13.9×10^{-5}	0 - 100	3

TABLE 2. Data From Thermomechanical Analysis of FEP Teflon-140.

BASELINE	THERMAL CYCLED
18.2×10^{-5}/°C	24.9×10^{-5}/°C
18.0×10^{-5}/°C	22.6×10^{-5}/°C
18.5×10^{-5}/°C	21.9×10^{-5}/°C

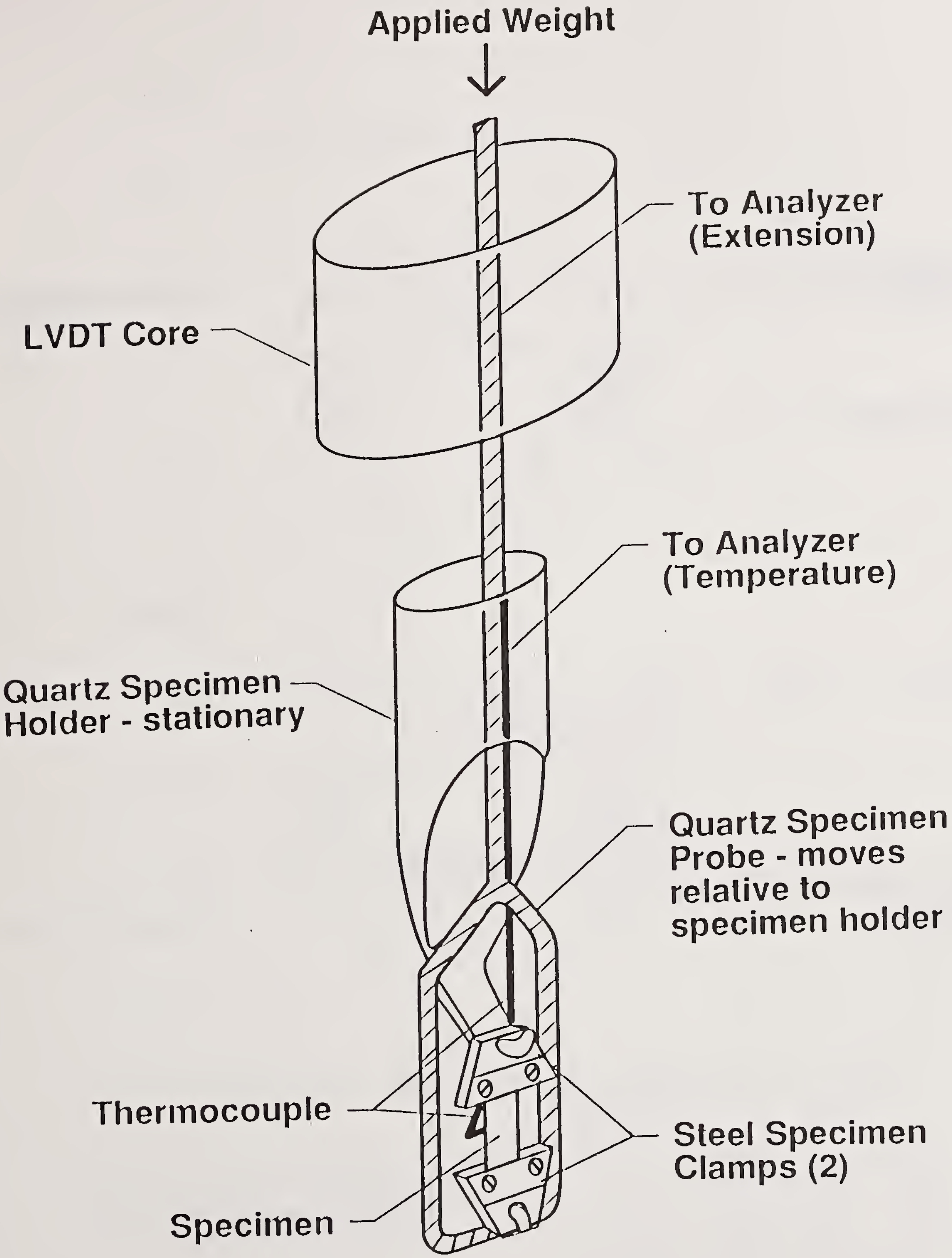


FIGURE 1. DuPont 940 Thermomechanical Analysis (TMA) Film Extension Apparatus (Reference 4).

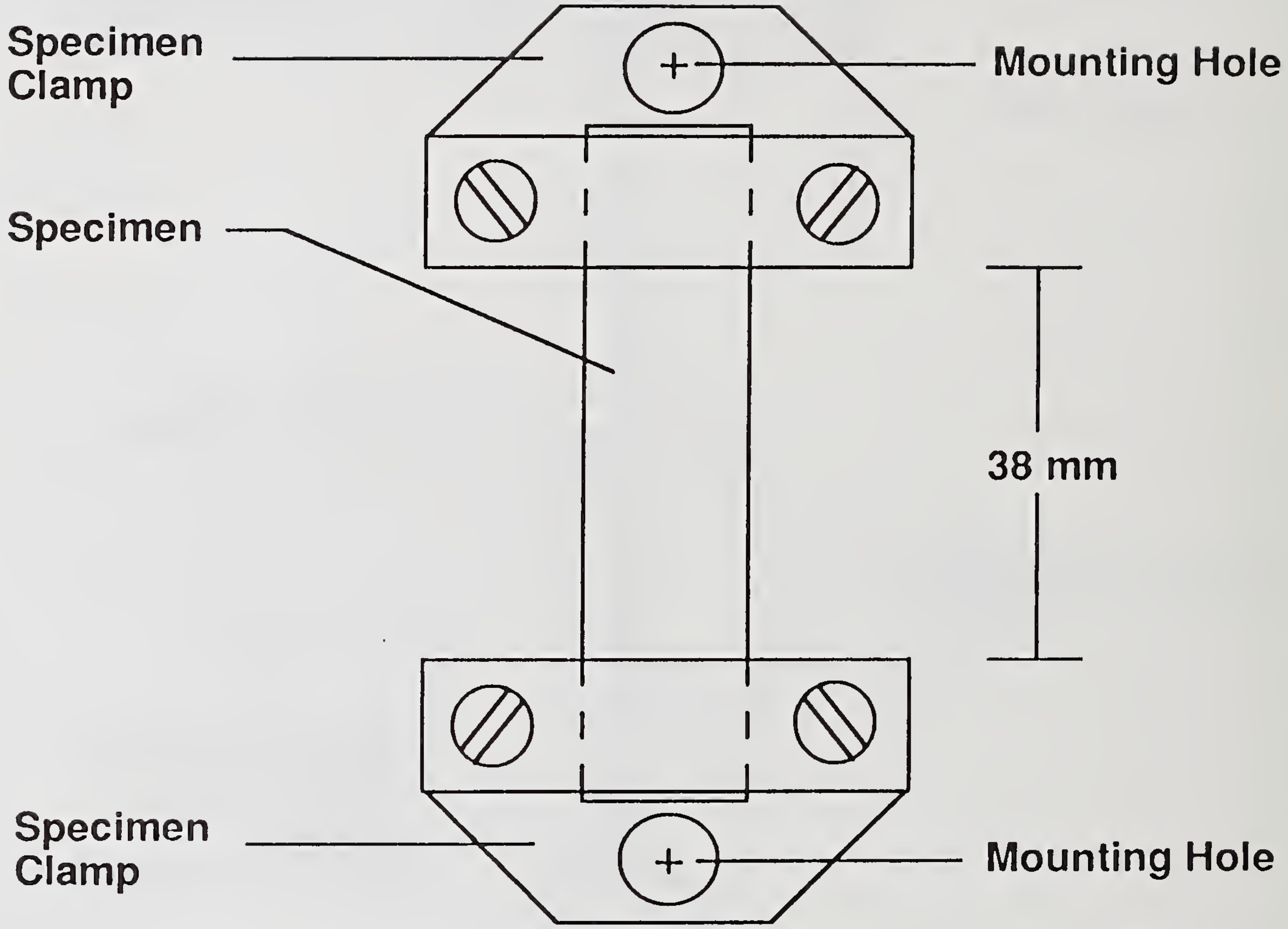


FIGURE 2. Film Sample with Steel TMA Clamps (Reference 4).

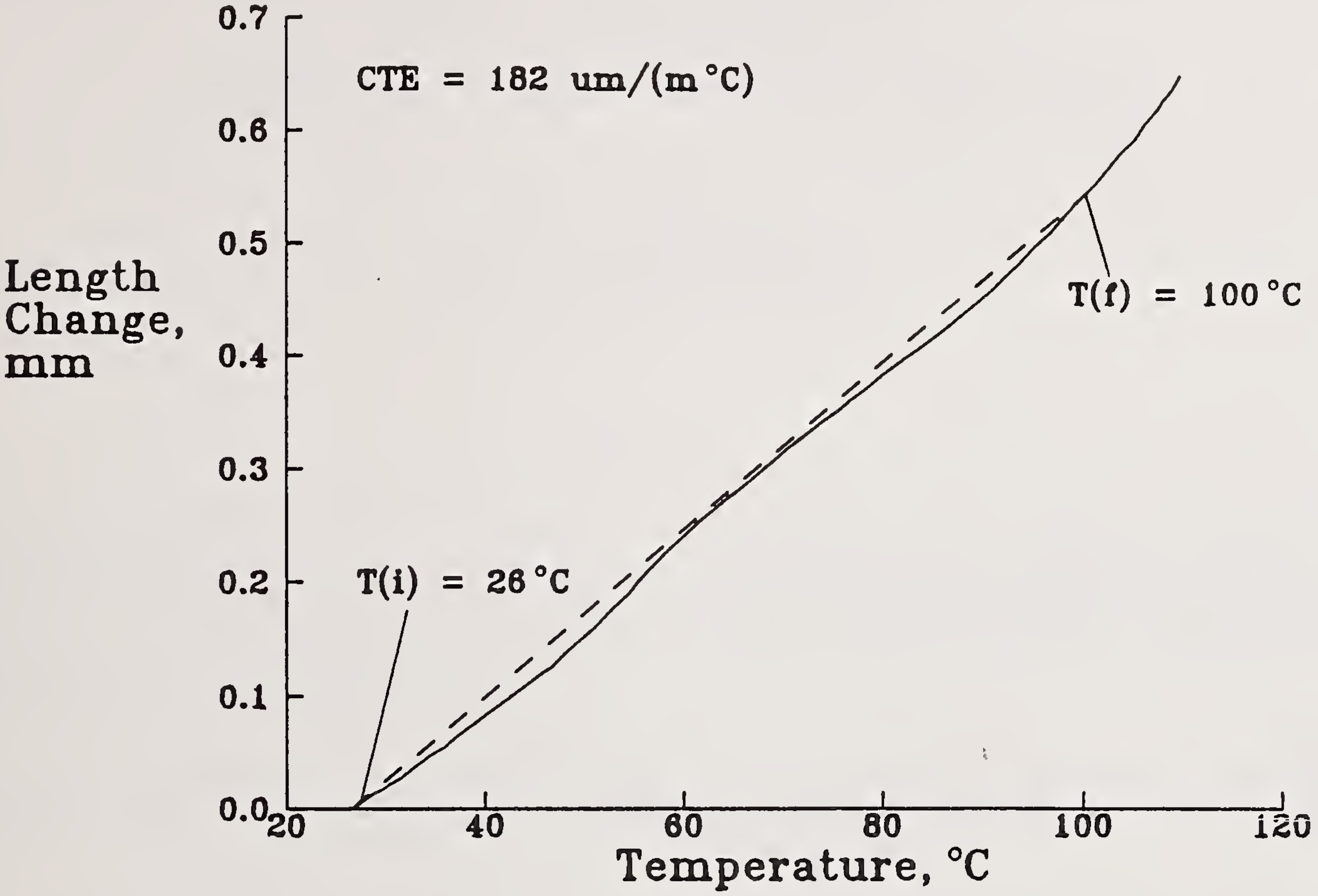
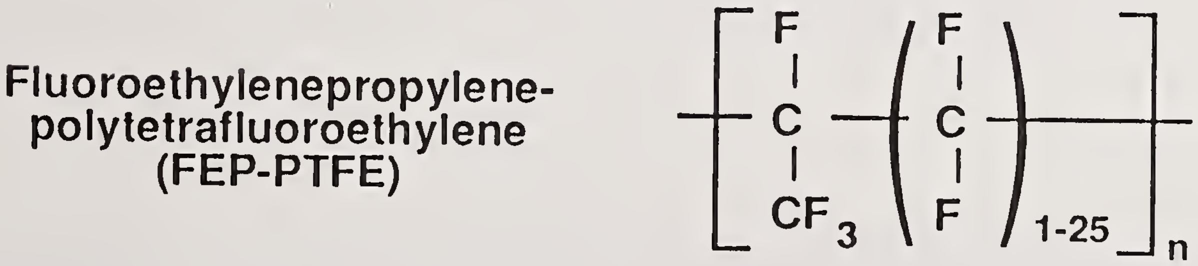


FIGURE 3. TMA Curve for Baseline FEP Teflon-140.



$$\left[-\underset{CF_3}{\overset{F}{C}}-\left(\underset{F}{\overset{F}{C}} \right)_{1-25}- \right]_n$$

FIGURE 4. Chemical Structure of FEP Teflon.

CHARPY V-NOTCH IMPACT TESTING
OF HOT ROLLED 1020 STEEL
TO EXPLORE
TEMPERATURE ~ IMPACT STRENGTH RELATIONSHIPS

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SUMMARY

Temperature has a profound effect on the ductility of plain carbon steels, especially within certain narrow temperature ranges known as ductile-brittle transition ranges. The exploration of this relationship provides students with empirical support for intuitive notions, and a great enhancement of the laboratory experience compared to the simple testing of isolated samples to verify properties.

This experimental procedure uses pendulum impact testing of metals at varying temperatures to explore the temperature-toughness relationship. The data from the tests are discussed, and alternatives to the procedures used are presented.

Students conducting these tests must attempt to explain the phenomena they observe, as well as relate test results to published toughness/temperature curves, in order to provide additional reality and meaning to the experience.

INTRODUCTION

The effect of temperature on impact strength can be observed easily in any laboratory with a pendulum type impact tester. The results of such experiments reinforce many fundamental concepts of materials science, including failure mechanisms, mechanisms for plastic deformation, ductile~brittle transition behavior, and basic concepts of energy and work. In addition, this experiment can be a starting point for other experiments relating to impact strength, such as the effect of striking velocity, notch sensitivity, and previous conditioning (hot or cold working, or heat treatments) of the metal samples.

PROCEDURE

Overview

In this laboratory experiment, ten samples of hot rolled steel (SAE/AISI 1020) are prepared for impact testing by machining to ASTM Standards (E23) (Charpy V-Notch sample type), then conditioned at five temperatures (2 samples at each temperature) between -193 and 100 degrees Celcius. A wide range of temperatures can be attained without special equipment through the use of liquid nitrogen, dry ice, an ordinary refrigerator, and a bunsen burner to heat ordinary tap water. If temperatures are chosen over a suitable range, the Transition Range of the metal will be made very clear through the results. In these tests, temperatures of 25, 10, 0, -10, -20, and -30 degrees Celcius were used.

Safety

Pendulum impact test machines can be among the most dangerous of all mechanical test devices in the materials laboratory (ASTM, E23-88). A careful explanation of the dangers of the moving hammer to testers and bystanders in the lab. is essential with this experiment. Students also love to leave the hammer swinging. This must also be discussed, both in terms of danger and interms of wear on the vital pendulum bearing.

Equipment

These tests are designed to require a minimum of specialized test equipment. Most items can be acquired easily and cheaply, but the test lab must have an impact tester capable of breaking all the samples in the study. For ASTM standard Charpy impact tests of SAE/AISI 1020 hot rolled steel, approximately 180 to 210 foot pounds will be required. If a tester with such capacity is not available, suitable modifications of this lab can include study of subsized specimens (note that there is no ASTM standard for subsized metal impact test specimens). Keyhole notch specimens can be used, though they are not usually used for such materials. This type of specimen will require substantially less energy to fracture (mostly below 60 foot pounds). Plastics can be used to study the glass transition temperature, if a much smaller tester is all that is available (range required is from 8 to 16 foot pounds).

Other equipment that is required include:

Beakers
Bunsen burner and ring stand
Water and Methanol
Ice
Salt
Dry ice
Thermometers or thermocouple temperature sensors
Tongs
Prick Punch and (ball peen) hammer
Vernier or dial calipers
A hand lens (5 to 10 power) is handy for macroscopic evaluation of the fracture surfaces.

Temperature Control

Temperature is controlled by using water baths that are either heated with the bunsen burner, or cooled in various combinations of water, ice and water, and ice-water-salt (ice brine). Additional cooling can be obtained by immersing dry ice in methanol. It will be ideal if temperatures between -40 and +100 degrees Celcius can be obtained.

Samples

These tests are usually done with low carbon (SAE 1020) hot rolled steel. It is important to be aware that metal processing (hot or cold) and carbon content have a profound effect on both the shape and location of the transition temperature range. Lower carbon steels show a slightly lower temprature range, and a much sharper drop. The drop for SAE 1008 steel is nearly a vertical line at close to -10 degree C.

Advanced students can explore:

curves for different types of metals
notch sensitivity
unnotched samples
plastics

ASTM requirements for timing must be adhered to while conditioning the samples and moving them from the cooling environment to the test fixture.

RESULTS

Data are compiled in tabular form, then reduced and presented in graphical form. Appropriate representative formulae and calculations are shown. The results are presented without comment about their meaning, to separate data and discussion.

The results from these tests are as follows:

Table 1. Impact Test Results

Specimen	Test Temp.	Energy.	% Lat. Exp.	%Cleav.
1	20	170	11	0
2.	10	142	10	0
3	10	140	10	0
4	0	53	6	41
5	0	57	7	40
6	-10	29	2	65
7	-10	25	3	60
8	-20	30	2	68
9	-20	22	2	65
10	-30	12.5	1	75

Students may be asked to prepare the graphs using computer software such as Excel or Lotus, and this can lead into a discussion of tables, charts, and graphs as means of communication. The graph of these data is presented in figure 1, below.

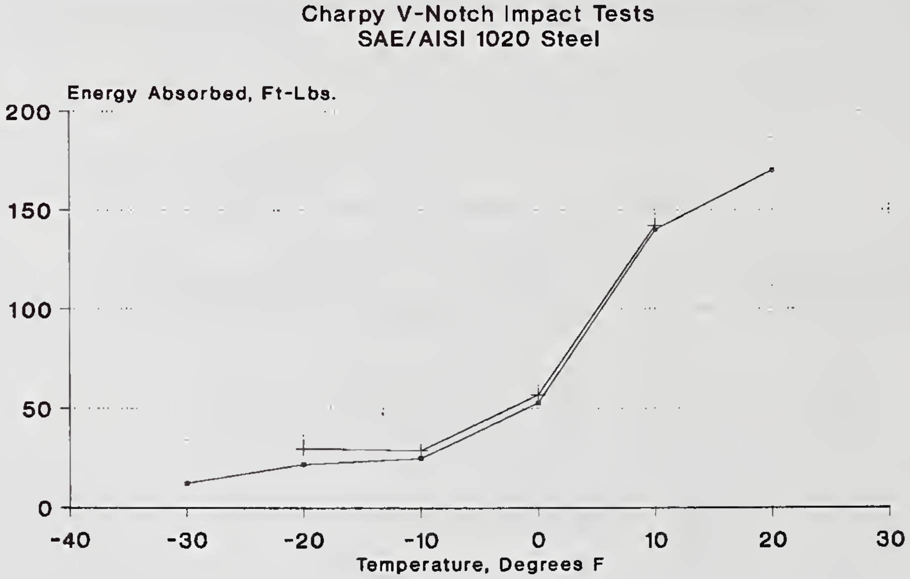
DISCUSSION

These data provide a confirmation of the ductile-to-brittle transformation for low carbon steel, and also show variations in the data that are to be expected from laboratory experiments. These variations can be upsetting to students, just as variations in Gregor Mendel's genetic studies of beans would have been upsetting to religious clergy in his day. After all, if God is perfect, then his created phenomena would be perfect as well.

The discussion of such test results gives students a chance to demonstrate understanding of concepts presented through readings and lectures. Students must explain the results in terms of what was expected and in terms of changes that are observed in the material under the varying conditions. The depth of such discussions will be determined by the level of the materials or metallurgy course in progress. This is a meaningful and satisfying experiment for students, based on direct feedback during and after the experiment is over.

This experiment can be used to lead into or support discussions of plastic and brittle failure mechanisms, and the effect of temperature on atoms in crystal lattice structures. In addition, Professors will enjoy the opportunity to challenge the students to consider the variables of concern to the tests, and what aspects of the test need to be understood simply because they weaken the validity of the test.

Figure 1. Temperature vs. Impact Strength



Machine Design students may also enjoy discussing the inherent weaknesses of this test and the equipment used to conduct it. Some relevant issues include:

friction in the pendulum bearing
windage, or friction between the moving pendulum and the air
friction between the sample and the anvil,
which goes through several stages
throw energy - the energy required to throw the sample after testing

ENHANCEMENTS OF THE METHOD

Needless to say, the results from such tests cannot be taken as highly precise. Variations in pendulum bearing friction, windage, sample friction against the anvils, and throw energy will result in numbers that include all of such errors. Today, technology exists which can eliminate such concerns. The tools are sold by Tinius Olsen as well as several other companies which manufacture or market impact testing equipment, but they can be built on-site by enterprising faculty with the help of electronics students or faculty.

The technique is to attach a Piezo-electric sensor to the hammer, to sense the impact itself and to provide an electronic impulse output that is exactly proportional to the energy absorbed by the sample during fracture. The output is read through a storage oscilloscope equipped with a triggering signal, and the area under the resulting curve can be integrated to determine its magnitude. Implementing such a system would make a suitable senior or graduate project for an enterprising student.

CONCLUDING REMARKS

The samples tested were of SAE/AISI hot rolled steel, tested in as-received condition. Because of the sensitivity of impact test results and of the transition temperature range to small variations in microconstituents and processing, it is advised that the instructor screen each lot of samples before the students begin their work. Also, if technicians are involved in sample preparation, it is important to watch that samples from different shipments or pieces of stock do not get thrown together. This could result in a random scattergram of results, rather than the exciting curve that should be discovered.

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SAN JOSE STATE UNIVERSITY
DIVISION OF TECHNOLOGY

Industrial Materials   Dr. Seth Bates

Lab Experiment: Impact Testing of Carbon Steels at Varying Temperatures

Introduction

The objectives of this experiment are:

1. To learn how to perform the Charpy impact test
2. To study how the impact toughness of a common engineering alloy varies with test temperature
3. To examine how the fracture appearance correlates with impact toughness.

References

ASTM. "Standard Methods for Notched Bar Impact Testing of Metallic Materials", E23-82, Annual Book of ASTM Standards, Author, Philadelphia.

Materials Tested

Ten steel bars supplied by your instructor will be tested at five temperatures between -40 and +100 degrees Celcius. Be sure to look up the published values for these materials. If you cannot find values, consult your instructor.

Procedure

NOTE: Be sure that the area of swing is clear before any use of the pendulum on this machine. It is very dangerous. Stop the pendulum after each single swing!

1. Obtain ten samples from your instructor, and complete their preparation according to ASTM specifications for the 'V Notch' Charpy impact test. Review the ASTM procedure carefully, noting definitions, conditions of testing, and data to be collected and reported.
2. **Due precaution** should be observed in the operation of the pendulum machine. Study the machine and its operation by reading the instruction manual. Study the mechanism for releasing the pendulum, making sure that it latches and locks properly and that you understand its operation.
3. Raise the pendulum to the latch position and note the proper positioning of the sample. Do not insert the sample yet. Set the energy indicator at the maximum scale reading, and allow the pendulum to swing through one full swing to determine the energy in the hammer. Record the reading.
3. Raise the pendulum to the latch position and set the energy indicator at the maximum scale reading again. Take the test specimen from its cooling or heating medium (see Note B); place it in the correct position in the specimen shroud.
4. Release the pendulum and record the energy absorbed. DO NOT allow the pendulum to swing more than once!

Notes:

A. Safety precautions should be taken to protect personnel from the swinging pendulum, flying specimen fragments, and hazards associated with specimen cooling and heating media.

B. While placing test specimens in the machine, **<u>be sure</u>** that the pendulum is latched in the safety position.

C. Should any specimen jam in the machine, disregard the results and examine the machine thoroughly for damage or maladjustment, which would affect its calibration.

Results

1. Inspect each broken specimen and sketch its fracture surface. Observe and calculate the percent cleavage (brittle fracture) and shear (ductile fracture).

2. Compare the obtained impact energies with respect to the temperature changes, and graph the results. Compare these results to published curves for this alloy.

3. Report measurements as indicated by ASTM guidelines.

4. Explain your conclusions regarding the behavior of this metal at different temperatures.

FIBER REINFORCED COMPOSITE MATERIALS

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Key Words

Composite, anisotropy, fibers, matrix, reinforcement, shear distortion

Prerequisite Knowledge

This laboratory requires a general knowledge of the concepts and parameters involved with the elastic behavior of materials, namely, stress, strain, elastic modulus, and Poisson's ratio.

Objectives

In this laboratory, data will be collected in order to accomplish the folowing analyses pertaining to the characterization and elastic behavior of fiber reinforced composite materials.

1) Calculate the fiber volume fraction, V_f, for a composite sample from sections oriented **a)** perpendicular to the fibers and **b)** 45° to the fibers. Compare these two values and account for any differences.

2) Calculate the longitunal and transverse moduli, E_L and E_T, from data provided for a nylon reinforced rubber composite and compare these values to those observed in the laboratory.

3) Calculate the elastic modulus, E_1, at different angles (i.e. θ=15°, 30°, 45°, 60°, and 75°) and plot the values as a function of θ. On the same graph, plot the theoretical curve from the equation given and compare the two.

4) Obtain the major Poisson's ratio ν_{12} measured at the different angles and plot the values along with a theoretical curve for comparison.

5) Plot the observed values of the shear coupling modulus, β, as a function of θ.

Background

This laboratory is concerned with the mechanical behavior of fiber reinforced composite materials. These types of materials are being used extensively in the aerospace industry and in many other applications where high strength and low weight are the primary design criteria. Generally, these materials consist of:

1) **Fibers -** long cylindrically shaped elements of high strength and/or high modulus materials such as graphite, glass, Kevlar®, boron, tungsten, aluminum oxide, etc., with diameters ranging from 6 microns to 100 microns (1 micron = 10^{-6}m).

2) **Matrix -** material which binds the fibers and transmits loads to the fibers through chemical or mechanical bonds, and usually has lower strength and modulus than the fiber material. The most common matrix materials in use today are polymers such as thermosetting epoxy resins and low-melting metals such as aluminum. Other matrix materials such as thermoplastics of considerable toughness and heat resistant ceramics are being developed for composite applications.

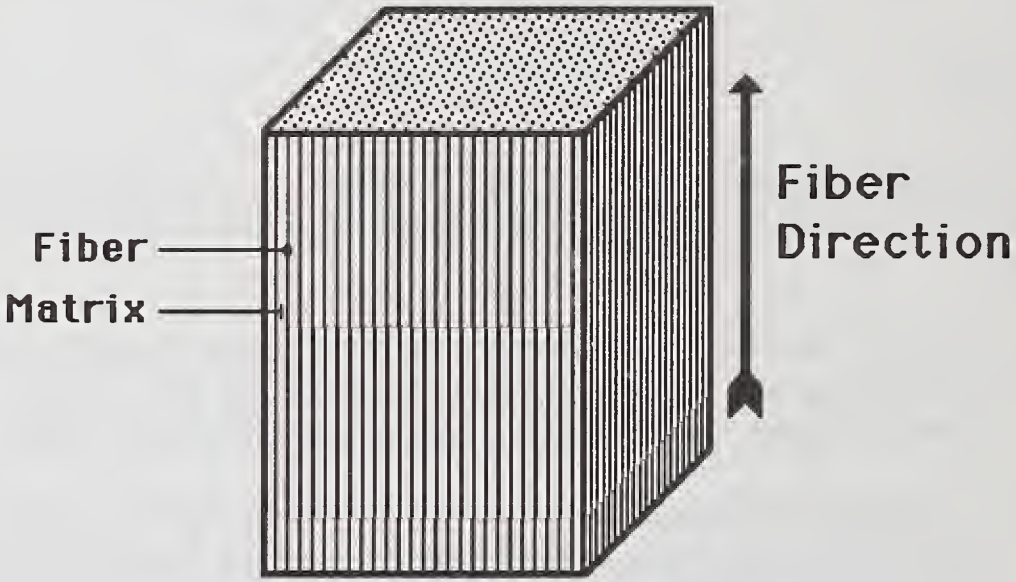


Fig. 1 Typical section of a fiber reinforced composite material with unidirectionally oriented continuous fibers.

These composite materials (i.e. materials in which both constituents retain their identities when "mixed" together) differ from most engineering materials in that their mechanical properties are highly dependent on the direction in which the loads are applied. Materials such as this are referred to as being *anisotropic* (just like a piece of wood). Therefore, it is of considerable importance to the engineer to have a feel for this directional dependence so that these materials may be utilized properly and safely.

DETERMINATION OF FIBER CONTENT

The fiber content of a particular composite is generally expressed as the volume fraction of fiber and is denoted V_f. Most structural composites contain between 50 to 60 percent fiber ($0.5 < V_f < 0.6$) while composites in other applications may contain less.

One method utilized for determining the fiber content is called the *point count method.* This consists of the placement of a grid or an array of points over a photomicrograph of a polished cross section of the composite material. Note that the points must be small in comparison to the fiber diameter for an accurate measurement. By counting the number of points or intersections which fall on the fibers and dividing by the total number of points, the area fraction A_f is calculated.

Referring to figure 2, the number of points falling within fiber area is around 155 (not everyone will count the same amount given the same picture). The total number of points in the array is 630 (exactly) so the area fraction of fiber in this example is $155/630 \approx 0.25$.

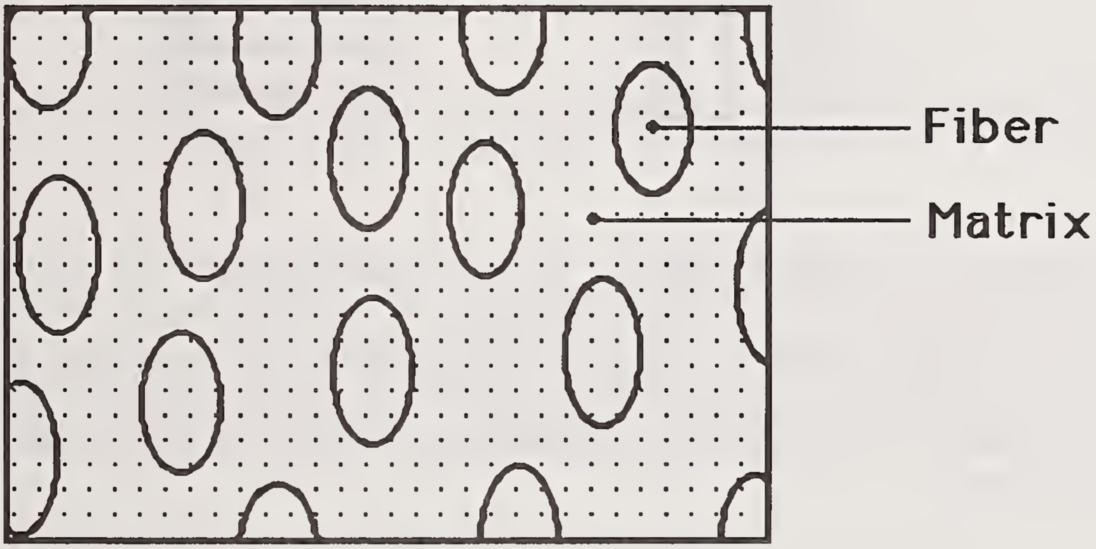


Fig. 2 Example of the point count method for a fiber-reinforced composite.

Notice that the fiber patches are elliptical; does this mean that these fibers are not circular in cross section? Could they be circular but cut at an angle? Can the angle at which the fibers are cut change the area fraction of fiber obtained by the point count method?

Consider the case where the fibers are cut at a right angle to their lengths. The patches would be circular and the area fraction could be obtained as above. Clearly, this area fraction A_f is the same as the volume fraction V_f. Furthermore, it does not matter at what angle the fibers are cut; even if the fibers are randomly oriented, such as in a composite containing chopped fibers, the point count method gives, directly, the volume fractions of the constituents.

In this lab, however, the total fiber area will be determined by counting the number of fibers intersecting the surface of the sample and multipying by the area of the fiber. This is possible due to the relatively large diameter of the nylon reinforcement and low fiber content. This method will be employed on one surface oriented perpendicular to the fibers and another oriented 45° to the fibers.

ELASTIC BEHAVIOR

As was previously mentioned, the mechanical behavior of fiber reinforced composite materials is highly dependent on the direction in which the particular property is measured. For instance, the elastic modulus along the fibers, $\mathbf{E_L}$, is drastically different from the elastic modulus transverse to the fibers, $\mathbf{E_T}$. First order approximations of these moduli can be caluclated from the elastic constants of the constituent materials by considering the *isostrain* and *isostress* models shown in Figure 3:

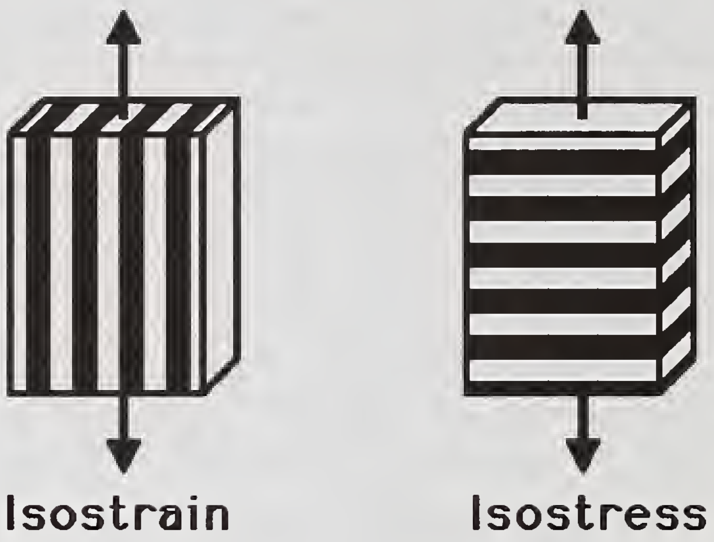


Fig. 3 Geometry of idealized unidirectional composite materials.

The equation for the longitudinal modulus (along the fibers) can be derived from the isostrain model as:

$$E_L = E_f V_f + E_m V_m$$

where,

E_f = elastic modulus of the fibers

V_f = volume fraction of fiber in the composite

E_m = elastic modulus of the matrix material

V_m = volume fraction of matrix = $(1 - V_f)$

This equation is known as the *rule of mixtures* and implies that the contribution of a constituent is directly proportional to its volume fraction. Clearly, the equation for the density of the composite would be of the same form.

The equation for the transverse modulus (perpendicular to the fiber direction) can be derived from the isostress model as:

$$\frac{1}{E_T} = \frac{V_f}{E_f} + \frac{V_m}{E_m}$$

This equation is known as the *inverse rule of mixtures*. Both equations are shown in Figure 4 to illustrate the effect of fiber content on the elastic moduli. Notice that the transverse modulus is not appreciably increased beyond the modulus of the less stiff constituent, the matrix, at the fiber contents usually encountered in engineered composites.

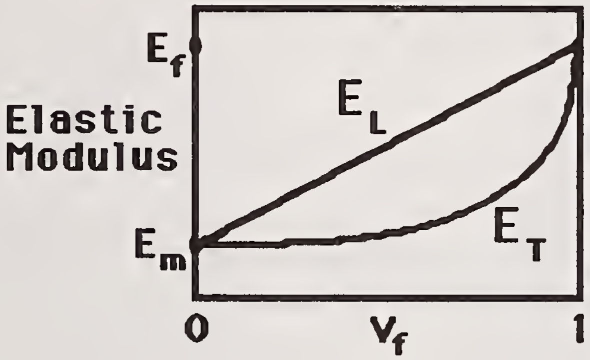


Fig. 4 Elastic modulus versus fiber volume fraction for the isostress and isostrain models.

We can refer to the directions **L** and **T** as the *material axes* and, if the composite is stressed in either of these directions, the corresponding strains can be calculated (on the left in Figure 5). However, if a stress is applied at an acute angle, θ, to the fibers (on the right in Figure 5), the elastic response along the *loading axes* (1 and 2) can be calculated from the properties measured along the *material axes* (L and T).

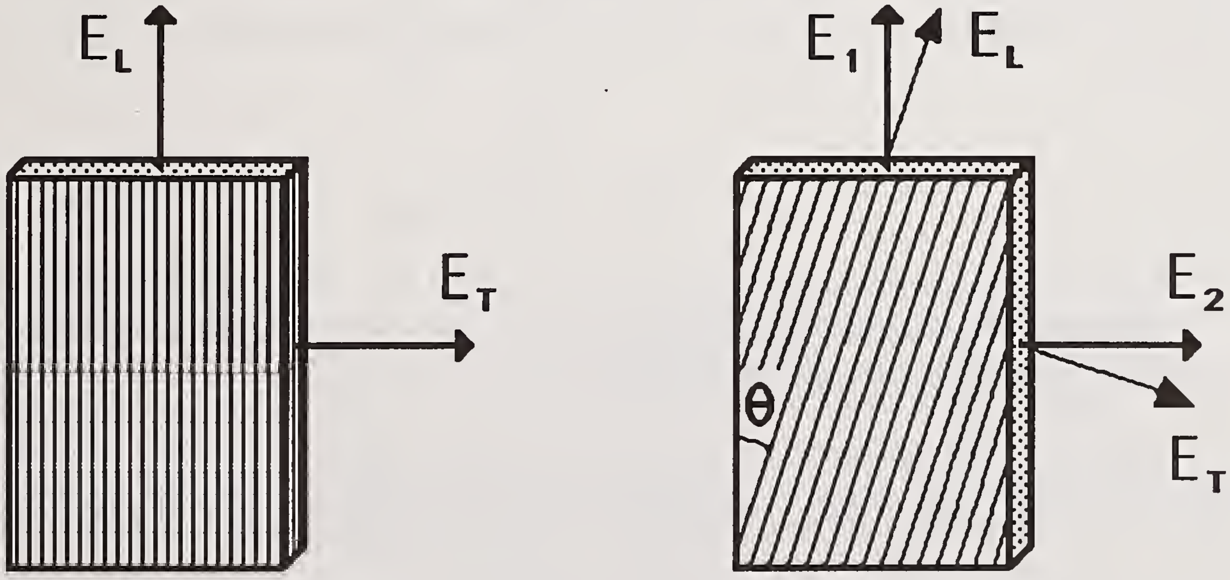


Fig. 5 Illustration of material (L and T)and loading (1 and 2) geometries.

$$E_1 = E_L\left[\cos^4\theta + \frac{E_L}{E_T}\sin^4\theta + \frac{1}{4}\left(\frac{E_L}{G_{LT}} - 2\nu_{LT}\right)\sin^2 2\theta\right]^{-1}$$

$$E_2 = E_L\left[\sin^4\theta + \frac{E_L}{E_T}\cos^4\theta + \frac{1}{4}\left(\frac{E_L}{G_{LT}} - 2\nu_{LT}\right)\sin^2 2\theta\right]^{-1}$$

Notice that in the above equations the material parameters needed include θ, G_{LT}, and ν_{LT}, as well as E_L and E_T. The value of E_L can be calculated from the rule of mixtures equation given previously and the value obtained will be satisfactorily close to reality (even though the composite is made of fibers in a matrix instead of alternating plates as utilized in the isostrain model). However, the value for E_T calculated from the inverse rule of mixtures equation would not be close to reality because the isostress model (stacked plates) is an oversimplification of a fiber composite loaded transversely. Similarly, equations for G_{LT} (the shear modulus obtained when a torque is applied in the plane perpendicular to the fiber direction) do not always give accurate values; therefore, E_T and G_{LT} should be measured in the laboratory. The major Poisson's ratio, ν_{LT} (a longitudinal stress causes a strain in the transverse direction), can be calculated reliably from a rule of mixtures type equation just like E_L.

$$\nu_{LT} = \nu_f V_f + \nu_m V_m$$

What about ν_{TL} (a transverse stress causing a strain in the longitudinal direction)? Would you expect it to be equal to ν_{LT}? It is indeed different but easily calculable as follows:

$$\nu_{TL} = \frac{E_T}{E_L}\nu_{LT}$$

The transformation of ν_{LT} (along the material axes) into ν_{12} (along the loading axes) is given in the following equation:

$$\nu_{12} = \frac{E_1}{E_L}\left[\nu_{LT} - \frac{1}{4}\left(1 + 2\nu_{LT} + \frac{E_L}{E_T} - \frac{E_L}{G_{LT}}\right)\sin^2 2\theta\right]$$

One of the most important phenomena which occur upon the off-axis loading of unidirectionally reinforced fiber composites is the production of a shear strain from a purely tensile stress state. As illustrated in Figure 6, if a 90° reference angle is enscribed on the sample (prior to loading), this angle will change as the sample is loaded uniaxially, indicating the existence of a shear strain. This clearly would not happen with an isotropic material (a square would become a rectangle but angles would not change) or on a composite sample loaded along the material axes (θ=O° or θ=90°); but when θ is between 0° and 90°, a shear strain usually occurs. It may be positive, or negative, or zero, depending on the value of θ, the elastic properties of the consitituents, and the fiber content of the composite.

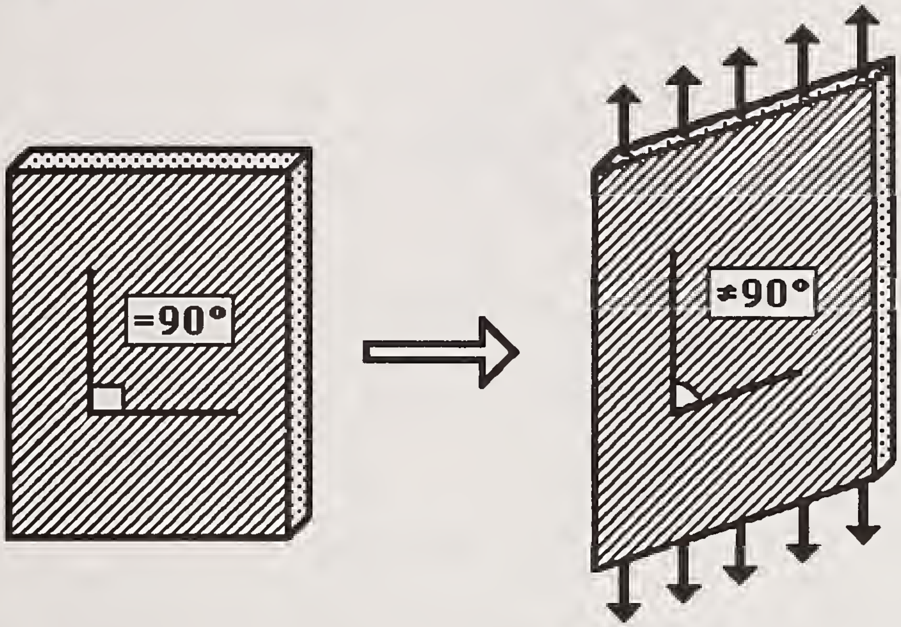


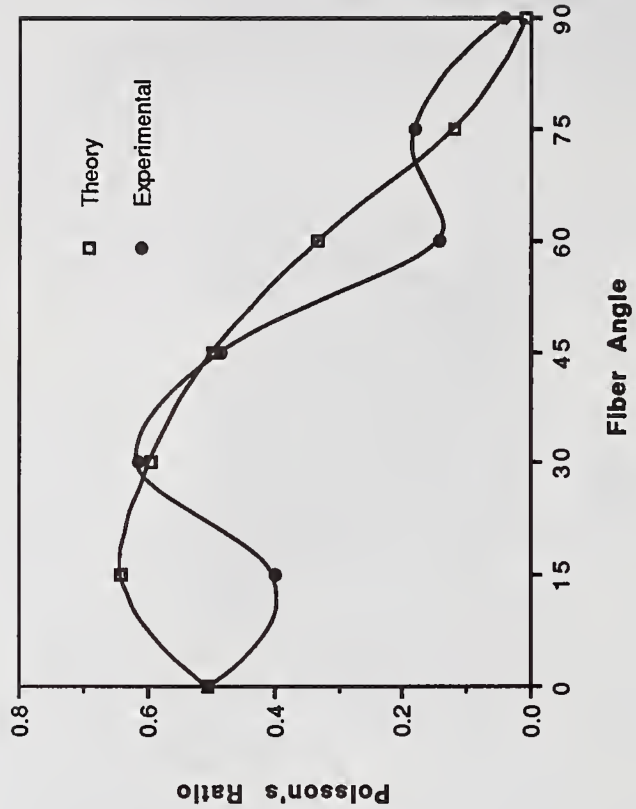
Fig. 6 Illustration of shear strain produced by tensile loading of a fiber composite.

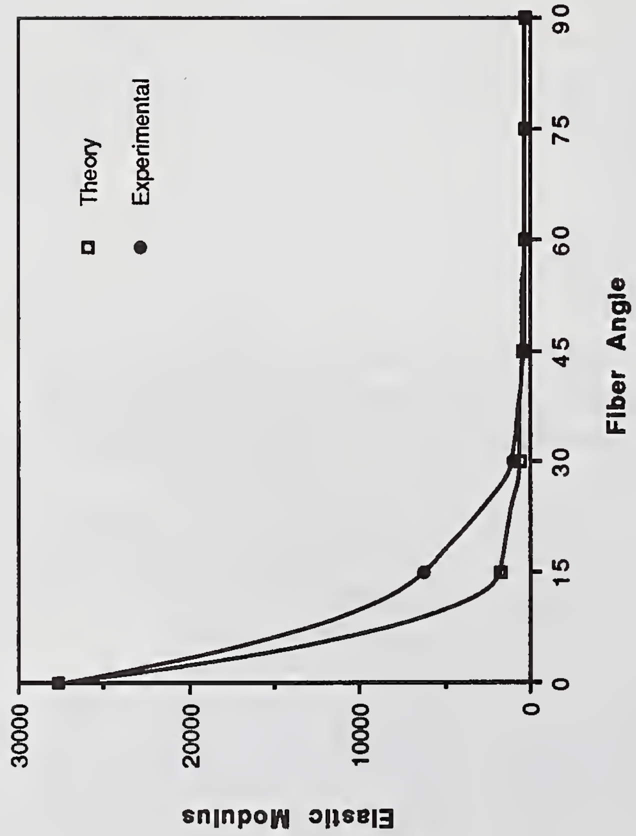
The amount of change in the 90° angle (measured in degrees) that occurs upon the stressing of the composite is the shear strain γ_{12}. For the purpose of our simplified experiment, we will define a *shear coupling coefficient*, β, to relate the the applied normal stress, σ_1, to the resulting shear strain as follows:

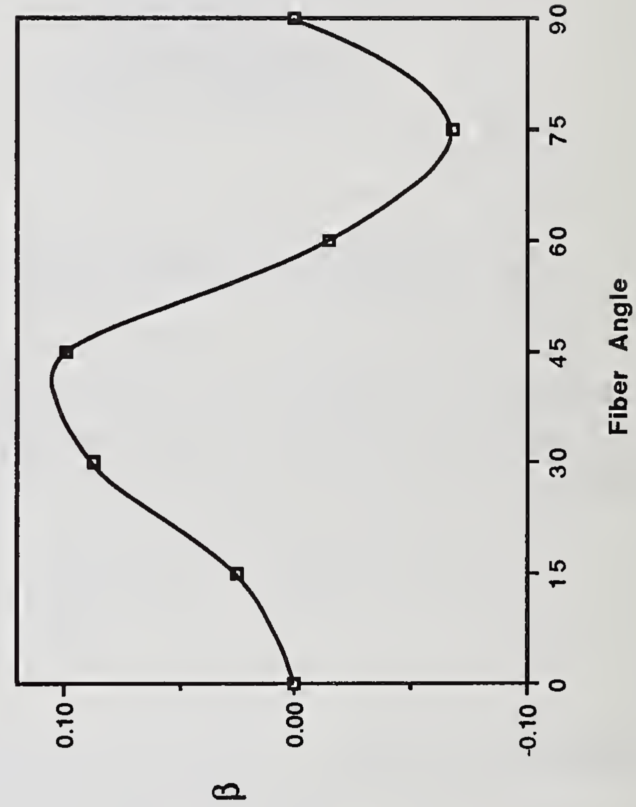
$$\beta = \frac{\gamma_{12}}{\sigma_1}$$

Instructor Notes

The composite samples currently used for this experiment consist of 7% by volume of nylon cord in a cured rubber matrix. The raw material was originally obtained for a senior project conducted by Mr. Yusef Fahmy. His investigation laid the groundwork for this laboratory exercise and his assistance was greatly appreciated. We cannot specify a current vendor for such a nylon/rubber composite system, but can only suggest contacting a tire manufacturer for uncured, unidirectionally reinforced material. A similar experiment could be performed with fiberglass/epoxy or graphite/epoxy, but the deflections would only be measureable with strain gages. Other commercially available reinforced sheet materials could suffice as long as the extensibility was high enough to permit hand measurement of strain and shear distortion. For this exercise, the students were able to measure the elongations (and transverse contractions) with dial calipers and the shear distortion angle was measured with a protractor. Samples of the results are given on the proceeding page.


Theory
Experimental
Poisson's Ratio
0.8
0.6
0.4
0.2
0.0
0
15
30
45
60
75
90
Fiber Angle


Theory
Experimental
Elastic Modulus
30000
20000
10000
0
0
15
30
45
60
75
90
Fiber Angle


β
0.10
0.00
-0.10
0
15
30
45
60
75
90
Fiber Angle

DIELECTRIC BEHAVIOR OF SUPERCONDUCTORS AT MICROWAVE FREQUENCIES

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KEYWORDS

Superconductors; Microwave Resonant Cavity; Dielectric Constant; and Dielectric Relaxation.

OBJECTIVES

1. To learn to operate a microwave spectrometer.
2. To tune the microwave resonant cavity at a certain frequency and get resonant signal and frequency markers
3. To take data: Frequency shifts and Q-changes as a function of temperature as the superconductor sample under investigation goes through its transition temperature.
4. To analyze the data using a computer program.

SUMMARY

A cylindrical microwave resonant cavity in TE_{011} mode was used as a probe to study the dielectric behavior of two superconductors: $Y_iBa_2Cu_3O_x$ and $Bi_2Sr_2Ca_2Cu_3$ at microwave frequency of 9.2 GHz. The microwave cavity was used in a self designed microwave spectrometer with a very efficient method of cooling. Dielectric relaxation of these super-conductors was studied by cooling the sample to the superconducting state. The frequency shifts and the Q-changes of the cavity were monitored as the cavity temperature was varied.

INTRODUCTION

Dielectric properties of solids, liquids and gases have been studied by different investigators (ref. 1-6). The present investigation involves the use of a microwave resonant cavity to study the dielectric response of two different samples of superconductors: $Y_iBa_2Cu_3O_x$ and $Bi_2Sr_2Ca_2Cu_3$.

The resonant circuit technique is a standard technique and has been used by several workers. Bussey et al (ref. 7) and S. Chattarjee (ref. 8) used a circular cavity resonating in TE_{010} mode with capillary tube specimen and tuning plunger. C. H. Collie et al (ref. 9) made use of a circular cavity resonating in TM_{01} mode with a capillary tube specimen. K. H Hong and J. A. Roberts (ref. 10) used a cylindrical cavity resonating near 10 GHz in TM_{010} mode to determine the dielectric properties of liquids and solids. Dahiya et al (ref. 11 and 12) studied the dielectric behavior of various polar and non-polar compounds using a microwave resonant cavity in TE_{011} mode.

Dielectric behavior of these superconductors was studied between 80 K and 240 K while their superconducting temperatures were between 90 K and 110 K. The microwave resonant cavity in this investigation was cooled by flushing very cold nitrogen gas around the cavity. The thermocouple was attached to the sample inside the cavity which in turn was computerized by using microcomputer interfacing techniques (ref. 13). The powdered sample of super-conductor was placed into the resonant cavity through a capillary tube attached to a micrometer drive which allowed a different mass of the sample into the cavity. Dielectric behavior of these superconductors was studied by introducing a fixed length of the sample into the resonant cavity and then the cavity was cooled to achieve temperatures lower than the superconducting temperatures. Slater's perturbation equations were used to calculate the frequency shifts and Q-changes of the cavity.

THEORY

In a static field the displacement vector $\vec{D}$ and the electric field vector $\vec{E}$ are proportional to each other.

$$\vec{D} = \epsilon\vec{E} \tag{1}$$

where ϵ is the dielectric constant.

When a dielectric is subjected to an alternating field the orientation of the dipoles, and hence the polarization, will tend to reverse every time the polarity of the field changes. As long as the field remains low ($<10^6$ Hz), the polarization follows the alternations of the field without any significant lag and the permittivity is independent of the frequency and has the same magnitude as in a static field. When the frequency is increased the dipoles will no longer be able to rotate sufficiently rapidly so that their oscillations will begin to lag behind those of the field. As the frequency is further raised the permanent dipoles will be completely unable to follow the field and the contribution to the static permittivity (ϵ) from this molecular process, the orientational polarization ceases and this usually occurs in the rf range (10^6 - 10^{11} Hz).

For frequencies in the infrared (10^{11} - 10^{14} Hz) range there is no contribution to ϵ from atomic or ionic polarization and the only polarization that contributes to ϵ is the electronic polarization. Therefore, the permittivity of a dielectric material decreases with increasing frequency. This process is known as dielectric dispersion.

At very high frequencies, E, D and ϵ become complex quantities given by

$$\hat{E} = E_o e^{j\omega t} \tag{2}$$

$$\hat{D} = D_o e^{j(\omega t-\delta)} \tag{3}$$

and

$$\hat{\epsilon} = \epsilon' - j\epsilon'' \tag{4}$$

where ϵ' and ϵ'' are the real and imaginary parts of the dielectric contant.

Since

$$\hat{D} = \hat{\epsilon}\hat{E} \tag{5}$$

$$\hat{\epsilon} = \frac{\hat{D}}{\hat{E}} = \frac{Do\ e^{j(\omega t-\delta)}}{Eo\ e^{j\omega t}} \tag{6}$$

or

$$\hat{\epsilon} = \frac{D_o}{E_o} e^{-j\delta} \tag{7}$$

or $$\hat{\epsilon} = \frac{D_0}{E_0} (\cos\delta - j \sin\delta) \tag{8}$$

Therefore, using eq. (4) $$\epsilon' = \frac{D_0}{E_0} \cos\delta \tag{9}$$

$$\epsilon'' = \frac{D_0}{E_0} \sin\delta \tag{10}$$

The dielectric loss at a certain frequency is indicated by tanδ given by equations (9) and (10) as follows

$$\tan\delta = \frac{\epsilon''}{\epsilon'} \tag{11}$$

The frequency shifts and the Q-changes of a perturbed cavity are functions of the dielectric properties of the cavity media and are given by Slater's perturbation equations (ref. 12).

$$\frac{\Delta f}{f_0} = - \frac{\epsilon' - 1}{2} \int \frac{\vec{E}_s \cdot \vec{E} \ dv}{\vec{E} \cdot \vec{E}_a \ dV} \tag{12}$$

and $$\Delta(1/Q) = \epsilon'' \int \frac{\vec{E}_s \cdot \vec{E} \ dv}{\vec{E} \cdot \vec{E}_a \ dV} \tag{13}$$

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, and v and V are the volumes of the sample and cavity respectively.

By defintion

$$Q = \frac{f_0}{W} \tag{14}$$

where f_0 is the resonant frequency of the system and W is the frequency separation in Hertz, of the resonance half-power points.

Therefore, we have the following relation (with modulation correction)

$$\Delta(1/Q) = \frac{\sqrt{3}\Delta W}{f_o} \tag{15}$$

The real and imaginary parts of the dielectric constant are further related to the relaxation time (τ) using Debye's equations (ref. 14) as follows.

$$\frac{\epsilon_s - \epsilon'}{\epsilon''} = \omega\tau \tag{16}$$

where $\omega = 2\pi f_o$, and ϵ_s is the value of the dielectric permittivity for static field.

EXPERIMENTAL METHOD

Materials

Superconductors $Y_1Ba_2Cu_3O_x$ and $Bi_2Sr_2Ca_2Cu_3$ in the powdered forms. The transition temperatures for these superconductors were around 95 K and 106 K respectively.

Equipment and Supplies

i) Western electric 2K25 klystron
(ii) Hewlett Packard 715 A klystron power supply
(iii) Webster G40P5158 directional coupler
(iv) OKI 8-11 GHz wavemeter
(v) Microwave resonant cavity in the TE_{011} mode designed in the physics workshop at Southeast Missouri State University
(vi) Hewlett Packard x 485B tunable detector mount
(vii) Kenwood R-1000 radio receiver
(viii) Sencore FC71, 10 Hz - 1 GHz frequency counter
(ix) Hewlett Packard 612A UHF (450 MHz-1250 MHz) signal generator
(x) Tektronix 533A dual channel oscilloscope
(xi) 31 KHz bandpass amplifier designed in the physics workshop at Southeast Missouri State University
(xii) Waveguides in the x-band of frequency (8-11 GHz)
(xiii) Waveline attenuator for varying microwave power
(xiv) Omega 6125A digital thermometer with a thermocouple attached
(xv) Large dewar to hold about 25 liters of liquid nitrogen for

cooling process.

(xvi) Thermal bath with copper coils to circulate cold dry nitrogen gas around the sample placed in the microwave resonant cavity.

Procedure

A microwave spectrometer in the x-band of frequencies was put together by collecting different components of the spectrometer and this system is relatively very inexpensive as compared to the ESR spectrometer. The details of the spectrometer have been discussed elsewhere (ref. 12). A block diagram of the spectrometer is shown in Fig. 1. A microwave signal of 9.2 GHz is produced by a 2K25 reflex kylstron. The signal is modulated by applying a horizontal sawtooth from the repeller of the klystron to give a broader frequency response of the microwave power. This signal is transmitted through a waveguide as co-ax cables cannot be used because of very high inductive reactance at that frequency. The directional coupler divides this signal in two parts. A part of the signal goes to the microwave resonant cavity and the reflected signal is detected by a microwave diode and the amplified signal looks like a butterfly. The other part of the signal from the directional coupler is mixed with a standard frequency signal to produce a series of markers. A radio receiver is used to detect these markers which in turn are used to study the frequency shifts and the width changes of the signal produced by the resonant cavity as the sample is introduced in the cavity. The frequency shifts and width changes or Q-changes of the signal are related to the real and imaginary parts of the dielectric constant through the Slater's perturbation equations 12 and 13.

The powdered samples of superconductors were provided by Superconductive Components, Inc., Columbus, Ohio, and the purity level was 99.9%. Each sample under investigation was taken into a capillary tube that was sealed with a fine thermocouple wire attached to the sample. It was then placed along the symmetry axis of the cavity via a micrometer drive. The cavity resonating in TE_{011} mode was placed into a thermal bath. A block diagram of the microwave resonant cavity is shown in Fig. 2. The cooling technique used in this investigation is very efficient and has been discussed elsewhere (ref. 12). Each sample of the superconductor was cooled below its superconducting state and the temperature allowed to rise in suitable steps with resonant frequency shifts Δf and microwave loss parameter $\Delta(1/Q)$ measured for each temperature until sufficient data were obtained to determine the behavior of the dielectric response of the system over the transition region. At each reading, sufficient time was allowed to elapse so that the sample and cavity came to temperature equilibrium.

RESULTS AND DISCUSSION

Microwave absorption technique has become very sensitive in explaining the properties of high T_c superconductors (ref. 15,16). The resonant cavity technique employed in this investigation successfully monitored the dielectric relaxation phenomenon in superconductors: $Y_iBa_2Cu_3O_x$ and $Bi_2Sr_2Ca_2Cu_3$. Each sample of the superconductor was found to show a dramatic behavior in the dielectric properties near the super-conducting temperature. This behavior can be seen in Figs. 3-6 with Figs. 3 and 4 showing the behavior of microwave loss $\Delta(1/Q)$ and frequency shift $\Delta f/f_o$ vs. temperature for the superconductor sample $Y_iBa_2Cu_3O_x$, and Figs. 5 and 6 showing the same behavior for the superconductor sample $Bi_2Sr_2Ca_2Cu_3$.

The microwave resonant cavity technique employed in this investigation clearly identifies the superconducting temperature. The microwave loss below the superconducting temperature is very interesting. According to the Meissner effect, the microwave frequency should be greater when the sample is in the superconducting state because the magnetic component of the microwave field applied is excluded from the sample. These measurements of microwave absorption were made in the absence of an external magnetic field. When a superconductor is in the normal state (above the superconducting temperature), this thickness is much greater than the skin depth. Therefore, in the normal state the microwave field does not penetrate the sample and only a fraction of it is absorbing the microwaves.

The detailed results of the microwave loss measurements and the frequency shifts in these superconductors will be presented elsewhere as a continuation of this experiment. This experiment is presented here as a technique to monitor the dielectric relaxation mechanism in these and other supercondcutors and to identify their superconducting temperatures.

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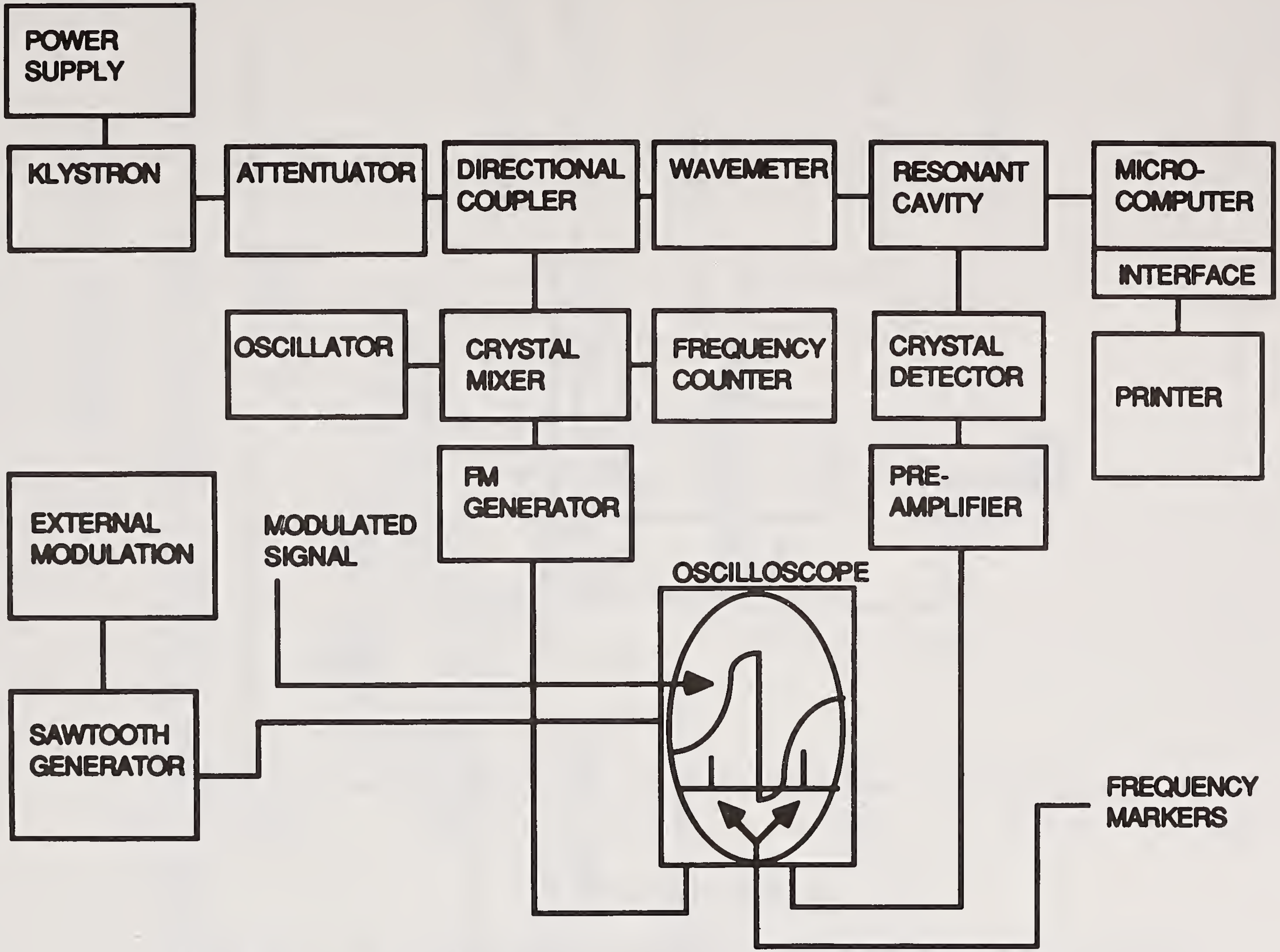
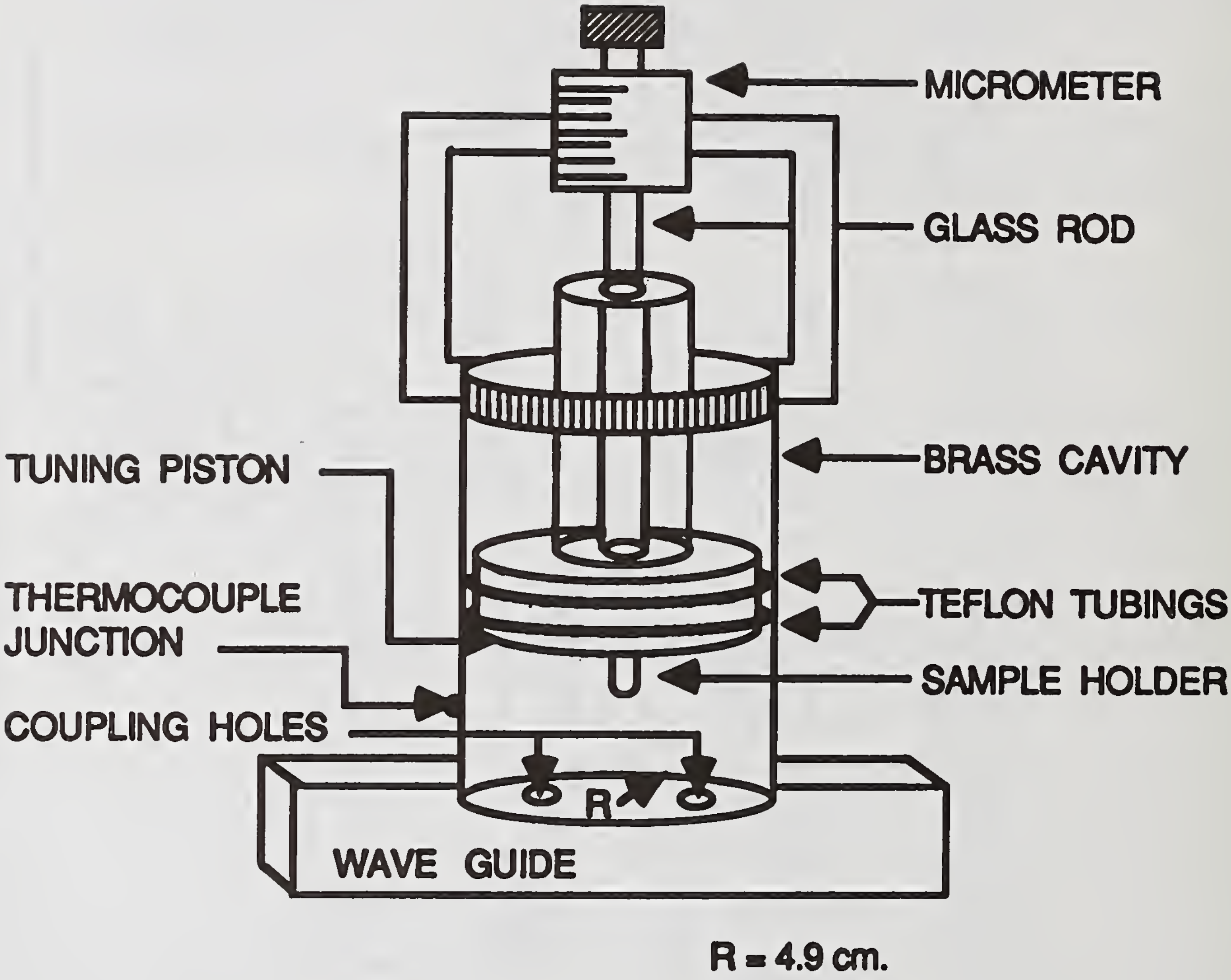
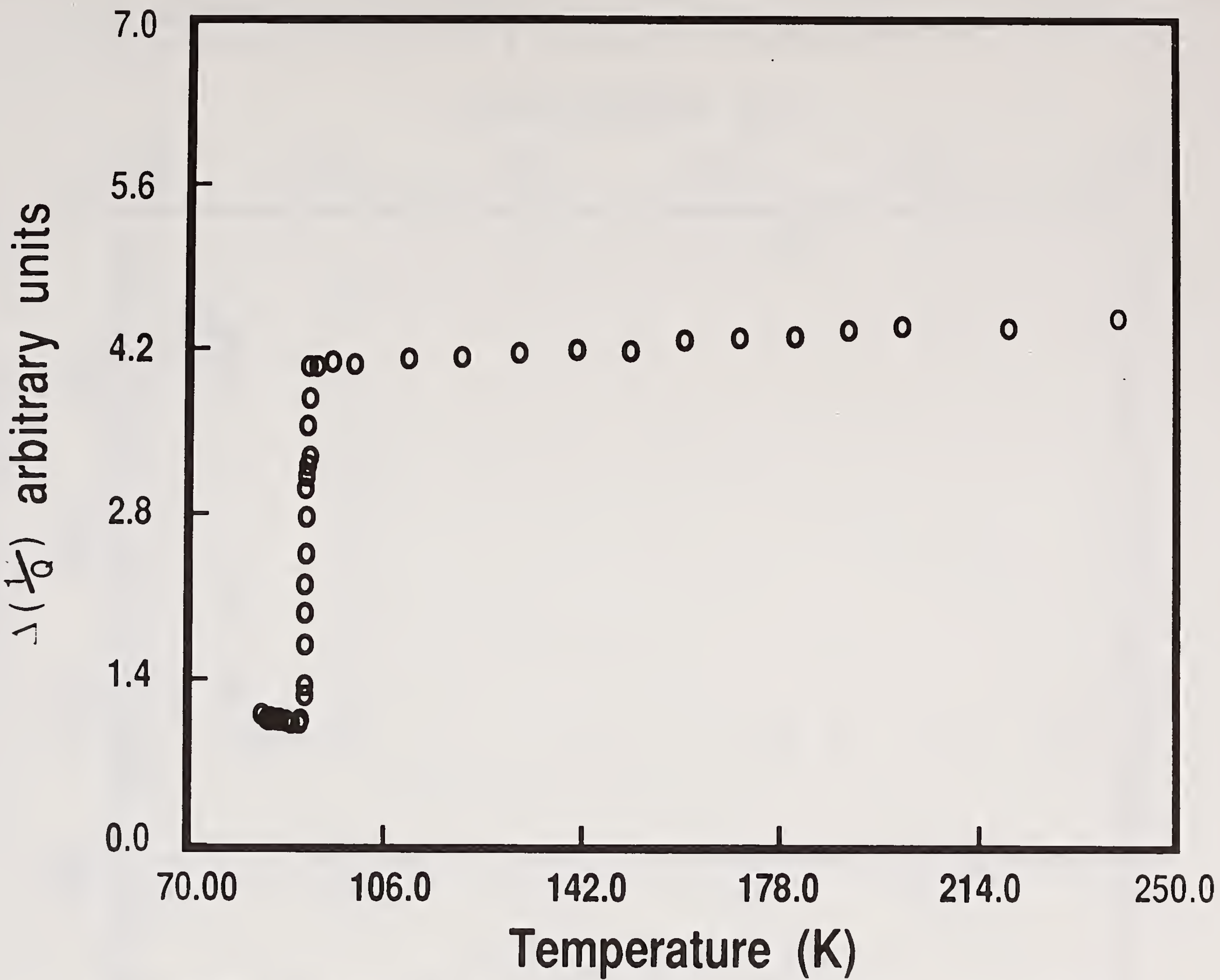


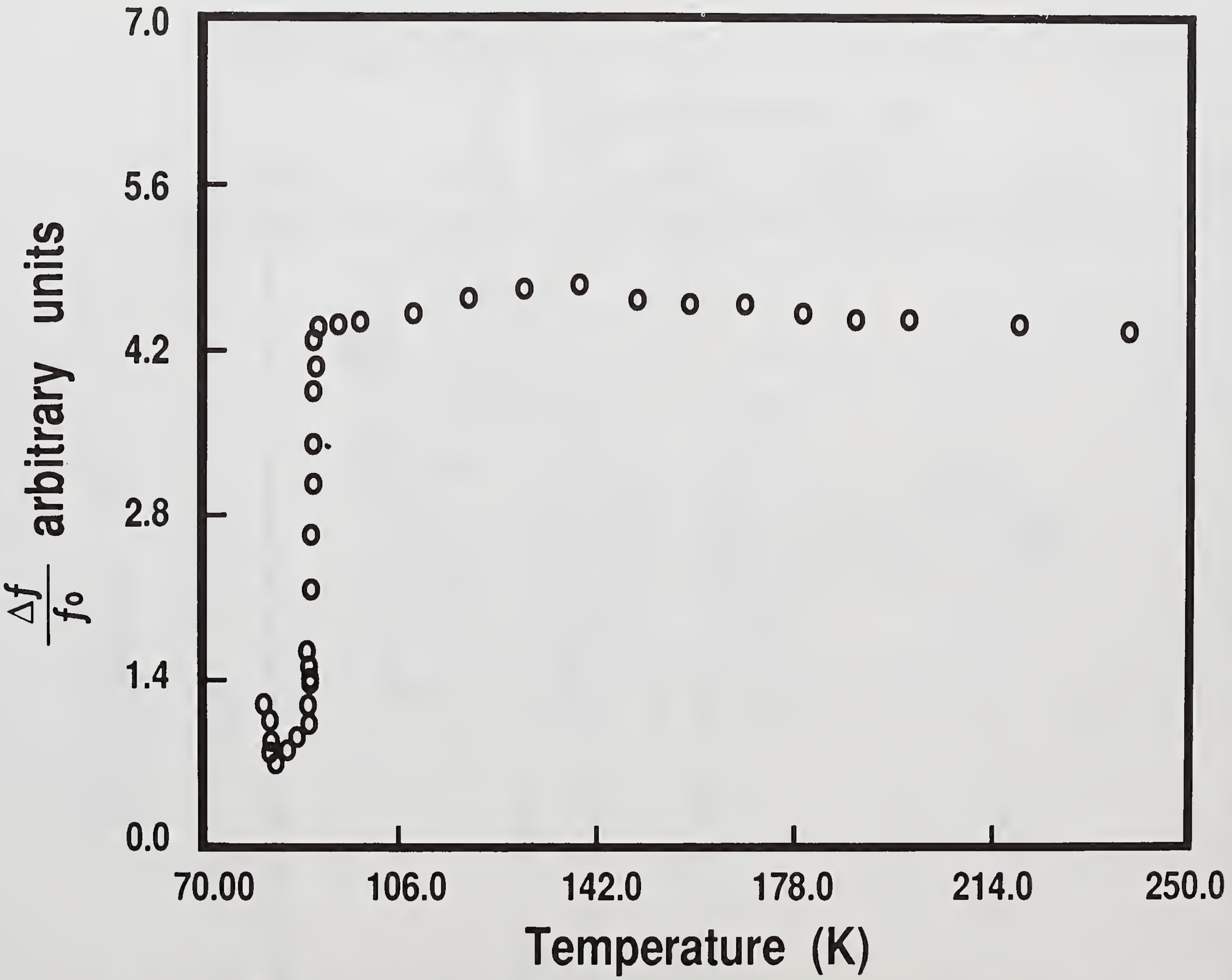
1. Block diagram of the microwave spectrometer used in this investigation.



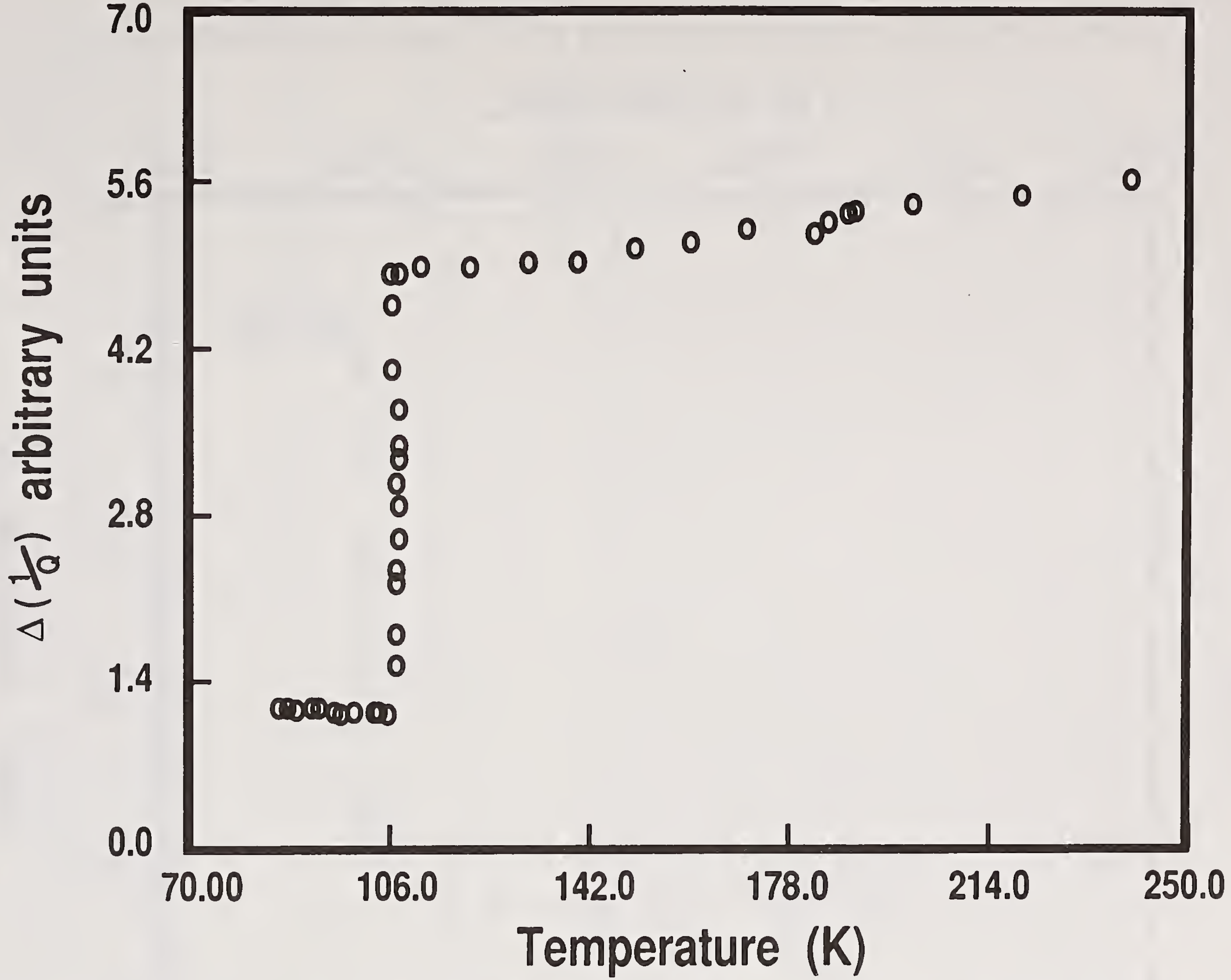
2. Block diagram of the tunable microwave resonant cavity operated in TE_{011} mode used in this investigation.



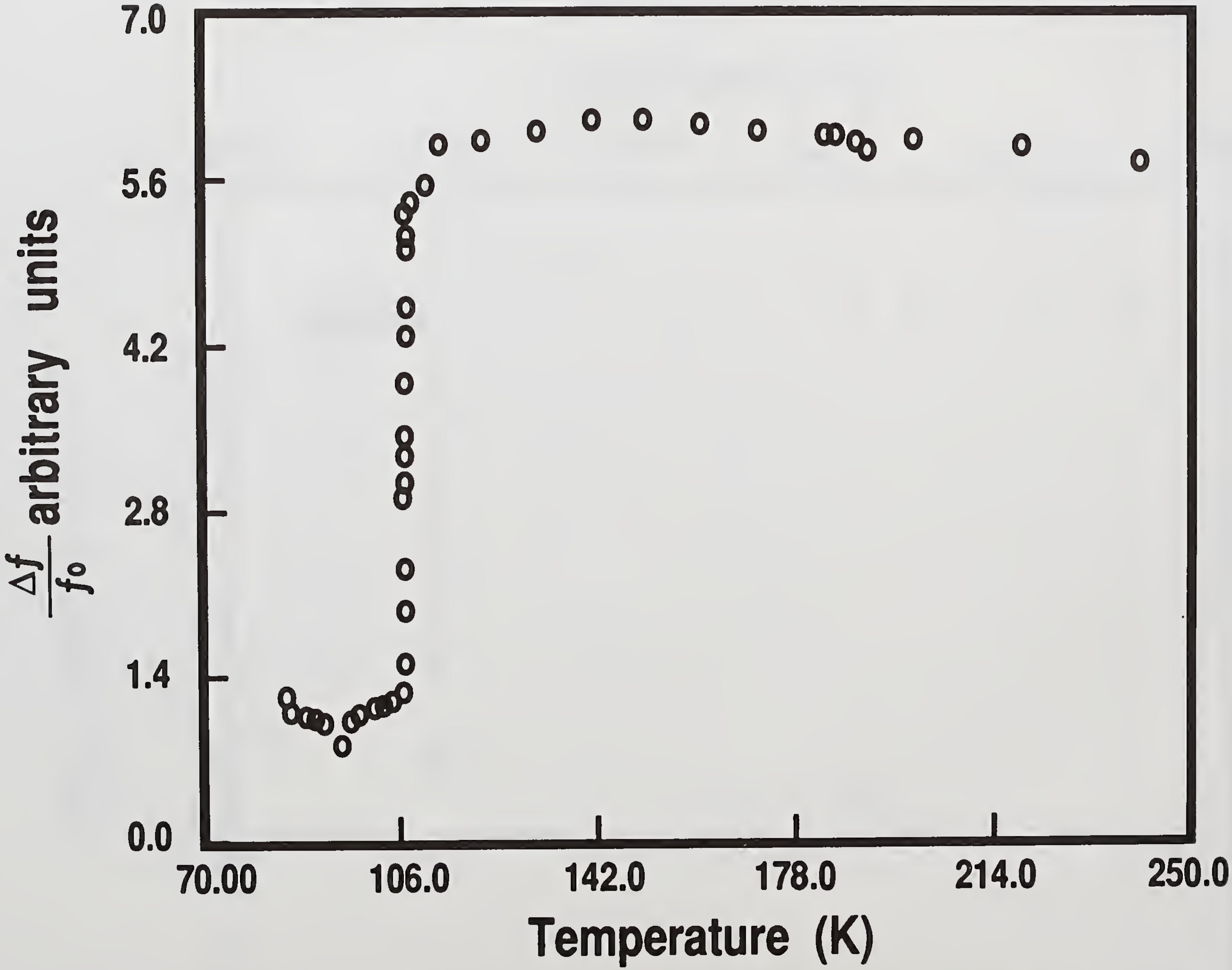
3. Microwave loss Δ(1/Q) as a function of temperature for a sample of $YiBa_2Cu_3O_x$ at 9.2 GHz.



4. Frequency shift $\Delta f/f_0$ as a function of temperature for a sample of $YiBa_2Cu_3O_x$ at 9.2 GHz.



5. Microwave loss Δ(1/Q) as a function of temperature for a sample of $Bi_2Sr_2Ca_2Cu_3$ at 9.2 GHz.



6. Frequency shift $\Delta f/f_o$ as a function of temperature for a sample of $Bi_2Sr_2Ca_2Cu_3$ at 9.2 GHz.

IT'S HARD TO TEST HARDNESS

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ABSTRACT

Hardness is essentially a mechanical property of uniform materials, a surface indicator of other properties. It is hard to define, hard to correlate different tests, and hard to compare different materials. Nevertheless, hardness is broadly considered as "resistance to penetration, elastic and/or plastic." For metal alloys, commercial tests typically measure plastic-strain or permanent-set for solids; resultant scratches or dents are relatively non-destructive, unless stress-concentrations in finished surfaces make trouble.

With dozens of hardness tests available, our conventional lab experiments tend to focus on a pair of popular industrial machines, Brinell and Rockwell, and to generate repetitive data for common metals by routine operations. Technologists are not clerks but problem-solvers, in need of an early exposure to a variety of tests, which involve diverse principles and definite applications. Selecting appropriate methods and following published standards are priority lab objectives. Details of several unconventional experiments, with simple equipment and familiar samples, are suggested to supplement certain well-established but lesser-known ASTM hardness tests.

KEY WORDS

Hardness; Mohs-scratch; Brinell-stress; Rockwell-strain; Plastic-dent; Elastic-rebound; Scleroscope.

PREREQUISITE KNOWLEGE

Definitions of Stress-Strain Test, Yield Point, Ultimate Strength, Resilience, and Toughness.

OBJECTIVE

It is short-sighted to limit our technology labs to mere measurements of "the plastic response of metals", typically using Brinell and Rockwell machines. Pupils need a broad view of the various ways to modify "materials" and a deeper understanding of the many methods to assess "hardness" as strength, firmness, rigidity, resilience, stiffness, solidity or toughness.

INTRODUCTION

Hardening processes include: oxidizing and anodizing, plating and coating, cold-working and heat-treating, allotropic and precipitation quenching, alloying and laminating, shot-peening and ball-peining, orienting and cross-linking, irradiating and catalyzing. Testing processes involve: filing and sawing, chiseling and tooling, twisting and scratching, tumbling and breaking, milling and drilling, grinding and sanding, sounding and pounding, magnetizing and sparking, indenting and rebounding.

Commercial tests with standard procedures and plastic strains include: Mohs scratch, Pencil number, File number, Cloudburst shotting, Wheelabrator tumble-blast, Bierbaum plow, Keep's drill, L.A. rattler, Deval weight-loss, Charpy-Izod toughness, Webster pliers, Barcol yoke, Sward rocker, Pfund indenter, Telebrineller indent, Monotron stress, Micro-character scratch width, Imbedded-ball Newtons, Penetrascope pressure, Wilson-King-Brinell pressure, Buehler-Krautkramer-Tukon-Vickers-Knoop micropressure, New Age-Wilson-Rockwell inverse strain, Superficial mini-Rockwell, New Age-Ames-Rockwell portable.

Commercial tests with elastic strains include: Zwick-Shore durometer, A.F. Shore scleroscope, Turner sclerometer, Barcol yoke, Equotip-Leeb rebound velocity, non-destructive sonagram, Boyer-Gall-Lissajou oscilloscope pattern, as well as the Rockwell test (minor-load remains as dial gage is read).

Indenters and penetrators include:

a) Brinell hard-steel ball, Hultgren cold-worked ball, Sintered-carbide ball (WC); all have 10mm diameter and 500-3000 kgf loads.

b) Rockwell hard-steel balls (1/16",1/8",1/4",1/2" diameters) and cone-shaped diamond crystal "Brale", with regular 60-100-150 kgf weights and superficial 15-30-45 kgf weights.

c) Micro-hardness crystals shaped as (⊠)[13](Vickers, Pfund) or (◇) lozenge[8](Knoop); and diamond-tipped points or carbide-disks (Shore, Turner, Leep). So, to say "diamond-indenter" is to confuse. Also, to distinguish between inventers, machines, trademarks, and generic terms is important.

SCRATCH TEST

Material removal (filing, sawing, drilling, milling, planing, sanding, grinding) by hand or machine will produce chips, noise, sparks, heat, odor, lost speed or power, and tool marks or damage. Quantitative evidence, gathered under controlled conditions to describe hardness variations, is elusive. Fortunately, a box of rocks collected by Friedrich Mohs (1812) established a "pecking order" (scratchability) for a wide range of materials (#1-10) from softest talc to hardest diamond.[9] A modified selection (1822) of 15 minerals (#1-15) provided better discrimination of materials. However, purchase of a certified "Rock Kit" is not required to run these scratch tests of HM (Hard Mohs' Numbers) or their half-points:

a) Hold a stick of chalk (say 10cm long) on a flat surface and mark with a pencil (one stroke, about 1cm). Typically, chalk (HM #2) scratches the softer graphite (HM #1.5) and gets a grey mark. However, even a "soft lead" (HM #2 pencil) will leave a "groove" when firmly scribed on the chalk (white chalk-dust coats the pencil point). Obviously, the lead also includes grit and binder (HM #6 to 8).

b) Mark the chalk with the tip of your thumbnail (HM #2.5) or the edge of a copper penny (HM #3.5). Since your nail elastically deflects, the "harder penny" cannot scratch it; however, if you try to scratch the penny's edge with your nail, the nail gets scraped.

c) Hold flat a piece of mild steel (HM #5) and mark with the sharp edge of a broken piece of hacksaw blade (HM #6), with one firm stroke (1cm). However, a broken piece of "white-iron" casting (chilled cementite) or "Duriron" pipe (hi-silicon) is not scratched (HM #7); but, a flat sawblade can be scratched with the sharp edge of such brittle iron.

d) Use other samples with broken edges: e.g., pottery, concrete, glass, tile, file, arrow-head, cut-off saw, or glass-cutter. Show how abrasives and refractories range between HM #7-10. The coating on a "tin-can" is about HM #2. Use all 5-senses to augment the "plastic-scratch" test; e.g., noting friability, sparks, and sounds.

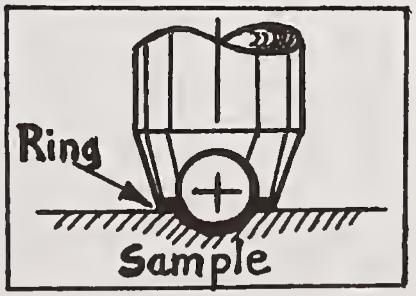
BRINELL STRESS TESTS

Ultra-hard metals (Martensite, Duriron) are highly elastic (stiff) and resilient (rebound), somewhat like elastomeric polymers. Thus, a Brinell hardness test[4] of plastic (permanent) indentation has serious limitations, which can be explored by the lab to supplement routine tests normally run on mild steel, brass, or aluminum:

a.) Rubber specimens (hockey-pucks, old-tires) are tested to show the dent disappears after a Brinell load (500-3,000 kg) is removed. The Brinell hardness is apparently infinite (load ÷ zero area) and patently useless. Refer to stress-strain curves of cast-iron, martensite, or ceramics to show how resilient energy is recovered; i.e., brittle materials are 75-95% "hockey-puck". Even if ultra-hard samples are un-cracked by quenching or testing, and if the test-ball is not damaged, the unrealistically small dents give too-high hardness readings. Save and exhibit any broken balls and samples, from deliberately overloaded trials.

b.) In hard rubber, a "scuff-mark" can be measured with the 20-power telescope (standard ASTM procedure #E 10). Usually, vulcanized rubber lets the Brinell ball (10mm diameter) sink to the holder (say 9mm) to establish a maximum limit for "bearing stress".

Thus BHN (kg per square millimetre) is calculated as load (kilograms) divided by contact area (sq mm), which is a spherical-surface (plus flat-ring) as shown in Figure I. Incidentally, remember steel quenched in cold "Brine" is hard as "'ell", and never mis-spell Brinell or Rockwell; mnemonics are an important part of lab.



c.) Of course, non-standard weights less than 500 kgf can be applied, so the ball-holder does not reach the specimen. While yet loaded, the actual dent-diameter (d) can be measured with thin calipers, or else marked with felt pen (fine point) for later measuring by scope. Often, pre-coating specimen or ball with chalk is sufficient to delineate dents.

d.) For supplementary exercises, measure a metric-ruler, with your "20-power scope", verifying length (not area) is magnified 20X and is accurately read to half-a-tenth (1/20 mm). This converts by "factor-label" method (mm/20 X cm/10mm X 1,000 mil/2.54cm) to 1.9685 mils. So this "2-mil scope" is about half as accurate as a machinist's micrometer.

e.) Another exercise involves weighing a "500 kgf" Brinell disk (about 19 pounds Avoirdupois) but calculating an 1,100 lbf load on the test-ball at 453.6 g/lbf, say 2.2 lbf/kgf). Measure disk-diameter (a boss offsets a recess) and height; subtract the rod-slot; calculate volume and then density (say 480 lb/cuft); probably the disk-material is "steel".

f.) Of course, when soft and/or resilient materials are tested with weights below 500 kgf, direct loads may be applied to specimens via an indenter-ball. Friction in machine-levers at low-loads is thus avoided.

g.) Deriving the "Brinell Stress" formula is instructive, as shown in <u>Figure II</u>:

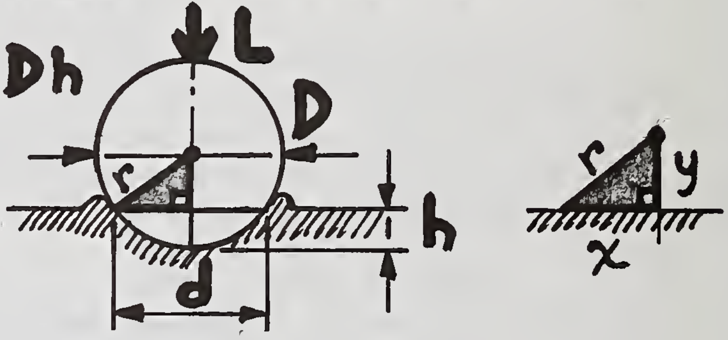
Spherical Dent Area = $\pi D h$

$$x^2+y^2=r^2,\quad h=r-y,$$

$$2r=D,\quad 2x=d,$$

$$\therefore 2rh=h^2+\frac{d^2}{4}=\frac{(4h^2+d^2)}{4}$$

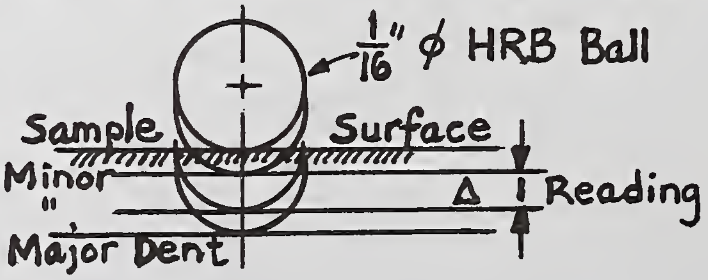
$$HB=L/A$$



ROCKWELL STRAIN TESTS

Similarly, standard tests of hardened-steels or cast-irons (C-setup) can be supplemented with unorthodox (useless) tests of aluminum and brass samples. A deep dent may read "HRC 88", with diamond-cone "Brale" and full 150kgf load. However, compared with "pin-pricks" in a standard C-gage block, this soft sample obviously betrays an off-scale reading (minus 12 or minus 112), depending on the position of the "little-hand", on the dial-gage. This is the time to warn against ultra-high (above 80) or low (below 20) numbers, on a 1-100 scale.

Then, the literal reading on the dial-indicator (<u>Figure III</u>) and the measuring principle (% inverse-strain) of the "**HR**" " reversed-scale can be explained:



BOUNCING BALL TESTS

A ball-bearing (about 10-mm diameter) is dropped through a vertical glass or plastic tube (15mm O.D. X 30cm tall) to rebound from the surface of a common commercial alloy (steel, brass, aluminum). This is the principle of the "Scleroscope Machine":[5]

a) Square bars (25mm X 25mm X 5cm long) or thick disks (25mm X 5cm diameter) are placed on a solid bench, granite block, or anvil.

b) Elastic rebound can be measured (approximately) by a metric-ruler, observing 5 or 10 trials and recording maximum value (less rubbing against tube-wall).

c) As a measure of elastic hardness, a "coefficient of restitution" (e) is calculated as the square-root of maximum rebound-height ÷ original drop-height. Velocity is the square-root of $2gh$, where g is the acceleration of gravity. So, elastic hardness may be expressed as the inverted restitution-coefficient, $1/e$.

d) As a measure of plastic hardness, a Brinell scope (20 X) is used to measure dent-diameter (nearest twentieth of a mm). The "Impact Value" can be calculated as Energy (ball-weight X drop-height) per Area (spherical contact). Thus I.V. = (kgf x mm) per mm-squared. Prepare smooth specimens (file or sand off any hacksaw marks). Students should know 20x power means 20-times length.

e) Compare both values (elastic and plastic) to determine which material is "harder". Generally, the higher the rebound the smaller the dent. Thus, the greatest sum($1/e$ + I.V.) identifies the hardest specimen.

f) Another plastic-strain test involves mounting a slender bar (say 5mm square X 5cm long) of mild steel (or copper, brass, bronze, aluminum) in a vise. Twisting 90° with a torque-wrench (fitted with a square-socket) gives an initial reading. Then, the return twist (minus 90°) gives a higher reading (from work-hardening). Report as torque (force X distance) per degree.

CONCLUSION

For useful correlations of various hardness tests (elastic and plastic), see "References"[7]. Absolute hardness of soft-steel (annealed) is about 100 kg/sq mm (minimum); precipitation-hard aluminum is about 100 kg/sq mm (maximum); alloyed copper (brass) is about 60 kg/sq mm; soft-aluminum (annealed) is about 30 kg/sq mm. These are comparable to conventional U.S. values of elastic-modulus (E); i.e., 30-million (psi) for steel, 20-million for brass, and 10-million for aluminum.

TERMS

1. Elastic strain - stress is proportional to strain (Hooke's Law); strain returns to zero, when stress is removed.
2. Force - time rate of change of momentum (mass times velocity); commonly mass times acceleration of constant mass; load or weight.
3. Hardness - resistance to penetration, elastic and/or plastic; related to strength, stiffness, resilience, toughness.
4. Hysteresis - energy left in a material, after elastic-strain and stress-removal.
5. Impact - Potential energy absorbed by rapid blow. (weight x height).
6. Mechanical - relating to forces (tension, compression, shear).
7. Plastic strain - stress exceeds a material's elastic-limit or yield-point); some permanent deformation then remains, when stress is removed.
8. Resilience - Elastic energy absorbed (area under linear plot).
9. Rigidity - Torsional stiffness.
10. Scleros - Greek word for "hard" (root of scleroscope, skeleton, multiple-sclerosis).
11. Stiffness - Slope (stress per strain) of an elastic linear plot.
12. Strain - relative change of a key dimension (length, area, volume); the result of stress.
13. Strength - Maximum or ultimate stress; so-called "Tensile Strength" (T.S.).

14. Stress - Reaction of material to pressure (load per unit area).
15. Toughness - Total energy absorbed (area under entire stress-strain curve).

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UNCONVENTIONAL IMPACT-TOUGHNESS EXPERIMENTS

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ABSTRACT

In the materials lab, various impact observations and exercises can illustrate basic concepts of mechanics (Work, Kinetic Energy, Potential Energy, Free-fall, and Friction), while defining Weight, Mass, Center-of-gravity, Velocity, and Acceleration. Unconventional impact-tests of un-notched specimens may exceed the capacity of a conventional but unanchored pendulum-machine. This leads to adjusting toughness readings, for kinetic and friction losses, before assessing the devastating effects of notching a test-bar or changing the temperature.

Keywords: Impact-toughness; Energy-absorption; Drop-weight; Pendulum-tup; Charpy-Izod Tests

Prerequisite Knowledge: Stress-Strain Test; Crystal Lattices (BCC, FCC); Notch Sensitivity; Potential-Kinetic-Frictional Energy

Objectives: 1. To distinguish between slow absorption of energy (tensile toughness) and fast absorption (impact toughness); 2. To compare notched vs. un-notched samples of steel (BCC iron).

INTRODUCTION

Plain carbon steel is conventionally tested in tension, with slowly increased axial-load at constant cross-head speed, per ASTM #A370 specification. Modulus-of-toughness is routinely measured as the area under an engineering plot of stress (load/original cross-sectional area) vs. strain (change in length/original gage length). Typical units are psi (pounds per square inch) or N/m^2 (Newtons per square meter); however, toughness is not to be confused with stress, elastic-modulus, or elastic-resilience (also psi). When reported as "inch-pounds per cubic inch" (or Newton-metre per cubic metre), then tensile-toughness is seen as total energy per unit-volume of test material. It involves both elastic and plastic energy, slowly-absorbed.

By contrast, impact-toughness is perceived as rapidly-absorbed energy per standard specimen, usually reported as "foot-pounds" (or Joule; Newton-metre) of total shock-resistance. This is commonly mis-labeled "impact-strength." Of the 14-different "Bravais" crystal types, a body-centered cubic (BCC) lattice of iron (steel) is exceptionally vulnerable to a shock-load in a cold environment, as blemishes bring stress-concentration or notch-sensitivity. Moreover, a coarser-grained metal, with higher area-volume ratio and lower strain-rate, tends toward lower impact-toughness. To avoid potential trouble in specific alloys or processes, a variety of impact tests were developed for a falling-weight (tup) or swinging pendulum (gravity) in several different setups:

1. Standard samples (notched or plain) are rapidly loaded in bending, tension, or torsion.

2. Samples are supported as simple beams (Charpy), cantilevers (Izod), or fixed beams (Both ends).

3. Notches are V, U, or keyhole shaped. Their surface smoothness is a critical factor.

4. Toughness vs. temperature (y vs. x) is plotted, with constant tup-speed, for identical samples at various temperatures.

CONVENTIONAL IMPACT-TESTING

Results from different test-setups generally cannot be converted. Probably the most popular test is with a gravity-pendulum, per ASTM #E23: Samples of a specific metal are tested under constant conditions (Charpy-supported, V-notched) except with varying temperatures; the objective is to determine any "critical temperature," where ductile-brittle transformation may occur. Common applications were the design of Alaska pipelines and the modification of North Atlantic ships, where brittleness was a severe problem in BCC steels.

Materials lab classes tend to neglect impact-testing, until a costly pendulum-machine (with digital readout) is available. Most demonstrations simply involve notching and breaking 1/4" x 1/4" x 2" steel bars (hot-medium-cold) to obtain readings of absorbed energy. Metric bars are typically larger (10 mm x 10 mm x 60 mm long). Conveniently, boiling-water (100°C) and dry-ice (-56°C) are equi-spaced (±78C° of temperature difference) from room-temperature (22°C). However, to contrast the notched bars (brittle) to thicker un-notched bars (tough) may be desirable but difficult: cross-sectional areas are unequal, machine-damage threatens the anchored-machine, and energy-losses beset an unanchored-machine.

UNCONVENTIONAL IMPACT-TESTING

Simplified demonstrations of dynamic material properties, varying with strain-rate, can be done in the classroom:

1. Pitch will shatter, when hit with a hammer; but ductile-flow ensues with slow pulling, even at room temperature.

2. "Silly-Putty" (trademarked silicone polymer) will snap like brittle chalk, when jerked or sharply twisted; but ductile necking will occur with slowl/4" loading. Also, a rolled-ball will bounce like rubber (fast compression); but the same ball slowly makes a puddle (viscous liquid) with gentle gravity.

3. Panes of window-glass will snap when notched, using a martensite file or diamond cutter,.especially when wetted; but an aged-pane may exhibit a marked increase in calipered-thickness, from top to bottom, with ductile-flow induced by gravity at moderate temperature.

4. Certain grades of glass-tubing, when heated red-hot, will snap if jerked but will neck ductilely if slowly pulled.

5. Sugar cubes may "fluoresce" in the dark, when hammered. Similarly, "Certs" (trademarked breath-mint) may "flash" with piezoelectricity, when crunched and viewed in a dark room.

Quantitative measurements of impact-toughness are feasible, using an inexpensive drop-weight tester. (See ASTM #E208.) The blunt "tup" slides like a guillotine down an upright pair of guide-rods, striking the test specimen placed on the base. Generally, this test precedes a standard "guided-bend" test of strips cut from welded pipe. The drop-height is increased until "work-to-break" is found (weight x height) when friction is ignored. For unsatisfactory joints, this avoids the expense of guided-bends. Obvious classroom variations are: comparing different metals, notches, temperatures, or processes; e.g., thick steel plates may be brittler than thin ones; cold-worked steel may be tougher than air-cooled products.

A primitive pendulum can be made from a ball-bearing, swung from nylon fish-line, supported by a ring-stand or easel-frame. Target samples could be boiled eggs or blown shells. Energy (weight x height) to crack or break the egg is determined by increasing the pendulum-angle and calculating drop-height (from radius and angle). The ball's center is the pendulum's "center-of-gravity," neglecting the fish-line. A large protractor can become a "direct-reading" quadrant, calibrated as N x m (or ft x lb). Threads, wires, or film-strips can be stretched across the pendulum's trajectory. Tear-resistances of papers and plastics (notched vs. plain) can be compared. If excessive energy is used, the sample's absorption is then figured as weight times loss-in-height (original-final).

A small inexpensive pendulum-machine (about $500) is available for testing plastics. Typical specimens are small "dumbbells" (about 1 cm long). Tensile-impact then decapitates one end and registers the energy expended (N x m). The specimens can be molded in the lab, using an auxiliary unit (another $200).

A plain pendulum-machine (costing $5,000 to $10,000) will directly indicate the energy absorbed by a struck specimen. Model #74 (Tinius-Olsen Co.) has 264 ft-lb capacity (center-of-gravity of 60 lb tup x 4.4 ft vertical fall). Its 1500 lb frame has a "Charpy" simple-beam support for the sample. Plastics bars are plied (say 30 mm x 10 mm) for adequate cross-section. Obviously, it is wrong to compare un-notched tensile-toughness (in-lb per cu in) with notched impact-toughness (ft-lb). Just comparing the un-notched vs. notched impact-toughness of standard (10 mm square) steel bars (5-6 cm long) is complicated:

1. The pendulum will jam in an un-notched bar, like an axe in a rotten stump, barely bending the sample bar and falsely reading 264 ft-lb (maximum).

2. Unless anchored, the machine will slide 2-4 cm (about 1") on a tile-floor; clearly the machine's capacity was exceeded, and pendulum-energy was dissipated. Anchoring the base may lead to a damaged pivot on the swing-arm, also giving false readings. Eventual shaft-fatigue and failure can occur.

3. Visually comparing the tested specimens (un-notched vs. notched) should convince students that "tough steel" (AISI #1040) became brittle (like cast-iron) when notched and hammered. But how can this subjective observation be objectively quantified?

PROCEDURE

Perhaps an impact-toughness reading (for notched bar) should be standardized, dividing absorbed energy (ft x lb x 12 in/ft) by the bar's volume (cu in). Comparison with tensile-toughness (psi) then seems logical; however, ASEE recommends "all-SI" classwork; and comparing bending mode vs. pulling is suspect. (See ASTM #E208.) With S.I. metric equipment, the standardized result would be in Pascals (N.m per cu m).

Perhaps a better approach is this:

a) Adjust the original "un-notched reading" (say 264 ft-lb) for kinetic and friction losses. Machine weight (1500 lb) times dynamic friction-coefficient (say 40%) times sliding distance (1/12') is 50 ft-lb of sliding-work. Moreover, 20% reduction of the indicated toughness for an un-notched (10 mm) bar (264 ft-lb) is 53 ft-lb. Thus the pendulum really lost 103 ft-lb (39%) of its original 264 ft-lb of potential energy. This lowers our "reference toughness" to 161 ft-lb (264-103). Steel is still seen to be plenty "tough," if not blemished.

b) Adjust the actual "notched reading" (say 7 ft-lb) for kinetic energy loss of a "flying sample," at room temperature. Thus, a 1.5 oz bar (by postal scale) with 12 fps observed velocity (about 6' per half a second) has KE of 0.21 ft-lb; this is a 3% inflation of the material's toughness.

c) Now compare "actual notched-toughness" (7 ft-lb) of Item (b) with "reference toughness" (161 ft-lb) of Item (a). We may conclude that V-notched steel is about 4% (7 vs 161) as tough as smooth steel, when struck at room temperature.

As a final exercise, the class can estimate or calculate velocities:

a) Average velocity of the sliding 1500 lb machine is estimated by equating kinetic energy with sliding-work (50 ft-lb) of Item (5a). Thus, V-squared is 64.4 (50/1500), and 1.47 fps is sliding-velocity.

b) Impact velocity of of the pendulum is estimated by equating kinetic energy with potential energy (minus 1% lost arm-friction). Thus, KE is (264-2.64) ft-lb; the pendulum velocity is 16.75 fps.

c) This velocity is checked, using Newton's "free-fall" formula, as Initial velocity squared + 2gh is Final velocity squared. With zero Initial velocity, the Final velocity squared is 64.4 times 4.4 ft per second squared; so, pendulum velocity is 16.83 fps. Then, figure the percent difference, based on 16.75 fps (more realistic).

d) All answers should then be converted to S.I. units.
Pound force (Avoirdupois) x 4.448222 is Newtons.
Feet x $0.304800\overline{0}$ is metres.

e) Metric machines are recommended for all future acquisitions.

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ADAPTING ARCHIMEDES' METHOD FOR DETERMINING DENSITIES AND POROSITIES OF SMALL CERAMIC SAMPLES

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Key words: Archimedes' Method, bulk density, apparent density, porosity

ABSTRACT

Nearly all materials contain at least two phases: solids and pores. Many standard methods for determining densities and porosities, especially for a variety of ceramic products, employ various adaptations of Archimedes' Method. This presentation demonstrates how the method can be extended using an analytical balance and several precautions to accurately determine physical properties of samples smaller than one gram.

INTRODUCTION

Literally hundreds of specific test methods exist for determining various densities of materials. Among those standardized by the American Society for Testing and Materials (ASTM), well over a dozen tests (ref. 1) employ Archimedes' Method directly to determine at least some of the several properties of materials that can be calculated using this simple technique. Many other tests for determining densities (such as hydrometer, pycnometer, and porosimeter methods) also use the principle of fluid displacement, although not in the manner described here.

The method is sometimes used for cemented carbides, high modulus fibers, electronic wire, asphalt, plastics, bituminous coal, and even peat. However, the most common and the most detailed uses occur for ceramic and related products (refs. 2-4). Typical ASTM tests include:

C 20	Burned refractory brick, by boiling water
C 373	Ceramic whitewares
C 725	Semidense mineral fiber siding
C 830	Burned refractory brick, by vacuum pressure
C 914	Solid refractories, by wax immersion
C 948	Glass-fiber reinforced concrete
C 1039	Graphite electrodes

These tests, as written, all require relatively large specimens with masses of at least 100 g to assure significant results using balances with accuracies of no better than 0.01 g.

PREREQUISITES

A rudimentary understanding of the types of volumes, densities, and porosities is required (refs. 5, 6). In addition, the ability to determine masses using an analytical balance is presumed.

OBJECTIVE

The purpose of this experiment is to demonstrate how Archimedes' Method and an analytical balance can be used to determine the mass bulk density and several other related physical properties of a small (approximately 1 g) ceramic specimen.

EQUIPMENT AND SUPPLIES

In addition to basic laboratory supplies such as tweezers and tissues, the following are required:

Analytical balance, mechanical, accurate to 0.0001 g
Bell jar (or vacuum type desiccator)
Vacuum pump
Thermometer, for room temperature
Copper or other non-corroding wire, 0.40 mm gauge or less
Jar or beaker, approximately 9 cm diameter x 9 cm high
  (Small peanut butter jar works well!)
Distilled water
3-legged stand

The 3-legged stand, which is used to hold the jar above and away from the balance pan, can be constructed of any materials on hand but needs to have: 1) a base approximately 8 cm x 14 cm, and 2) legs approximately 30 mm long.

PROCEDURE

1. Tare analytical balance. Then measure dry weight, W_D, to the nearest 0.0001 g; record.

2. Place specimen in its support in the jar and fill to within approximately 1 cm of full with distilled water.

3. Place jar of water containing specimen inside of bell jar.

4. Connect vacuum pump to bell jar. (It is desirable to place a trap in the line to prevent contaminating the vacuum pump oil.)

5. Turn on vacuum pump for at least fifteen (15) minutes so that the water boils, and water enters all open pores within the specimen.

6. Remove the jar containing the specimen, and allow the temperature to approach equilibrium. Record the temperature.

7. Place the 3-legged stand over the pan of the balance, making sure it does not contact the pan. Then tare the balance.

8. Place the jar containing the specimen and its support on the 3-legged stand.

9. Hang the support wire over the second hook below where the pan bail is connected.

10. Measure the gross saturated suspended weight of the specimen plus its support, $W_{ss,G}$, to the nearest 0.0001 g; record.

11. Use tweezers to dislodge the specimen from its support, and either transfer it from the jar to another container of water at the same temperature or allow it to fall to the bottom of the jar.

12. Measure the tare weight of the support, $W_{ss,T}$ while suspended; record.

13. Remove the jar and 3-legged stand from the balance, and tare the balance again.

14. Daub surface of specimen with a wet tissue or cloth to remove any droplets of water on the surface. Then promptly measure the saturated weight, W_S; record.

15. Measure the temperature of the water in the jar. If this has changed more than 0.2°C, then repeat steps 7-14. Otherwise, determine the water density, d_L, corresponding to the average water temperature during the determinations of the various saturated weights.

16. Calculate the bulk volume, bulk density, apparent volume, apparent density, apparent porosity, and water absorption using the equations provided.

SAMPLE DATA AND RESULTS

For a specimen of aluminum oxide, which had been sintered at 1667°C for ten (10) hours, the following were measured:

Dry weight, g	3.1127
Gross saturated suspended weight, g	2.7182
Tare weight, g	0.3869
Saturated weight, g	3.5281

Physical properties calculated from the above include:

Bulk volume, cm^3	1.197
Bulk density, g/cm^3	2.601
Apparent volume, cm^3	0.7814
Apparent density, g/cm^3	3.983
% apparent porosity	34.71
% water absorption	13.35

Although not part of the scope of this experiment, it can further be shown that for a theoretical density of aluminum oxide equal to 3.98 g/cm^3, other properties are:

% closed porosity	0.00
% total porosity	34.7
% apparent theoretical density	100.0
% theoretical density	65.3

INSTRUCTOR NOTES

Density and Porosity Relationships

Archimedes' Method permits several physical properties relating absorption, densities, and porosities of a specimen to be calculated from just three weight measurements: dry weight, saturated weight, and the net saturated suspended weight.

The table on the following page shows six (6) of the commonly calculated properties using different notations.

Property	ASTM	Subscripted
Bulk volume	$V = M - S$	$V_b = \frac{W_S - W_{SS}}{d_L}$
Bulk density	$B = \frac{D}{V} = \frac{D}{M - S}$	$d_b = \frac{W_D}{V_b} = \frac{W_D \times d_L}{W_S - W_{SS}}$
Apparent volume (Impervious material)	$= D - S$	$V_a = \frac{W_D - W_{SS}}{d_L}$
Apparent density	$T = \frac{D}{D - S}$	$d_a = \frac{W_D}{V_a} = \frac{W_D \times d_L}{W_D - W_{SS}}$
Apparent porosity	$P = \frac{M - D}{V}$	$\%P_a = \frac{W_S - W_D}{W_S - W_{SS}} \times 100$
Water absorption	$A = \frac{M - D}{D}$	$\%A = \frac{W_{WS} - W_D}{W_D} \times 100$

where: $M = W_S$ = Saturated weight
$S = W_{SS}$ = Saturated suspended weight
= Gross weight $W_{SS,G}$ - Tare weight $W_{SS,T}$
$D = W_D$ = Dry weight
d_L = Density of saturating liquid
W_{WS} = Water saturated weight

Note that for the first four (4) ASTM relations the units do not seem to be consistent. This is because the saturating liquid is assumed to be water with a density of unity. For other saturating liquids (such as for example when kerosene is used for water soluble refractories), the complete subscripted equations containing the liquid density factor must be used.

For an excellent discussion of these and other related properties, see Reference 6. This clearly explains the differences between open (or apparent or intergranular) and closed (or intragranular) porosities. It also clarifies the distinctions among bulk, apparent, and true densities.

Sources of Error

Primary causes of erroneous results include inadequate attention to the following precautions:

1. Allow at least the minimum time (15 minutes) for the "boiling in vacuum" absorption step so that the specimen absorbs water (or other immersion liquid) into as many of its open pores as possible.

2. Allow the water and specimens sufficient time to approach a temperature equilibrium following the absorption step -- usually at least twenty (20) minutes.

3. Be sure to tare the balance, after placing the stand over the pan, before determining the gross saturated suspended weight and the tare weight of the support.

4. Do not take any more time than necessary to measure the saturated weight; otherwise, some water from the open pores may migrate to the surface and evaporate.

5. Take care that no water droplets fall onto the balance pan. Also be sure that no water droplets cling to the support wire loop.

6. If a basket is tied to the support wire, make sure that only a single wire breaks the surface of the water.

7. Check that no air bubbles adhere to the specimens or support. This is often the case if a mesh basket is used. Light tapping will suffice to release these to the surface.

Variations

This basic technique can be used for a variety of experiments such as:

1. Compare the results obtained for a small piece of refractory brick with those obtained using ASTM test method C 20 on a half brick.

2. Determine gradient in density and porosity properties between center and outside of a fired ceramic article.

3. Analyze errors and significant figures of results. For example, what error is introduced if the immersion density is assumed to be unity and the effect of ambient temperature is neglected?

   (Obviously, the calculations need not be done manually, and the data can provide practice for writing dedicated computer or spreadsheet macro programs. In particular, the dependence of water density upon temperature provides an excellent exercise for fitting an empirical equation for use in such programs.)

4. For refractory specimens that slake, hydrate, or are soluble in water, compare the results obtained by using another liquid (such as kerosene) with the results using ASTM test method C 914, which involves coating the specimen with paraffin.

5. Compare the results with those obtained by using geometric, pycnometric, sink-float, or other determinations.

6. If the solid material is single phase (or ideally so), then determine the theoretical density from X-ray diffraction or other sources, and calculate the percent theoretical density and the percent closed porosity.

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EFFECT OF HEAT TREATMENT ON MAGNETIC PROPERTIES OF A METAL ALLOY

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ABSTRACT

An experiment is proposed using a non-destructive test method, Gaussmeter, to observe the effects of heat treatment on the magnetic flux inductance of a soft magnetic material. The material chosen for this exercise is a nickel alloy steel known as Alloy 48. The chemical composition of this alloy is; 48% nickel, 0.25% manganese, 0.02% carbon, 0.001% sulfur, and 51.73% iron.

In this study, the Alloy 48 will be heat treated in an oven for four hours at 927°C. Test samples will then be air cooled in the lab environment at ten minute intervals. Time factor of heating is the test variable. Finally these heat treated samples will be magnetized using a neodymium-iron hard magnet. The magnetic flux density or induction will be measured using a Gaussmeter.

The results of this experiment could be used to help material technologists and engineers determine heat treatment time factor needed for optimum permeability of a metal alloy such as Alloy 48.

INTRODUCTION

Ferromagnetic materials are generally classified into two distinct groups, soft and hard magnets. Soft magnets are the metals can be easily magnetized and demagnetized. However, hard magnetic materials maintain a net magnetization after a magnetization process. When a soft magnet is placed near a hard magnet, there will be attraction between the two. The soft magnet will then become magnetized due to the induction of the magnetic flux from the hard magnet. When the two magnets are separated, the soft magnet will return to its original unmagnetized state.

Permeability, is the ratio of magnetic flux induction and magnetizing force, or the ease at which a material will set up a magnetic field. The gaussmeter is an instrument that measures the magnetic induction using the unit "Gauss" of the CGS system.

Alloy 48 is a nickel base metal alloy, designed for high permeability, high field strength, and low core losses. It finds excellent applications in many industries such as instrument transformers, electronics relays, communication equipment devices and magnetic shieldings. This experiment is designed to observe the effects of heat treatment on the inductance of a soft magnetic material, Alloy 48. On the other hand, a hard magnetic material, known as Neodymium-Iron alloy, is used as a magnetising force to induce magnetic flux in the soft magnet. After heat treatment, both soft and hard magnets will be examined using a Gaussmeter to see how would heating hours affect the future magnetization process. The results of this experiment could be used in industry to indicate the appropriate heat treatment time factor needed for optimum permeability of the material , Alloy 48.

EXPERIMENTAL PROCEDURE

Heat Treatment

Eleven metal samples were cut from a hot-rolled 0.25 inch thick Alloy 48 sheet stock. After referring to ASTM standards and taking into account of the size of the available furnace in the laboratory, the metal samples were further cut into 4.0 inch by 1.5 inch strips. The samples were then deburred, stamped with numbers 0-10. Sample 0 was left untreated for comparison. According to the recommendation of materials supplier, samples 1-10 were normalized in the furnace for 4+ hours at 927°C (1700°F). At four hours the samples were taken out one by one, at ten minute intervals, so that they would have varying time factors of heating. All heat-treated samples were allowed to have sufficient air cooling. The attached oxidation was gently sanded off, and the samples were further cleaned with rubbing alcohol (isopropyl alcohol). Six 0.75 inch square by 0.375 inch thick neodymium magnets were selected to be used as the magnetizers.

Magnetic Measurements

The gaussmeter Model 615 manufactured by F. W. Bell was employed to take readings at specific locations on the surfaces of the Alloy 48 samples. It was decided to use sample numbers 1, 3, 5, 7, and 9, in the test so that the intervals of heat treating would be twenty minutes. The gaussmeter was calibrated to zero, then readings were taken off of the six neodymium-iron magnetizers to characterize them. Readings were recorded at 0.25 inch intervals using an axial probe and a template to determine accurate locations (Figure 1). The soft magnets (Alloy 48 samples) were then characterized and the readings were found to be zero. The magnetizers were then placed on top of the six heat treated Alloy 48 samples, directly in the center of the large surface. The magnets were then left to sit for one hour so that the soft magnets hopefully could become magnetized. Magnetic measurements were conducted using the probe at twelve points along the perimeter of the soft magnets on both sides (see Figure 2). Hysteresis loops are plotted in Figure 3.

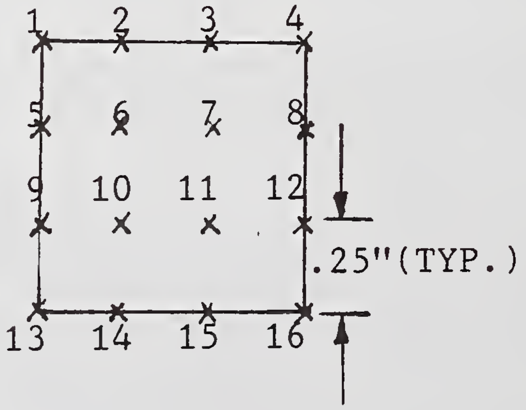

Figure 1. Points Locations Across Hard Magnet

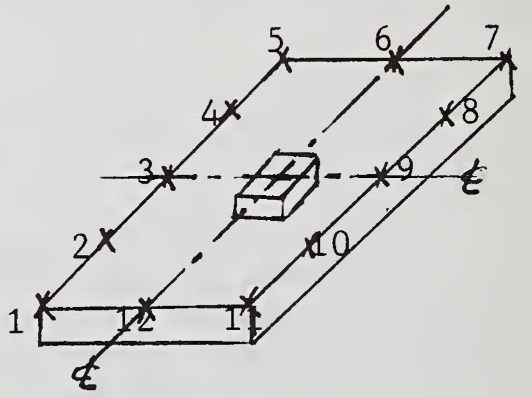

Figure 2. Positions of Hard Magnet on Soft Magnet

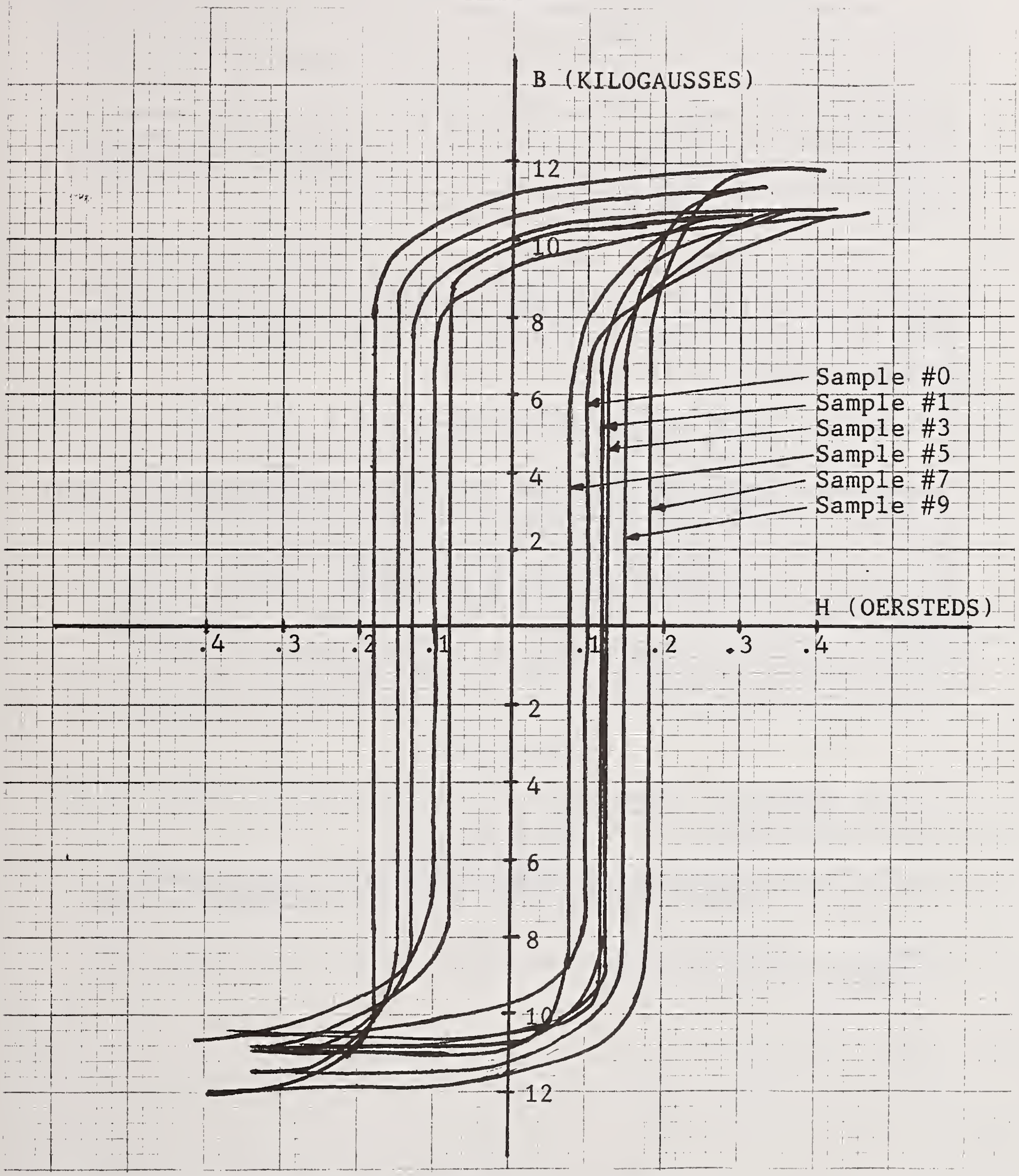

Figure 3. Hysteresis Loops for Samples 0, 1, 3, 5, 7 and 9.

DISCUSSION

From analyzing the magnetic induction readings taken off the smaller hard magnet alone, and the readings from when the hard magnet is situated on the soft magnet, it can be determined that the magnetic induction across the hard magnet increased quite consistently at corresponding points. The enhanced magnetic induction could be due to the sum of the magnetic flux density of hard magnet itself and the external magnetic flux density of the magnetized bar (soft magnet). It is also evident that there is no clear pattern between the heat treated samples themselves, or in comparison to the untreated sample (Group 0). In general, the readings lowered as the probe got closer to the center line, where the polarity transformation occurs. Group 5 sample after being heated for five hours was found to have lowest readings along the perimeter of the hard magnet.

Plots shown in Figure 3 reveal that there is a significant increase in the magnetic induction over its control counterpart (Group 0). The best group in this experiment was Group 1, which had been heat treated for four hours and air cooled. Sample 5 has least amount of energy loss during the magnetization process. When samples were cooked too long in an oven, they seemed difficult to be magnetized.

CONCLUSION

It is found in this study that normalizing of the Alloy 48 material can increase a material's permeability during a magnetization process. The optimum condition was found in the sample one which had been heat treated for four hours and then immediately air cooled, if the core loss is not considered. Sample 5 has the least amount of core loss. Other groups of samples generally have lower permeabilities and higher core losses because of longer heating times. This experiment is found not a great determiner of the variance of magnetization with heat treatment. Because it is difficult to see any changes corresponding to the time/heat treatment factor. A systematic approach employing annealing and quenching techniques and different heat treatment parameters can be utilized to help understand the relationship between magnetization and heat treatment work.

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ASTM

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DETERMINING THE IMPACT OF ADJUSTING TEMPERATURE PROFILES ON PHOTODEGRADABILITY OF LDPE/STARCH BLOWN FILM

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ABSTRACT

The major objective of this study was to develop a test procedure to determine the impact of modifying temperature profiles of extruded blown film on photodegradability characteristics of a low density polyethylene (LDPE)/15 percent starch blend.

Four sequential temperature adjustments, each of 2 percent from the manufacturers recommended processing temperature for polyethylene were used. Three barrel temperatures and two die temperatures were adjusted accordingly throughout the test. The blown film bubble and final film gage were stabilized previous to the study to minimize any influences beyond typical processing control.

Three levels of lighting were used to determine the impact of light on LDPE/starch film. The three conditions were as follows: 1) control specimens were kept in total darkness, 2) typical four foot office fluorescent lights with acrylic diffusers were used to represent an average office environment, and 3) an accelerated weathering unit was used to represent the more intensive ultraviolet (UV) lighting conditions. Six samples per processing temperature setting, per week, per environment were prepared anticipating tensile tests relative to machine direction of the blown film. Sample preparation and testing procedures followed ASTM as close as practically possible. Total time of exposure in all environments was limited to six weeks with testing of 90 selected specimens weekly. This required a minimum of 540 test specimens to be processed, documented, handled, tested, and evaluated.

The test procedure provided information relative to the UV light sensitivity of an LDPE/starch blend over a variety of light conditions and processing temperatures sensitivity and photodegradability characteristics. The study has the potential to provide information for the processing characteristics of temperature controllers relative to quality control.

INTRODUCTION

The development of the following test procedure, to determine the impact of modifying temperature profiles of photodegradable extruded blown film evolved as a result of various discussions centered around plastics packaging and the environment. Blown film was used as the processing method for several reasons:

* the material was thin enough to show changes in tensile test results in one 15 week semester
* the process could be stabilized and controlled
* large numbers of samples could be produced in a short period of time
* test equipment was readily available
* storage, handling, and testing specimens presented the fewest problems using blown film
* the film produced from LDPE represented the typical packaging material.

Instead of the traditional processing and testing, the environmental/photodegradability issue causes one to view the dilemma from a systems approach. It forces students involved with plastics manufacturing to consider the inevitable, to design a product that is, in the long run, a greater part of the solution to the environmental problem.

The exact amount of plastic that goes into Americas' sanitary landfills is unknown but estimates vary from a low of 4 percent to a high of 35 percent by weight. Plastics are as pervasive in landfills as they are in our daily lives. Any material which is destined for a landfill should be either photodegradable (light sensitive) or biodegradable (microorganism sensitive). Regardless of the environmental attitudes (pro or con) on the part of the student we wanted to develop a test procedure that caused the participants to think more analytically.

When considering the equipment available, we had to acknowledge what test procedures could be developed to adequately evaluate the photodegradability of an LDPE/starch blend? Equipment eventually selected consisted of:

* Davis Standard 1.250" extruder with blown film die and tower
* Instron universal test center (model 1011)
* Q panel for accelerated exposure

Additional materials necessary to insure repeatability consisted of various ASTM standards, sampling procedures, statistical process control tools, and assorted handheld measuring instruments.

Material used throughout the test was an 85 percent blend by weight of Dow polyethylene 533 low density film extrusion resin and 15 percent by weight of Archer Danials Midland's (ADM) Polyclean 012-400, general purpose starch based additive. The additive is formulated such that it can be either extruded or injection molded.

The major objective of this study was to develop a test procedure to determine the impact of modifying temperature profiles of extruded blown film on photodegradability characteristics of an LDPE/15 percent starch blend. The film was produced using five sets of processing parameters. Each parameter set had an increase or decrease in the temperature profile and screw speed in increments of 2 percent from the manufacturers recommended settings. Three levels of lighting were used to determine the impact of light on biodegradable film. The three lighting conditions were as follows: 1) control specimens kept in total darkness, 2) normal four foot fluorescent tubes, and 3) an accelerated weathering unit. Six samples per processing temperature setting, per week, per environment were prepared anticipating tensile tests relative to the machine direction of the blown film (Table 1).

PROCESSING MATERIAL PREPARATION

The resin and additive obtained both represented typical materials as used in the production arena. The LDPE extrusion film resin 533 was obtained from DOW Chemical company. The following represents a brief listing of processing conditions, and select physical and mechanical properties of resin 533 (Table 2).

It was assumed that the values from Table 2 represented averages drawn from a normal distribution. Melt temperatures, blow-up ratio and optimum gauge range were used to represent the standard from which all tests were run.

The additive obtained from Archer Danials Midland's (ADM) was Polyclean 012-400. It was recommended by representatives at ADM that a 15 percent by weight blend of this material added to the LDPE would represent the typical blends as used in industry. 7.5 lbs. of Polyclean were added to 50 lbs. of LDPE in a clean 55 gallon barrel and sealed. Care was taken to minimize exposure to any form of light. The drum was rotated slowly (1 revolution every 2 seconds) to thoroughly blend the LDPE and Polyclean. ASTM D1898, Standard Practice for Sampling of Plastics was used to insure the blend was homogeneous. If the blend was not homogeneous, processing time and temperature would vary excessively, material in contact with the screw and barrel walls would not be uniform, gauge thickness would not be consistent and the photodegradability results would be inconsistent.

MATERIAL PROCESSING

The LDPE/Polyclean blend was processed in a Davis Standard 1.250" (DSR-125) extruder with a Barber-Colman control system and a Maco 8000 processor. The air ring, side fed die, cooling ladder and mandrel were built from various industrial designs. The air ring has eight equally spaced air ports for greater control when producing the blown bubble. The ladder was leveled and centered with the die before each production run. A LDPE curtain was placed around the cooling ladder because of the adverse affects of air from not only the air conditioning system but also personnel in the area. Final layflat width was kept as close as possible to 5" while film thickness varied from 0.0021" to 0.0024". ASTM D1248, Standard Specification for Polyethylene Plastic Molding and Extrusion Materials was adhered to as close as practically possible for an educational environment.

The film was produced using the following five sets of processing parameters (Tables 3-7). Each processing parameter had an increase or decrease in the temperature profile and screw speed in increments of 2 percent from the manufacturers recommended settings. Through previous experiments it was determined that a 2 percent adjustment was adequate to produce results desired for this experiment.

TESTING MATERIAL PREPARATION

Subsequent to the processing of each of the five rolls of blown film, samples were kept in darkened environments. As needed each set of samples were prepared following ASTM 882, Standard Test Methods for Tensile Properties of Thin Plastic Sheeting using parallel knives designed specifically for thin film sample preparation. The parallel knives provided specimens with a uniform width of 1.0", care was taken to remove any samples with notched or upset edges. Folded seams created at the cooling ladder were avoided when cutting specimens, all specimens were cut from the center portion of either the front or back of the layflat. The width of each specimen should be no less than 0.02" and no greater than 1.0". The overall length of each specimen was 6.5".

Each sample was then labeled with an alpha-numeric code for identification. Table 8 represents the labeling procedure used.

ACCELERATED UV UNIT

ASTM G 53-84, Standard Practice for Operating Light- and Water-Exposure Apparatus (Fluorescent UV-Condensation Type) for Exposure of Nonmetallic Materials was referenced for use with the Q-panel. The Q-panel is not intended to simulate the deterioration caused by localized weather phenomena, such as atmospheric pollution, biological attack, and water exposure. Rather, it is intended to simulate the deterioration caused by water as rain or dew and the ultraviolet (UV) energy of the sun.

The apparatus consists of: A test chamber made of corrosive resistant material with a heated water pan and fluorescent UV lamps, specimen racks, and provisions for controlling and indicating operating times and temperatures. The lamps are rapid start, medium bipin fluorescent UV lamp with a nominal rating of 40 Watts when in operation. Lamps used were UV-A 340 which produces wavelengths in the 315 to 400 nanometers range. UV-A 340 represents sunlight not filtered through glass. The UV-A causes slower degradation than the UV-B. UV-B lamps will produce wavelengths in the 280 to 315 nanometers range. The eight lamps were rotated every four-hundred hours as per manufacturers recommendations. Samples in the Q Panel were exposed to lighted conditions and condensation 12 hours per day for the duration of the study. A condensation mechanism produced water vapor generated by heating the water pan located under the entire specimen area. The water supply in the water pan was regulated with an automatic controller to provide a constant water level. Specimens were mounted and secured with surgical tape. Surgical tape was used because it provided the best method of securing the samples when exposed to the UV light and water condensation.

NORMAL FLUORESCENT LIGHTING UNIT

The fixture was designed to secure samples to be placed in a typical fluorescent lighted environment. The fixture was a wood frame, 8.5" X 43.5", secured to a piece of hardboard of the same size. The center section, 4.5" X 40.25" was removed with 30 wooden clothespins secured every 1.5" along the length of each side. Each fixture secured 30 samples between the clothespins. Six of these fixtures were required to provide the 180 test samples. Each unit was hung directly beneath normal fluorescent 40 Watt light fixtures at a distance of 12". A constant temperature of 72°F was maintained while the relative humidity was a constant 45 percent. Samples were exposed to lighted conditions for an average of 70 hours per week for the duration of the study.

CONTROL

A set (180) of control samples were kept in a darkened environment at 72°F while the relative humidity was 45 percent. A total of 180 control samples were prepared such that each week at each processing setting the exposed samples could be compared to an equal number of control samples.

MATERIAL TESTING

An Instron Model 1011 was used in conjunction with ASTM 882 and the testing of the thin film. As per ASTM 882 the Instron provided a device for recording the tensile load and the amount of separation of the grips. As per ASTM 882 the rate of separation of the jaws was to be uniform and capable of adjustment from approximately 0.5 to 20.0 inches per minute. The rate of grip separation used in this test was 12.0 inches/minute.

This was the accepted rate of grip separation used in related industry tests. Data collected consisted of load, load extension, and load at break. All data was analyzed and plotted using a PC based statistics package. Numerical analysis consisted of analysis of variance, hypothesis testing, regression, and statistical process control procedures.

SUMMARY

Six samples were prepared for each processing parameter per each week. Averages and standard deviations were calculated for each set of six samples. The averages were arranged in a matrix for each of the lighting conditions. The results of each test consisted of load, load at break and elongation at break. It was found that for our purposes elongation at break was the most valuable. The matrices representing elongation at break had a vertical axis of the five processing parameters while the horizontal axis was represented by weeks 1 through 6.

This method of data representation allowed for analysis of within processing parameter variation and differences, between lighting condition variation and differences through the use of analysis of variance, linear regression, and statistical process control methodology.

RECOMMENDATIONS

1. The effect of the natural weather would be valuable to the study. A roof top fixture integrated with this study is essential.

2. A chemical analysis of the film would be beneficial. Any or all of the following would bring in valuable information.

    Infrared Spectroscopy
    X-Ray Defraction
    UV Spectrophotometry
    Gas Chromatography

3. A dedicated compressed air source would eliminate any variation in air supply to the blown bubble and further reduce processing variation.

4. Replicate this test using UV-B lamps in the Q-panel.

5. Six weeks as the exposure time using normal fluorescent lights needs to be extended. Perhaps a separate study emphasizing this portion could be further developed.

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WEEK NUMBER	1	2	3	4	5	6
Q-PANEL	30	30	30	30	30	30
TOTAL DARKNESS	30	30	30	30	30	30
OFFICE LIGHT	30	30	30	30	30	30

TABLE 1.-QUANTITY OF SAMPLES REQUIRED PER WEEK/ENVIRONMENT

Melt Index	2.0
Density	.922
Coefficient of Friction	0.20
Slip Additive	**Med**
Antiblock Additive	**Low**

Outstanding Properties
- ●Excellent Processability
- ●Excellent Drawdown
- ●Good Toughness
- ●Good Optical Properties

Fabrication Conditions
Tubular Film Extrusion
Melt Temperature .... 370°F/188°C
Blow-up Ratio 2.25:1
Optimum Gauge Range 0.8-2.0 mils

Physical Properties[1]	**ASTM Method**	**Value**
Melt Index, gms/10 min	D 1238	2.0
Density, gms/cc	D 792[2]	.922
Vicat Softening Point, °F/°C	D 1525	207/97
Tensile Yield†, psi	D 638	1625
Ultimate Tensile†, psi	D 638	1375
Ultimate Elongation†, %	D 638	630
Tensile Modulus†, 2% secant, psi	D 638	34,000
Film Properties	**1.5 mil**	
Dart Impact, gms	D 1709	130
Elmendorf Tear Strength, gms		
Machine Direction	D 1922	85
Cross Direction	D 1922	175
Tensile Yield, psi		
Machine Direction	D 882	1475
Cross Direction	D 882	1440
Ultimate Tensile, psi		
Machine Direction	D 882	2930
Cross Direction	D 882	2725
Ultimate Elongation, %		
Machine Direction	D 882	470
Cross Direction	D 882	585
Transparency, %	D 1746	37
Gloss, 45°	D 2457	66
Haze, %	D 1003	6.5
Heat Seal Range, °F		250-350

†Compression molded samples

[1] These typical property values are intended as guides only, and not as specification limits.
[2] Method B Dow modification.

TABLE 2.-DOW POLYETHYLENE RESIN 533

RUN ONE

BARREL 1	BARREL 2	BARREL 3	DIE 1	DIE 2
288	336	346	356	361

SCREW SPEED==> 25 RPM

MELT PRESSURE==> 800 psi

AIR RING PRESSURE==> 43 psi

AIR RING OPENING==> 2

NIP SPEED==> 20%

NIP TORQUE==> 100%

WIND SPEED==> 40%

WIND TORQUE==> 85%

FILM WIDTH==> 5"

FILM THICKNESS==> .0021"

TABLE 3.-PROCESSING CONDITION AT MANUFACTURERS RECOMMENDED TEMPERATURE SETTINGS MINUS 4 PERCENT

RUN TWO

BARREL 1	BARREL 2	BARREL 3	DIE 1	DIE 2
294	343	353	363	368

SCREW SPEED==> 30 RPM

MELT PRESSURE==> 755 psi

AIR RING PRESSURE==> 51 psi

AIR RING OPENING==> 2

NIP SPEED==> 20%

NIP TORQUE==> 100%

WIND SPEED==> 40%

WIND TORQUE==> 85%

FILM WIDTH==> 5"

FILM THICKNESS==> .0023"

TABLE 4.-PROCESSING CONDITIONS AT MANUFACTURERS RECOMMENDED TEMPERATURE SETTINGS MINUS 2 PERCENT

RUN THREE

BARREL 1	BARREL 2	BARREL 3	DIE 1	DIE 2
300	350	360	370	375

SCREW SPEED==> 35 RPM

MELT PRESSURE==> 850 psi

AIR RING PRESSURE==> 62 psi

AIR RING OPENING==> 2

NIP SPEED==> 20%

NIP TORQUE==> 100%

WIND SPEED==> 40%

WIND TORQUE==> 85%

FILM WIDTH==> 5"

FILM THICKNESS==> .0023"

TABLE 5.-PROCESSING CONDITIONS AT MANUFACTURERS RECOMMENDED TEMPERATURE SETTINGS

RUN FOUR

BARREL 1	BARREL 2	BARREL 3	DIE 1	DIE 2
306	357	367	377	383

SCREW SPEED==> 40 RPM

MELT PRESSURE==> 850 psi

AIR RING PRESSURE==> 74 psi

AIR RING OPENING==> 2

NIP SPEED==> 30%

NIP TORQUE==> 100%

WIND SPEED==> 65%

WIND TORQUE==> 85%

FILM WIDTH==> 5"

FILM THICKNESS==> .0024"

TABLE 6.-PROCESSING CONDITIONS AT MANUFACTURERS RECOMMENDED TEMPERATURE SETTINGS PLUS 2 PERCENT

RUN FIVE

BARREL 1	BARREL 2	BARREL 3	DIE 1	DIE 2
312	364	374	385	391

SCREW SPEED==> 45 RPM
MELT PRESSURE==> 850 psi

AIR RING PRESSURE==> 78 psi
AIR RING OPENING==> 3.5

NIP SPEED==> 30%
NIP TORQUE==> 100%

WIND SPEED==> 65%
WIND TORQUE==> 85%

FILM WIDTH==> 5"
FILM THICKNESS==> .0024"

TABLE 7.-PROCESSING CONDITIONS AT MANUFACTURERS RECOMMENDED TEMPERATURE SETTINGS PLUS 4 PERCENT

A-216

A - CONTROLLED CONDITION
  A - OFFICE LIGHT
  B - Q PANEL (Accelerated UV Exposure)
  C - TOTAL DARKNESS
2 - WEEK NUMBER
1 - PROCESSING PARAMETER
6 - SAMPLE NUMBER

TABLE 8.-ALPHA-NUMERIC LABELING PROCEDURE USED IN PHOTODEGRADABILITY STUDY

TITLE: DEMONSTRATION OF A SIMPLE SCREENING STRATEGY FOR MULTIFACTOR EXPERIMENTS IN ENGINEERING

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INTRODUCTION

One of the more serious problems in engineering today is the widespread use of poorly designed experiments in engineering investigations. Most of us use the strategies learned in our college lab courses, but these are often ill-suited for engineering research. In college labs, as in most basic research, a well-defined theoretical idea is being investigated and this greatly simplifies the task of data interpretation. We use the data used to obtain best fit values for the parameters in our favorite theoretical formulas, but rarely consider how well alternative theories and their formulas might have explained the data. Usually such alternatives will contain other independent variables and this could greatly expand the scope of the investigation, not to mention the level of confusion. Indeed most projects in basic research and college labs are designed to be narrow in scope for precisely these reasons.

In engineering studies almost none of the above applies. Often there is no well-defined theory or formula for relating the variables one can control to the responses one hopes to influence. In some cases it turns out that none of the factors that we can control will significantly influence the important responses in the process of interest. Moreover, we often have to consider a large number of candidate control variables, at least in the early stages of the study, and the experimental strategies used in college labs do not teach one how to efficiently sort the "vital few" controllable variables from the "trivial many," particularly when no theory or well-defined fitting formula are available.

Fortunately, some very efficient, statistically-based strategies exist which can handle these kinds of problems, but engineers are gaining little or no exposure to them during their college years. This is probably because their mentors are not well versed in them either. At present, almost all the engineers using statistically designed methods of experimentation have learned them after graduating from college and probably at considerable expense to their employer. We hope this will change soon and that this paper helps speed the transition.

THE PAPER HELICOPTER EXPERIMENT: ITS FEATURES AND PURPOSE

Described below is an easy to run project called the "paper helicopter experiment."[1] We think it originated with Mr. C. B. "Kip" Rogers, a consultant quality engineer with Digital Equipment Corp., Maynard MA 01754, who conceived it as a teaching tool for courses in the design of experiments.

We chose it because it is simple, quick, and inexpensive to run and because it entails many of the bewidering experimental problems encountered in engineering studies. There is no "pat answer" that can be looked up in a physics book and,in our version, a total of eight controllable variables are to be sorted into the vital few as opposed to the trivial many. (Instructors can easily modify the project, however, so as to either shrink or expand the number of candidate control factors.) In real life, these control factors may act alone or in concert with one or more of the others to dramatically affect the response variable; in such cases we speak of interaction effects. Interaction effects are investigated after sorting, especially when four or more control factors are in the offing, so we willleave the treatment of interactions for another time. Also, only one response variable-- the flight timeupon dropping the helicopter-- will be considered here, tough we shall comment later on how to simultaneously monitor others as well.

The sorting strategy employed here is called a linear screening design. Its primary purpose is to distinguish the most important control variables from those which are of such minor importance that studying them any further would (probably) be a waste of time and effort.

EXPERIMENTAL PROCEDURE

Figure 1 shows a small drawing of a typical helicopter, along with a generic pattern for making the crafts. Only four of the eight control factors are clearly described in figure 1; namely, Body Width (B), Body Length (L), Wing Length (W) and Fold (F). Whereas these all change in value from craft to craft, the dimensions shown as 3" and 8.5" on Figure 1 are kept the same on all the crafts. (We chose 3" and 8.5" only because they happen to fit conveniently on 8.5" x 11" paper. However, other values could certainly be substituted if desired, or if another size of paper is used.) In addition, we also included four other factors as follows: Paper Type (P), Tape on the Body (T) and Tape on the Wings (M). Table 1 lists all eight of the design (control) factors and gives the high and low settings we used for each.

The design matrix of Table 2 lists all sixteen combinations of control settings used in our version of this experiment. For example,craft #1 is made with the settings given in row #1 of the design matrix, craft #2 is made with those of row #2 and so on for all 16. Because we are only doing a linear screening experiment, each variable has only two settings; one high and one low, which are designated respectively as +'s and -'s in the table. There is one helicopter for each trial in the design and we recommend that each team construct all sixteen and number them accordingly. That way the results of the various teams can be compared and disussed afterwards.

Figures 2 (A) through (C) can be photocopied as templates to save time. Together they contain all the unique patterns needed for this experiment; this is eight in total. One set is fabricated from regular paper and the other from construction paper, bringing the grand total to sixteen. Each pattern has two numbers associated with it, one odd and one even. The odd numbered crafts are fashioned from regular paper (-) and the evens from construction paper (+). So much for the P factor column of Table 2.

The next three columns-- B, L and W-- should be fairly obvious in that they only involve cutting and folding to get the appropriate high (+) and low (-) settings. The last four factors, however, may need a little further explanation. We added a paper clip at the bottom or foot of the body, essentially as a means of increasing the weight of the craft. So, a "+" in the W column means "add a paper clip," whereas a "-" means "no paper clip". A "+" in the F column means fold the bottom inch of the body upwards and tape it in place. (We attached a similar piece of tape to each body whether or not it has been folded to avoid a possible confounding effect between the fold and weight variables.) A "+" in the Taped Body or the Taped Wing columns simply means the body or wing was stiffened by covering it with scotch tape.

COLLECTING THE RESPONSE DATA

When all sixteen crafts are in hand, the teams are ready to collect their data. Again, we recommend labeling the helicopters from 1 - 16 at the outset. They are then to be dropped (from the same height!) and timed, but in a randomized order. Randomization is important for it randomly distributes the noise from background or uncontrolled factors among the various high and low settings. This minimizes the possibility that chance noise variations will contribute systematically to one or more of the main effects. Because the runs were so easy and inexpensive to execute, we dropped each helicopter five times and used the average of the five flight times as the response value. The best place to enter each response value is to the immediate right of the appropriate row in the design matrix of Table 2. (This will be the row whose number also appears on the helicopter being dropped.) These entries will form a column and should be labled "response" or "ave. t" as was done in Table 2. We suggest distributing photocopies of Table 2, with our response values blanked out, for use as data recording sheets.

Incidentally, it should now be clear how one can follow any number of responses simultaneously. Had we wished to also study, say, the variation in flight time as a second response variable, we could have calculated the standard deviation for each sample of five drops and entered that value under a second response column named "S.D. 5 DROPS". It would be located to the immediate right of the "AVE. 5 DROPS" column of our Table 2. We might also have considered a kind of signal-to-noise ratio as yet another response variable. To do this, we would simply add a third response column to receive the ratios-- "AVE. 5 DROPS" divided by the "S.D. 5 DROPS"-- as could be readily calculated from entries in the previous two response columns. Clearly any number of other response variables can be added in a similar fashion, but in our version of the experiment, we considered only the average flight time so that only one response column appears in our Table 2.

ANALYSIS OF THE RESPONSE DATA

After all the response values have been entered, there begins the task of evaluating the effect that each control factor has on the flight time response. Our detailed calculations are given in Table 3.[2] To begin the analysis, pick any particular control factor, say L, and merely add all the response values for the high (+) settings-- for L this would be eight runs (5 through 8 and 13 through 16) as can be seen from Table 2-- and then divide by the number of +'s or eight in this case. This gives a kind of "average of the highs". Subtracting from this the same kind of "average of the lows (-)" gives the main effect of control factor L on the flight time response. It is the difference .142 in Table 3.

For the variable P, eight other runs (helicopters) have P set high (+) and eight with P low (-). Hence to get the overall effect on flight time of changing P from low to high, we proceed as before: simply add all the responses with P high and divide by eight, then do the same for all the helicopters with P low and then subtract the latter average from the former. For our data, the effect of P on flight time turned out to be the difference of .011 in Table 3.

When the effects for all the control variables have been calculated in this fashion, a so-called scree plot (a kind of "Pareto Diagram", actually) is constructed with the effects plotted in descending order. "Scree" is from a Scandinavian word meaning "rock slide" or "pebble debris." The "vital few" factors are those located at the upper left, while the "trivial many" show up as the "scree" points or "debris" at the lower right. The scree plots of Figure 3 were constructed from effects as calculated in Table 3. In fact the heights of the solid black bars in Figure 3 are precisely equal to the "diff." values reported in Table 3, all of which are based on five-run samples. The other bars in Figure 3 are included only to show that the scree plots didn't change much when we cut the number of drops per sample back to three, two and even to one. (None of the data analyses from those additional experiments have been included here.)

For us, the most significant control factor by far was the wing length, with taped body being the next most important. All the other factors are clearly relegated to the debris or "scree" part of the plot.

CONCLUDING REMARKS

Once the vital few control factors have been identified as outlined above, one may go on to consider a more detailed study. Does the response exhibit any nonlinear dependences on the control factos? Are there significant interaction effects among the most important control variables? We prefer to use ECHIP--a software package of computer-assisted response surface methods[3]-- for handling the various nonlinear and interaction effects that can show up. Computer assistance is recommended because such analyses require rather advanced and time consuming algorithms and these, too, are well beyond the intended scope of this paper. However it should be noted that if one were to attempt to ferret out such effects before reducing the control factors from eight to our vital two, the number of trials (helicopters or flights in this case) could prove too much even for the most sophisticated software packages.

One final point. Had the effort and expense of gathering all the flight time data been beyond our research budget, we might have considered using fewer flights for each helicopter. After all, Figure 2 shows that cutting back the number of flights, at least in this experiment, would not have changed the conclusions we reached as to which control factors are the most important. However, there are penalties to be paid for doing this. For example, one could totally undermine certain tests of statistical significance that statisticians consider to be very important. Such significance tests, however, are also beyond the intended scope of this paper.

REFERENCES

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TABLE 1. EIGHT CONTROL FACTORS WHOSE EFFECTS ARE TO BE STUDIED

Variable	Low setting (-)	High Setting (+)
1) Paper (P)	Regular	Construction
2) Body Width (B)	1"	1.5"
3) Body Length (L)	1.5"	3"
4) Wing Length (W)	2"	4"
5) Paper Clip (C)	no	yes
6) Fold (F)	no	yes
7) Taped Body (T)	no	yes
8) Taped Wing (M)	no	yes

TABLE 2. THE RESPONSE VALUES AND DESIGN MATRIX OF SIXTEEN UNIQUE COMBINATIONS OF THE CONTROL FACTOR SETTINGS -- ONE FOR EACH HELICOPTER USED --

Number	P	B	L	W	C	F	T	M	RESPONSE AVE. 5 DROPS
1	-	-	-	-	-	-	-	-	1.474
2	+	-	-	-	-	+	+	+	1.102
3	-	+	-	-	+	-	+	+	1.084
4	+	+	-	-	+	+	-	-	1.260
5	-	-	+	-	+	+	+	-	0.928
6	+	-	+	-	+	-	-	+	1.232
7	-	+	+	-	-	+	-	+	1.310
8	+	+	+	-	-	-	+	-	1.306
9	-	-	-	+	+	+	-	+	2.078
10	+	-	-	+	+	-	+	-	1.808
11	-	+	-	+	-	+	+	-	2.006
12	+	+	-	+	-	-	-	+	2.122
13	-	-	+	+	-	-	+	+	1.530
14	+	-	+	+	-	+	-	-	1.988
15	-	+	+	+	+	-	-	-	2.000
16	+	+	+	+	+	+	+	+	1.502

TABLE 3. TABULATION OF HIGH VS. LOW SETTINGS

P HIGH = (2+4+6+8+10+12+14+16)/8

(1.102+1.260+1.232+1.306+1.808+2.122+1.988+1.502)/8=1.540

P LOW =(1+3+5+7+9+11+13+15)/8 DIFF = .011

(1.474+1.084+0.928+1.310+2.078+2.006+1.530+2.000)/8=1.551

B HIGH = (3+4+7+8+11+12+15+16)/8

(1.084+1.260+1.310+1.306+2.006+2.122+2.000+1.502)/8=1.574

B LOW = (1+2+5+6+9+10+13+14)/8 DIFF = .056

(1.474+1.102+0.928+1.232+2.078+1.808+1.530+1.988)/8=1.518

L HIGH = (5+6+7+8+13+14+15+16)/8

(0.928+1.232+1.310+1.306+1.530+1.988+2.000+1.502)/8=1.475

L LOW = (1+2+3+4+9+10+11+12)/8 DIFF = .142

(1.474+1.102+1.084+1.260+2.078+1.808+2.006+2.122)/8=1.617

W HIGH = (9+10+11+12+13+14+15+16)/8

(2.078+1.808+2.006+2.122+1.530+1.988+2.000+1.502)/8=1.879

W LOW = (1+2+3+4+5+6+7+8)/8 DIFF = .667

(1.474+1.102+1.084+1.260+0.928+1.232+1.310+1.306)/8=1.212

C HIGH = (3+4+5+6+9+10+15+16)/8

(1.084+1.260+0.928+1.232+2.078+1.808+2.000+1.502)/8=1.487

C LOW = (1+2+7+8+11+12+13+14)/8 DIFF = .118

(1.474+1.102+1.310+1.306+2.006+2.122+1.530+1.988)/8=1.605

F HIGH = (2+4+5+7+9+11+14+16)/8

(1.102+1.260+0.928+1.310+2.078+2.006+1.988+1.502)/8=1.523

F LOW = (1+3+6+8+10+12+13+15)/8 DIFF = .048

(1.474+1.084+1.232+1.306+1.808+2.122+1.530+2.000)/8=1.570

T HIGH = (2+3+5+8+10+11+13+16)/8

(1.102+1.084+0.928+1.306+1.808+2.006+1.530+1.502)/8=1.408

T LOW = (1+4+6+7+9+12+14+15)/8 DIFF = .275

(1.474+1.260+1.232+1.310+2.078+2.122+1.988+2.000)/8=1.683

M HIGH = (2+3+6+7+9+12+13+16)/8

(1.102+1.084+1.232+1.310+2.078+2.122+1.530+1.502)/8=1.495

M LOW = (1+4+5+8+10+11+14+15)/8 DIFF = .101

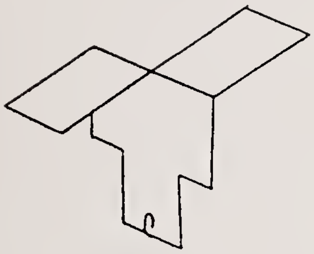
(1.474+1.260+0.928+1.306+1.808+2.006+1.988+2.000)/8=1.596

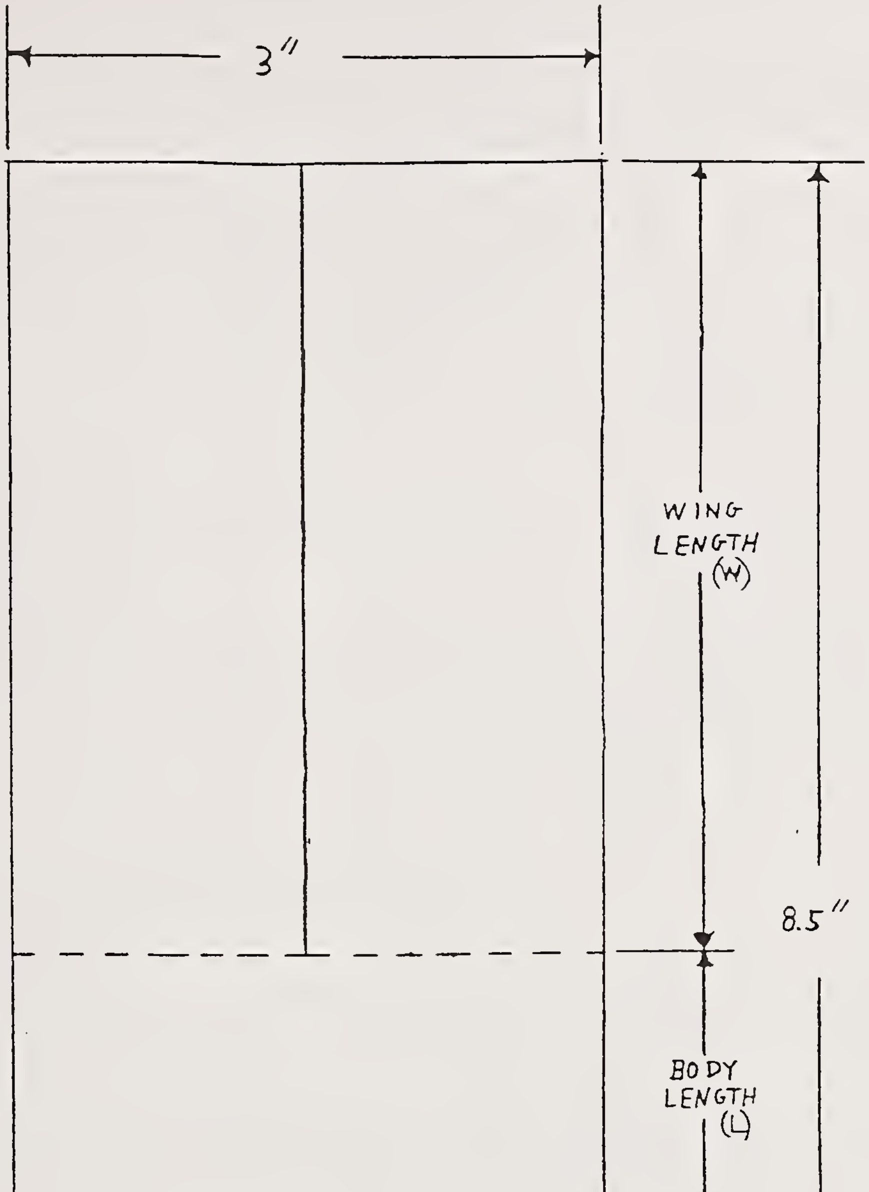
FIGURE CAPTIONS

FIGURE 1. DRAWING AND GENERALIZED TEMPLATE FOR THE HELICOPTERS

FIGURE 2. TEMPLATES FOR CONSTRUCTING ALL SIXTEEN HELICOPTERS

FIGURE 3. SCREE PLOTS FOR VARIOUS NUMBERS OF DROPS PER SAMPLE





--- FOLD
— CUT

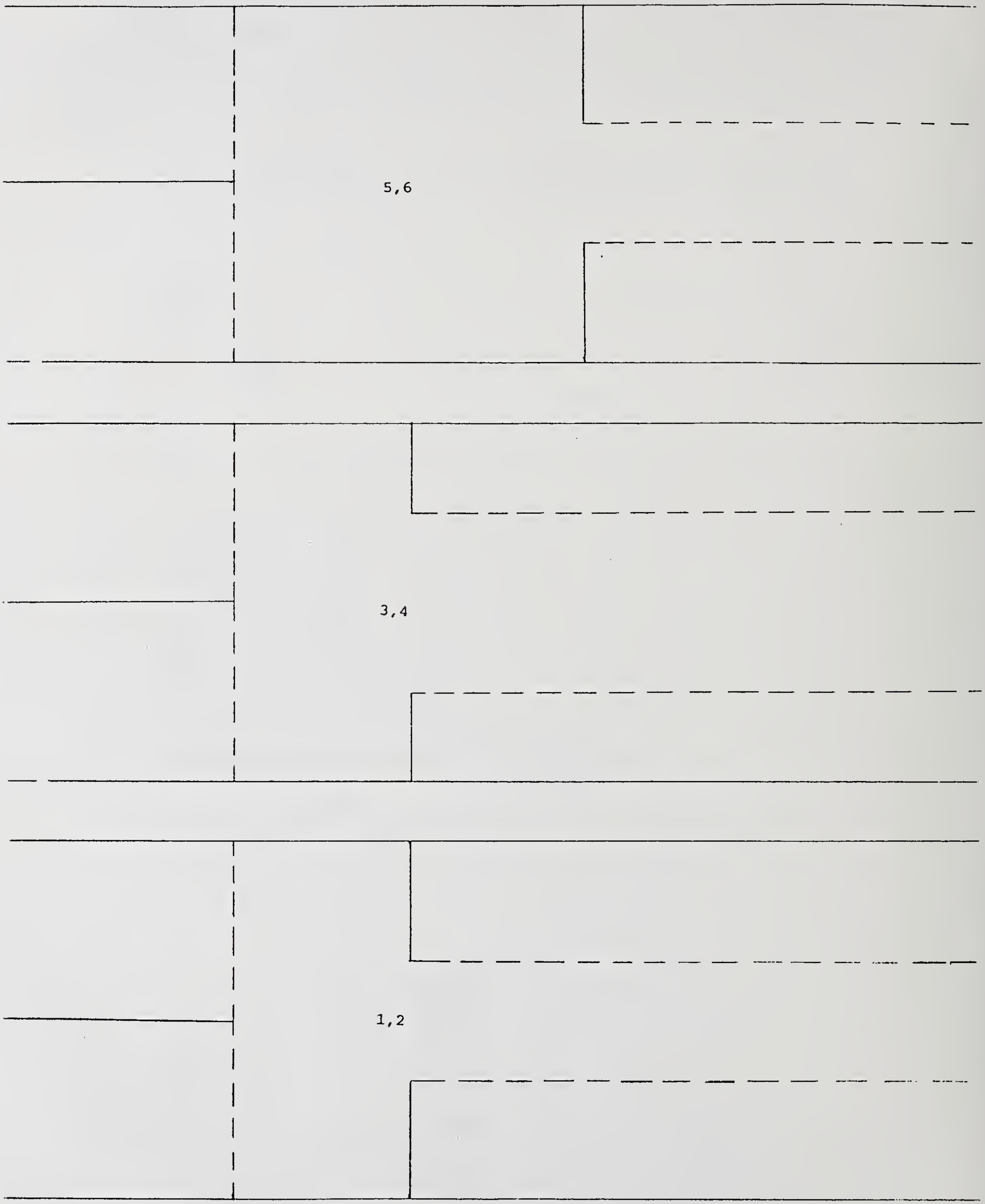


FIGURE 2(A)
(Reduced 80%)

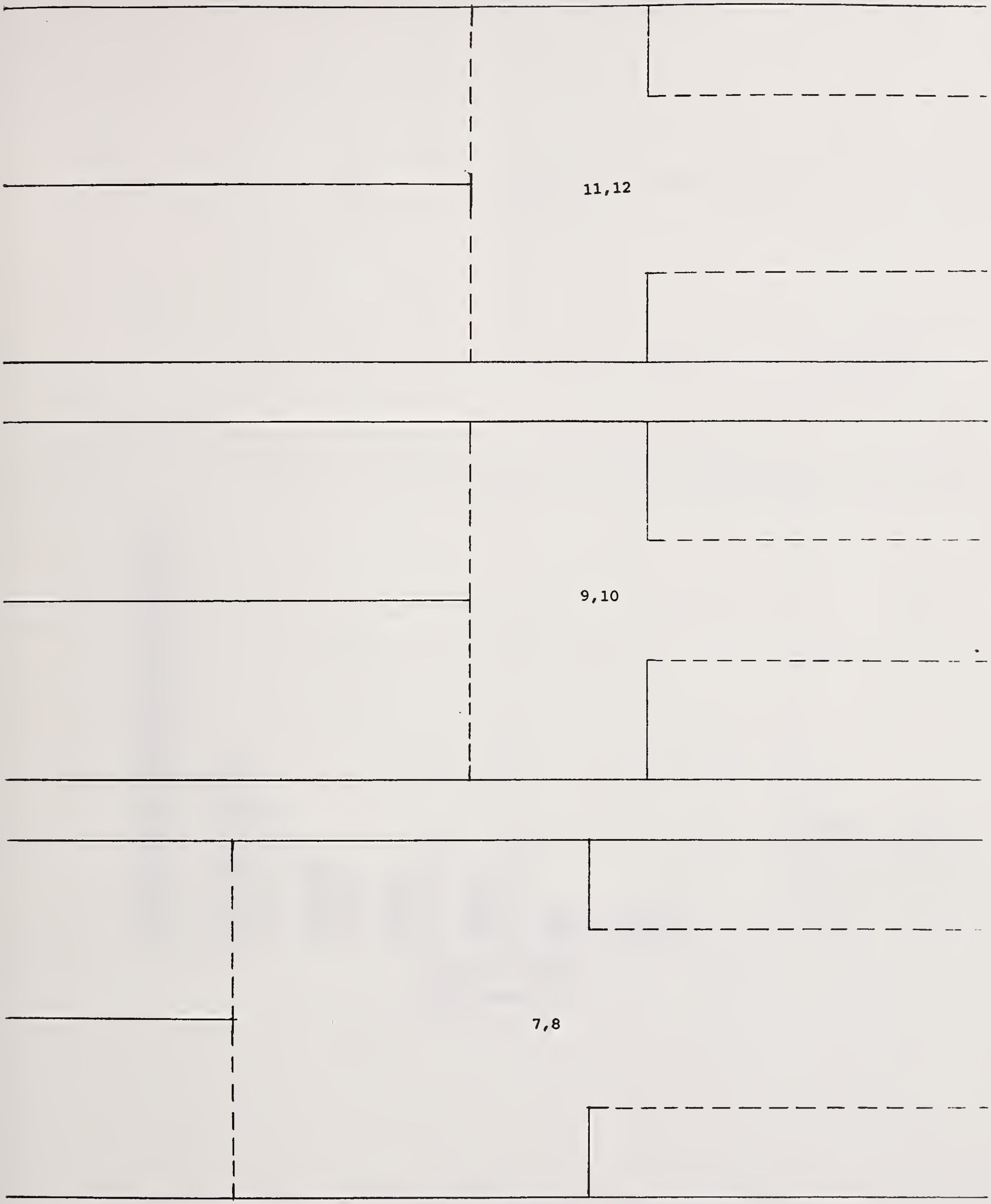


FIGURE 2(B)
(Reduced 80%)

FIGURE 2(C)
(Reduced 80%)

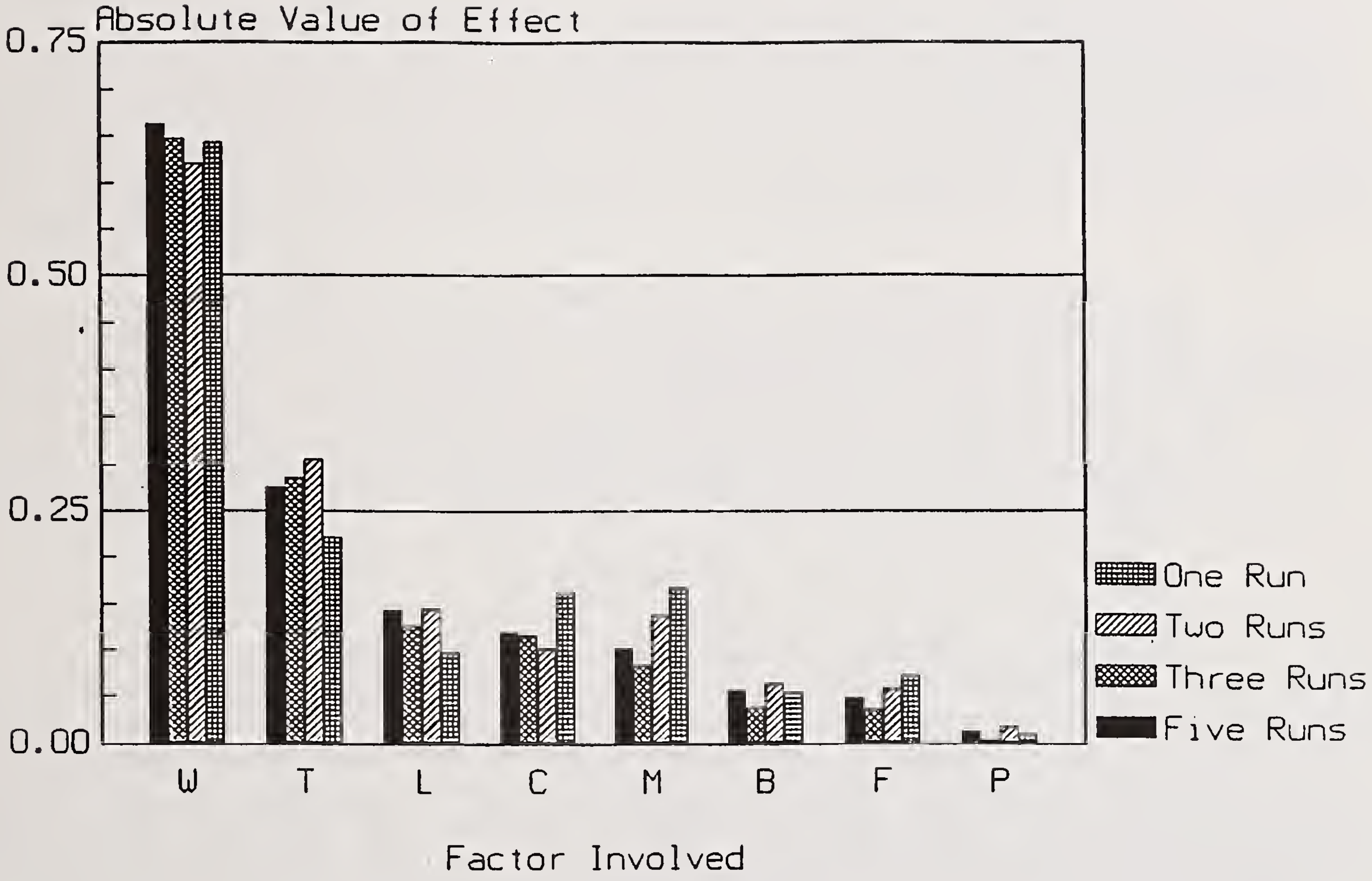


FIGURE 3. SCREE PLOTS FOR VARIOUS NUMBERS OF DROPS PER SAMPLE

ENVIRONMENTAL STRESS CRACKING OF RECYCLED THERMOPLASTICS

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SUMMARY

The high processing temperature of polymers cause what is called thermo-oxidative degradation which results in the rapid decrease in molecular weights. Due to various processing parameters all materials in fabricated form contain microscopic defects in the form of voids, contamination, or material discontinuities and grain boundaries. Testing for environmental stress cracking involves inducing mechanical stress in the test specimens under a controlled environment in the presence of specific reagents. Application of stress causes localized failures at the defects, which acts like a stress concentrator and promotes crack propagation.

The purpose of this experiment is to investigate the environmental stress cracking characteristics of recycled thermoplastics. Two types of polycarbonate materials were selected for investigation, one plain polycarbonate and one 30% glass fiber reinforced polycarbonate (abbreviated as PC). The degree of environmental stress cracking is expressed by the flexural strength with the level of induced stress as a parameter.

INTRODUCTION

As the polymer degrades to a lower molecular species, it is weakened to the point where it will no longer stand the load and will fail catastrophically. Mechanical properties such as flexural strength and impact resistance deteriorate drastically with repeated processing. Test specimens were molded according to DIN 53449 and stress was induced simply by pressing an oversized steel ball into a drilled hole in the specimen. Tests were performed under controlled conditions. The damage to the specimens caused by a specific reagent was determined by the flexural strength of the treated specimens.

Test specimens of a plain PC and a 30% glass fiber reinforced PC were molded at molding temperature of 300°C and 310°C. Then the specimens were tested and compared for their sensitivity towards environmental stress cracking. Variations in the stress level demonstrate the relationship of thermal damage to molecular chains with molding temperatures.

TESTING OF FLEXURAL STRENGTH

The purpose of this experiment was to determine the flexural strength or the stress level at which cracks propagates to ultimate failure in presence of a reagent as a medium. The medium selected was toluol normal propanol(1:10) which simulates an extended testing period in a normal environment.

EQUIPMENT

Flexural stress tester and a mechanical device for introducing oversized balls into the test specimens

TEST SPECIMENS

50mmx6mmx4mm test specimens were molded according to DIN 53449 in an injection molding machine at molding temperatures of 300C and 310°C. A 3.0 mm diameter hole was drilled at the center of the specimen as shown in fig.1. And a 150μm oversized steel ball was introduced into the drilled hole to induce stress. Four sets of specimens were prepared as follows:

A. Plain PC molded at 300°C
   a. with 150μm oversized balls
   b. without balls
B. Plain PC molded at 310°C
   a. with 150μm oversized balls
   b. withot balls
C. 30% glass fiber reinforced PC molded at 300°C
   a. with 150μm oversized balls
   b. without balls
D. 30% glass fiber reinforced PC molded at 310°C
   a. with 150μm oversized balls
   b. without balls

TEST PROCEDURE

For every processing five specimens were tested for flexural strength. The specimens were placed on supports at both ends and load applied at the center until failure. The flexural stress was calculated using the applied load, the bending moment, and the moment of inertia of the sample.

Flexural Stress $\sigma_b = M/(I/C)$

$$I/C = bh^2/6$$

b=width, h=height, M=bending moment.

RESULTS AND DISCUSSION

One set of sample test results for only the first moldings are given in table 1 and 2. Table 1 shows the flexural test results for plain PC molded at 300°C with and without induced stress. Table 2 shows the same for plain PC specimens molded at 310°C. A summary of test results for plain PC is given in table 3 and for 30% glass fiber reinforced PC is given in table 4.

Fig 2A and 2B shows the change in flexural strength as a function of repeated processing. No.1 refers to the very first processing and no.2 refers to the second processing or the first recycle. Samples from the first molding are assumed to have 100% strength and the subsequent recycling are rated accordingly.

In case of plain PC specimens from 300°C molding temperature show gradual drop in strength. The specimens from 310°C molding temperature show some strength upto the second processing only, which suggests higher thermal damage and faster development and propagation of environmental stress cracking. Specimens without induced stress does not show any drop in strength upto the third processing or second recycle. Even at the third recycle considerable amount of strength was maintained.

Glassfiber reinforced PC suffers sharper drop in strength due to induced stress. The specimens without induced stress maintained their strength through the recycle.

Some samples failed instantly when came in contact with the reagent. Plain PC showed far more resistance to stress cracking than the reinforced type. Plain PC with induced stress (molded at 300°C) maintained 76% of its original strength after the first recycle and the reinforced PC (molded at 300°C) maintained 56% of its original strength after the first recycle.

In reinforced PC due to the presence of glass as a foreign material, the crack propagation was drastic, and the specimens could not be tested for the second recycle. Fig.3 and fig.4 show flexural strength as a function of excess ball diameter which is an indirect measure of the induced stresses.

Fig.5 shows specimens of plain PC for all four processings. Fig.6 shows the gradual progress of failure in plain PC specimens of subsequent processings and fig.7 shows a drastic development of environmental stress cracking of a sample of fourth processing even without any induced stress. This is due to the residence stresses developed during repeated processing.

In practice environmental stresses develop due to adverse environmental conditions that leads to stress cracking and eventual failure of plastic parts.

ENVIRONMENTAL STRESS CRACKING
FLEXURAL TESTS

Table 1

Makrolon 3000W
First Molding
Molding Temp 310°C
(Excess ball dia 150µm)

Specimen no.	Load(N)
1	37.28
2	25.51
3	37.28
4	37.37
5	39.24

σ_b = 42.91 MPa

(Excess ball dia 0µm)

Specimen no.	Load(N)
1	76.52
2	78.48
3	77.50
4	76.52
5	80.44

σ_b = 97.34 MPa

Table 2

Molding Temp 300°C
(Excess ball dia 150µm)

Specimen no.	Load(N)
1	80.44
2	69.65
3	70.63
4	73.58
5	79.46

σ_b = 93.18

(Excess ball dia 0µm)

Specimen no.	Load(N)
1	73.58
2	82.40
3	74.56
4	76.52
5	82.40

σ_b = 97.34 MPa

Table 3
SUMMARY OF TEST RESULTS/MAKROLON 3000W

Molding Temp 300°C	150µm excess ball dia		Without ball	
Molding	Flexural Strength(MPa)		Flexural Strength(MPa)	
1	93.18	100%	97.34	100%
2	71.11	76%	95.63	98%
3	7.36	8%	98.08	101%
4			56.40	58%
Molding Temp 310C				
1	42.91	100%	97.34	100%
2	29.42	69%	106.05	109%

Table 4
SUMMARY OF TEST RESULTS/ MAKROLON GV 30

Molding Temp 300C	(150µm excess ball dia)		(Without ball)	
Molding	Flexural Strength(MPa)		Flexural Strength(MPa)	
1	96.85	100%	111.81	100%
2	53.94	56%	109.11	98%
Molding Temp 310C				
1	90.97	100%	112.79	100%
2	36.78	40%	113.28	100%

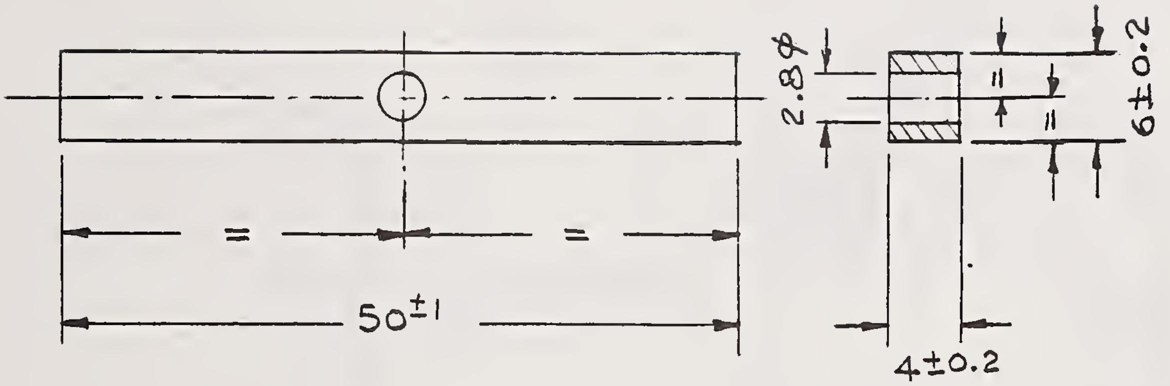


FIG 1

ENVIRONMENTAL STRESS CRACKING WITH INDUCED STRESS

PERCENT OF FLEXURAL STRENGTH MAINTAINED VS. RECYCLING

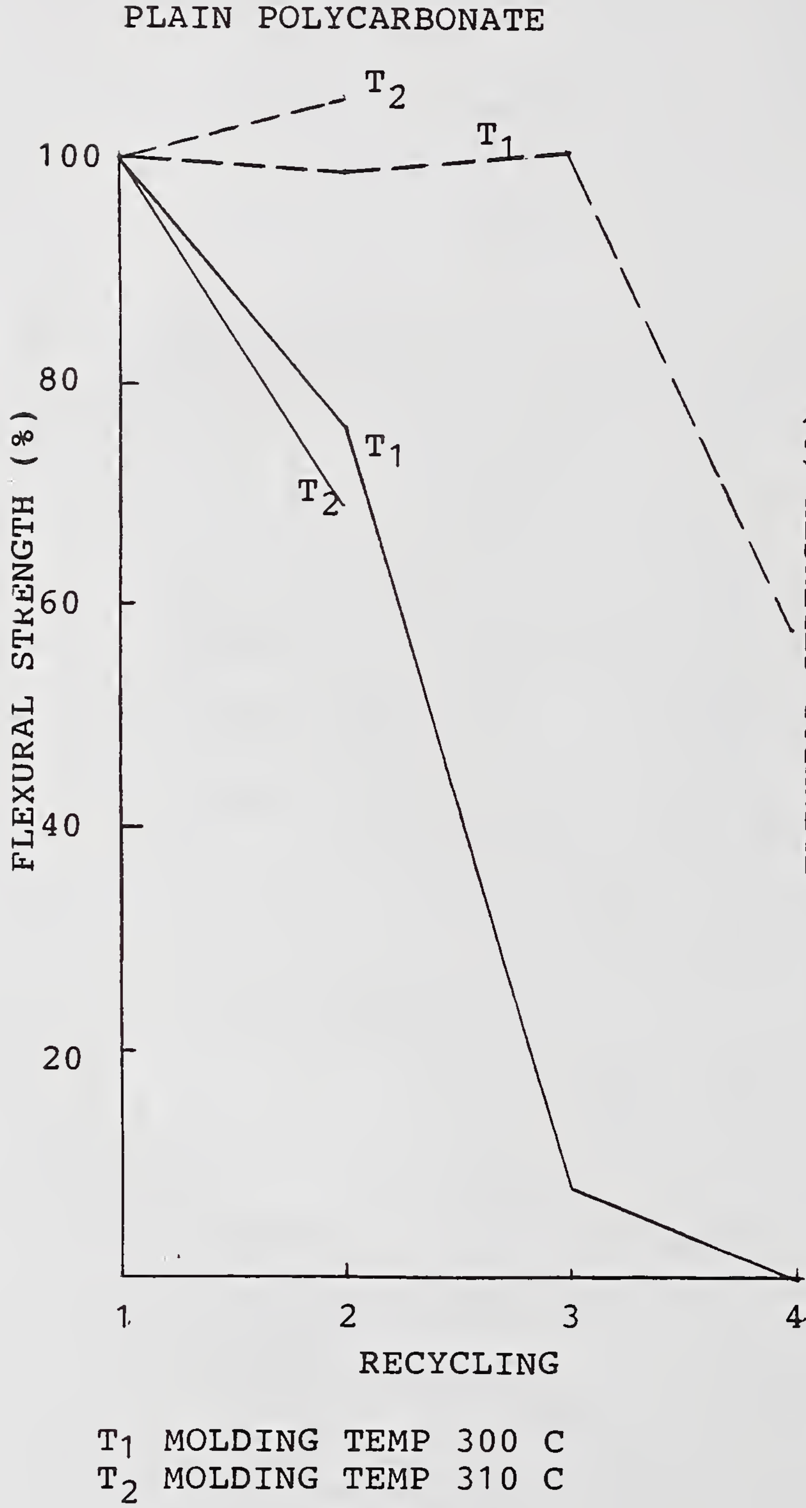


FIG 2A

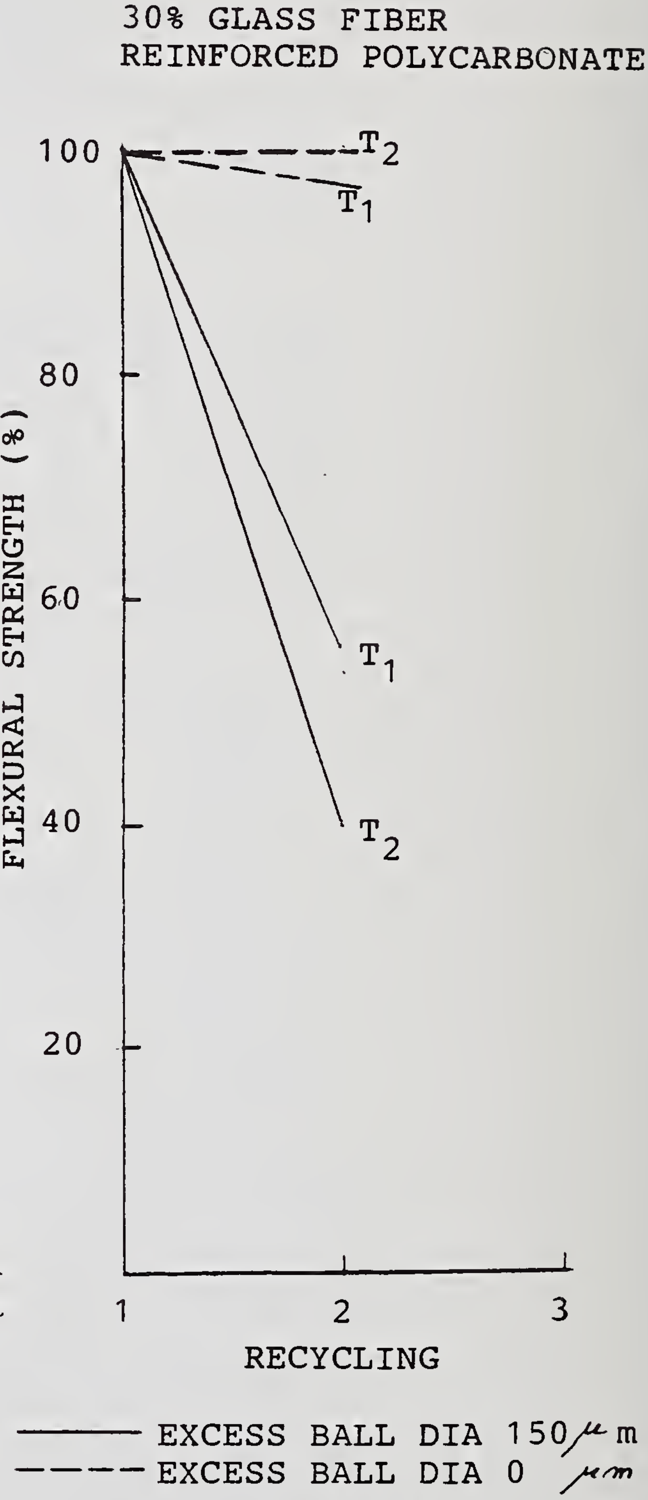
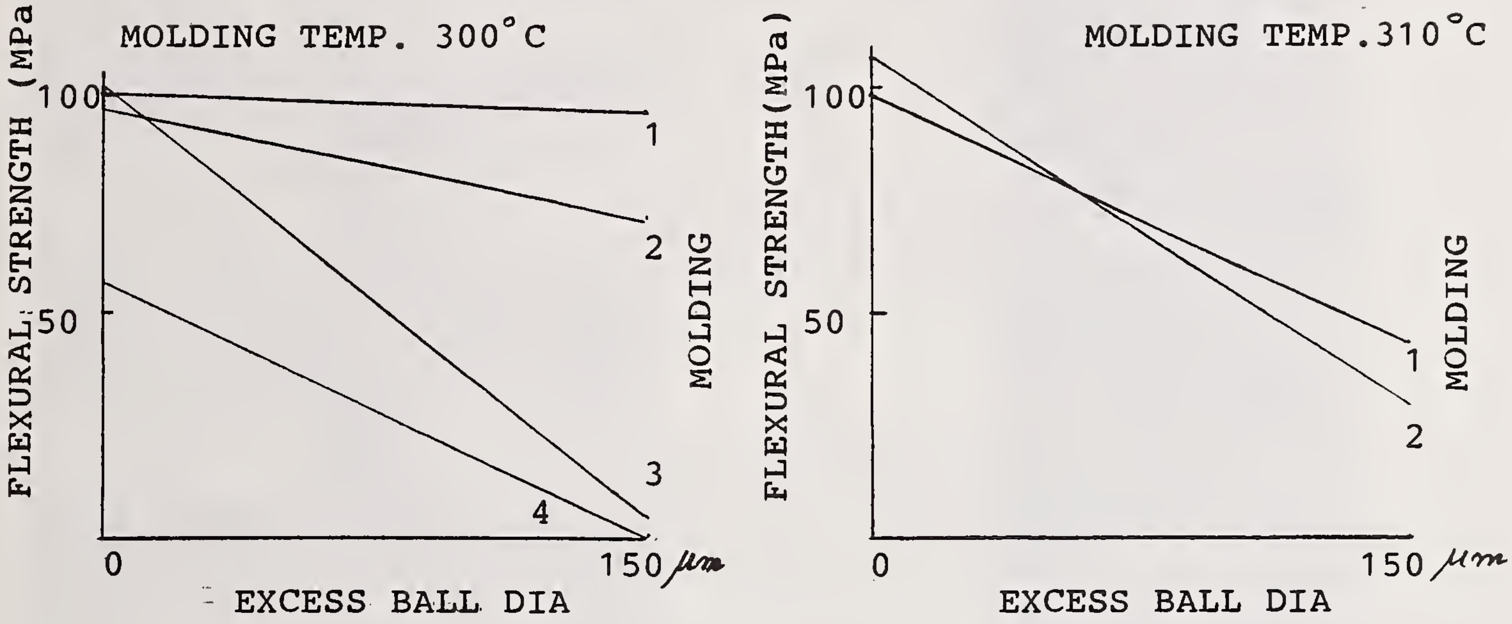
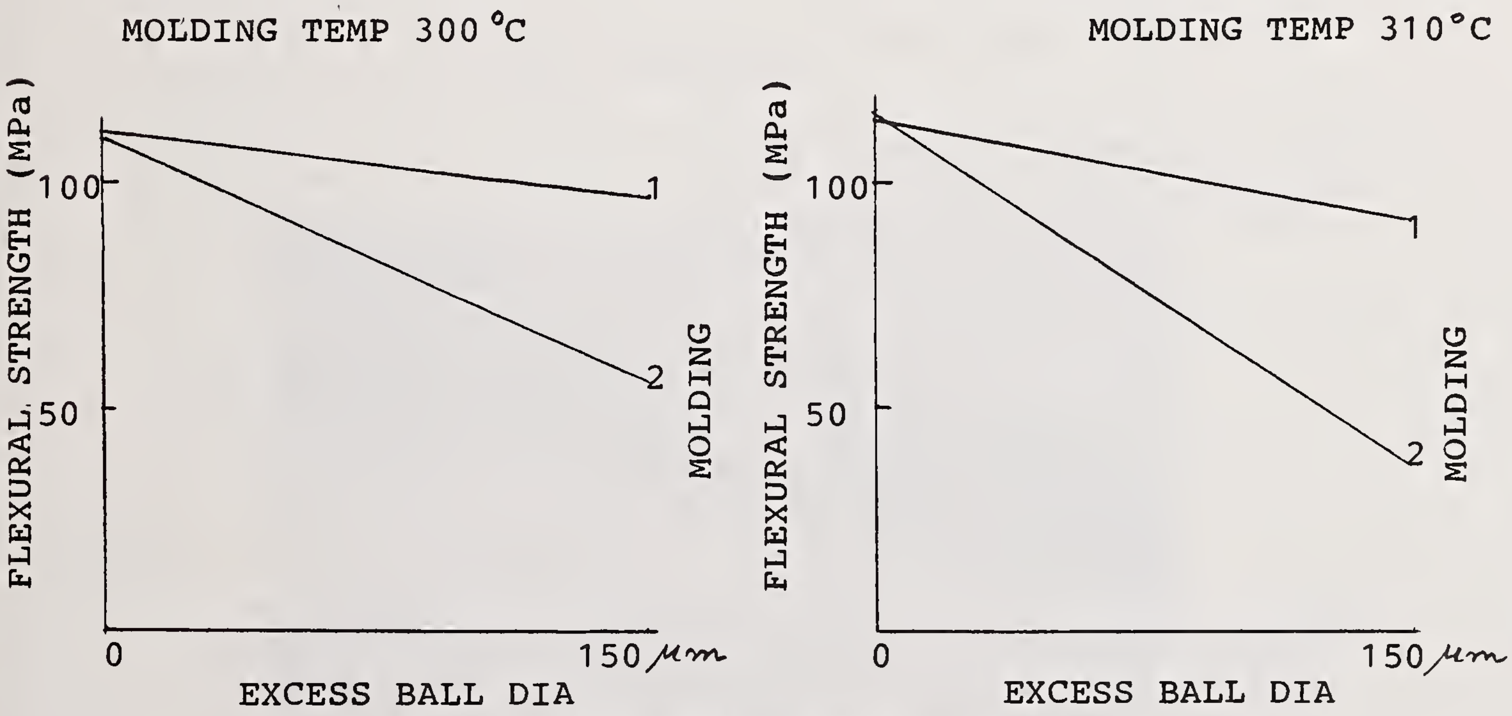


FIG 2B

FLEXURAL STRENGTH OF SPECIMENS WITH INDUCED STRESS



PLAIN POLYCARBONATE
FIG 3



30% FIBER GLASS REINFORCED POLYCARBONATE
FIG 4

FIG 5

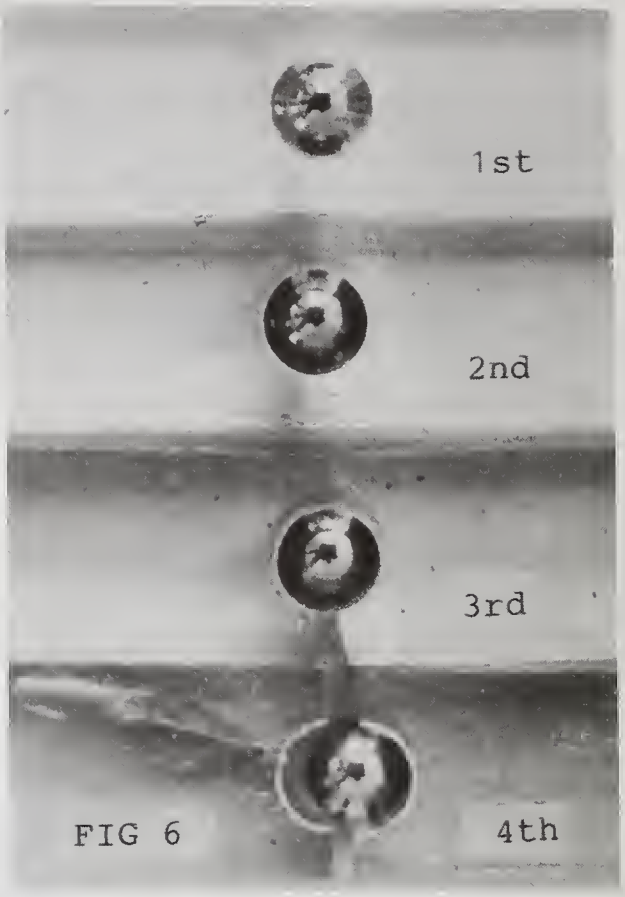


FIG 6

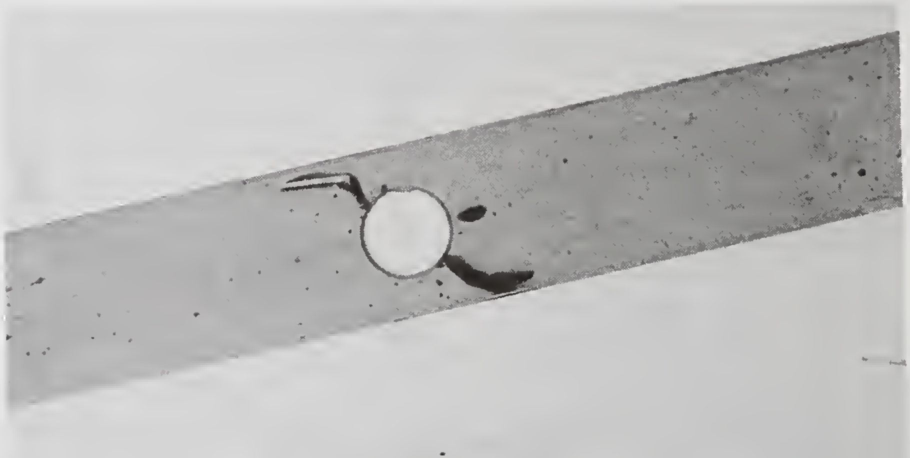


FIG 7

HOW DOES CHANGE IN TEMPERATURE AFFECT RESISTANCE?

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KEY WORDS

Resistance, conductivity as function of temperature.

PREREQUISITE KNOWLEDGE

Chemical bonding types, specifically metallic and covalent. This experiment illustrates elementary band gap theory. Familiarity of the equation relating resistivity to measured resistance is assumed.

OBJECTIVE

The two materials you are going to investigate are silicon (the shiny rod) and copper (a coil made of fine copper wire stolen from a computer corpse). Before you leave this station, you should be able to say how resistance and resistivity change with temperature for these two materials.

EQUIPMENT AND SUPPLIES

Piece of a silicon boule a few inches long with low megohms resistance.

Coil made of fine copper wire long enough to have resistance measureable on available ohmmeters.

Either a hot plate or an ice bag to change the temperature of the boule about 20 degrees C. It might be possible to cool the boule in the same medium used for the coil but the problem of making electrical contact to the wet surface is not trivial.

Both a thermos of ice water and a Dewar of liquid nitrogen.

Ohmmeter with appropriate leads

Safety glasses

PROCEDURE

1. The shiny rod is a single crystal of silicon, called a boule. Adjust the meter to measure ohms and hold the probes against the silver painted spots on the end. Be gentle, force will break the probes and not improve your measurement. You may also flake off the silver painted electrodes.

   If the area of the electrode is decreased, what happens to the measured Resistance?

The silver electrodes painted on the ends improve the contact between the Si and the probes. Whenever electrons have to leave one materials and enter another, problems can occur.

The reading should be stable and less than one megohm (substitute for actual sample here) if the T is about 20 C. (If not, call for help.)

a. Record the room temperature value of resistance and then set the boule on the hot plate, being careful not to let it roll off. (The hot plate should be only medium warm, not hot.)
b. Record the resistance at a slightly warmer temperature. It should not be necessary to heat the boule for more than a few minutes to see the relationship. (If when you get to this station, the boule is already warm to the touch, how can you adjust the procedure to suit the situation?*)

a. Room temperature value for Si boule:_________

b. Warmed boule:___________

2. Hook the meter onto the ends of the Cu wire that is wound in the coil.
   a. Adjust the meter to measure Resistance; and record the room temperature value.
   b. Carefully lower the coil into the ice water that should be available nearby and get a value of R after the coil cools to 0 C.

   PUT ON SAFETY GLASSES BEFORE PROCEEDING.

   c. Carefully lower the coil into the Dewar of liquid nitrogen, using the wires from the meter as a sky hook. Cool until the nitrogen quits boiling vigorously and the R value has stabilized. Record the R values below.

a. Room temperature R for Cu coil:_______________

b. 0 C Resistance for Cu coil: _______________

c. 77 K Resistance for Cu coil: _______________

REPORT

1. In one or two short paragraphs, explain how the resistivity of these two materials changes with temperature and explain why they change the way they do.
2. Graph the R vs. T curve for Cu using the 3 different temperatures for which you have data. Do you think this curve is truly representative?

* Take a measurement immediately and let the boule cool while you mess with the coil; then take a measurement of the cooled boule.

INSTRUCTOR NOTES

Resistance of Si boule may change because the Ag paint flakes off, readings become unstable and high.

As Area decreases, R increases.

Question about graph: not enough points.

A hair dryer is handy to warm the coil between groups.

The boule might crack if put in the liquid nitrogen.

The wires from the coil frequently break so stabilizing the coil in a plastic holder of some kind to reduce stress is advised.

Good follow up to the graphing question is to collect data from a superconductor or present R vs T data from a superconductor showing the difference of several orders of magnitude at room T with Cu lower followed by the switch to zero resistance by the superconductor. Comparison of same three points for superconductor makes the futility of three point graphs apparent.

REFERENCES

For basic theory about band gap, any introductory materials science book, such as Shackleford or Van Vlack.

An equation describing the change in R of Cu with temperature is usually found in the same text or in CRC Handbook of Chemistry and Physics.

SOURCE OF SUPPLIES

A company that works with semiconductors should be able to help locate a boule of Si. Richard Lane at Rochester Institute of Technology's Microelectronics Laboratory might be able to suggest a source. (716) 475-6911 (Don't tell him where you got his name.)

Air dry silver paint: GC Electronics, Rockford, IL 61125
Cat. #22-246.

DEMONSTRATION OF KINETIC RELATIONSHIPS BY PRECIPITATION-HARDENING EXPERIMENTS

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Abstract

The laboratory experiment on age-hardening that is included in many undergraduate metallurgy courses considers the changes in hardness that occur as Guinier-Preston zones form, grow, and eventually become noncoherent precipitates. If the solution and aging temperatures are carefully controlled, the resulting hardness/time data clearly show how mechanical properties can be manipulated by changing the microstructure of an alloy.

If we expand the experimental matrix to include additional aging temperatures, the family of hardness/time curves that results can also be used to illustrate diffusion kinetics. Aluminum alloy 2024 is particularly well suited to this scheme because aging can be retarded by storage in a freezer; natural aging starts to develop within the time limits of a laboratory session; and overaging can be developed in a simple oven. A two week sequence of laboratory instructions thus provides sufficient data for a thorough discussion and explanation of the kinetic principles underlying precipitation hardening and the more general consideration of the effect of temperature on diffusion rates.

Key Words:	Aluminum Alloy, Precipitation Hardening, Kinetic Relationships, Laboratory Experiments.
Prerequisite Knowledge:	Concurrent with classroom lectures on precipitation hardening, subsequent to laboratory and classroom discussions on cold working and hardness measurement.
Objective:	Reinforce understanding of age-hardening and demonstrate the effect of temperature on diffusion rates.
Equipment and Supplies:	Aluminum alloy 2024-T4 bar stock. Suggested dimensions - 25mm x 5mm x 3m. Quench Tank - Water Furnace for solution-treating - 650°C capability Furnaces for artificial aging - 220°C, 175°C capability Tongs and Gloves Freezer - 0°C Rolling Mill Shear or Bandsaw Abrasive Belt Grinder Hardness Testing Machines - Rockwell B scale

Procedure:

Two separate laboratory sessions are required to complete the three elements of the experiment:

> Artificially age solution-treated aluminum alloy samples, measuring their hardness after selected times,
>
> Suppress aging by holding solution-treated samples in a freezer for an extended time,
>
> Cold work solution-treated samples to accelerate the age hardening reaction.

First Session:

Prior to the session, the instructor should place four pieces of aluminum alloy (suggested dimensions 150mm x 25mm x 5mm) in the 650°C solution-treating furnace. The pieces should be in the furnace for about one hour.

Begin the session by acquainting the students with the nomenclature for aluminum alloy tempers - O, T3, T4 and T6. Then ask one of the students to take the aluminum alloy pieces from the furnace and immediately quench each one in room temperature water. As promptly as possible, one piece should be put into the freezer until the next session,

one piece should be sawed or sheared into six smaller pieces and all of these should be put into the 220°C furnace, and

one piece should be sawed or sheared into six smaller pieces and all of these should be put into the 175°C furnace.

one piece will be used to establish the hardness in the solution-treated condition and the initial portion of the natural age hardening response.

After quenching the last piece, carefully grind opposite surfaces flat with an abrasive belt grinder. Then measure the hardness of the sample in at least three places. Record the elapsed time between quenching and hardness testing, which should be mo more than five minutes. Continue to measure the hardness of the piece in at least three places at time intervals of 15, 30, 60, 90, 120 and 150 minutes after quenching. This piece should be stored by the instructor at room temperature until the next session.

Remove one sample from each of the furnaces after soaking times of 15, 30, 60, 90, 120 and 150 minutes. The samples can be cooled in water. Again, carefully grind opposite surfaces flat with an abrasive belt. Measure the hardness of each sample in at least three places.

Post each hardness measurement on a blackboard so that all participants can record and discuss the data.

Second Session:

For this session, the instructor should place three pieces of aluminum alloy (suggested dimensions 150mm x 25mm x 5mm) in the 650°C solution-treating furnace prior to the session. The pieces should be in the furnace for about one hour and then individually quenched in room temperature water. As promptly as possible,

cold roll one piece about 5%, measure its thickness after rolling, cut or saw it into six pieces and put all of them into the 220°C furnace,

cold roll one piece about 5%, measure its thickness after rolling, cut or saw it into six pieces and put all of them into the 175°C furnace,

cold roll one piece about 5%, measure its thickness after rolling,grind opposite faces flat and then measure its hardness in at least three places; make subsequent hardness measurements at intervals of 15, 30, 60, 90, 120 and 150 minutes after cold rolling.

Remove one sample from each furnace after soaking times of 15, 30, 60, 90, 120 and 150 minutes, cool it in water, grind opposite surfaces flat and then measure the hardness in at least three places.

Retrieve the sample that has been held in the freezer for one week, warm it to room temperature by immersing it in water, grind opposite surfaces flat and then measure the hardness in at least three places immediately and after 15, 30, 60, 90, 120 and 150 minutes.

Measure the hardness of the sample from the previous session that was held by the instructor. This sample has now been naturally aged one week.

Post all hardness measurements on a blackboard so that all participants can record the data and discuss the trends.

Student's Reports:

Summary

Description of the Experiment

Draw a simple block diagram flowsheet that shows the steps used to develop each temper, including the grinding and the hardness measurements. Explain each step carefully. Include a discussion of

phase solubility changes with temperature,

the change in solute atom diffusion rate that occurs as temperatures are lowered, and

the effect that work hardening has on the age hardening reaction.

This discussion should reference the textbook and other selected handbooks and journals that may be available.

Analysis of Data

Construct a graph that shows each hardness reading on the ordinate and the logarithm of the elapsed time on the abscissa. Draw a curve through the family of points that represents each aging treatment.

Conclusion

Note the time required to achieve peak hardness for each aging treatment and the value of that hardness. Discuss the agreement of these experimental results with theory and give reasonable explanations for any gross discrepancies. Do not attribute discrepancies to "variability" of the sample.

****

Notes for the Instructor

The underlying theory for the experiment is covered in all undergraduate metallurgy texts and will not be repeated here. You may wish to refer to the Metals Handbook Volume 2 for a particularly good explanation of the strength and hardness changes that can be expected for 2024 alloy.

We have found that students will obtain confusing and contradictory data if certain precautions are not taken.

> Hardness tests have a tolerance band of at least 4 points when the Rockwell B scale is used. Students will likely record hardness readings to one decimal place accuracy and will take more than one reading only if prompted. Take full advantage of the potential in this exercise to introduce the real world of measurement accuracy and experimental variation. Explain that at least four hardness readings and preferably six should be taken of each sample. Take as many readings as possible given limitations on sample size and hardness testing machine availability for other samples. Students should list all readings in the report and use their average for all graphs.
>
> The hardness reading variation problem will be exacerbated if sample faces are not parallel, freshly ground and dry. A few low hardness readings indicate poor sample preparation, especially if they are the initial hardness readings on a given sample.
>
> Laboratory furnaces commonly have a low thermal mass and inexpensive temperature controllers. Therefore, large temperature excursions can occur when cold samples are placed into a furnace or when the furnace is opened to remove samples. Aging may be accelerated if the furnace temperature overshoots. If a 9mm thick piece of stainless steel is laid onto the hearth of the furnace, the added thermal mass will lessen this problem.
>
> The aging temperatures that we list above are arbitrary. However, if you use temperatures higher than 220°C, overaging may occur too quickly to catch the peak hardness. If temperatures lower than 175°C are used, overaging may not occur within the time limits of a laboratory session.
>
> Although the recommended solution treating temperature for 2024 Aluminum is given as 493°C, we found it necessary to exceed that temperature considerably in order to effect solution treatment within one hour. The high reflectivity of aluminum, as well as its high heat capacity, makes thermal equilibrium difficult to achieve. Even though the phase diagram indicates that 650°C is well above the solidus line and into the liquid-solid 2-phase region for aluminum - 4.4% copper, we found that full solution

treatment did not occur at lower furnace temperatures within a reasonable length of time. We recommend that persons replicating this experiment devote some time to finding solution treatment conditions appropriate to their own situation.

The effect of cold work on aging kinetics is interesting but optional if you do not have access to a small rolling mill. If you do, determine the roll opening that corresponds to about 5% cold work with a scrap piece of aluminum alloy before quenching the sample from the solution-treating furnace.

The microstructural changes that occur in the samples of this experiment are on too fine a scale to observe with optical microscopy. Therefore, the discussion portion of the student reports should include, for each of the various processing schemes, a simple flow sheet and a discussion of the fundamental theory of the precipitation hardening process. A suggested format for the flow sheets is shown in Fig. 1. Require a hypothesis statement--for the given process scheme, will the peak hardness be higher or lower than that for other aging treatments and will the peak hardness be achieved in less or more time? Encourage the use of the textbook and selected references for the preparation of this section.

The kinetic relationships among the various aging schemes are best understood if the data are graphed as the average hardness versus the elapsed aging time with the latter on a logarithmic scale. You may wish to briefly review with your students the first-order kinetics basis for using a logarithmic transformation of the data. To avoid confusion over plotting the point which represents the solution-treated condition, do not use zero but instead use the time at which the hardness readings were taken, presumably two or three minutes. A sample graph is shown in Fig. 2. This graph was constructed using spreadsheet software. Encourage students to use this tool for enhanced productivity and accuracy.

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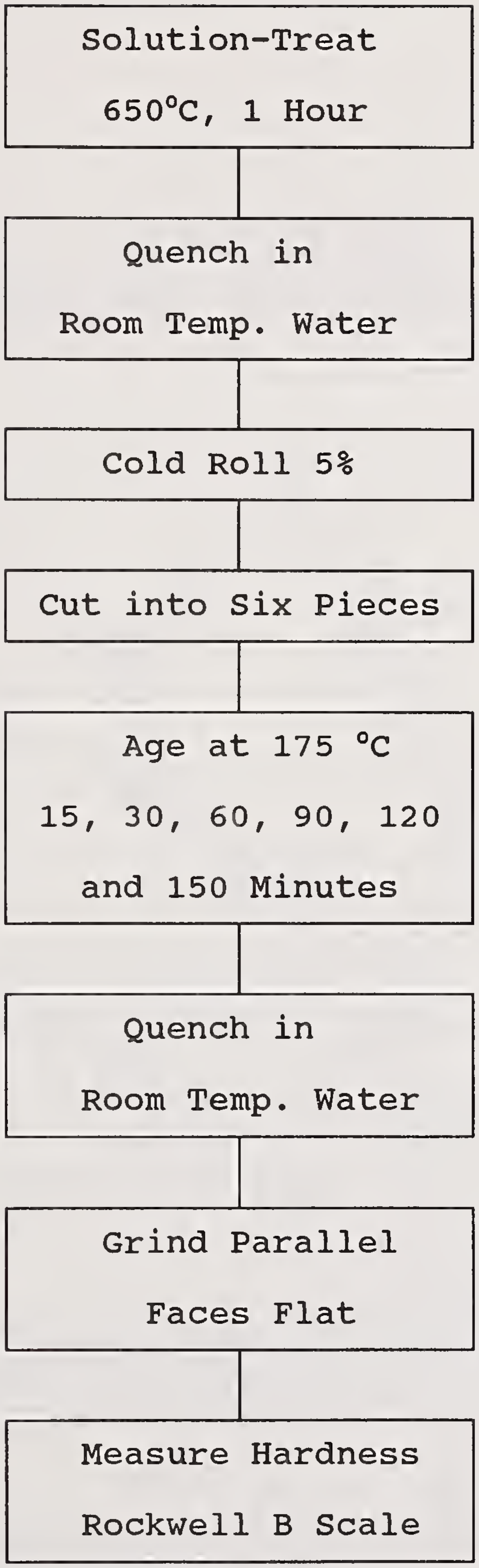

Fig. 1. Sample of format for flow sheets; T8 temper shown.

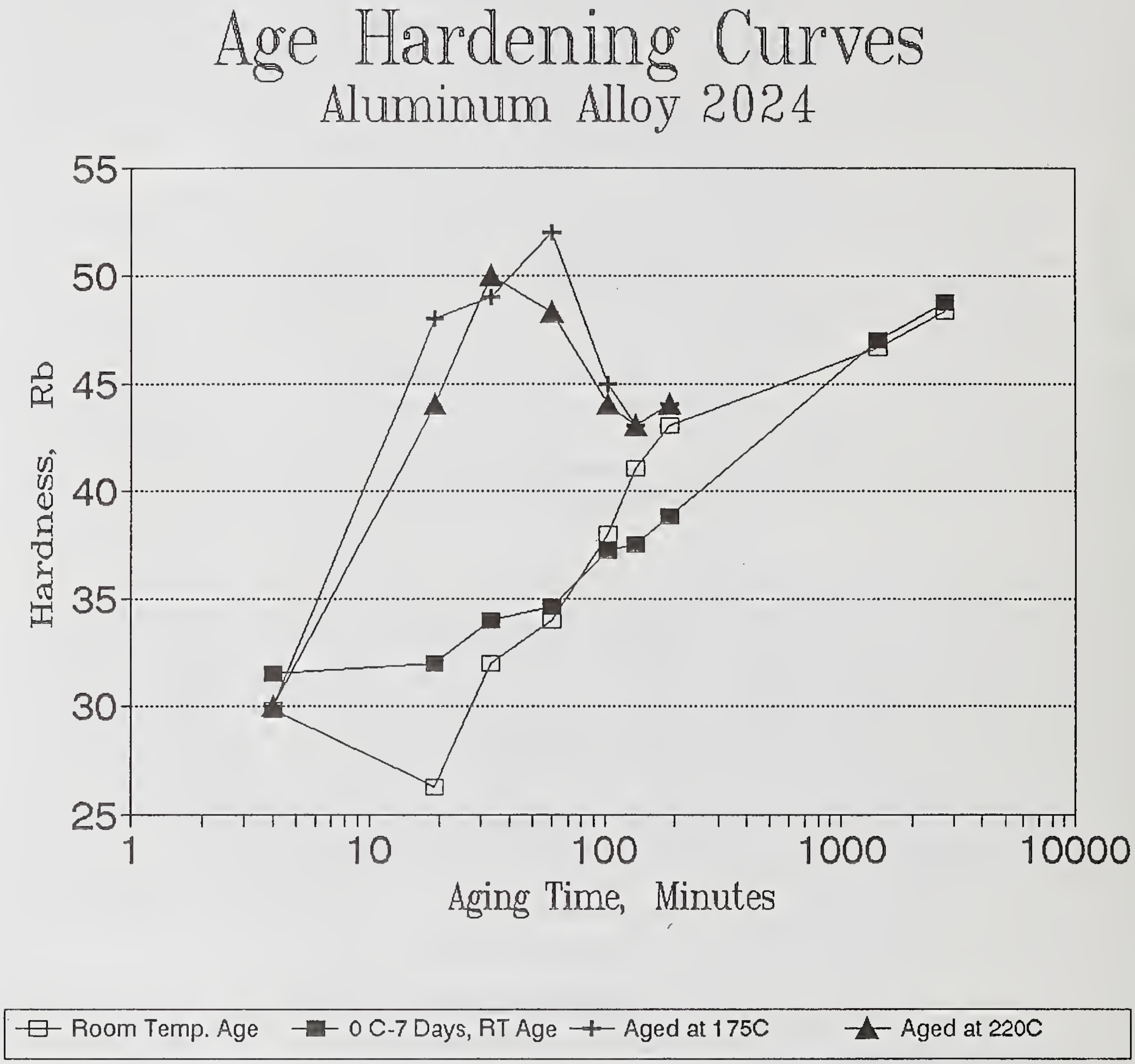

Fig. 2 Suggested format for age hardening curves.
T4 and T6 tempers shown

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SIMPLE STRESSED-SKIN COMPOSITES USING PAPER REINFORCEMENT

L. R. Bunnell

October 1990

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Gaithersburg, Maryland

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Pacific Northwest Laboratory
Richland, Washington 99352

SIMPLE STRESSED-SKIN COMPOSITES USING PAPER REINFORCEMENT

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KEY WORDS: composite, stressed skin, polyurethane foam, paper, cantilever beam, Young's modulus, elasticity.

PREREQUISITE KNOWLEDGE: This material could be taught to a typical student of materials science, at the high school level or above. The student should understand the concept of Young's modulus of elasticity, which is a measure of a material's stiffness.

OBJECTIVES: To demonstrate the composite reinforcement concept in a hands-on manner, using readily available materials. To demonstrate the consequences of certain defects in these structures. To quantify the gains made by engineered composite construction, using a simple measurement of Young's modulus of elasticity.

EQUIPMENT AND SUPPLIES: 1) Foam rubber beam about 8 cm by 8 cm by 30 cm, with vertical lines on all of the 8-cm faces; 2) polystyrene or polyurethane insulating foam; 3) heavy paper such as construction paper; 4) waxed paper or polyethylene; 5) slow-setting (>3 h) non-allergenic epoxy resin curable at room temperature; 6) cantilever beam loading device constructed as per instructions in this writeup; 7) known weights of about 100 g; 8) dial-gauge indicator capable of measuring to 0.025 mm (although most will measure in thousandths of an inch); 9) calculator.

PROCEDURE: Demonstrate by bending the foam rubber beam that the initially parallel lines get further apart on one side (the tensile side) and closer together on the other side (the compressive side), see Figure 1. Introduce the concept of stressed-skin composites by stating that a strong and stiff material, if attached to these faces, will provide substantial reinforcement to the structure by resisting such tensile or compressive forces. Next, students bend by hand precut pieces of polystyrene or polyurethane foam insulation 1 cm by 3 cm by 18 cm, bending the beam in the 1-cm direction. They will soon note that the beam is not very stiff and will not bend very far before breaking. Students will then weigh four more foam beams identical to the first, to a precision of 0.1 gram, and record the weights permanently on the ends of the beams. They should then prepare stressed-skin composites as follows: one beam is left as is; one beam has construction paper bonded to one of its 3 x 18 faces; one beam has construction paper bonded to both of its 3 x 18 faces; the fourth beam is made just as the third but has an intentional disbond that is made by placing a piece of waxed paper or polyethylene 3 cm by 6 cm at the midpoint of one of its paper-reinforced faces. To achieve the best possible bond, minimal epoxy should be used and the beams should be weighted during the curing process. Use waxed paper or polyethylene to separate the composites from surfaces such as tabletops where bonding is not desired.

The first part of the testing will be done by hand. First, the students weigh the beams after any necessary trimming and record the weight gains for reference. Students then bend the nonreinforced beams again for calibration purposes. The single-sided beam is next bent, with the nonreinforced face first on the tensile side; the beam should be bent only slightly, taking care not to break it. Note that

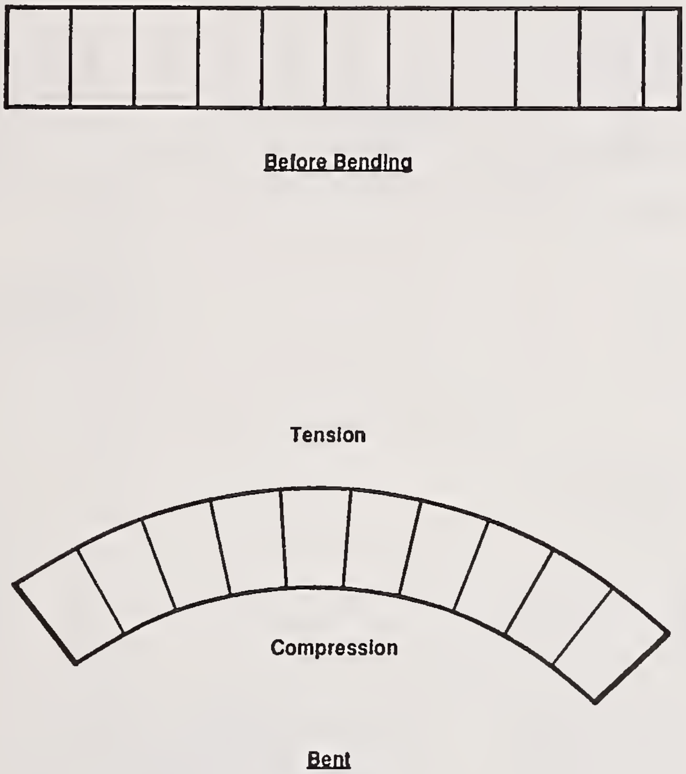

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FIGURE 1. Foam-Rubber Beam Used to Illustrate Tensile and Compressive Forces Resulting From Bending

this one-sided reinforcement does not have much effect on the stiffness. Finally, the beam should be bent so that the nonreinforced face is on the compressive side until it breaks. Note that the foam collapses on the compressive side. This is because the reinforcement has made the beam much stronger on the tensile side. Now the students should bend the two-faced reinforced beam without the intentional debond; it is noticeably stiffer than either of the two preceding beams. Some of the students may want to break these beams to observe whether failure occurs on the tensile or compressive face. Next, the defected beam should be bent but not to the breaking point, with the defect on the tensile side; note that the defect has essentially no effect. Finally, the defected beam should be bent with the defect on the compressive side until it fails. Note that the nonbonded paper pops away from the foam in what is known as buckling. Buckling is a fairly common failure mode for this kind of composite and can be avoided by close attention to construction.

Students may want to quantify the gains made by reinforcing the beams. This can be done by using the simple testing device in Figure 2. Start with a nonreinforced beam, clamping it in place as shown and barely contacting its bottom face with the dial indicator. Zero the indicator, or note its reading before loading. Next, place a weight of 50-100 g on the beam, directly in line with the dial indicator. If washers are used as weights, it may be useful to push a nail into the

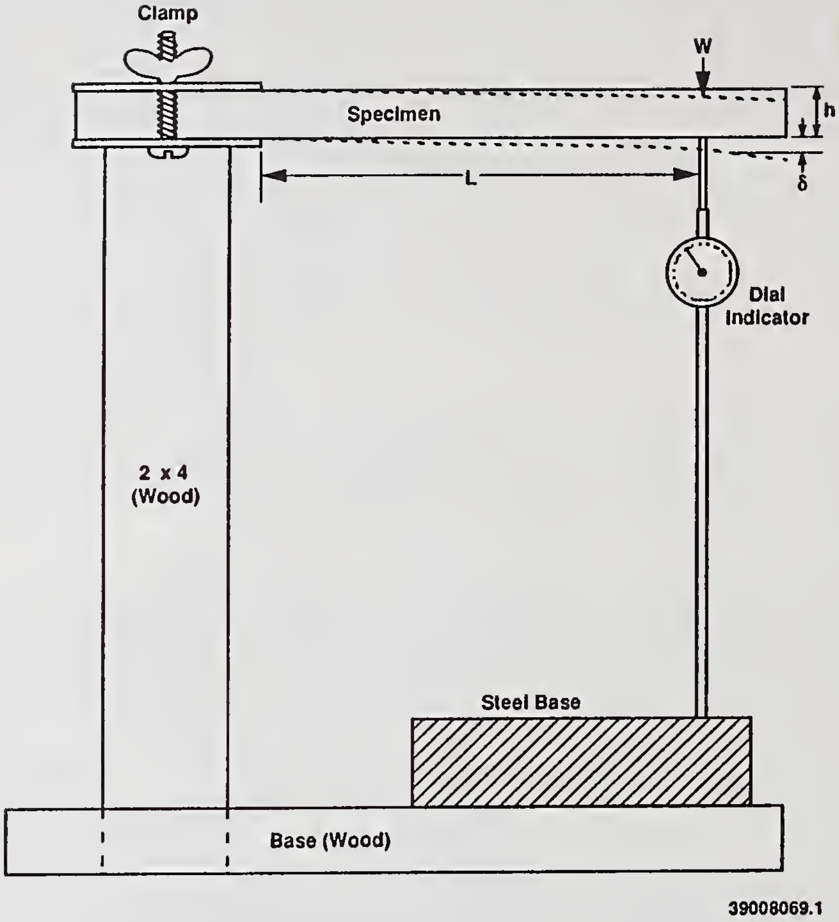

FIGURE 2. Apparatus Used to Evaluate Stiffness of Composite Beams by Measuring Deflection of a Cantilever Beam in Bending

beam to prevent them from sliding off. The beam will then bend, and the deflection should be noted and converted to centimeters. Using the formula, calculate the Young's modulus of the beam:

$$\text{Young's Modulus (in Pascals, Pa)} = 4\ (98)\ W\ L^3/D\ B\ H^3$$

where W = weight, g, L = unsupported length, cm, D = deflection, cm; B = width, cm; H = height, cm; and 98 is the conversion factor necessary to change from g/cm^2 to Pascals, the SI units for elastic modulus or pressure.

Do the same with one-sided and two-sided reinforced beams. The deflection should be at least 0.25 mm; if not, apply more weight until it is. It will be noted that the beam reinforced on one side is not a great deal stiffer than the one without reinforcement, just as was learned by hand bending. Similarly, the two-sided reinforcement produces impressive gains in stiffness. Some students may wish to relate the stiffness gains to the weight gains involved in the various reinforcements. Although the stiffness of the foam beams has been increased greatly by using only paper reinforcement, the resulting composites are not very stiff when compared with other materials. However, the density of the foam beams is very low compared with other solid materials. The following values of Young's modulus for some common materials may be useful for comparison:

Material	E, GPa
Aluminum	69
Steel	207
Many Solid Polymers	3
Glass	69

Note: 1 GPa = 10^9 Pa.

SAMPLE DATA SHEETS: Self-Evident.

INSTRUCTOR NOTES: The experiment outlined here is designed for minimal expense per concept learned. If students are interested in exploring the capabilities of different reinforcement fibers, such materials as Fiberglass cloth, woven Kevlar, or woven graphite fibers can all be used to make additional beams that can be evaluated by the cantilever beam test above. Additionally, other fabrication techniques such as vacuum bagging may be used to achieve better bonding while using even less epoxy. To save on supplies and time, the instructor may wish to prepare demonstration beams using the more exotic materials rather than have each student make all the beams. These demo beams may then be measured for stiffness using the cantilever beam apparatus.

REFERENCES: Timoshenko, S., and D. H. Young, Elements of Strength of Materials, Fourth Edition, Van Nostrand, 1962. p. 212.

SOURCES OF SUPPLIES: Polystyrene or polyurethane foam--available from any building supply store, in pieces about 2.5 m by 0.6 m by several different thicknesses. The pink board is much more uniform than the white type made from expanded beads and is much preferred. The test beams can be cut with a table saw or band saw, using a fine, sharp blade to produce a good surface finish. Epoxy--one epoxy, used successfully with no signs of skin reactions to date, is Vinacron Epoxy, available from Iasco, 5724 W. 36th St., Minneapolis, MN 55416.

Dial Indicator and Support--available from Sears Catalog Sales, both for a total of about $125.

Weights--use calibrated weights made for that purpose, or use large preweighed steel washers with their weights written on them.

EFFECT OF STRAIN RATE ON TENSILE PROPERTIES OF PLASTICS

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SUMMARY

Polypropylene specimens were tested in a uniaxial tensile testing machine to illustrate the effects of varying strain rates on the final elongation and yield strength of the material. As the rate increases, the strain to failure decreases while the yield stress increases. These results provide a nice contrast with metallic systems which are nearly independent of strain rate effect, at least at room temparature. All of the tests were conducted at 300° K which is above polypropylene's glass transition temperature of 253° K.

INTRODUCTION

The purpose of this experiment is to demonstrate the variation in tensile properties as a function of crosshead rate on uniaxial tensile machine. A specimen is prepared in the form of a sheet tensile specimens and pulled to failure in a uniaxial tensile test machine(Instron). This provides an excellent demonstration of the sensitivity of polymeric materials to the loading conditions.[1]

PROCEDURE

Sheet tensile specimens of common plastic materials are formed in a Boy injection molding machine. We have had made specimens from polyethylene, polypropylene, and polystyrene. For demonstrating the effect of varying crosshead rate, polyethylene is probably the most dramatic. The overall dimensions of the specimen are 5" long, 0.125" thick, and 0.500" wide in the gage length which is 1.875". The students remove the flash along the parting line and measure each specimen. Sheet metal tensile grips are used to hold the specimens. For convenience, the gage length is taken from the ends of the specimen holders. The crosshead rate is set and then the test is begun. It is crucial to record the temperature of the room.* A copy of the laboratory handout is given in Appendix 1.

DATA

Table 1 provides the crosshead speed, the final elongation and yield stress, the true strain, and the true strain rate for the polypropylene specimens. Figure 1 a and b gives the stress-strain curves for polypropylene at crosshead rates of 0.4 to 20 in/min.

DISCUSSION

As the data in Table 1 and Figure 2 show the strain to failure decreases as the crosshead rate or true strain increases. For example, at a crosshead rate of 0.2 in/min the strain to failure was between 140 and 170%, while at a crosshead rate of 20 in/min the strain to failure was between 54 to 66%. There is also a steady increase in the yield strength from about 3170 lb/in^2 at 0.4 in/min crosshead rate to 3620 lb/in^2 at 20 in/min crosshead rate.

Polymers have a range of mechanical property behavior. Depending on the glass transition temperature (T_g), the properties can vary from brittle-elastic at low temperatures, to plastic, to viscoelastic or leathery, to rubbery, and finally viscous at high temperature.[2] The T_g for-polypropylene is 253° K and laboratory tests were conducted at about 300° K. The polypropylene will be in the glass transition or the rubbery region during testing.

The modulus of elasticity for the 0.4 in/min specimen is approximately 55000 lb/in^2(0.38 GPa). Compared with published results, this value is about half the smallest value reported for polypropylene, 10000 lb/in^2 (0.69 GPa). This great inaccuracy is attributed to the lack of direct strain measurements using either an extensiometer or a strain gage mounted on the specimen. Without the direct strain measurements, specimen elongation will be overestimated due to structural deflection in the tensile testing machine, thus resulting in underestimate of the modulus of elasticity.

Since polymeric materials are strain rate sensitive, the strain is a function of both time and temperature. As the strain rate is increased, the strength of the material will increase while the strain to failure will decrease. Figure 2 demonstrates the decrease that occurs in the final elongation as the crosshead rate is increased and correspondingly the true strain rate is increased as shown in Table 1. The true stain rate can be obtained from the following expression,[3]

$$\dot{\varepsilon}_t = \frac{V}{l_o e^{\varepsilon_t}}$$

$\dot{\varepsilon}_t$ = *true strain*
l_o = *original gage length*
V = *crosshead speed*

Although the above experiment does not hold the strain rate constant during the entire test, using the crosshead rate is representative of the actual strain rate and plotting this versus the final elongation provides a very useful demonstration of the effect of varying loading rates on a polymeric material. Figure 3 illustrates the increase in yield strength that occur as the crosshead rate is increased.

Very similar data can be obtained for polyethylene and Figure 4 shows the final strain plotted against the crosshead rate for polyethylene. The strain decreases from more than 600% at the lowest rates to less than 10% at the highest crosshead rates. A simple explanation of the processes occurring in the structure is given to the students following Ashby and Jones.[2]

CONCLUSIONS

This experiment demonstrates the significant effect varying the crosshead rate (strain rate) has on the elongation and yield strength for the polyolefins. In particular, this experiment used polypropylene. There are several additional things that could be done to improve or enhance the experiment. A constant temperature cell could be built to surround the specimen and then the data as a function of temperature could be obtained. With more sophisticated test machine, one could actually perform the tests at a constant strain rate. Another possibility would be to obtain the strain rate exponent by performing rate change tests.[3]

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2. M. F. Ashby and D. R. H. Jones, Engineering Materials 2: An Introduction to Microstructure, Processing and Design, Pergamon Press, Elmsford, NY,1986.

3 G. E. Dieter, Mechanical Metallurgy, 3rd ed.,McGraw-Hill, 1986.

4. ASM Engineered Materials Reference Book, ASM International, Metal Park, OH.

* We had an interesting experience when one lab session took place when the room was about 10 degrees cooler than normal. The results differed dramatically from previous tests and we concluded that the temperature was the reason the results were different.

TABLE 1. MECHANICAL PROPERTIES OF POLYPROPYLENE.

Crosshead rate (in/min)	Engr. Strain (in/in)	Yield Strength (lb/in^2)	True strain	True Strain Rate (s^{-1})
0.2	1.681	3172	0.986	0.050
0.2	1.391	3161	0.872	0.056
0.4	1.402	3141	0.876	0.111
0.4	0.951	3200	0.668	0.137
0.6	0.667	3271	0.511	0.240
0.6	0.712	3203	0.537	0.234
0.8	0.657	3318	0.505	0.322
0.8	0.936	3234	0.661	0.336
1.0	0.981	3383	0.684	0.336
1.0	0.661	3318	0.507	0.402
5.0	0.667	3402	0.511	2.000
5.0	0.653	3454	0.503	2.016
10.0	0.510	3498	0.412	4.415
10.0	0.596	3517	0.468	4.175
15.0	0.594	3588	0.466	6.275
15.0	0.458	3563	0.377	6.859
20.0	0.537	3576	0.430	8.673
20.0	0.661	3666	0.507	8.031

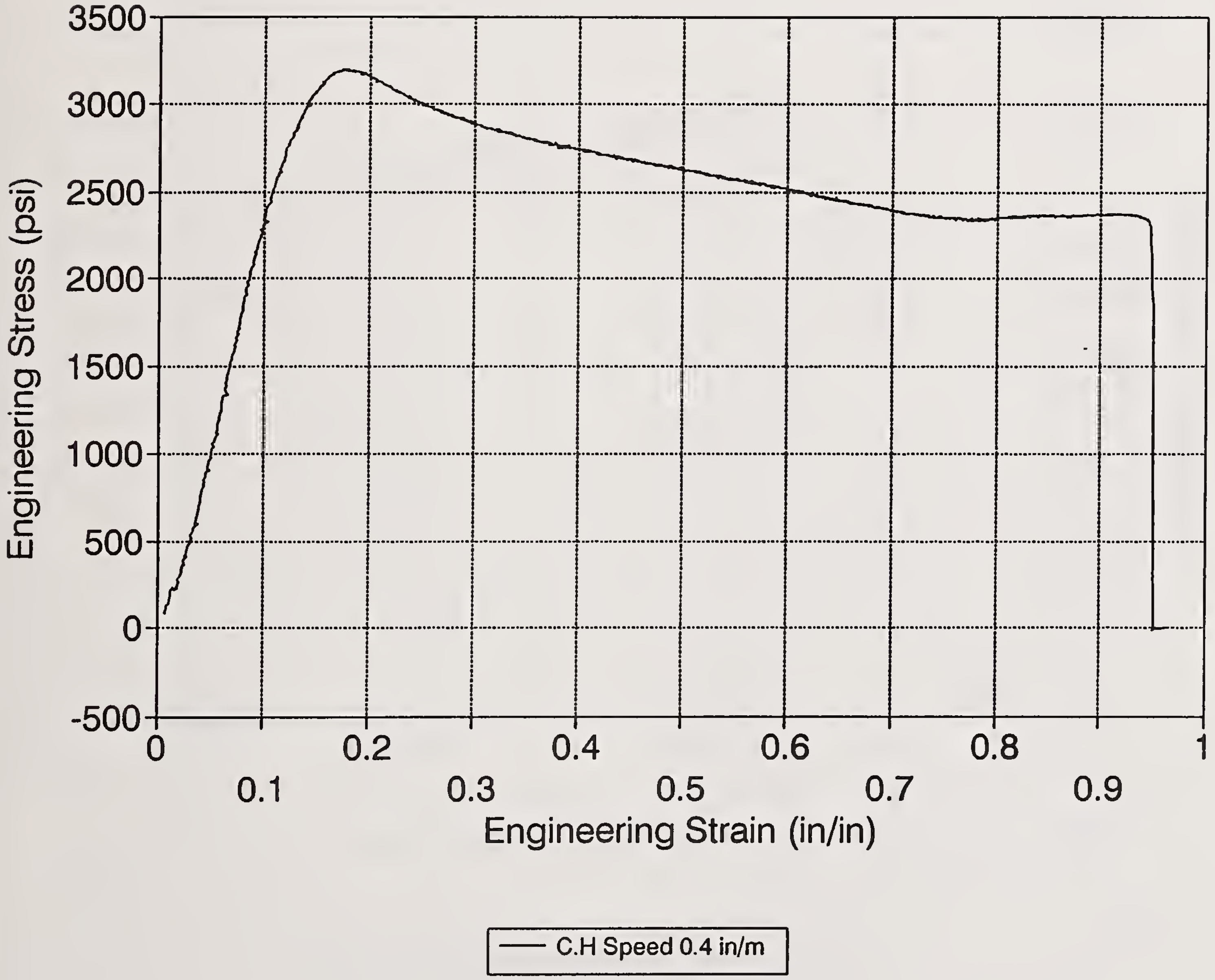

Fig 1 a: Engineering stress strain curve for polypropylene, crosshead speed of 0.4 in./min.

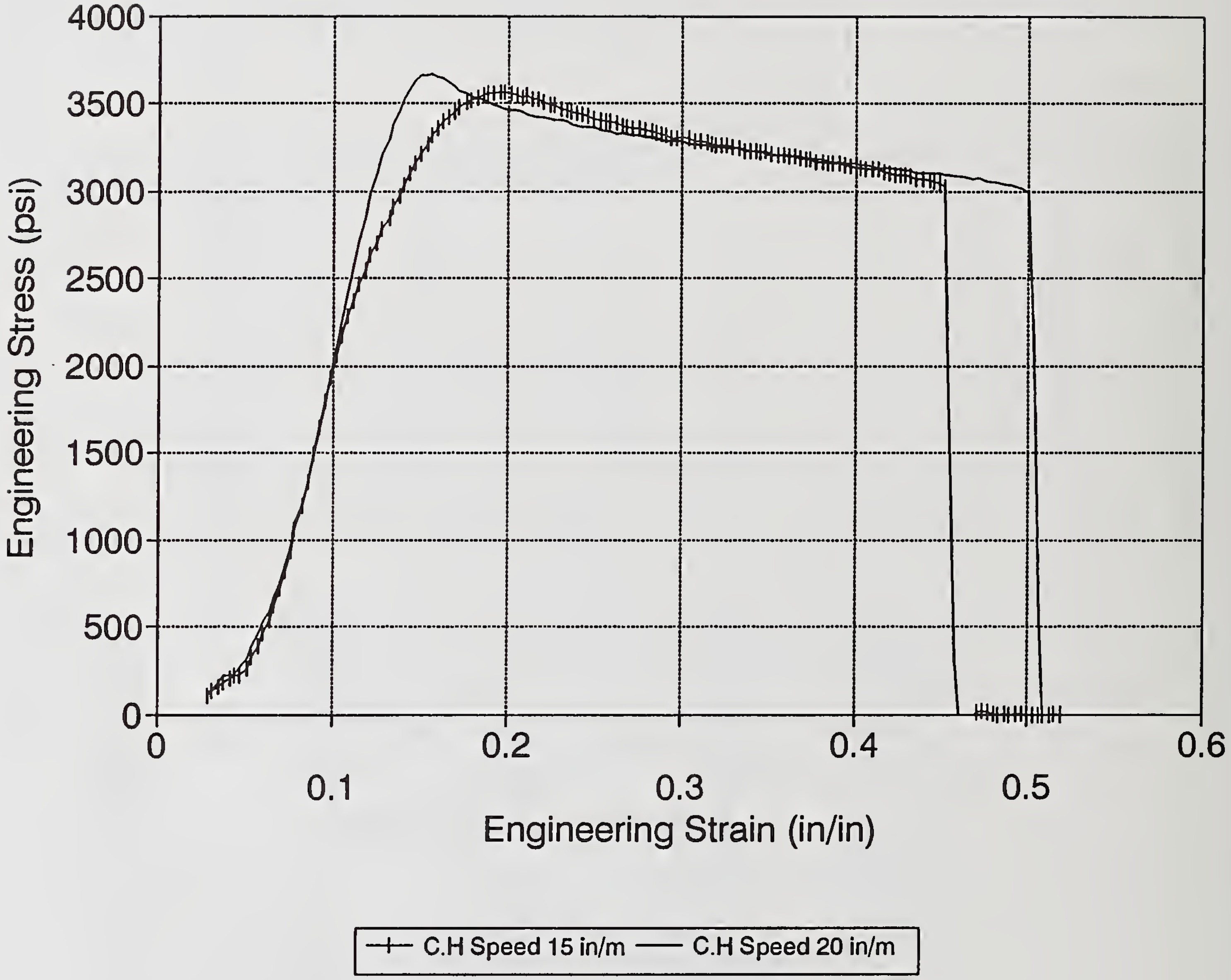

Fig 1 b: Engineering stress strain curve for polypropylene, crosshead speeds of 15 and 20 in./min.

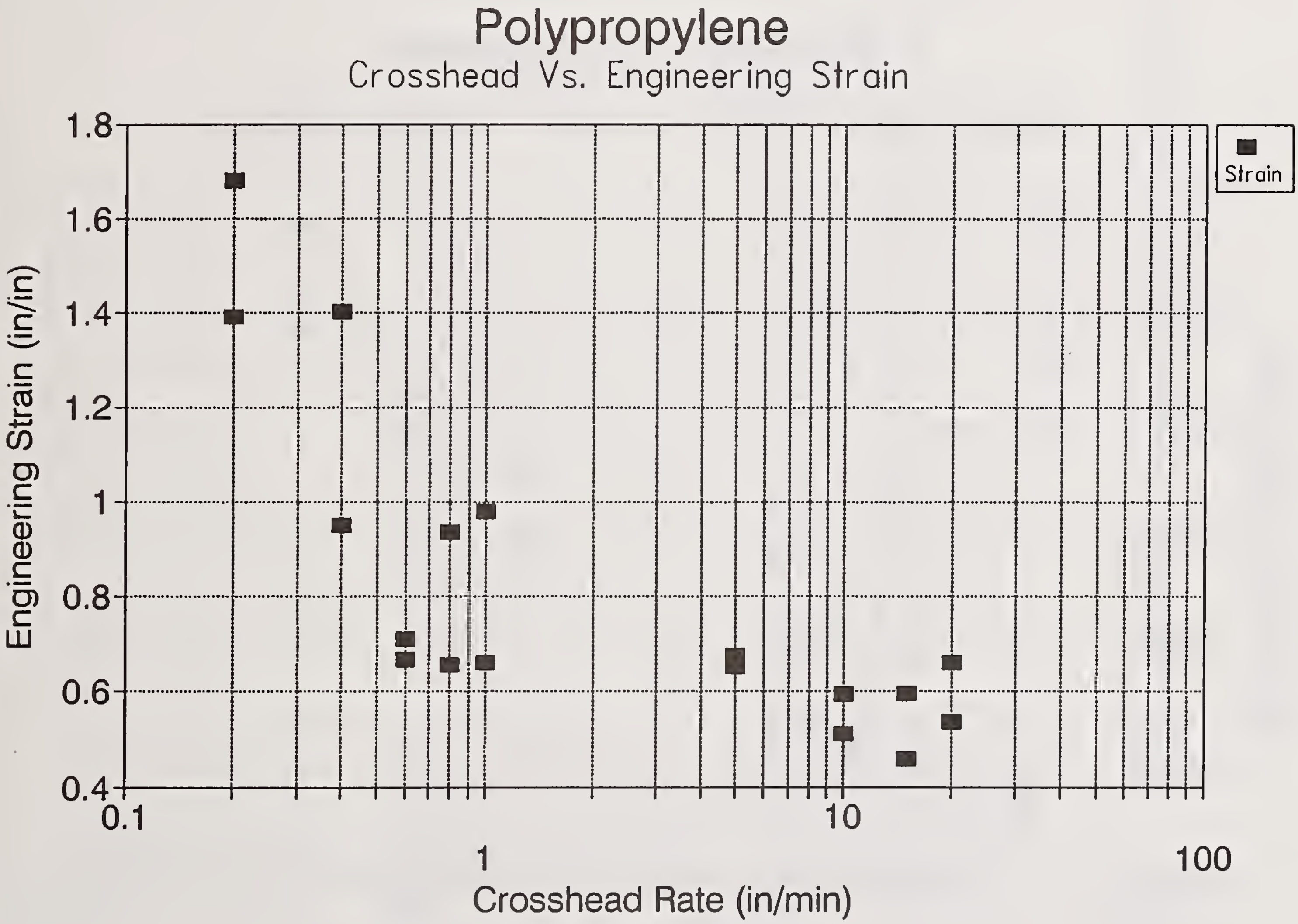

Fig 2: Plot of engineering strain as a funcion of crosshead rate for polypropylene

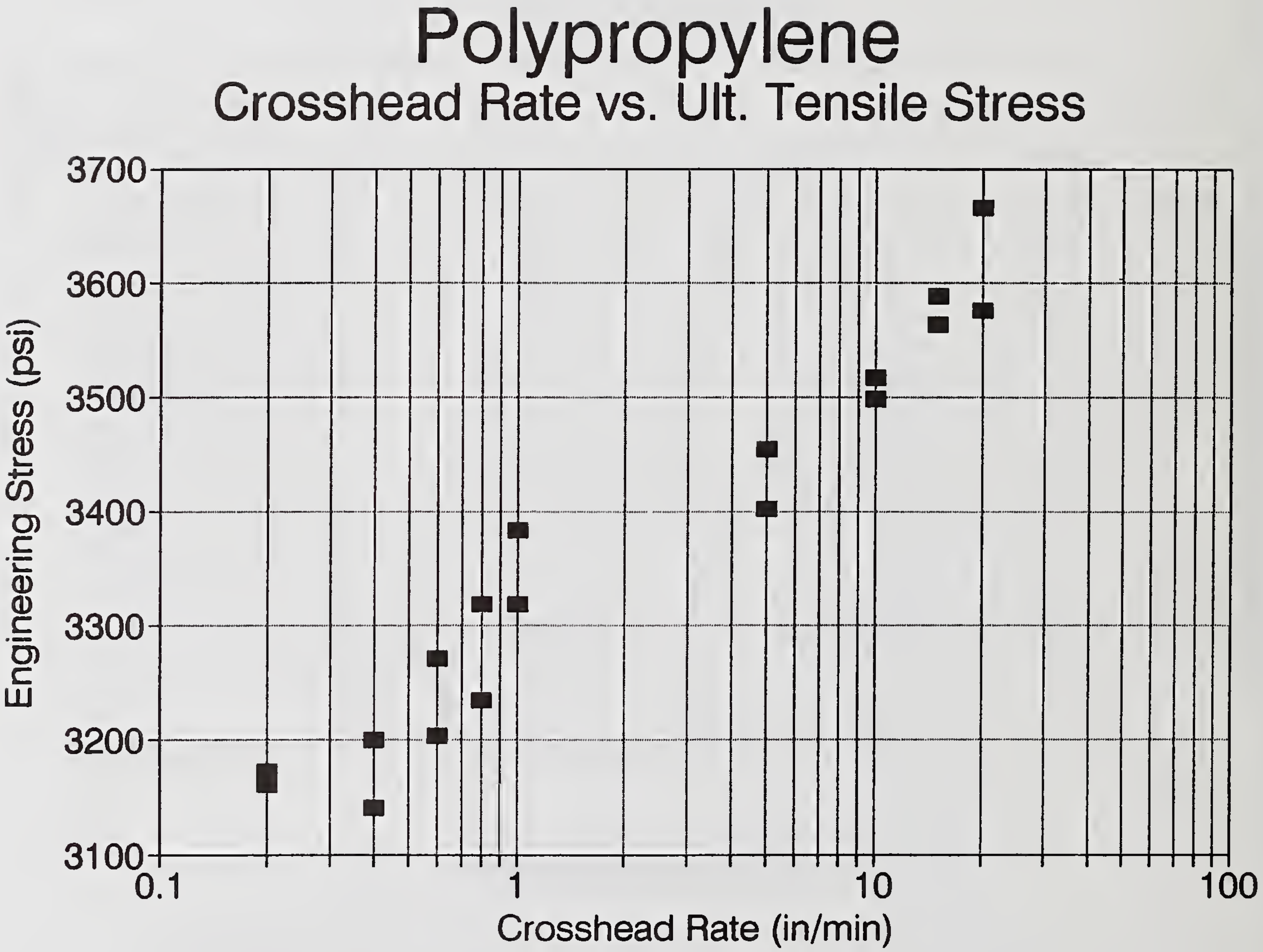

Fig 3: Plot of UTS of polypropylene as a function of crosshead rate.

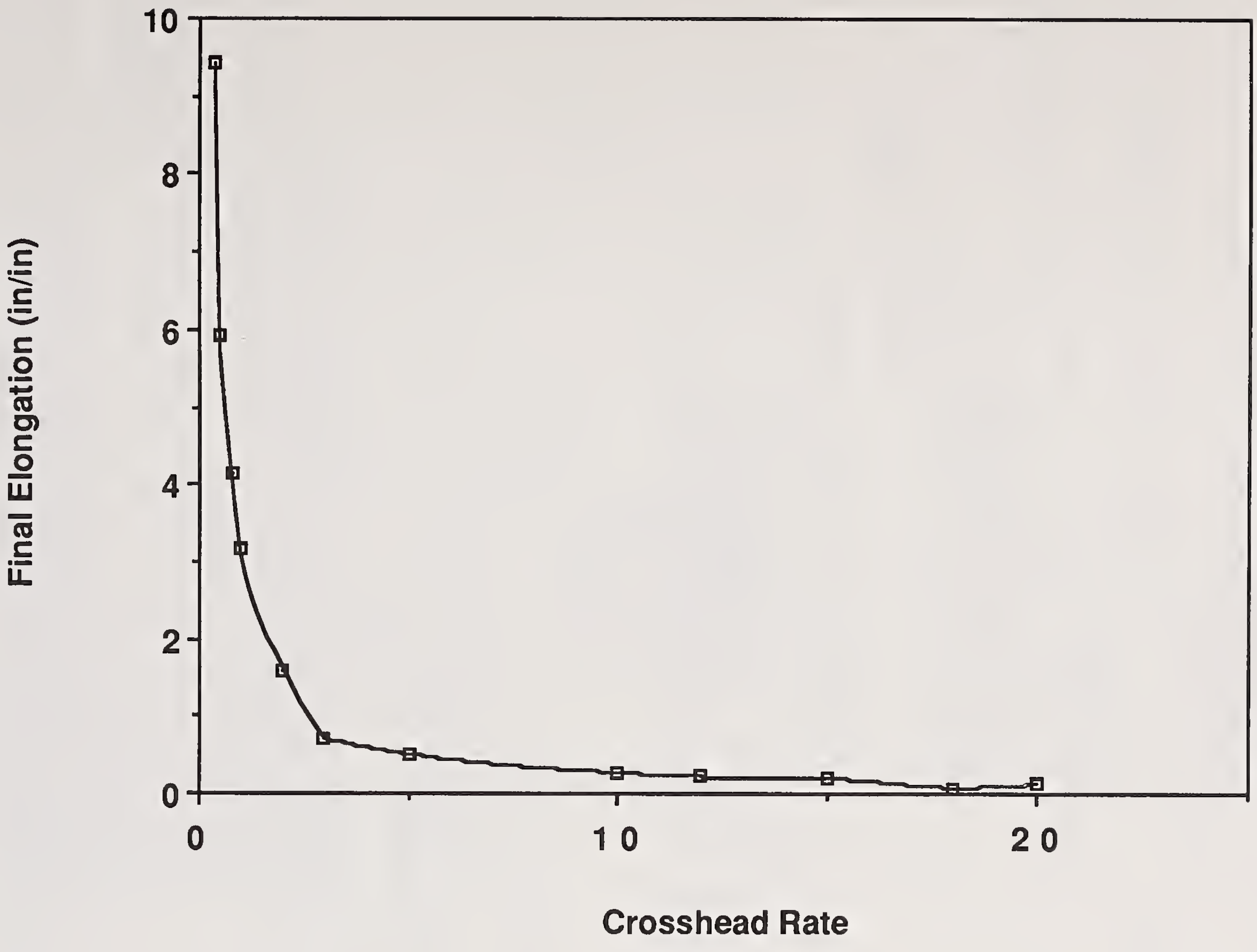

Figure 4. Final elongation vs crosshead rate for PE.

TENSILE TESTING OF PLASTICS

OBJECTIVE: To determine the effect of varying strain rates on the mechanical properties of an engineering plastic.

EQUIPMENT:

- Plastic tensile specimens.
- Instron test machine.
- Micrometer and scale.
- Thermometer.

PROCEDURE:

- Measure the gauge length and cross sectional area of specimens.
- Determine required crosshead speed testing range, tabulate speeds required, and then select a random sequence for performing the tests.
- Setup Instron and recording computer.
- After test, measure final elongation and area.

ANALYSIS: Determine the following properties:

- * maximum elongation
- * % reduction area
- * modules of elasticity
- * yield strength
- * fracture stress
- * flow stress

REPORT:

○ INTRODUCTION:(25%)

- Importance of plastics as an engineering material.
- Discuss the different mechanical properties of plastics and how they are utilized in selecting appropriate materials for a given application. give examples.
- Discuss the particular type of plastic used in this experiment including structure, type (thermoset vs. thermoplastic), applications ..etc.
- Discuss any environmental issues associated with the tested material including ease of recycling, biodegradability, toxic byproducts ...etc (**EXTRA CREDIT).**

- EXPERIMENTAL PROCEDURE:(15%)
    - Mention all testing parameters including material type, crosshead speed range and increments, and room temperature.
    - Briefly, describe the test setup including tensile testing machine, and data recording method.
    - Describe test specimens, including all the relevant dimensions, use an illustration.
    - Did the test conform with any ASTM standard?

- ANALYSIS OF DATA:(15%)
    - Describe data reduction procedure including:
        - all relevant formulas.
        - software used.

- RESULTS:(10%)
    - Tabulate test results and relevant published data. Include measured temperature as part of your results.
    - Generate plots displaying the relationship between the different mechanical properties and crosshead speed.
    - Describe the necking behavior at different crosshead speeds.

- DISCUSSION OF RESULTS:(25%)
    - Explain the necking behavior. Use a stress-strain curve generated during the experiment as an example.
    - Compare data for various crosshead speeds.
    - Explain the effect of crosshead rate on the material.
    - Contrast with tabulated values.
    - Compare and contrast the behavior of the polymer used with that of a metal of your choice.
    - Explain the effect of temperature on the test results.

- REPORT FORMAT (10%)

REFERENCES:

- ASM Engineered Materials Handbook- Plastics.
- Engineering Materials and Their application, Flinn/Trojan.
- The Science and Engineering of Materials, Askland.

BIODEGRADABLE PLASTICS:
AN INFORMATIVE LABORATORY APPROACH

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ABSTRACT

Simple laboratory procedures are outlined for class participative sessions on the subject of testing the new claims of polymer biodegradation. These claims are being brought out by major chemical companies pertaining to their new polymer products. Both long term and short term, accelerated tests are cited using published standards.

INTRODUCTION

Due to the increasing awareness of the public toward disposable trash items, the plastics industry has begun to respond to ever increasing pressures from both the public and politicians to enhance the biodegradation of polymeric materials. This is very problematic for the industry as polymers are virtually stable and inert to the elements when added to land fills. By their very chemical nature, polymeric materials are resistant to degradation by sunlight, water exposure or oxygenation. Only polymers that contain more unstable components in their chemical structure, such as Vinyls or Chlorides tend to degrade over time when exposed to weathering. Normally only the excessive energy exerted by short wave ultraviolet light (UV-B) emitted by the sun can destroy the bond integrity and thus destroy the polymer. Polymers ("plastics") such as Polypropylene (disposable squeeze bottles), Polyethylene (disposable bottles and films), Polystyrene (disposable utensils and dinnerware) and other food packaging materials are for all practical purposes chemically stable and will withstand the tests of time and elements very well.

The solution then, must come from some alternative method of material manufacture. The industry has responded with an organic additive to the polymer matrix called corn starch. This material is touted as being totally biodegradable and as having the ability to rot and fade away in a few short years. The question to the student and instructor alike is whether the claims are accurate. The questions are answered by following national standard test procedures for weathering (in order to expose the material to the elements to be encountered in a dump, excluding bacteria and micro-organisms which are very important to the "degradation" of the new polymer matrices) and for testing physical characteristics (in order to detect changes in matrix integrity, i.e. biodegradation).

EQUIPMENT

1. An outside weathering apparatus built in accordance to ASTM test number D1435-85.
2. Tensile testing apparatus meeting ASTM standards for pulling the material apart in order to simulate polymer matrix integrity.
3. Accelerated weathering apparatus capable of generating UV-B light and of varying both the relative humidity and heat in the chamber.
4. Dial micrometer reading to .0001".
5. Digital scales with at least .001 gram accuracy.
6. New household biodegradable trash bags.

EXPERIMENTAL

The most effective visual aid for demonstrating the effects of adding corn starch to a polymer matrix is to wrap a ball of any composition (representing the starch particle) with string (representative of polymer chains). This polymeric theory is made more apparent by wrapping a sugar cube (the starch) with stiff wire (the molecular chains) and submerging the model in water. The theory states that as the starch is removed from the system, the molecular structure falls apart. This is to promote biodegradation.

Using a sharp razor or knife, the trash bag must be cut into sample strips that are in accordance with ASTM D882-83 (Test Methods for Tensile Properties of Thin Plastic Sheet). These strips are for use in the tensile tester which will allow us to test the pull strength of the polymer. This will give an indication of the matrix integrity and allow conclusions to be drawn as to the effect of starch loss.

The prepared samples must then be mounted on an environmental test stand as described in ASTM D1435-85 (Practice for Outdoor Weathering of Plastics). This stand must set in a place free of any objects that might cast a shadow on the samples at any time during the day. The angle of the apparatus should be at 45 degrees. The stand should point to the South so as to give it the maximum exposure to the Sun (it is interesting to note that the UV absorption of a black trash bag is greater than that of a lighter colored bag. The time factor can be shortened by simply using a dark colored bag so that more UV energy is absorbed). If the stand is erected and the samples prepared in the Fall of the school year, the sample testing will occur throughout the full year and quite possible into the next year. This factor of a long exposure period of time in order to affect a change is the one major drawback to the testing procedures.

The answer to the problem of excessive time limitations is to be found with the uses of accelerated testing apparatuses. An apparatus of this type can significantly reduce the amount of time that it takes to get significant changes in the properties of the polymer to occur. A proper weather accelerator will expose the samples to UV-B light and will vary the temperature and the humidity levels within the chamber its self. This has the effect of simulating conditions on the outside of the building, but due to the severity of the conditions within the chamber, the time factor is greatly reduced (reference ASTM D756-78, Practice for Determination of Weight and Shape Changes of Plastics Under Accelerated Service Conditions for proper accelerated testing procedures).

As the sample progresses in the elements and "degrades" we can pull samples off of the test stand or out of the accelerated chamber and run a simple tensile test on them (or a test for Initial Tear Resistance of Plastic Film and Sheet, ASTM D1004-66, could be substituted as it uses the tensile tester as well). The output of the tester will show that the material starts to lose it's strength as the starch is dissolved. The amount of starch loss can be calculated by weighing the samples before testing in the tensile machine. Accurate numbering of the samples when they are first placed on the stand or in the chamber is essential and accurate weights must be made so that future determination of weight loss can be accounted for. This accounting for the starch loss may not show immediate correlation to the tensile data as the actual amount lost may be too small to accurately determine.

It is extremely interesting to keep record of the temperature and humidity of the test site so that these conditions can be correlated with the speed of degradation. Simple, periodic checks will suffice in getting the information needed to determine whether changing weather conditions drastically effect the rate of biodegradation (variables such as amount of rain, temperature of the specimen, relative humidity, length of time frozen in air or in ice, etc.)

One of the easiest ways of determining the concentration and therefore the biodegradability of the polymer is to take the trash bag and boil it in water. The reason is obviously to remove the starch additive. Weigh the remaining polymer structure to see what amount of loss occurs over the life of the material in the ground. By boiling the material and continually weighing the sample until a constant weight is achieved, we can calculate the percentage of additive used. Care must be taken when boiling the material as some Polyethylene materials that are used in trash bags will melt totally at this temperature. The molecules will re-form as the temperature lowers to below the melting point of the bag.

Whatever the preferred method of analyzing the effects of corn starch loss may be, the ultimate goal is to determine how long it would take for the material to completely degrade. This is a paradox in the testing as the polymer matrix may fall apart, but it will never completely dissolve under the conditions found in public land fills. The test stand and the accelerated chamber have one thing in common with each other which is that we are supplying all of the necessary ingredients necessary for complete degradation of any substance: light, heat, moisture and oxygen (air). Public land fills on the other hand do not supply these ingredients after we have buried materials over a certain depth (approximately six feet down). The testing performed with these procedures are of the type that assume the "best case" scenario which is when all of the necessary components are present.

EXPERIMENTAL ORDER

1.--PURCHASE BIODEGRADABLE TRASH BAGS
2.--CONDITION BAG SAMPLES ACCORDING TO ASTM STANDARDS
3.--CONSTRUCT ASTM WEATHERING SAMPLE FIXTURE OR PREPARE THE ACCELERATED WEATHERING APPARATUS IN ACCORDANCE TO THE RECOMMENDED MANUFACTURERS DIRECTIVES
4.--WEIGH EACH SAMPLE ACCURATELY BEFORE AND AFTER LABELING IN ORDER TO DETERMINE ACCURATE WEIGHT LOSS OF THE SAMPLE DURING TESTING (CORN STARCH LOSS)
5.--RECORD TEST CONDITIONS AT THE BEGINNING OF THE TEST AND AT EACH SAMPLE POINT ALONG WITH THE SAMPLES EXPOSURE LIMITS DURING TESTING (TEMP., WEATHER CONDITIONS, ETC.)
6.--PULL A REPRESENTATIVE SAMPLE OF PRE-TEST CONDITIONED SAMPLES AND TEST THEM USING THE TENSILE TESTER IN ORDER TO GET A STARTING POINT FOR THE DATA COLLECTION
7.--MOUNT THE TEST STRIPS TO THE WEATHERING APPARATUS IN ACCORDANCE TO ASTM PROCEDURE
8.--PULL A REPRESENTATIVE SAMPLE FROM THE TEST APPARATUS AT REGULAR INTERVALS AND CHECK FOR WEIGHT LOSS. IF NO MEASURABLE LOSS HAS OCCURED, RE-MOUNT THE SAMPLES AND CONTINUE TO LET THEM AGE. IF WEIGHT LOSS IS DETECTED, CONDITION THE SAMPLES AND TEST USING THE TENSILE TESTER. KEEP CORRECT DATA CORRELATIONS WITH THE TIME, TEMPS; AND CONDITIONS IF POSSIBLE (THIS IS WHERE THE ACCELERATED TESTING APPARATUS IS IDEAL AS YOU CAN RECORD THE EXACT CONDITIONS EXPERIENCED BY THE SAMPLES.).
9.--CONTINUE TO TEST UNTIL COMPLETE LOSS OF STRENGTH IS EXPERIENCED ON A CONSTANT BASIS (LOSS OF POLYMER MATRIX INTEGRITY) AT WHICH TIME YOU MAY CONSIDER THE MATERIAL TO BE DEGRADED ENOUGH TO END THE EXPERIMENT. YOU WILL NO DOUBT NOTICE THAT ALTHOUGH THE PROPERTIES ARE TOTALLY RUINED, THE MATERIAL IS STILL INTACT AS FAR AS IT'S PHYSICAL BULK IS CONCERNED.

NOTE: FOR AN INTERESTING COMPARISON, PERFORM THE EXACT SAME TESTING THAT IS OUTLINED ABOVE WITH A REGULAR TRASH BAG TO CORRELATE THE DIFFERENCES ENCOUNTERED WITH THE "BIO-DEGRADABLE" MATERIAL.

RESULTS AND DISCUSSION

What the students (and the un-informed instructor) will ultimately conclude is that unlike food stuffs, polymer materials, even with the corn starch additive are for the most part resistant to biodegradation. Even when the additive is removed the polymeric chains are still present in a far less space consuming form, but they are still present. As the plastics industry is reported to only contribute approximately 5% of the total land fill composition (by weight), we find that the problem of eliminating the materials through biodegradation is of questionable benefit. The one positive solution to the problem is to be found in such programs as source recycling whereby the materials are re-used. This is, in fact, one of the plastics industry's most endearing qualities with it's ability to recycle it's waste materials.

The test will however, demonstrate the effects of using special additives to polymeric materials to help achieve desired end results. The corn starch additive it must be recognized, does not alter the molecular structure of the polymer. It is instead simply being used as a sort of "space or place holder" that is later dissolved and eaten away by micro-organisms. When the starch dissolves, the hole left by the eroded mass is responsible for the collapse of the structure, but not the decomposition of the polymer matrix its self. We have had samples hanging outside for nearly a year now and have seen only minimal changes in a disposable (biodegradable) trash bag that is used in automobiles. This item was given to me to use when I took aluminum cans to a recycling drive. The item obviously had a curious attraction for me to test the properties of the "new" biodegradable polymers. The bag has proved to be as strong as the non-biodegradable bag that was hung next to it. The other bag is being used as a standard with which we have to compare to. The data for these tests is still forthcoming and I therefore have no sound correlations to report.

PERSONAL INSTRUCTION NOTES

If it if feasible from a financial standpoint, I feel strongly that the accelerated chamber method of sample preparation is the most effective. The amount of time required to get good results by hanging the samples outside is prohibitive to the success of these tests.

This test provides the essential elements of student interaction and curiosity as the subject matter is a newer, more current issue that has been given maximum air time on the different media services. Most people have already formed an opinion as to the causes of public land fill problems and are therefore startled to find that the plastic or polymer content of the dump is relatively small. The problem still exists for the industry to address, however. Additives in the matrix of polymeric materials is only one of the answers to a very complex problem.

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Freshman Chemistry and Materials Science:
Merging the Microscopic With the Macroscopic

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Introduction

Among the most exciting prospects for the benefit of society are high temperature superconductors, organic thin films for optical communication, and polymers which can function as prostheses. Each presents a "materials problem," and engineering undergraduates from virtually all disciplines will encounter problems like these. Solutions to these challenges demand an appreciation of many issues, including the chemical route(s) by which a material is synthesized, the effect the preparation and processing steps have on various levels of structure (atomic to 'bulk'), and how structure ultimately affects properties. Thus, a solution to a materials problem requires understanding of both microscopic (e.g., electronic structure and bonding) and macroscopic (e.g., fracture strength, electrical conductivity) phenomena. What we wish to advocate in this article is a *merging of the microscopic with the macroscopic in undergraduate chemical education, beginning with freshman chemistry*. The key difference in a freshman chemistry course with a more macroscopic emphasis is the set of examples selected to illustrate chemical principles. We present here motivations for this shift in direction, and offer an approach to implement the integration of the microscopic with the macroscopic.

Chemistry and materials science are inextricably linked. In fact, in the National Research Council's "Opportunities in Chemistry," familiarly known as the Pimentel Report,[1] can be found the following definitions from Webster: a *material* is "the substance or substances out of which a thing is constructed," and *chemistry* is "the science that deals with the composition, properties, and changes of properties of substances." However, there is a perception that chemistry is concerned primarily with microscopic phenomena. Nowhere is this attitude more evident than in a freshman chemistry textbook. While occasional chapters on the solid state can be found, these typically dwell on atomic packing. Virtually no mention is made of material properties such as modulus, electrical conductivity, and transparency, and how these are dictated by atomic and molecular composition and structure. If solids are discussed, imperfections such as point and line defects are almost never mentioned, and yet these dictate, for example, mechanical properties. This approach is unfortunate, as it has been pointed out quite clearly that tremendous opportunities exist for chemists to participate in the exciting area of materials science. For example, in the Pimentel Report, materials chemistry played a prominent role, and the interdisciplinary nature of materials chemistry was stressed. It was stated that "chemists are increasingly joining and expanding the specialist communities concerned with glasses, ceramics, polymers, alloys, and refractory materials." Furthermore, the report predicted that "coming years will see entirely new structural materials, liquids with orientational regularity, self-organizing solids, organic and ionic conductors, acentric and refractory materials." It should be noted that a companion report, "Frontiers in Chemical Engineering" (National Research Council, 1988), expressed similar opportunities in the materials area for chemical engineers.[2] That more and more chemical research activity is indeed being directed toward the materials area was responsible for the American Chemical Society's launching of a new journal, *Chemistry of Materials*, in 1989. Dr. Mary Good, former ACS President, recently edited a book published by the American Chemical Society which speaks directly to the point.[3] The title? "Biotechnology and Materials Science: Chemistry for the Future." In order to meet these challenges, it has been suggested that changes in chemical education may be needed "to keep chemists in the center of revolutions in materials and biological sciences."[4]

Perhaps the most compelling arguement for at least considering a shift toward a more macroscopic focus in freshman chemistry comes from engineering faculty. At a number of institutions, engineering faculty have complained that introductory chemistry is not terribly relevant to the needs of their undergraduates. What they mean (in part) is that their students are not learning enough about the solid state and, hence, about materials. For example, many electrical engineering faculty believe that students should be exposed to semiconductors and how simple devices can be constructed from them. Another way of saying it is that freshman chemistry is not applied enough for engineering needs. But is it not, afterall, the application of chemistry that fascinates chemists?

The reluctance of chemistry faculty to respond to these needs has led to the initiation of 'alternative' chemistry courses taught by engineering faculty. For example, for the about the last 15 years, freshmen at MIT may elect to take their one-semester requirement in chemistry in the form of "Introduction to Solid State Chemistry." This course is taught exclusively by the Materials Science and Engineering Department. Roughly one-half of the freshman class takes this course instead of the Chemistry Department's offering. Northeastern University now teaches a second semester freshman course emphasizing materials chemistry for honors students. At the University of Pennsylvania and the University of Arizona, freshmen engineering majors can take their second semester chemistry course in solid state chemistry, again taught by materials science and engineering faculty. While these are programs are ones with which the authors are familiar, there are likely to be many more in progress or in the planning stages.

Should this shift toward materials chemistry be taken seriously? The answer in brief is 'yes' if students are consulted - materials topics bring vitality to freshman chemistry, which should be fascinating but many times falls short of this goal.

An Approach to the Microscopic/Macroscopic Merger

At Rensselaer Polytechnic Institute, we are in the third year of teaching a two-semester freshman chemistry course which emphasizes materials chemistry. This 'pilot' course, being taken by about 180 engineering students selected at random in 1990-1991, is co-taught by faculty from the

Departments of Chemistry and Materials Engineering. The development of course materials, as well as the staffing of both lectures and recitations, are shared equally by the two Departments, as this is in the truest sense a cooperative venture. Summarized in this section are the philosophies about the operation of the course and an outline the subject matter.

The organization of a typical freshman chemistry textbook is inappropriate for a course aimed at teaching the basics of chemistry using materials examples. Chapters on bonding, for example, are frequently in the middle of a book, whereas we insist that bonding be the first subject discussed in detail. We strongly believe that what is required is a hierarchical approach which begins with the structure of the atom and continues to molecules and then collections of atoms or molecules into various superstructures (i.e., condensed phases). The fundamentals of thermodynamics, kinetics and the solid state are discussed next to prepare students for the remainder of the course. Students will learn, for example, in detail why metals are electrical conductors and malleable, whereas most ceramics are insulators and are brittle. They also learn why structural metals are not as strong as one might predict based upon an ideal (perfect) crystal lattice, and how it is possible for solid-state chemical reactions to alter the strength of an alloy. To understand macroscopic properties, it is absolutely essential that the microscopic world of atoms and electrons be understood. This *is* modern chemistry, and it epitomizes what is meant by a microscopic/macroscopic merger.

Because no chemistry textbook had an order of presentation which was deemed appropriate, we developed the course outline (Table 1) without reference to any particular text. It reflects the hierarchical approach mentioned earlier. Once this was established, readings were assigned from the texts selected (Gillespie et al., *Chemistry*, 2nd ed., Allyn & Bacon, 1989; Shackelford, *Introduction to Materials Science for Engineers*, 2nd ed., Macmillan (1988)). In many instances, the texts' treatment of a particular subject may be weak or non-existant, and therefore we provide students with detailed lecture notes. Students meet for two large lectures per week and two smaller (ca. 25 student) recitations. The first of the two recitations takes on the character of a small but more personal lecture, where concepts presented in the texts and in the main lectures are built upon. The relevance of the course material to the 'real world' is particularly stressed. The second recitation serves as a problem-solving session, and weekly quizzes are given here to monitor student progress.

Table 1. Abbreviated RPI Freshman Chemistry/Materials Course Outline

Part 1: Structure and Bonding

Nuclear and electronic structure; properties of light; simple quantum theory; the periodic table; creation of elements; bonding; hybridization and molecular geometry; resonance; the states of matter.

Part 2: Thermodynamics and Kinetics

Equilibrium; acids and bases; thermochemistry and bond energies; the laws of thermodynamics; kinetics: simple collision theory and activation energy; chain vs. non-chain reactions; catalysis.

Part 3: The 'Ideal' Solid State

Atomic, ionic, covalent and network solids; bonding and packing of spheres; band theory; cubic crystals; evidence for periodicity (x-ray diffraction); metallurgy; mechanical properties of 'ideal' crystals.

Part 4: The Imperfect Solid State

'Real' crystals. Compositional and structural imperfections; their influence on material properties. Glasses.

Part 5: Phase Diagrams: Equlibrium 'Maps'

Two-component diagrams with and without solid solubility; the Gibbs phase rule; lever rule; illustrative systems (e.g., Pb/Sn solder, and Fe/FeC (steel)).

Part 6: Kinetic Processes in Solids

Diffusion; reactions in/on solids; sintering.

Part 7: Classes of Materials

Metals (conduction, mechanical properties); ceramics (brittle fracture, glasses); polymers (synthesis and properties of macromolecules); electronic materials (semiconductors, simple electronic devices, device fabrication).

Part 8: Materials Synthesis

Inorganic and organic reactions; use of thermodynamic and kinetic arguments to select reaction conditions; influence of synthesis pathway on structure.

It should be emphasized that the order of the presentation allows continuous reinforcement of concepts. For example, Part 6 calls for an appreciation of activation energy, a concept already discussed in Part 2 using gas-phase kinetics but now applied to kinetic processes in solids. Parts 7 and 8, which last about eight weeks, provide the opportunity to 'put it all together.' For example, among the many phenomena discussed in Part 7 is the fracturing of glass.[5] The rate of crack propagation is known to be enhanced in the presence of water. Hydrolysis of Si-O-Si bonds occurs and is facilitated by bond angle strain at the crack tip, the latter being dependent on the magnitude of the applied stress. A detailed discussion serves to illustrate, among other things, (1) nucleophilic attack by water on Si and why silicon is more reactive than carbon towards nucleophilic attack, (2) the effect of bond angle strain on chemical reactivity, (3) the effect of molecular size on reactivity at the restricted geometry of the crack tip (methanol and aniline react more slowly than water), and (4) a macroscopic phenomenon is dictated by events occurring at the microscopic (atomic) level. These ideas are important, and the interesting example of glass fracture provides a vehicle to promote an interest in learning chemistry. Part 8 attempts to merge what chemists know how to do particularly well, that is to make things, with a materials scientists' detailed knowledge of how atomic and molecular structure affects properties. Virtually all of the concepts presented earlier are reinforced here.

The outline mentioned above is obviously quite different from that of a typical freshman course. While many traditional experiments can be readily employed, the course content calls for new experiments. Our goal for the coming year is to develop interesting laboratory experiments which are relevant to the lecture material and which can be performed with the large throughput of students in a freshman course. Experiments under development include precipitation hardening of an aluminum alloy, the synthesis of diamonds, the growth of a metal/semiconductor junction and its electrical properties, anisotropic etching of Si single crystals, absorption spectroscopy of transition metal ions in glasses, and construction of a phase diagram. Each is intended to provide a conceptual link between a macroscopic property and its atomic/molecular level origin. The near-term plan is to offer the course in 1991-1992 to all engineering freshmen at Rensselaer (approximately 700 students). Our longer-range goals are to develop a textbook based upon the outline in Table 1 along with a laboratory manual.

Additional details and course material will be provided for the asking.

Acknowledgements

The authors wish to thank their colleagues D. A. Aikens, R. H. Doremus, D. J. Duquette, J. B. Hudson, K. J. Miller, J. A. Moore, C. T. Moynihan, S. Murarka, K. Rajan, R. R. Reeves, C. Steinbruchel, R. L. Strong, and S. E. Wiberley for their effort in making the course described a reality. Financial support from Rensselaer's Office of the Provost, the Deans of the Schools of Science and Engineering and the Departments of Chemistry and Materials Engineering, and the General Electric Company Foundation is gratefully acknowledged. We thank our 'pilot' students for their thoughtful comments and suggestions.

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3. M. Good, "Biotechnology and Materials Science: Chemistry for the Future," ACS, Washington, 1987.
4. B. Hileman, *Chemical and Engineering News*, Oct. 26, 1987, p. 34.
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INTRODUCTORY MATERIALS LAB

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INTRODUCTION

Freshmen in Engineering Technology at Purdue must take a basic "core" course in Materials and Processes (MET 141) with two one-hour lectures and one two-hour laboratory. As usually scheduled, our initial lab session precedes the first lecture. Also half of these students have had little or no math and science since junior high school. The first lab is designed to serve as an orientation to draw students' attention and create a need for understanding engineering materials; to arouse students' curiosity and give insight into some principles of science; and to encourage students to develop general technical skills in the field of materials technology. The objectives of this lab are: (1) establish a broad outlook for classification of engineering materials; (2) introduce basic concepts and vocabulary in materials technology; (3) make qualitative observations and measure or gage quantitatively; (4) use and review basic math and science.

PROCEDURE

The instructor gives an overview of engineering materials, classifications, and properties. Complex tests like specific gravity and thermal conductivity may be demonstrated for the whole class. Then with 12-students per lab, individual students (or teams) may select a task (or setup). Observations (or data) are recorded on the WORKSHEET (see page 2). Students then move to the next setup. Some tasks are "fill-in-the-blank"; others require "homework calculations," neatly and legibly written on separate sheets, stapled to the "WORKSHEET."

Grading is based on logical answers, correct conversions, and appearance. Scoring (24 points) is about 10% of total lab points (25% of course grade). Different samples and types of materials are introduced each semester, to encourage "student initiative" and "foil the files." Alternative setups can be introduced as desired or required to elucidate other "PHENOMENA" (see page 3).

COSTS

Materials and exhibits may be selected from discarded test specimens, broken parts, or household items. Each setup can be stored in boxes or stacked trays. Standard sets of metallics, plastics, and ceramics may be purchased:

Metals kit (36 pieces)	$200
Plastics kit (25 pieces)	60
Ceramics kit (12 pieces)	10
Baby scale, for weighing kilograms, or pounds	50
Beam balance, for buoyancy/density experiments	200
Magnifiers (10x)	5
Brinell scope (20x)	20-300
Micrometer caliper	50
Vernier caliper	50
Torque wrench (with socket)	50

WORKSHEET

MET 141L, DIV. #________ DATE:________ NAME:____________________

INTRODUCTION TO MATERIALS

1. METALS:	Al	Brass	Cu	Mg	Nb	Sn	Ta	Ti
Main Color?	________	________	________	________	________	________	________	________
Oxide Tint?	________	________	________	________	________	________	________	________

2. DENSITY:	Metal (steel)	Ceramic (glass)	Plastic (acrylic)
Weight?	________________	________________	________________
Volume?	________________	________________	________________
∴ lb/ft^3 =	________________	________________	________________

3. SPECIFIC GRAVITY/RELATIVE DENSITY:
Dry Sample = ____________kg, Wet Sample = ____________kg, Lost = ____________kg.
∴ S.G. = _________. Sample is __________ (Mg = 1.7, Al = 2.7, Ti = 4.5 Fe = 7.9).

4. GRAINS:	Galv. Zn Plate	Brass	Al Elbow	ASTM Chart
Grains/in^2 = N =	______________	N = __________	N = __________	N = __________
If N = $2^{(n-1)}$, then n =	________	n = __________	n = __________	n = __________

5. MAGNET: List the materials which the magnet attracts. ______________________
____

6. BRINELL SCOPE: What is its magnifying power?______________________
Does the scope read (a) cm and tenths, or (b) mm and tenths?______________________
Does "power" multiply (c) Length, or (d) Area?______________________

7. MICROMETER: Read mils (1/1,000 in) and tenths (1/10,000 in).
The dry IBM card is __________ mils. The wet IBM card is __________ mils.
(thickness) (thickness)

8. TEMPERATURE vs. COLOR: Heated steel will oxidize and cool to various colors, so light yellow forms at _________°C, and light blue forms at _________°C.
∴ Plain steel conducts heat (a) faster, or (b) slower than stainless?____________

9. METRIC RULER (Stainless Steel): Measure a large paper clip.
Width = _____ cm, Length = ______ cm, Wire dia. = _____ mm. And 1-inch = _____ cm.
(S.I. metric units).

10. SHEARED EDGE: (a) a <u>shiny-cut</u> occurs in ductile-metal.
(b) a <u>dull-break</u> occurs in work-hardened-metal. In a punch-disk, what occurs first? ______________________________

11. TENSILE BAR: D_F = __________", D_O = __________", L_F = __________", L_O = 2".
Ductility index = % area reduction = 100 (A_O - A_F) ÷ A_O = __________%.
Elongation = linear strain = (L_F - L_O) ÷ L_O = ___________in/in.

12. SPARKS: Using electrical grinder and chart, identify the "unknown metal bar" as ______________________________?

PHENOMENA

I. **Surface Grains**--Frost, zinc, brass, aluminum.

II. **Fractures**--Cast iron, Duriron (with silicon), Aluminum plug, Brass piece. Welded steel pieces; Blistered Steel. Fatigue rods (steel, brass, aluminum); Torsion shaft and bolt.

III. **Machining**--Stainless steel, Mild steel, Brass, Aluminum, Copper.

IV. **Shearing and Punching**--Aluminum and Steel strips, discs.

V. **Sounds**--Hit and drop specimens.

VI. **Ball and Tube**--Simple rebound scleroscope; Rough surfaces; Dents.

VII. **Penetration**--Using hammer, awl, knife, chisel, file, saw. Making dent, scratch, cut, chip/burr, dust/groove.

IX. **Vise**--Bending tin specimens (creaky) and solder bar (silent). Sparking cerium specimen (Ce).

X. **Grinder**--Shower of sparks; vary Carbon %; need chart and experience.

XI. **Magnet**--Ferrous metals, Nickel electrode, Stainless steels; Non-ferrous metals; Non-metals; Ceramics.

XII. **Specific Gravity**--Zero the scale; Weigh sample in air and in water. Calculate S.G. = Air Wt./Wt. Loss. Compare published values (ABS, Steel, Cu, Pb, Brass).

XIII. **Thermal Conductivity**--Time to melt wax at end of rod. Specimens of Stainless and Mild Steel, Cu, Brass and Aluminum. Compare published values (thermal and electric).

XIV. **Thermal Heat Capacity**--Constant weight specimens (Cu, Al, Brass, Steel, Stainless) and constant volumes of water; measure maximum temperature rise; compare published values.

XV. **Melting Temperatures**--Time to melt same size ball (Pb, Sn, Solder of 50% Lead/50% Tin). Refer to "Equilibrium Phase Diagram" and predict melting point of "Eutectic mix" (63% Tin, 37% Lead). Discuss wiping solder (non-eutectic) and potable water (no lead) in copper pipes (95% Sn, 5% Sb).

XVI. **Oxidation and Corrosion**--Hot steel colors; Cold oxide colors; Crayons ("Tempil Stix"); Rust and Tarnish (Fe, Cu, Brass); Brass tubing (Sulfur in Natural Gas); PVC (plastic degradation).

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3. Widener, E. L., "Materials Labs: MET Department, Purdue University," Kinko's Copies, West Lafayette, IN, 1989.

DIAMOND SYNTHESIS WITH A COMMERCIAL OXYGEN-ACETYLENE TORCH

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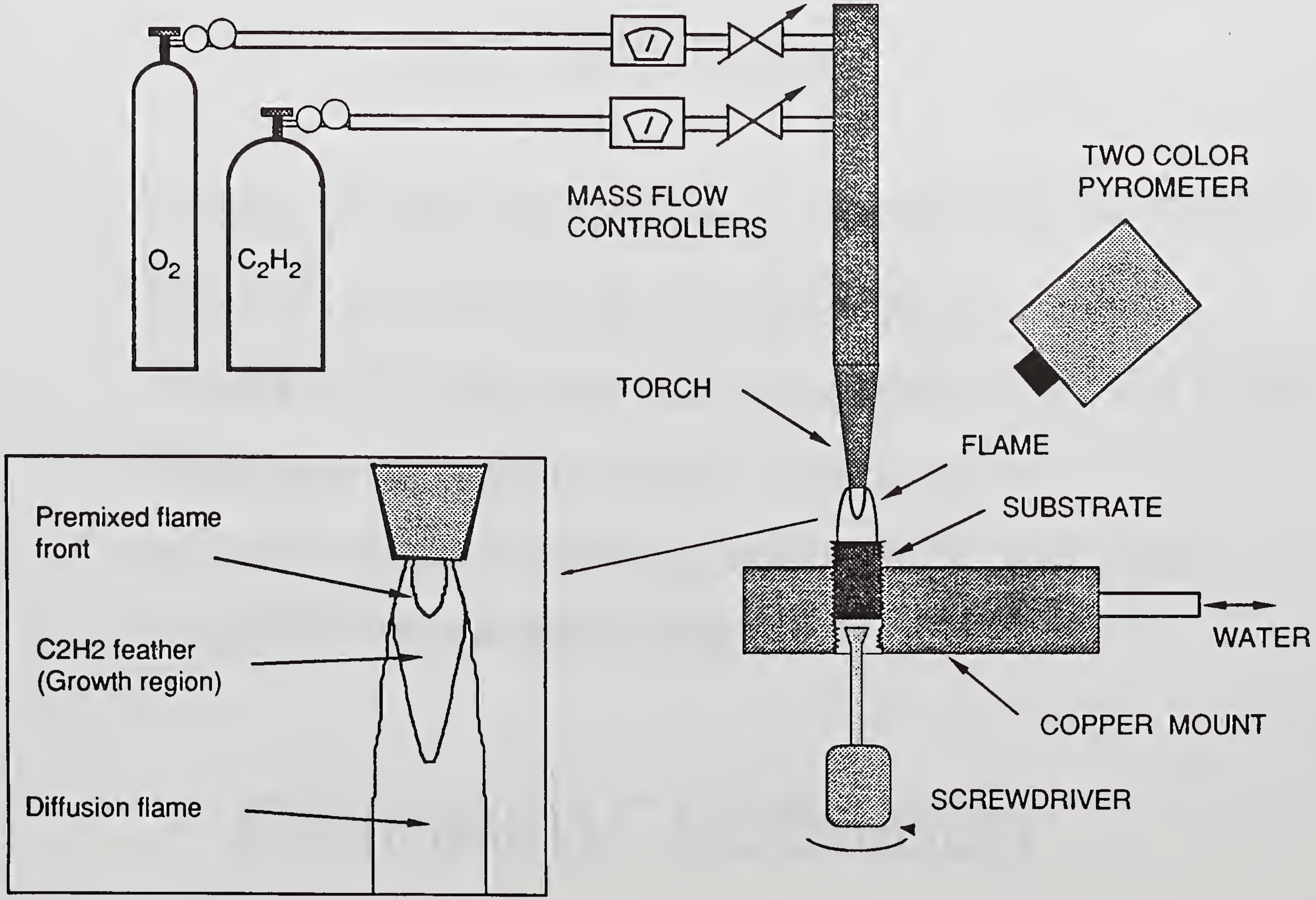
ADVANTAGES

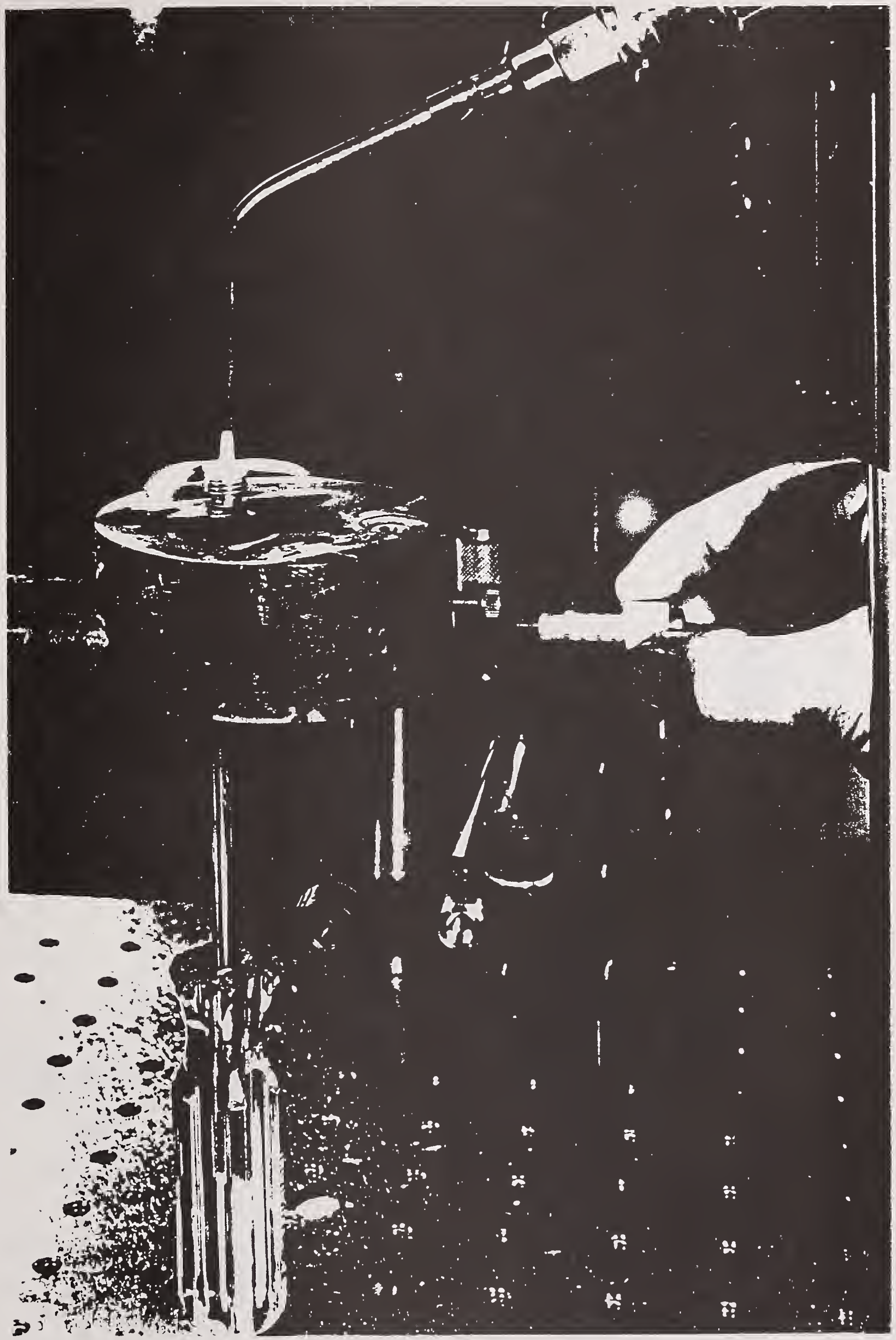
- VERY LOW COST (< $500)
- HIGH GROWTH RATES (~50 μm/hr)
- NO VACUUM SYSTEM REQUIRED
- NO ELECTRICAL POWER REQUIRED
- CRYSTALS OBSERVABLE WITH STANDARD OPTICAL MICROSCOPE

EQUIPMENT REQUIRED

- OXYGEN-ACETYLENE TORCH
- WATER COOLED COPPER HEAT SINK
- SUBSTRATES (Mo, Si, etc.)
- GASES (O_2, C_2H_2), TUBING, FITTINGS & REGULATORS
- BALL-IN-TUBE IN-LINE FLOWMETERS
- DISAPPEARING FILAMENT PYROMETER (OPTIONAL)

EXPERIMENTAL SETUP





(See schematic on page 312.)

FACTORS AFFECTING DIAMOND GROWTH

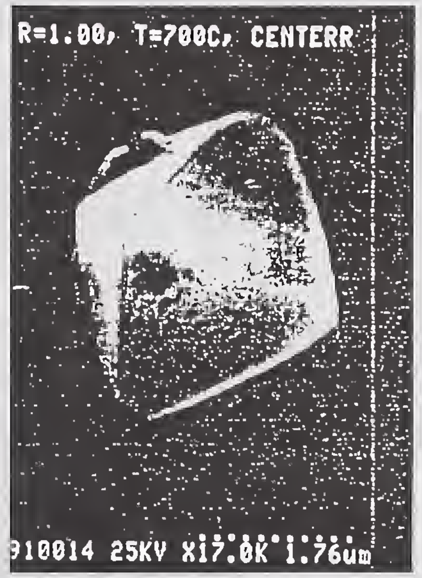
- SUBSTRATE TEMPERATURE
- OXYGEN / ACETYLENE FLOW RATIO
- SUBSTRATE MATERIAL (Mo, Si, etc.)
- SUBSTRATE FINISH
- SUBSTRATE POSITION IN FLAME
- POSITION ON SUBSTRATE

USEFUL TIPS

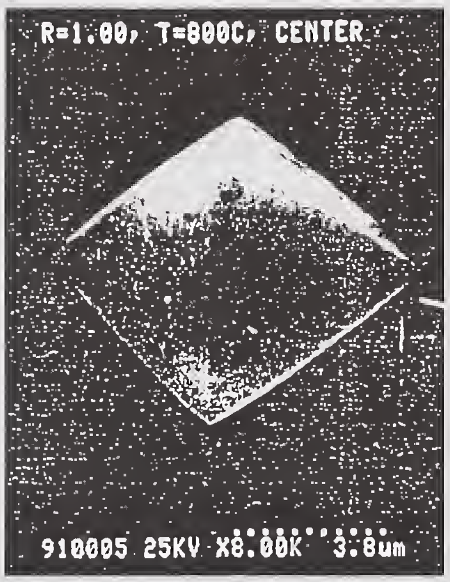
- USE THREADED ROD FOR SUBSTRATE - ADJUST TEMP. VIA PENETRATION INTO WATER COOLED Cu BLOCK
- PRE-SCRATCH SUBSTRATE WITH DIAMOND GRIT
- USE CARBIDE FORMING SUBSTRATE (e.g. Mo, Si, W, Ta, etc.)
- IN LIEU OF PYROMETER, ADJUST TEMP. SO SUBSTRATE JUST BEGINS TO EMIT VISIBLE LIGHT (~900°C)
- ADJUST FLAME SO EXCESS C2H2 FEATHER IS ABOUT 2x LENGTH OF PREMIXED, CONICAL FLAME FRONT
- DO NOT USE LARGE ORIFICE TIP (CAUSES TURBULENCE)

MORPHOLOGY vs. DEPOSITION TEMPERATURE

(O_2/C_2H_2 = 1.0, total flow = 3.0 slm, center of substrate)

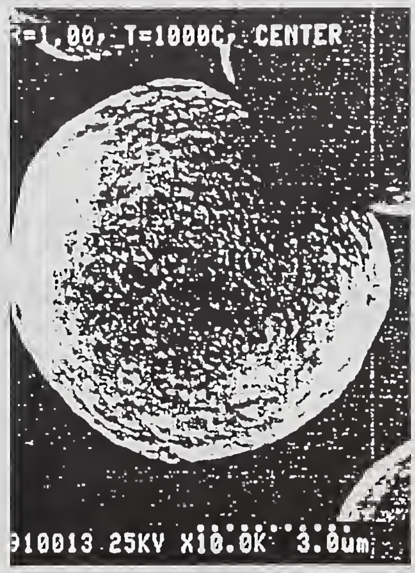


700°C

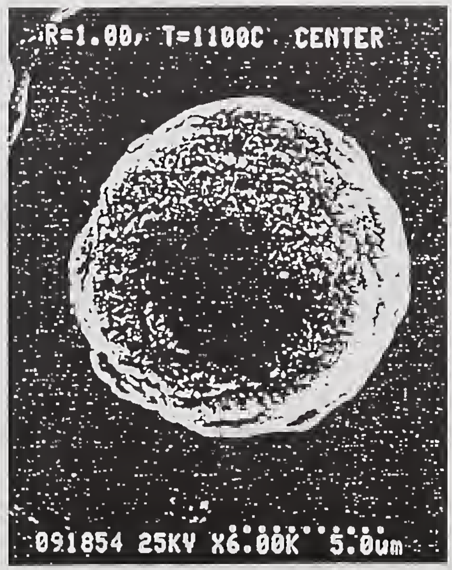


800°C

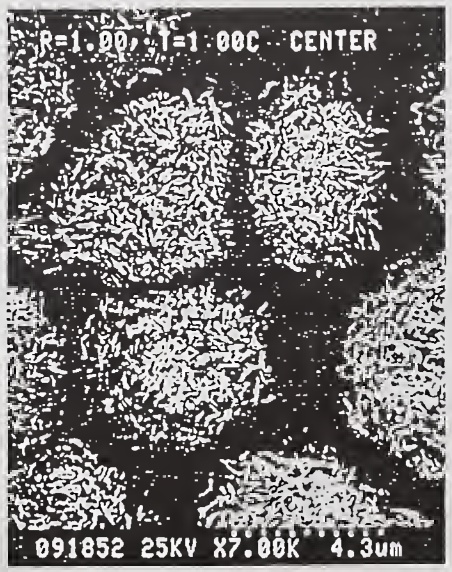
900°C



1000°C



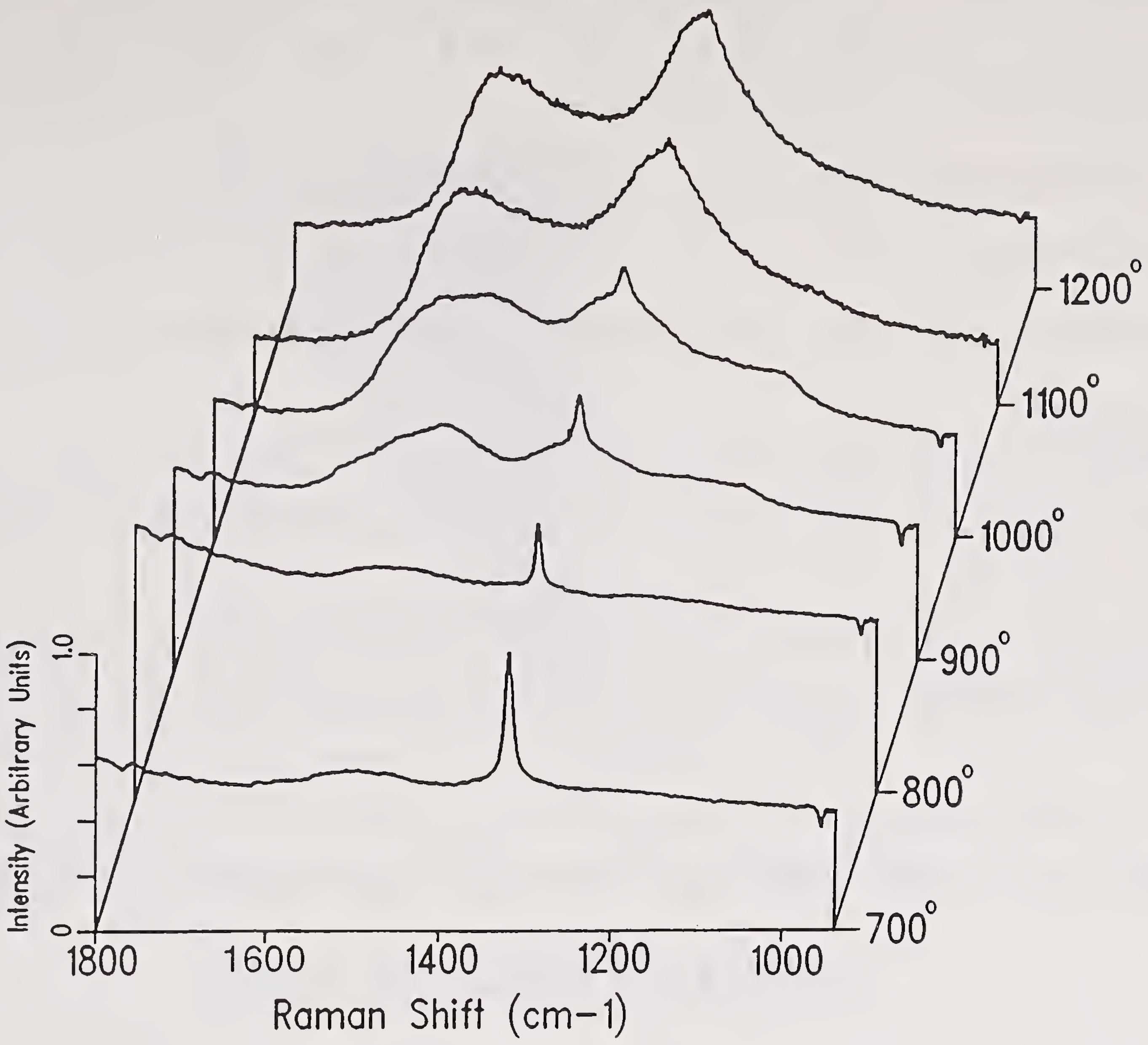
1100°C



1200°C

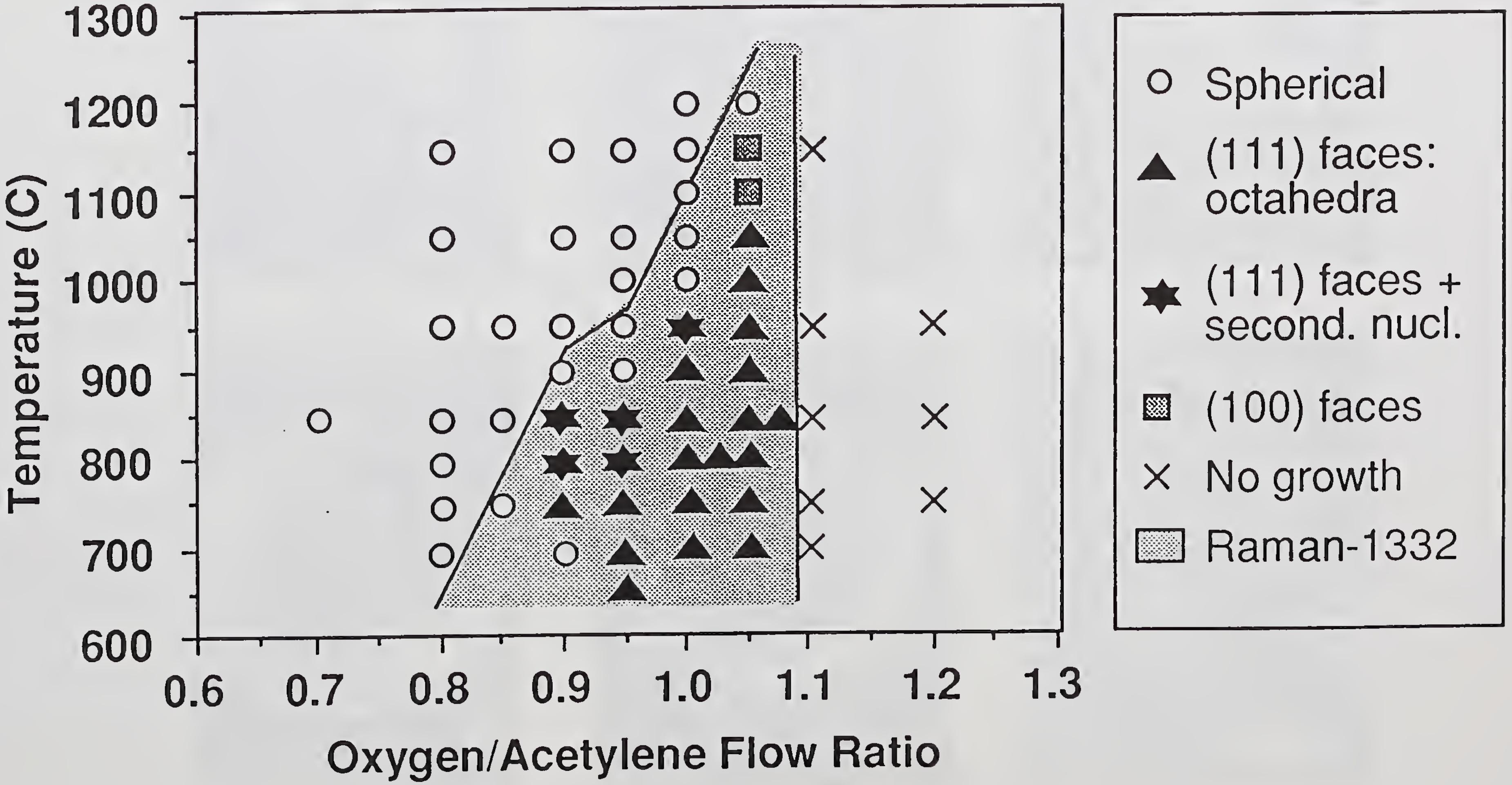
RAMAN vs. DEPOSITION TEMPERATURE

(O_2/C_2H_2 = 1.0, total flow = 3.0 slm, center of substrate)



Morphology Map

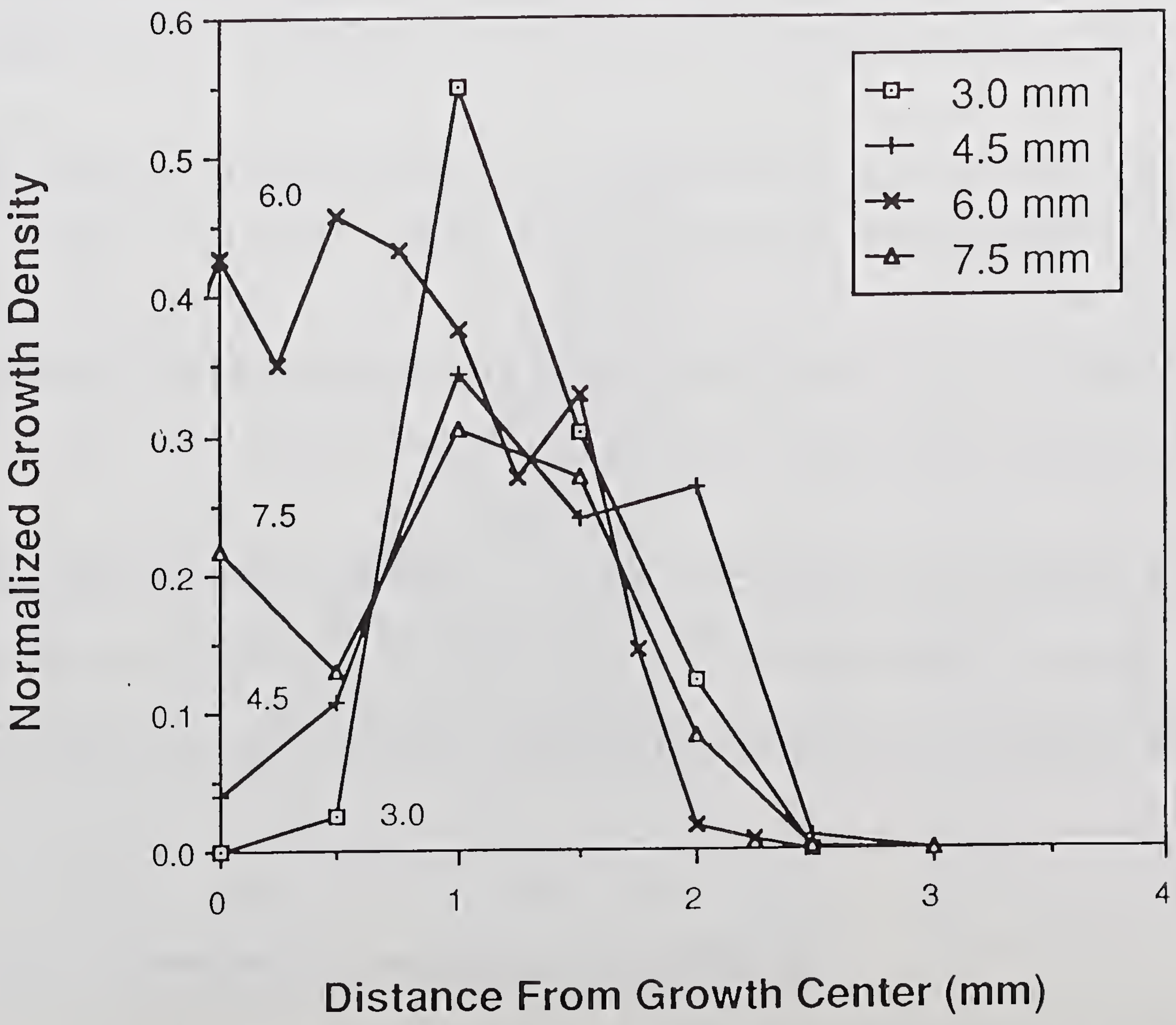
(O2-C2H2 flame, ctr. of substrate, total flow = 3.0 slm)



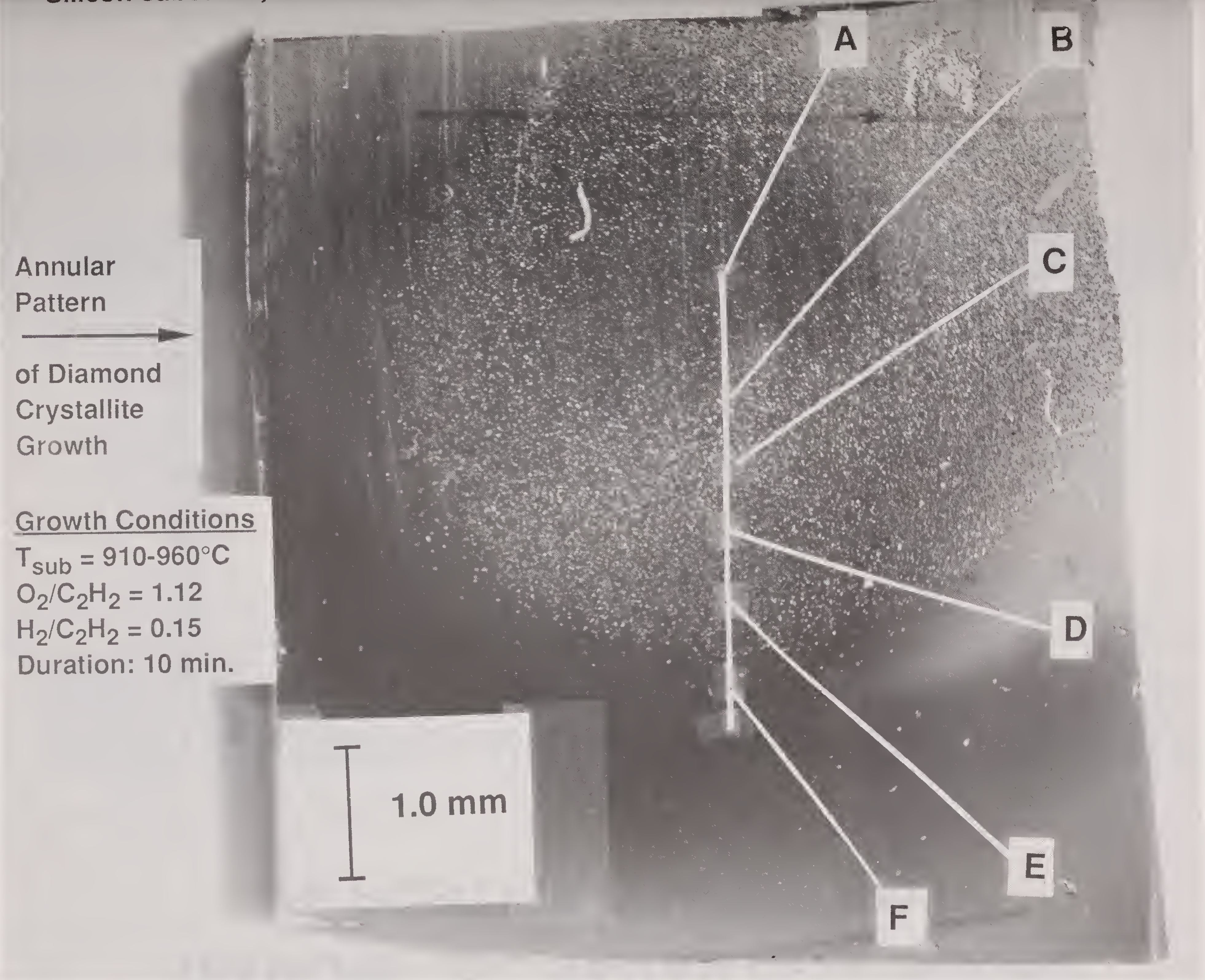
VARIATION WITH POSITION IN FLAME

- SERIES OF 10 min. GROWTHS PERFORMED ON Si IN AN $(O_2+C_2H_2)$ FLAME WITH $O_2/C_2H_2 = 1.12$ and $T_{sub} = 800\text{-}890°C$ FOR DIFFERENT SUBSTRATE - FLAME FRONT DISTANCES (D)
- FOR D = 3.0 - 4.5 (±0.5) mm, CRYSTALLITE GROWTH DENSITY WAS HIGH IN AN ANNULAR REGION AND LOW ELSEWHERE
- FOR D = 6.0 - 7.5 mm, GROWTH DENSITY WAS HIGH DIRECTLY UNDER THE FLAME AXIS & DECREASED WITH RADIAL DISTANCE
- TEMPERATURE GRADIENT ACROSS GROWTH REGION ON SUBSTRATE DECREASED WITH INCREASING D

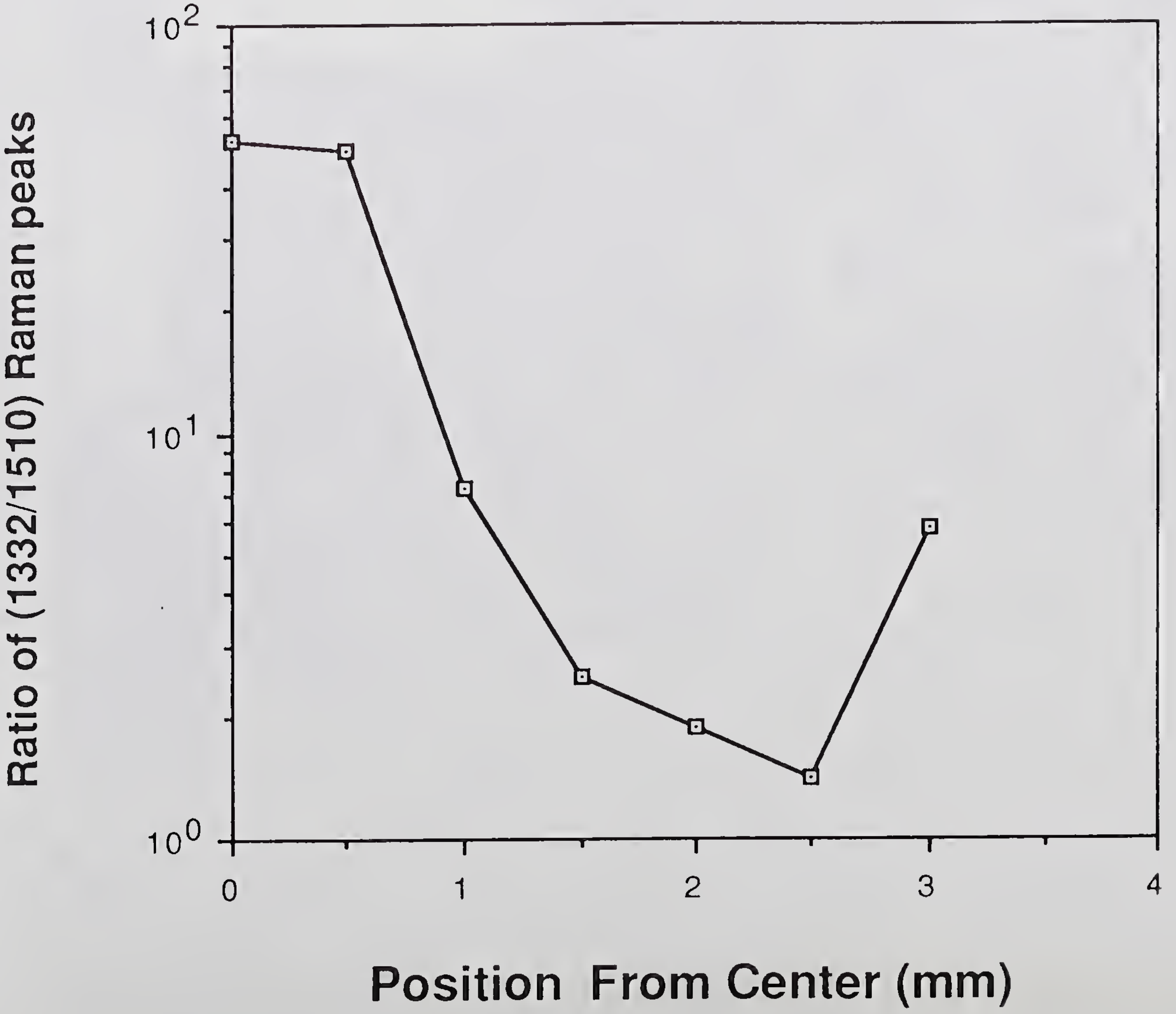
GROWTH DENSITY vs. POSITION IN FLAME



Silicon substrate, scratched with 5 μm diamond paste

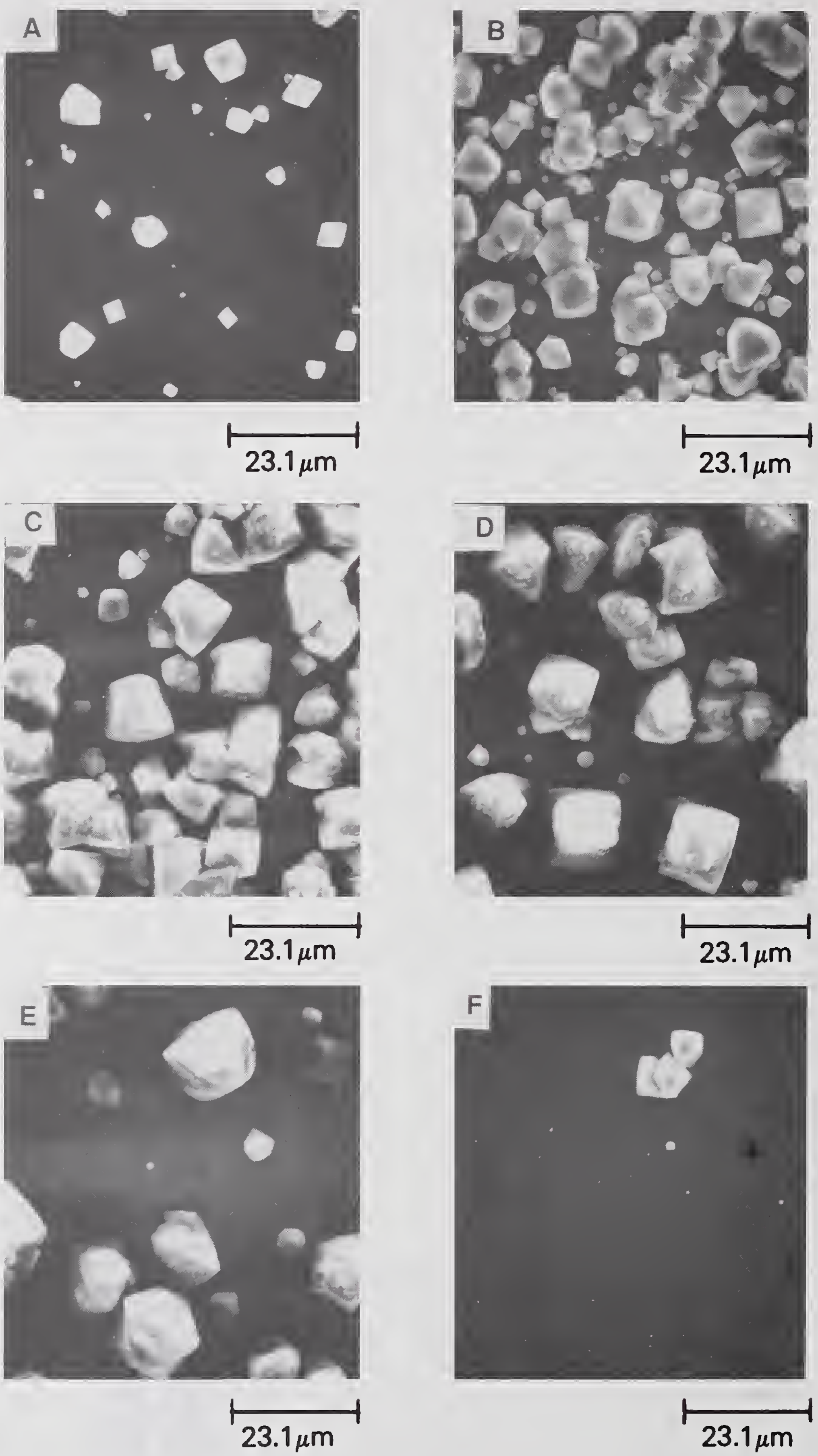


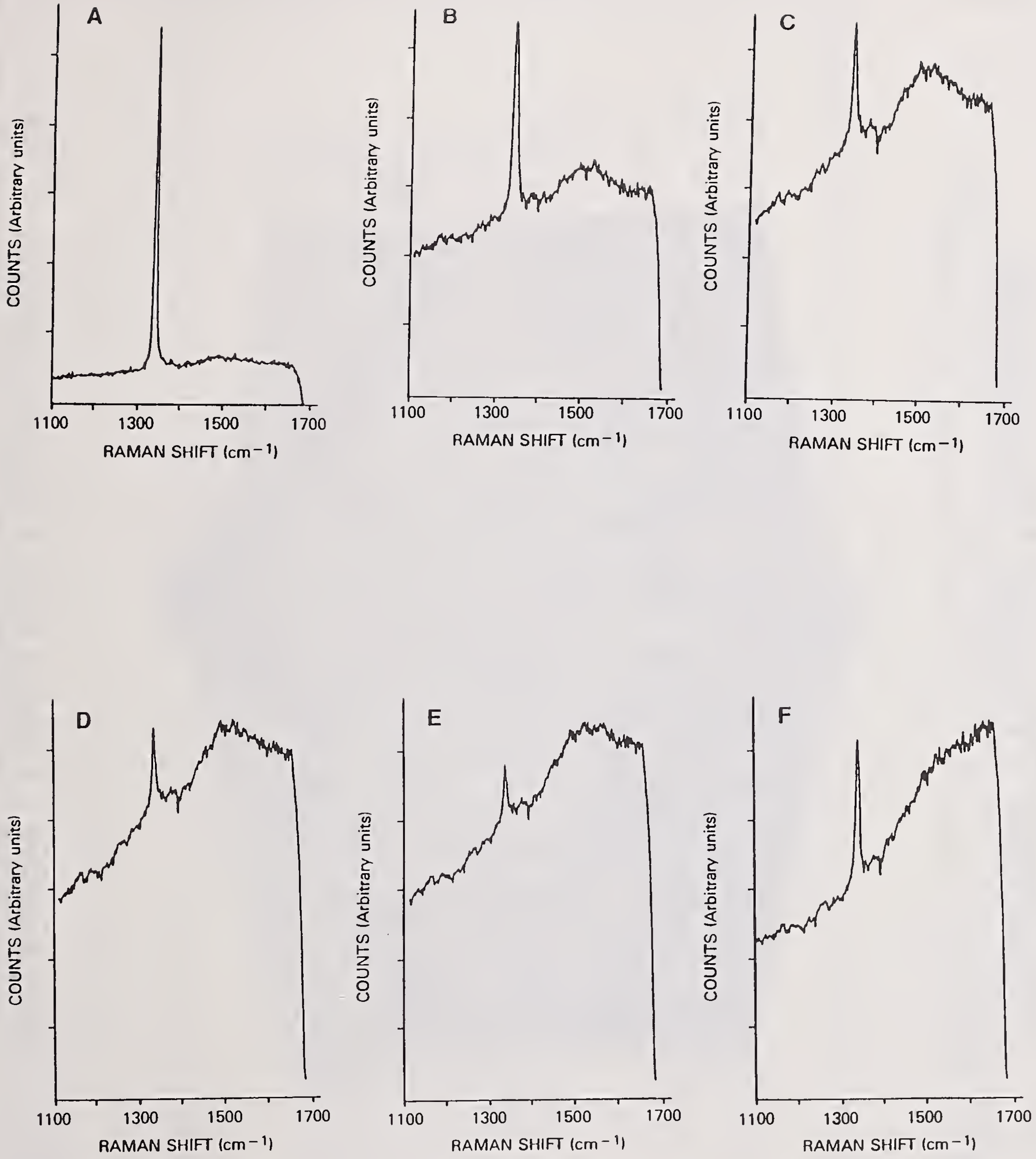
DIAMOND / a-C RAMAN PEAK HEIGHT vs. POSITION ON SUBSTRATE



IS NON-UNIFORM GROWTH DUE TO
TEMPERATURE OR SPECIES INHOMOGENEITIES?

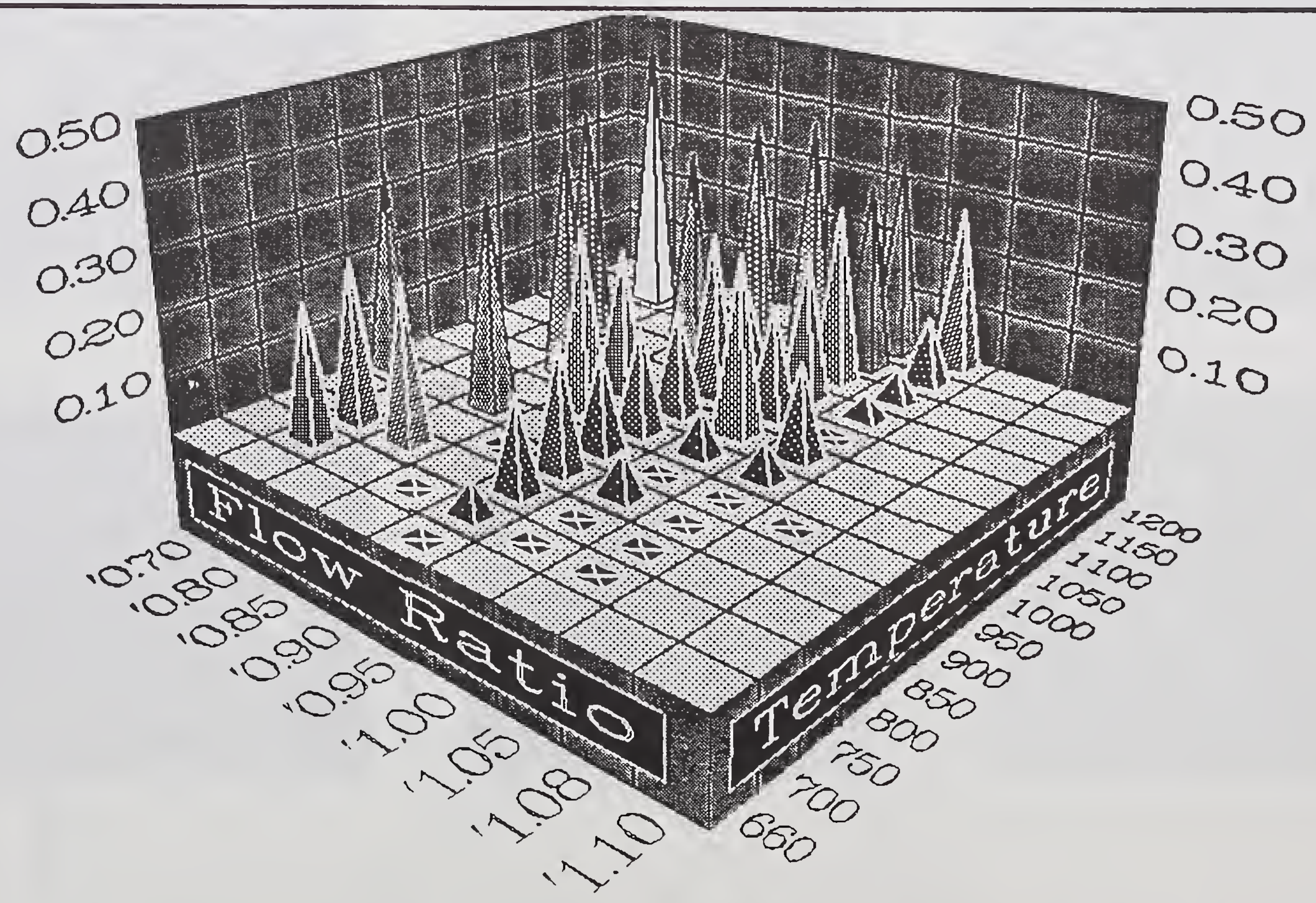
SEM AND MICRO-RAMAN STUDY OF VARIATIONS IN
CRYSTAL QUALITY WITH POSITION ON SUBSTRATE


A
23.1μm
B
23.1μm
C
23.1μm
D
23.1μm
E
23.1μm
F
23.1μm


A
B
C
D
E
F
COUNTS (Arbitrary units)
RAMAN SHIFT (cm^{-1})
1100
1300
1500
1700

RAMAN PHASE DIAGRAM

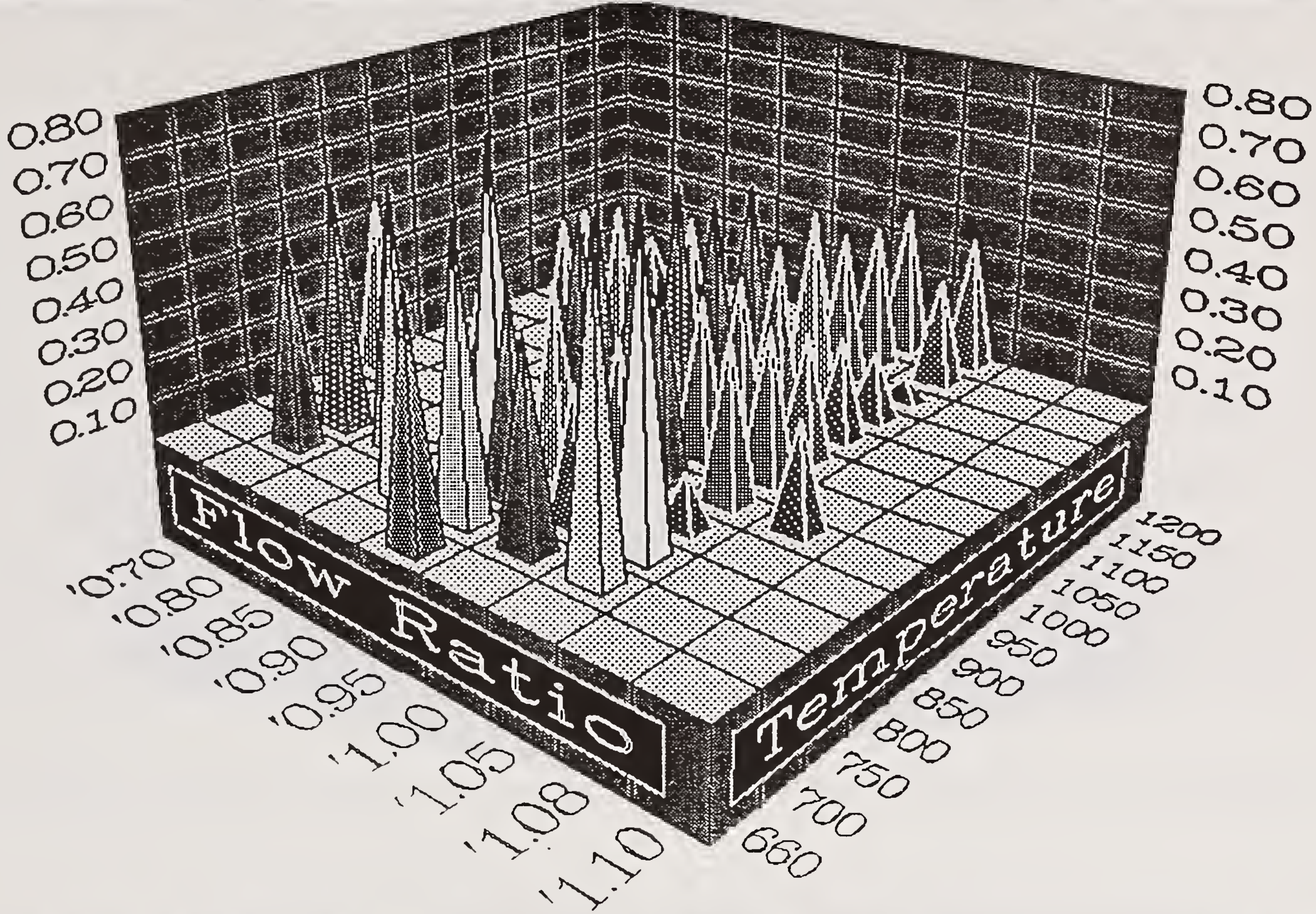
(O_2+C_2H_2, total flow = 3.0 slm, center of substrate)



MICRO–XTAL GRAPHITE (1350 CM–1)

RAMAN PHASE DIAGRAM

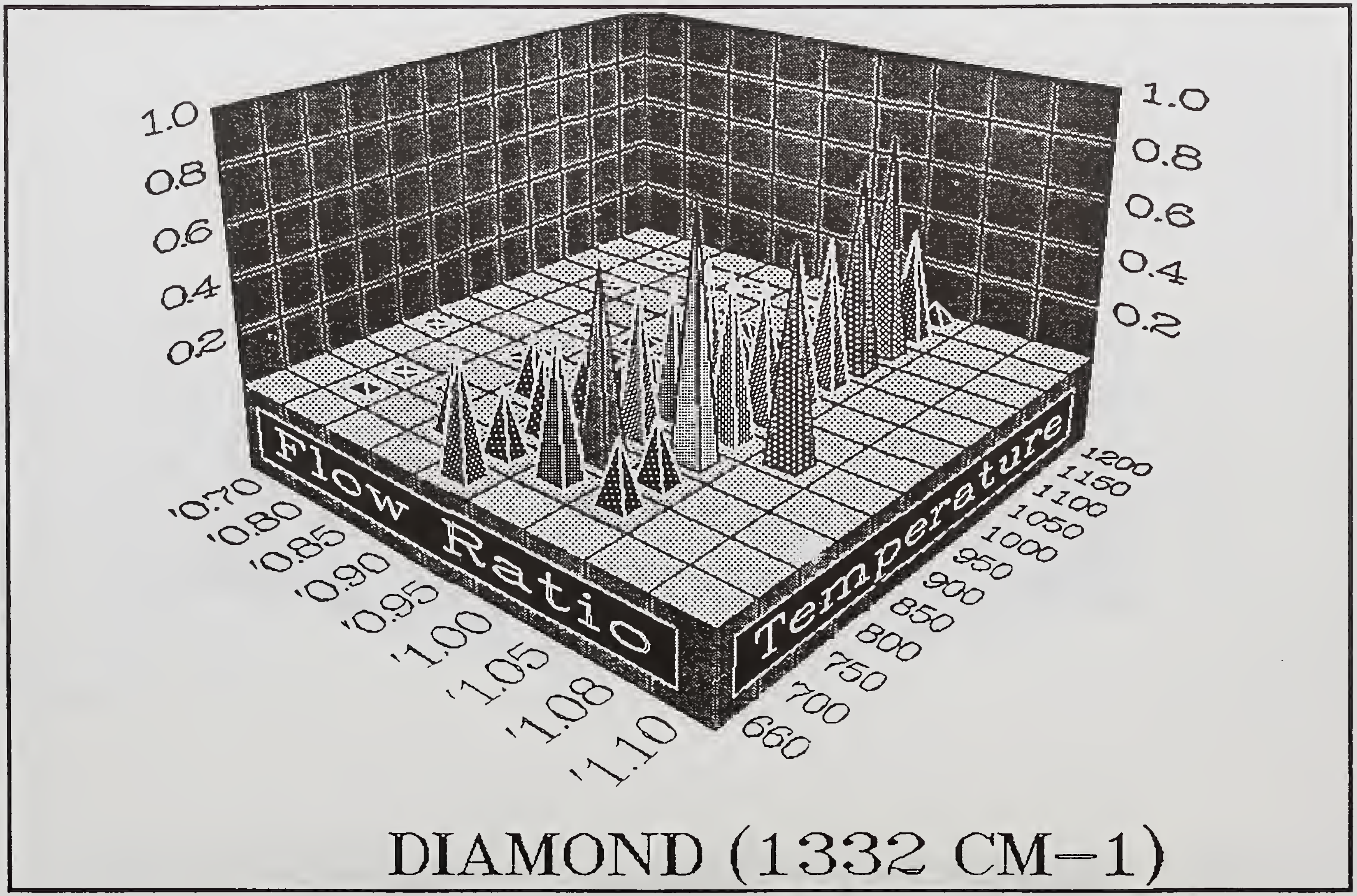
(O_2+C_2H_2, total flow = 3.0 slm, center of substrate)



AMORPHOUS–CARBON (1510 CM–1)

RAMAN PHASE DIAGRAM

(O_2+C_2H_2, total flow = 3.0 slm, center of substrate)



WHY ARE THE RAMAN PHASE DIAGRAMS AND MORPHOLOGY MAPS THE WAY THEY ARE?

- VARIATIONS WITH TEMPERATURE
  - Desorption of hydrogen (reconstruction of diamond surfaces at high temperatures)
  - Rate constants for surface reactions
  - Stability of graphite vs. diamond nuclei
  - Mobility of adsorbed species (defects @ low temps??)

- VARIATIONS WITH OXYGEN/ACETYLENE RATIO
  - Lack of growth species (no growth region)
  - Excess of growth species wrt H level (ball region)
  - Changes in flame temperature

POSTER SUMMARY

- DIAMOND DEPOSITION WITH OXY-ACETYLENE TORCH IS APPROPRIATE TOPIC FOR COLLEGE MATLS. SCI. LABS

- SIMPLE, LOW COST SETUP + HIGH GROWTH RATES

- MORPHOLOGY, QUALITY & GROWTH RATE VARY WITH:
  - SUBSTRATE TEMPERATURE
  - OXYGEN-ACETYLENE FLOW RATIO
  - POSITION IN FLAME & ON SUBSTRATE
  - SUBSTRATE MATERIAL, FINISH

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Simple Strategies for Improving Retention of Women In Technical Studies

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The United States is facing a serious shortage of engineers and decreasing numbers of young people interested in pursuing technical careers. To alleviate this situation, engineering students must be recruited from populations not traditionally well represented. Women are one such group.

Despite the best intentions and determined efforts made by engineering schools over the last ten years, women still account for only 7% of the American engineering workforce. [1] This paper addresses two facets of this situation. One aspect is related to the cultural attitudes that are responsible for the problem. The second is a qualitative description of painless techniques that can be implemented by engineering faculty to provide moral support for the women who do make it to college in an engineering major.

I. The Roots of the Problem

We are all products of our upbringing and under scrutiny will find that much of our interpretation of the behavior of others is based on the premise that their motives are the same as ours. Often, this not the case. We of the Caucasian majority in the U.S. tend to think that culture is something only minorities have. Try standing less than 28 inches from a typical American and you are aware that something is wrong; a manifestation of your enculturation. For a WASP, a person who encroaches on your personal space is sending a message, with sexual and/or sinister overtones. More likely, the person is hard of hearing or comes from a subculture where twenty inches or less is the appropriate distance between people in conversation.

We have definite cultural values that condition us to expect certain patterns of behavior that are often gender-specific. I would like to mention a few of these, on the grounds that being aware of them will in itself be helpful in understanding why so few women become engineers.

In years past, it has been a popular conception that women are not as good at math and science as men. When men study young males and females to ascertain whether there is a measureable difference in mathematical ability, the conclusion is usually that there is. [2] When women conduct the study, no difference can be documented. [3] This prejudice still influences many, properly enculturated individuals, both men and women. Math phobia among young females is a recognized problem; a manifestation of a self-fulfilling prophesy.

As small children, males and females in our culture are usually trained to express anger in different fashions. If a three or four year old child lets go with a stream of obsenities, the response of the adults in the immediate proximity will depend to some extent on what gender the child is believed to possess. Since, at that age, gender is mostly determined by apparel or name, the perception of gender is no more than exactly that. A girl child who indulges in such expressions of anger or hurt will be considered the unfortunate product of poor parenting, probably a profane mother. A boy child might be remonstrated, depending on the relationship of those within hearing distance, but the behavior will be considered much less shameful, perhaps even boyish. The girl can cry with complete immunity but the boy is likely to be told that men do not cry and keep a stiff upper lip, etc. Which behavior is going to be considered acceptable in young engineering students running up against a frustrating problem or bad grade? After years of training to be typical females, young women suddenly find their cultural conditioning is crippling them. Men are not sure how to work with a person who expresses anger or frustration by crying.

Similarly, most children are taught not to interrupt other people when they are speaking. However, consideration for others and nurturant behavior is part of what our culture considers especially proper female training. Boys are taught to be polite also but are treated much less severely when they are not. Some recent studies [4] showed men start more than 90% of their conversations by interrupting whoever has the floor. Women seldom begin a conversation by interruption. They wait for a break to occur which means in some cases they can wait a long time. Of course there are exceptions to this cultural pattern; men who find it difficult if not impossible to interrupt a speaker and women who have no problem initiating a statement in the middle of another's discourse.

I was present at a meeting of about ten faculty discussing the selection of a new chair not long ago and was able to observe this phenomenon in action. In the first twenty minutes of the meeting, eight people spoke, not one began his comments at a period. The two of us who did not feel comfortable with that procedure waited hopefully for order to prevail. (I was the only woman; the other nonspeaker was a male.) Reluctance to begin speaking by interruption does not mean that a person has less to say, which is the conclusion drawn by typically enculturated males. In some cases, probably a direct result of child rearing practice, the cultural prohibition on rudeness has been engrained more thoroughly.

Some years ago, one of the more perceptive psychologists studying our culture presented his observations on the natural proclivity of preadolescent girls to plan the interior of rooms and boys to build rockets and tall buildings, when encouraged to design an environment with a collection of props. [5] From these observations, he concluded that the presence of the womb in even young girls led them to an interest in the interior of domiciles while the young boys were taken with phallic images. This study was widely accepted and considered a good example of how men and women are naturally different. I was rearing young girls at the time and was appalled at the lack of sensitivity the author showed to the extent of enculturation that girls had been subjected to by the time they were preadolescent. When asked why this was the case, the daughter of this eminent phychologist said he was too busy developing his theories to notice how she was subjected to cultural values. Girls are "nice" when they are quiet, clean, helpful; boys are "all boy" when they are active, loud, dirty, investigating everything. (Which set of character traits do you suppose will lead a young person to be interested in engineering?)

The above examples of how our culture influences our response to the gender of a person have been cited only to heighten awareness on the part of engineering educators (who are predominatly male). I am not trying to make males feel guilty because they are a product of their upbringing. A more appropriate goal is to help formulate patterns of behavior that will make the four years (minimum) a young woman spends in the classroom more pleasant, so the word will spread to younger students that engineering is OK.

II. Cultural Remediation in the Engineering Classroom

Some techniques suggest themselves, based on the examples above. Allow uninterrupted speaking time to both men and women. You will never know what good ideas some people have if they have to interrupt or bear interruption to share them. It can be argued that these people should just learn to function like "real men" but there is a fair amount of evidence accumulating that competition for the floor does not result in very good communication in the best of times. [6] Tentative broaching of a solution or question is more common from females than males and must be handled carefully to avoid squelching participation. Listening for the idea rather than dismissing the contribution because of overly polite phrasing or questioning intonation is not easy for a typical, properly brought-up male of my generation. The immediate impulse is to interrupt and rephrase, regardless of the validity of the response. [6] Women are taught to be indirect in their approach by our culture. Ask a question that brings up your point rather than make the point bluntly; good psychology for smart females but poor practice in a male-dominated classroom. A professor might respond with a tentative expression or ask supporting questions when faced with encouraging a non-assertive student. Students of both sexes have been shown to participate significantly more often in classes taught by women, who are more accepting of the tentative "female" approach. [6]

EXAMPLE: The instructor asks: "What determines thermal shock resistance in a ceramic?" (Looking for thermal expansion, thermal conductivity and tensile strength) A tentative answer from a student: "The thermal gradient across the sample is important?" (Misinterpreted question; missed "property" and is not thinking in the same path as the professor) Positive response by professor: "Yes, that will certainly affect the likelihood of fracture due to thermal shock. Could we divide the important properties in environmental and material related? Thermal gradient is the most important environmental factor. What would be the most important material related factors? The properties of the material." Note the use of some of the same words the student used; that is great for the self concept of the student. So is reference to the student by name. "As Joan mentioned, thermal gradient must always be considered when evaluating the likelihood of thermal shock cracking." The professor has in this reply interpreted a response as correct in the manner the student intended it, encouraged that student and others to ask questions, helped the class make an important distinction, and steered them in the direction s/he wanted to go.

Professors should not be afraid to call on women for fear of embarrassing them, even if there are only two or three in the class. Female students should be treated as individuals and expected to respond in the same manner as the man, keeping in mind that many women (and some men) will not respond well to being interrupted and may be "unbecomingly tentative" (cultural interpretation) in their responses. Encouraging the classroom attitude that every student is worthwhile and probably has good ideas will result in more learning and will make females more comfortable.

Be sensitive to your proxemic behavior. Male professors tend to aviod eye contact with female students. [6] The message this gives the women is that they probably do not have anything to contribute, because our culture encourages eye contact during serious conversation. If you would not put your arm around a male student who was talking to you as you walked down the hall, don't do it to a female. Many of us automatically respond differently to males as opposed to females; it is part of our enculturation and we must be very sensitive to avoid creating the wrong impression.

EXAMPLE: Talking about how different people act often relaxes tension and allows for open discussion of what can be very disturbing behavior patterns. I recently taught a lab class to senior engineers with two teaching assistants who represented both extremes in the need for personal space. One was Latin and always had his body next to whomever he was talking to, touched everyone and generally could have been perceived as a real menace by both genders. The other T.A. was a typical American, 28 to 30 inches and never touch anyone. The first semester I worked with these two gentlemen I got many complaints about the Latin T.A. from student evaluations. Knowing the person well, I was quite confident that his intentions were honorable and friendly. The second semester I introduced them with comments about the spectrum represented by the three of us. I was somewhere in the middle, often touching when I should not have. I told the students that the Latin couldn't help himself. After 25 years of being trained by his culture, he wasn't going to change and they should just kick him right above the ankle if he bothered them by his proxemic behavior. I was a hopeless case also, having been a mother for almost 25 years (a difficult disease from which most people never fully recover) and they should likewise make it clear to me that I was bothering them. They all laughed at this and with subsequent encouragement, were able to joke about proxemic behavior during the semester. The important point is to make discussion easy because a strained classroom is not an easy place to learn. No one complained about the Latin T.A. the second semester.

Another culturally initiated difference that can be addressed in the classroom with perfect honesty is how men and women respond to failure. Men tend to blame the textbook, the professor, time spent on sports, and similar external influences. Women almost always blame themselves. They think they are not smart enough or something serious is wrong with their background. [7] I have found joking about this when someone (male) makes excuses a good way to bring it up and a source of tremendous interest to the women. It is easier to address directly in a meeting of female undergraduates, but that format is often unavailable. Knowing this reaction is common from females as opposed to males often allows the women to build defenses against self-doubt, since most modern young women are not willing to allow their success to be hindered by gender-specific behavior.

To support this point, a recent study at the University of Illinois revealed that after two years of college, women who were top high school students have significantly lower self-images than do their male counterparts, and that their perceptions had no correlation to the students' actual academic performance. The study also revealed that, by their sophomore year in college, women had lowered their career aspirations more than men had. [7]

Well enculturated women tend to feel that they are obligated to nurture the world. This conditioning has been very helpful to our society over the years but results in women seeking service professions rather than what are perceived as materialistic endeavors. A recent Office of Technology Assessment study reported that young people in general would be more attracted to the field if engineering were perceived as directly serving people and the public interest. [8] The idealism of the young (may it never fail!) leads to questions like: What is engineering? What does engineering contribute to humanity? What does it stand for? A big picture description of engineers as problem solvers - people who enjoy trying to develop new and better ways of doing things - is often helpful in addressing these concerns, which are expressed by both genders.

Since women usually picture themselves as being nurturant they may respond more actively to this picture of engineering as a profession that contributes significantly to the well being of humanity. When asked what an engineer does, most of us tend to focus on our own branch and start outlining the various specializations when the question is generally prompted by more global concerns.

EXAMPLE: Engineers work at solving problems such as environmental pollution. Ceramic engineers are trying to make a ceramic engine that will run at higher temperatures to get more energy from a gallon of gasoline. Automotive engineers are trying to design a more efficient engine. Petroleum engineers are trying to find better ways of extracting gasoline from oil. Chemical engineers are trying to develop alternative fuels that are less polluting.

Be sensitive to the fact that the male gender dominates in all textbooks, articles, examples and problems in engineering. When you make up problems, include reference to females.

EXAMPLE: When the engineer evaluated the glass batch as specified by the customer, SHE discovered the weight percents did not add up to 100.

III. Summary

The number of women who begin engineering studies and the number who finish and go on to work in the field are influenced by cultural attitudes; their own, their peers' and their professors'. I believe we can, as engineering faculty, have a significant, positive impact on the percentage of women actually working in the field by being more sensitive to our gender-specific behavior, as described above. In addition, the following general approaches are recommended. [6]

1. Pay particular attention to classroom interaction patterns during the first few weeks of class and make a special effort to draw women into discussion during that time. The pattern that is estalished early in the term will likely continue.

2. Notice whether the "feminine" or "masculine" style of a student's comments, question or response affects your own perception of its importance.
3. Intervene in communication patterns among students that may shut out women.
4. Note patterns of interruption to determine if women students are interrupted more than men, by yourself or by other students.
5. Ask women and men qualitatively similar questions; critical and factual.
6. Avoid using the generic "he" whenever possible.
7. Watch for and respond to nonverbal cues that indicate women students' readiness to participate in class.
8. Use the same tone of voice when talking to women as with men.
9. Ensure that women are not squeezed out by male classmates from viewing a laboratory demonstration or engaging in other group activities.
10. Assume an attentive posture when responding to women's questions or listening to their comments.
11. Ask a colleague to visit your classroom and observe; watching for inadvertent gender-specific behavior on your part.

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Proceedings of a workshop sponsored jointly by NIST and the Schools of Technology and Science, Norfolk State University, Norfolk, Virginia, and held in Gaithersburg, Maryland, November 13-15, 1990. The workshop theme was strengthening materials education. Material in this publication can serve as a valuable guide to faculty who are interested in useful experiments for their students. There was a blend of experiments on new materials and traditional materials. Uses of computers in MS&E, experimental design and an approach to systematic materials are among the topics presented.

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