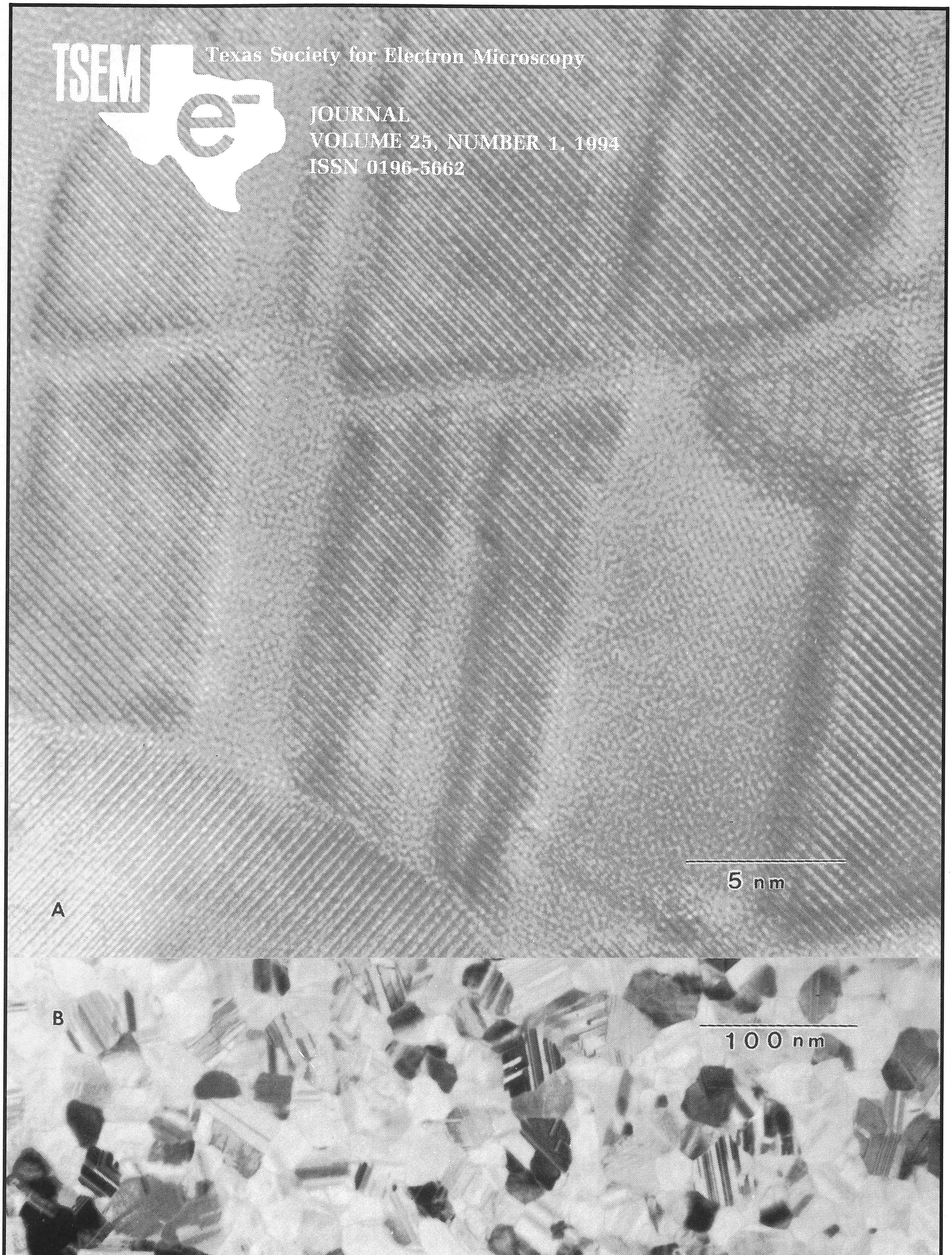
TSEM
Texas Society for Electron Microscopy

JOURNAL
VOLUME 25, NUMBER 1, 1994
ISSN 0196-5662



TSEM OFFICERS
1994-1995

Contents

TEXAS SOCIETY FOR ELECTRON MICROSCOPY JOURNAL
VOLUME 25, NUMBER 1, 1994
ISSN 0196-5662

David C. Garrett, Editor
Department of Biological Sciences, University of North Texas, Denton, TX 76203

Texas Society for Electron Microscopy
"For the purpose of dissemination of research with the electron microscope."

ON THE COVER
Electron Microscopy of $BaTiO_3$ deposited on a Si(100) substrate using pulsed laser ablation in vacuum at 750°C. Micrograph B shows the polycrystalline structure of $BaTiO_3$. Micrograph A shows the internal grain structure and may represent nanometer-size ferroelectric domains or twinning. Photo — Young Gyu Rho, Department of Physics/CMC, University of North Texas, Denton, TX. Sample supplied by S. R. Summerfelt, Texas Instruments, Dallas, TX.

WANTED

PREFERABLY ALIVE

Manuscripts

Techniques

Commentaries

for the

TSEM JOURNAL

REWARD!

The satisfaction of knowing you have
helped support the
Texas Society for Electron Microscopy

President's Message

1994 is fulfilling its promise as a period of continuing growth for the Texas Society of Electron Microscopy and for yours truly. The meeting in Galveston was quite successful. It was very enjoyable for me to welcome many of you to the island, and the Tremont Hotel staff did their usual superb job in providing arrangements for the group. We are currently working on setting up the technique of in situ hybridization using the methods so kindly described to us by Dr. Gwen Childs.

The spring meeting in Denton is upon us; it seems incredible that so much time has passed since the fall meeting. Time really does fly when you have fun and stay busy. In spite of the late call for papers, for which I must take most of the responsibility, the membership has responded admirably by submitting a number of abstracts for the meeting, and I look forward to welcoming many of you to Denton. As is often the case at national MSA meetings, this should be an opportunity for those of us who work on biological tissues to have our horizons expanded by hearing how our friends in the materials sciences use powerful techniques to characterize other kinds of specimens. Dr. Russell Pinnizzotto will be hosting the meeting and providing a workshop on high-resolution transmission electron microscopy to be held, in a welcome break with tradition, on the campus of the University of North Texas. We also have an outstanding speaker from Baylor College of Medicine, Dr. Kimon Angelides, who will discuss with us the newest methods for computer enhancements and analysis of images. His work is of great interest to electron microscopists (and light microscopists) working in both the biological and physical sciences. Paula Williamson, our program chair, is responsible for organizing the Galveston and Denton meetings and deserves our warmest thanks. Mitch McCartney is already hard at work preparing the fall 1994 and spring 1995 meetings. We will be visiting with Nancy Smith in San Antonio in the fall, and favorable arrangements have already been made with a hotel there.

A momentous event took place last fall, too late to be noted in the *Journal*. At long last, TSEM has been awarded tax exempt status by the federal government. Wayne Sampson deserves enormous credit for his many hours of hard work on this project, and I will be joining you in expressing my gratitude for his efforts and his success.

As I look back over my term in office and look forward to becoming an ex-President, I wish I could have accomplished more but feel a great sense of gratitude for the experience. It has always been rewarding and highly pleasurable to work with the members of this society. I recommend active participation to anyone. It's a really enjoyable and productive way to spend some time. I hope you enjoy the meeting in Denton!

Sincerely,

Hal K. Hawkins
President, 1993-1994

Treasurer's Report

TEXAS SOCIETY FOR ELECTRON MICROSCOPY
TREASURER'S REPORT
For Period Ending December 31, 1993

ASSETS ON JANUARY 1, 1993:		
Certificate of Deposit No. 113515	$3,651.24	
Certificate of Deposit No. 2414483036	1,688.30	
Certificate of Deposit No. 9005997	4,109,97	
Checking Account No. 44059412	1,197.43	
TOTAL		**$10,646.94**
CHECKING ACCOUNT RECEIPTS:		
Dues	$3,243.00	
Spring 1993 Meeting Registration	965.00	
Workshop	45.00	
Exhibitors	850.00	
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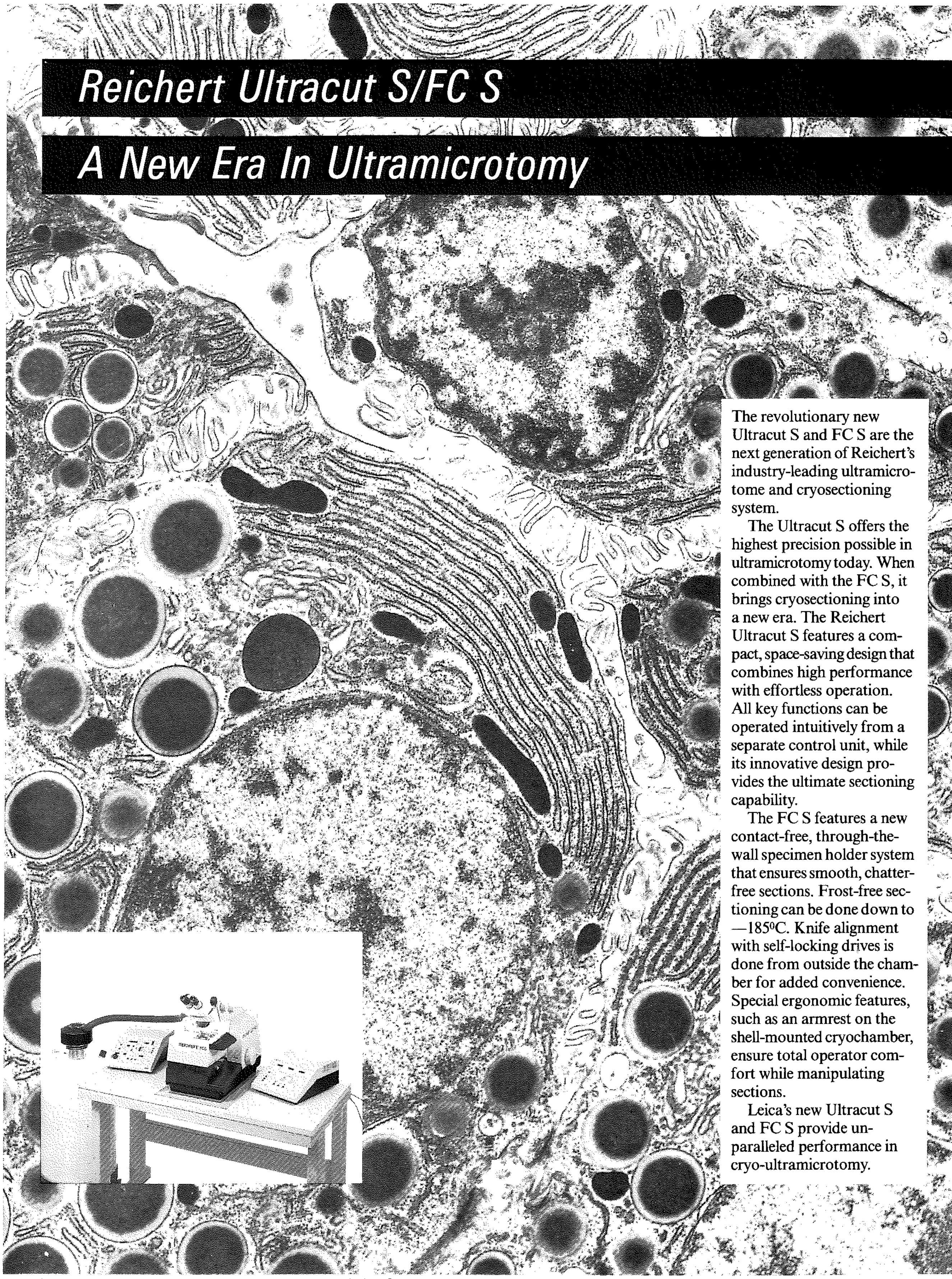
Guest	120.00	
Fall 1993 Meeting Registration	1,380.00	
Workshop	1,050.00	
Exhibitors	1,245.00	
Donations and Grants	450.00	
Guest	250.00	
Journal Advertisements 23:2	260.00	
24:1	2,625.00	
24:2	2,500.00	
Miscellaneous	178.82	
Checking Account Interest (Account No. 70072962)	92.39	
Certificate of Deposit Interest (No. 9005997 and No. 2414483036)	80.70	
TOTAL		**$15,508.66**
C.D. No. 9005997 to Checking Account No. 70072962	4,109.97	
C.D. No. 2414483036 to Checking Account No. 70072962	1,688.30	
Certificate of Deposit Interest (No. 113515)	239.28	
EXPENSES:		
Journal, 24:1	$2,240.31	
24:2	2,378.96	
Office Expenses	152.69	
Mailouts	585.55	
Spring 1993 Meeting	2,205.84	
Workshop Refund	45.00	
Student Competition/Travel	385.00	
Miscellaneous	74.40	
Invited Speaker	420.07	
Fall 1993 Meeting	3,447.85	
Workshop	232.00	
Student Competition/Travel	395.00	
Invited Speaker	624.02	
Workshop Refund	87.00	
Legal Fees	5,457.82	
Checking Account Service Charge (Account No. 44059412)	52.10	
Miscellaneous	74.40	
TOTAL		**$18,783.61**
ASSETS AS OF DECEMBER 31, 1993:		
Certificate of Deposit No. 113515	$3,890.52	
Checking Account No. 70072962	3,720.75	
TOTAL		**$7,611.27**



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Signature of nominating MSA Member: ______________________

Signature of advisor *(for student applicants)*: ______________________

Signature of applicant: ______________________ Date: ______________________

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- ☐ Appalachian Regional Electron Microscope Society
- ☐ Arizona Society for Electron Microscopy and Microbeam Analysis
- ☐ Central States Electron Microscopy Society
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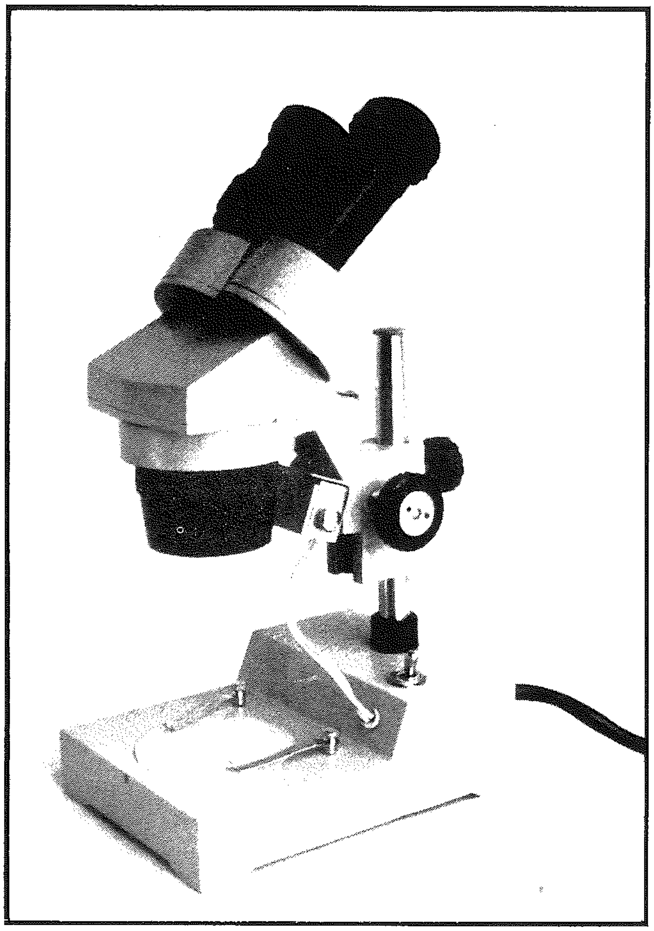

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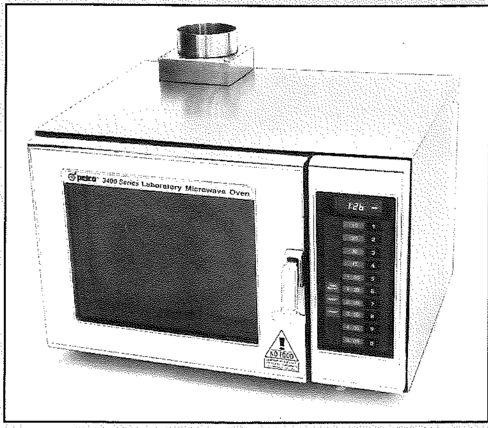

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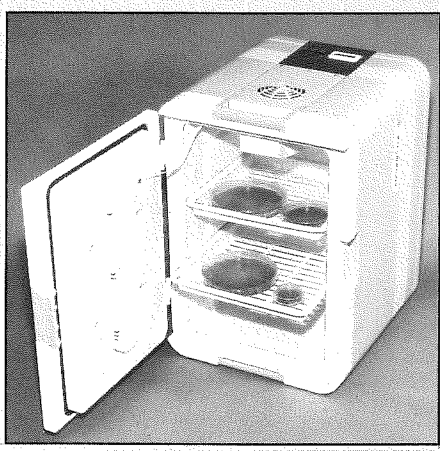

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CALENDAR OF MEETINGS

FALL MEETING OF TSEM

October 20-22, 1994
San Antonio, Texas

See Details Elsewhere In This Journal

FOOD STRUCTURE ANNUAL MEETING

May 7-12, 1994
Toronto, Canada

Contact: Dr. Om Johari (708) 529-6677

SCANNING '94 (FAMS & SEEMS)

May 17-20, 1994
Charleston, South Carolina

Contact: Mary K. Sullivan (201) 818-1010

13th INTERNATIONAL CONGRESS ON ELECTRON MICROSCOPY

July 17-22, 1994
Paris, France

Contact: Secretariat ICEM, 13,67 rue Maurice Gunsbourgh, 94205 Ivry sur Siene cedex, France

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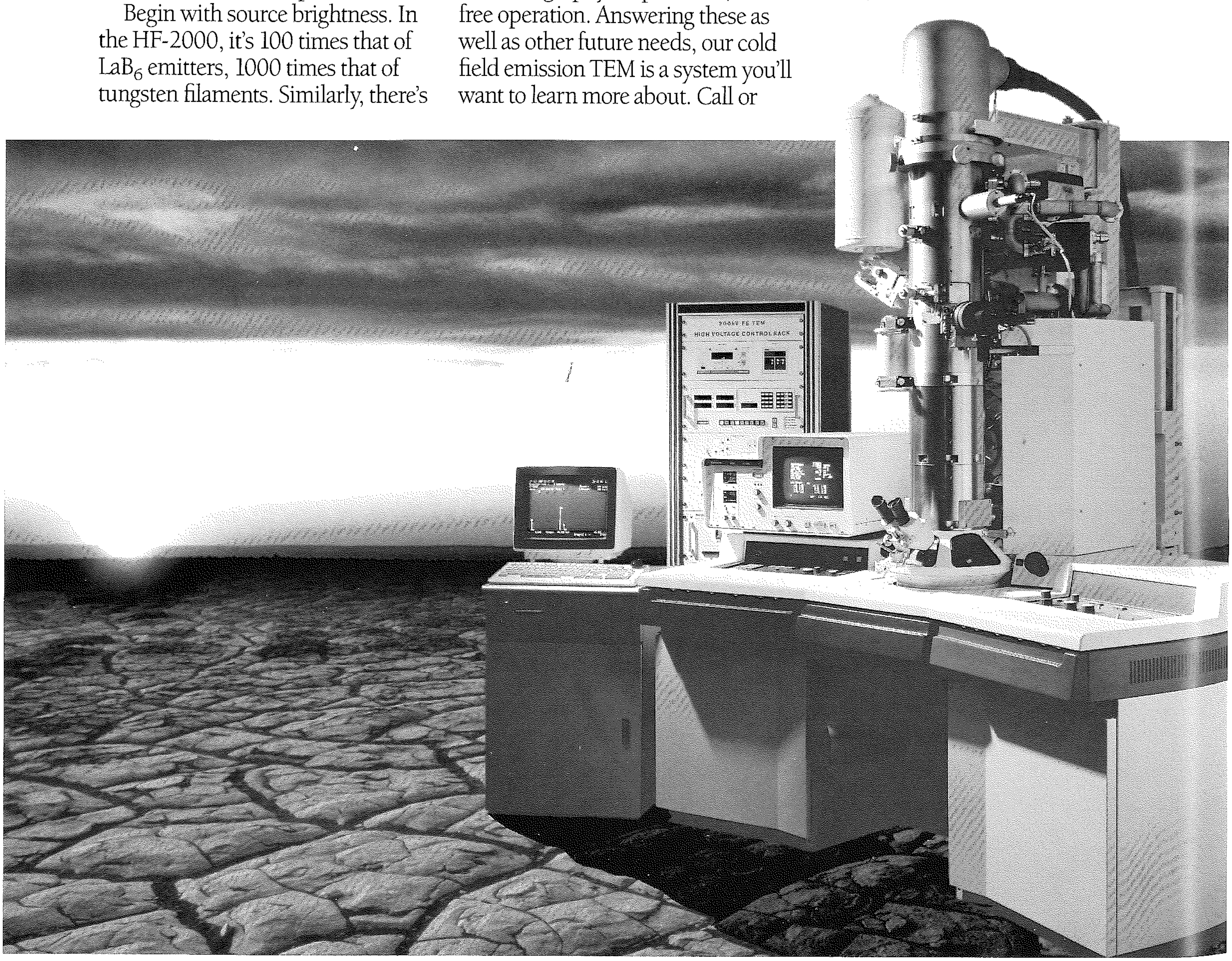

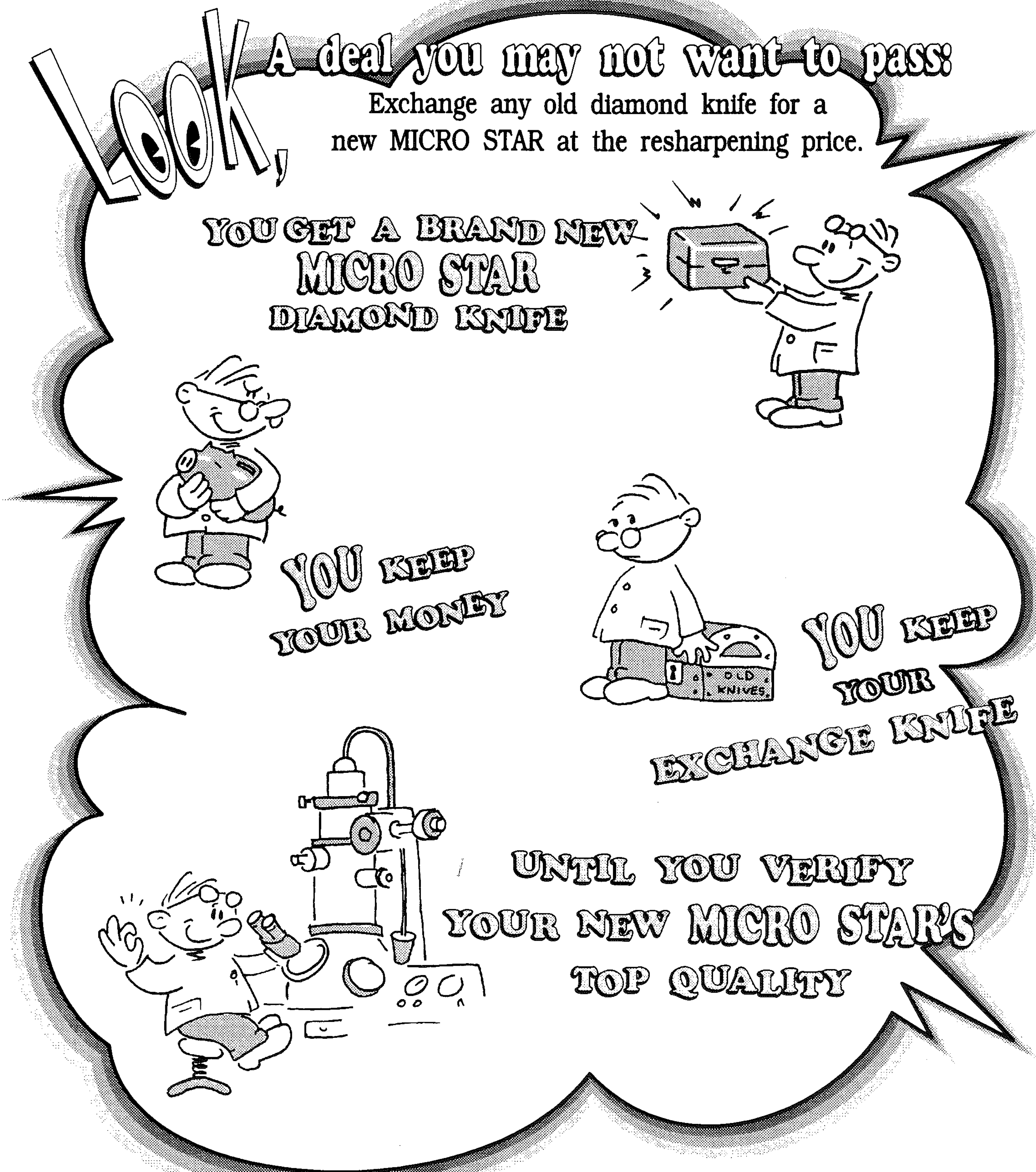
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ARTICLE — STUDENT COMPETITION WINNER

DIFFUSION BEHAVIOR OF TIN IN GOLD COMPOSITE SOLDER

Yujing Wu
Center for Materials Characterization
University of North Texas, Denton, TX 76203-5308

Sn/Pb solder alloys are used by the electronics industry to provide electrical and mechanical interconnections between components and substrate materials. The formation and growth of the intermetallic compounds Cu_6Sn_5 and Cu_3Sn at the solder/copper substrate interface, both during soldering operation and system use,[1] have been proposed as controlling mechanisms for the solderability and reliability of electronic solder joints.[2] In applications such as surface mount technology (SMT), solders with enhanced mechanical properties are required for high reliability. One approach is to add metallic or intermetallic particles to eutectic 63Sn/37Pb solder to form composite solder. The goal of this research is to study the influence of Au particle additions to the Sn/Pb eutectic solder matrix on the diffusion behavior of Sn, and on the growth kinetics of intermetallics at the solder/copper interface.

Intermetallic formation and growth at the solder/copper interface were studied for 4 wt% Au composite solder as a function of time and temperature, and compared to the behavior of eutectic solder alone. By using SEM, TSEM, STEM and XEDS, it was found that the addition of Au particles to eutectic Sn/Pb solder strongly affects the diffusion behavior of Sn, and therefore affects the kinetics of imtermetallic formation and growth. The activation energies for the formation of Cu_6Sn_5 and Cu_3Sn at the Au composite solder/copper interface are 0.65 and 0.85 eV, respectively, which are smaller than the values of 0.84 and 1.63 eV for the eutectic solder alone. The diffusion data in the literature were examined and it was found that Au rapidly diffuses by an interstitial diffusion mechanism in the Au-Sn system. During soldering, all Au particles in the solder matrix react completely with Sn to form $AuSn_4$

FIGURES:

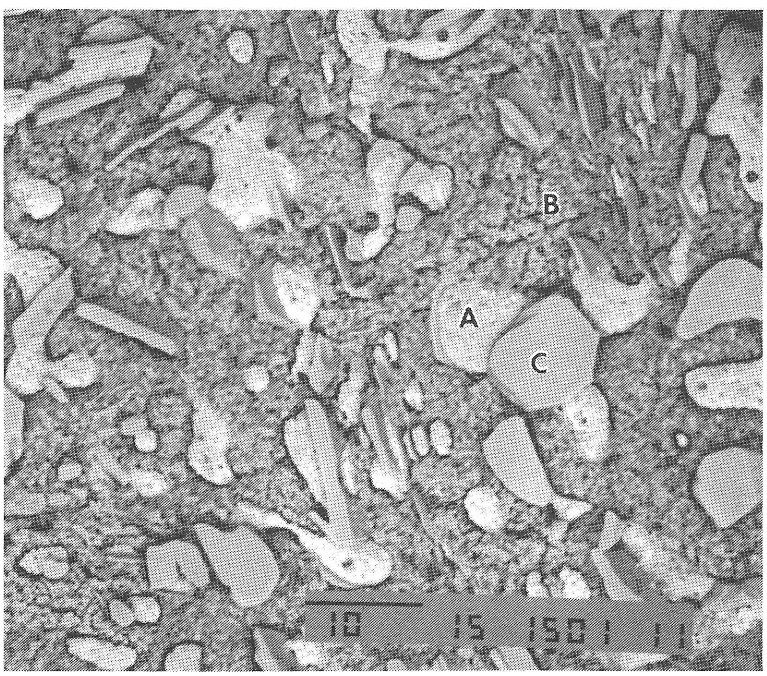

Figure 1

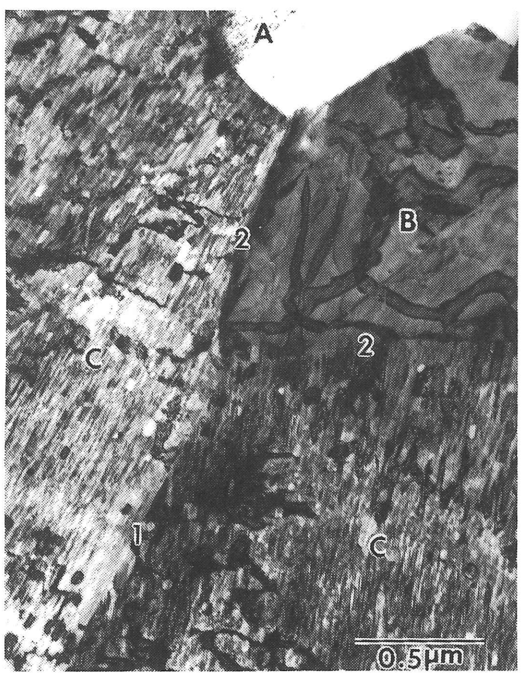

Figure 2

FIGURE CAPTIONS:

Figure 1 — SEM micrograph of 4 wt% Au composite solder matrix after 4 days at 140°C: (A) Pb-rich phase, (B) Sn-rich phase and (C) $AuSn_4$ intermetallic. The magnification line indicates 10 μm.

Figure 2 — TEM micrograph of 4 wt% Au composite solder matrix after 4 days at 140°C. Two $AuSn_4$ particles and the solder phases are clearly visible. (A) Pb-rich phase, (B) Sn-rich phase, (C) $AuSn_4$ intermetallic, (1) $AuSn_4$/$AuSn_4$ grain boundary and (2) $AuSn_4$/Sn phase boundary.

Article *continued from Page 17*

intermetallic, thus reducing the amount of Sn that is able to reach the solder/copper interface. The initial thicknesses of Cu-Sn intermetallics after the soldering operation are thinner than for the eutectic solder alone. As shown in Figure 1, after soldering with $AuSn_4$ particles are distributed uniformly in the solder matrix. Figure 2 is a TEM micrograph which shows the Au composite solder matrix after annealing at 140°C for 96 hours. There are two $AuSn_4$ particles in contact with each other. Both $AuSn_4$ grains contact the Sn-rich phase of the solder. All these phases were identified using selected area electron diffraction patterns. The $AuSn_4/AuSn_4$ grain boundary (labeled as 1) and $AuSn_4/Sn$ phase boundary (labeled as 2) are clearly visible. XEDS in STEM mode was used to determine the Sn/Au ratios along the $AuSn_4/AuSn_4$ grain boundary and $AuSn_4/Sn$ phase boundary, compared to the Sn/Au ratios within the $AuSn_4$ phase (measured 150 Å away from the boundary toward the center of the $AuSn_4$ grain). The average XEDS peak intensity ratio of Sn to Au at the boundaries is 2.3, much larger than the average value of 1.1 inside the $AuSn_4$ grains. This means that the $AuSn_4$ grain boundaries and the $AuSn_4/Sn$ phase boundaries may act as enhanced diffusion pathways for Sn. Since the Sn can diffuse easily through these boundaries after soldering, the Sn supply to the solder/copper interface is greater than for eutectic solder alone. The Sn moves via normal bulk diffusion of the Sn in the areas without $AuSn_4$, and the much faster boundary diffusion through the $AuSn_4/AuSn_4$ grain boundaries and $AuSn_4/Sn$ phase boundaries. This enhanced Sn diffusion reduces the activation energies for the formation of both Cu_6Sn_5 and Cu_3Sn at the Au composite solder/copper interface compared to the eutectic solder alone. The diffusion of Sn in 20 wt% Cu_6Sn_5 and 10 wt% Cu_6Sn_5 composite solders was also examined. It was found that the thicknesses of Cu_6Sn_5 and Cu_3Sn at the 20 wt% Cu_6Sn_5 composite solder/copper interface after annealing at 150°C for 4, 8, 16 and 32 days are thicker than the corresponding thicknesses for 10 wt% Cu_6Sn_5 composite solder. Therefore the diffusion coefficient is larger for 20 wt% Cu_6Sn_5 composite solder than for 10 wt% Cu_6Sn_5 composite solder. Since the Cu_6Sn_5 phase is the terminal phase in the system, the Cu_6Sn_5 phases do not react with Sn. The only difference between these two solders is that there are more Cu_6Sn_5 grains in the 20 wt% Cu_6Sn_5 composite solder and therefore more Cu_6Sn_5 grain boundaries. These boundaries also appear to enhace Sn diffusion.

REFERENCES:

1. D.S. Dunn, T.F. Marinis, W.M. Sherry and C.J. Williams, Mat. Res. Soc. Symp. Proc., **40**, p. 129, (1985).
2. P.E. Davis, M.E. Warwick and S.J. Muckett, Plating and Surface Finishing, **70**, p. 49, (1983).

ADVERTISERS INDEX

EDITORIAL POLICY

LETTERS TO THE EDITOR

Letters to the editor are printed as they are received in the order of their arrival. These letters reflect the opinion of the individual TSEM member and do not necessarily reflect the opinions of the editor or the society. The content of the letters should be concerned with the philosophical or operational aspects of the TSEM, the Journal and its contents, academic or national policies as they apply to TSEM and/or its members and electron microscopy in general. Editorial privilege may be evoked to insure that the LETTERS SECTION will neither be used as a political forum nor violate the memberships' trust.

ELECTRON MICROGRAPHS AND COVER PHOTOS

Micrographs submitted for cover photos should be marked as such. The choice of photographs will be made by the editor. Photograph receipt and/or dispensation will not be acknowledged. Photographs will not be returned. Electron micrographs to be used for cover photos and text fillers are welcome and should be selected with some attention to aesthetic appeal as well as excellence both in technique and in scientific information content.

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The JOB OPPORTUNITIES section will be comprised of a "Jobs Available" and a "Jobs Wanted" sub-section. Anonymity of individuals listing in the Jobs Wanted or Jobs Available sub-sections may be maintained by correspondence routed through the News Editor's office.

TECHNICAL SECTION

The Technical Section will publish TECHNIQUES PAPERS, and HELPFUL HINTS. The TECHNICAL PAPERS will describe new or improved methods for existing techniques and give examples of the results obtained with methods. The format of the Technique Papers will be the same as that used for regular research reports. HELPFUL HINTS will be in the form of a brief report with an accompanying illustration, if required for clarity. Helpful Hints should embody techniques which will improve or expedite processes and/or procedures used in EM.

PUBLICATION PRIVILEGES

The right to publish in the TSEMJ is restricted to TSEM members or to those whose membership is pending. A membership application form can usually be found in each issue of the TSEMJ. Membership dues are as follows: student $2.00; regular members $15.00; Corporate members $75.00. Individuals who belong to TSEM by virtue of a corporate membership are invited to participate in Journal submissions as are our regular or student members. However, papers of a commercial nature, either stated or implied, will not be accepted for publication as a Research Report or Techniques Paper. Such papers may be acceptable as advertising copy.

TSEM STUDENT COMPETITION

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Competition is open to all student members of T.S.E.M. who are actively seeking a degree at an accredited institution. The term student member will also include those students with a membership application pending. To be eligible to compete, all competition requirements must be fulfilled by the designated deadlines given in the first call for papers preceding the Fall meeting. In addition, to be considered for the top award you must, (1) be a student at the time of the next MSA meeting, (2) apply for a Presidential Student Award, and (3) present your paper at that meeting.

REQUIREMENTS:

You must be the sole author, personally present your paper from the platform, and submit a student competition application signed by a regular T.S.E.M. member, if possible your supervising professor. Two abstracts must be submitted by the designated deadlines; a regular T.S.E.M. abstract following normal procedures submitted to the current *Journal* editor, and an MSA style two page abstract with an application for student travel submitted to the current secretary. Since it is assumed that your professor has supervised your work and others may have contributed in various ways, you must acknowledge these contributions on your application as well as in your platform presentation.

SPECIAL ABSTRACT FORMAT

1. The paper must be two pages each 8½″ by 11″. Margins should be 1″ top and bottom and ¾″ left to right. Text should be 12 characters per inch IBM LETTER GOTHIC or 11 point TIMES ROMAN with 12 point spacing each font at 6 lines per vertical inch.
2. The first page will have text only. Title on first line in all capitals except chemical symbols, single spaced if more than one line is needed. Leave one line of space; then your name and address skipping one line between each. Leave one line blank and start text with no indentions and skip one line between paragraphs. Group all references at the end on the text before illustrations.
3. Page two will include pictures and text. Micrographs should be numbered, have an appropriate scale marker, and be trimmed to form a rectangle with no gaps. Figure captions should follow the micrographs and come last.
4. Examples and additional guidelines may be found by consulting an MSA call for papers.

AWARDS:

Up to 3 awards (0-3) may be given at each Fall meeting. These awards may be cash or prizes as determined by the Executive Council. The top award that can be given is substanial support towards competing in MSA's Presidential Student Award program. This award can only be given if you meet MSA qualifications and compete at the next MSA meeting.

JUDGING:

Judging will be by a panel of regular T.S.E.M. members. You will be judged 50% on the quality of your special abstract and 50% on the quality of your presentation, including how well you answer questions from the audience. The regular abstract you submit for publication in the *Journal* will not be judged. Because of additional demands of disclosure each entrant will be given an additional 5 minutes of podium time.

TSEM STUDENT COMPETITION APPLICATION

Student's Name: ____________________

Mailing Address: ____________________

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Phone: ____________________

University: ____________________

Department: ____________________ Supervising Professor: ____________________

Degree Program: ____________________ Anticipated Date of Degree: ____________________

Title of Paper: ____________________

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Contributions from Others: ____________________

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Do you wish to be considered for travel support to the next MSA meeting? By answering "YES", you agree to meet MSA guidelines pertaining to the Presidential Student Award program. YES ____ NO ____

I certify that the work being reported is my own.

Student's Signature ____________________

I certify that the work being reported is that of the student.

Professor's Signature ____________________

Information for Authors

GENERAL INFORMATION

PURPOSE: The goal of the TSEM Journal is to inform members of the society and the Journal's readers of significant advances in electron microscopy, research, education, and technology. Original articles on any aspect of electron microscopy are invited for publication. Guidelines for submission of articles are given below. The views expressed in the articles, editorials and letters represent the opinions of the author(s) and do not reflect the official policy of the institution with which the author is affiliated or the Texas Society for Electron Microscopy. Acceptance by this Journal of advertisements for products or services does not imply endorsement. Manuscripts and related correspondence should be addressed to David C. Garrett, Editor, TEXAS SOCIETY FOR ELECTRON MICROSCOPY JOURNAL, Department of Biological Sciences, University of North Texas, Denton, Texas 76203-5218

GUIDELINES: Manuscripts written in English will be considered for publication in the form of original articles, historical and current reviews, case reports and descriptions of new and innovative EM techniques. It is understood that the submitted papers will not have been previously published. Accepted manuscripts become property of the TEXAS SOCIETY FOR ELECTRON MICROSCOPY JOURNAL and may not be published elsewhere without written consent of the Editor. The author should retain one complete copy of the manuscript. The JOURNAL is not responsible for manuscripts lost in the mail.

PAGE PROOFS/REPRINTS: The editor will be responsible for proof-reading the type-set article. Reprints may be ordered from the printer.

MANUSCRIPT PREPARATION: Manuscripts should conform with the following guidelines:

FORMAT: Submit an original and two copies of the entire manuscript, typed, double-spaced, on 8½ x 11 white paper, leaving ample margins. Number each page and identify the article by placing, at the top left of the page, a shortened form of the title, followed by the last name of the first author.

TITLE PAGE: Include:

a. Full title of the article
b. Initials and last names of all authors
c. Current positions of each author (department, institution, city)
d. Full name, telephone number and address of the author to whom reprint requests are to be sent.

SECTIONS: The text of each original article and technical report should be divided into four major sections entitled INTRODUCTION; METHODS AND MATERIALS; RESULTS; AND DISCUSSION.

Historical and current reviews and case reports do not need to be divided into the aforementioned sections.

ABSTRACT: Summarize the article in no more than 150 words. This takes the place of a final summary paragraph.

REFERENCES to other work should be consecutively numbered in the text using parentheses and listed at the end, as in the following examples:

(1) A. Glauert, Practical Methods in Electron Microscopy. Vol. 2 (North-Holland. Amsterdam, 1974) 82-88.
(2) P.S. Baur, Jr., G.F. Barratt, G.M. Brown and D.H. Parks. Ultrastructural Evidence for the Presence of "Fibroclasts" and "myofibroclasts" in Wound Healing Tissues. J. of Trauma. 19 (1979) 774-756.
(3) D. Gabor. Information Theory in Electron Microscopy, in: Quantitative Electron Microscopy. Eds. G.F. Bahr and E. Zeitler (Williams and Wilkins, Baltimore, 1956) 63-68.

(NOTE: Authors are responsible for the accuracy of references.)

TABLES:

a. Type double-spaced each table on a separate sheet.
b. Number in order in which they are referred to in the text.

ILLUSTRATIONS:

A. Submit three complete sets of illustrations. Copy machine reproductions of photographs will not be accepted. Indicate which set is the original photograph or illustration.
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NOMENCLATURE AND ABBREVIATIONS: Journal abbreviations used should be those listed by the "Index Medicus." Nomenclature abbreviations should be similarly standardized.

ACKNOWLEDGEMENTS should appear as a footnote which will appear at the top of the first page of the article.

MSA UNDERGRADUATE SCHOLARSHIP PROGRAM

Description and Eligibility Requirements

1. The Microscopy Society of America (MSA) has established a scholarship fund for undergraduate research. The purpose of the scholarship is to further educational and research potential in full time undergraduate students intent on pursuing electron microscopy as a career. Applications in all areas of electron microscopy will be accepted for review.
2. Scholarships will be awarded to full time undergraduate students. Maximum total dollar amount awarded each year will be $10,000.00.
3. When possible at least one scholarship will be awarded to an under-represented minority applicant.
4. Preference will be given to those scholarship proposals which utilize a facility other than the one at which the student is currently enrolled.
5. Applicant must be a full time undergraduate student and a U.S. citizen or resident alien (green card required). Research programs for which scholarship funds are awarded must be carried out in a U.S. laboratory.
6. Awarded funds must be used within a designated time period not to exceed one year from the award date. Students are eligible to receive an award only once.
7. Applications must be received by 15 November of each year to be awarded by 1 March of the following year.
8. Complete application forms should include the following items:
   - **A.** Provided form with requested information.
   - **B.** A research proposal not to exceed 3 pages in length. The proposal should include a brief introduction, a short methods section, and itemized goals of the study.
   - **C.** A budget proposal detailing how awards will be utilized. If the proposed project will exceed the requested amount, a statement should be provided indicating sources of additional funding.
   - **D.** Two letters of reference from academic and/or industrial personnel familiar with the student's competence are required.
   - **E.** A letter from the laboratory supervisor where the proposed research will be performed indicating the applicant will be accepted in the laboratory to work on the proposed project. A laboratory must be designated in the proposal for funds to be awarded.
   - **F.** A cirriculum vitae detailing previous education and/or experience in electron microscopy, and a brief statement of career goals.
9. Complete applications, reference letters, and a letter from the laboratory supervisor where the research will be performed should be sent to:

   Dr. M.G. Burke
   Westinghouse Science & Technology Center
   1310 Beulah Road
   Pittsburgh, PA 15235-5098
10. Following completion of the scholarship period a statement detailing status of the research project should be given to the MSA Council. Publications resulting from the scholarship should acknowledge the award. A reprint should be given to the MSA Education Committee Library when they become available.

MSA UNDERGRADUATE SCHOLARSHIP APPLICATION

1. Name (Last, First, Middle): ______________________________

2. Date of Birth: ____________________ **3.** Social Security Number: ____________________

4. U.S. Citizen: ☐Yes ☐No Resident Visa: ☐Yes ☐No

5. Telephone Number (Area Code, Number, Extension): ______________________________

6. Present Address: Permanent Address:

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7. University Affiliation: ______________________________

8. Academic Status (Year Completed by Spring): ☐Fr. ☐So. ☐Jr. ☐Sr.

9. Title of Proposed Research: ______________________________

10. Site where proposed research will be conducted: ______________________________

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11. Amount of funding requested (Maximum = $2,500): ______________________________
Include a detailed budget with sources of additional support for project on a separate page.

12. Name, title, and addresses of two academic or industrial personnel familar with the student who will provide letters of reference. Applicant is responsible for having all letters sent before the application deadline. Letters must be received before application is considered for funding.

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13. Name, title, and address of laboratory supervisor who will supervise the project and provide a letter of reference.

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14. Optional Information: Racial/Ethnic Background: ______________________________
(Note: When possible, at least one award will be given to an under-represented minority applicant.)

15. Give a brief statement concerning your chosen academic major, and your career objective.

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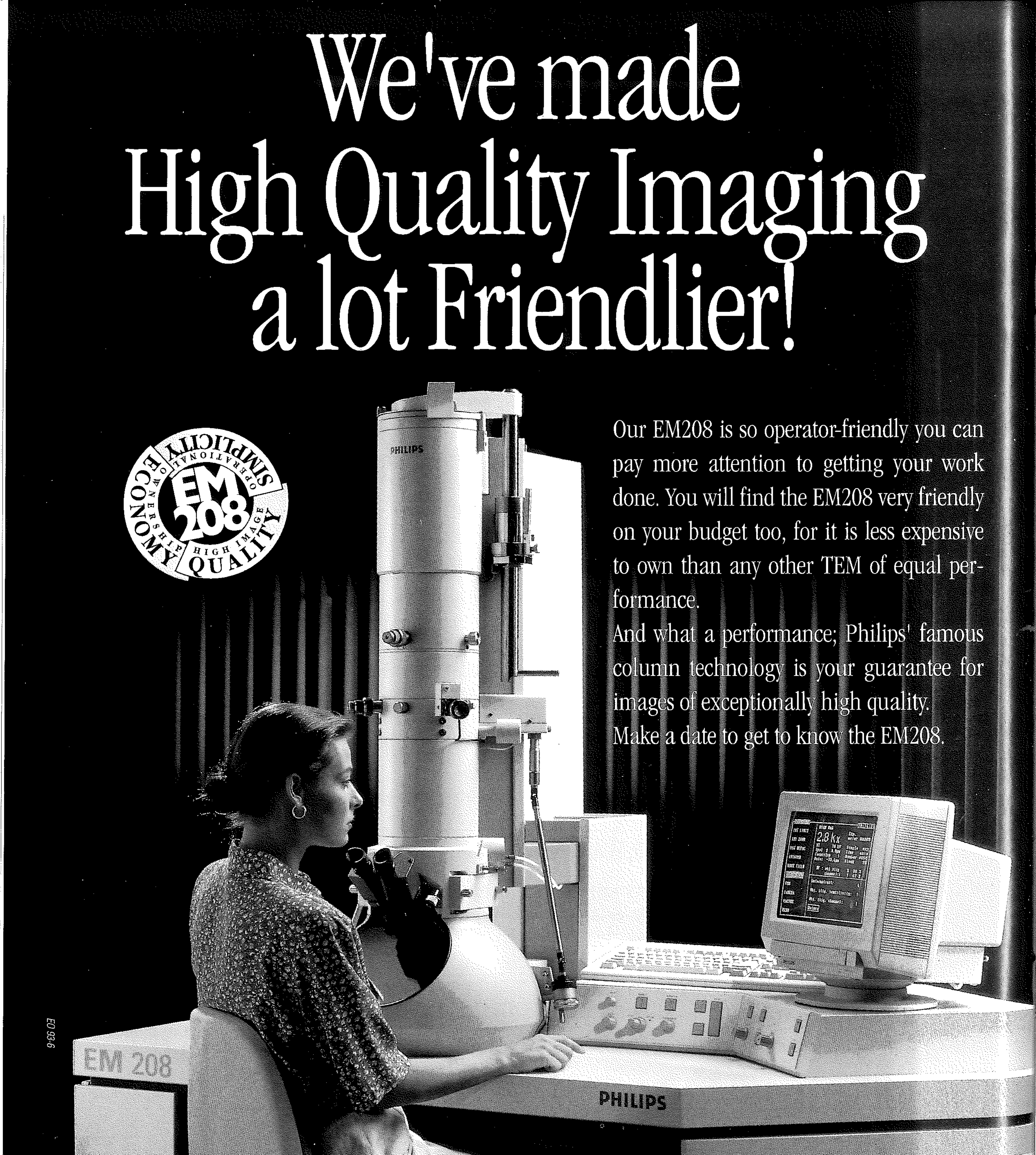
16. I certify that I am a full time undergraduate student, and that all information provided in this application is true. Any false information may result in forfeiture of awarded funds.

Applicant's Signature: ______________________________ Date: ____________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Abstracts

BIOLOGICAL SCIENCES

POSTER PRESENTATION — SPRING 1994

RECOVERY OF INSECTS BY USING FROTH FLOTATION DEVICES. B.B. PENDLETON & M.W. PENDLETON, Department of Entomology, Texas A&M University, College Station,TX 77843-2475.

The sorghum midge, *Contarinia sorghicola* (Coquillett) is one of the most damaging insects of sorghum, *Sorghum bicolor* (L.) Moench. Female sorghum midges oviposit in flowering sorghum spikelets. Sorghum midge larvae hatch and feed on developing sorghum ovaries preventing kernel development. Some sorghum midge larvae overwinter in spikelets that have fallen to the ground. In early spring, the first generations feed on johnsongrass, *Sorghum halepense* (L.) Pers., until flowering sorghum becomes available. A froth flotation device constructed of welded high-density polypropylene plastic was used to recover sorghum midge larvae from samples of cropland soil. During froth flotation, pressurized air is bubbled through a column of liquid containing the soil sample. Due in part to the attachment of minute air bubbles to objects suspended in the liquid column and to the addition of water to the column tank, seeds and insect parts are separated and overflow from the top of the column into a series of screens for collection. Special reverse-flow impellers forced jets of water along the underside of the collection screens to prevent clay particles from forming barriers to screening. Sorghum spikelets with overwintering sorghum midges were recovered by this device and identified by using light and scanning electron microscopy.

A Grant-in-aid provided by the Department of Anthropology, California State University, Chico facilitated the construction of this device. Electron microscopy was performed at the Electron Microscopy Center, Texas A&M University, College Station, Texas.

Dorsal Glands of *Alligator mississippiensis*. M.S. CANNON, R.W. DAVIS, and P.J. WELDON* Departments of Anatomy and Neurobiology, and Biology*, Texas A&M University, College Station, Texas 77843

The North American alligator (*Alligator mississippiensis)* suffered a major population depletion in this century that led it to be considered an endangered species. After considerable effort on the part of environmentalists and naturalists this animal has made an amazing recovery and is no longer considered endangered. Along with this re-establishment of sizable populations has come renewed research interest in its behavior, anatomy, and histology.

The gross anatomy of the dorsal glands of crocodilians has been described in only a couple of species. We investigated those of the alligator . These organs lie on the deep surface of the dermis nearly at the superficial fascia and usually occur in 2 rows of 20-22 glands. Each is lined by a stratified cuboidal to stratified columnar epithelium in varying stages of degeneration indicating a holocrine-type of secretion. Surrounding each gland are moderate to very dense collagenous fibers and numerous short elastic fibers. Skeletal muscle surrounds this connective tissue. The epithelial cells and secretory product contain slight to moderate amounts of lipid and are periodic acid-schiff positive with no reduction in staining following diastase digestion. Unique, elongate crystals, sometimes demonstrating a dense core and/or layering, occur in the epithelial cells and secretory product. Moreover, more than one type of crystal may be present. By energy dispersive x-ray analysis the crystals appear to contain lead, calcium, zinc, iron, copper, and potassium. Some interdigitation of epithelial cell membranes occurs and desmosomes are occasionally observed. Mitochondria, some vacuole-like structures, and possible short segments of rough endoplasmic reticulum also occur in the cells.

THE STOMATA OF THE MOSS LORENTZIELLA IMBRICATA. ANN E. RUSHING, Department of Biology, Baylor University, Waco, TX 76798-7388.

Stomata of Lorentziella imbricata (Mitt.) Broth. are located in the epidermis of the lower portion of the sporophyte capsule. Mature stomata give access to a substomatal cavity continuous with a large internal air space within the capsule. Each stoma consists of a single, binucleate guard cell with a centrally located pore in the anticlinal, ventral wall. The binucleate condition results from nuclear division followed by incomplete formation of the anticlinal, ventral wall during cytokinesis. Paradermal sections reveal, however, that this anticlinal, ventral pore forming wall is connected to the dorsal, anticlinal end walls throughout some of the depth of the cell. Initially the ventral pore forming wall is similar in thickness to the remainder of the guard cell walls. As development proceeds the wall becomes thickened centrally. Rough endoplasmic reticulum and dictyosomes are found in the cytoplasm surrounding areas of wall thickening and wall extension. Pore formation occurs in the anticlinal, ventral wall by the splitting of the middle lamella, perhaps linked to cuticularization of the middle lamella and pore wall, resulting in a centrally located pore. Guard cells contain many vacuoles and numerous starch filled chloroplasts.

BIOLOGICAL SCIENCES

PLATFORM PRESENTATION — SPRING 1994

A MICROSCOPIC AND X-RAY STUDY OF THE HYPHOPODIA OF THE TAKEALL DISEASE ORGANISM, *GAEUMANNOMYCES GRAMINIS*, GROWN UNDER DIFFERENT MINERAL REGIMES.

Arnott, H. J., Jones, E. G. and Lopez, L. E. Department of Biology, University of Texas at Arlington, Arlington TX 76019.

We used several types of microscopy to study the structure and chemical variation found in the hyphopodia of *Gaeumannomyces graminis* var. *graminis* grown with added metals. Sterile culture dishes containing potato dextrose agar either alone (controls) or with various concentrations of Mn, Mg, Co, Fe, Cu Zn were inoculated with strain 501 IL, provided by Don Huber of Purdue University, and grown at room temperature. The growth rates of runner hyphae and the patterns of hyphopodial development were examined using LM; fixed preparations were examined by SEM and thin sections were examined by STEM and x-ray analysis. This investigation confirmed previous observations (Arnott, *et al*, 1992) showing manganese deposition associated with the walls of hyphopodia and short lateral dendric branches but not with runner hyphae. The manganese deposits are entirely external and interrelated with a fibrous material apparently produced by the hyphal walls. The manganese mineralization process begins in almost mature or mature hyphopodia. The deposition starts with patch-like zones which gradually grow together and cover the entire free surface of the hyphopodia with a golden-brown coat. STEM and x-ray analysis show that the manganese compounds are deposited outside the hyphal cell wall Other metals, at same concentration level that produce abundant deposits of manganese, cause reduced or complete cessation of growth from the inoculum.

ULTRASTRUCTURE OF ROOT APICAL MERISTEM OF *PINUS KORAIENSIS* TWO DAYS AFTER SEED GERMINATION. Z. H. NING, Jarvis Christian College, Hawkins, TX 75765.

Root tips of *Pinus koraiensis* were fixed two days after seed germination. Root meristems were observed using a transmission electron microscope. There was considerable cell division and elongation in the meristems. During seed germination a great amount of protein that had accumulated in the protein bodies was broken down and utilized as energy forming small vacuoles within the protein bodies. Gradually the small vacuoles fused to form larger vacuoles.

THE INFECTION OF *CYNODON DACTYLON* BY *GAEUMANNOMYCES GRAMINIS*.

Huffine, M. S., Arnott, H. J. Department of Biology, University of Texas at Arlington, Arlington, TX 76019.

Using light microscopy (LM) and scanning electron microscopy (SEM) we studied the infection process of *G. graminis* var. *graminis* (a root infecting fungus) on *Cynodon dactylon* (bermudagrass). Turfgrass seedlings were grown with and without fungal inoculum in sterile chambers maintained at room temperature. Seedlings of varying ages were fixed in glutaraldehyde, embedded in paraffin, stained, and sectioned with a microtome for LM or freeze fractured, critical point dried, and coated for SEM. All seedlings, except controls, became infected with the fungus. Hyphae grew on all surfaces of the host and formed mycelial mats and hyphopodia on leaf, coleoptile, and stem surfaces. Specialized forms of hyphal cells directly penetrated epidermal cells of the roots, stem, and coleoptile. Following root and stem penetration, hyphae grew within the cortex, endodermis, and stele. Numerous lignituber-like structures were observed within the cortex. Hyphal growth within the coleoptile was restricted to mesophyll tissue, there was little or no hyphal growth observed inside the leaves. Direct penetration of the host may be the result of a combination of enzymatic and mechanical activities by the specialized hyphae. The disintegration of stelar tissues of roots causes the eventual death of the plant.

AN ULTRASTRUCTURAL STUDY OF THE SPOROPHYTE-GAMETOPHYTE JUNCTION IN EPHEMERUM (MUSCI, EPHEMERACEAE). K. L. YIP and A. E. RUSHING, Biology Dept., Baylor University, Waco TX 76798-7388.

The young sporophyte foot of Ephemerum cohaerens is conical and tapered with a large basal tip cell. The placental region of the sporophyte-gametophyte junction consists of the epidermal layer of the sporophyte foot, the space between the two generations, and the innermost layer of the gametophyte. Characteristic transfer cells are found in this junction. For those transfer cells in the gametophyte, their ingrowths are not as extensive as their sporophyte counterparts and no ingrowths are found in their radial walls. Mitochondria, starch-filled plastids, and extensive lipid deposits are characteristics of these cells. Extensive ingrowths of wall materials line the tangential as well as the radial walls of sporophytic transfer cells. Ingrowths develop first in the basal tip cell. Inner tangential walls of these cells have some elaborations. An interesting feature is the contrast of the ingrowth density between the upper wall of the tip cell (less ingrowths) and the adjacent walls of the cells above (more ingrowths). Transfer cells in the sporophyte are characterized by pleomorphic plastids, mitochondria, small vacuoles, and strands of rough endoplasmic reticulum. These cells also have some starch granules within the plastids, and small lipid droplets in the cytoplasm.

A FORENSIC ANALYSIS OF THE COMPONENTS OF TEA PACKAGING.

Arnott, H. J., Tennant, M.M. Department of Biology, University of Texas at Arlington, Arlington TX 76019.

We used light microscopy (LM), scanning electron microscopy (SEM) and x-ray analysis to investigate the structure and chemical nature of several different components involved the packaging of tea used by Ceylon Tea Merchants and marketed in the USA under the label "Terra Teas". In this study we were particularly interested in determining the nature of the wood used to manufacture the tea boxes as well as the nature of the external and internal wrapping and display materials utilized these products. Investigation of the wood using LM and SEM revealed a diffuse porous wood having a few widely spaced vessels. The vessel members had simple perforation plates and were ca. 200 µm in diameter and 350 µm in length; they were occasionly occluded by tyloses. The remaining wood was composed of tracheids, having a diameter of 20-30 µm, separated by numerous uniseriate rays. Using literature descriptions and wood samples we identified this wood as *Ochroma lagopus* (balsa wood). Both the tea box wood and samples of balsa wood exhibited similar structure including occasional multiseriate rays in which each cell contained crystals of calcium oxalate. Examination of the inner lining of the box revealed a metallic coated paper, x-ray analysis showed that the metal coating was pure aluminum. The string attaching the tea bag to its tag was made of man made fibers. The paper forming the tea bag was made of loosely woven flattened fibers, apparently of natural origin. The paper contained many pores between 50-125 µm in diameter. Other papers used in this packaging were also examined revealing a variety of coats, structures and densities.

CONTINUING APPLICATIONS OF DIAGNOSTIC ELECTRON MICROSCOPY IN NEUROLOGIC AND NEUROMUSCULAR DISEASES. S.C. BAUSERMAN, Dept. Pathology, Scott and White Clinic and Texas A&M University, Temple TX 76508.

In spite of the immense popularity of immunohistochemistry and immunocytochemistry in diagnostic pathology, there is a continuing role for electron microscopy (predominantly TEM) especially in diagnostic neuropathology. Certain neurodegenerative diseases and some hereditary conditions remain without biochemical markers or other means for establishing a diagnosis. Selected case vignettes are presented with emphasis on the role played by diagnostic electron microscopy in establishing diagnoses and directing therapeutic plans. Examples include: Neuronal Ceroid Lipofuscinosis (NCL) studying peripheral blood lymphocytes, conjunctiva and selected tissues; Mitochondrial Myopathies; Neuroaxonal dystrophy and certain collagen disorders. Additional applications for the diagnosis of certain central nervous system tumors remain significant, especially in children.

MATERIALS SCIENCES

PLATFORM PRESENTATION — SPRING 1994

DEFORMATION MICROSTRUCTURES IN TANTALUM EXPLOSIVELY FORMED PENETRATORS, C-S. NIOU, L. E. MURR, AND C. FENG*, Department of Metallurgical and Materials Engineering, The University of Texas at El Paso, El Paso, TX 79968, and *U.S. Army Armament Research, Development, and Engineering Center, Picatinny Arsenal, NJ 07806

A systematic optical and transmission electron microscope investigation of recovered tantalum explosively formed penetrators (EFP's) has been conducted. Variations of straining range from essentially zero to in excess of 300% in various EFP locations. Corresponding observations depict an evolution of microstructures involving dislocation and dislocation loop development, dislocation cells which elongate to form sub-boundaries or dynamically recrystallized regions having boundary misorientations (Θ) in excess of 5°. Dark-field TEM is used to differentiate these regions and examine the microstructural evolution in terms of misorientation angles observable in the selected-area electron diffraction patterns for $0<\Theta>5°$. The micro structural features of interfacial evolution corresponding to this range of misorientations is also critically examined in the context of dislocation cell walls, dense dislocation cell walls, and high-angle grain boundaries or so-called geometrically necessary boundaries characteristic of recrystallized regions in the most heavily deformed regions of the EFP. The implications of these detailed microstructural observations in the development and design of self-forging projectiles will be described with reference to popular constitutive equations and their applications.

INTERMETALLIC PHASES OF EUTECTIC Ag/Sn SOLDER. D.R. FLANDERS, E.G. Jacobs, R.F. Pinizzotto, Center for Materials Characterization, University of North Texas, Denton, TX 76203.

Copper strips approximately 2mm wide and 10mm long were soldered with eutectic 4Ag/96Sn solder to form sandwich-like structures. The goals of this study are to characterize the intermetallic phases formed at the Cu/solder interface, to study the diffusion behavior of Sn, and to calculate the activation energies for intermetallic formation. Samples were annealed in dry box ovens at 110, 130, 140, 150, 160 and 170°C for 0, 1, 2, 4, 8, 16, 32 and 64 days. This gave an overall sample matrix of 48 samples. The solder and intermetallic phases were examined using SEM/XEDS. The intermetallic layers at the Cu/solder interface were Cu_3Sn, Cu_6Sn_5 and Ag_3Sn. Average thicknesses of the intermetallic layers were calculated by encoding a minimum of 100 thickness measurements into a PC for data analysis. Diffusion coefficients, D, for each intermetallic layer were calculated at each temperature assuming a rate equation of the form $x=\sqrt{Dt}$. Using this data, the activation energies of the Cu_3Sn and Cu_6Sn_5 layers were determined. The activation energies of eutectic Ag/Sn were compared to the activation energies of eutectic Sn/Pb.

FUNDAMENTAL AND COMPARATIVE STUDIES OF $M_{23}C_6$ CARBIDE GROWTH ASSOCIATED WITH TWIN BOUNDARIES AND GRAIN BOUNDARIES IN 304 STAINLESS STEEL, ROBERTA J. ROMERO AND L. E. MURR, Department of Metallurgical and Materials Engineering, The University of Texas at El Paso, El Paso, TX 79968

It is well known that $M_{23}C_6$ carbide precipitation occurs first on general grain boundaries, then an non-coherent twin boundaries, and finally on coherent twin boundaries as evidenced by heat treatment of 304 stainless steel. This aging-related sequence for carbide growth kinetics is apparently related to the associated interfacial free energies: $\gamma_{gb} \cong 800$ mJ/m^2, $\gamma_{TB} \cong 200$ mJ/m^2, $\gamma_{tb} \cong 20$ mJ/m^2. We have observed some rather unique growth features for carbides associated with these distinct interfaces using transmission electron microscopy (TEM). Growth from regular grain boundaries, while often along {111} trace directions is confined to regions connected to the interface and carbides often form large, faceted precipitate blocks along the interface. Growth from non-coherent twin boundaries is usually lamellar with the long lamellar carbide interphase boundaries along specific {111} trace directions dependent upon the grain (or twin) surface orientation. Lamellar growth associated with the coherent twin boundaries is observed to be either parallel to the {111} coherent boundary trace or non-coplanar, but along another {111} trace direction. In addition, carbides growing parallel or coincident with the coherent twin interface are often observed to be growing from non-coherent microsteps in the boundary plane. These growth-specific features for carbides in heat-treated 304 stainless steel can be rationalized in large part by examining the crystallographic/geometric features through a configurational equilibrium theory for relative interfacial torques associated phenomenologically with coherent twin boundary - grain boundary intersections. Numerous examples of these growth-specific carbide features in heat treated 304 stainless steel will be presented and the implications discussed in terms of both theory and practice. Supported by NSF-RIMI Grant HRD 9105065.

THERMOMECHANICAL AND MATERIAL PARAMETER EFFECTS ON CARBIDE PRECIPITATION IN 304 SS VERIFIED BY ELECTRON MICROSCOPY, E. A.TRILLO, L. E. MURR and W. W. FISHER, Department of Metallurgical and Materials Engineering, The University of Texas at El Paso, El Paso, TX 79968

Thermomechanical processing and chemical composition are two crucial parameters concerning carbide precipitation and sensitization development in 304 SS. This research compares the effect that several variables (temperature, carbon content, and strain) have on carbide nucleation and growth. Four carbon contents were chosen: 0.01, 0.025, 0.05, and 0.07 wt%C to document the carbon content effect on carbide precipitation. The temperature regimes that were utilized in this study were 775°C and 625°C. They illustrate the differences in high and moderate temperatures. The effect of strain was also recorded in this study. Samples were deformed 0, 10, and 20% true strain and treated at 625°C. Electron microscopy verifies all effects that were recorded with the electrochemical potentiokinetic reactivation (EPR) test. Supported by NSF-RIMI Grant HRD 9105065.

TEM SAMPLE PREPARATION FOR SPECIFIC-AREA ANALYSIS. P.B. BASHAM, M. COVIELLO, J.L. WALLER, AND H.L. TSAI, Texas Instruments Incorporated, Materials Science Laboratory, Dallas, TX 75265

In advanced microelectronic devices, failure analysis is often conducted on specific locations. These locations can be very small such that they are not visible with the naked eye. Using Transmission Electron Microscopy (TEM) for this analysis can be especially difficult since the specific areas need to be polished thin enough for electron transmission.

The selection of the proper preparation technique is the key to accurately analyzing TEM samples. Various samples require different methods of preparation. These are prepared by carefully controlling the polishing and the ion milling of the sample. Other techniques are the single-bit and the modified techniques both of which target a specific area as small as .5 microns. A detailed discussion will be presented on each of the techniques.

FIBROUS CLAY SUPPORT FOR HIGH RESOLUTION TRANSMISSION ELECTRON MICROSCOPY SPECIMENS OF SMALL CRYSTALS. J. B. DIXON, Soil & Crop Sciences Department, Texas A&M University, College Station, TX 77843.

The examination of small ($<<1$ μm) particles with rounded shapes such as those in many clays and nodules from soils is complicated by the similarity of the subject to lumps of extraneous carbon that are common on holey C films. Also, small crystals are seldom positioned in space for ideal viewing on C films. A recent report in Australia of a fibrous halloysite describes a suitable material to use in solving the problem (Norrish, 1993). A dilute suspension of fibrous halloysite is mixed with the specimen in water and drop mounted on a holey C film with large holes. Such open holey C films are commonly discarded, but are effective here. The tubular fibers are thin walled and straight to slightly curved with a diameter or width of about 50 nm. The mineral is unstable in the electron beam and exposure of about two minutes destroys the crystal structure. The low mass of the thin fibers provides little interference by absorption or diffraction. Thus the halloysite provides an almost perfectly clean mesh that contrasts with irregularly shaped soil particles and makes them easy to find and image. The halloysite fibers are much less absorbing than the typical holey carbon film. The composite technique has been tried on manganese oxides and morphology and lattice images were easily obtained with much less searching time than with typical holey carbon films. The discovery of natural fibrous clay for use as a mounting substrate may lead to pure synthetic fibers designed for particular specimens.

Norrish, K. 1993. An unusual fibrous halloysite. Abstracts of Keynote Addresses, Oral and Poster Papers, 10th International Clay Conference, Adelaide, Australia. p. K-2.

ANSWER TO "WHAT IS IT"

from TSEM JOURNAL 24:2

Scanning EM view of rat trachea after treatment with EDTA solution, which removes the ciliated and nonciliated epithelial cells, leaving predominantly basal cells attached to the basal lamina. The pavement-like pattern of basal cells is visible at the bottom, while the ciliated and Clara cells are present on the top. (Magnification X 1500)

Micrograph — Robert A. Cox, Shriners Burns Institute, Galveston, TX 77550

DYNAMIC RECRYSTALLIZATION IN BALLISTIC AND HYPERVELOCITY PENETRATION AND CRATER FORMATION IN METALS, JESUS M. RIVAS, STELLA QUINONES, AND L. E. MURR, Department of Metallurgical and Materials Engineering, The University of Texas at El Paso, El Paso, Texas 79968

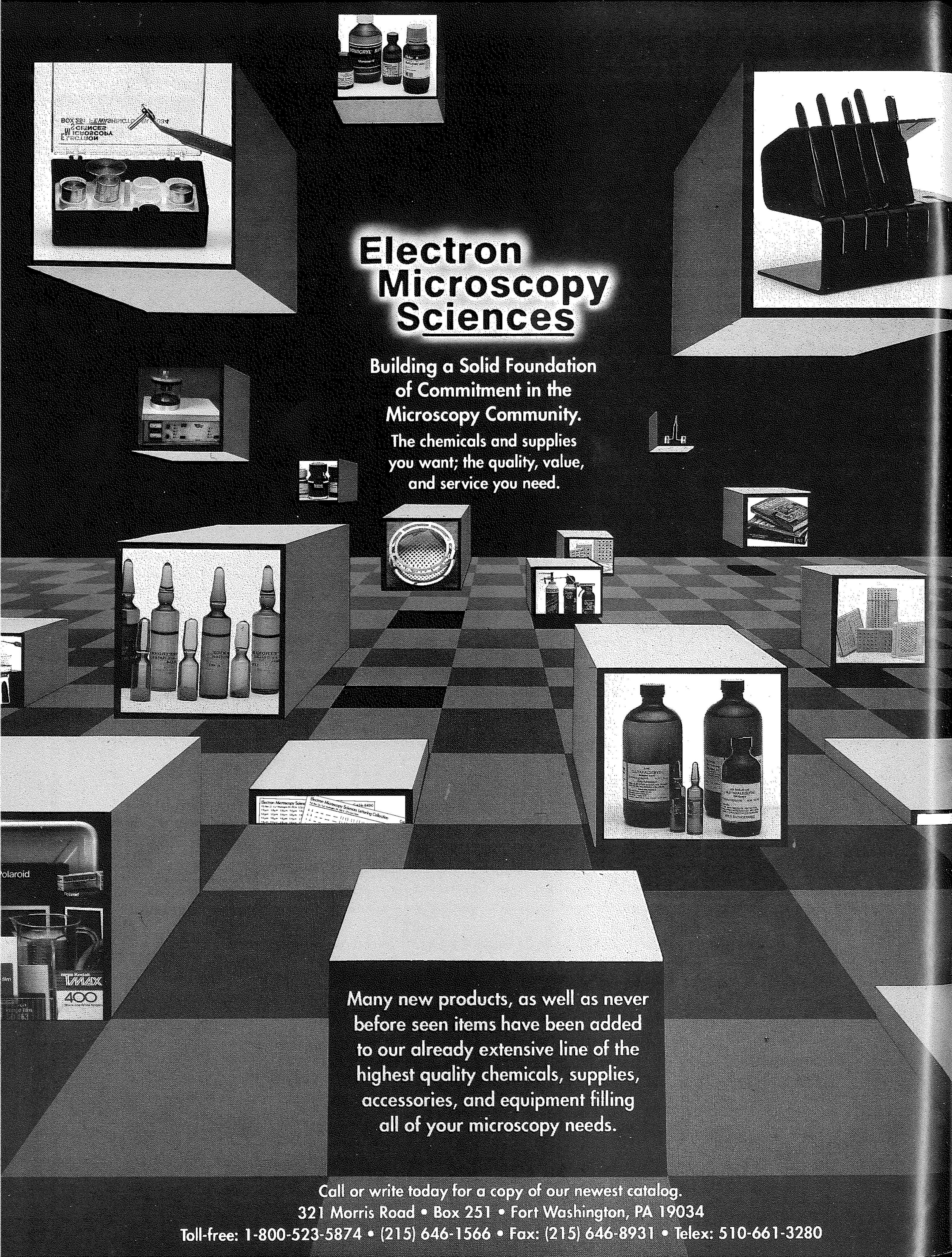
Studies of ballistic and hypervelocity penetration, especially laboratory simulations using high velocity guns, have been a popular means of studying cratering in spacecraft materials for more than 40 years. It is remarkable to realize that while data regarding parametric relationships between impacting particles and the residual crater geometries have been well established, there is not a single, systematic metallurgical analysis of crater-related deformation phenomena in metallic targets. Here we report on the first, detailed observations of dynamic recrystallization in impact craters in 1100 aluminum and copper by preparing electron transparent disc samples from thin slices through laboratory-produced craters. In addition to grain refinement in copper craters near the crater wall, the recrystallized grains have no annealing twins, and these observations suggest fundamental mechanisms for crater-related deformation processes. Supported by NASA-Johnson Space Center Grant NAG 9-481.

RECRYSTALLIZATION PHENOMENA IN HIGHLY DEFORMED AND SENSITIZED 304 STAINLESS STEEL, J. G. MALDONADO AND L. E. MURR, Department of Metallurgical and Materials Engineering, The University of Texas at El Paso, El Paso, TX 79968

Austenitic 304 stainless steel uniaxially deformed to a 40% true strain level has shown some interesting characteristics in terms of the microstructural changes occurring during heat treatment at 670°C (for aging times up to 10h) to produce sensitization in the material. The primary objective of this study was to investigate the effects of strain-induced martensite (α'–martensite) on transgranular (TG) carbide precipitation. Strain-induced α'–martensite was found not to be a precursor to TG carbide precipitation and in fact it was observed to undergo a transformation/annihilation with thermal treatment. A new phase was observed to have formed in some of the previously strain-induced α'–martensite locations. Diffraction studies showed that the new observed phase was also martensite, but its morphological characteristics were indicative of the lath-martensite phase and a product of aging-induced recrystallization and growth. After the shortest thermal treatment (0.1h) the microstructure showed very significant changes in which fine-intermixed regions containing fine-grained austenite and lath martensite were observed. This apparent recrystallization phenomena, which continues to occur in longer heat treatments, appears to have a marked importance, and plays a major role in the sensitization behavior of austenitic 304 stainless steel. The highly deformed condition of the material provides a suitable mechanistic environment for the recrystallization process to occur. Also, the intermixing of the strain-induced α'-martensite phase in the austenitic matrix may have a significant bearing in the observed recrystallization phenomena. Supported by NSF-RIMI Grant HRD 9105065.

TIME SERIES IMAGING OF CHEMICAL VAPOR DEPOSITION DIAMOND NUCLEATION SITES ON SILICON AND THEIR BEHAVIOR DURING THE GROWTH PERIOD USING SCANNING ELECTRON MICROSCOPY. Richard Stallcup, Jose Perez, Wayner Rivera, Albert Aviles, University of North Texas, Physics Department, Denton, Texas 76203 and Mike Gilbert, Dept. Computer Education and Cognitive Systems Program

Chemical Vapor Deposition (CVD) diamond crystals grow and interact with one another on a substrate of silicon (100). Over time the morphology may change and crystals obtain secondary nucleation sites. In order to observe the diamond to diamond interaction and secondary nucleations the substrate is imaged using a Scanning Electron Microscope (SEM) then exposed to the CVD process and then re-imaged. This was done multiple times in order to obtain a time series set of Polaroid pictures. The SEM Polaroid pictures were then digitized using a scanner and a PC. The digital files were converted into a computer slide show.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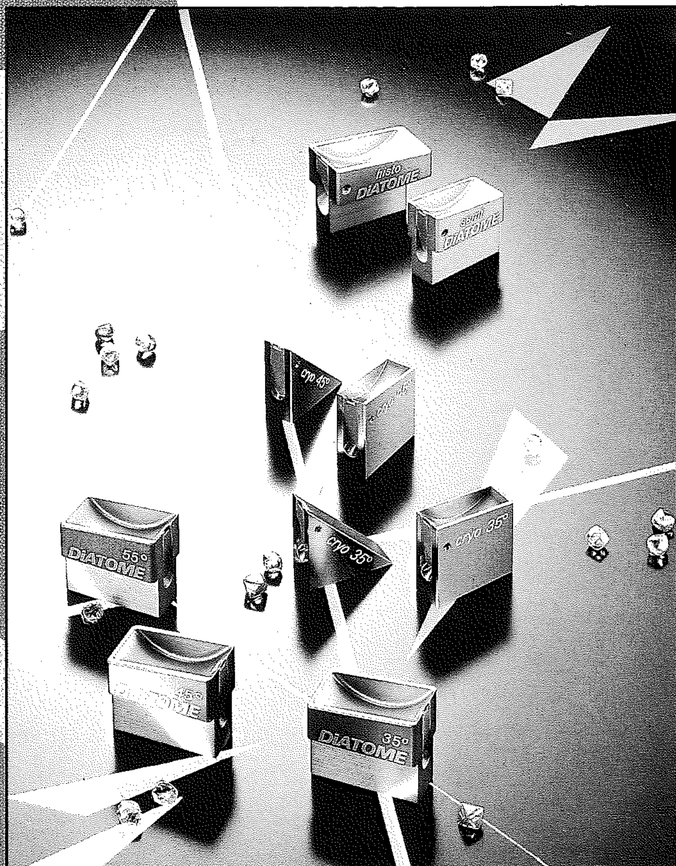
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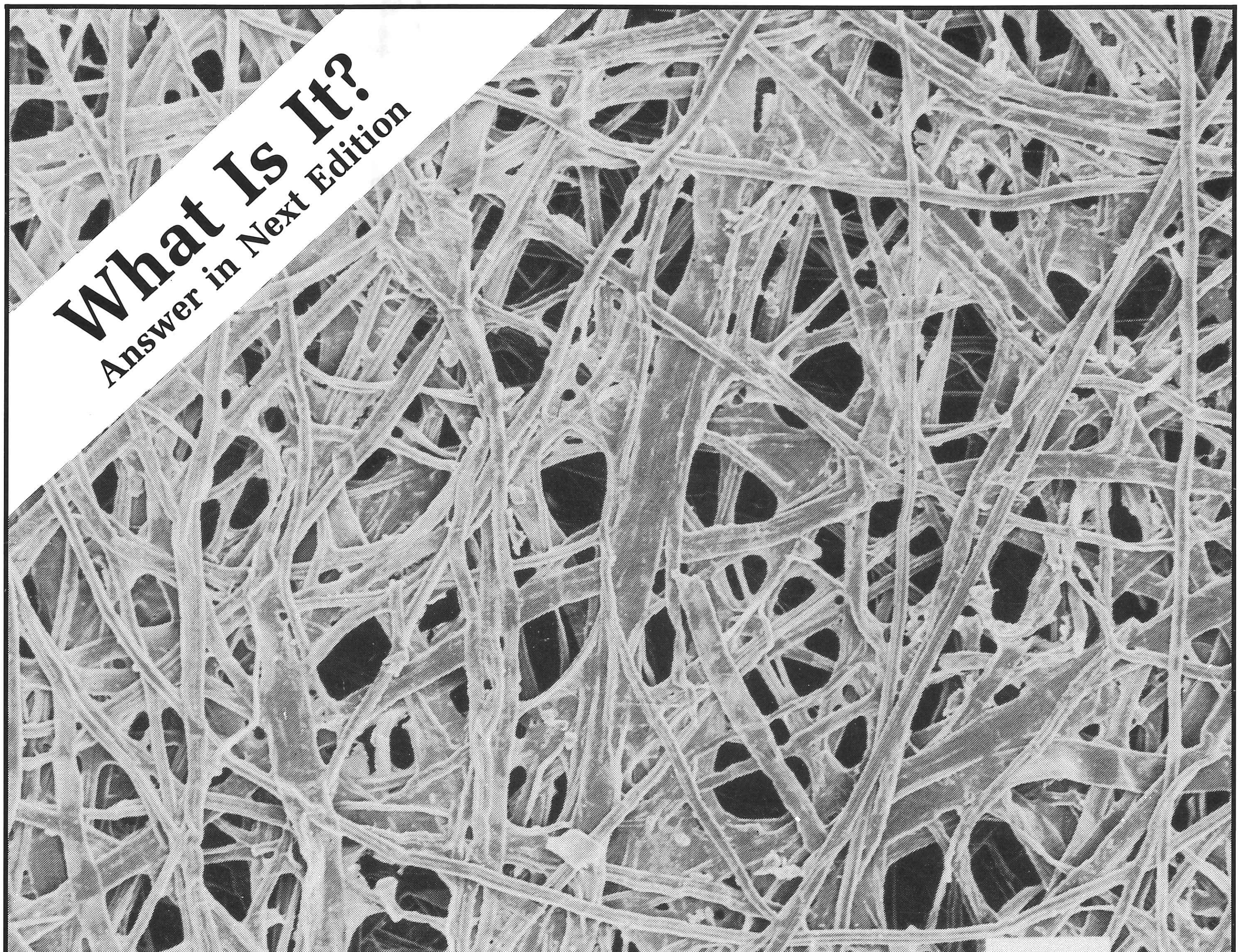

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