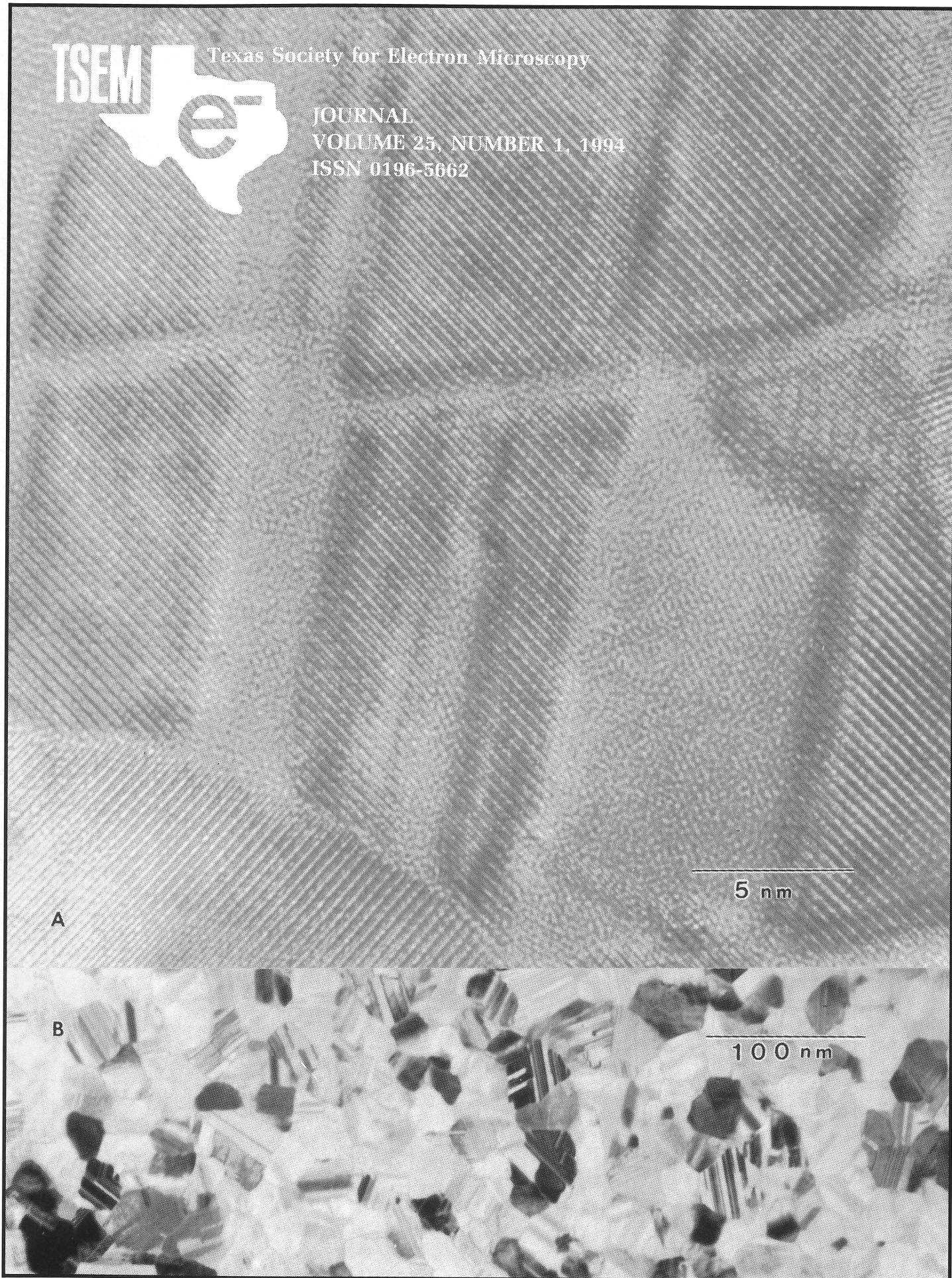




Texas Society for Electron Microscopy

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



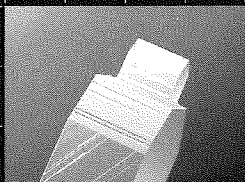
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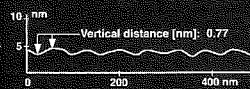
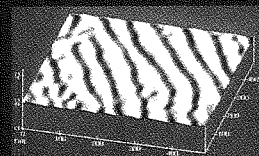
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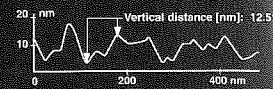
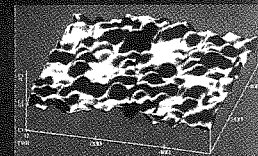
Only gem diamond crystals of the highest purity are selected to be crafted into MICRO STAR knives. The diamonds are cleaved, laser aligned to the crystal plane and welded to the metal shank assuring permanent stability and chatter free performance.



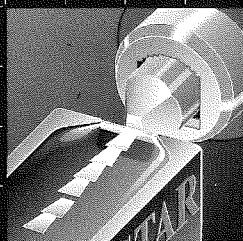
MICRO STAR knives are tested in our own full service EM laboratory which includes two TEM's, one SEM and one AFM.



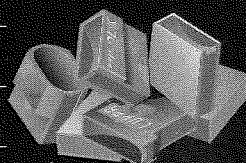
ATOMIC FORCE MICROSCOPE VIEW OF A MICRO STAR DIAMOND KNIFE SURFACE ADJACENT TO THE EDGE. ROUGHNESS IS LESS THAN 5 CARBON ATOMS.



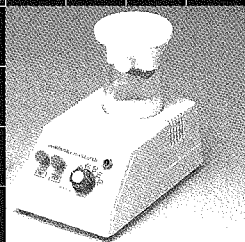
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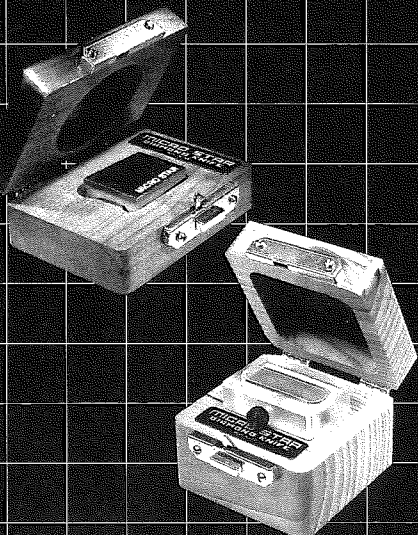
15

TYPE-ANGLE COMBINATIONS



59

EDGE LENGTH SIZES



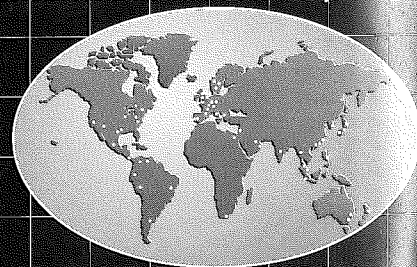
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David C. Garrett, Editor

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."

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ON THE COVER

Electron Microscopy of BaTiO₃ deposited on a Si(100) substrate using pulsed laser ablation in vacuum at 750°C. Micrograph B shows the polycrystalline structure of BaTiO₃. 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. Summelfelt, Texas Instruments, Dallas, TX.

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Commentaries
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TSEM JOURNAL**

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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

TEXAS SOCIETY FOR ELECTRON MICROSCOPY FALL MEETING 1994

Thursday, October 20 - Saturday, October 22

Best Western - Oak Hills Motor Inn
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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
Donations and Grants	173.75
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

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Certificate of Deposit No. 113515	\$3,890.52
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Reichert Ultracut S/FC S

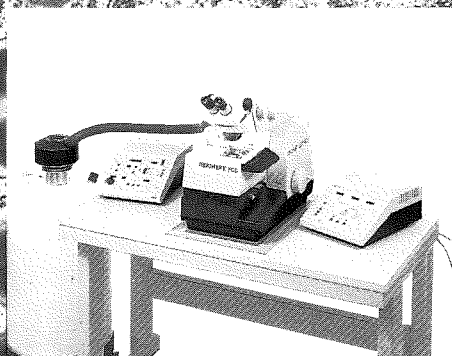
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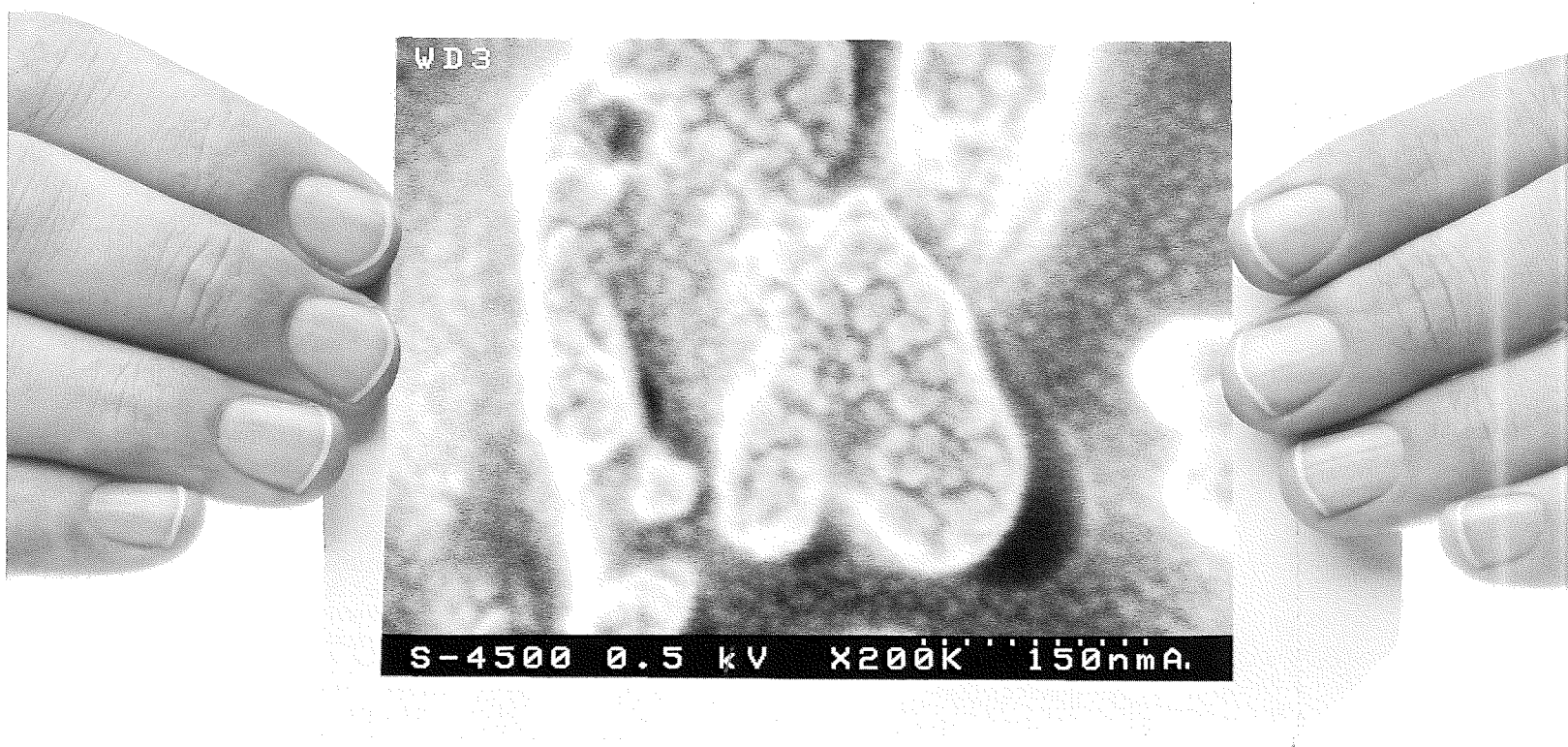


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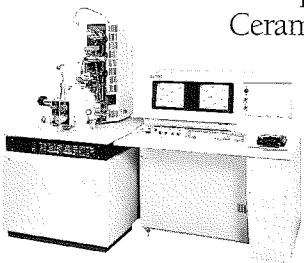
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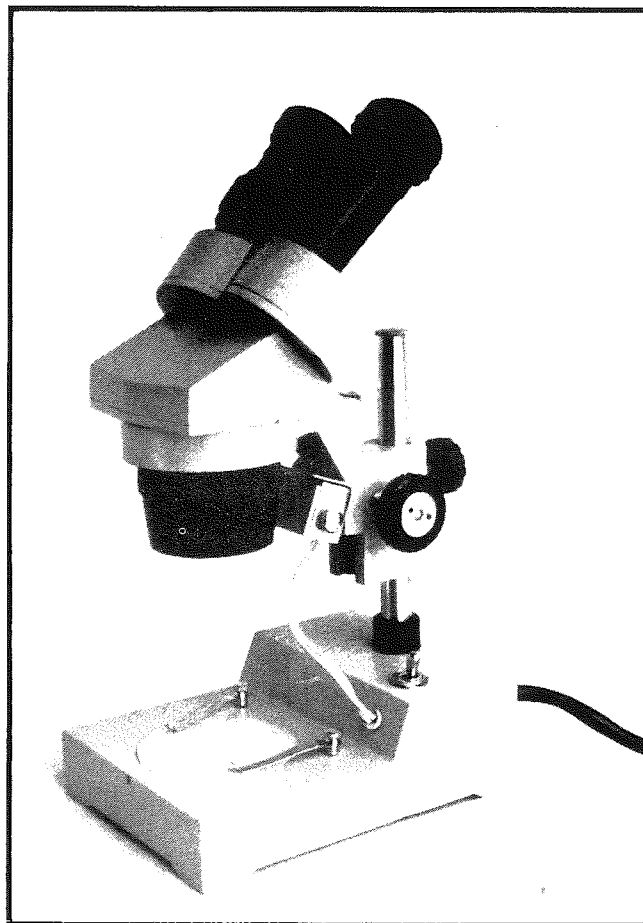
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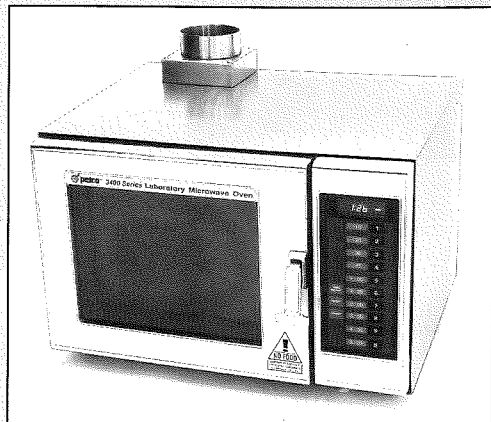
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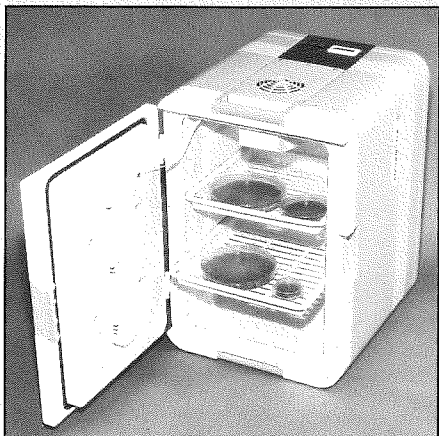
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FALL MEETING OF TSEM

October 20-22, 1994

San Antonio, Texas

See Details Elsewhere In This Journal

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May 7-12, 1994

Toronto, Canada

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

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May 17-20, 1994

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July 17-22, 1994

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**MICROSCOPY SOCIETY OF AMERICA
52nd ANNUAL MEETING**

and

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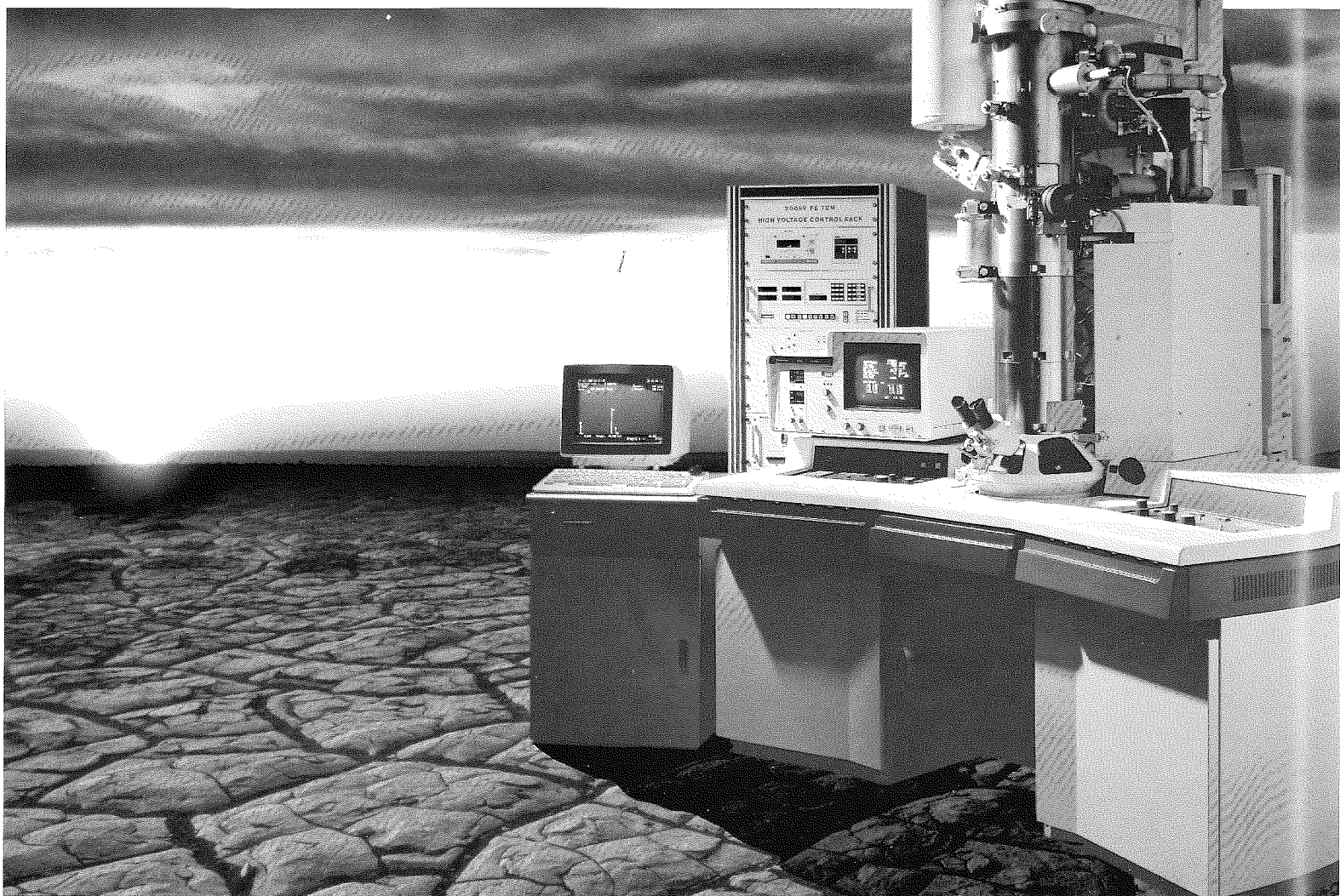
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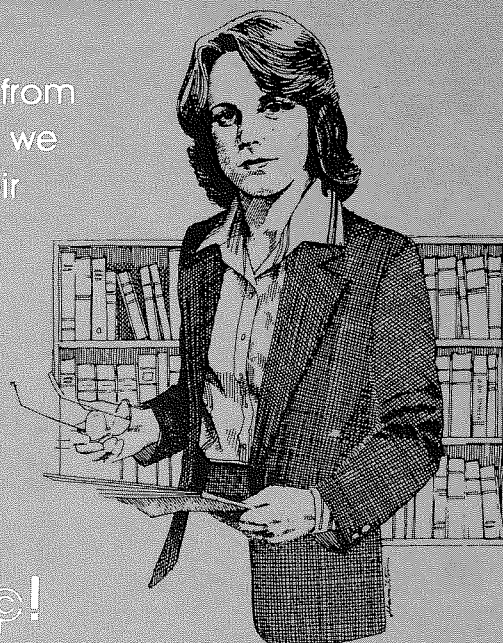


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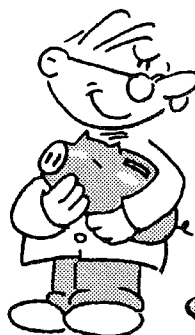
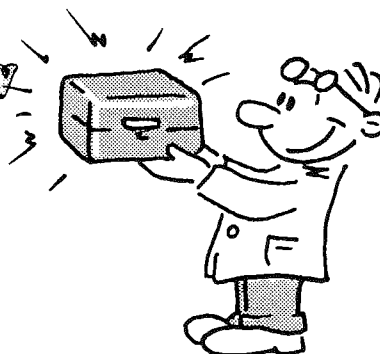
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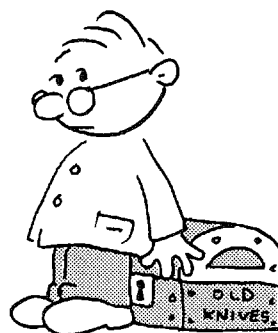
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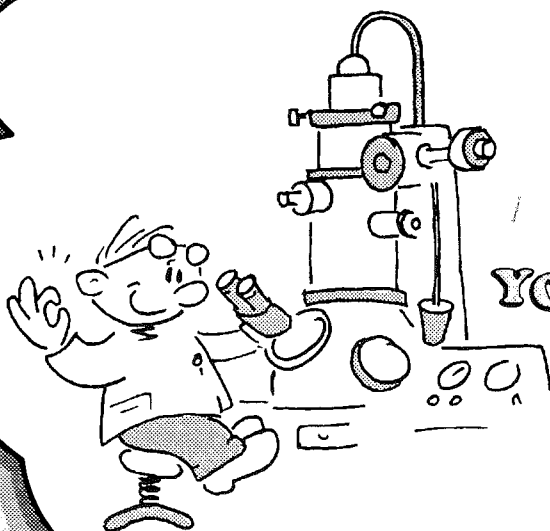
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ARTICLE — STUDENT COMPETITION WINNER

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 intermetallic 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:

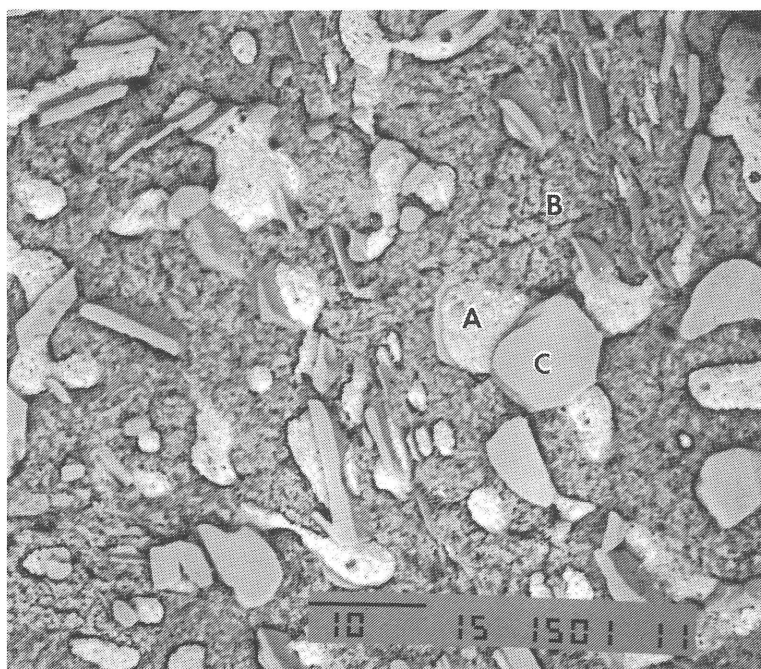


Figure 1



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) $\text{AuSn}_4/\text{AuSn}_4$ grain boundary and (2) AuSn_4/Sn phase boundary.

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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 AuSn₄ 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 AuSn₄ particles in contact with each other. Both AuSn₄ grains contact the Sn-rich phase of the solder. All these phases were identified using selected area electron diffraction patterns. The AuSn₄/AuSn₄ grain boundary (labeled as 1) and AuSn₄/Sn phase boundary (labeled as 2) are clearly visible. XEDS in STEM mode was used to determine the Sn/Au ratios along the AuSn₄/AuSn₄ grain boundary and AuSn₄/Sn phase boundary, compared to the Sn/Au ratios within the AuSn₄ phase (measured 150 Å away from the boundary toward the center of the AuSn₄ 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 AuSn₄ grains. This means that the AuSn₄ grain boundaries and the AuSn₄/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 AuSn₄, and the much faster boundary diffusion through the AuSn₄/AuSn₄ grain boundaries and AuSn₄/Sn phase boundaries. This enhanced Sn diffusion reduces the activation energies for the formation of both Cu₆Sn₅ and Cu₃Sn at the Au composite solder/copper interface compared to the eutectic solder alone. The diffusion of Sn in 20 wt% Cu₆Sn₅ and 10 wt% Cu₆Sn₅ composite solders was also examined. It was found that the thicknesses of Cu₆Sn₅ and Cu₃Sn at the 20 wt% Cu₆Sn₅ 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% Cu₆Sn₅ composite solder. Therefore the diffusion coefficient is larger for 20 wt% Cu₆Sn₅ composite solder than for 10 wt% Cu₆Sn₅ composite solder. Since the Cu₆Sn₅ phase is the terminal phase in the system, the Cu₆Sn₅ phases do not react with Sn. The only difference between these two solders is that there are more Cu₆Sn₅ grains in the 20 wt% Cu₆Sn₅ composite solder and therefore more Cu₆Sn₅ grain boundaries. These boundaries also appear to enhance 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).

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Include a detailed budget with sources of additional support for project on a separate page.
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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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PHILIPS

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 dendritic 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.

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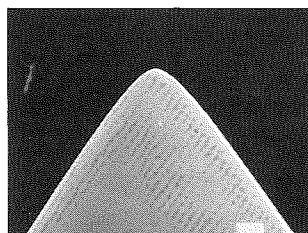
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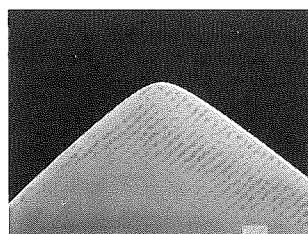
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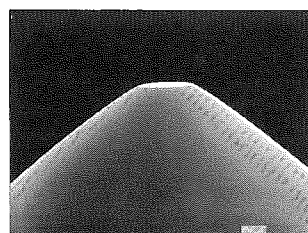
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THE INFECTION OF *CYNODON DACTYLON* BY *GAEUMANNOMYCES GRAMINIS*.

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 in 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 μ m in diameter and 350 μ m in length; they were occasionally occluded by tyloses. The remaining wood was composed of tracheids, having a diameter of 20-30 μ m, 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 μ m 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 $0 < \theta < 5^\circ$. The microstructural 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.

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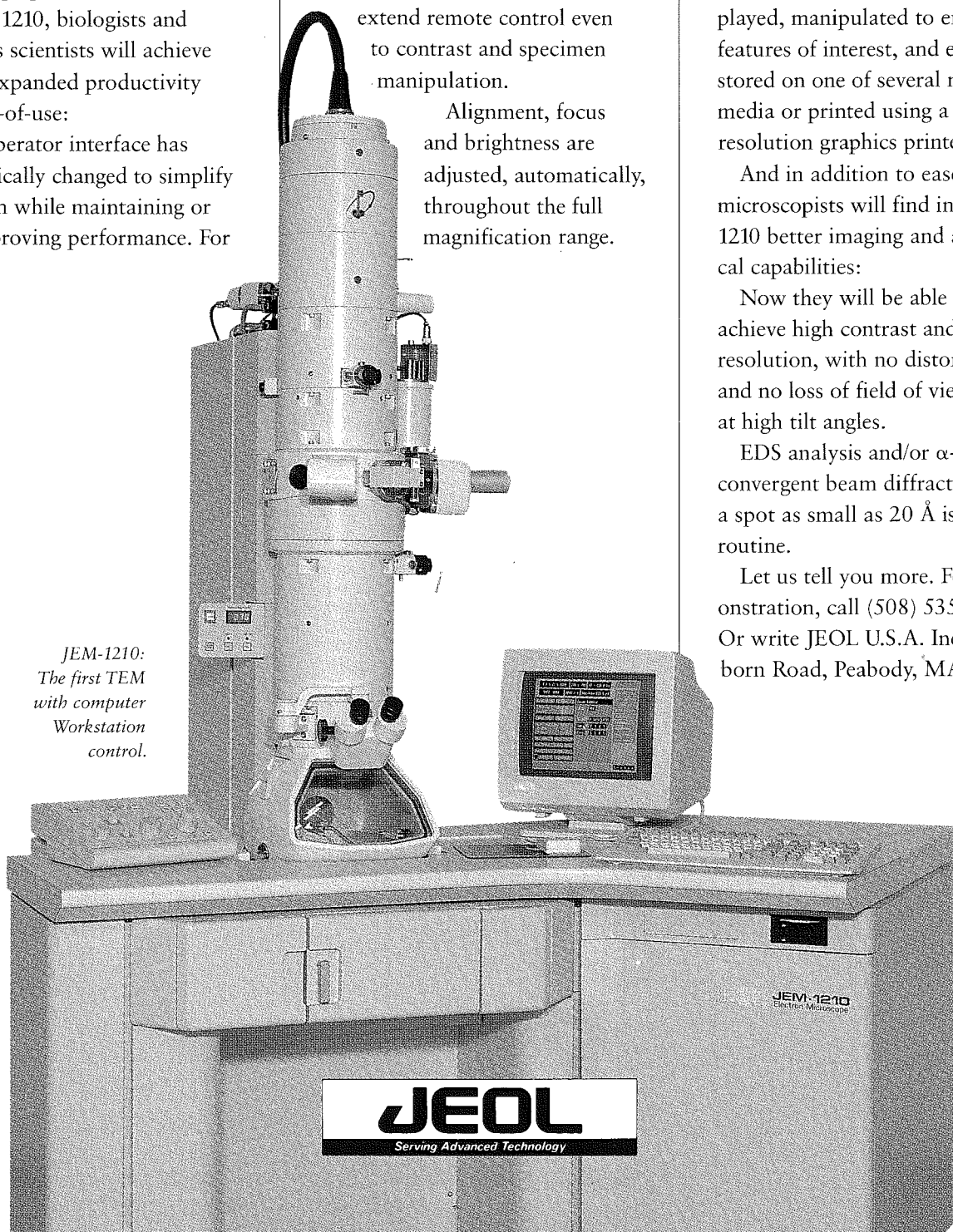
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FUNDAMENTAL AND COMPARATIVE STUDIES OF $M_{23}C_6$ CARBIDE GROWTH ASSOCIATED WITH TWIN BOUNDARIES AND GRAIN BOUNDARIES IN 304 STAINLESS STEEL, ROBERTA J. ROMERO AND L. E. MURR, Department of Metallurgical and Materials Engineering, The University of 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 on 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: $\gamma_{gb} \approx 800 \text{ mJ/m}^2$, $\gamma_{TB} \approx 200 \text{ mJ/m}^2$, $\gamma_{TB} \approx 20 \text{ mJ/m}^2$. 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 ($< 1 \mu\text{m}$) 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 (Norris, 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.

Norris, 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

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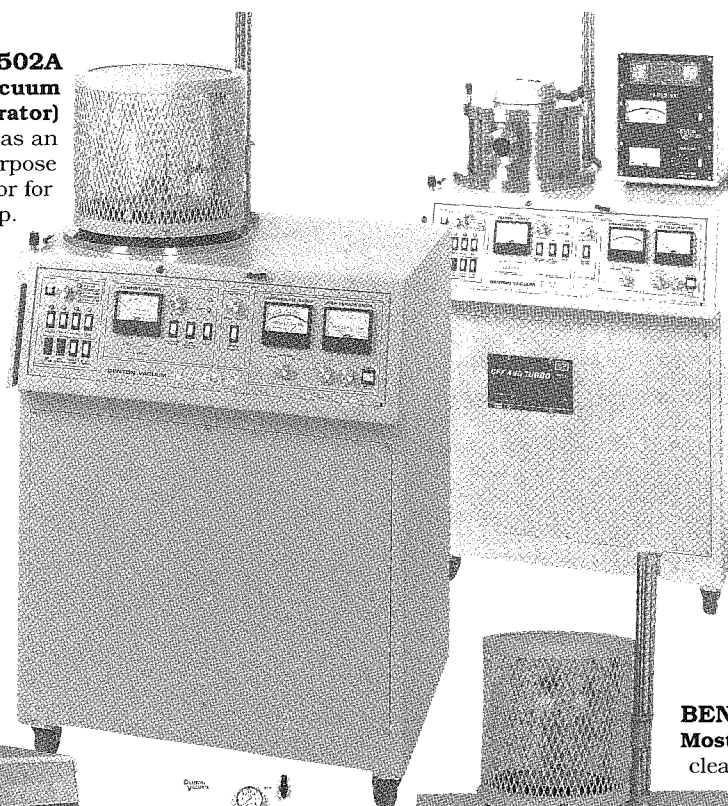
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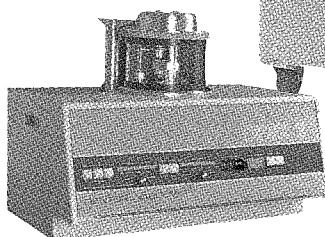
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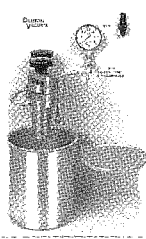
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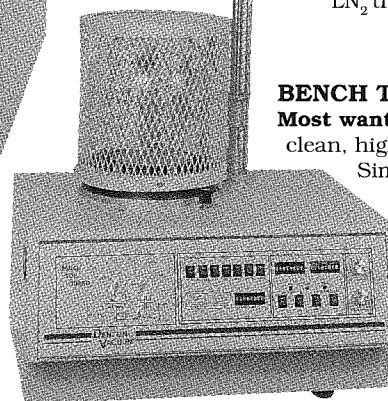
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DYNAMIC RECRYSTALLIZATION IN BALLISTIC AND HYPERVELOCITY PENETRATION AND CRATER FORMATION IN METALS, JESUS M. RIVAS, STELLA QUINONES, AND L. E. MURR, Department of Metallurgical and Materials Engineering, The University of Texas at El Paso, El Paso,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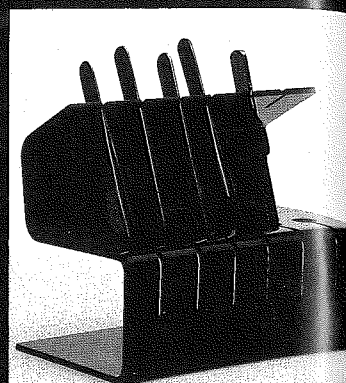
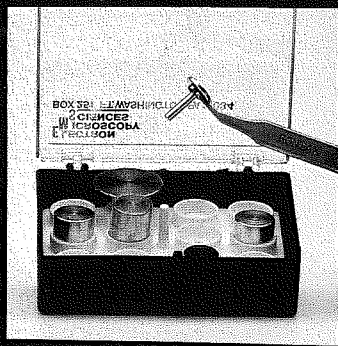
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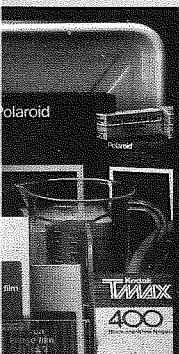
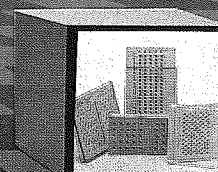
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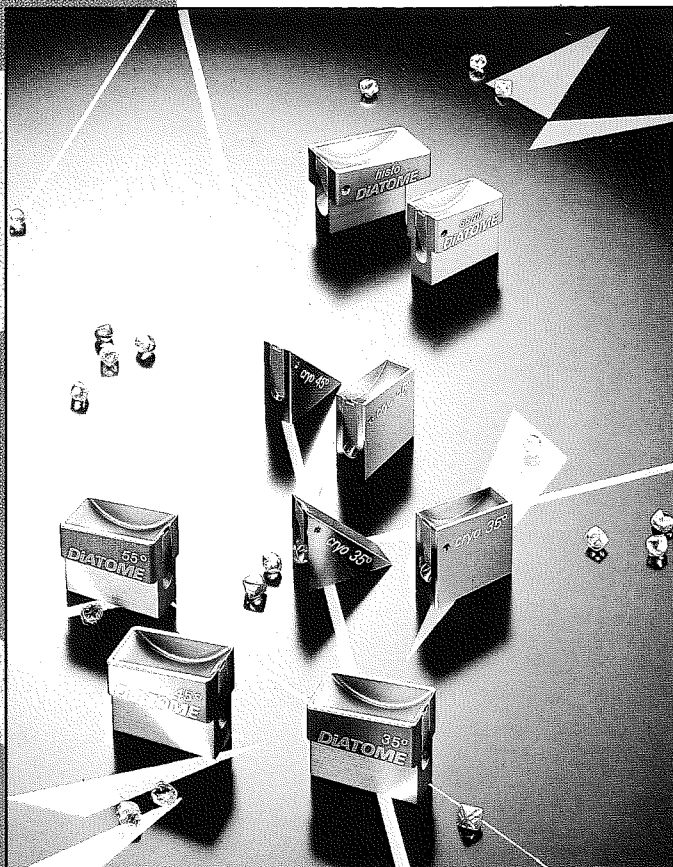
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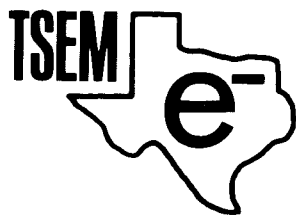
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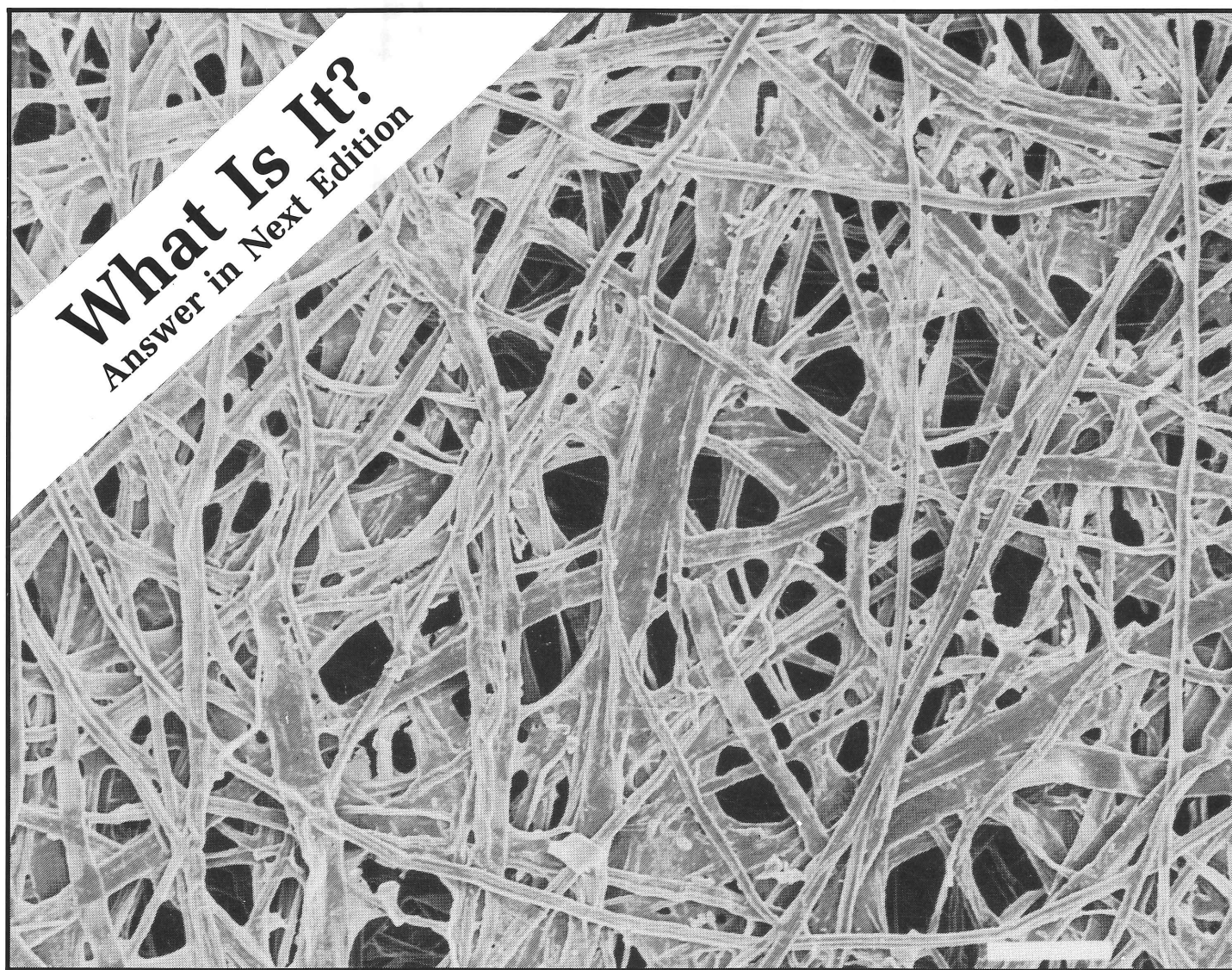


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