Volume 22, Number 5

October 2016

Microscopy AND Microsanalysis



CAMBRIDGE UNIVERSITY PRESS ISSN 1431-9276



"NEW" trimtool 90

Many requests from customers doing FIB cutting of biological and technical sample blocks have motivated us to relaunch the trim 90 blade: With the trim 90 blade the surface of the blocks as well as the 90° inclined block sides may be trimmed for the following FIB processing.

Please contact us for more information.

Over 40 years of development, manufacturing, and customer service

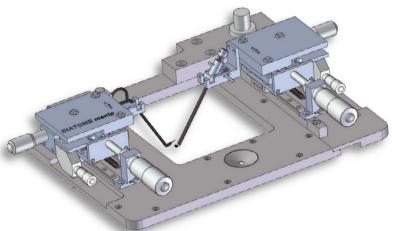
DiATOME U.S.

P.O. Box 550 • 1560 Industry Rd. • Hatfield, Pa 19440 Tel: (215) 412-8390 • Fax: (215) 412-8450 email: sgkcck@aol.com • www.emsdiasum.com

DIATOME diamond knives

Incomparable...

...and still innovating



NEW DIATOME manip

For easy handling and mounting of section ribbons.

The mounting of the manipulator is easy. The original plate on the cryochamber is removed. The manipulator is fixed in 5 minutes.

Applications

- Frozen hydrated biological samples (CEMOVIS)
- Room temperature sectioning of water sensitive samples Dry resin sectioning of biological samples for chemical analysis Dry sectioning of industrial samples such as polymers

Functionality

The left manipulator moves a conductive fibre on which the sections are attached by electrostatic force; the growing ribbon is guided.

The right manipulator guides the grid very precisely below the section ribbon, the sections are attached to the carbon film with electrostatic force.

Compatability

Leica cryochambers FC6 and FC7 equipped with a Crion ionizer/charger adaption on other cryochambers upon request

A section ribbon attached to a fiber is depicted emerging from a diamond knife edge (left) beneath the ribbon the grid attached to a holder touches the section ribbon.



Microscopy and Microanalysis

An International Journal for the Biological and Physical Sciences

THE OFFICIAL JOURNAL OF

MICROSCOPY SOCIETY OF AMERICA MICROANALYSIS SOCIETY MICROSCOPICAL SOCIETY OF CANADA / SOCIÉTÉ DE MICROSCOPIE DU CANADA MEXICAN MICROSCOPY SOCIETY

BRAZILIAN SOCIETY FOR MICROSCOPY AND MICROANALYSIS VENEZUELAN SOCIETY OF ELECTRON MICROSCOPY

EUROPEAN MICROBEAM ANALYSIS SOCIETY

AUSTRALIAN MICROSCOPY AND MICROANALYSIS SOCIETY

PORTUGUESE SOCIETY FOR MICROSCOPY

PUBLISHED IN AFFILIATION WITH

ROYAL MICROSCOPICAL SOCIETY
GERMAN SOCIETY FOR ELECTRON MICROSCOPY
BELGIAN SOCIETY FOR MICROSCOPY
MICROSCOPY SOCIETY OF SOUTHERN AFRICA

Editor in Chief

Robert L. Price Cell and Developmental Biology and Anatomy University of South Carolina Columbia, SC 29209 e-mail: Bob.Price@uscmed.sc.edu

Associate Editor-in-Chief

John Mansfield Michigan Center for Materials Characterization University of Michigan Ann Arbor, MI 48109-2800 e-mail: jfmjfm@umich.edu

Administrative Editor

John Shields University of Georgia Athens, GA 30602 e-mail: jpshield@uga.edu

Biological Sciences Applications Editors

W. Gray (Jay) Jerome
Department of Pathology, Microbiology and
Immunology
U-2206 MCN
Nashville, TN 37232-2561
e-mail: jay.jerome@vanderbilt.edu

Heide Schatten Department of Veterinary Pathobiology University of Missouri-Columbia Columbia, MO 65211 e-mail: SchattenH@missouri.edu

Rosemary White CSIRO Plant Industry Canberra, ACT 2601, Australia e-mail: Rosemary.white@csiro.au Elizabeth Wright Department of Pediatrics School of Medicine Emory University Atlanta, GA 30322 e-mail: erwrigh@emory.edu

Materials Sciences Applications Editors

Vinayak Dravid Materials Science and Engineering Northwestern University Evanston, IL 60208-3105 e-mail: v-dravid@nortwestern.edu

Georg E. Fantner Interfaculty Institute for Bioengineering École Polytechnique Fédéral de Lausanne Lausanne, 1015 Switzerland. e-mail: georg.fantner@epfl.ch

Brian Gorman Metallurgical and Materials Engineering Colorado Center for Advanced Ceramics Colorado School of Mines Golden, CO 80401 e-mail: bgorman@mines.edu

David J. Larson CAMECA 5500 Nobel Drive Madison, WI 53711 e-mail: david.larson@ametek.com

Ross Marceau Institute for Frontier Materials Deakin University Geelong, VIC 3216, Australia e-mail: r.marceau@deakin.edu.au Yoosuf N. Picard Materials Science & Engineering Carnegie Mellon University Pittsburgh, PA 15213 e-mail: ypicard@cmu.edu

Masashi Watanabe Dept. of Mater. Sci. & Eng. Lehigh University Bethlehem, PA 18015 e-mail: masashi.watanabe@lehigh.edu

Special Issues and Reviews Editor

David J. Smith Department of Physics Arizona State University Tempe, AZ 85287-1504 e-mail: david.smith@asu.edu

Book Review Editor

Cynthia Goldsmith Centers for Disease Control Atlanta, GA 30333 e-mail: csg1@cdc.gov

M&M Program Guide Editor

Richard L. Martens 1013 Bevill Building Box 870164 Tuscaloosa, AL 35487-0164 e-mail: rmartens@caf.ua.edu

Proceedings Editor

Gail Celio University of Minnesota St. Paul, MN 55108 e-mail: celio001@umn.edu

Editorial Board

Ralph Albrecht
University of Wisconsin, Madison, Wisconsin
Ilke Arslan
Pacific Northwest Laboratory, Richland, Washington
Barry Carter
University of Connecticut, Storrs, Connecticut
Wah Chiu
Baylor College of Medicine, Houston, Texas

Marc De Graef Carnegie Mellon University, Pittsburgh, Pennsylvania
Niels de Jonge INM Institute for New Materials, Saarbrücken, Germany

Alberto Diaspro University of Genoa, Italy

Elizabeth Dickey North Carolina State University, Raleigh

Mark Ellisman University of California at San Diego, San Diego, California

Pratibha Gai University of York, United Kingdom

Marija Gajdardziska-Josifovska University of Wisconsin-Milwaukee, Milwaukee, Wisconsin

Paul Kotula Sandia National Labs, Albuquerque, New Mexico

William Landis University of Akron, Akron, Ohio

Charles Lyman Lehigh University, Bethlehem, Pennsylvania

Dale Newbury National Institute of Standards and Technology, Gaithersburg, Maryland

Jean-Paul Revel California Institute of Technology, Pasadena, California

David SmithArizona State University, Tempe, ArizonaNan YaoPrinceton University, Princeton, New JerseyNestor ZaluzecArgonne National Laboratory, Argonne, Illinois

Editorial Board Representatives from Affiliated Societies

Masashi Watanabe Lehigh University (MAS)

Gautam Kumar Dey Bhabha Atomic Research Centre (EMSI)

Gema Gonzalez Venezuelan Institute for Scientific Investigation (Venezuela)

Michael Robertson Acadia University, Wolfville, Nova Scotia (Canada)

Brendan Griffin University of Western Australia (AMMS)

Guillermo Solorzano Pontificia Universidade Catolica, Rio de Janeiro (Brazil)

Mike Matthews Atomic Weapons Establishment, Reading, Great Britain (EMAS)

Miguel Yacaman Mexico Institute for Nuclear Research (Mexico)

Henrique Almeida Universidade do Porto (Portugal)

Founding Editor

Jean-Paul Revel California Institute of Technology, Pasadena, California

Previous Editors-in-Chief

Dale Johnson University of South Florida, Tampa, Florida Charles Lyman Lehigh University, Bethlehem, Pennsylvania

This journal is part of the **Cambridge Journals Online** service. Access to online tables of contents and article abstracts is available to all researchers at no cost. Access to full-text articles online is provided to those with online subscription. Online subscriptions must be activated. Once your subscription is activated, free access to past, present, and forthcoming articles is available at:

Microscopy and Microanalysis website: journals.cambridge.org/MAM.

Instructions for authors submitting manuscripts may be found at journals.cambridge.org/MAM. Select "Further Information" then select "Instructions for Contributors." An abbreviated version of these instructions will be published in the first issue (February) of each volume.



Octane Elite Silicon Drift Detectors **Elite Technology for Elite Results**

- Light element sensitivity increased up to 60% using new Si₃N₄ window
- Highest throughput SDD on the market
- Unparalleled resolution stability
- Highly reliable and moisture tolerant
- Safe for plasma cleaning
- Octane Elite Plus and Octane Elite Super models now available















Microscopy AND Microanalysis

Microscopy and Microanalysis publishes original research papers dealing with a broad range of topics in microscopy and microanalysis. These include articles describing new techniques or instrumentation and their applications, as well as papers in which established methods of microscopy or microanalysis are applied to important problems in the fields of biology or materials science. Microscopy and microanalysis are defined here in a broad sense, and include all current and developing approaches to the imaging and analysis of microstructure. The criteria for acceptance of manuscripts are the originality and significance of the research, the quality of the microscopy or microanalysis involved, and the interest for our readership.

Four types of communications are published in the Journal. **Regular Articles** are of substantial length and describe the findings of an original research project that satisfies the aims and scope of the Journal, described above. **Review Articles** summarize the current status of an important area within the aims and scope of the Journal. **Letters to the Editor** usually contain comments on recent articles that have appeared in the Journal. **Book Reviews** are also published, but these are solicited only through the Book Review Editor.

Instructions for Contributors

Instructions for authors contributing manuscripts may be found at http://mc.manuscriptcentral.com/mam under "Resources: Instructions and Forms." Authors may also visit http://www.journals.cambridge.org/jid_MAM, select "Further Information," and then select "Instructions for Contributors." An abbreviated version of these instructions will be published in the first issue (February) of each volume.

Copyright Information

Submission of a manuscript implies: that the work described has not been published before (except in the form of an abstract or as part of a published lecture, review, or thesis); that it is not under consideration for publication elsewhere; that its publication has been approved by all coauthors, if any, as well as by the responsible authorities at the institute where the work has been carried out; that, if and when the manuscript is accepted for publication, the authors agree to automatic transfer of the copyright to the Microscopy Society of America; that the manuscript will not be published elsewhere in any language without the consent of the copyright holders; and that written permission of the copyrighted sources.

All articles published in this journal are protected by copyright, which covers the exclusive rights to reproduce and distribute the article (e.g., as offprints), as well as all translation rights. No material published in this journal may be reproduced photographically or stored on microfilm, in electronic data bases, video disks, etc., without first obtaining written permission from the publisher.

The use of general descriptive names, trade names, trademarks, etc., in this publication, even if not specifically identified, does not imply that these names lack protection by the relevant laws and regulation.

Authorization to photocopy items for internal or personal use, or the internal or personal use of specific clients, is granted by Cambridge University Press, provided that the appropriate fee is paid directly to Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, USA (Tel: (508) 750-8400), stating the ISSN (1431-9276), the volume, and the first and last page numbers of each article copied. The copyright owner's consent does not include copying for general distribution, promotion, new works, or resale. In these cases, specific written permission must first be obtained from the publisher.

Disclaimer

The Microscopy Society of America, the other societies stated, and Cambridge University Press cannot be held responsible for errors or for any consequences arising from the use of the information contained in this journal. The appearance of scientific reports and/or workshops, or any other material in *Microscopy and Microanalysis* does not constitute an endorsement or approval by The Microscopy Society of America of the findings, data, conclusions, recommendations, procedures, results, or any other aspect of the content of such articles. The appearance of advertising in *Microscopy and Microanalysis* does not constitute an

endorsement or approval by The Microscopy Society of America of the quality or value of the products advertised or any of the claims, data, conclusions, recommendations, procedures, results, or any other information included in the advertisements.

While the advice and information in this journal is believed to be true and accurate at the date of its going to press, neither the authors, the editors, nor the publisher can accept any legal responsibility for any errors or omissions that may be made.

Subscription Information

Microscopy and Microanalysis is published bimonthly in February, April, June, August, October, and December by Cambridge University Press. Three supplements (Meeting Guide, Program Guide, and Proceedings) are published in June and August.

Society Rates: Members of the Microscopy Society of America should contact the MSA Business Office for all subscription inquiries: Microscopy Society of America, Hachero Hill, Inc., 11260 Roger Bacon Drive, Suite 402, Reston, VA 20190, Tel.: (703) 964-1240, Ext. 14, E-mail: nicoleguy@mindspring.com, URL: www.msa.microscopy.org. Members of other affiliated societies should contact their respective society business offices for all subscription inquiries.

Subscription Rates: Institutions print and electronic: US \$1876.00 in the USA, Canada, and Mexico; UK £1128.00 + VAT elsewhere. Institutions online only: US \$1327.00 in the USA, Canada, and Mexico; UK £803.00 + VAT elsewhere. Individuals print plus online: US \$548.00 in the USA, Canada, and Mexico; UK £333.00 + VAT elsewhere. Prices include postage and insurance.

USA, Canada, and Mexico: Subscribers in the USA, Canada, and Mexico should send their orders, with payment in US dollars or the equivalent value in Canadian dollars, to: Cambridge University Press, Customer Services Department (Journals), 1 Liberty Plaza, New York, NY 10006, USA. Tel: (845) 353-7500. Fax: (845) 353-4141. Orders may be phoned direct (toll free): (800) 872-7423. E-mail: journals_subscriptions@cup.org.

Outside North America: Subscribers elsewhere should send their orders, with payment in sterling, to: Customer Services Department (Journals), Cambridge University Press, The Edinburgh Building, Shaftesbury Road, Cambridge, CB2 8RU, UK. Tel: +44 (0)1223 326070. Fax: 44 (0)1223 325150. E-mail: journals@cambridge.org

Change of address: Allow six weeks for all changes to become effective. All communications should include both old and new addresses (with postal codes) and should be accompanied by a mailing label from a recent issue. Society members should contact their respective society business offices to inform them of address changes.

Editorial Office

Robert L. Price, Editor in Chief, Department of Cell and Developmental Biology and Anatomy, School of Medicine, University of South Carolina, 6439 Garner's Ferry Road, Bldg. 1 B-60, Columbia, SC 29209, USA; Tel: (803) 216-3824; Fax: (803) 733-3212; E-mail: Bob.Price@uscmed.sc.edu.

Office of Publication

Cambridge University Press, 1 Liberty Plaza, New York, NY 10006, USA; Tel: (212) 337-5000; Fax: (212) 337-5959.

Advertising Sales & Production

Kelly Miller, M.J. Mrvica Associates, Inc., 2 West Taunton Avenue, Berlin, NJ 08009, USA; Tel: (856) 768-9360; Fax: (856) 753-0064.

© 2016 by Microscopy Society of America. Printed in the United States on acid-free paper. Periodicals postage paid at New York, NY, and additional mailing offices. Return postage guaranteed. Postmaster: Send address changes in the U.S.A. and Canada to *Microscopy and Microanalysis*, Subscription Department, Cambridge University Press, 1 Liberty Plaza, New York, NY 10006.



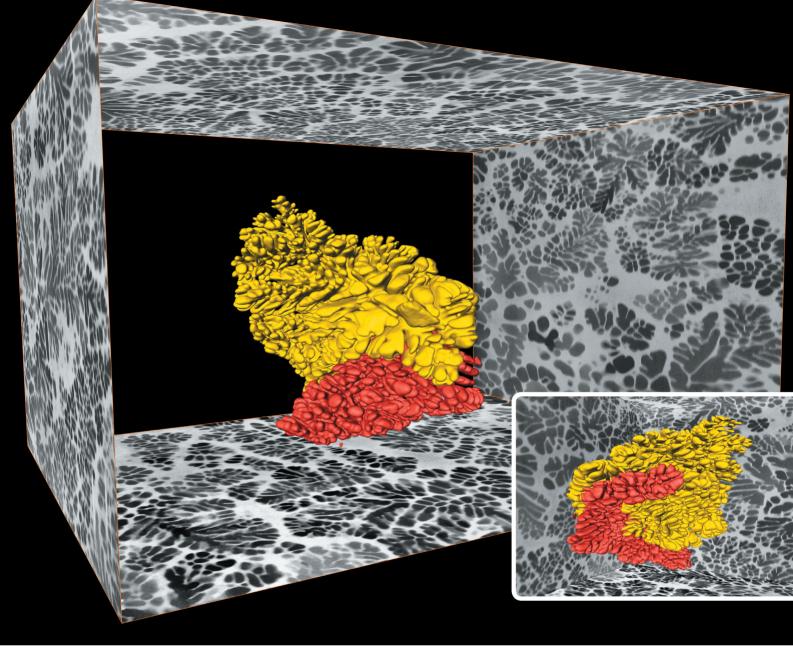
TESCAN TriglavTM

New SEM column technology for the new UHR models 2016



Find out more about this new cutting-edge technology at

www.tescan-usa.com/triglav



FEI Avizo® 3D visualization of two large adjacent crystalline dendrites of a bulk-metallic-glass matrix composite $(Zr_{sas}^2Ti_{14.3}Nb_{s.2}Cu_{s.1}Ni_{4.9}Be_{n.0})$. Data was obtained by large volume serial sectioning tomography using the Helios PFIB DualBeam. The sectioned block is about $90\times80\times70~\mu\text{m}^3$. Sample from The University of Tennessee, USA. Images courtesy of The University of Manchester.

Large 3D volumes with unprecedented surface resolution

Until recently, available technologies have limited the volumes and depths of materials that can be analyzed at high resolution, ultimately restricting the insight into structural, crystallographic, and chemical properties. This is no longer the case. The Helios™ PFIB DualBeam™ offers unrivaled access to regions of interest deep below the surface—combining serial section tomography with statistically relevant data analysis.



Discover more at FEI.com/Helios-PFIB

Microscopy and Microanalysis

An International Journal for the Biological and Physical Sciences

Volume 22, Number 5
October 2016

Instrumentation and Techniques Development Novel Polarizing Method for Light Microscopy 933 Irina G. Palchikova, Evgenii S. Smirnov, and Natalia V. Kamanina Removing Beam Current Artifacts in Helium Ion Microscopy: A Comparison of **Image Processing Techniques** 939 Anders J. Barlow, Jose F. Portoles, Naoko Sano, and Peter J. Cumpson Annular Focused Electron/Ion Beams for Combining High Spatial Resolution with 948 **High Probe Current** Anjam Khursheed and Wei Kean Ang Thin-Film Phase Plates for Transmission Electron Microscopy Fabricated from 955 Manuel Dries, Simon Hettler, Tina Schulze, Winfried Send, Erich Müller, Reinhard Schneider, Dagmar Gerthsen, Yuansu Luo and Konrad Samwer Polyallylamine as an Adhesion Promoter for SU-8 Photoresist 964 Shiladitya Chatterjee, George H. Major, Barry M. Lunt, Massoud Kaykhaii and Matthew R. Linford Why Do We Need to Use Three-Dimensional (3D) Fourier Transform (FT) Analysis to Evaluate a High-Performance Transmission Electron Microscope (TEM)? 971 Kazuo Ishizuka and Koji Kimoto Decontamination in the Electron Probe Microanalysis with a Peltier-Cooled 981 Ben Buse, Stuart Kearns, Charles Clapham and Donovan Hawley

On the Cover: X-ray fluorescence map of human lung exposed to asbestos. For further information see Pascolo et al., pp 1062–1071.

Characterization of Amorphous Oxide Nano-Thick Layers on 316L Stainless Steel by Electron Channeling Contrast Imaging and Electron Backscatter Diffraction 997 Mahrokh Dorri, Stéphane Turgeon, Nicolas Brodusch, Maxime Cloutier, Pascale Chevallier, Raynald Gauvin and Diego Mantovani On the Chemical Signature and Origin of Dicoppertrihydroxyformate (Cu₂(OH)₃HCOO) Formed on Copper Miniatures of 17th and 18th Centuries

987

Identification of Nanocrystalline Inclusions in the Bismuth-Doped Silica Fibers

Liudmila D. Iskhakova, Filipp O. Milovich, Valery M. Mashinsky, Alexander S. Zlenko,

BIOLOGICAL APPLICATIONS

MATERIALS APPLICATIONS

and Preforms

and Jorge Ginja Teixeira

Cytotoxicity of Experimental Resin Composites on Mesenchymal Stem Cells Isolated from Two Oral Sources

Alexandra Roman, Emöke Páll, Mărioara Moldovan, Darian Rusu, Olga Şoriţău, Dana Feştilă and Mihaela Lupşe

Microscopy and Microanalysis website: http://www.journals.cambridge.org/MAM Indexed in Chemical Abstracts, Current Contents, BIOSIS, and MEDLINE (PubMed)

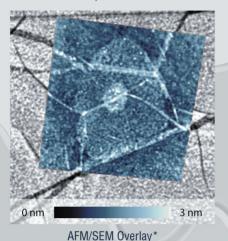
Alfredina Veiga, Dora Martins Teixeira, António J. Candeias, José Mirão, Paulo Simões Rodrigues

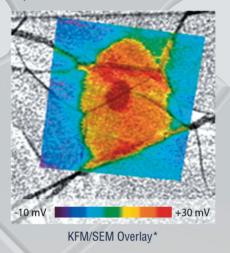
Contents continued

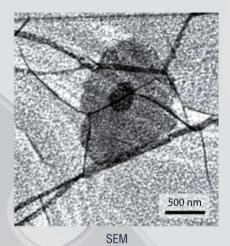
Zinc-Containing Restorations Create Amorphous Biogenic Apatite at the Carious Dentin Interface: A X-Ray Diffraction (XRD) Crystal Lattice Analysis Manuel Toledano, Fátima S. Aguilera, Modesto T. López-López, Estrella Osorio, Manuel Toledano-Osorio and Raquel Osorio	1034
Aberration-Corrected Transmission Electron Microscopic Study of the Central Dark Line Defect in Human Tooth Enamel Crystals José Reyes-Gasga, Joseph Hémmerlé and Etienne F. Brès	1047
Electron Microscopy Findings in N-Methyl-N-Nitrosourea-Induced Mammary Tumors Ana I. Faustino-Rocha, Ana M. Calado, Adelina Gama, Rita Ferreira, Mário Ginja and Paula A. Oliveira	1056
Focused X-Ray Histological Analyses to Reveal Asbestos Fibers and Bodies in Lungs and Pleura of Asbestos Exposed Subjects Lorella Pascolo, Alessandra Gianoncelli, Clara Rizzardi, Martin de Jonge, Daryl Howard, David Paterson, Francesca Cammisuli, Murielle Salomé, Paolo De Paoli, Mauro Melato and Vincenzo Canzonieri	1062
Polarization Second Harmonic Generation Discriminates Between Fresh and Aged Starch-Based Adhesives Used in Cultural Heritage Sotiris Psilodimitrakopoulos, Evaggelia Gavgiotaki, Kristallia Melessanaki, Vassilis Tsafas and George Filippidis	1072
Spectroscopic and Microscopic Study of Peroxyformic Pulping of Agave Waste Hilda M. Hernández-Hernández, Jorge J. Chanona-Pérez, Alberto Vega, Pablo Ligero, Reynold R. Farrera-Rebollo, Jorge A. Mendoza-Pérez, Georgina Calderón-Domínguez and Norma Güemes Vera	1084
Diplopods as Soil Bioindicators of Toxicity After Application of Residues From Sewage Treatment Plants and Ethanol Industry Cintya A. Christofoletti, Annelise Francisco, Janaína Pedro-Escher, Vinícius D. Gastaldi and Carmem S. Fontanetti	1098
BOOK REVIEW	
Correlative Light and Electron Microscopy II Nadine Soplop	1111

HITACHI Inspire the Next

AFM/SEM Comparative Measurements of Graphene







*Overlay images created by using AZblend (ASTRON, Inc.).

New Release AFM5500M

Automation

- Automated sample navigation via 4-inch motorized, fully addressable, and programmable stage
- Automated cantilever loading and laser alignment
- Automated and self-optimizing data acquisition

Accuracy

- Piezo-flexure-based scanner design rendering flat and orthogonal scans within the full 200-µm range
- High-resolution and accurate imaging via closed-loop scanner with low sensor noise

Correlative Imaging

- Hitachi-proprietary AFM/SEM shared alignment sample holder
- Sample location preserved for true correlative AFM/SEM measurements

Learn more about the AFM5500M and correlative AFM/SEM systems at: http://www.hitachi-hightech.com/us/afm





Think Outside the Lab

Visit Hitachi at 2016 MRS Fall Meeting Booth #1117, Nov. 29-Dec. 2, Boston, MA

NO COMPROMISE

ATOMIC RESOLUTION + FAST ANALYSIS



www.jeolusa.com salesinfo@jeol.com • 978-535-5900

Learn more at www.jeolusa.com/F2

FUSION



FIND OUT MORE:

www.protochips.com

Toll-Free Phone (USA/Canada): 844.INSITU1 (844.467.4881) International Phone: 001.919.377.0800 E-mail: contact@protochips.com



Expanding the imaging range with only one microscope.

ZEISS LSM 800



// PRECISION

MADE BY ZEISS

Your versatile confocal microscope for materials research and failure analysis

Expand your imaging range. ZEISS LSM 800 is the one instrument you will need for materials analysis. Characterize 3D surfaces precisely. With no need to change microscopes, you'll save time on set-up. Combine light microscopical and confocal imaging for your 3D analyses.

ZEISS

www.zeiss.com/lsm800-mat