

# Minnesota Microscopy Society

Local affiliate of the *Microscopy Society of America*  
and the *Microbeam Analysis Society*



Newsletter

April 2010



FOCUS  
ON  
SCIENCE

Minnesota Microscopy Society  
Spring Symposium

Date: Friday, May 7, 2010

Location: Science Museum of Minnesota  
120 W. Kellogg Blvd., St. Paul  
Discovery Hall  
([www.sci.mus.mn.us](http://www.sci.mus.mn.us))

## Schedule:

|                  |   |
|------------------|---|
| 7:30 - 8:15 AM   | Registration, Continental Breakfast, and Vendor Displays  |
| 8:15 - 8:30 AM   | Welcome   |
| 8:30 - 9:30 AM   | Paul Kotula<br><i>Microanalysis and the FBI's Amerithrax Investigation of the 2001 Anthrax Attacks</i>  |
| 9:30 - 10:30 AM  | Sarah Cousineau<br><i>A Hitchhiker's Guide to Accelerators</i>  |
| 10:30 - 11:00 AM | Break and Vendor Displays   |
| 11:00 - 12:00 PM | Lucille Giannuzzi<br><i>FIB-based Techniques for Specimen Preparation, Materials Characterization, and Prototyping</i>  |
| 12:00 - 1:30 PM  | Lunch and Vendor Displays   |
| 1:30 - 1:45 PM   | Business Meeting  |
| 1:45 - 2:45 PM   | Andre Mkhoyan<br><i>Atomic and Electronic Structure of Graphene-Oxide Films Measured by STEM and Electron Energy Loss Spectroscopy</i>                        |
| 2:45 - 3:45 PM   | Scott Sitzman<br><i>Beyond Imaging and Chemistry: Characterizing the Microstructure of Materials using Electron BackScatter Diffraction (EBSD) in the SEM</i> |
| 3:45 - 4:00 PM   | Door Prizes, Closing Remarks  |

## Special Note to Attendees:

Due to the Dead Sea Scrolls exhibit at the Science Museum, added security measures are in place for museum patrons. *Please be prepared to have all bags, purses, and boxes checked upon entry.* Thank you for your patience and understanding.

**Registration:**

The cost of the meeting will be \$80 for MMS members\*\*, \$90 for nonmembers, and \$25 for students and K-12 teachers. This fee includes the meeting, buffet lunch, breakfast, coffee breaks, and a free pass to the Museum exhibits (a \$7 value). Registrants can pay at the door, but **reservations must be made no later than Wednesday, April 28th**. Register by e-mail (preferred) to Bede Willenbring at [reservations@mnmicroscopy.org](mailto:reservations@mnmicroscopy.org), or by phone at 651-236-5470. Include your name, company, phone number, and email address.

**Luncheon Buffet**

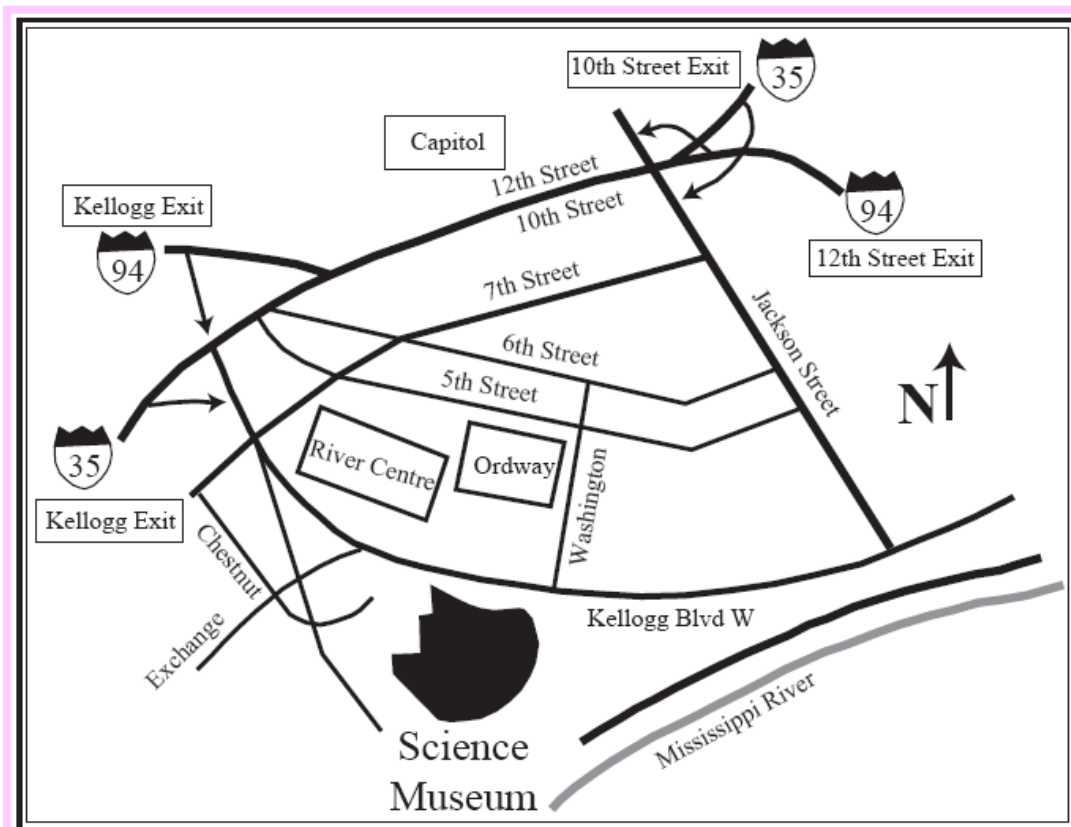
- Platters of cold roast beef, roast turkey, and smoked ham.
- Slices of swiss, cheddar, and pepper jack cheeses.
- Fresh lettuce, sliced tomatoes, onions, and pickles.
- Freshly baked bread, rolls and petite croissants.
- Country potato salad, penne pasta salad primavera, and kettle chips.
- Fresh sliced fruits of the season.
- Chocolate Torte.

**\*\*Unemployed MMS member?  
Please contact Bede for information  
on reduced registration fees.**

**Location of the Science Museum,  
Parking, and Meeting Room**

The Science Museum is located at 120 W. Kellogg Blvd., St. Paul. The meeting will be held in Discovery Hall. Enter the Museum from Kellogg Boulevard or via the hallway from the parking ramp. After passing through Security, go through the Lobby, angle left just after the Box Office, and continue to the stairs/elevators. Discovery Hall is one floor down.

The Science Museum’s parking ramp can be accessed from either Kellogg Boulevard or Chestnut Street. Enter the museum by taking the parking ramp elevator to the Lobby level. The River Centre ramp is an alternative to the Science Museum’s ramp.



## MMS Spring Symposium 2010 – Speakers



### *Microanalysis and the FBI's Amerithrax Investigation of the 2001 Anthrax Attacks*

Paul Kotula,  
Sandia National Laboratories

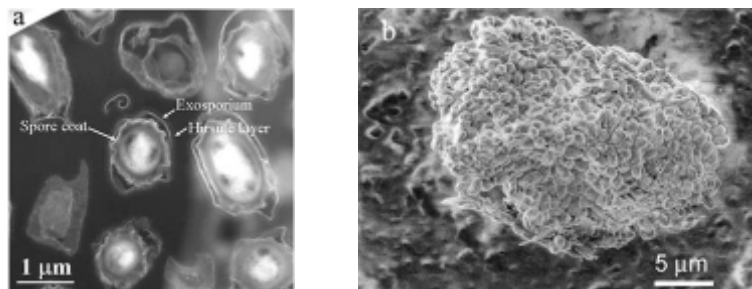


Figure1. a. Annular dark-field STEM image of a fixed and stained, microtomed section of *B. Anthracis* from the New York Post letter. b. SEM image of a clump of *B. Anthracis* spores from the Leahy letter.

#### Biography

Paul Kotula is a Principal Member of Technical Staff in the Materials Characterization Department at Sandia National Laboratories in Albuquerque, NM. Paul received his B.S. from Cornell University and Ph.D. from the University of Minnesota, both in Materials Science and Engineering. Before joining Sandia, he was a Postdoctoral Fellow at Los Alamos National Laboratory. His work at Sandia includes analytical electron microscopy support for microelectronic and micro-electromechanical device development, welding, brazing, soldering, forensics, process feedback, failure analysis, and 3D materials characterization and microanalysis. He has helped build a research program on spectral imaging and automated multivariate statistical analysis. The software developed from this work for x-ray microanalysis is commercially available from Thermo Fisher Scientific and is now in over 500-labs worldwide. It is also in research-form in over 25-labs worldwide. Paul's work has also garnered several awards over the years, among them an R&D 100 Award in 2002, two Best Analytical Techniques paper of the year in the journal *Microscopy and Microanalysis* (2003, 2006) and the Heinrich Award for outstanding young scientist from MAS in 2008.

Paul has been an Adjunct Professor in the Department of Materials Science and Engineering at North Carolina State University since 2001 and has authored or co-authored over 70 journal articles on a wide variety of topics involving electron microscopy and microanalysis as well as two patents and two book chapters. He was also a Director of MAS (2002-2004), a Tour Speaker (2003-2004) and President of the Society (2006-2007).

#### Abstract

The Anthrax attacks of 2001 in the US killed 5, sickened 22 others and caused a significant disruption of mail and other government facilities. Although the attack materials were for the most part recovered (*Bacillus Anthracis*) in powder form in sealed envelopes, the US Federal Bureau of Investigation (FBI) was unprepared to perform the needed forensic analyses on these bio-weapon materials. In particular, it was identified that microanalysis from the micro- to nano-scale was a key missing piece of their capabilities. As a result, Sandia was asked to analyze the materials from the attacks by early 2002 and we reached our general conclusions within a few months. We also analyzed over 200 samples of *B. anthracis* between 2002 and 2008 in an attempt to discern the method of manufacture of the attack materials.

This talk will describe Sandia's involvement in the FBI's investigation and in particular the power of microanalysis in answering several critical questions: Was the *Bacillus Anthracis* intentionally weaponized (i.e., contain an additive to make it disperse predictably) and were the materials from the attacks from the same source? In particular x-ray spectral imaging (in the SEM and STEM) combined with multivariate statistical analysis [1-3] were used to answer these questions. Specimen preparation was both by conventional microtomy and focused ion beam (FIB) sectioning of spore preparations. In addition, significant advances in analytical throughput were achieved by modification of a FE-SEM with an annular Si-drift detector with a solid angle of over 1 steradian. STEM in SEM was then performed with this new hybrid instrument in order to analyze large numbers of spores in a short time.

## MMS Spring Symposium 2010 – Speakers

### *A Hitchhiker's Guide to Accelerators*

Sarah Cousineau,  
Oak Ridge National Laboratory



#### **Biography**

Dr. Sarah Cousineau is an accelerator physicist at the Spallation Neutron Source accelerator at Oak Ridge National Laboratory. She attended graduate school at Indiana University with the support of a Women in Science Fellowship, and received her PhD in 2003. She joined the accelerator physics group at the Spallation Neutron Source in 2003, where she has been involved in the commissioning and power ramp up of the Spallation Neutron Source accelerator. Her research is focused on issues of beam transport, beam instabilities, and collective interactions in high intensity particle beams.

In addition to her research, she active in science outreach and education activities, for which she has received mentoring and teaching awards. She served as vice-chair of the Oak Ridge National Laboratory Committee for Women, and has recently been elected to the Executive Committee of the American Physical Society Division of Particles and Beams.

#### **Abstract**

Since their advent in the early part of the 20th century, particle accelerators have given rise to some of the most important scientific discoveries in history. Today, accelerators are used for a wide variety of applications, from the study of the fundamental constituents of matter, to medical therapy machines and industrial applications. In this talk we will review the basic principles of acceleration and how particle accelerators have evolved. We will discuss the technological challenges associated with accelerating particles to velocities near the speed of light, and will highlight a few of today's most prominent accelerators. Finally, we will end with a discussion of the world's highest intensity hadron accelerator, the Spallation Neutron Source at Oak Ridge National Laboratory.

## MMS Spring Symposium 2010 – Speakers

### *FIB-based Techniques for Specimen Preparation, Materials Characterization, and Prototyping*

Lucille Giannuzzi,  
FEI Company



#### **Biography**

Lucille A. Giannuzzi received her B.E. and M.S. from SUNY Stony Brook and her Ph.D. from The Pennsylvania State University. She joined the University of Central Florida in 1994, ending her career at UCF as Professor, Department of Mechanical Materials and Aerospace Engineering. She joined FEI Company in 2003 as a product marketing engineer for FIB/DualBeam where she continues to perform FIB-related research and product marketing. She is active in professional societies, is a Fellow of AVS, and has over 100 publications.

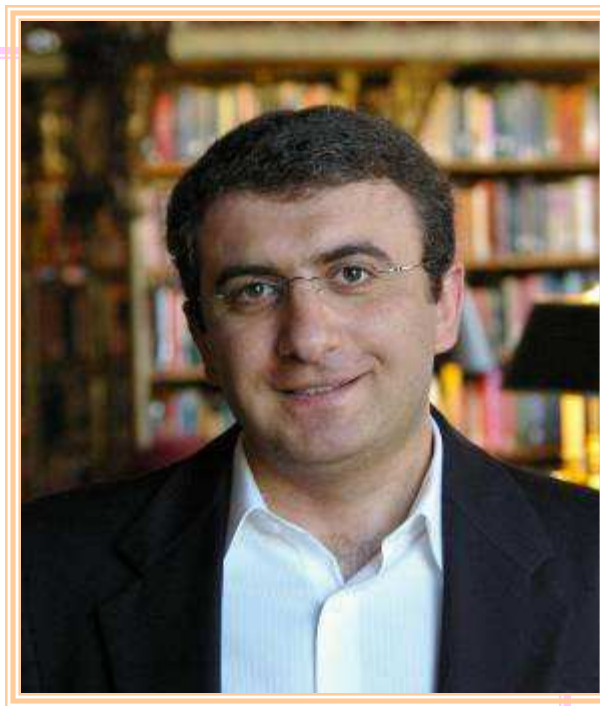
#### **Abstract**

Focused ion beam (FIB) based instruments are now routinely used for specimen preparation for a range of analytical instruments. DualBeam instruments (a FIB column and a scanning electron microscope (SEM) column on the same platform) can be used for unattended and automated site specific cross-sectioning or TEM specimen preparation. The SEM can be used for end-pointing any FIB technique via live or intermittent imaging. In addition, the synergistic use of the FIB with the SEM allows for automated acquisition of serial slices for subsequent 3D reconstruction and tomography of microstructure (via SEM imaging), crystallography (via EBSD) and/or elemental composition (via EDS). Automated and advanced digital patterning capabilities can be used to either remove or deposit material lithography or micro- and nanoprototyping. Examples of the methods and techniques mentioned above will be presented from a variety of materials.

## MMS Spring Symposium 2010 – Speakers

### *Atomic and Electronic Structure of Graphene-Oxide Films Measured by STEM and Electron Energy Loss Spectroscopy*

Andre Mkhoyan,  
University of Minnesota



#### **Biography**

K. Andre Mkhoyan was born in Yerevan, Armenia. In 1991 he graduated with honors from the Physics and Mathematics oriented high school in Yerevan and enrolled into Yerevan State University. There he received B.S. and M.S. in Physics in 1996 with honors specializing in Solid State Physics. From 1998 to 1999 he was working as a researcher at Bell Laboratories of Lucent Technologies in Murray Hill, NJ. He entered the Graduate School at Cornell University in a fall of 1999 in School of Applied and Engineering Physics and joined the research group of Prof. John Silcox. He received the M.S. in May 2003 and Ph.D. in January 2004, both in Applied Physics. From 2004 to 2008 he was Postdoctoral Research Associate at Cornell and Visiting Scientist at T.J. Watson Lab of IBM working with Dr. Phil Batson. He joined Department of Chemical Engineering and Materials Science in August of 2008 as Assistant professor.

#### **Abstract**

Thin sheets of graphene oxide (GO), which can be deposited over large areas, has recently emerged as a material alternative to graphene. The solubility of graphene oxide in water and other solvents allows it to be deposited onto wide ranging substrates uniformly in the form of thin films or networks which makes them useful for microelectronics. Graphene oxide is an insulator but controlled oxidation provides tunability of the electronic and mechanical properties including the possibility of accessing zero-band-gap graphene via complete removal of the C-O bonds. The structure of GO is often simplistically assumed to be a graphene sheet bonded to oxygen in the form of carboxyl, hydroxyl or epoxide groups. We elucidated the atomic and electronic structure of GO using composition sensitive annular dark field low and high resolution imaging of single and multilayer sheets and electron energy loss spectroscopy for measuring the fine structure of the carbon and oxygen K-edges as well as low-loss electronic excitations in a scanning transmission electron microscopy. The results reveal that the GO sheet are rough and the structure is predominantly amorphous due to distortions from the high fraction of sp<sup>3</sup> C-O bonds. Our results suggest that chemical removal of oxygen may leave behind a highly distorted reduced graphene oxide sheet which is likely to have substantially lower carrier mobilities than pure graphene.

## MMS Spring Symposium 2010 – Speakers



### *Beyond Imaging and Chemistry: Characterizing the Microstructure of Materials using Electron BackScatter Diffraction (EBSD) in the SEM*

Scott Sitzman,  
Oxford Instruments

#### **Biography**

Scott Sitzman is the lead Electron BackScatter Diffraction (EBSD) applications scientist in North America for Oxford Instruments. He holds a B.S. in geology from the University of California – Santa Barbara and a M.S. in geology with a minor in materials science from the University of Wisconsin – Madison, where his graduate work focused on TEM analysis of defect microstructures in a naturally occurring iron oxide (magnetite). Scott went on to work as an EBSD specialist at General Electric's Global Research Center in upstate New York, primarily analyzing aircraft engine titanium alloys, nickel-base superalloys, ceramics and metal-ceramic *in situ* composites. In 2002 he joined HKL Technology, Inc., supplier of the EBSD systems he used at GE, since acquired by Oxford Instruments. In addition to his applications work, he is involved in collaborative research efforts on nanowires and nanoparticles with a materials science group at UCLA.

#### **Abstract**

Electron BackScatter Diffraction (EBSD) is a powerful, versatile technique that significantly expands the analytical capabilities of SEMs for characterization of metals, ceramics and geological materials. The technique uses Kikuchi diffraction patterns generated from damage free, usually polished, surfaces of bulk samples using an SEM's focused electron probe. Patterns emanating from a surface are automatically analyzed for phase and crystallographic orientation by comparison with simulated patterns generated from potential match phases. In EBSD phase identification, elemental information collected by an integrated EDS system at points of interest are used to cull possible match phases from crystallographic phase databases, followed by indexing against those phases using simultaneously collected Kikuchi patterns, to arrive at solutions based on both chemistry and crystallography. In EBSD mapping, points over a designed grid are automatically visited by the electron beam, diffraction patterns collected and indexed, and (optionally) EDS data simultaneously collected. Modern EBSD systems are capable of collecting and indexing patterns at rates up to many hundreds of points per second, allowing characterization of relatively large sample areas at high resolutions, SEM conditions and materials dependent. Raw data thus collected may be analyzed for quantitative and visual analysis (via EBSD maps) of grain size and shape, phase distribution and area %, grain boundary position and character (e.g., low angle, high angle, and twin/special boundaries), crystallographic texture/preferred orientation, orientation relationships between phases, strain, and other modes of analysis. Point analysis of non-polished materials are also possible, for phase and orientation determinations in nanowires, nanostructures, particles and fracture surfaces. Recent advances in the technique include 3-D EBSD on FIB-SEMs, high speed data acquisition, high sensitivity strain analysis and ultra-high sensitivity topographic imaging using the forescatter electron detectors, which are used commonly used in conjunction with EBSD detectors.

**An Invitation from Paul Voyles, Associate Professor  
Materials Science and Engineering  
University of Wisconsin, Madison**

Dear Colleagues,

The University of Wisconsin, Madison Materials Science Center invites you to a series of events to celebrate the opening of its FEI Titan (S)TEM Laboratory. The Titan has a CEOS probe-side aberration corrector, which provides revolutionary performance in STEM imaging and microanalysis. The events are:

April 22-23: Workshop on EELS and EFTEM characterization using the Gatan Imaging Filter, presented by Dr. Ray Twesten, Gatan Product Manager for Analytical Instruments and Gatan EELS School instructor.

April 29th: Titan grand opening, featuring presentations on materials and biological applications of the Titan and a Materials Science Center open house with live Titan demo.

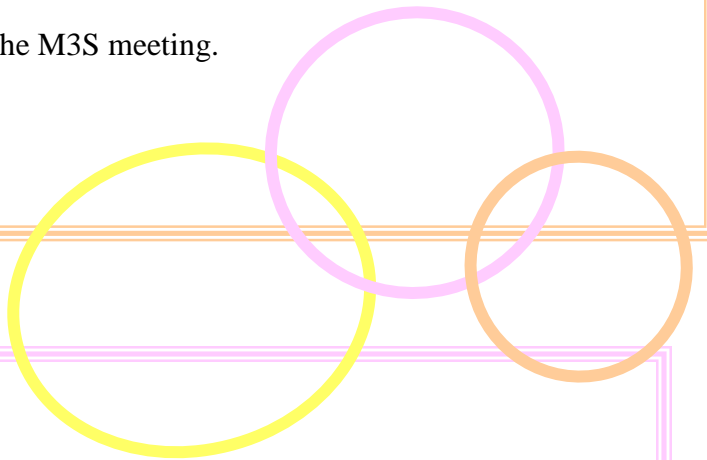
April 30th: Midwest Microscopy and Microanalysis Society meeting "Microscopy and Microanalysis for Materials and Biology", featuring a morning plenary session and parallel afternoon sessions on physical and biological sciences.

Limited opportunities for visitors to examine their samples in the Titan will also be available. All the events are on the UW Madison campus. The EELS workshop and Titan grand opening are free to attend. The M3S meeting is free for M3S members. For further information and event registration, see

<http://tem.msae.wisc.edu/STEM/announcement.htm> for the EELS workshop and Titan opening and

<http://www.midwestmicroscopy.org/meetings.htm> for the M3S meeting.

Sincerely,  
Paul Voyles



For those of you in the medical devices industry....

**"Design of Medical Devices Conference"**

*April 13-15, Radisson University Hotel, 615 Washington Ave. S.E., Minneapolis*

*Presented by the U of MN Academic Health Center and the Institute for Engineering in Medicine's Medical Devices Center, the world's premier medical devices conference consists of multiple scientific poster sessions and technical/scientific sessions. Early bird registration ends March 28.*

[Learn more and register](http://www.dmd.umn.edu/)

<http://www.dmd.umn.edu/>



## Honeywell Aerospace Tour

**2600 Ridgway Parkway, Minneapolis, MN 55413**

<http://www.honeywell.com>

April 22nd 2010

4:30 – 6:00 PM

Honeywell has been a major supplier for the aerospace industry for several decades. We will have the opportunity to have a closer look at its facility in Minneapolis and listen to the latest development in the industry, at least any that we are authorized to hear!

The tour will cover the manufacturing areas for ring laser gyroscopes and related systems. There will also be a short discussion of several areas of technology as they relate to the aerospace industry.

We need to provide in advance the names of the attendees. Please bring a photo ID and (if applicable) Green Card. We also need the country of origin of any foreign national so we can get access. Please send an e-mail to: [torij001@umn.edu](mailto:torij001@umn.edu) if you wish to join us in touring this interesting company. You do NOT need to be a member of AVS to attend.



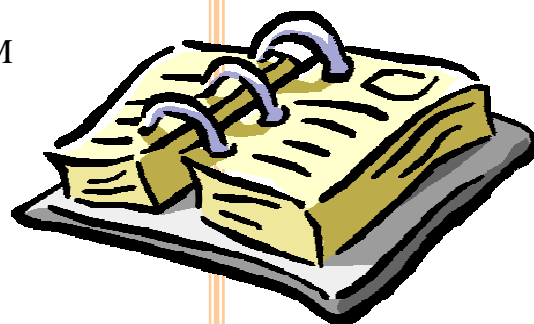
### Upcoming Events

**April 13-15** Design of Medical Devices Conference, U of M

**April 22** Honeywell Aerospace Tour, Minneapolis

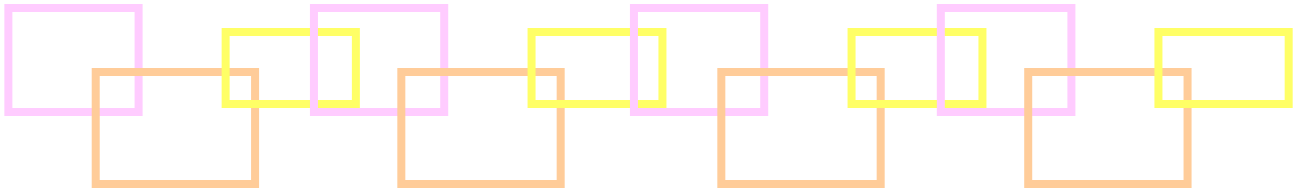
**May 7** MMS Spring Symposium, Science Museum

**May 19** JEOL /Thermo Users meeting at Medtronic

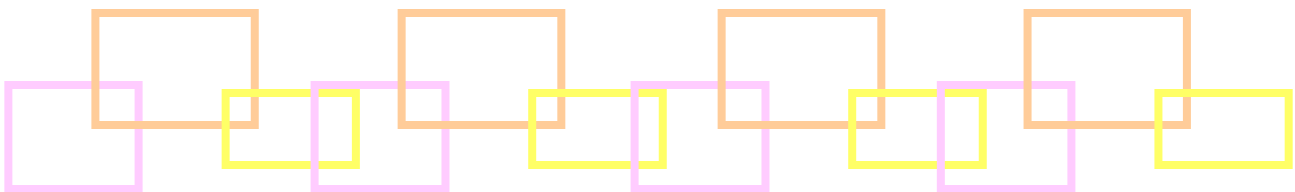


**Vendor Announcements:**

If you are a local vendor with information to share, please contact Jason Heffelfinger ([jason.r.heffelfinger@medtronic.com](mailto:jason.r.heffelfinger@medtronic.com)) for deadline and content guidelines.

**Networking... MMS Seeks a Database Specialist**

In the past the MMS website included a member database that allowed members to look up various bits of information about each other. We would like to re-established a similar database. We are looking for a volunteer with experience setting up such a database. The database would be maintained at the MMS website ISP (GoDaddy.com). A MySQL database server is available. Anyone interested contact the MMS Treasurer at [Treasurer@MnMicroscopy.org](mailto:Treasurer@MnMicroscopy.org) for more detailed information.

**Like to be 'in the know' about upcoming events? Have a creative streak looking for an outlet? Please consider becoming the MMS Newsletter Editor.**

Kathy Hough will be leaving the Newsletter after this edition. The job typically requires ~2 hours of data gathering and organizing per month... a little extra for big events. A comfort level with computer work (Word, Excel, email, PowerPoint, pdf's, and some database access) is needed. Guidance cheerfully provided! Please contact the incoming MMS President if you are interested.

## Corporate Sponsors

*Corporate Sponsors are the backbone of financial support for the Society.*

*These members make it possible for the Society to support Project Micro and to cover many expenses of the regular meetings and the Spring Symposium. MMS gratefully acknowledges the Corporate sponsorships provided by the following companies in 2009-2010. Please take the time to visit those attending this year's Spring Symposium.*

|   |  |   |
|---|--|---|
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**Webmaster:** Stuart McKernan, 3M Center,  
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**Reminder:** Membership in MMS runs from January through December.  
**Have you paid your 2010 dues?**

Please visit the MMS website, [www.MNmicroscopy.org](http://www.MNmicroscopy.org), for a fill-and-print pdf version of the membership form...

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**Minnesota Microscopy Society – Membership Form**

All microscopists are urged to support their Society at one of the membership levels offered below. Often, supervisors will support MMS memberships out of their project budget because they recognize that it is a very inexpensive way to maintain and increase the skills of their microscopists. If you have been a member over the years and recognize the value of MMS to the community of microscopists it serves, consider upgrading your membership this year to the patron or sustaining level. Thank you.

Name \_\_\_\_\_ Dr \_\_\_\_\_ Mr \_\_\_\_\_ Ms \_\_\_\_\_ Phone (\_\_\_\_) \_\_\_\_\_

Affiliation \_\_\_\_\_ Position \_\_\_\_\_

Address \_\_\_\_\_ ZIP \_\_\_\_\_

E-mail address \_\_\_\_\_

**\*A legible e-mail address is critical for delivery of your newsletter.  
 Newsletters are sent via email only.**

Check here \_\_\_\_\_ if you do NOT want your name and address to appear in the Society directory.

Are you an MSA member? \_\_\_\_\_ MAS Member? \_\_\_\_\_ Other Professional groups? \_\_\_\_\_

Student (\$5) \_\_\_\_\_ Basic (\$10-\$24) \_\_\_\_\_ Patron (\$25-\$99) \_\_\_\_\_ Corporate Sustaining (\$100- ) \_\_\_\_\_

Payment: \_\_\_\_\_ Check \_\_\_\_\_ Visa \_\_\_\_\_ MasterCard \_\_\_\_\_ American Express \_\_\_\_\_

Credit Card Number: \_\_\_\_\_ - \_\_\_\_\_ - \_\_\_\_\_ - \_\_\_\_\_ Exp. Date: \_\_\_\_\_ / \_\_\_\_\_

Signature (for credit card payment): \_\_\_\_\_

Make checks payable to MMS and mail to our Treasurer:

Bede Willenbring, MMS Treasurer, 4763 Decatur Ave. North, New Hope, MN 55428-4402