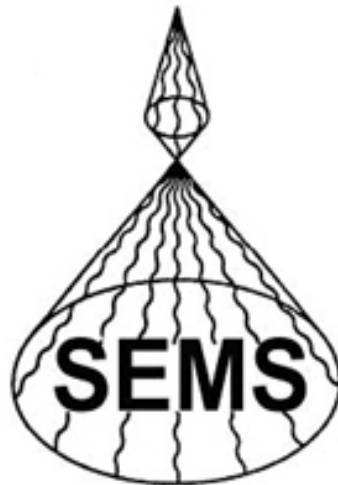


Proceedings of the Southeastern Microscopy Society

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of the
Southeastern Microscopy Society

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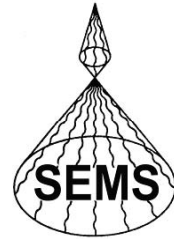
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Acknowledgements

As an affiliate of MSA and MAS we benefit by support for MSA and MAS invited speakers and meeting expenses.

Our **Corporate Members and Exhibitors** are an important part of our organization and make it possible for SEMS to have outstanding meetings and to publish the SEMS Proceedings. We thank them for their excellent service over the years and look forward to a bright and productive future.

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Welcome From the President

Dear SEMS members,

Welcome to the 2017 Annual Meeting of the Southeastern Microscopy Society in charming Athens, Georgia thanks to our local arrangements crew, **Mary Ard** and **John Shields**. This year we have an outstanding program that includes workshops, presentations, the Ruska Award competition, commercial exhibit and demos, and social opportunities for getting to network and meet this year's attendees.

On Wednesday, May 24, registration opens at 8:00 a.m. for a full day of workshops and demos, some that will provide the opportunity to visit the Georgia Electron Microscopy Lab in Barrow Hall. **Fisher Scientific/FEI** is having a STEM workshop, and **Zeiss** is providing CLSM workshops at the Coverdell Bldg. There will also be a Negative Staining workshop with our own **Sara Miller and Mary Ard** sponsored by **JEOL**. The commercial exhibit runs all afternoon and it will be followed by a Corporate Mixer. Thanks to our Program Chair, **Terri Bruce**, our formal program of presentations takes place on Thursday all day and Friday morning, starting with this year's RUSKA competition. On Thursday evening our annual banquet will provide a special and unique opportunity to deliver a tribute to **Ann Ellis** who was one of the longest and most cherished members of SEMS. We will also honor a longtime corporate member, **Dave Roberts** of RMC. I encourage you to share your thoughts and stories about Ann and Dave during the tribute.

The society's business breakfast is on Friday starting at 9:00 a.m. which will be followed by MSA speaker, **Dr. Mike Marko** of the Wadsworth Center, NY, and **Dr. Vincent Smentkowski** of GE Global Research before officially closing the meeting at noon.

It has been a great honor and privilege to get to know and work this last two years, as a council member of SEMS, with the outstanding group of microscopists, researchers and lab technicians that belong to this society. SEMS welcomes new members with open arms, engaging its membership in a supportive, responsive and encouraging environment that clearly defines it as an unparalleled professional home for microscopists. If this is your first time attending SEMS, please take the opportunity to get to know the society, its history and members, and consider joining us as we strive to deliver a better meeting each year and more opportunities for professional growth and development for our community.

Please also take the opportunity to get to know the gorgeous "Classic City" of Athens and University of Georgia campus! Finally, I want to thank each of you for attending and participating in SEMS 2017. We have a great venue, program and an exceptional group of speakers and exhibiting corporate members. I truly hope to see each and everyone one of you enjoying the meeting!

With warm regards,

Luisa Amelia Dempere, SEMS President 2017

SEMS 2017 PROGRAM

TUESDAY EVENING, MAY 23

6:00pm EXECUTIVE COUNCIL MEETING ROOK & PAWN

WEDNESDAY MORNING, MAY 24

REGISTRATION – 8am to 4:30pm HALLWAY

WORKSHOPS: (NOTE: LUNCH ON YOUR OWN)

9am – 11am FEI STEM Workshop and Demo GEM Rm 155
Van to transport from Holiday Inn

8:30am – 12:00 Negative Staining Workshop GEM Rm 154
Sponsored by JEOL USA & Rm 165
Sara Miller and Mary Ard, at the EM labs
Van to transport from Holiday Inn

8:30am – 11:00am Zeiss LSM 710 Confocal Microscopy 106B COVERDELL BLG.
UGA Family Housing or East/West Bus to Coverdell

WEDNESDAY AFTERNOON

1pm – 5pm Commercial Exhibits GEORGIA BALLROOM

1pm – 3pm FEI STEM Workshop and Demo GEM Rm 155
Van to transport from Holiday Inn

1pm – 3pm Zeiss LSM 710 Confocal Microscopy 106B Coverdell Blg.
UGA Family Housing or East/West Bus to Coverdell

6:30pm – 8:00pm CORPORATE MIXER GEORGIA BALLROOM

THURSDAY MORNING, MAY 25

Registration – 8:30am to 5pm

HALLWAY

9:00 am **Opening Remarks – *Amelia Dempere, SEMS President***

PRESENTATIONS:

ATHENA 1

9:15am [MSA Invited] *Historical Perspectives on Biological EM*

M Marko, Wadsworth Center, Empire State Plaza, PO Box 508, Albany, NY

RUSKA COMPETITION

Moderator: *Brandon Walker*

10:00am *Correlative Precession Electron Diffraction and Atom Probe Tomography Characterization of Cluster Formations in Nanocrystalline Cu(V)*

XZhou¹, T Kaub¹, F Vogel¹, R Anthony², and GB Thompson¹

¹ Department of Metallurgical & Materials Engineering, The University of Alabama Tuscaloosa, AL ². Northridge High School, Northport, AL

10:15am *Lithiation and Exfoliation of Metal Hexaborides Using Solution-based Methods*

R Ramachandran and TT Salguero, Department of Chemistry, University of Georgia, Athens, GA

10:30am *Highly conductive reduced graphene oxide (rGO) heterostructures*

M Savchak¹, M Anayee¹, R Burtovyy¹, N Borodinov¹, K Hu², Ruilong Ma², V Tsukruk², I Luzinov¹

¹Department of Materials Science and Engineering, Clemson University, Clemson, SC 29634 ²School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA

10:45am *Investigation of a New Mn₃(PO₄)₂ Polymorph with Electron Microscopy*

GR Neher and TT Salguero, Department of Chemistry, University of Georgia, Athens, GA

10:45am – 11:15am BREAK (PLEASE VISIT EXHIBITORS) GEORGIA BALLROOM

11:15 *Quantitative Phase Imaging for in vitro Observation of Live Cells*
HA Holdaway, Life Sciences Specialist at TESCAN USA Inc., Warrendale, PA

11:30 *Determination of How Morphology Affects the Aging Mechanism of Ag Nanostructures in Aquatic Environs*

EV Formo¹, CB Potter¹, M Yang², RR Unocic³, DN Leonard³, M Pawel³. ¹Georgia Electron Microscopy, University of Georgia, ²Department of Biomedical Engineering, Georgia Institute of Technology, ³Center for Nanophase Materials Sciences, Oak Ridge National Laboratory

11:45am – 1:30pm **LUNCH (ON YOUR OWN)**

THURSDAY AFTERNOON, MAY 25

PRESENTATIONS **Moderator:** *Robert B. Simmons* ATHENA 1

1:30pm *Unique SDD... the Ultimate Analytical Approach*

T Juzwak and J Mastovich, Bruker AXS Inc., 5465 East Cheryl Parkway, Madison, WI

1:45pm *Validation and Application of an Automated Nanoparticle Sizing and Analysis System*

J Martin, MW Rigler, B Jiang, and W Hill, Materials Department, MAS, LLC, (Materials Analytical Services) Suwanee, GA

2:00pm *Simultaneous Atomic-Resolution Secondary Electron and Transmitted Electron Imaging of Nanocrystals*

J Howe, Hitachi High-Technologies Canada Inc, Toronto, Canada

2:15pm *New Diagnostic Observations for Concrete Petrography*

SJ Stokowski, B Wolfe, A Abdkahar, and K Garrett, TEC Services, Inc., 235 Buford Drive, Lawrenceville, GA

2:30pm – 4:00pm **BREAK AND POSTERS** GEORGIA BALLROOM

4:00pm *Determination and analysis of external and internal sulfate attack in concrete from the southeastern United States*

K Garrett, TEC Services, Inc., 235 Buford Drive, Lawrenceville, GA

4:15pm *Case History: Epoxy and Latex Finish Deterioration on Precast Concrete*

SJ Stokowski, TEC Services, Inc., 235 Buford Drive, Lawrenceville, GA

6:00pm-7:00pm

SOCIAL

GEORGIA BALLROOM/HALLWAY

7:00pm-9:00pm

BANQUET

UNIVERSITY ROOM

SPECIAL PRESENTATION:

Remembering Ann Ellis and Dave Roberts - a Tribute

FRIDAY MORNING, MAY 26

9:00am-10:30am

BUSINESS BREAKFAST

UNIVERSITY ROOM

PRESENTATIONS

Moderator: *Terri Bruce*

ATHENA 1

10:30am [MAS INVITED] *Surface Microscopy and Microanalysis in an Industrial Research and Development Laboratory: General Electric Global Research Center*

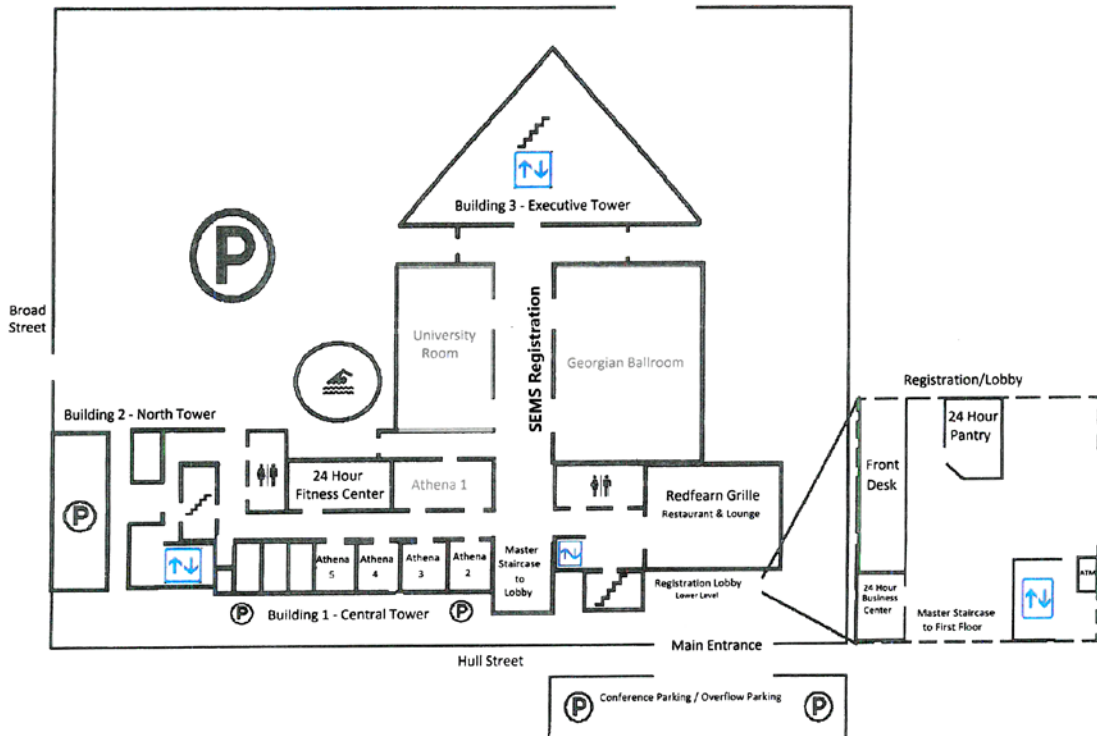
V Smentkowski, GE Global Research, Niskayuna, NY

11:15

CLOSING REMARKS:

TERRI BRUCE, PRESIDENT-ELECT

Holiday Inn Athens Hotel Property Map



All Buildings/Towers connect on the 1st Floor. The Lobby is located on the Lower Level.

Presentations	Athena 1
Exhibitors	Georgian Ballroom
Registration	Hallway
Poster Sessions	Georgian Ballroom
Corporate Mixer	Georgian Ballroom
Wednesday Night Social	Georgian Ballroom
Banquet	University Room
Business Breakfast	University Room
Breaks	Georgian Ballroom
Executive Council Meeting	Tuesday Evening, Rook & Pawn, 294 W Washington St #300, Athens, GA 30601

Workshops on UGA Campus:

Zeiss Confocal:	Coverdell Bldg, D.W. Brooks Drive
FEI and Neg Stain:	Georgia Electron Microscopy Barrow Hall, D.W. Brooks Drive

RUSKA AWARD WINNERS

1972	Danny Akin Univ. of Georgia	1989	Josephine Taylor Univ. of Georgia	2003	Patrick Brown Univ. of Georgia
1973	John Wolosewick Univ. of Georgia	1989	Graham Piper Clemson Univ.	2003	Heather Evans Univ. of S.C. Medical College
1974	Murray Bakst Univ. of Georgia	1990	Chi-Guang Wu Univ. of Florida	2005	Janet Donaldson MS State Univ.
1975	William Henk Univ. of Georgia	1991	Karen Snetselaar Univ. of Georgia	2006	Sangmi Lee MS State Univ.
1976	Durland Fish Univ. of Florida	1992	Yun-Tao Ma Clemson Univ.	2007	Jennifer Seltzer MS State Univ.
1978	Dwayne Findley N.C. State Univ.	1992	Theresa Singer Univ. of Georgia	2007	Tao Wu Georgia Tech
1979	Glen Watkins N.C. State Univ.	1992	Kerry Robinson Clemson Univ.	2008	Katherine Mills-Lujan Univ. of Georgia
1979	John Weldon Univ. of Georgia	1993	Julia Kerrigan Univ. of Georgia	2009	Shanna Hanes Auburn Univ.
1980	Michael Dresser Duke University	1994	John Shields Univ. of Georgia	2010	Kirthi Yadagiri Clemson Univ.
1981	Michael Short W. Georgia Coll.	1994	Meral Keskin-tepe Univ. of Georgia	2011	Maria Mazillo-May Auburn Univ.
1982	Mark Rigler Univ. of Georgia	1995	Katalin Enkerli Univ. of Georgia	2012	David Lovett Univ. of Florida
1982	Chris Sunderman Univ. of Georgia	1996	Rhonda C. Vann MS State Univ.	2013	S. Patnaik MS State Univ.
1983	Patricia Jansma Univ. of Georgia	1997	K.J. Aryana MS State Univ.	2014	Katherine Dye Univ. of Georgia
1985	Mark Brown Univ. of Georgia	1998	Timothy Wakefield Auburn Univ.	2015	Zulong Ke Georgia Tech
1986	Judy King E. Tenn State Univ.	1999	Wendy Riggs Univ. of Georgia	2015	A.A. Trofimov Clemson Univ.
1986	Peter Smith Clemson Univ.	2000	Gail J. Celio Univ. of Georgia	2016	Wren Gregory Clemson Univ.
1987	Robert Roberson Univ. of Georgia	2001	Joanne Maki Univ. of Georgia	2016	James Kizziah Univ. Alabama, Birmingham
1988	Rajendra Chaubal Univ. of Georgia	2002	Rocio Rivera Univ. of Florida		

DISTINGUISHED SCIENTISTS

Jerome Paulin	1984
Ben Spurlock	1985
Ivan Roth	1986
Gene Michaels	1987
Sara Miller	1991
Raymond Hart	1993
James Hubbard	1995
Charles Humphrey	1996
Johnny L. Carson	2000
W. Gray (Jay) Jerome III	2000
Charles W. Mims	2001
Danny Akin	2002
Robert Price	2003
E. Ann Ellis	2009
Glenn Cohen	2010

DISTINGUISHED CORPORATE**MEMBERS**

Harvey Merrill	1989
Charles Sutlive	1989
Ted Wilmarth	1989
Ray Gundersdorff	1997
Charles and Betty Sutlive	2000
John Bonnici	2002
Doug Griffith	2007
Robert Hirche	2008
Ron Snow	2009
Al Coritz	2011

ROTH-MICHAELS TEACHING AWARD

James Sheetz	2005
Charles Mims	2006

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1964-65	Anthony Kittane
1965-66	John Brown
1966-67	William Callahan
1967-68	Ronald Fraser
1968-69	Ivan Roth
1969-70	Emilio Mora
1970-71	Ralph Ramsey
1971-72	N.M. McClung
1972-73	Walter Humphreys
1973-75	Jim Hubbard
1975-76	Edward DeLamater
1976-77	Eleanor Smithwick
1977-78	Gene Michaels
1978-79	Edith McRae
1979-80	Jerome Paulin
1980-81	Ken Muse
1981-82	Mary Beth Thomas
1982-83	Jack Munnell
1983-84	Sara Miller
1984-86	Ray Hart
1986-87	Glenn Cohen
1987-88	Gerry Carner
1988-89	Danny Akin
1989-90	Johnny Carson

1990-91	Janet Woodward
1991-92	Charles Mims
1992-93	Charles Humphrey
1993-94	Sandra Silvers
1994-95	JoAn Hudson
1995-96	Jay Jerome
1996-97	Mark Farmer
1997-98	Robert Simmons
1998-99	Robert Price
1999-2000	Buddy Stephens
2000-01	Jim Sheetz
2001-02	Glenn Cohen
2002-03	Charles Mims
2003-04	Greg Erdos
2004-05	John Shields
2005-06	Judy King
2006-07	Johnny Carson
2007-08	Robert Simmons
2008-09	Giselle Thibeadeau
2009-10	Robert Price
2010-11	Michael Miller
2011-12	E. Ann Ellis
2012-13	Richard Brown
2013-14	W.Gray (Jay) Jerome
2014-15	Mary Ard
2015-16	Amelia Dempere

MSA INVITED SPEAKER

Historical Perspectives on Biological EM

Mike Marko

Wadsworth Center, Empire State Plaza, PO Box 508, Albany, NY 22201-0509

Biological TEM started in 1934, not long after the development of the first TEM that surpassed the light microscope in resolution, Ernst Ruska's 1933 version. Ruska eventually shared the Nobel Prize in Physics in 1986 for this work. Ruska's innovation was the use of iron polepieces to increase the magnetic-field strength so that the magnification could be increased without making the column impractically long. Pioneering TEMs were built in the US and Canada, as well. Series-production of the TEM occurred first in Germany, and then in the US. Immediate specimen damage to biological specimens was thought at first to preclude the use of the EM, but fixation, embedding, and microtomy were eventually developed, and TEM became routine in biology; these developments will be covered. Significantly, early biological TEM was directly responsible for establishing the name and the discipline of Cell Biology, by Porter and Claude in 1956. The 1974 Nobel Prize in Physiology and Medicine was given to Albert Claude, Christian de Duve and George Palade, basically for their studies of cell ultrastructure. The era of the million-volt electron microscope ("HVEM") in biology (mid-1970s to mid-1980s) will be described.

Cryo-TEM, currently the most high-profile technique in biological TEM, started in the mid- 1960s, including the use of liquid helium and superconducting lenses. This work started even before the first description (by Jacques Dubochet) of vitreous ice in the EM in the early 1980s. This aspect of cryo-EM will be covered.

Microanalysis started in the 1940s, and biological applications with freeze-prepared samples appeared in the 1980s. Examples of biological microanalysis will be shown. The concepts relating to three-dimensional reconstruction were introduced already in the late 1960s. Electron crystallography, helical reconstruction, low-dose imaging, 2-D correlation, and 3-D "single-particle" approaches for asymmetric particles were developed in the 1970s and 1980s. The pioneering work in EM structural biology by Aaron Klug was recognized by his Nobel Prize in chemistry in 1982. The sequence of this development, and several of the early groundbreaking papers, will be mentioned. While not strictly a "History", the talk will include a selection of highlights describing many of the advances over the past 80-plus years, with some emphasis on work from the author's own Institution over the past 40 years.

The author is the Archivist of the Microscopy Society of America, and much of the material is in our Archives. The author's own scientific contributions are currently supported by NIH grant R35GM119023.

**Correlative Precession Electron Diffraction and Atom Probe
Tomography Characterization of Cluster Formations in Nanocrystalline
Cu(V)**

Xuyang Zhou¹, Tyler Kaub¹, Florian Vogel¹, Ryan Anthony², and Gregory B. Thompson¹

¹. Department of Metallurgical & Materials Engineering, The University of Alabama
Tuscaloosa, AL 35401.

². Northridge High School, Northport, AL 35406.

It has been shown that solutes can be used to stabilize nanocrystalline grains from high-temperature growth. The enthalpies of mixing and segregation have been used to ascertain the competitive balance for this stabilization. Even in highly segregating systems, where the solute cluster and precipitate as a secondary phase, nanocrystalline stability has been seen. However, much of this work has been done in ‘bulk’ balled milled nanocrystalline materials. The present study aims at producing nanocrystalline grains by thin film deposition to mitigate this extraneous contamination to better elucidate the stability. To initiate these studies, a series of Cu(V) alloys which are predicted to be nanocrystalline stable were grown and subsequently annealed at temperatures up to 800°C for 1 hour. A cross-correlative precession electron diffraction (PED) – atom probe tomography (APT) technique was employed to study the solute grain boundary segregation specificity. The results of which are framed in how solute segregation and migration to specific boundary types and surfaces leads to stability or instability of the grain size.

The authors gratefully recognize support from the Army Research Office, grant 911NF1310436, as well as the University of Alabama’s Central Analytical Facility.

RUSKA

Lithiation and Exfoliation of Metal Hexaborides Using Solution-based Methods

Roshini Ramachandran and Tina T. Salguero

Department of Chemistry, University of Georgia, Athens, GA 30602

Our research has targeted the metal borides owing to their exceptional properties such as high melting points, chemical inertness and extreme hardness. The industrial use of metal borides is limited because they are difficult to process, however, their nanostructuring would open up a wide range of solution-processing options. We used a novel lithiation-exfoliation chemistry to synthesize various nano-morphologies of strontium hexaboride and lanthanum hexaboride. The process entails doping lithium ions into the metal hexaboride structure, and subsequently reacting the lithiated intermediate with water to cause a disassembly and subsequent nanostructuring of the bulk metal hexaboride. Powder x-ray diffraction, multinuclear solid-state nuclear magnetic resonance spectroscopy, transmission electron microscopy, and scanning electron microscopy with elemental mapping were used to characterize the morphology and composition of the lithiated intermediates and exfoliated products. The nanoproducts retain their metal hexaboride composition and structure; proving that this top-down, solution-based synthetic approach paves the way towards low temperature, facile nanostructuring of metal hexaborides.

This work was supported by the US Office of Naval Research.

RUSKA

Highly Conductive Reduced Graphene Oxide (rGO) Heterostructures

Mykhailo Savchak¹, Mark Anayee, Ruslan Burtovyy¹, Nikolay Borodinov¹, Kesong Hu², Ruilong Ma², Vladimir Tsukruk², Igor Luzinov¹

¹Department of Materials Science and Engineering, Clemson University, Clemson, SC 29634

²School of Materials Science and Engineering, Georgia Institute of Technology, Atlanta, GA

The goal of the present research is to fabricate and investigate opto-electrical properties of conductive reduced graphene oxide (rGO) bi-layer films with reduced intersheet resistance. In our approach, we first modified the surface of pristine graphene oxide (GO) sheets with surface active copolymer poly(GMA-ran-OEGMA) [GMA: glycidyl methacrylate; OEGMA: oligo ethylene glycol methyl ether methacrylate], which allowed us to obtain a GO monolayer with high sheet/surface density over a large surface area of silicon wafer substrate. Then we let polyacrylic acid adsorb onto the surface of formed GO layer and finally the second layer of GO was formed by dip-coating. Afterwards such heterostructure has been subjected to thermal reduction. The presence of double GO/rGO layers on the surface of silicon wafer was confirmed by AFM imaging. Also, we employed TGA, FTIR, SEM and XPS for materials characterization. We demonstrated that the thermal reduction of GO to rGO led to obtaining quite transparent material with high electrical conductivity on the order of 10^3 S/cm.

This work is supported by National Science Foundation # 2015001055.

Investigation of a New $\text{Mn}_3(\text{PO}_4)_2$ Polymorph With Electron Microscopy

Gregory R. Neher and Tina T. Salguero, Department of Chemistry, University of Georgia, Athens, GA 30602

Metal phosphate compounds have received much attention in recent years due to their applications in supercapacitors, water oxidation catalysts, and precursors to cathode materials for Li-ion batteries. We recently synthesized a fourth manganese orthophosphate polymorph and characterized the crystal structure with single crystal X-ray diffraction. The unique structure of $\delta\text{-Mn}_3(\text{PO}_4)_2$ is highlighted by planes of edge-shared MnO_5 pentrahedra adjoined to chains of PO_4 tetrahedra, forming a microporous open-framework structure. Both the crystal growth and morphology of this new compound were investigated with scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Microcrystals of $\delta\text{-Mn}_3(\text{PO}_4)_2$ with dimensions of $>100\ \mu\text{m}$ were synthesized by reacting $\text{Mn}_5(\text{PO}_4)_2(\text{PO}_3\text{OH})_2\cdot 4\text{H}_2\text{O}$ at $250\ ^\circ\text{C}$ for 1-6 h. An SEM time study reveals that the microcrystals grow into flower-like microstructures, via a dissolution-renucleation pathway. Interestingly, changing the precursor to $\text{Mn}_3(\text{PO}_4)_2\cdot 3\text{H}_2\text{O}$ and applying similar reaction conditions yields microplates of $\delta\text{-Mn}_3(\text{PO}_4)_2$ approximately $10\text{-}40\ \mu\text{m}$ in length and $150\ \text{nm}$ in thickness. Selected area electron diffraction (SAED) and high resolution TEM confirm the preferred growth of the microplates along the (002) plane, illustrating the anisotropic structure of $\delta\text{-Mn}_3(\text{PO}_4)_2$. Hydrothermal reaction of $\delta\text{-Mn}_3(\text{PO}_4)_2$ with Li_3PO_4 forms nano-morphologies of LiMnPO_4 after 6 h at $250\ ^\circ\text{C}$ aimed. Ongoing work is focused on studying the incorporation of lithium into this structure for energy applications.

This work was supported by the U.S. Department of Energy, Office of Basic Energy Sciences, DE-SC0008065

Quantitative Phase Imaging for *in vitro* Observation of Live Cells

Heather A. Holdaway

Life Sciences Specialist, TESCAN USA Inc., Warrendale, PA 15086

Live cell imaging is an integral component in better understanding cellular dynamics, for instance, differentiation, senescence, disease and death (such as apoptosis, necrosis, autophagy, and external treatment). One key characteristic in observing cellular dynamics is a change in cellular morphology. Current cell imaging techniques lack several important components required for live cell imaging. For example, brightfield, phase contrast and differential interference contrast imaging all lack clear cell boundaries and quantitative data. Fluorescence imaging provides good boundary detection and quantitative data for segmentation, however, it is not a natural system (requires labeling) and has a high photo toxicity. Quantitative phase imaging (QPI) provides better boundary detection and quantitative data in a natural state. Imaging in scattering media (e.g. phospholipid emulsions, extracellular matrices) still presents an issue with *in vitro* observation of live cells. New patented technology of Coherence-controlled holographic microscopy allows for excellent boundary detection and quantitative data collection of live cells in scattering media. This allows for testing reactions of cells to a specific treatment. Examples include cancer cells and immune cells.

Determination of How Morphology Affects the Aging Mechanism of Ag Nanostructures in Aquatic Environments

Eric V. Formo¹, Caroline B. Potterf¹, Miaoxin Yang², Raymond R. Unocic³, Donovan N. Leonard³, Michelle Pawel³

¹. Georgia Electron Microscopy, University of Georgia

². Department of Biomedical Engineering, Georgia Institute of Technology

³. Center for Nanophase Materials Sciences, Oak Ridge National Laboratory

In studying the fate and aging of nanomaterials exposed to the natural environment, electron microscopy techniques for analyzing such dynamic processes have been limited. Herein we show the remarkable findings related to how the shape of a nanostructure affects its aggregation and dissolution. We obtained these results via the use of electron microscopy by comparing the aging profiles of silver nanocubes (NCs) and silver nanoparticles (NPs) after exposure to aquatic systems. (S)TEM in particular was critical in understanding the aging mechanism of both of these nanostructures once they were placed in seawater, pond water, or synthetic hard water. Specifically, we observed that the silver NCs do not undergo aggregation but instead maintain their nano-size domain until the silver atoms have undergone complete dissolution from the NC in all of our test waters. This result runs counter to what takes place in the case of silver NPs which simply aggregate as expected. Further HR-TEM analysis clearly details that the exposed $\langle 100 \rangle$ surface facets of the NCs caused the enhanced robustness of the Ag NCs. Moreover this ability of Ag NCs to maintain their nano size domain for an extremely long period of time in aquatic environments, potentially makes them more environmentally harmful than their NP analogues.

Unique SDD... the Ultimate Analytical Approach

Ted Juzwak and John Mastovich,
Bruker AXS Inc., 5465 East Cheryl Parkway, Madison, WI 53711

The Silicon Drift Diode (SDD) EDS detector has become the industry standard, as it has superior throughput while maintaining resolution and light element sensitivity, and holds calibration from low to high count rates. Today, larger active areas and multiple-detector configurations for SEMs and TEMs are available, providing very rapid analysis. An alternative and unique approach is the implementation of an SDD array between the pole piece and the sample (like a backscatter detector), providing the highest solid angle possible and the ultimate throughput. Numerous applications will be discussed to illustrate the analytical benefits of the Flat Quad SDD technology.

This work has been supported by a variety of Bruker employees and clients.

Validation and Application of an Automated Nanoparticle Sizing and Analysis System

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FDA requires that analytical systems and methods be validated for performance in order to be used by GMP/GLP testing labs. All FDA registered labs must show documented proof of precision and accuracy for each analytical system used for drug and medical device testing. We describe a method for validating automated particle analysis using a Hitachi SU 8230 cold field emission scanning electron microscope with a Bruker Flat Quad detector and the Bruker Esprit automated particle analysis software. The Bruker Esprit software was first calibrated using an MRS-2 SEM magnification standard and stage micrometer. For each of 6 sample runs, NIST certified gold coated silica, borosilicate and soda lime particles (500 nm – 100 um) on polycarbonate filters were used to determine precision and accuracy. The microscope settings were as follows: accelerating voltage was 15KV and the working distance (WD) was set to 8 mm. The magnification setting for each sample was dependent on the particle size and distribution of the particles. Sample fields were randomly analyzed automatically field-to-field in backscatter mode using the photodiode backscattered (PD-BSE) detector with brightness and contrast adjusted so that particles appeared bright on a dark background. The bright particles were sized using the bright phase particle setting found in the Esprit software. Filters were added to separate touching particles. Several random areas of the sample preparation were examined to ensure that a total of at least 2,500 or more particles were sized according to the average diameter. Data from all runs was compiled and summarized into tables of analytical results. Accuracy ranged from 96% to 100% across all size ranges and the precision ranged from 7.3 to 14.5%. A correlation coefficient of 1.0 was determined for the standard curve over the wide particle size range analyzed. Specific nanoparticle analysis applications, including sizing, particle identification, and analysis time will be discussed.

New Diagnostic Observations for Concrete Petrography

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Changes in concrete technology make this an exciting time to be a concrete petrographer, as illustrated by four case histories. First, we investigated many cases of early scaling of the top of concrete sidewalks and a road. These projects used concrete with a state-mandated mix design containing 70% GGBFS (ground-granulated blast furnace slag, a pozzolanic replacement for portland cement, PC), but had different contractors. The scaling was due to leaching and migration downward of portlandite ($\text{Ca}(\text{OH})_2$) before it reacted the GGBFS, which had greater crystallinity than normally observed, so was unlikely to be very reactive. Portlandite is a PC hydration product essential for the pozzolanic chemical reaction with GGBFS that results in concrete durability. We suspect that the GGBFS was a defective import, instead of the product previously made at the Sparrows Point Steel Mill in Baltimore, which closed. The second case history involves Alkali Carbonate Reactivity (ACR). A precast, prestressed concrete product exhibited pattern cracking in less than one year, an unusual situation, especially given that the coarse aggregate was relatively pure dolomite. ACR occurs primarily with argillic dolomitic limestones. There are a few reported cases of ACR with pure dolomites in the US and worldwide that are debated for a variety of technical and economic reasons. In this case, the dolomite aggregate in the concrete contains partially dissolved dolomite crystals, microcracks filled with calcite ($(\text{CaCO}_3)^2$) that extend from the aggregate into the cement paste, the aggregate and surrounding cement paste has an elevated potassium (K) content suggestive of a chemical reaction with the K component of the cement, and there are isolated concentrations of EDX-identified magnesium (Mg) suggestive of Brucite. In the third case history, we used minor occurrences of ASR (Alkali Silica Reactivity) in concrete as an indicator of minimum relative humidity. The associated projects investigated the widespread deterioration of concrete foundations made with aggregate containing pyrrhotite (Po). Po is an unstable mineral with time in the presence of oxygen and moisture. While it is reasonable to presume that a concrete foundation would have a high relative internal humidity, this is not a given because the foundations have a bituminous moisture barrier adjacent to the soil. Work by Stenger (1982¹) established that 50% is the minimum relative humidity for Po oxidation. Observation of minor ASR reactions with slate in the PC allowed the historic relative humidity to be great enough to oxidize Po because Stark (1991²) established that the minimum relative humidity in concrete for ASR at 70%. In the fourth case history, in super-plasticized, low w/c mixes we recognized an association of low cement hydration and concrete microcracking adjacent to coarse aggregate that was batched dry and absorbed water from the mix. This is essentially the opposite of the concept of internal curing, where wet, absorptive rock causes better hydration of cement.

¹ Stenger, H. F., 1982, Oxidation of Sulfide Minerals VII. Effect of Temperature and Relative Humidity on the Oxidation of Pyrrhotite: *Chemical Geology*, V. 35, p. 281-295

² Stark, D., 1991, The Moisture Condition of Field Concrete Exhibiting Alkali-Silica Reactivity: CANMET/ACI Second International Conference on Durability of Concrete, SP-126, American Concrete Institute, p 973-987

Determination and Analysis of External and Internal Sulfate Attack in Concrete from the Southeastern United States

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Sulfate attack is a severe form of concrete degradation. It often leads to irreparable damage such as expansion, cracking, and softening of the cementitious matrix. The types of sulfate attack include external, in which the source of sulfate exists outside of the concrete such as in the soil or groundwater, and internal where the source of sulfate comes from the components in the concrete mix. The type of sulfate attack, as well as the severity, can be determined petrographically using transmitted polarized light microscopy and reflected light microscopy, skills often practiced by well-trained geologists. Two case histories illustrating TEC's work in the southeast United States explore the petrographic observations that establish the presence of sulfate attack and the source of the sulfate and moisture necessary for the reaction. The case histories exemplify the presence and significance of gypsum, ettringite, and thaumasite in deteriorated concrete, the diagnostic crystal forms and spatial distribution of the secondary minerals, the placement environment, and how key geologic principals are important to the diagnosis and resolution of this materials problem.

Case History: Epoxy and Latex Finish Deterioration on Precast Concrete

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Optical and SEM/EDS microscopy were essential tools for evaluating why epoxy and latex finishes on precast concrete prison-cells deteriorated. The concrete finish system essentially consists of: first, bonding agent, then, a filled sealer coat, and last, a finish coat (epoxy or latex paints). Expansion of the filled sealer coat caused alligator cracking of the epoxy finish and delamination at the base of the epoxy or latex finish. The expansion resulted because of confined hydration of the white, Type I cement substituted for the originally-specified ground marble.

The $\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$ efflorescence enhanced the expansive hydration of the cement filler when it dissolved into the acrylic emulsion sealer. Chloride is present in the concrete and traces of calcium chloride are present in and on the concrete, and in the bonding agent, filled sealer coat, and as unusual molds of twinned crystals in the latex paint on the exterior of the cells.

Subsequent coating delamination occurred primarily as micro-cracking tensile failures in the underlying, weak concrete; minor adhesive failure occurred over aggregate particles.

MAS INVITED SPEAKER

Surface Microscopy and Microanalysis in an Industrial Research and Development Laboratory: General Electric Global Research Center

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The top few nanometers of a sample is defined as the surface. The surface is where most chemical reactions take place. There are many instances where the surface of materials are designed/functionalized in order to optimize properties and improve device performance; there are other instances where the surface becomes compromised and the material/device performance degrades. Auger Electron Spectroscopy (AES), X-ray Photoelectron Spectroscopy (XPS), and Time of Flight Secondary Ion Mass Spectrometry (ToF-SIMS) are the three most common, and commercially available, surface analysis techniques. These techniques provide complimentary information regarding the composition/microstructure of the surface of a sample. In this presentation, we will introduce AES, XPS, and ToF-SIMS, show typical data, and discuss how the data helped understand mechanisms and/or resolve material problems. We will also introduce techniques which we do not have in-house, but have access to via external collaborations.

POSTER

Self-Assembly of a Tandem Repeat Protein Protomer into a Sequence Flexible Solenoid

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Tandem Repeat Proteins (TRPs) represent an attractive scaffold for the development of tunable biomaterials due to their simplistic sequence to structure relationship, and high sequence flexibility. TRPs comprising helix-turn-helix subunits are particularly useful due to the minimal effect of sequence mutations on the 3-dimensional protein fold. A TRP protomer was chemically synthesized and self-assembled *in vitro* into extended solenoidal structures with a defined external diameter and a solvent accessible pore. The nanotubes were shown via negative stain TEM to have a diameter of 8.4 nm, and a pore diameter of 4.8 nm. Mass per Length measurements derived from STEM showed that there were 21.3 subunits in a superhelical turn, and linear power spectra taken from the negative-stained STEM images gave a superhelical pitch of 30.7 Å. Cryo-EM and iterative helical reconstruction were used to generate a sub 5Å-resolution structure of the nanotubes, and revealed an unexpected dimerization of the assembly protomer. The inherent sequence flexibility of the inner and outer walls of the structure could allow the binding of carbon nanotubes, small molecules, and even other protein moieties.