6th FIB SEM Workshop
March 1, 2013
Cambridge Massachusetts

Over six years ago a group of focused ion beam (FIB) and FIB-SEM (scanning electron microscope) users from the greater Washington DC area held their first meeting of its kind. It grew rapidly and by the 5th year well over 100 users attended from areas no longer confined to greater DC but also internationally. The organizers decided it was time to "take the show on the road" and held the 6th meeting in Cambridge, MA. Again, well over 100 users attended the 18 paper presentations, 10 poster s and exhibits by a dozen companies.

The program was packed with technical information, opportunities for networking and also equipment demonstrations. Harvard University's Center for Nanoscale Systems co-sponsored this meeting along with 14 equipment and services suppliers. A tour of Harvard's CNS facilities was offered in the afternoon and many attendees participated.

The technical program opened up with a tribute to Oliver Wells by Lynne Gignac of IBM. Oliver was Sir Charles Oatley's 2nd PhD student at Cambridge University to have worked on the SEM (after Dennis McMullan) and is considered one of the founding fathers of the field. Oliver passed away the week prior to this meeting.

The first two papers of the program introduced us to new applications of cryogenic FIB-SEM. Yu Zhu of IBM presented results of cryogenic FIB-SEM preparation of photovoltaic material for TEM imaging and analysis. It was discovered that cryogenically FIB milling the Cu2ZnSn(S,Se)4 material eliminated artifacts observed in room temperature FIB milling. Cyro-FIB samples had almost no particle formation during sample preparation, and the TEM sample showed no Cu diffusion/enrichment in the CdS layer by EDX.

Yu Zhu*, Nicholas Antoniou**, Jemima Gonsalves* & David Mitzi*
*IBM TJ Watson Research Center, **Harvard University
Cryo-FIB TEM Sample Preparation for Cu2ZnSn(S,Se)4 Solar Cell

The next paper, presented by Cheryl Hartfield, outlined the effort and results in the development of a universal cryogenic FIB-SEM sample preparation technique and equipment for TEM imaging and analysis. Minor modifications of the Autoprobe™ tip for cryogenic use and off-the-shelf cryogenic stage and prep equipment were used to prepare samples in cryo FIB-SEM.

Cheryl Hartfield* & Nicholas Antoniou**
*Oxford Instruments, **Harvard University
Progress towards Cryo FIB Lift-Out

Still on the topic of TEM sample preparation but at room temperature, the next paper by Solomon Boakye-Yiadom & Nabil Bassim (father of one of the organizers of this meeting!) presented a new approach using FIB for preparing thin foil sections from adiabatic shear bands in selected impacted steel specimens for transmission electron microscopy examinations. It was found that the emergence of dislocations, carbide dissolution, evolution of subgrain/subgrain boundaries and dynamic recovery/recrystallization, contribute simultaneously to the formation of adiabatic shear bands in quench-hardened steels.

Solomon Boakye-Yiadom & **Nabil Bassim**
University of Manitoba
Use of FIB to Study the Impact Properties of Materials

FIB-SEM instruments are now being used for a new type of sample preparation, Atom Probe Tips (APT). Matthew I. Hartshorne showed us how FIB instruments enable site-specific APT specimens to be made from grain boundaries and other regions of interest. A technique has been developed to target specific grain boundaries in these materials using a combination FIB and TEM, and the method was validated by targeting a prior austenite grain boundary in a martensitic steel. The technique involves first preparing a TEM sample perpendicular to the grain boundary, then transferring the sample to an electropolished stub compatible with a Fischione on-axis tomography holder. The TEM sample is then rough cut to position the grain boundary near the end of the sample, then annular milled to shape the atom probe specimen. This procedure produces samples with a grain boundary near the tip, on a sample stub suitable for use in an on-axis tomography, and has provided a higher success rate than experienced with traditional techniques for materials with grains in this size range.

Matthew I. Hartshorne*, Dieter Isheim**, David N. Seidman** & Mitra L. Taheri*
*Drexel University, **Northwestern University
Atom Probe Tomography: A Novel Double-Liftout Method for Site-Specific Preparation of Specimens for Correlating TEM and APT in Fine Grained Materials

Andrew Smith of Kleindiek Nanotechnik presented how the integration of an atomic force microscope (AFM) in a FIB-SEM can add 3-D surface imaging to the already highly capable two beam system. The AFM's main advantage is its ability to obtain 3D information, the downsides are that it is hard to find the target area and image generation is slow. Combining these two tools into one setup - putting an AFM inside an SEM - gives quick access to a more complete data set. Additionally, FIB-milled or FIB-deposited structures can be characterized using this combination of tools in a FIB-SEM system.

A.J. Smith*, G. Renka*, K. Schock*, A. Lieb**, M. Dadras*** & S. Kleindiek*
*Kleindiek Nanotechnik GmbH, ** Nanosurf AG, ***Centre Suisse d'Electronique et de Microtechnique
Addinf 3D Surface Imaging to Your FIB/SEM

Harvard University researcher Aaron Kuan presented the technique he developed to image solid state nanopores used in single molecule DNA-sequencing. A cross sectional TEM image of asingle nanopore is difficult to achieve because of the nanometer precision required in localization, integrity of the structure as it is being milled and overall scale (sub nanometer resolution required along with nanometer scale elemental analysis).

Aaron Kuan
Harvard University
FIB-SEM Techniques for Solid-State Nanopore Characterization

Continuing on the subject of nanopores, Joe Klingfus introduced us to newly developed ion lithography system. Using the Raith ionLiNE, a FIB tool designed exclusively and foremost as a lithography nanofabrication instrument, Joe and his team were able to fabricate arrays of nanopores across a 4 inch wafer in an automated step-and-repeat fashion. High quality, repeatable 20 nm pores were milled in a 100 nm Si3N5 membrane and distributed across a 4 inch wafer.

J. Klingfus*, A. Nadzeyka**, S. Bauerdick**, T. Albrecht*** & J.B. Edel***
*Raith USA, **Raith GmbH, ***Imperial College London
Wafer-scale Ion Beam Lithography of Nanopore Devices

Until a few years ago, FIB systems used gallium exclusively as the ion species. The few but important limitations of gallium have led to developments such as the helium focused ion beam, with a smaller probe size relative to that of the electron beam and Ga-FIB. H. Wu of Zeiss presented to us the capability of fabricating sub-10 nm patterns at high densities using the He ion FIB. The resulting metal deposits were directly inspected for their geometry and size by helium ion microscopy (HIM) because of its high imaging resolution, and further characterized by high resolution transmission electron microscope (HR-TEM) and electron energy-loss spectroscopy (EELS). Electrical property measurements show these metal lines have less than 100 μΩcm resistivity and less than 100 Ω contact resistance on gold pads. HR-TEM images reveal that these metal lines are composed of 5-10 nm crystallite grains; and EELS analysis shows that no measurable carbon signal was observed. By reducing the size of the beam limited aperture, 10 nm metal lines at 25 nm half pitch were deposited by He ion beam.

H. Wu*, L.A. Stern*, K. Klein*, D. Xia*, D. Ferranti*, B. Thompson*, P.D. Rack** & C.M. Gonzalez**
*Carl Zeiss NTS, **University of Tennessee
Fabrication of 10 nm Metal Lines with Low Resistivity by Helium Ion Beam Induced Deposition

Mike Phaneuf of Fibics gave us a glimpse into what can be done beyond the column and species improvements in FIB with advanced beam control mechanisms, often coupled with direct signal feedback from the perspective of the beam(s) being patterned. Such beam controls and feedback have enabled a range of new developments that step outside what has traditionally been thought of as "the limits of FIB".
Through the use of "non-standard" patterning and beam control methods Mike and his team have pushed the boundaries of what is typically considered achievable,. Examples

from LMIS Ga-FIB, FIB-SEM and GFIS instruments from the fields of large area imaging, three-dimensional analysis, circuit editing and nanofabrication, with and without gas assisted processes were presented.

Michael W. Phaneuf
Fibics Inc.
Enabling Next-Generation Focused Ion Beam Applications Through Advanced Beam Control

In the spirit of advances to the FIB-SEM technology next was Brandon VanLeer who presented what is new from FEI in beam chemistry and patterning on FIB-SEM. Chemical precursors used in conjunction with the DualBeam can provide an expanded capability to create 2D and 3D nano- and micro-scale structures. FEI has designed a new gas delivery module that allows control of pumping speed and independent gas mixing. Together with the FEI DualBeam platform, users can precisely control each parameter of deposition or etch process in an automated format.

Time-of-flight correction and differential pumping provide for more accurate direct writing at short dwell times and improved resolution while using gas injection for deposition and selective etch. Integration of a high-resolution 16-bit patterning engine allows the user to access up to 65,000 X 65,000 points for a given field width and a pattern memory of up to 8 million points. Nanoprototyping software that automates multi-site, complex direct-write processes can be an invaluable tool for pattern and process optimization. Nanobuilder provides an environment that automates beam conditions including energy and current, patterning parameters, the use of beam chemistry with the use of onboard patterns GSDII files or stream files using an optional layer-by-layer alignment step.

Brandon Van Leer & Laurent Roussel
FEI Company
Fast Prototyping of Functional Devices Using a DualBeam

A regular at the FIB-SEM meetings is Leonidas Ocola has been sharing with this group his developments in the fabrication of microfluidic devises using FIB. Unlike electron beam lithography, in ion beam lithography one can program arbitrary depths by manipulating the dwell time, or dose, of a particular structure. Leo and his team have been working on this topic for several years now and are able to show that the ion beam tool can be used for real microfluidic applications where the bottom of serpentine mixers are texturized dramatically using the FIB/SEM. Some of the lessons learned and pros and cons of different patterning strategies were presented.

Leonidas E. Ocola
Argonne National Laboratory
Advances in Ion Beam Micromachining for Complex 3D Microfluidics

Carl Kamp from MIT shared with us his experience with introducing FIB-SEM to the aspects of the automotive industry.

Carl Kamp, Alexander Sappok & Victor Wong
Massachusetts Institute of Technology
Investigations of Automotive Aftertreatment Component Aging by Focused Ion Beam (FIB) Milling: the Catalyst-Substrate Interface and Intra-and Inter-Layer Interactions

William Rice from the New York Structural Biology Center shared images and applications results concentrated on sectioning of biological samples embedded in resin. Examples include: Ultrastructure analysis of filamentous bacteria in mouse intestine through FIB/SEM, use of EM to analyze how the morphology of dendritic and/or t cells are affected by presence of HIV virus, characterization of organelles in identified cells in the nematode, C. elegans, use of large scale tomographic imaging to study synapse morphology in rat amygdala and to quantify the total number of synapses into the different compartments of layer 4C of macaque primary visual cortex and other.

William J. Rice
New York Structural Biology Center
Overview of projects collected on the Helios FIB/SEM at the NYSBC

Kedar Narayan of NIH dazzled us with animated 3-D reconstructions and tested our A/V capabilities with his elaborate movies. Ultrastructural imaging across large volume scales is a significant challenge in biology, as traditional methods force a trade-off between image resolution and size. Using novel automated procedures in Focused Ion Beam Scanning Electron Microscopy (FIB-SEM), his team was able to simultaneously image large resin embedded biological samples rapidly at intermediate resolutions, and selected sub-volumes at high (up to 3x3x5 nm) resolutions. Using a combination of fluorescently labeled HIV core proteins, TRIM5a (a cellular protein that binds HIV) and cell membranes, they first produce a "target map" of an HIV infected cell by fluorescence microscopy. They then generate a correlated high-quality 3D EM volume of the entire cell, and simultaneously, target high-resolution 3D images of cytoplasmic TRIM5a, endocytosed HIV virions, and individual HIV cores. This represents a size differential of 10^9-fold in a single dataset collected in a one automated overnight run. These results represent significant progress toward "point-and-click" imaging where one can efficiently visualize the nanoscale local structure as well as mesoscale global context of biological objects of interest.

Kedar Narayan
National Institutes of Health
Simultaneous Correlative 3D Imaging of Nanoscale Targets and Entire Cells by Focused Ion Beam Scanning Electron Microscopy

Continuing with 3-D recosnstruction was Luiz F. G. Morales of the German Research Centre for Geosciences Potsdam but applied to geological material. In addition to the now well-known methods of TEM foil preparation, this group has been using FIB techniques mainly for nanotomography and micromachining of materials. The nanotomography, conducted via FIB-SEM, has been applied in a number of studies, such as (i) 3D reconstruction of open grain and phase boundaries in rocks; (ii) 3D phase quantification and distribution of microstructures, such as symplectites, characterized by the complex intergrowth of two or more phases; (iii) 3D modeling of porosity and organic matter distribution in shales, a very fine grained rock that may potentially store oil and gas. Micromachining on the other hand has been used for the preparation of µm samples of single and polycrystals for ultrahigh pressure experiments in diamond anvil cells, and to cut mechanically anisotropic crystals that are not possible to cut and polish by standard methods.

Luiz F.G. Morales & Richard Wirth
German Research Centre for Geosciences Potsdam
Applications of FIB Coupled with SEM and TEM in Geological Materials

Last but not least on the 3-D reconstruction section was Shawn Zhang from VSG. His presentation focused on challenges in 3-D reconstruction, specifically the difficulty with empty space in structures. Most of the time, it is very difficult or impossible to fill the pore space with epoxy before image acquisition, hence the electrons will go behind the current imaging plane and hit the back-side of the pore. The signal reflected from the pore-back created contrast artificially brighter than that of a pore space. Strategies on dealing with this "pore-back" artifact were presented with applications in porous shale rock and fuel cells. While FIB-SEM tomography provide unprecedented resolution, the volume of the specimen being studied is typically very small and unlikely to be representative. It was shown how the microscale information from FIB-SEM can be integrated with macroscale data via a multi-scale imaging and modeling framework in subsurface transport applications.

Shawn Zhang
Visualization Sciences Group
Advanced Processing and Modeling Methods for Porous Material FIB-SEM Data

Ed Principe from Tescan presented on the latest Tescan Workstation, The FERA, featuring the plasma ion column in combination with a third generation field emission electron column. He provided an overview of the ion source hardware and performance with a comparison with LMIG ion sources.He also described the unique and complementary analytical methods compatible with the FERA platform such as the integration of FIB-SEM with ToF-SIMS. FIB based ToF-SIMS represents a favorable nano-analytical method in terms of lateral resolution, analytical volume and detectability. Other interesting hardware accessories he discussed include in-situ AFM and the opportunity for cathode luminescence (CL) as a correlative microscopy method in biological applications.

E.L. Principe
Tescan
FERA Plasma Ion Source Workstation: Applications and Possibilities

To close the meeting John Notte of Zeiss presented on gas filed ion source making or jokingly: "Why Some Noble Gases are More Noble Than Others". In the last 8 years, the gas field ion source (GFIS) has been commercialized with the purpose of producing a focused helium ion beam useful for both sub-nanometer imaging and a variety of nanofabrication techniques. While this new technology is gaining wider acceptance, many enthusiasts suggest that the same GFIS might be put to use for other gas species to permit an ion source in which virtually any gas species can be selected. The reality is that helium is a remarkable gas species that is uniquely suited to the GFIS. To date, the 'beyond helium' efforts have concentrated on making a neon beam function effectively. For the GFIS, neon is the second favorite gas species, but still there are monumental differences compared to its lighter brother, helium. Neon will ionize at about 75% of helium's field strength requiring a lowering of the field that otherswise serves to protect the ion source. And while neon is not quite able to condense on the surface of the cryogenic emitter (75 Kelvin), it does tend to randomly adsorb and desorb on the emitter causing emission fluctuations over time. In this talk John presented an overview of the latest results with an emphasis on the technical challenges to its stability and lifetime.

John Notte
Carl Zeiss
The Gas Field Ion Source: Extension to Species Beyond Helium

The authors and titles of the posters presented are below.

Soeren Eyhusen & Roland Salzer, Carl Zeiss Microscopy
Expanding the range of the FIB: From rapid material removal to ultra-thin TEM lamella preparation

Lucille A. Giannuzzi, L.A. Giannuzzi & Associates LLC
EXpressLO™ for High Throughput Lift-Out

Zachary D. Harms, Daniel G. Haywood & Stephen C. Jacobson, Indiana University
Fabrication of In-Plane Nanofluidic Devices in Glass Substrates by Flood Gun Assisted FIB Milling

Gavin Murphy, Indiana University
Indiana University's state-of-the-art FIB-SEM

Konrad Rykaczewski, NIST
Seeing Below the Drop: Cryo-FIB/SEM Imaging of Complex Nanoscale Interfaces involving Solid, Liquid, and Gas Phases

S.D. Sitzman & C.T. Chou, Oxford Instruments America
Method for Grain Boundary Triple Junction Analysis by 3D EBSD

Anna Weber, Rahul Thakar, Celeste Morris, Kirstin Morton, Chiao-Chen Chen & Lane A. Baker, Indiana University
Focused Ion Beam Milling of Nano Scale Probes for Scanned Probe Microscopy

Richard G. White, Tim S. Nunney, Paul Mack & Brian R. Strohmeier, Thermo Fisher Scientific
Advanced Depth Profiling Characterization of Mixed Organic/Inorganic Multipayer Devices Using X-Ray Photoelectron Spectroscopy (XPS) and a Combined Monatomic and Gas Cluster Argon Ion Source

Efrat Moyal & Janet Teshima, Lattice Gear LLC.
New, Innovative Method for Accurate Cleaving of Samples with Single Crystal Substrate

George Wetzel* & Jamil Clarke**, *Clemson University, ** Hitachi High Technologies America
Three-Dimensional Microanalysis of Advanced Materials Using FIB-SEM Instrumentation