The ORION Helium Ion Microscope: Imaging and Modification of Bio-Materials

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Outline:

1. HIM Technology
2. HIM Sample Interaction Advantages Compared to SEM
   A. Higher resolution
   B. Greater depth of focus
   C. More surface sensitivity
   D. Reduced charging artifacts
   E. Reduced sample damage
3. HIM Imaging Examples
4. HIM Modification Capabilities

Verbal Accompaniment: The ORION helium ion microscope is a new technology - not unlike the SEM was in 1950. It has undergone a long evolution beginning at its conception in 1955. And it was just in 2005, that the technology achieved sufficient promise to warrant the establishment of ALIS Corporation (Atomic Level Ion Source) in Peabody, MA in 2005. By 2006, a commercial product was produced, and shortly thereafter Carl Zeiss SMT acquired ALIS.
Now (2010) the ALIS technology is past its infancy. It has proven itself in a range of applications, and continues to find new capabilities in imaging and nanofabrication.
History of the Gas Field Ion Source:

Verbal Accompaniment: The ancestry of the helium ion microscope can be traced to Erwin Muller. Despite his obscurity, Muller was the first person to see atoms. Actually it was his graduate student who insisted on doing this experiment on Oct 11, 1955 at Penn State. Muller would probably have been awarded the Nobel prize if he lived long enough. Amazingly, this simple blown glass instrument (pictured right) is all he needed to achieve this milestone!

Erwin Müller (1911-1977)

History of the Gas Field Ion Source:

Verbal Accompaniment: Muller’s simple instrument is shown in the diagram above. Called the Field Ion Microscope, it produces a projection image of the most-protruding atoms at the end of a pointy needle. At the time, Muller required dark adapted eyes and photographic plates. The Atom Probe is also a descendent of this technology, making it a cousin of the ORION microscope.
History of the Gas Field Ion Source:

Verbal Accompaniment: This slide shows the one to one correspondence between the most protruding atoms on the metal needle, and the projection image. The metal needle is crystalline, so although its end form is roughly spherical, the most protruding atoms form a geometric pattern. Usually thousands of little beamlets are produced, each created in the ionizations disks above the most protruding atoms.
History of the Gas Field Ion Source:

Verbal Accompaniment: Muller’s FIM, when oriented vertically, becomes the ion source for the helium ion microscope. The rest of the microscope involves many other technical challenges, but can be briefly described as being like a conventional SEM or FIB. To be specific, the ion beam is brought to a focus on the sample. The focused probe that is just 2.5 angstroms in size. The probe is rastered across the sample one location at time, corresponding to each pixel of the final image. The grey scale of each pixel is determined by the abundance of secondary electrons (for example) that are collected at that location.
Overview of the Helium Ion Microscope

Verbal Accompaniment: Today, 4 years after the introduction of the technology, the ORION has established itself as the pinnacle of imaging by virtue of its high resolution, surface sensitivity, and other capabilities. The high magnification image of asbestos fibers, carbon nanotubes, and graphene, reveal details not visible in the SEM.

FOV: 300 nm
Single Walled Carbon Nanotube
Why is Helium So Different?

Verbal Accompaniment: The previous slides described the ORION technology that we are very proud of (rightfully I think). The remaining slides describe the amazing physics of what happens when the ion beam meets the sample.

The diagram on the left shows most of the important comparisons between SEM and ORION. First, it is easily seen that the SEM has a bigger focused probe size (by about 4x). Also the helium beam has a smaller convergence angle which leads to its longer depth of focus.

Under the surface, the electron beam rapidly spreads as it interacts with the electrons from the sample (equal mass collisions produce lots of backscatter events). In contrast, the helium ions stay relatively collimated under the surface because many collisions with electrons are required before they are appreciably deflected. The helium ions are rarely backscattered.

All this means that the secondary electrons that escape from the helium beam are produced very near the surface, and right where the beam enters. Whereas the electrons are often created from deep scattered, and convey deeper, non-local information (SE2).

Verbal Accompaniment: Also, because the electron beam is stopped to close to the surface, almost all of the energy of the beam is deposited in a small volume. For the helium beam, the energy is spread out of a larger volume. For some samples, this means that the helium beam produces less damage.
Charging Advantage with Helium Ions: How?

Three Distinct Differences:
1. The ORION requires only very little probe current. (Typically 0.2 pA)
2. The ORION produces only positive charging (+ ions in – electrons out)
3. The ORION produces only surface charging (relatively easy to neutralize)

Verbal Accompaniment: As this slide describes, the charging of a SEM is fundamentally different from the charging from the helium ion beam. This fundamental difference comes from the fact that an electron cannot leave its charge behind. Whereas, it is generally understood, that most helium ions become neutral right at the surface continue on the rest of their trajectories in a mostly neutral state. The ORION’s surface charging (which will always be positive) can easily be addressed with an electron flood gun. The sub-surface charging of the electron beam cannot be so easily addressed.
Case Study: Growth of Bone minerals on PLLA
(Ian Smith U. of Michigan)

Substrate is a thin coat layer of PLLA. Then immersed in simulated bio-fluid for 1 to 3 hours.

SEM images are not revealing the important surface details due to charging problems, polymer damage.

Verbal Accompaniment: We learned that Ian Smith was having imaging difficulty when using the SEM to imaging HydroxyApatite mineral growth on a PLLA polymer substrate. The SEM had difficulties getting good images due to charging, image drift, and polymer damage. He brought some samples to us to test out in the ORION.
Case Study: Growth of Bone minerals on PLLA
(Ian Smith U. of Michigan)

- High resolution image of micron size growths
- HA growth nucleates on top of previous mineral.
- Not observed at 1H
- New individual crystals were also observed. (arrows)
Case Study: Growth of Bone minerals on PLLA

(Ian Smith U. of Michigan)

- In some cases, intimate contact with surface is seen (left)
- Larger conglomerates show minimal surface contact (nucleation on right)
Case Study: Growth of Bone minerals on PLLA
(Ian Smith U. of Michigan)

- New tests with a spun network of PLLA fibers.
- High open area ratio allows more room for cellular uptake.
- PLLA surface still should be favorable for mineralization.
- What does growth look like?

(1 hour)
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Case Study: Imaging Structures in the Inner Ear

Three areas were studied, with the goal of obtaining direct observation and/or measurement of nano-scale structures that are related to the functioning of the ear.

1. Tectorial Membrane: structure and layout of collagen fibers.
2. Stereocilia, (sound transducers) including
   - The nature of their membranes
   - The tip links, which connect the tips of stereocilia in adjacent rows in their bundles
3. Otoconia: nanostructure of the calcite-containing minerals employed in the ear for balance.

Observe these in HIM without usual staining (OTOTO method) and conductive coating.
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50 nm
Helium Ion Microscope: Images of Biological Materials

Verbal Accompaniment: These are some other collected examples of biological materials imaged in the ORION. In each case, the ORION offers some advantage compared to the SEM in terms of resolution, depth of focus, surface detail, charging, or damage.

As a side note: The ORION gets its highest resolution images from secondary electrons. But other generated particles can also be used. We have experimented with backscattered helium, photons, transmitted ions (from thin sample), etc. There is a lot to explore! We also have an energy spectrometer for backscattered helium that helps us to measure film thickness or identify unknown materials (e.g. distinguish Ni vs. Cu).
Helium Ion Microscope: What is there beyond imaging?

Verbal Accompaniment: To be accurate, the ORION is classified as a focused ion beam (FIB) and this means that it can do a lot of useful things that we normally associate with gallium FIBs. At first, we didn’t recognize or appreciate or promote the fact that the helium beam could be used for nanofabrication. However, some of the early adopters of this technology began developing these applications. We expect to see this one instrument serving two valuable roles: providing unequalled imaging, and providing precision nanofabrication.
Helium Ion Microscope: What is there beyond imaging?

- Lithography with a helium beam
- Beam chemistry with a helium beam
- Implantation of helium atoms
- Hole drilling by helium sputtering
- Milling with a neon beam

A standard resist was patterned with the helium beam to produce this dot array


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FOV: 6 μm
Structures (including a high aspect ratio pillar) were created by patterning the helium beam in the presence of a tungsten precursor gas.

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Membrane folding was achieved by implanting stress in the Silicon Nitride film. Higher (lower) beam energies bent the membrane up (down).

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FOV: 500 nm
This hole was drilled with the helium beam in a MgO crystal. It is 10 nm wide and 300 nm deep.

(McVey ...to be published)
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- Milling with a neon beam

FOV: 1.9 um
A neon beam was used to produce this cross section of an aluminum post.

(Shida Tan, Richard Livengood, Darryl Shima, John Notte, Shawn McVey, GFIS & LMIS Charged Particle Material Interaction Study for Semiconductor Nanomachining Applications ...submitted for publication in JVSTB)
Helium Ion Microscope: What is there beyond imaging?

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FOV: 2.5 um
The neon beam was used to produce this lamella of a semiconductor device.

(Farkas, Rahman ...to be published)
Summary:

1. The Helium Ion Microscope is a new technology presently being evaluated for various imaging applications
2. The interaction of the helium ion beam with the sample offers some unique advantages relative to the traditional electron beam:
   - Higher Resolution
   - Greater Depth of Focus
   - More surface specific imaging
   - Reduced charging artifacts
   - Reduced sample damage
3. The same technology can be used for fabrication of structures at the nanoscale.

Verbal Accompaniment: Thank you very much for your attention and your interest. Please do not distribute this presentation beyond the registered attendees of the 2010 Biointerface Conference in Atlanta.

Updated literature concerning the ORION and other Zeiss products can be attained through the Carl Zeiss web page.