



Challenges and Perspectives in FIB Workshop Programme

10.00 - Registration + Tea & Coffee

10.30 - FIB SIMS

11.00 - Developments in Instrumentation - an FEI Perspective

11.30 - Use of Cryo-FIB & Cryo-SIMS in Analysis of Biomaterials

12.00 - Lunch

1.15 - FIB Instrumentation for Space Developments

1.45 - FIB for Biomaterials & Tissue Engineering

2.15 - Short Presentations

3.00 - Tea & Coffee

3.30 - FIB in Materials Science

4.00 - Developments in Instrumentation - a Hitachi Perspective

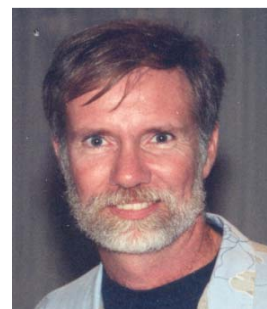
4.30 - Different Application Fields for Ion Beams

5.00 - End

Challenges in FIB –SIMS

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FIB-SIMS is one of a limited number of small area elemental analysis techniques and a very limited number of small area methods that can detect hydrogen and provide isotopic information. Improvement of detection sensitivity would significantly enhance the utility of this method. In general, the statistics of detection for regions less than 100nm limit most applications to matrix elements. However, if it is possible to enhance secondary ion yields by a factor of 30 or more, then most of the transition metals would fall into the easily detected regime.

A review of prior FIB-SIMS analyses is provided, particularly the mineral and biological applications by R. Levi-Setti and J. Chabala at the University of Chicago, semiconductor cross section and depth profiling analyses by FEI Company, and the 3-dimensional analyses by Tomiyasu et al. at Tokyo University. Use of cross section specimens from ion implanted materials allows quantification of the FIB-SIMS elemental analysis images.

Earlier experiments with a flood gas, such as oxygen or water vapor, showed significant secondary ion yield enhancement and a time-of-flight SIMS study using cesium beam as enhancing species showed greater than factor of 100 increase for analysis of arsenic in silicon. Initial measurements at North Carolina State University using 5 keV O_2^+ enhancement for analysis with a 25 keV Ga^+ beam on a bulk doped boron in silicon sample showed a secondary ion yield increase of a factor of approximately 50 for boron and silicon. A detailed investigation is planned for the secondary ion yield enhancement as a function of the bombardment parameters for both O_2^+ and Cs^+ primary beams and for various flood gas species.

Focused Ion Beam Application Drivers and Solutions

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In the last twenty years focused ion beams (FIB) systems have moved from being systems built primarily to investigate fundamental FIB capabilities such as lithography, milling, imaging and gas chemistry into systems targeted at an ever increasing array of applications. As these applications have become more demanding the result has also been the increased specialization of system capabilities and configurations.

A key driver in these changes has been the needs of the electronics industry. Shrinking dimensions, increased complexity, and shorter design cycles have fueled a need for systems that can help understand how the nano-scale devices are put together, identify sources of defects, and get processes and products to market faster. In all these areas, FIB based systems have played a significant role.

FIB capabilities have also expanded into other applications, such as materials research and “NanoTechnology”, especially where small 3D structures need to be created or analyzed. Like the semiconductor applications, the unique capabilities of the FIB for high resolution imaging and localized material removal and addition have been key to their adoption.

This presentation will discuss the drivers that will push further developments in FIB capabilities. These include the need for higher throughput analysis, higher quality transmission electron microscope (TEM) data, and 3D analysis.

The Use of Cryo-FIB and Cryo-SIMS in the Analysis of Biomaterials

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Chemical fixation procedures are often used prior to the analysis of soft biological specimens. These procedures remove the water and render them suitable for use in a high vacuum. There are many protocols that are designed to preserve the physical or chemical attributes of the specimen. However, none are available that are applicable to all sample types and that preserve all aspects of the sample.

An alternative approach is to use cryo-preparation, where samples are plunged into nitrogen slush, inserted onto a cryo-stage in the instrument and maintained close to liquid-nitrogen temperature. This technique is well known for SEM/EDX analysis, but has not been exploited for FIB and SIMS.

Here we describe the results of cryo-FIB and cryo-SIMS analysis applied to plant cells, and to human fibroblast cells that have been exposed to CoCr metal particles similar to those shed from hip prostheses. In the former technique, a cryo-preparation system has been incorporated into an FEI DualBeam instrument and used to section and image samples. The technique is particularly effective in sectioning material containing components with different hardness, such as metal particles in cells. The usual advantages of the FIB technique such as site specificity are retained. In the latter technique, the cryo-preparation system has been incorporated into a magnetic sector SIMS instrument containing a gallium ion gun. High-resolution SIMS maps from the samples are presented.

Subjects to be covered:

- Cryo-FIB technique.
- Cryo-SIMS technique.
- Analysis of Brake Fern plant, *Pteris vittata*.
Analysis of human fibroblasts exposed to CoCr metal particles.

LIMS and FIB Applications in Aerospace Science – Today and Tomorrow

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The connection between the NANOFIB community and the subjects touched in this talk is the use of a common device in both areas, namely the liquid metal ion source (LMIS). This device is used as a micromachining tool in NANOFIB; in space technology (the subject of this talk) the LMIS is used either simply as a supplier of positively charged atomic particles or as a charged particle source for microanalytical instruments. The former application is concerned with the active control of charging potential of a spacecraft, the latter application in particular represents the use of the LMIS as a primary ion source in an onboard space - probe TOF – SIMS spectrometer for chemical microanalysis of cometary material.

The FIB at ARCS has only recently been installed. Planned applications include: investigation of nanostructured alloys and composites used in the aerospace field, e.g. Ti and Al alloys reinforced by nanotubes and nanoparticles and wear – and temperature resistant coatings. FIB offers outstanding capabilities for preparation of very precisely defined areas like interfaces, inclusions and precipitates. An intended feedback to space science is the micromachining of LMIS tips, used e.g. as micronewton electrical thrusters, for better capillary properties in our 1540 XB FIB workstation.

High Resolution Investigation Biological Cell Tissue using CrossBeam Technology

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To investigate the embedding of implants into bone tissue and connective tissue, or the coupling of recording or stimulation electrodes to neural tissue, the precise knowledge of the interface (structure, chemical composition, internal layers, etc.) between cell tissue and implant or electrode material is essential. In electro-physiology the exact knowledge of the interface between cell tissue and stimulation or recording electrodes contributes to the interpretation of the recorded signals [1]. Furthermore the understanding of mechanisms influencing the behavior of cells on micro structured surfaces could help to optimize the surface of future implants.

The investigation of the interface between cell tissue and hard materials like silicon etc. is mostly limited to the investigation of epoxy replicas [3]. This is due to the limitations of the ultramicrotomy technique for cutting hard materials like silicon, glass, ceramics etc.

Other, previously used, techniques allowing to investigate the real interface in the TEM [2] are very time consuming and not very site specific.

To overcome the disadvantages of the microtome (only replicas can be investigated), we have developed a fast preparation technique that allows a site specific, high resolution investigation the real interface between silicon microstructures and cell tissue at high resolution in a CrossBeam (FIB / FESEM) instrument.

Due to the combination of a high resolution field emission SEM for superb imaging capabilities and a high resolution FIB for cutting this technique allows site specific preparation and high resolution investigation of the internal interface at the nm level. The CrossBeam instrument can also be used to cut a thin lamella out of the substrate, that can be used for high resolution STEM investigation inside the CrossBeam instrument.

References

[1] A. Stett, S. Weiss, P. Gnauck, M. Stelzle, W. Nisch, H. Haemmerle and E. Zrenner, *Investigative Ophthalmology & Visual Science (Suppl.)* 38:41, 1997.

[2] P. Gnauck, W. Nisch, *18th Annual International Conference of the IEEE Engineering in Medicine and Biology Society*, 31. October - 3. November 1996, Amsterdam, The Netherlands, Vol. 2, 686

[3] F. Pfeiffer, B. Herzog, D. Kern, L. Scheideler, J. Geis-Gerstorfer, H. Wolburg, *Microelectronic Engineering* 67-68 (2003) 913-922

Short Presentations from Current FIB Users

Dorothee Petit - "Nanomemmer scale patterning using Focused Ion Beam milling"

Jon Fenton - "Three-dimensional FIB patterning of sub-micron TI-Ba-Ca-Cu-O structures and *in-situ* current-voltage measurements with a liquid-helium-cooled sample stage"

Ooi Thian - "Characterisation of oxidation mechanism of bond-coated superalloys using FIB-SIMS technique"

Jon Silk - "The application of FIB milling, polishing & imaging techniques to an aluminium metal matrix composite"

Kristel Fobelets - "Processing electrical/optical devices"

Focused Ion Beam SIMS in Materials Science

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The interaction of the FIB gallium ion gun with the material surface leads to the emission of a broad variety of species including photons, electrons, ions and neutral atoms. All the 'secondaries' can be used analytically. In this talk we will focus on the secondary ions which are the basis of secondary ion mass spectrometry.

Our FIB system is fitted with a quadrupole mass spectrometer and thus we can perform SIMS imaging and SIMS depth profiling of selected areas of interest.

In this talk we will give a general overview of the SIMS process describing the key SIMS equation that relates the secondary ion intensity to the concentration of the species of interest.

We will present case studies illustrating different ways in which the FIB SIMS capability can be used. These are:

- ^{18}O tracer mapping in SOFC materials
- Corrosion of Venetian glass
- Analysis of Oxygen in aerospace alloys
- Yield enhancement when analysing VLSI chips
- Ion channelling contrast
- Analysis of Raqqa glass
- Superconductor multilayers

RECENT DEVELOPMENTS IN FIB TECHNIQUES IN SITE SPECIFIC 3D STRUCTURAL AND ELEMENTAL ANALYSES

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Capability of focused ion beam (FIB) technique has been drastically improved after the development of the FIB micro-sampling technique^[1]. In particular, positional accuracy in transmission electron microscope (TEM) sample preparation is now 100nm or better and sample extracted from a specific site can be fabricated in any shape and thickness. Recently, we developed a FIB-STEM (TEM) compatible sample rotation holder which allows rotation and tilting of an extracted micro-sample in both instruments^[2]. The holder has a needle-shaped sample stub, which is inserted in the center of rotation mechanism of the holder. In this technique, TEM sample is shaped into a pillar and mounted on a sharpened tip of the sample stub perpendicularly. The shapes of the sample, the stub and the holder allow TEM observation of a sample from an azimuth of 360-degree and multi-directionally observed images are used for 3D structural analysis. For quick characterization of the 3D structure, animated image was made by Hitachi EMIP software. Electron tomography using SIRT(simultaneous iterative reconstruction technique) was also carried out for 3D reconstruction. Depending on the materials and the purpose, imaging mode was chosen from among bright field (BF)-, dark field (DF)- and high angle dark field(HAADF) -STEM(TEM) . BF-STEM mode was used for the imaging of resin embedded biological tissues^[3] and polymers. Metals, ceramics and semiconductor devices, containing crystallized materials, were mainly observed by HAADF-STEM. Distribution of nano-crystalline in an amorphous metal matrix of same composition as the crystalline was observed by DF-TEM using a hollow-cone beam illumination. , Energy dispersive X-ray (EDX) mapping was used in the reconstruction of the distribution of elements of low contrast and/or low concentration. 3D distribution of dopant As in a Si device successfully reconstructed by this method. Low energy Ar ion beam of 200V was applied to clean a surface of FIB prepared Si sample and multi-directional observation of high resolution TEM images as well as electron diffraction patterns were successfully carried out. Thus, the dedicated FIB system combined with the micro-sampling technique enabled us to carry out 3D analysis of elemental distribution and 3D structural analysis of a specific site at atomic resolution.

References

1. T.Ohnishi et al. *Int. Symposium for Testing and Failure Analysis*(1999) 449-453
2. T.Kamino et al. *Microsc.Microanal.*6,(2000) 510-511
3. T.Kamino et al. *J. Electron Microscopy* 53(5) (2004) 563-566

Different application fields for ion beams

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FIB is a well established technology for nano and micro fabrication tasks since many years. The NanoFIB EC growth project allowed us studying application fields besides well trodden paths and creating a new nanofabrication proof of concept tool. The main focus have been "high speed" surface modification applications (10^{12} to 10^{14} ions/cm²) [1].

E.g.:

- Creating magnetic domain walls by locally destroying the magnetic properties of thin films.
- Using gold cluster films as negative tone resist.
- Locally altering surfaces properties as seeds for selective growth techniques.
- Classically locally remove thin shielding surface layers followed by a CVD process.

As a result the application areas for focused ion beams have been significantly enlarged offering new processes maybe also for your applications fields.

[1] J. Gierak et al., *Applied Phys. A* 80(1) 2005, p. 187-194