Focused Ion Beam Technology Offers New Potential for Rapid Analysis of Hot Samples

Jean-Jacques DUPUY, Lloyd PETO FEI Company
Jean-Jacques.Dupuy@fr.feico.com FEI Company
LPeto@uk.feico.com FEI Company

Focused Ion Beam microscopes (FIBs) have long been used within the Semiconductor, Data Storage Industries and within Academic Institutions. This paper will introduce how FIB technology may benefit the remote handling of hot samples in 5 separate ways. The use of FIB's within the "hot" community is very limited, and by outlining some potential areas of use we hope to learn and develop the applications we have identified here.

Introducing Focused Ion Beam Microscopy

FIB's are very similar to SEM's (Scanning Electron Microscopes) both with respect to their operating principles and the types of results they produce. A charged particle beam is accelerated towards the sample surface at 30kV and images are obtained by collecting the secondary electrons that are produced at the sample surface. The intensity of the signal is matched with the scanning of the spot and the scanning of the image on the microscope monitor.
FIB systems employ a Ga+ liquid metal ION source instead of a Tungsten filament and the accelerating voltage is reversed to reflect the opposite polarity of the charged particles. Electro-static lenses are used in the place of electro-magnetic as the ions have considerably more momentum and require stronger forces to focus them onto the sample surface.
Ion beam imaging is similar in resolution to a conventional Tungsten SEM at 5nm as opposed to 3.5nm. There are some differences in the contrast mechanisms however that are note worthy. Ion Beam images tend to have stronger materials contrast and voltage contrast information, they also have much stronger crystal grain contrasts which is an important quality for metallurgists. There are some images in the presentation showing ION beam imaging resolution and the grain channeling contrast effects available with ion beam microscopy.

There are some other differences caused by the particle surface interaction. The ion-surface reaction differs to the electron-surface interaction in the following ways.

- The ion does not penetrate more than 30nm and so the information collected is truly surface specific
- The ion causes substrate ions to be ejected from the surface (the exact amount is dependant on the ION beam current), thus causing different amounts of controlled milling
- Some crystalline amorphisation will occur at the very top surface (<30nm) due to the impact of the ion

By increasing the ion beam current by many thousands of times(1pA to 20nA), it is possible to switch a FIB from a high resolution imaging mode to a powerful machining tool. This enables the ion beam system to control many aspects of the surface of the sample.
Ion Beams can be used to prepare a metallurgical surface for analysis and imaging, in-situ to the microscope. A field of view of the microscope can be modified without affecting the rest of the sample surface and the analysis to be performed directly to that area (max 1x1mm, min 1x1 micron). Oxides and contaminants can be removed without exposing the rest of the sample to any mechanical preparation or broad ion beam techniques, thus preserving the sample integrity and minimizing the material removed before analysis.
Even oxide layers that are many microns thick can be quickly and easily removed ready for subsequent analysis.

Once a surface is prepared the ion beam system can produce high contrast metal grain images of the surface.

Ion Beam imaging shows the strongest metallic grain contrast of any imaging technique available, with a low image acquisition time. This enables rapid assessments to be made of annealing condition, cracking, corrosion and many other metallurgical factors.

Higher current Ion beams can be used to make precision cross sections through sites that have been identified inside the microscope. This guarantees a cross section placement to an accuracy of no less than 200nm, and to a depth of up to 50 microns from the sample surface.
This enables the user to see the vertical information at any particular position. The face of this cross section is very smooth and no mechanical polishing process is required. The amount of material removed is very small and the sample is not modified in any way during the process (like heating with a laser).
This cross sectioning process is very fast (a few minutes) and the vertical face is immediately available for high resolution imaging with the ion beam. This cross sectioning process takes place inside the microscope and so is able to be done without any additional handling steps.

By combining this site-specific ability to make cross sections into the surface of the sample with the high contrast imaging and grain information contrast, it is now possible to image metallurgical samples in a very important new way. There are some examples here of cross sections taken through the inside and outside surfaces of a coated fuel rod, showing the coating de-laminating, and a plastic deformation region inside the rod, and nano-structured metal grains at the surface of the metal. These can be compared directly with the grain information taken from cross sections made on the outside surface where no coating is visible and where no plastic deformation can be seen.
Both these sets of data were acquired form the same sample which was not prepared in any way other than to cut a 10cm length of rod from the total length.

We have been able to take this process one step further and perform 2 micro-cross-sections at the same location, one transverse, one longitudinal to the length of the rod. Two “corner” sections of the different sides of this rod are shown here.

Site specific cross sectioning can be taken another step further by performing 2 cross sectioning operations back-to-back. This leaves a thin wall of material that stands vertically at the sample surface. By using the ion beam to polish this vertical wall to a thickness of less than 100nm and then to cut around the foil, it is possible to manufacture a TEM specimen from the surface of a bulk sample without having to machine the bulk material in any way. TEM sample preparation can be performed from any surface to a depth of up to 30 microns, this process removes not more than 2.5 x10-5 mm^3 of material and requires no mechanical preparation, no polishing and no broad-beam milling steps.
Once a free standing thin foil has been machined into the surface, a micro manipulator inside the microscope can be used to lift the foil away from the surface and transfer it onto a copper TEM grid inside the FIB and then this can be handled separately. The dimensions of the foil are 20 microns x10 microns x 100nm or less. The volume of material contained in the TEM foil is not more than 10 cubic microns. This combination of imaging, machining and micro-manipulation has the potential to provide a significant improvement in the time and effort required to obtain the highest quality results from ‘hot’ material samples and related materials.

One other type of benefit that FIB systems may lend to these sample types is that FIB also permits Quadrapole SIMS maps to be collected with a spatial resolution of 50nm. This technique may be employed for investigating materials that congregate at grain boundaries in metals (boron for example) This technique is only currently available with a mass range up to 200amu's, and so requires further development before is could be used for isotope distribution studies (for example) but does shows promise because of its lack of sensitivity to radiation when compared to EDX analysis equipment.
The examples shown here are the imaging of BORON concentrations at the grain boundaries of a Nickel alloy (InCo).