Imperial College London

EPSRC

Engineering and Physical Sciences Research Council

NanoFIB Training Day Programme

10.00 - Registration + Tea & Coffee

10.30 - Basic Instrumentation

11.15 - Basic Operation

12.00 - 50% Lunch + 50% FIB Demos

1.00 - 50% Lunch + 50% FIB Demos

2.00 - TEM Specimen Preparation

2.45 - FIB as a Sample Prep Technique for AES, EDS & SIMS

3.30 - Tea & Coffee

4.00 - Materials Structure

4.30 - Chemical Analysis

5.15 - End

Basic FIB Instrumentation and Capabilities

R.J. Young

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This presentation will introduce the basic hardware and capabilities of focused ion beam (FIB) systems. FIB systems are now used in a very wide variety of applications from basic materials and "nanotech" research to the manufacture of semiconductor and data storage components. Although system configurations and performance needs vary between applications, the various FIB systems share the same fundamental hardware and capabilities.

Presentation outline:

- System overview
  - Ion source
  - Focusing column
  - Detectors
  - Imaging and patterning
  - Gas delivery & chemistry
  - Stages and sample handling/manipulation

- Basic capabilities
  - Beam-sample interactions
  - Image contrast mechanisms
  - Sputtering
  - Beam chemistry – etching and deposition
  - Modelling of FIB processes

Basic Operation

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[2] SiVenture (A division of NDS)

The beam-target interaction causes sputtering of the target but also microstructural damage, amorphisation and gallium implantation. These basics aspects will be covered in the context of producing TEM thin sections and the sputtering of bulk insulators in the FIB that often causes problems associated with surface charging.

FIB Methods for TEM Specimen Preparation

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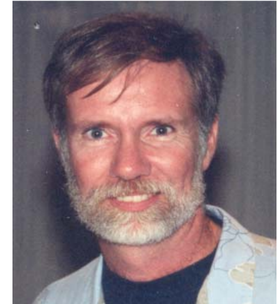
Transmission Electron Microscopy (TEM) can provide a unique means of determining the localised structure and composition of a material, and from there knowledge of structure-property relationships. A potential stumbling block to the use of TEM for materials characterisation, however, lies in the difficulty of making suitable electron transparent samples which generally need to have a thickness 15-20% of the wavelength of light. In recent years, TEM sample preparation has become considerably easier with the development and application of Focused Ion Beam (FIB) sectioning and this in turn has increased the repertoire of material characterisation problems that have been addressed using TEM. Here some of the basic FIB strategies that can be used to prepare TEM samples – both cross-sections and plan-views - will be described and comparisons made between trench and lift-out methods. Such methods will be illustrated with a number of TEM micrographs, and some of the potential pitfalls of FIB sectioning and the ways in which artefacts can be minimised will be discussed.

FIB as a Sample Preparation Technique for AES, EDS & SIMS



F. A. Stevie

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FIB has become an established method for specimen preparation for microscopy analysis using SEM and TEM, but can also provide significant capability in sample preparation for elemental analysis methods. The site specific nature of FIB and the development of lift-out methods, both in-situ and ex-situ, make possible the rapid generation of specimens from a variety of specimen types, including particles.

Defect detection methods for AES analysis of particles provide detection of sub-micrometer particles even on large (200 – 300mm diameter) wafers. With FIB, material can be removed to expose and thus allow AES analysis of buried particles. Once the particle has been exposed, AES survey and image analyses provide elemental identification. The sputtering capability available with most AES instrumentation permits removal of any surface gallium contamination and provides the ability to profile through the defect.

EDS analysis for SEM can be improved by removal of most of the material underneath a feature located at the surface to reduce unwanted x-ray emission originating from surrounding material. For improvement of STEM-EDS, by using lift-out to remove a specimen with thickness on the order of 500nm, the defect or particle can be encapsulated in the lift-out. Quantification of EDS data can be obtained by analysis of a similar sample ion implanted at high dose (on order of 1E16 atoms/cm^2). Detection limits for EDS are not compromised using this approach.

FIB preparation for SIMS using lift-out provides the ability to study cross section specimens at high lateral resolution (<100nm) using instruments such as the CAMECA NanoSIMS 50. Using FIB for removal of material adjacent to the detected area may be useful to improve dynamic range (mesa approach) and to study small areas.

3D characterisation of materials using focused ion beams

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Focused ion beams (FIB) have been used now for over 20 years to sputter holes at specific sites into surfaces (see earlier lectures). The excellent spatial control of focused ion beam sputtering now available means that materials can be serially sectioned using focused ion beams with <100nm precision. This has enabled a new technique of 3D FIB tomography to be developed, which complements more established 3D analysis methods such as TEM and X-ray tomographies, which operate at different lengthscales.

This tutorial will cover the following topics:

- Methods of 3D analysis
- Focused Ion Beam sectioning
- Analysis of 3D FIB tomographic data
- Resolution and errors
- Variants of FIB tomography
- Materials applications
- Use of FIB in other 3D nanocharacterisation techniques

Focused Ion Beam SIMS

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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 I 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 I will give a general overview of the SIMS process describing the key SIMS equation that related the secondary ion intensity to the concentration of the species of interest.

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

- Corrosion of Venetian glass
- Analysis of Oxygen in aerospace alloys
- Yield enhancement when analysing VLSI chips
- Ion channelling contrast

Chemical Analysis

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Secondary ions, their detection and mass analysis provides a powerful method of compositional analysis of the target surface. This topic is covered briefly from the basics and an example given of the use of stable isotope tracers with bulk ceramic and composite materials.