Equipment Name:	**Category:**
Scanning Electron Microscopes: 2 Field Emission SEMS (one with Environmental Capabilities) 1 Variable Pressure SEM 1 Dedicated Electron Microprobe 1 Dual Beam Focused Ion Beam 1 Nanoprober (SEM with 4 Scanning Tunnelling Microscope tips)	**C. Particle Characterisation in and ex-situ** **Institute: University of Leeds** **Location: IMR, Engineering Building, Leeds. LS2 9JT. UK** **Contact Details of Technology Expert:** **Name, Rik Brydson** **Phone, +44 113 3432369** **Fax, +44 1133432348** **E-mail mtlrmdb@leeds.ac.uk**

Short technology description/Overview *(approx 300 words)*:

The SEM is used to generate images of the surface and subsurface of a specimen at magnifications typically in the range 20x – 20,000x. This is achieved by scanning a small electron probe across a specimen and simultaneously monitoring the intensity of either *secondary* emitted or *backscattered* electrons, light or X-rays from the specimen at each pixel point – it produces a scanned or *serial image*. The source of the electron probe is normally a "hairpin" filament, but for higher magnification ranges (down to a few nanometres) pointed field emission sources are employed. For successful operation, SEMs require high vacuum conditions inside the microscope column.

Apart from the magnification, the biggest advantage of the SEM over the LM is the much increased depth of field. This arises because the N_A of the SEM (expressed in terms of the SEM *working distance*) is much smaller than in a LM. A further benefit of the SEM is the ability to analyse the spectrum of X-rays generated in the specimen (alluded to above) during electron bombardment and to determine *the chemical composition* of the sample at each point.

Since the specimen is bombarded with electrons it must be conducting; hence non-conducting specimens must be coated (and earthed) with a thin (ca. 10 nm) layer of a conducting material such as gold or carbon. This also improves the image quality of specimens with low atomic number. Certain environmental SEMs can operate with a partial pressure of water vapour near the sample which can allow the sample to be kept hydrated.

There are a number of basic modes of operation of the SEM:

(i) Secondary electron imaging
(ii) Backscattered electron imaging
(iii) X-ray point analysis and mapping

Secondary electron imaging uses low energy (< 50 eV) secondary electrons which have escaped from the top 1-30 nm (depending on the incident beam energy) of the sample to image the *surface topography* of the sample at each point. Principally these secondary electrons arise from the ionization of electrons associated with atoms in the sample surface caused by the impact of the incident electron beam. The yield of secondary electrons which reach the detector is increased at surface asperities and from those sides of the sample which are angled towards the detector.

By tilting the sample and recording two secondary electron images at differing tilts, *stereo pair* images may be formed which provide a 3D view of the sample surface.

Backscattered electron imaging uses much higher energy (up to the incident beam energy i.e. 1-20 keV) electrons which have been backscattered from the top 10-100 nm of the sample

to image the subsurface detail. The yield of backscattered electrons (while also dependent on topography) is strongly dependent on the average atomic number at each point (and to a lesser extent the crystal orientation). Consequently backscattered images provide *atomic number contrast* provided the effects of surface topography have been removed by specimen polishing. Since backscattered electrons are produced from much deeper in the sample than secondary electrons, the image resolution of backscattered electron images is degraded with respect to secondary electron images – this is because the incident electron beam, although focused to a small probe, progressively spreads out with increasing depth within the sample.

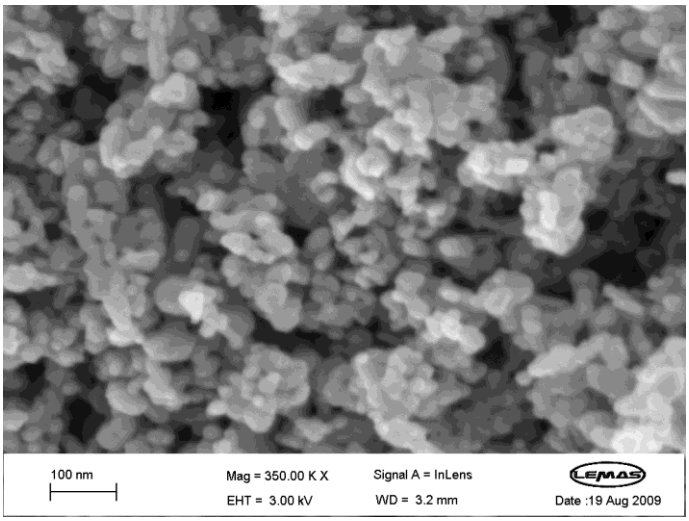
Energy (or wavelength) dispersive X-ray maps are formed by recording the energy (or wavelength) spectrum of X-rays emitted at each point as the electron beam is scanned across the sample. These are produced from typically up to 1 micron below the sample surface and therefore these X-ray maps have a much poorer image resolution than either secondary or backscattered electron images. Different elements in the sample produce X-rays of characteristic energies (or wavelengths), hence it is possible to map the distribution of elements in the sample surface/subsurface.

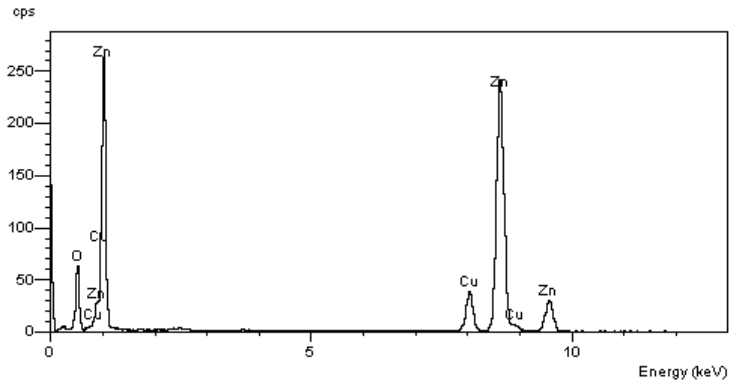
Main Features (Equipment Capabilities):

- Secondary electron imaging
- Backscattered electron imaging
- EDX and WDX X-ray point analysis and mapping
- Heating Stage
- Environmental Capabilities
- 4 probe Conductivity Measurements of nanorods and wires
- Ability to deposit metals and to cut cross-sections in substrates and particles using focused ion be Nanomanipulation possibilities.

Typical Samples & Images:

Powders dispersed in suitable liquid, drop caste and dried onto a conducting stub.





Any further Information: