ELECTRON MICROSCOPY WITH PLYMOUTH UNIVERSITY

Plymouth Materials Characterisation Project (PMCP)

Bringing 3D analysis and imaging, on the nano-scale, to the Heart of the South West, through Plymouth Electron Microscopy Centre.

Plymouth Electron Microscopy Centre (PEMC) provides support and access to a wide range of microscopy solutions by producing extremely high magnification analysis and images at high resolution. PEMC has been working with industry for over 30 years, providing an effective outsourced lab facility for companies throughout the UK, alongside its academic research users; supporting a broad user group, including aerospace, biomedical, microelectronics, photonics, engineering, food, geology and mining, manufacturing, renewable energy and textiles.

We are continually updating our equipment and instrumentation to enhance our capability and create new opportunities for collaboration and the latest of these is the **Plymouth Materials Characterisation Project** which is designed to bring the "big company technology" of FIB-SEM to businesses in Devon (focused ion beam, scanning electron microscope).

As shown overleaf, FIB-SEM can be used to verify product quality, identify causes of failure and inform new product development, by giving users a 3D view of their specimen.

Thanks to European Funding, PEMC is able to offer 40 Devon businesses free use of the FIB-SEM and our Technical Specialists, to aid their company, using an R&D approach to new product development.

To find out whether your companies is eligible for 30 hours of free support from PMCP and to discuss how you could make use of the FIB-SEM, please contact:

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What Could FIB-SEM Do For Your Company?

Focused ion beam–scanning electron microscopy (FIB-SEM) can be applied to a broad range of industrial applications, such as aerospace, automotive, bio-medical, construction, energy, geology and mining, life sciences, marine, micro-electronics, packaging, photonics and renewables. Such examples involving 3D analysis include:

- Studying pore size and sintering levels in ceramic components
- Studying grain size and orientation distribution in metals
- Studying alloying constituents and inclusions
- Measuring and confirming layers in semi-conductors
- Studying phase distributions in mining rocks
- 3D investigation of biological tissue for medical research

Nano-fabrication

There are several different types of nanofabrication that can be utilised with FIB-SEM, such as:

- Nano-patterning to create nano-fluidic channels in a silicon master stamp (see Figure 1)
- Lamellae sample preparation for use with the STEM detector or TEM instrument (see Figure 2). Lamellae samples can be used to study different phases from a specific area with high spatial resolution
- In-situ deposition with various gaseous sources:
    - For the protection of the region of interest
    - Create conductive pathways
    - Assisted ion beam milling with XeF_2

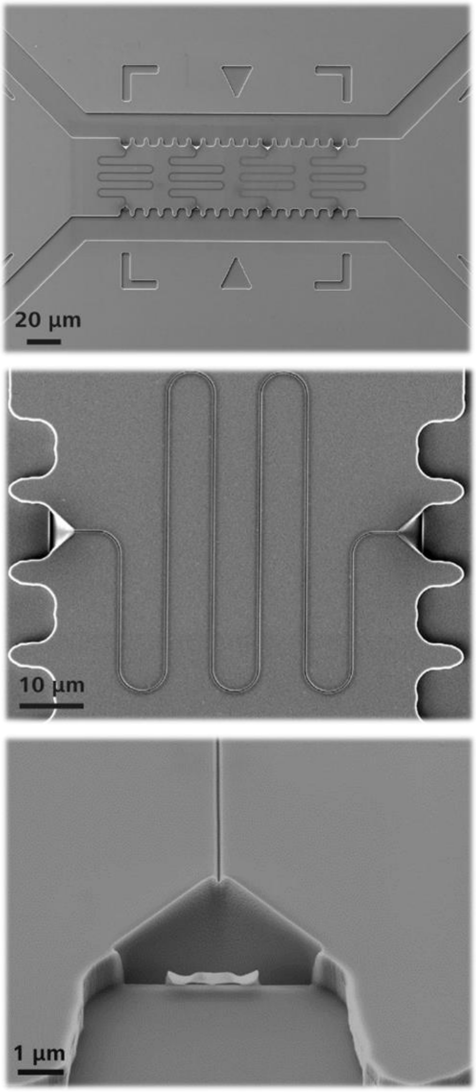

Figure 1 - Nano-fluidics channels fabricated by FIB in a silicon master stamp

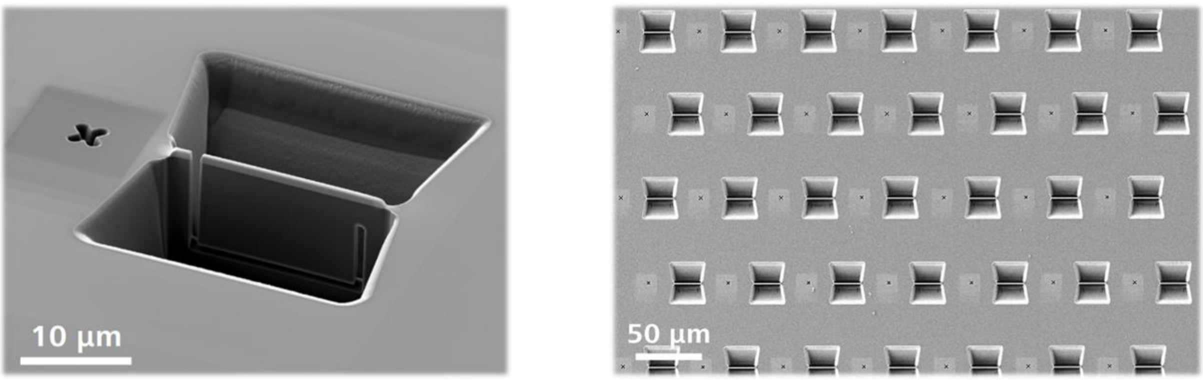

Figure 2 - TEM sample preparation (left) and an array of TEM lamellae prepared automatically (right)

Cross-Sectional Chemical Analysis

The FIB-SEM has the capability to create and analyse cross sections in almost any type of sample, through various electron imaging and chemical analysis.

Figure 3 shows a working example of studying a cathode from a lithium ion battery. Image a) shows a cross-section of the cathode, produced by the FIB. Images b) and c) show the cross-section being imaged in secondary electron (SE) mode, for morphology, and backscattered electron (BSE) mode, for compositional distribution. Image d) shows an energy dispersive x-ray spectroscopy (EDS) map, illustrating the distribution on manganese (Mn) and lanthanum (La).

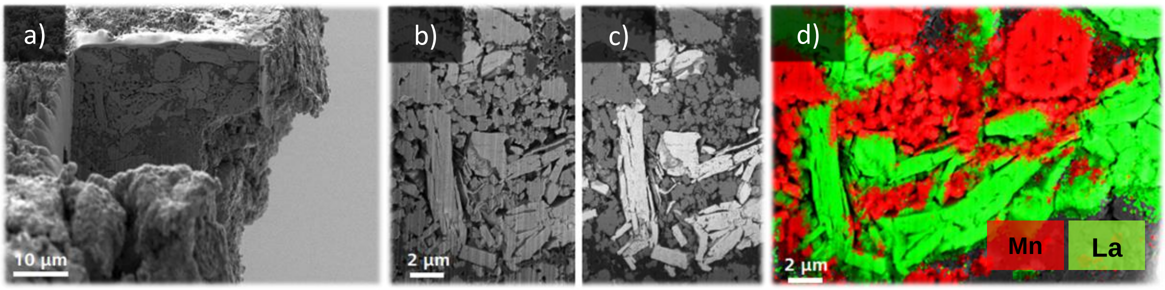

Figure 3 - Cross-sectional analysis of $LiMn_2O_4$ cathode material of a lithium ion battery, using different modes

3D EBSD

The FIB-SEM has the capability of analysing crystalline structures in 3D using electron backscatter diffraction (EBSD), to obtain information about grain size, orientation and distribution. This mode can be used within a range of fields, including metallurgy, mineralogy and ceramics.

Figure 4 shows a working example of studying the grain structure of a copper (Cu) sample. Image a) shows a reconstructed 3D orientation map. Image b) shows an example of viewing individual slices in each of the three planes. Image c) shows the selection of a single grain.

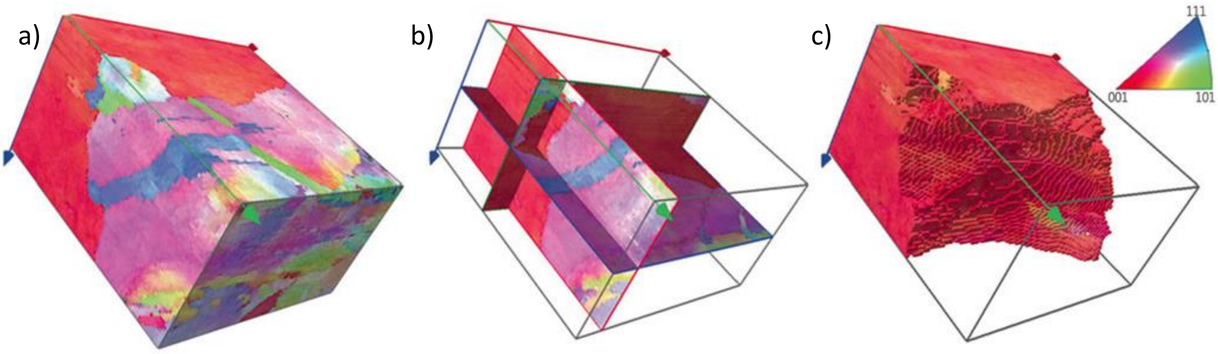

Figure 4 - 3D EBSD analysis of a Cu sample

Tomography

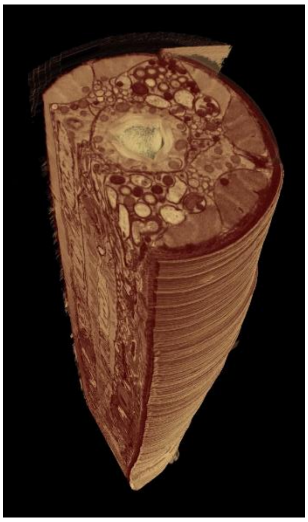

Figure 5 - Tomographic image of a nematode worm

The FIB-SEM has the capability of reconstructing a representative volume with nanoscale resolution at selected areas of interest. This tomography technique is achieved through a slice-and-view process that can be combined with EDS analysis and can be used with any solid material.

Figure 5 shows a tomographic reconstruction of a nematode worm ~1 mm in length and ~50 µm in diameter, showing the internal structure in its entirety.

The tomography technique has been shown to support a wide range of applications, for example; determining alloying element distributions, studying inclusions, measuring pore size and distribution and analysing fillers in composite materials.

Scanning Transmission Electron Microscopy (STEM) in FIB-SEM

Previously prepared thin lamellaes can be imaged with the STEM detector in a variety of modes, including bright field (BF), dark field (DF) and high angle anullar dark field (HAADF). BF mode can be used for cystalline samples, DF and HAADF modes can be used for amorphous materials. Figure 6 is a schematic showing STEM mode.

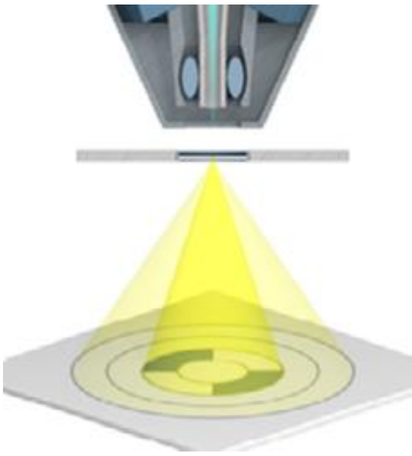

Figure 6 - Schematic showing STEM mode

The STEM detector can also be used simultaneously with EDS. Figure 7 shows a working example of analysing chromium depletion in stainless steel. Image a) shows a STEM image of a grain boundary, with chromium carbide particles along the grain boundary. Images b) and c) show the same area using EDS, indicating the chromium (Cr) and iron (Fe) distribution.

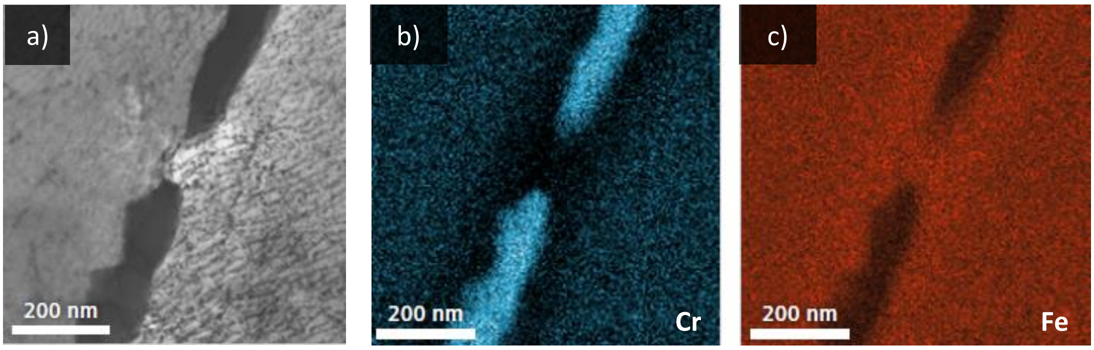

Figure 7 - STEM-BF image and EDS maps showing chromium carbide particles along grain boundary