Scanning electron microscopy

Aims of this experiment

- Understand the principles and operation of a scanning electron microscope.
- Prepare and image physically interesting samples.
- Interpret, as quantitatively as possible, the physical properties of the samples in the light of their microstructure.

1 Principles of the SEM

The principle of operation of the scanning electron microscope (SEM) is widely described, see the bibliography. Briefly, a beam of electrons is focussed to a small spot on the surface of a sample, and the spot is scanned in a raster over a part of the surface. Any localised response of the sample to this scanning spot can be used to form an image, by recording the intensity of the response as a function of spot position. In our microscope, as is typical, the signal is provided by the detection of secondary electrons ejected from the sample by the beam.

At a convenient point when working with the SEM, you should use the literature to find out about it. The web-based training module would be a reasonable place to start. You need not understand the workings of the microscope in great detail, but you should be able to answer the following kinds of question:

- How is the electron beam generated, focused, and scanned?
- Why is a vacuum needed, and how good must it be?
- How are the secondary electrons detected, and why is there contrast between various surface features?
- Why must the sample be electrically conductive?
- What determines the magnification, and the spatial resolution?
- What advantages does an electron microscope have over an optical microscope?

The Leica S430 in the lab is a *scanning* (rather than transmission) electron microscope, having a thermionic electron gun, Everhart-Thornley secondary electron detector, and a dry turbo-pumped vacuum system. It also has an EDX (Energy Dispersive X-ray spectroscopy) accessory, which can analyse the characteristic X-rays coming from the sample.

2 Experimental method

2.1 Outline of SEM procedure

The details of sample preparation depend very much on your sample, but the operating procedure for the SEM is similar in many cases, so you should get familiar with the SEM by imaging a couple of easy samples. A staff member will show you the procedure the first time. The list below should serve as an *aide memoire*.

- **Do not introduce samples that may contaminate the chamber or come loose.** Always check your proposed sample with staff. A bad sample can damage the microscope.
- **Always watch the sample stage for collisions** while slowly moving the door or the stage.
- Mount the sample on an aluminium sample stub, using a conductive tab or silver paint. If the sample is insulating, you normally need to coat it with a thin conductive layer in the sputter system ($\sim$2 minutes sputtering).
- Use gloves or tweezers to avoid contamination the sample and stub with moisture from your fingers; this all has to be pumped away by the microscope.
- Use the camera to check that the sample stage can be moved without hitting anything.
- Cautiously open the chamber door, Place the stub in the sample carousel and gently tighten its retaining screw
- Check the seal is clean, then close the chamber, again watching with the camera.
- Select Vacuum » Status » Pump in the computer interface.
- When the vacuum status is Ready ($\sim$ 10 minutes), turn on the beam: Beam » Beam on
- Start imaging. File » Load state » Setup3.MLC is a good starting setup. Adjust the position, magnification, distance (focus), brightness and contrast to suit. When moving the sample stage, always watch the monitor for an impending collision.
- Use a small scanning window to make adjustments, for rapid feedback.
- When you have a nice but grainy image, increase the data acquisition time to 30-60 s to get a clean image. Save any images you want on the network drive `\\DESKPC\\semdata`
- To change samples, turn off the electron gun, wait until it is fully off, then select Vacuum » Vent. **Move the stage to a safe, low position**. Loosen the chamber door fastening, and wait for the door to open with a finger tip (again, watching the camera). Do not apply force to open the door.
- Minimise the amount of time the chamber is open to the (moist) atmosphere.

2.2 Suggested initial samples

You only need $\sim$1 mm of sample to fill the field of view of the SEM. The sample should fit stably on the stub. Anything that falls off could cause catastrophic damage to the pump, so make sure this can't happen. You could try one or two of the following.

- A small piece of blue Morpho butterfly wing, see further reading.
- A crumb of sintered copper powder taken from the dilution refrigerator development project.
- A broken piece of vinyl record or pressed CD.
- A light dusting of lycopodium powder.
- Anything else with a fine enough texture, approved by staff.

3 Suggested further investigations

You may find enough physics interest suggested by one of your initial samples to do further work on it. Alternatively:

3.1 Structural colours

The recommended reading discusses how the ordered physical structure of iridescent natural materials such as mother-of-pearl, opals etc. (as well as butterflies) gives rise to their unique optical properties. Try some samples of these materials, but be aware there are some tricks in the sample preparation of the very nice images you will find on the web. Your work should go beyond attractive pictures, for example identifying the structures and length scales in your sample that are relevant to the optical and other properties of the sample.

3.2 Imaging domains

Some papers in the recommended reading discuss a relatively simple way of imaging magnetic domains at the surface of a ferromagnet using the SEM. Try this with a single crystal of cobalt, which has a favourable domain structure. Alternatively, you could decorate a piece of computer hard disk with a dilute suspension of magnetic particles and image this. You will also find literature regarding the imaging of electric domains in ferroelectric materials.

3.3 EDX

If you have a suitable sample in mind, staff will show you how to use EDX to identify the composition of different regions of the sample.

4 Recommended Reading

WWW

Australian Microscopy and Microanalysis Research Facility, *Myscope Scanning electron microscope training module*, http://www.ammrf.org.au/myscope/sem/introduction/ (accessed Aug 2019) http://www.ammrf.org.au/myscope/analysis/eds/

Textbooks

D. Chescoe and P. J. Goodhew, *The operation of transmission and scanning electron microscopes*, OUP (1990). A slim, practical book. QH212.S3C44.

L. Reimer, *scanning electron microscopy*, Springer (1998). Thorough. §8.1.1 describes magnetic contrast. QC778R.

J. W. S. Hearle *et al.*, *The use of the scanning electron microscope*, Pergamon Press (1972). Old, but good on sample preparation. QH212.S3H4.

Ian M. Watt, *The Principles and Practice of Electron Microscopy*, cambridge University Press (1997). ebook via library.

P. J. Goodhew and F. J. Humphreys, *Electron microscopy and analysis*, Taylor and Francis (1988). Short. QH212.E4G62.

R. D. Tilley, *Colour and optical properties of materials*, Wiley (1999). §4.12 Mentions blue butterflies. QC355.2

Research Papers

N. Stephant *et al.*, *Investigation of Hidden Periodic Structures on SEM Images of Opal-Like Materials Using FFT and IFFT*, Scanning (2014), pp. 487-499.

F. Schenk *Nature's fluctuating colour captured on canvas?* pp. 98-108 in *Art, Design & Nature*, eds. C. A. Brabbia *et al.* WIT press (2011). By an artist. QC494c

W. Szmaja, *Digitally enhanced type-I magnetic contrast in SEM as a method of domain investigation*, Journal of Magnetism and Magnetic Materials 202 (1999) 201 – 219.

D. C. Joy and J. P. Jakubovics *Scanning electron microscope study of the magnetic domain structure of cobalt single crystals*, J. Phys. D: Appl. Phys. 2 1367 (1969),

G. W. Kammlott, *Observation of Ferromagnetic Domains with the Scanning Electron Microscope*, Journal of Applied Physics 42, 5156 (1971).

Appendix: Fourier transforms

During this experiment, you may wish to apply a Fourier transform (FT) to some of the images that you collect. For example, the image analysis program ImageJ provides a two-dimensional fast Fourier transform (FFT) option. This process samples the entire image, and converts it into spatial frequencies. If a particular periodic repeat exists in the image, strong intensity will be seen at the corresponding spatial frequency $2\pi/l$, where l is the distance in the original image. For the images you are transforming, the smallest unit is one pixel, and the image has a width $W = N_w p_l$ and length $L = N_l p_l$, where p_l is the pixel size and $N_{w,l}$ is the relevant number of pixels.

The largest spatial frequency in the Fourier transform corresponds to variations on the length scale of one pixel in the original image, and the smallest spatial frequency (i.e. one pixel in the FT) corresponds to the largest length scale in the original image, i.e. the width of the total image. The pixel length $p_{FT} = \frac{2\pi}{L} = \frac{2\pi}{N_l p_l}$. The total number of pixels in the image is $L_{FT} = \frac{2\pi}{p_l}$. So, if a particular strong point is found n pixels from the centre of the FT, the characteristic distance in the original image is $l = \frac{2\pi}{n p_{FT}} = \frac{N_l}{n} p_l$.

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