Practical 2P8

Transmission Electron Microscopy

Originators: Dr. N.P. Young and Prof. J. M. Titchmarsh

What you should learn from this practical

Science

This practical ties-in with the lecture course on Microstructural Characterisation of Materials. Its aim is:

1) to help you to understand how transmission electron microscopes (TEMs) operate ;
2) to show you how to form electron diffraction patterns and diffraction contrast images;
3) to enable you to "see" directly features such as dislocations and precipitates;
4) to give you an idea of how such features can be characterised in more detail;
5) to introduce you to the technique of energy-dispersive X-ray microanalysis (EDX).

Practical skills

The JEOL-2100 transmission electron microscope will be operated at first by a demonstrator, but you will expected to be familiar with the operating principles of transmission microscopes (covered in the lectures) and you will be encouraged to take over some aspects of the operation as the experiment proceeds. You will learn basic TEM and EDX operating procedures, how to take micrographs using the digital camera, recording both images and diffraction patterns.

Overview of practical

1) You will examine a thin foil of Inconel-600 alloy, and observe and record images of dislocations, grain-boundaries and any other features of interest under well-defined diffraction conditions.
2) You will make some qualitative chemical analyses of precipitates using energy-dispersive X-ray microanalysis and use this and other information to try to identify them.

Experimental details

1. Transmission Electron Microscopy of Inconel

Load the specimen into a holder and introduce into the microscope. Observe at 200kV.

(i) Obtain a bright-field image. Estimate the grain size of the specimen, varying the magnification as necessary. Record images of typical areas.

(ii) Use the microscope at magnifications 10k-100k. Obtain selected area diffraction patterns from large grains. Tilt specimens and deduce the crystal orientation in one or two cases. Record both Kikuchi line patterns and some typical diffraction spot patterns. Set up 2-beam diffraction conditions with one Bragg reflection strongly excited and observe and photograph in both bright- and dark-field some or all of the following:

- Dislocations and dislocation networks.

- Stacking faults (if present) and micro twins. Again take diffraction patterns.

- Grain boundaries and grain boundary contrast fringes. Low-angle grain boundaries composed of dislocation networks.

- Thickness fringes, bend contours and areas which exhibit both 'mixed-up'.

- Precipitates.

- Any other features of interest.

For each of the above, observe the effect of small tilts on the image contrast. If time permits, explore how the image contrast of dislocations varies when different Bragg reflections are excited. (If you can find a reflection where a particular dislocation is not visible, then its Burgers vector can be deduced. The theory of this is covered in the Advanced Microstructural Characterisation course).

2. X-ray microanalysis

Use the EDX system to investigate the bulk composition and the local composition of precipitates. Identify which types of precipitate are present and make comments on the shape of them.

Safety considerations

The electron microscope operates at high voltage but is quite safe provided that you follow the normal operating procedures as advised by the demonstrators.

Rough timetable

Day 1: Microscopy of the Inconel 600 specimen

Day 2: Complete microscopy and perform EDX analysis. Obtain digital data from the demonstrator.

What should be in the report

Structure and length

As for other practicals.

Questions you should try to answer

You should concentrate on describing and interpreting the results obtained, including as much quantitative data as possible. Address the following questions:

What is the general microstructures of the specimen? Include estimates of grain sizes and dislocation densities. Identify as many microstructural or imaging features as you can (e.g. grain boundary fringes, thickness fringes, bend contours) and describe how their contrast changes with diffraction conditions. Comment on any textures revealed by diffraction patterns.

What directions do stacking faults or slip traces lie along? (Remember the rotation between diffraction pattern and image).

What types of precipitate are present? Where are they situated: within grains, along dislocations, at grain boundaries? What are their sizes, shapes and habit planes? Can you make a guess at their type from the EDX composition information? What diffraction pattern features do they produce (streaks, extra spots?), and why?

Not wanted in the report

You should be able to show knowledge and understanding of the design, operation and characteristics of the electron microscope, the required specimen preparation procedures, and the various mechanisms whereby image contrast arises. However you should **not** devote a major part of your write-up to any of this.