Practical 2P11 - Transmission Electron Microscopy

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What you should learn from this practical

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 diffraction contrast images;
3. to enable you to "see" directly features such as dislocations and precipitates;
4. to introduce you to the technique of energy-dispersive X-ray (EDX).

Practical skills

The JEOL-2100L (200kV) transmission electron microscope will be operated by a demonstrator, but you will expected to be familiar with the operating principles of transmission microscopes (covered in the lectures). You will be shown basic TEM, how to set up various conditions and imaging modes, recording both images and diffraction patterns, and how to use EDX to analyse parts of the microstructure.

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.

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

Key measurements

- Estimate the grain size of the specimen
- Observe twin boundaries
- Observe and photograph in both bright- and dark-field the following:
    - Thickness fringes, bend contours and areas which exhibit both 'mixed-up'. Use the thickness fringes to estimate the sample thickness in this region.
    - Dislocations and dislocation networks,
    - Grain boundaries and grain boundary contrast fringes.
    - Precipitates.
    - Any other features of interest

2. X-ray microanalysis

Key measurements

Use the EDX system to investigate the chemistry at various regions of the material:

- Observe the general appearance of the EDX spectrum in thin and thick parts of the foil
- Measure the bulk composition, and the local composition of precipitates and grain boundaries
- 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.

Under normal circumstances, the microscope room is limited to 5 students plus demonstrators to prevent over-crowding, we are limiting occupancy further this year until the ventilation can be improved.

Additional Video contents

- Introduction (+bonus video of the sample being loaded into the holder)
- Experiment part 1: Focus on diffraction contrast
    - Measuring grain size
    - Selected area diffraction patterns on zone axes
    - Twins
    - Thickness fringes and Bend contours
    - Dislocations
    - Precipitates
- Experiment part 2: Focus on EDX
    - EDX spectra from thin and thick regions
    - EDX spectra from intra-granular (within the grain) precipitates
    - EDX spectra from inter-granular (between grains) precipitates
    - EDX spectra from grain boundaries
- Outro

What should be in the write-up

Style

- Log-book
- Structure and length should be 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.
- 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?

Not wanted in the report

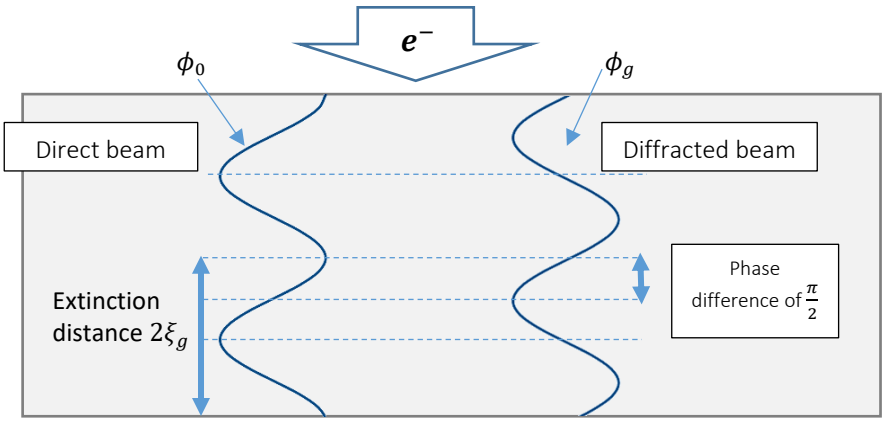
You should be able to show knowledge and understanding of the design, operation and characteristics of the electron microscope, the required specimen preparation procedures, and the various mechanisms whereby image contrast arises. However you should not devote a major part of your write-up to any of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

Appendix

Table 1: Extinction distance table for FCC Nickel, a=3.499 Å

h	k	l	d (Å)	1/d (Å^{-1})	Bragg angle (mrad)	Extinction distance ξ (nm)
1	1	-1	2.02	0.50	6.21	66.17
2	0	0	1.75	0.57	7.17	73.15
2	2	0	1.24	0.81	10.14	103.78
1	1	3	1.05	0.95	11.89	121.20

Figure 1: Wave-function of electrons in a crystal in the two-beam approximation

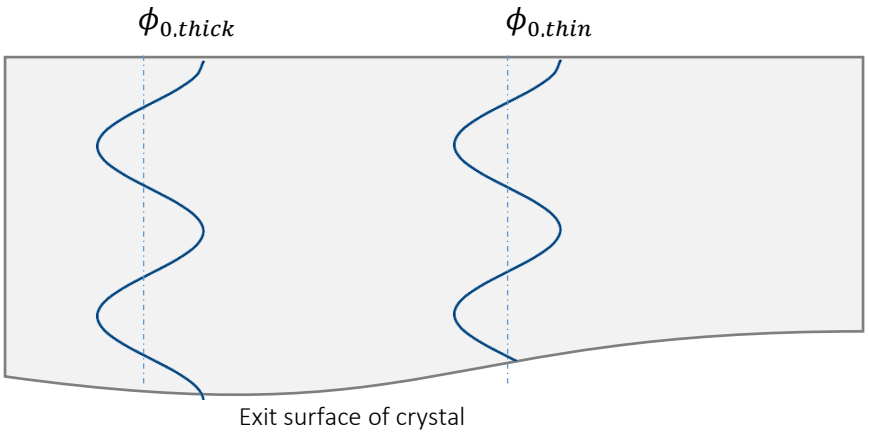


Howie-Whelan Equations

$$\frac{d\phi_0(\boldsymbol{r})}{dz} = \frac{\pi i}{\xi_g}\phi_g(\boldsymbol{r})\, e^{i\,\boldsymbol{g}\cdot\boldsymbol{R}(r)} \qquad \frac{d\phi_g(\boldsymbol{r})}{dz} = \frac{\pi i}{\xi_g}\phi_0(\boldsymbol{r})\, e^{i\,\boldsymbol{g}\cdot\boldsymbol{R}(r)} - 2\pi i s_g \phi_g(\boldsymbol{r})$$

You do not need to know these equations, but it is useful to appreciate that their solutions for the perfect two-beam condition (s_g= 0) and in a distortion-free crystal ($\boldsymbol{g}\cdot\boldsymbol{R}(\boldsymbol{r})$ = 0) reveal a sinusoidal oscillation of the electron wavefunction as it propagates through the crystal, oscillating between the direct beam and reflection **g**. We should see that in this condition, the images we set up from the direct beam (bright field) and from the strongly diffracted beam (dark field) will be reciprocal to each other, since ϕ_0 and ϕ_g are out of phase by $\frac{\pi}{2}$, and the intensity of the electron beam $I = |\phi|^2$.

Figure 2: Origin of thickness fringes



Consider ϕ_0 at two points in the crystal which have different thicknesses. The intensity of the electrons that make up our image depends on the wavefunction at the exit surface of the foil. Therefore, in two different positions of the crystal, one thick and one thin, the wavefunction at the exit surface will be at a different phase. The intensity of the electron beam is $|\phi_0|^2$ so cutting off the wavefunction at a maxima or minima will result in brighter intensity than a region that cuts the wavefunction when it crosses zero. This is what gives rise to thickness fringes, which have a periodicity equal to the extinction distance for that reflection ξ_g when s_g= 0 (perfect Bragg condition).

Figure 3: What's what on the microscope