Introduction to microstructure

1.1 What is microstructure?

When describing the structure of a material, we make a clear distinction between its crystal structure and its microstructure. The term 'crystal structure' is used to describe the average positions of atoms within the unit cell, and is completely specified by the lattice type and the fractional coordinates of the atoms (as determined, for example, by X-ray diffraction). In other words, the crystal structure describes the appearance of the material on an ***atomic (or Å) length scale***. The term 'microstructure' is used to describe the appearance of the material on the ***nm-cm length scale***. A reasonable working definition of microstructure is:

"The arrangement of phases and defects within a material."

Microstructure can be observed using a range of microscopy techniques. The microstructural features of a given material may vary greatly when observed at different length scales. For this reason, it is crucial to consider the length scale of the observations you are making when describing the microstructure of a material.

In this course you will learn about how and why microstructures form, and how microstructures are observed experimentally. Most importantly, microstructures affect the physical properties and behaviour of a material, and we can tailor the microstructure of a material to give it specific properties (this is the subject of the next course). The microstructures of natural minerals provide information about their complex geological history. Microstructure is a fundamental part of all materials and minerals science, and these themes will be expanded on in subsequent courses.

This practical is in three parts. You should aim to spend a total of 60 minutes on parts 1 and 2, and 60 minutes on part 3.

What you should know by the end of the practical!

i. What is meant by the terms 'microstructure', 'phase', 'component', and 'defect'

ii. Some of the different processes that can lead to the formation of microstructures

iii. How we observe microstructures experimentally, and how the length scale of the microstructure observed depends on the resolution of the experimental technique used

iv. Basic principles of imaging and resolution

1.2 Phase/Component/Defect

A 'phase' is taken to be any part of a material with a distinct crystal structure and/or chemical composition. Different phases in a material are separated from one another by distinct boundaries.

A pure substance with a uniquely-defined chemical composition is said to consist of one chemical 'component'. The chemical composition of some materials can be varied continuously between two or more extremes (often referred to as 'endmembers'). These materials must contain, therefore, two or more chemical components. Note that a multi-component material can exist as a single phase if the different chemical components are intimately mixed at the atomic length scale. In the solid state, such mixtures are called '***solid solutions***'.

A 'defect' is taken to mean any disruption to the perfect periodicity of the crystal structure. This includes ***point defects*** such as vacancies and interstitials, ***planar defects*** such as surfaces, twin boundaries, and grain boundaries, and as we will investigate in Course D, ***dislocations.***

You are provided with several photographs and hand specimens. In each case, write down the number of components and phases present, and identify the types of defect (if any) that are present. Make a labelled sketch of each one and add an appropriate scale bar. The examples provided are:

(a) A single crystal of quartz (SiO_2)

(b) A sheet of galvanized steel (Zn surface layer)

(c) An Fe-Ni meteorite

(d) A partially crystallised wollastonite ($CaSiO_3$) glass

(e) Granite

2.1 How microstructures form

Microstructures form through a variety of different processes. Microstructures are almost always generated when a material undergoes a phase transformation brought about by changing temperature and/or pressure (e.g. a melt crystallising to a solid on cooling). Microstructures can be created through deformation or processing of the material (e.g. rolling, pressing, welding). Finally, microstructures can be created artificially by combining different materials to form a composite material (e.g. carbon-fibre reinforced plastic).

Here we will examine some examples of microstructures formed by different processes. The materials will be examined using both reflected-light and transmitted-light microscopes. Ask your demonstrators for advice on setting up the two different types of microscope. Guidelines are printed on separate sheets. Remember to look at the materials at a range of magnifications: some microstructural details may only be visible at high magnification!

a) ***Solidification***. Solidification of a crystal from a melt occurs through a process of nucleation and growth. Below the freezing temperature, small clusters of atoms in the melt come together through random chance to form a small crystalline particle (a nucleus). The nucleus forms a template onto which other atoms can attach. Each nucleus grows into an individual grain of the crystal. When adjacent grains impinge they form grain boundaries. Since individual nuclei form in different orientations, there is no orientational relationship between adjacent grains.

Sample 1 shows the grain microstructure obtained when single-component crystals of olivine (Mg_2SiO_4) solidify slowly from a melt. Examine the sample using transmitted light under crossed polars. Make a labelled sketch of the characteristic grain microstructure and add an appropriate scale bar (focus the microscope on a transparent ruler to get an estimate of the field of view). What factors do you think affect the size of the grains? What other microstructural features can you identify?

b) Phase separation (exsolution, precipitation). A multi-component material can exist as a single phase if the components are intimately mixed (i.e. ***miscible***) at the atomic scale (forming a solid solution). In many materials, miscibility is restricted to a limited range of compositions. The range of miscibility is a strong function of temperature: a material that is happy to form a single phase at high temperature might be forced to unmix into two phases at lower temperature (i.e. the components become ***immiscible***). This process is known as ***phase separation***, ***exsolution*** or ***precipitation.*** We have already seen a classic example of this phenomenon in the case of the Fe-Ni meteorite in Part 1.2.

Sample 2 is a micrograph of a material that has exsolved to yield a two-phase intergrowth. What do you notice about the shapes and orientations of the exsolved phases? What could be the reason for these observations?

When a melt with limited miscibility between components solidifies, one often finds that different phases solidify at different temperatures on cooling. In some cases, the different phases form contemporaneously and become intimately intergrown with each other to form complex (and often quite beautiful) microstructures. We will meet many examples of this behaviour throughout this course.

Sample 3 shows an example of a slowly cooled multi-component melt with very limited miscibility between components. Different phases formed at different temperatures as the melt was cooled. Make a sketch of the microstructure, paying particular attention to the relationship between different phases (i.e. does one phase appear to fill in the gaps between the other phase?). From your observations, can you deduce the order in which the phases crystallised from the melt? Look carefully at the phases at high magnification. What can you say about the phase that crystallised last?

Microscopy

3.1 Abbe theory of imaging and resolution

You have met the principles of diffraction from lattice planes in Course A. Here we revise some of the basic concepts and how they relate to the formation of images in a transmission electron microscope (TEM). We will then use the laser benches to explore the resolution of a system. The basic principles of image formation in a microscope (either an optical or an electron microscope) are illustrated in Fig. 1.

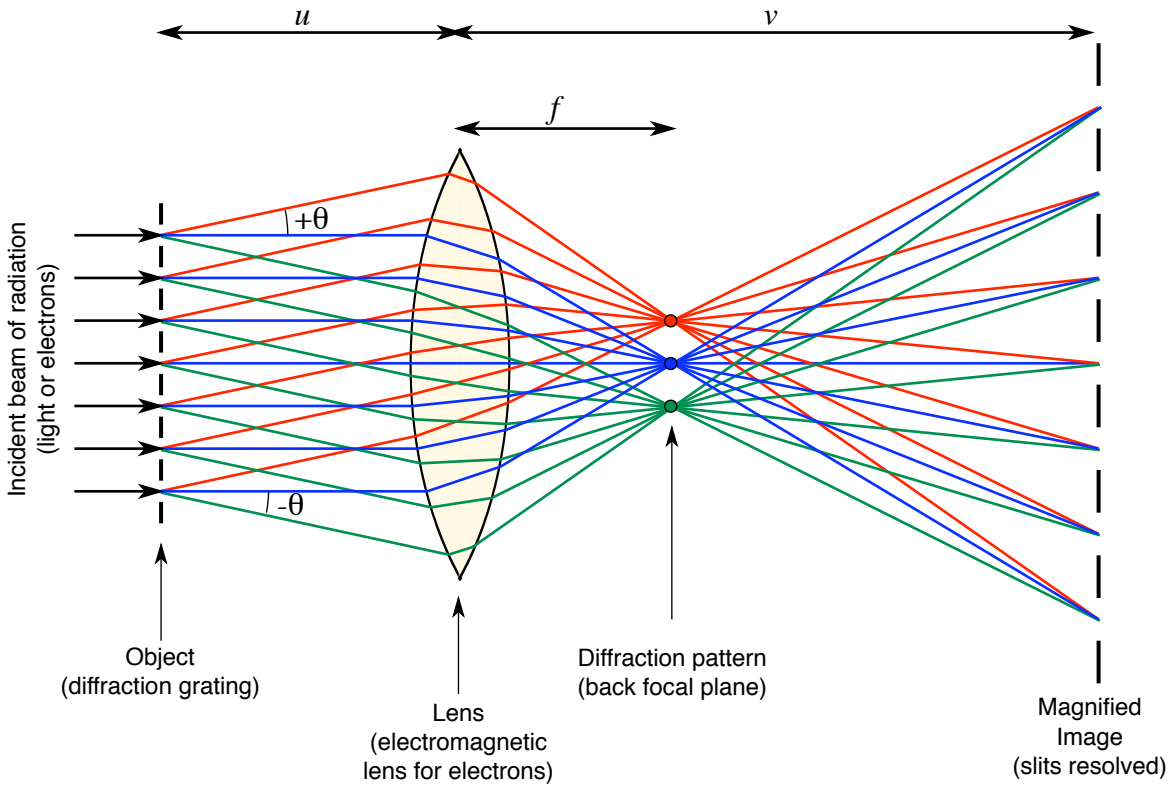

Fig. 1. Abbe theory of imaging using all diffracted spots

Radiation with wavelength λ is incident on the object (in this case a diffraction grating with slit spacing d). Each slit in the grating scatters radiation in a variety of directions. Radiation scattered in a given direction is collected by a lens placed at a distance u from the object and focussed into a point in the back focal plane, located at a distance f from the lens. If the condition $d\sin\theta = n\lambda$ is satisfied (see Course A) then constructive interference occurs and a bright diffraction spot will appear at that point. The image is formed at a distance v from the lens. The image can be considered as the diffraction pattern of the diffraction pattern in the back focal plane.

An image is formed when the equation:

$$1/u + 1/v = 1/f$$

is satisfied. Here f is the focal length of the lens.

3.2 Observation of diffraction gratings in an optical microscope

Transmitted light microscopes are set up on a side bench to examine diffraction gratings of different spacings. When making these observations, it is important that none of the settings of the microscopes are changed except as indicated below.

Three diffraction gratings are set in a single mount. Observe the grating with widest spacing (100 lines/mm) taking care to focus precisely. Next look at the 300 lines/mm grating and determine whether the lines are resolvable. (Any adjustment to the focus should be extremely slight.)

With all the settings untouched, carefully remove the microscope eyepiece and look down the tube with the eye several inches away from it. This gives a view of the back focal plane of the microscope. Observe and sketch the diffraction pattern. Is your observation consistent with that made when the eyepiece was in place?

With the eyepiece still removed, move the 100 lines/mm grating under the objective. How many orders can be seen in the diffraction pattern? (Note: under some circumstances this pattern can be difficult to see at first. It can help to move the eye from side to side.)

Finally, observe both the diffraction pattern and the image of the fine grating (600 lines/mm). Are the lines resolvable, and could they be if the quality of the lens system was improved?

From all your observations, estimate the resolution of the microscope as set up. Compare this with the theoretical limit of resolution, d_{min}, for an optical microscope, which is given by

$$d_{min} \approx \frac{\lambda}{n \sin \alpha}$$

where λ is the wavelength of light (~0.5 μm), n is the refractive index of the medium between the specimen and the objective lens ($n \approx 1$ for air), and α is the acceptance angle of the objective lens. The value of $n \sin \alpha$ is usually printed on the side of each lens as the numerical aperture, N. A.

Essentially the microscope is a tube whose diameter puts a maximum limit on the orders of diffraction maxima which manage to exit the tube to form an image. Making the tube wider increases the maximum resolution, but then the larger lenses suffer from more aberrations which in turn need correction.

Warning: Laser Light is Very Bright

The He-Ne lasers you will be using are of comparatively low power, nominally 0.5 mW, and thus not particularly dangerous. The intensity of the light in the main laser beam is similar to sunlight, and thus the damage which it could inflict on the retina is closely equivalent to that which would result from forcing oneself to look directly into the sun. For this reason never look along the laser beam and be careful to prevent it from reaching anybody else in the laboratory.

3.3 Forming a high-resolution image

Insert mask 12 (a zebra with unusually uniform stripes - see Fig. 2) in the holder in front of the laser and set the screen of the optical bench at approximately 1 m from the mask. Adjust the position of the lens (between 0 and 0.4 m from the mask) so as to form a sharp image on the screen. Calculate the focal length of the lens.

(One or two sheets of paper under the lens mount will reduce the strength of the magnetic clamping and make fine position adjustments easier.)

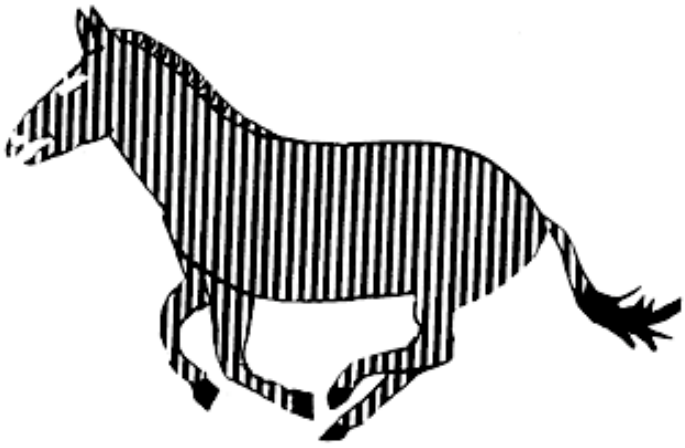

Fig. 2. Zebra mask. Note black lines correspond to clear regions of the actual mask

Replace the screen on the bench with the mirror provided, so that the beam is centred on the second screen at the laser end of the bench (this is to increase the magnification). Adjust the position of the lens so as to focus the zebra on the screen. It will help to focus first on the edge of the zebra.

Briefly place the original screen in the back focal plane, which is one focal length away from the lens, to observe the zebra's diffraction pattern.

From your observations of the limits of resolution of an optical microscope, how could you make the zebra lose its stripes?

Try out your solution (given the adjustable aperture provided – clue!). What is the minimum amount of the diffraction pattern necessary to give the zebra a suggestion of stripes? Draw a sketch of the 'minimum pattern'.

Images formed from a single diffraction spot will not contain any information about the periodicity of the object (i.e. its crystal structure), but they will contain information about the general shape, size, and spatial distribution of phases and defects in the object (i.e. its microstructure).

Images formed from all the diffraction spots are high resolution images which allow us to visualise the periodicity of the underlying crystal structure.

3.4 Real example: Microstructures in 1 billion year old ilmenite-hematite

On the next page are three images of the microstructure observed in a sample of the mineral titanohematite (a solid solution containing hematite, Fe_2O_3, and ilmenite, $FeTiO_3$). As this mineral cooled very slowly over a period of 1 billion years, thin platelets (lamellae) of ilmenite and hematite exsolved. Note that the length scale of the microstructure can vary over several orders of magnitude within the same sample, with platelet widths varying from mm to nm. This is caused by the gradual reduction in the distance over which atoms can diffuse as the temperature decreases - platelets formed at high temperature are thick and widely spaced, whereas those formed at low temperatures are thin and closely spaced.

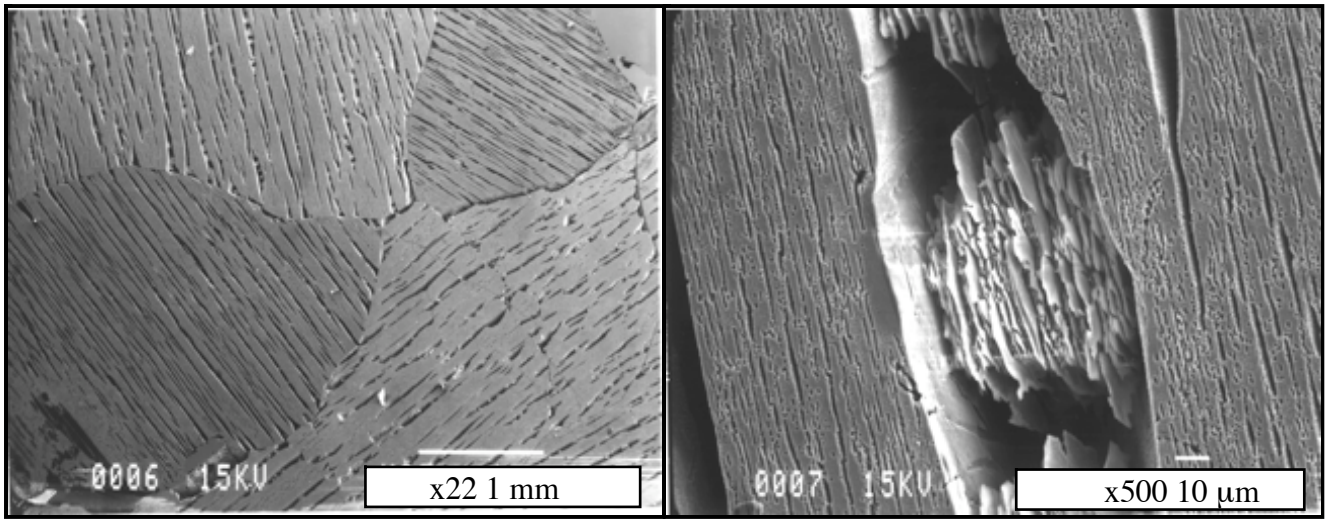

Fig. 3. (a) 'Low' magnification scanning electron microscope images of two-phase intergrowths in etched ilmenite-hematite. Dark patches show where hematite lamellae have been preferentially dissolved out of the ilmenite host. (b) 'Medium' magnification image of a dissolved hematite lamella, which itself contained finer-scale ilmenite platelets.

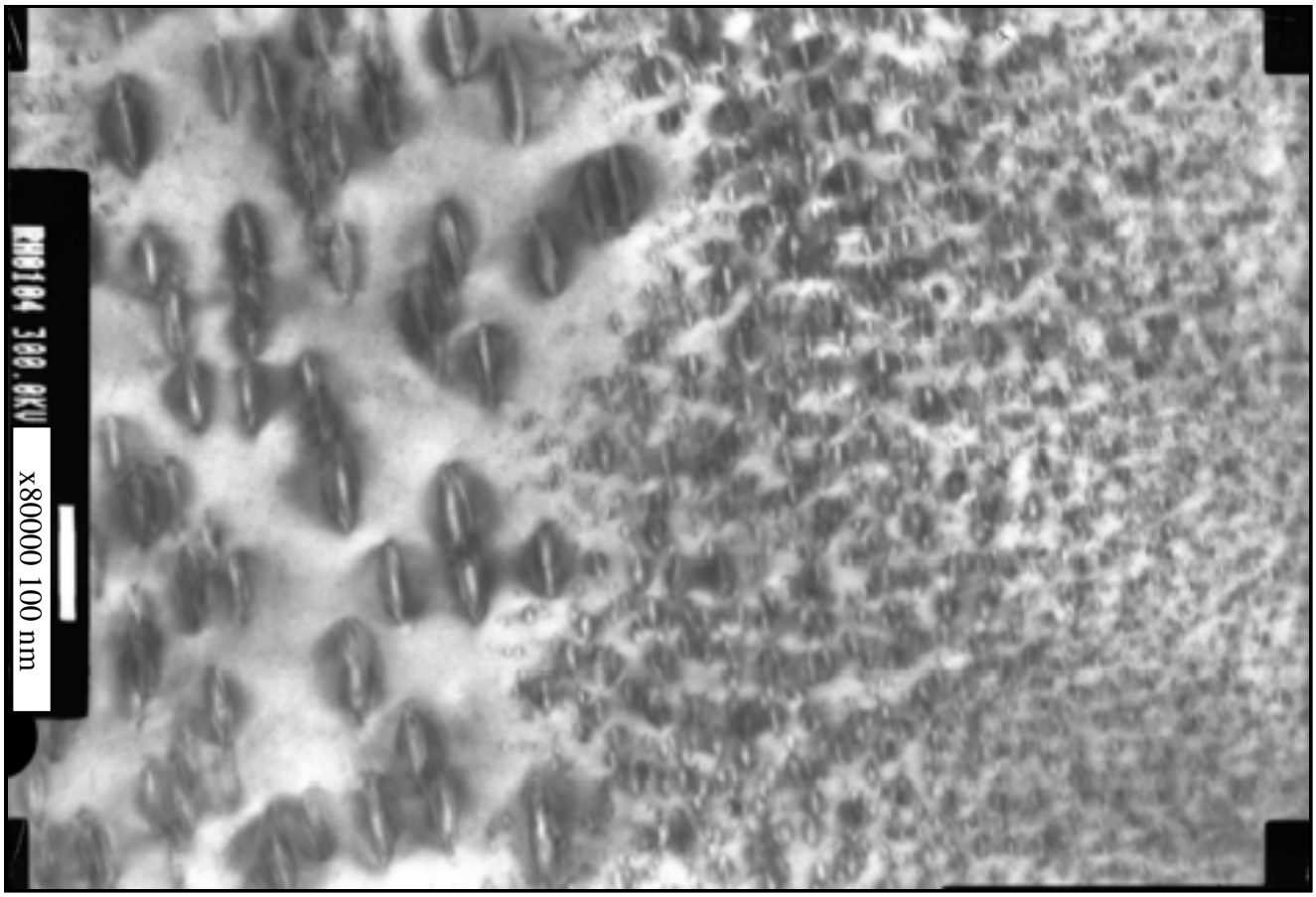

Fig. 4. 'High' magnification transmission electron microscope image of hematite platelets in an ilmenite host. Note that length scale of platelets decreases from left to right, with sizes reaching 1-2 nm (the unit cell length of hematite is 1.4 nm!).