# ORE MICROSCOPY Lab. No. 1

- Introduction: The optical microscope has a wide variety of applications. When examining a range of materials, it is useful to consider using both reflected-light and transmitted-light modes.
- Reflected-light microscopy is used for a range of materials, including metals, ceramics and composites.
- Transmission mode can be used when the specimen is transparent. The specimen is usually in the form of a thin slice (e.g. tens of microns thick).

## The reflected light microscope

- Place the mounted specimen on the stage. The area to be viewed should be positioned approximately beneath the objective lens.
- Switch on the illumination, starting at a low power setting and gradually increase until the desired light level is reached. This can best be judged by increasing the power until a clear spot of light is seen on the specimen surface.
- Use the lowest magnification objective lens to coarsely focus the microscope. Without looking down the microscope, lower the objective lens close to the specimen surface, then use the coarse focus knob to slowly raise it until the circle of light on the specimen appears reasonably sharp. Be careful not to 'crash' the lens into the specimen when focusing ALWAYS focus coarsely by pulling the lens AWAY from the sample.
- The following instructions assume the user is working with a binocular microscope a microscope with two eyepieces.
- Adjust the eyepieces so that they are the right distance apart. This is done by carefully sliding them apart or closer together so that a single image can be seen comfortably.
- Looking through the eyepieces, use the fine focus knob to bring the image into sharp focus.
- The eyepieces should now be further adjusted one of them will focus separately, as many people have slightly different focus in each eye. The image should be brought into focus in the non adjustable eyepiece first using the fine focus knob, and then the other eyepiece adjusted so that it is also in focus.
- If a higher magnification is required the appropriate lens can be moved into place, and the image can be refocused using the *fine* focus knob.
- If focus is lost while at a high magnification, do not adjust the focus using the coarse focus knob, as it would be easy to crash the lens into the specimen. Change the objective lens to a lower magnification and then re-focus as described previously. The magnification can be increased again once the specimen is back in focus.
- Further tips for reflection microscopy

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A further adjustment needed while using the reflection microscope is the position of the specimen. This is adjusted by using the mechanical stage controls, which move the specimen in two directions at right angles to each other. These allow different areas to be observed whilst still looking down the microscope.









**FIGURE 1.2** Schematic cross sections of microscopes illustrating essential components and the path of light through the systems involving (a) whole-field glass plate reflector; (b) half-field prism; (c) whole-field glass plate reflector for conoscopic viewing.

FIGURE 1.4 Schematic illustration of the three reflector units: 45° plane-glass [1]; Smith (2); totally reflecting prism (3).

### Ore & Metallographic specimen preparation

#### 1. Cutting a specimen

When cutting a specimen from a larger piece of material, care must be taken to ensure that it is representative of the features found in the larger sample, or that it contains all the information required to investigate a feature of interest.

One problem is that preparation of the specimen may change the microstructure of the material, for example through heating, chemical attack, or mechanical damage. The amount of damage depends on the method by which the specimen is cut and the material itself.

Cutting with abrasives may cause a high amount of damage, while the use of a low-speed diamond saw can lessen the problems. There are many different cutting methods, although some are used only for specific specimen types.

#### 2. Mounting

## Mounting of specimens is usually necessary to allow them to be handled easily. It also minimises the amount of damage likely to be caused to the specimen itself.

The mounting material used should not influence the specimen as a result of chemical reaction or mechanical stresses. It should adhere well to the specimen.

Specimens can be hot mounted (about 150 °C) using a mounting press either in a thermosetting plastic, *e.g.* phenolic resin,. If hot mounting will alter the structure of the specimen a cold-setting resin can be used, *e.g.* epoxy, acrylic or polyester resin. Porous materials must be impregnated by resin before mounting or polishing, to prevent grit, polishing media or etchant being trapped in the pores, and to preserve the open structure of the material.

A mounted specimen usually has a thickness of about half its diameter, to prevent rocking during grinding and polishing. The edges of the mounted specimen should also be rounded to minimise the damage to grinding and polishing discs.

# A mounted specimen (shows typical dimensions)



## 3. Grinding

Surface layers damaged by cutting must be removed by grinding. Mounted specimens are ground with rotating discs of abrasive paper, for example wet silicon carbide paper. The coarseness of the paper is indicated by a number: the number of grains of silicon carbide per square inch. So, for example, 180 grit paper is coarser than 1200.

The grinding procedure involves several stages, using a finer paper (higher number) each time. Each grinding stage removes the scratches from the previous coarser paper. This can be easily achieved by orienting the specimen perpendicular to the previous scratches. Between each grade the specimen is washed thoroughly with soapy water to prevent contamination from coarser grit present on the specimen surface. Typically, the finest grade of paper used is the 1200, and once the only scratches left on the specimen are from this grade, the specimen is thoroughly washed with water, followed by alcohol and then allowed to dry. The drying can be made quicker using a hot air drier.

Cleaning specimens in an ultrasonic bath can also be helpful, but is not essential.

## 4. Polishing

Polishing discs are covered with soft cloth impregnated with abrasive diamond particles and an oily lubricant. Particles of two different grades are used : a coarser polish - typically with diamond particles 6 microns in diameter which should remove the scratches produced from the finest grinding stage, and a finer polish – typically with diamond particles 1 micron in diameter, to produce a smooth surface. Before using a finer polishing wheel the specimen should be washed thoroughly with warm soapy water followed by alcohol to prevent contamination of the disc.

# Specimen leveling press

Ideally the surface to be examined ٠ optically should be perfectly flat and level. If not, then as the viewing area is moved across the surface it will pass in and out of focus. In addition, it will make it difficult to have the whole of the field of view in focus - while the centre is focused, the sides will be out of focus. By using a specimen leveling press (shown below) this problem can be avoided, as it presses the mounted specimen into plasticene on a microscope slide, making it level. A small piece of paper or cloth covers the surface of the specimen to avoid scratching.

