Metallographic Preparation

Background

Materials engineers can predict the general behavior of materials by observing their microstructure. Besides the crystallographic nature of a material, imperfections inside a material have an even greater influence on the mechanical properties, i.e. tensile, fatigue, creep, fracture toughness, impact properties. Some defects such as missing planes of atoms, called **dislocations**, are responsible for plastic deformation of crystalline solids. Others such as **grain boundaries**, **precipitates**, **twins** and **cracks** alter stress distribution in a material and the accompanying motion of dislocations. Some defects such as missing atoms and dislocations cannot be observed optically except by their effects, i.e. strain, etch pits, slip lines. Other defects such as grain boundaries, twins, precipitates, can be observed readily in the microscope.

Procedures

Metallography is essentially the study of the structural characteristics or constitution of a metal or an alloy in relation to its physical and mechanical properties. The most important part of metallography deals with the microscopic examination of a prepared metal specimen. The metallographic microscope is described in Appendix D on the MSE 227L webpage. Correct preparation begins with the selection of a suitable specimen and continues to the etching stage where the structure of the specimen is revealed. The microscopic examination then defines clearly such structural characteristics as **grain size**, the size, shape and distribution of secondary **phases** and non-metallic **inclusions**; and segregation and other heterogeneous conditions.

These characteristics profoundly influence the **mechanical properties and physical behavior** of the metal. Metallographic examination can provide quantitative information about specimen grain sizes, amount of interfacial area per unit volume, and the amount and distribution of phases. When these and other constitutional features are determined by microscopic examination and the extent to which they exist in the microstructure is known, it is then possible to **predict with considerable accuracy the expected behavior of the metal when used for a specific purpose**. Of equal importance is the fact that, **within limits**, the **microstructure can provide an accurate picture of the mechanical and thermal treatments that a metal has received**.

Preparation of Specimens

The technique for preparing metal sections can be divided into two groups, those processes involving the use of emery papers and coarse abrasives (**grinding)** and the subsequent operations using fine abrasives (**polishing** treatments). Grinding must be carried out carefully in such a way that all microscopic constituents in the surface are preserved and that the grinding medium is not embedded in the sample. To achieve this, the specimen is ground on successively finer grades of emery (sand) paper. **During grinding, the specimen is held with the newly formed scratches at right angles to the scratches introduced on the preceding paper**. Undue pressure should be avoided since the disturbed layer this produces on the surface can extend to considerable depth. For some heat treated alloys and in particular for many of the soft metals, it is an advantage to use paper **thoroughly wetted**. Grinding also

removes surface deformations. After grinding, the specimen is washed thoroughly in water and then polished. Mechanical polishing can best be carried out by holding the specimen against a rotating disc covered with a suitable pad that is impregnated with either a suspension of **polishing alumina in water** or diamond dust oil.

Mounting of Specimens

It is frequently convenient to mount small specimens in bakelite or acrylic to aid specimen preparation, grinding, polishing and etching. You will be given instructions on the use of the apparatus for mounting specimens. The basic idea is that bakelite powder is thermosetting. Therefore the specimen is placed in a tube 2/3 filled with powder. The tube is heated while the powder is compressed. The pressure and heat are removed when the powder has completely melted and the bakelite has set. To maintain orientation small shot are sometimes placed next to the specimen in some identifying arrangement.

Grinding and Polishing Procedure

The following instructions indicate the general method to be used in specimen preparation. The edges of cylindrical metal specimens must first be beveled off to avoid damaging the polishing cloths. For Mg and Al and their alloys, use the aluminum polishing wheels.

Stage	Abrasive	Lubricant	Cloth
Rough Grinding	Silicon Carbide	Tap Water	
Fine Grinding	**Grit 240**		
	Grit 320		
	Grit 400		
	Grit 600		
Rough Polishing	Gamma **Alumina 1.0µ**	Tap Water	Rayon
Final Polishing	Gamma **Alumina 0.05µ**	Tap Water	Rayon

It is important that abrasive is not carried from one part of a sequence to another. Therefore, you must wash both the specimens and your hands between each step. When grinding the specimens, they are rubbed forward in one direction until the surface is completely ground, that is, until only grinding marks due to the particular paper can be seen on the whole surface. For soft metals, further grinding for a short time is advisable after this condition is reached to remove any sub-surface deformation produced in previous operations. The direction of grinding is changed from paper to paper so that the **removal of previous grinding marks is easily observed.**

Polishing is carried out on cloth covered rotating wheels. During the polishing, the specimen should be held firmly in contact with the polishing wheel, undue pressure should be avoided. During polishing, the specimen should be rotated or moved around the wheel to give an even polish. **The specimens must be washed and dried before both polishing steps.**

Etching

Etching is done to bring out the structure of the polished specimen. It is usually performed by subjecting the polished surface to the chemical action of an appropriate reagent. However, the **polished specimen should first be examined unetched**. **Inclusions, flaws, scratches** and

other **defects** can be observed in this way, and if they are identified before etching, subsequent confusion and misinterpretation can be largely avoided. The specimen to be etched is treated by immersion in, or by swabbing with, the appropriate reagent. It is impossible to lay down general rules for the **time of etching**. Usually the desired effect will be produced between **ten seconds and two minutes**. The **specimen after etching should be washed in a stream of running water**. The surface should be **dried** untouched by holding in air current. **When selecting etching times, it is more desirable to under-etch than to over-etch.** If a specimen, after a first attempt is found to be insufficiently etched, the etching process can usually be repeated without further preparation of the surface. A specimen that is over-etched can only be corrected by repolishing and then reetching for a shorter time.

Etching reagents can produce effects in several different ways:
1. The etching of a **pure metal** or a **single phase alloy** is a process of chemical solution of the metal by the reagent wherein the **grains are attacked** at rates dependent upon their orientations with respect to the polished section plane. Because the rate of solution of any one grain differs among different crystallographic planes, during etching a series of well-defined facets develop that are similarly oriented for any one grain, but as a group are differently oriented from those on neighboring grains. The different facet orientations on the respective grains reflect the incident light from the microscope in different amounts. This gives rise to contrast in the reflected light intensities and, thus, the respective grains can readily be identified. In general, grain boundary regions (and twin boundaries) are also delineated because of their higher energies and, correspondingly, higher dissolution rates.

2. The mechanism of etching multiphase alloys is essentially electrochemical in nature. Because of a difference in potential between the structural components when the specimen is brought into contact within the etching reagent, one phase tends to go into solution more readily than the others. The **preferential dissolution** causes the phase to be somewhat roughened and depressed, especially at the boundaries. When observed microscopically, shadow effects are seen and the various structural features are delineated.

3. The operative characteristics of some reagents reveal structural details by preferential dissolution and selectively discolor or stain certain phases of the structure.

<u>The Metallurgical Microscope</u>
Having already described in some detail the methods by which a metallographic specimen is best prepared and subsequently etched for microscopic examination, it is now appropriate to discuss the principles of the metallurgical microscope. A metallurgical microscope differs from a biological microscope in the manner by which the specimen is illuminated. Because of the inability of visible radiation to propagate through a metal specimen, observations are made using **light reflected from the polished surface**. A horizontal beam of light is deflected by a plane glass reflector, upward and through a microscope objective onto the surface of the specimen. A certain amount of incident light will be reflected from the specimen surface back through the objective lens system and then through a second lens system, the microscope eyepiece.

The total visual magnification obtained by the combination of a given eyepiece and objective is equal to the product of the magnifications of the two systems. These magnifications are usually marked clearly on the appropriate parts. When examining a metallographic specimen, the **objective of lowest magnifying power should first be used**. Subsequently, greater detail of particular areas can be obtained by using progressively higher magnifications. The different objectives are mounted on a rotating head, so that their focal planes are very nearly at the same level. After focusing at the lowest magnification, only small adjustments should be necessary at higher magnifications. For examining very fine details, oil immersion objectives can be used. Here a film of oil of greater reflective index than air is placed between the front of the objective and the specimen. A wider angle of light rays and a correspondingly greater amount of reflected light is received by the objective. The oil immersion objectives are usually marked and will need to be used only rarely during the present course. After using immersion oil, the specimen should be cleaned by swabbing gently with cotton wool soaked in the solvent provided.

Photomicrographic Techniques

Materials engineers frequently need to photograph the metallographic work. The image desired is projected into the binocular lens. Once proper focus is obtained, the computer software (Buehler Omnimet Imaging system) is used to further analyze the microstructure.

Glossary of Terms

Alloys. Combinations of metals and elements which enhance the general characteristics of metals.

Dislocation. A line imperfection in the lattice of a crystalline material. Movement of dislocations helps explain how materials deform. Interference with the movement of dislocations helps explain how materials are strengthened.

Etching. Use of a chemical reagent to bring out the micro-structure of a polished metallographic specimen.

Grain boundary. A surface defect representing the boundary between two grains. The lattice has a different orientation on either side of the grain boundary.

Metallography. Study of metallic microstructures. This can be used to determine a number of things including: Heat treatment, mechanical processing, material properties and phases present.

Phase. A material having the same composition, structure, and properties everywhere under equilibrium conditions.

Plastic deformation. Permanent deformation of the material when a load is applied, then removed.

Precipitate. A solid phase that forms from the original matrix phase when the solubility limit is exceeded. In most cases, we try to control the formation of the precipitate to produce the optimum dispersion strengthening.

Twin boundary. A surface defect across which there is a mirror image misorientation of the lattice. Twin boundaries can also move a cause deformation of the material.