Metallography.

Introduction

Metallography is the study of the structure of metals. These days it is also applied to non-metallic materials. Carbon composites, wood, glass, ceramics, and polymers fall into this last category. The examination of a materials structure ranges from extremely coarse to very fine. Viewing by eye to observe rust, cracks, holes are coarse techniques. Identification of individual atoms and dislocations by electron microscopy and X-ray analysis are extremely sensitive and accurate techniques. Normal metallographic examination usually falls in between these two extremes. Grain size and shape, porosity, cracks, second phases, and fracture processes are investigated. From a brief review of these parameters, metallography can be shown to be an extremely powerful analytical tool. For example, the mechanical properties of materials depend upon grain size and shape, and the amount, nature, and distribution of second phases. Thus metallography can 'predict' the mechanical properties of given material. Phase diagram determination utilizes quantitative metallography. The objectives of the experiment are to provide experience of metallographic preparation techniques together with practical examples of fracture analysis and Quantitative metallography.

Metallographic Preparation Techniques

Mounting a Specimen

The first priority is correct selection of the specimen to be examined. Often material is sectioned from components by cutting either by hacksaw or more delicately by a diamond wafering saw. The area to be eventually examined is placed face down on a die; a hollow cylinder slipped over the die and filled 1/3 full with a thermosetting plastic. A ram compresses the powder, and is forced by a hydraulic press into the powder at a pressure of 5,000 psi. A heating jacket around the ram/die melts the plastic, and the pressure removes all porosity and voids. The plastic mounted specimen is then cooled by a finned brass cooler, and removed from the mounting press.

For samples that are heat sensitive, the same process can be used but employing a low temperature hardening epoxy or polyester mounting material. In this case the sample is again sectioned the same way, then placed face down on a bottom of a plastic container. A tube to make up a cylinder is then placed above the sample. After mixing the individual portions of the polymer, it is then poured into the cylinder and left to harden. Hardening can take a short or long time depending on the polymer used.

Specimen Grinding.

Specimens are ground on successively, finer Sic papers lubricated by water. The water also removes "loose SiC particles and metal debris. Coarse 240 grit SiC is used first. Specimen surfaces must be unidirectionally scratched by longitudinal motion along the SiC papers. Once this is achieved, rotations of 9G ensure only 240 grit scratches are on the surface. A condition to be avoided is 'beveling' when the specimen surface is not flat. A ridge on the specimen surface and scratches at slight angles to each other across the ridge are symptoms of it. Provided beveling is not present after 360° of rotations, ie 4-90° rotations, the procedure is repeated on 320 grit, 400, and finally 600 grit. Be careful to wash the specimen between each successive paper to avoid transfer of 240 grit to 320 paper etc. To remove beveling, grind at 90° to the non-flat direction until unidirectional scratches are present. Follow the procedure as usual once beveling is removed.
Specimen Polishing

Polishing is carried out in cloths which have a suspension of alumina and water on them. Diamond is also used, for example on steels. $\text{Al}_2\text{O}_3$, 1.0 $\mu\text{m}$ size is used initially. After squirting same $\text{Al}_2\text{O}_3$ and water on the cloth, turn the wheel on, gently place the specimen on the cloth as the wheel rotates. Do not push specimen into the wheel, just let it gently rest upon, it. Once the 600 grit scratches are removed, repeat the polishing up to the 0.3$\mu\text{m}$ wheel.

Specimen examination

Optical metallographic microscopes are used. These are reflecting rather than transmitting microscopes. Light is reflected from highly polished specimen, and collected which forms contrasting features. Features which do not reflect light back into the objective lens such as scratches appear dark areas, fig. 1. Flat areas act as mirrors and reflect, so appear light. Second phases and non-metal inclusions, whose reflectivity is different from that of the remainder, can be also imaged in the reflecting microscope. These will appear as darker areas. Such materials should be examined unetched. However most materials require etching, which is a process of chemical attack to induce some irregularities on the surfaces which can be imaged. This is how grain boundaries and dislocations art detailed.

Resolution of a microscope, or shortest distance between two points that can be distinguished, is given by:

$$\delta_{\text{min}} = \left(\frac{0.5 \times \lambda}{\text{NA}}\right)$$

$\lambda$ - wavelength of the light

$\text{N.A.}$ - numerical aperture of the objective lens

$\text{N.A.} = \nu \sin \alpha$

$\nu$ - refractive index of medium between lens and object ($\nu$ for air =1, $\nu$ for cedar oil = 1.5)

$2\alpha$ angle subtended by lens diameter at the object.

For green light, $\lambda = 5000$ $\text{A}$, and $\delta_{\text{min}} = 2500$ $\text{A}$ for $2\alpha = 180^\circ$

Practical limitations on $\alpha$ and lens aberrations, chromatic and spherical, usually reduce resolution to 5000 $\text{A}$ or worse. Oil immersion, where cedar oil is placed between the object and the lens improving resolution by 50%.

Modern metallography utilizes Scanning Electron Microscopes which have resolutions of 40 $\text{A}$ and large depths of focus at magnifications approaching 50,000. The same specimen preparation techniques are utilized. However, specimens to be viewed are placed on a mount and must be electrically conducting. The source of illumination in the SEM is electrons. A beam of electrons is excited from an electron source, usually a small tungsten filament. Condenser lenses produce a small beam, which is focused on the specimen by an objective lens. The incident electron beam ejects secondary electrons from the specimen surface. These are collected by a detector which measures the intensity of electrons ejected from the specimen by incident electrons. The incident electron beam 'scans' the specimen across. At the end of the traverse it goes down one line. In this manner the beam scans the area of interest. The electron detector is linked to an oscilloscope which scans at the same rate at the incident beam. Thus, if during scanning the incident beam ejects many secondary electrons from a point on the specimen, a
bright area is shown on the oscilloscope. Similarly, if few electrons are ejected, a dark region appears on the oscilloscope. In this way an image is formed similar to a TV picture.

The wavelength of electrons is 0.0359 Å at 20KV accelerating voltage. However practical limitations of lens manufacture keeps resolution at 30 Å for SEM, but around 1.4 Å for TEM. For the latter machine, atoms can be imaged and individual defects such as a void or a dislocation. This is of extreme importance in the electronics industry, where defects control the properties of integrated circuits.

Specimen Etching

SAFETY is the most important part of etching as very aggressive chemicals are often required to attack the surface of many materials. Always check the Standard Operating Procedure (SOP) prior to any etching of samples.

Etching is chemical attack of a metal. High energy areas are defects, grain boundaries, dislocations and voids for example, which are easily attacked. Thus these are outlined by etching. Different orientations of crystal planes have different spacings from atomic packing, and this leads to some atoms being less loosely held. Thus orientation effects are induced by etching because. Some grains are more easily etched than others.

Different phases have different solubilities in chemical solutions, eg ferrite is dissolved in dilute nitric acid, but carbide phases such as cementite are not. Therefore, etching of plain carbon steels is carried out by a solution of 5% HNO₃ (nitric acid) in methanol, usually called nital.

Care should always be uppermost when etching as it uses solutions which are potentially very dangerous. Two basic techniques exist, swab etching and immersion etching. A ball of cotton is wet by the etching agent and gently rubbed over the specimen in the first method. In the second method, the whole specimen is immersed in etchant. After etching, specimens should be washed in water, rinsed with methanol, and dried prior to examination. Always wash your hands after etching specimens, and wear protective gloves, goggles and lab coat during etching.

References

ASM Handbook on Metallography.
Fig 1a. Light reflects back from polished surface and is collected – bright image.

Fig 1b. Light reflected by scratch is not collected and so appears as a dark region.
## Metallographic Etchants.

### Steel

<table>
<thead>
<tr>
<th>Reagent</th>
<th>Concentration</th>
<th>Use</th>
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<tbody>
<tr>
<td>2% Nital</td>
<td>2 ml Ethyl or Methyl Alcohol 98 ml</td>
<td>Carbon steel General purpose- reveals grain boundaries Ferrite, pearlite and martensite</td>
</tr>
<tr>
<td>Picral</td>
<td>4 gm Ethyl or Methyl Alcohol 96 ml</td>
<td>For all grades of carbon steel</td>
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<tr>
<td>HCl - Picric (Vilella’s)</td>
<td>5 ml Ethyl or Methyl Alcohol 100 ml</td>
<td>Reveals austenitic grain size in quenched and quenched and tempered steel.</td>
</tr>
<tr>
<td>Aqua Regia (dilute)</td>
<td>3 parts Ethyl or Methyl Alcohol 2 parts</td>
<td>Reveals grain structure in Cr-Ni steels. H₂O May be varied to increase or decrease etching time.</td>
</tr>
<tr>
<td>Marble’s Reagent</td>
<td>4 gm Ethyl or Methyl Alcohol 50 ml</td>
<td>For stainless steel and nitrided case.</td>
</tr>
<tr>
<td>Ferric chloride - HCl</td>
<td>5 gm Ethyl or Methyl Alcohol 100 ml</td>
<td>General reagent, reveals structure of austenitic nickel and stainless steels.</td>
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</tbody>
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Note – Picric Acid in dried crystal form is explosive.

### Titanium and alloys.

<table>
<thead>
<tr>
<th>Reagent</th>
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<th>Use</th>
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</thead>
<tbody>
<tr>
<td>Kroll’s etch</td>
<td>2 ml Ethyl or Methyl Alcohol 10 ml 88 ml</td>
<td>General purpose- reveals grain boundaries and phases</td>
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