

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.

Specimen Preparation

SECTIONING, the removal of a conveniently sized and representative specimen from a larger piece, is the first major operation in the preparation of metallographic specimens. The other operations, discussed in subsequent articles, include mounting (not always performed), grinding/polishing, etching (not always performed), and examination of the specimen. Metallurgical sectioning may also be used to prepare specimens for tests other than macrostructure or microstructure evaluation. In this case, the term specimen extraction may be more appropriate. Many of the techniques described in this article apply to extracting metallurgical test coupons for hardness or other mechanical tests, or for scanning electron microscopy or energy-dispersive spectroscopy or other microchemical analysis procedures that may precede metallography in complete materials characterization programs or failure analysis protocols.



Fig 1. An example sectioning device (ref: www.struers.com)

The most widely used sectioning device (fig 1) is the abrasive cutoff machine, ranging from units using thin diamond-rimmed wafering blades to those using wheels that are more than 1.5 mm (1/16 in.) thick, 30 to 45 cm (12 to 18 in.) in diameter, containing silicon carbide particles. Heat is generated during abrasive cutting, and the material just below the abraded surface is deformed. To minimize burning and deformation, a lubricant or coolant is typically used. Wet cutting yields a flat relatively smooth surface. However, because of the abrasion associated with cutting, the structure of the metal or alloy is damaged to a depth of approximately 1 mm (0.04 in.). The exact depth of damage depends on the type of cutoff wheel used, the cutting speed, and the hardness of the specimen. The harder the specimen, the shallower the depth of damage. This damaged layer must be removed by grinding. However, before the specimen can be conveniently ground, it often must be mounted.

MOUNTING facilitates handling of the specimen. A procedure that does not damage the specimen should be selected. Because large specimens are generally more difficult to prepare than small ones, specimen size should be minimized. Standard or typical specimen mounts are right circular cylinders 25 to 50 mm (1 to 2 in.) in diameter. Mounting mediums should be compatible with the specimen regarding hardness and abrasion resistance. Two common mounting materials are thermosetting phenolics, such as Bakelite, and thermoplastic materials, such as methyl methacrylate (Lucite). A thermosetting polymer develops a rigid three-dimensional structure upon being heated and held at 200 to 300 °C (390 to 570 °F). A thermoplastic polymer softens when held at elevated temperatures. 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.

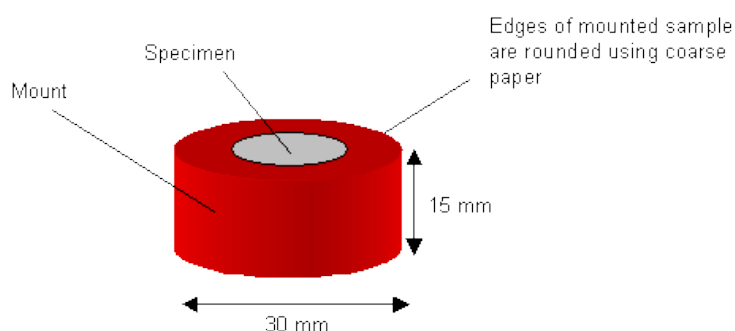
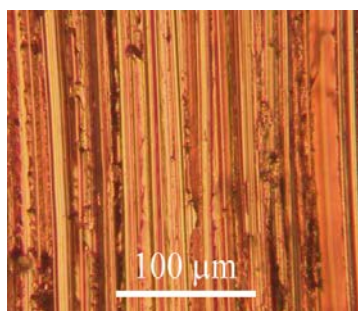
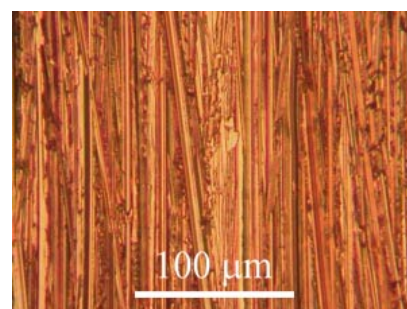


Fig 2. A mounted specimen (shows typical dimensions) and examples mounted specimens (Ref: <http://www.icbl.hw.ac.uk> and <http://phoenixlaser.com>)

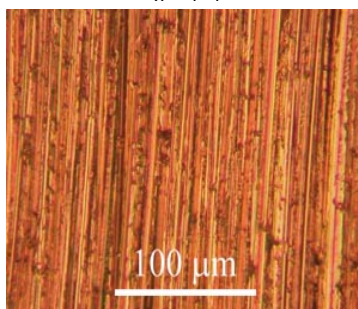
GRINDING is generally considered the most important step in specimen preparation. Care must be taken to minimize mechanical surface damage. Grinding is generally performed by the abrasion of the specimen surface against water-lubricated abrasive wheels (assuming water does not adversely affect the metal). Grinding develops a flat surface with a minimum depth of deformed metal and usually is accomplished by using progressively finer abrasive grits on the grinding wheels. A typical sequence might begin with 180- or 240-grit papers and proceed to 240, 320, 400, 600, 800, 1000 and 1200 grits. Scratches and damage to the specimen surface from each grit must be removed by the next finer grinding step.



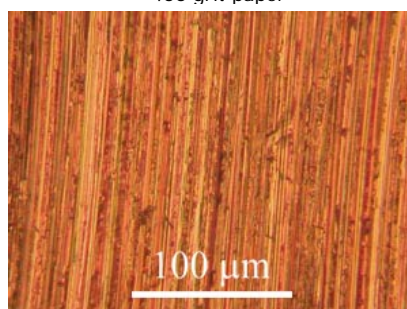
Copper specimen ground with 180 grit paper



Copper specimen ground with 400 grit paper



Copper specimen ground with 800 grit paper



Copper specimen ground with 1200 grit paper

Fig 3. Grinding device and microscopic images (ref: <http://www.icbl.hw.ac.uk>)

POLISHING of the metallographic specimen generally involves rough polishing and fine polishing. In rough polishing, the cloth covering on a wheel is impregnated with a fine (often as small as 1 μm) diamond paste or a slurry of powdered Al_2O_3 in water, and the specimen is held against the rotating wheel. The cloth for rough polishing is frequently napless, providing easy access of the polishing abrasive to the specimen surface. Fine polishing is conducted similarly, but with finer abrasives (down to 0.05 μm in diameter) on a napped cloth.

ETCHING includes any process used to reveal the microstructure of a metal or alloy. Because many microstructural details are not observable on an as-polished specimen, the specimen surface must be treated to reveal such structural features as grains, grain boundaries, twins, slip lines, and phase boundaries. Etchants attack at different rates areas of different crystal orientation, crystalline imperfections, or different composition. The resulting surface irregularities differentially reflect the incident light, producing contrast, coloration, polarization, etc. Various etching techniques are available, including chemical attack, electrochemical attack, thermal treatments, vacuum cathodic etching, and mechanical treatments.

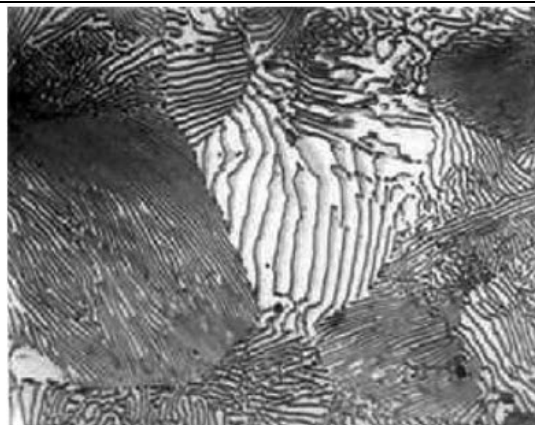
METAL	REAGENT	ACTION
Aluminum and Alloys	0.5% HF in water, 15 seconds, wash in water.	Cleaning and grain boundary etchant.
Aluminum and Alloys	45% HCl, 15% HNO_3 15% HF in water, until grain structure appears, wash in water.	Grain contrast and pitting etch for macroscopic examination only.
Iron and Carbon Steels	1-5% HNO_3 in alcohol, wash in alcohol.	Outlines grains, cleans surface, develops pearlite, does not attack cementite.
Iron and Carbon Steels	%5 picric acid in alcohol, wash in alcohol.	Develops pearlite and related structures.
Austenitic Stainless Steels	25 parts HCl, 5-50 parts of 10% CrO_3 in water.	Microstructure of heat treated steels.
Stainless Steel	FeCl_3 in HCl (saturated solution), add few drops of HNO_3 .	Structure of stainless steel.
Nickel and Alloys	10% HNO_3 , 5% CH_3COOH in water, (electrolytic 1.5 volts), 20-60 seconds, wash in water.	Contrast etch for grain boundaries and microconstituents.
General Purpose In, Sn, Pb, Cu, Ni, Al, Mg, W, Mo and their Alloys	Solution A: $\text{K}_2\text{Cr}_2\text{O}_7$ - 6 grams, NCl - 12cc saturated soln. H_2SO_4 - 24 cc. H_2O - 300cc. Solution B: CrO_3 - 10% in H_2O . Use 50-50 solutions in A and B.	To bring out grain boundaries and to outline microconstituents. The proportions of the A and B solutions may be varied and water may be added to obtain slower attack.

Table 1. List of Etchants Used for Various Metal / Alloys

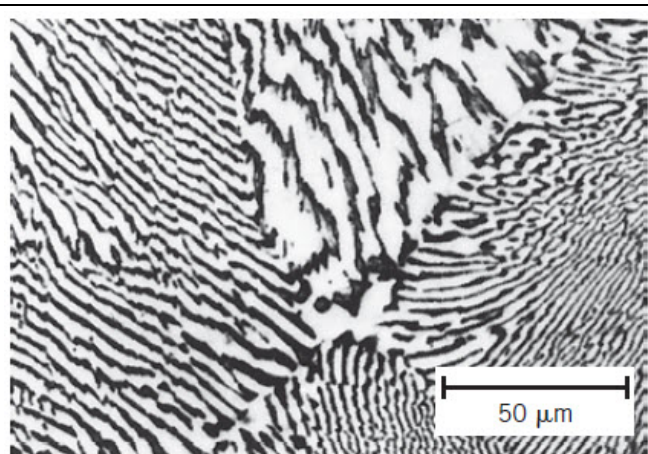
Example Microstructures



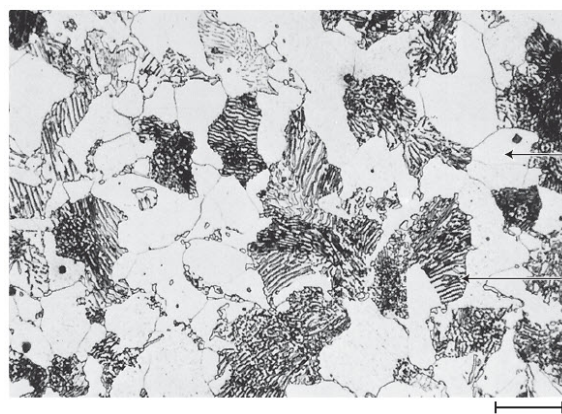
0.2% C steel Photo: J.Gibson



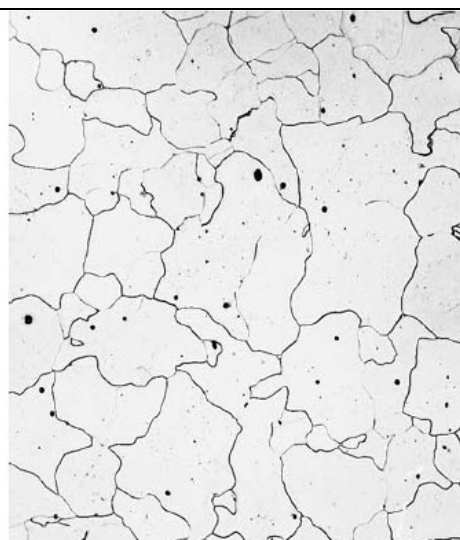
Pearlite in steel. Microstructure of an 0.8% C annealed Photo: J.Gibson



Photomicrograph showing the microstructure of a lead-tin alloy of eutectic composition. This microstructure consists of alternating layers of a lead-rich β -phase solid solution (dark layers), and a tin-rich α -phase solid solution (light layers). 375 X. (Reproduced with permission from *Metals Handbook*, 9th edition, Vol. 9, *Metallography and Microstructures*, American Society for Metals, Materials Park, OH, 1985.)

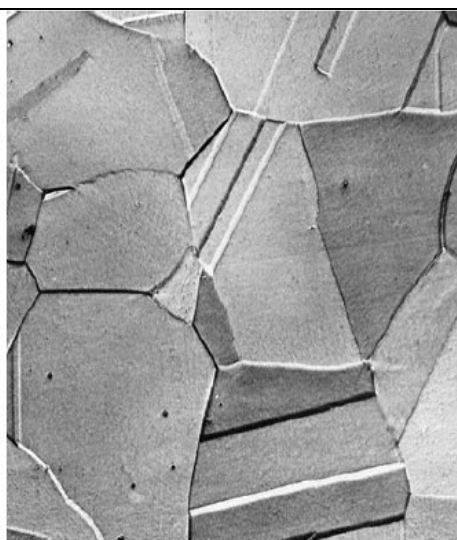


Photomicrograph of a 0.38 wt% C steel having a microstructure consisting of pearlite and proeutectoid ferrite. 635X. (Photomicrograph courtesy of Republic Steel Corporation.)



(a)

200 μm



(b)

50 μm

Photomicrographs of (a) α -ferrite (90x) and (b) austenite (325x). (Copyright 1971 by United States Steel Corporation.)

References

- ASM Handbook Volume 9 *Metallography and Microstructures*
- ASM Handbook, Volume 10: *Materials Characterizations*
- *Materials Science and Engineering*, Eighth Edition, William D. Callister and David G. Rethwisch
- Web

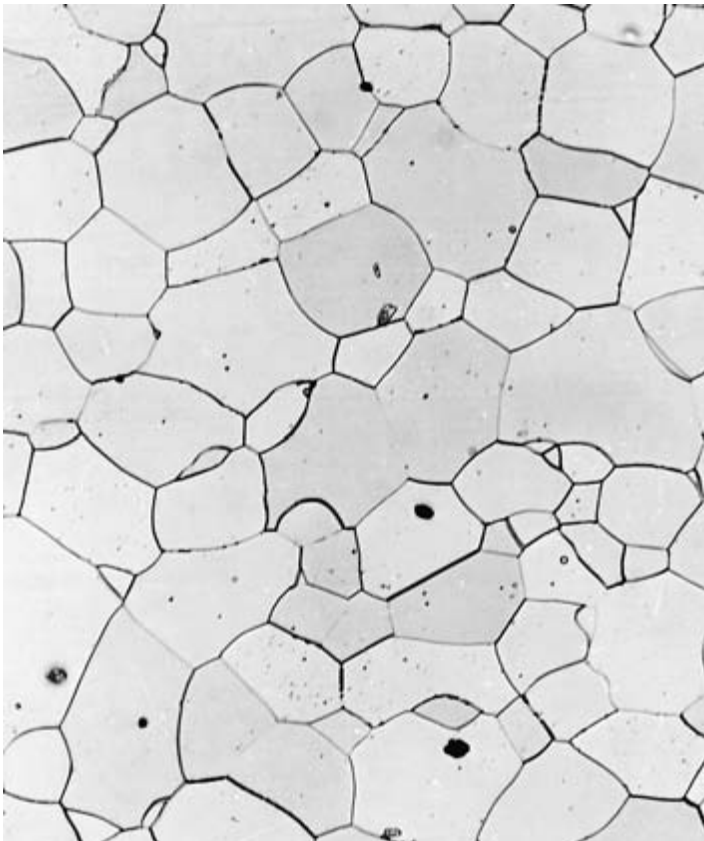
Tasks

1- Give definitions of the following terms:

Imperfection, alloy, solid solution, solute, solvent, interstitial, substitutional, grain, grain boundary, dislocation, point defect, atom percent.

2- Describe and compare the working principles of the SEM, TEM, light microscopy, and AFM (max. 1 page)

3- **(a)** Using the intercept method, determine the average grain size, in millimeters, of the specimen whose microstructure is shown in following figure; use at least seven straight-line segments. **(b)** Estimate the ASTM grain size number for this material.



4. For an ASTM grain size of 8, approximately how many grains would there be per square inch **(a)** at a magnification of 100 \times , and **(b)** without any magnification?