Practical 1P8
Metallography

What you should learn from this practical

Science
This practical ties-in with the lecture course on Microstructure of Materials. It will help you to understand:
1) How to use a phase diagram to predict the microstructure of a material.
2) The effect of rolling and annealing on the microstructure of Cu.
4) The effect of solidification on the microstructure of two-phase Al-Cu alloys.
5) The effect of annealing followed by cooling at different rates on the microstructure of a carbon steel.

Practical skills
You will learn:
1) How to polish and etch metallographic sections of Cu, Al and Fe based alloys.
2) How to use a reflection optical microscope to investigate microstructures at a range of magnifications.

Overview of practical
You will polish and etch a total of 8 metallographic specimens for examination in a reflection optical microscope, recording the observed microstructures with a hand drawn sketch or by photomicrography. You will then use the relevant phase diagram to explain in each case the mechanism of formation of the microstructure and the detailed microstructural features observed. It will be best to use the Olympus BX60M microscopes and digital cameras; do not use DIC. Print and annotate the pictures using Adobe Photoshop and the colour laser printer.

Experimental details
Divide the samples equally so that both of you develop competence in polishing and etching techniques.

Metallographic preparation:
The preparation of each specimen involves grinding, polishing and etching in that order. Most of the specimens have been used previously for this experiment, and only need polishing and etching, but new or damaged specimens will need to be ground first. The experimental techniques of grinding, polishing and etching will be shown to you by the senior and/or junior demonstrators. Make sure that you are careful in using the grinders and polishers, and handling the etching acids.

Optical microscopy:
After preparation, each specimen should be mounted on a glass slide with plasticine, levelled, and then examined in one of the reflection optical microscopes. The senior and junior demonstrators will show you how to use the microscopes. Always examine specimens at low magnification first, and then with progressively higher magnifications. Much information can be lost by not examining at a low magnification.

Recording the microstructure:
Each member of the group should examine each specimen and record the observed microstructure by making a hand sketch or by taking a microphotograph. Structures should be drawn or photographed at a magnification which shows important features clearly. Make sure that you record the magnification in each case.
Specimens:
There are a total of 8 specimens to be examined, listed as follows:

I Copper alloys

A4. Impure copper, chill cast (rapidly cooled in copper mould). Polish with Brasso. Etching: alcoholic FeCl₃ solutions produce a faceted finish and distinguish grains clearly, aqueous FeCl₃ tends to attack or stain impure regions in the material. A low magnification will suffice.

A5. As above, after cold rolling and annealing. Polish with Brasso, etch in FeCl₃. Note the fine regular grain structure and the appearance of annealing twins which were absent in the cast material.

A6. Cu-30wt.%Zn. α-Brass, as cast. At low magnifications a coarse grain structure is apparent, but the outlines of grains are ragged (why?). Within each grain a fine criss-cross pattern of dendrites is to be seen - dendrites being lower in zinc content and relatively bright. Between the dendrites the zinc content is higher and therefore these regions are more heavily etched. Note that although dendrites are all of the same preferred growth orientation, the different sections produce a wide variety of patterns.

II Aluminium alloys

A9-11. Al - 25, 33 and 40wt.%Cu. Slow cooled from the melt. Polish on diamond wheel; etch in aqueous 1%HF + 1%HCl + 1%HNO₃ - take extreme care with these dangerous chemicals. Note the various proportions of the primary phases and the finer eutectic structure which is visible at high magnification.

III Steels

S6. Fe-0.85wt%C. Three samples in the same mount
   (i) Larger diameter sample - normalised from 1100K
   (ii) Middle sample - as (i) but re-annealed at 1100K for 30 min and furnace cooled
   (iii) Third sample - as (i) but re-annealed at 1220K for 30 min and cooled at 1K per minute to room temperature.

Polish using the diamond wheel, etch in nital. Initial large grains of γ (austenite) decompose with a eutectic like reaction (called eutectoid - solid solutions) to form plates of α (ferrite) and Fe₃C (cementite). The specific eutectoid reaction at 1000K is:

\[ γ (0.77\%C) ⇔ α (0.02\%C) + Fe₃C (6.7\%C) \]

This structure is called pearlite (ferrite and cementite) and is only formed during slow cooling (why?). Try and determine where the original γ grain boundaries were and whether growth of the pearlite is directed away from these boundaries. Also, look at the rim of each sample and explain any differences cp central region.

S17. Fe-0.8%C steel quenched into water from 1273K to give an acicular martensite which can be revealed by a relatively heavy etch in nital.

Safety considerations
- The etching is only to be done under supervision.
- Always carry out etching in a fume cupboard, wearing a lab coat, safety glasses and plastic gloves, and always use tongs to handle the specimen.
HF solution: very dangerous, corrosive and toxic. Must use nitrile gloves.

Nital: corrosive, toxic and flammable. Use either vinyl (normally) or nitrile gloves.

Alc FeCl₃: corrosive, toxic and flammable. Use either vinyl (normally) or nitrile gloves.

Splashes on the skin or eyes must be washed with copious amounts of water. Get immediate help from PCT (Paula or Pat). HF splashes require hospital treatment.
What should be in the report

- Give brief details of the experimental methods.
- The annotated sketch or photograph of the microstructure of each specimen should be included in the report, with the magnification indicated at the corner, preferably by a scale marker.
- The relevant phase diagram for each specimen should be consulted and also included in the write up.
- Give a brief description of the main features of each microstructure, and try to explain how it formed. A few sentences for each specimen should be sufficient.

References

Hansen - The Constitution of Binary Alloys
Smithells - Metals Reference Book
A.S.M. - Metals Handbook
W. Hume-Rothery - The Structure of Alloys of Iron
V. Voort - Metallography Principles & Practice
G.A. Chadwick - Metallography of Phase Transformations
Higgins - Engineering Metallurgy