

**UNIVERSITY OF MAURITIUS**  
**FACULTY OF ENGINEERING**  
**MECHANICAL AND PRODUCTION ENGINEERING DEPARTMENT**

**METALLURGY LAB.**  
**MATERIALS SCIENCE MODULE- MECH 2121**  
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**METALLOGRAPHY:**

Metallography is the study of metals by optical and electron microscopes. Structures which are coarse enough to be discernible by the naked eye or under low magnifications are termed macrostructures. Useful information can often be gained by examination with the naked eye of the surface of metal objects or polished and etched sections.

Those which require high magnification to be visible are termed microstructures. Microscopes are required for the examination of the microstructure of the metals. Optical microscopes are used for resolutions down to roughly the wavelength of light (about half a micron) and electron microscope are used for detail below this level, down to atomic resolution.

The most commonly used microscope is the conventional light microscope. In principle, optical microscopes may be used to look through specimens ('in transmission') as well as at them ('in reflection'). Many materials, however, do not transmit light and so we are restricted to looking at the surface of the specimens with an optical microscope. Electron microscope can be used in the transmission e.g. Transmission Electron Microscope (TEM) and to look at the surfaces e.g. Scanning Electron Microscope (SEM)

Microscopy can give information concerning a material's composition, previous treatment and properties. Particular features of interest are

- (I) grain size
- (II) phases present
- (III) Chemical homogeneity
- (IV) distribution of phases
- (V) elongated structures formed by plastic deformation

**Optical Microscopy:**

With optical microscopy, the light microscope is used to study the microstructure; optical illumination systems are its basic elements. For materials that are opaque to visible light (all metals, many ceramics and polymers), only the surface is subject to observation, and the light microscope must be used in a reflective mode. Contrasts in the image produced result from differences in reflectivity of the various regions of the microstructure.

Careful and meticulous surface preparations are necessary to reveal the important details of the microstructure. The specimen surface must first be ground and polished to a smooth and mirror like finish. This is accomplished by using successively finer abrasive papers and powders. The microstructure is revealed by a surface treatment using an appropriate chemical reagent in a procedure termed etching. The etching reagents depend on the material used and after etching the specimen must be washed with alcohol and ether to remove the grease. The atoms at the grain boundaries are chemically more active, and consequently dissolve more readily than those within the grains forming small grooves. These grooves become discernible when viewed under a microscope because they reflect light at an angle different from that of the grains themselves.

When the microstructure of a two phase alloy is to be examined, an etchant is chosen that produces a different texture for each phase so that the different phases may be distinguished from each other.

The maximum possible magnification with an electron microscope is approximately 2000 diameters.

### **Electron Microscopy:**

#### ***Transmission Electron Microscopy (TEM):***

The image seen with a TEM is formed by an electron beam that passes through the specimen. Details of the internal microstructural features are accessible to observation; contrasts in the image are produced by differences in beam scattering or diffraction produced between various elements of the microstructure or defect.

In the TEM, electrons are focused on an extremely thin foil of the material; the beam of electrons interacts with imperfections in the material, causing differences in the fraction of electrons that are transmitted. The transmitted beam is projected onto a fluorescent screen or a photographic film so that the image may be viewed.

Magnifications approaching 1000000x are possible with TEM. The TEM is used to observe dislocations.

#### ***Scanning Electron Microscopy (SEM):***

The surface to be examined is scanned with an electron beam, and the reflected beam of electrons is collected, then displayed at the same scanning rate on a cathode ray tube. The image that appears on the screen, which may be photographed, represents the surface features of the specimen. The surface may or may not be polished and etched, but it must be electrically conductive; a very thin metallic coating must be applied to non conductive materials.

Magnifications ranging from 10 to in excess of 50 000 diameters and also very great depths of field are possible.

### **GRAIN SIZE DETERMINATION:**

The grain size of metals are usually expressed as the American Society for Testing and Materials (ASTM) grain size number. The ASTM has prepared 10 standards comparison charts, all having different average grain sizes. To each is assigned a grain size number,  $n$ , ranging from 1 to 10, the larger the number, the smaller the grains. The designation is based on the equation,

$$N = 2^{n-1}$$

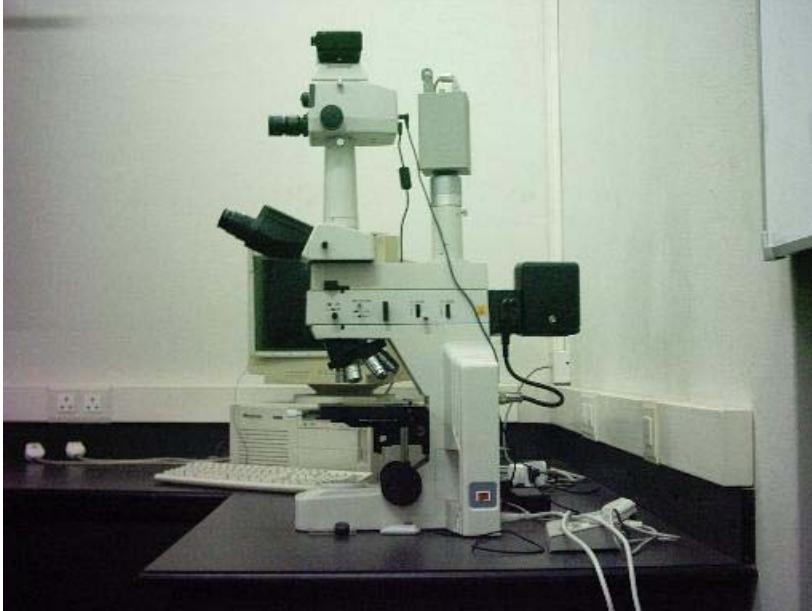
where  $N$  is the number of grains in an area of 1 sq. in at 100x magnification. A specimen must be properly prepared to reveal the grain structure, which is photographed at a magnification of 100x. Thus a steel with  $N=6$  has, on average, 32 grains in an area of 1 sq. in. at 100x. Grain size is expressed as the grain size number of the chart that most nearly matches the grains in the micrograph.

Grain size may also be determined using an intercept method described below.

Straight lines all of the same length are drawn through several photomicrographs that show the grain structure. The grains intersected by each line segment are counted; the line length is then divided by an average of the number of grains intersected, taken over all the line segments. The average grain diameter is found by dividing this result by the linear magnification of the micrographs.

## **SPECIMEN PREPARATION FOR OPTICAL MICROSCOPY**

The examination of materials by optical microscopy is essential in order to understand the relationship between properties and microstructure. Metallography is the study of metals by optical examination. This is most commonly done using a conventional light microscope. However useful information can be gained by examination with the naked eye of the surface of metal objects or of polished and etched sections. Structures which are coarse enough to be discernible by the naked eye are termed macrostructures. Those which require magnification to be visible are termed microstructures.



**Fig 1: Optical Microscope Fitted with 35mm Camera and Digital Camera**

## **MICROSTRUCTURES.**

The preparation of a specimen to reveal its microstructure involves.

- ❖ Sawing the section to be examined
- ❖ Mounting in resins (if sample is too small)
- ❖ Coarse grinding
- ❖ Grinding on progressively finer emery paper
- ❖ Polishing using alumina powder or diamond paste on rotating wheel
- ❖ Etching in dilute acid (2% Nital for steel)
- ❖ Washing in Alcohol and drying

Typical magnifications used are between 50x and 1000x

## **ROUGH PREPARATION.**

The specimen is ground on progressively finer SiC waterproof papers from 120 to 1000 grit, to produce a reasonably flat surface, it is lubricated with water to keep it cool and to remove the grinding products. If the sample is not flat, it might be necessary to remove some material on the lathe or grinding machine first. The sample should be moved forward and backward on the paper until the whole surface is covered with unidirectional scratches. It is then washed with running water to remove debris associated with the grade of paper used. It is then ground on the next finer paper such that the scratches produced are at right angles to those formed by the previous paper. This enables you to easily decide when the scratches from the coarser paper have been completely removed. This procedure is repeated through the range of papers available.

When the specimen has been ground on the final paper, it is generally worthwhile rotating it through and grinding again with less pressure than before. This technique can decrease the time required for the next stage, which is polishing. Before polishing, the specimen and your hands must be washed and dried to remove any SiC particles.

### **POLISHING**

Due to the very small depth of field obtained from an optical microscope it is essential that the surface is flat, in fact it needs to be optically flat, acting as a perfect mirror. The specimen therefore has to be “polished”. This is done using rotating wheels covered with a cloth impregnated with a very fine abrasive compound. The common compounds used are diamond and alumina.



**Fig 2 : An Automatic Polishing Machine with Two Rotating Discs**

### **DIAMOND POLISHING.**

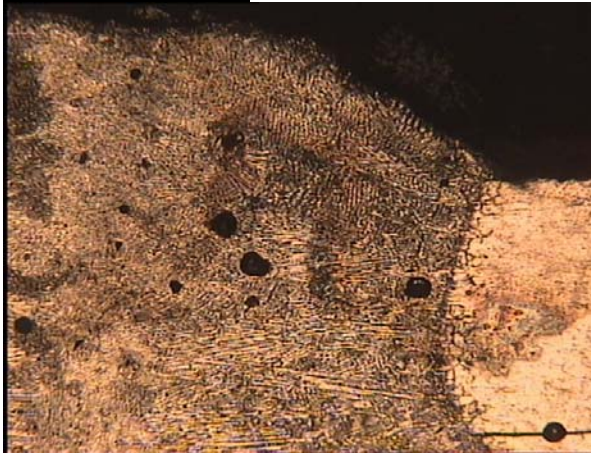
The Diamond paste is available in various sizes 25  $\mu\text{m}$ , 15  $\mu\text{m}$ , 6  $\mu\text{m}$ , 1  $\mu\text{m}$ ,  $\frac{1}{4}$   $\mu\text{m}$  and if required, polishing may be started with 25  $\mu\text{m}$ . When the surface is of acceptable quality, polishing is continued with diamond paste of lower sizes. Separate wheels must be used for the different sizes. Check the appropriateness of diamond paste for the polishing process for a particular material. Other Grinding paste are available.

In most cases two polishing should be sufficient i.e. the 6 $\mu\text{m}$  and the 1 $\mu\text{m}$ . Two separate wheels are used, the first with 6 $\mu\text{m}$  diamond paste and the second 1  $\mu\text{m}$  diamond paste. Both are lubricated with an organic liquid and water must not come into contact with these polishing wheels. The 6  $\mu\text{m}$  diamond wheel is used first. The specimen is pressed down onto the lubricated rotating wheel. It is important not to hold the specimen in one orientation for an extended time as it will cause “dragging” of some microstructural components. For example, small spherical particles can develop “tail” rather like a comet. To avoid this, the specimen is oscillated about its vertical axis continuously. After 20-30 seconds the specimen is removed and rotated through 90<sup>0</sup> in the hand, placed back on the wheel and then again oscillated. This process is continued until the fine scratches from the final paper have been removed.

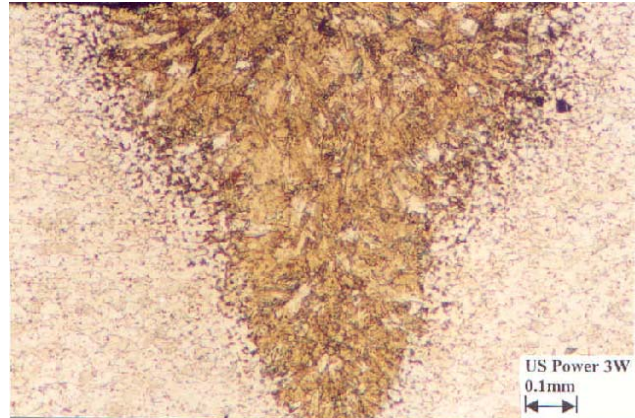
The specimen and your hands, particularly finger nails, should be thoroughly washed to remove all traces of lubricant and the 6  $\mu\text{m}$  diamond. The specimen should be rinsed in alcohol and dried. The above technique is repeated using the 1  $\mu\text{m}$  diamond wheel. After polishing on this wheel, the surface should be optically flat, you should be able to use it as a mirror. With many

specimens the 1  $\mu\text{m}$  diamond finish will be adequate. On occasions where it is not possible, the specimen is further polished on an alumina wheel lubricated with water or diamond wheel with finer abrasives.

### **EXAMINATION.**



**Fig 3: Microstructure of part of a laser weld bead in brass (Original mag X100)**



**Fig 4 : Microstructure of a laser weld bead in mild steel sheet**

Specimens should always be examined in the “as polished” condition to assess the quality of polishing and to observe any features showing contrast. After examination and noting any features, the specimen should be etched to develop additional contrast to reveal the microstructure.

For mild steels, high Carbon steel in part (a) and b (3), the specimen must be etched for about 10-15 seconds in Nital. For high carbon steel in part b (1) and b (2), the specimen must be etched for about 5 seconds. Analyse the specimen under the microscope and etch for a few more seconds if required.

*(For other alloys, different etching reagents may be required)*

### **MOUNTING.**

With very small or irregularly shaped specimens it is generally more convenient to mount them in a resin. Bakelite is commonly used. Also, more than one specimen from a single component can be mounted and therefore polished at the same time. Never mount dissimilar metals in the same mount.

A further advantage of mounting is that the edges of the specimen will be reasonably well polished and not “bevelled” by the preparation process. In certain instances the very edge of the specimen is most important. In these circumstances the specimen would be electroplated with nickel before mounting. Any slight degradation will then take place on the nickel coating and not the specimen.



**Fig 5: A Hot Mounting Press**

### **ASSIGNMENT.**

***(If in doubt please contact the technician or your lecturer)***

***Please be careful while using the equipment in the lab. Switch off all equipment and tidy the room before you leave.***

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**Chief Tech**: Mr Choo Ping Kwan (*Metallurgy Lab.*)

**Technician** : Messrs. Banji Soni & Seetohul (*Metallurgy Lab.*), Mr Manoga (*Structural Mechanics Lab.*)

Make sure that you note all your observations and readings as you will be using the same specimens for all the tests. Each group will be provided with a mild steel (up to 0.3 %C) and a tool steel (approx. 0.87 % C) specimen.

(a) You will be required to prepare the specimens for macrostructural and microstructural analysis and sketch the grain structure. Perform (note the results) some hardness tests (Structural Mechanics Lab.) at various locations on the specimen.

(b) The properties of materials depend on the type, size, distribution and amount of the different phases present. By the proper heat treatment process, the properties of some materials can be changed. With reference to steel, briefly explain what you understand by (i) Hardening, (ii) Tempering (iii) Annealing and (iv) Normalising.

1. You will be required to harden the 2 specimens. Use the Iron-Iron Carbide Phase Equilibrium Diagram, determine the hardening temperature for each specimen. Contact the Technician and heat the specimen in the furnace. Allow the specimen/s to soak in the furnace for some time and then quickly remove the specimen/s and quench it/them in oil or water.

Repeat step (a) for the microstructural analysis and hardness tests.

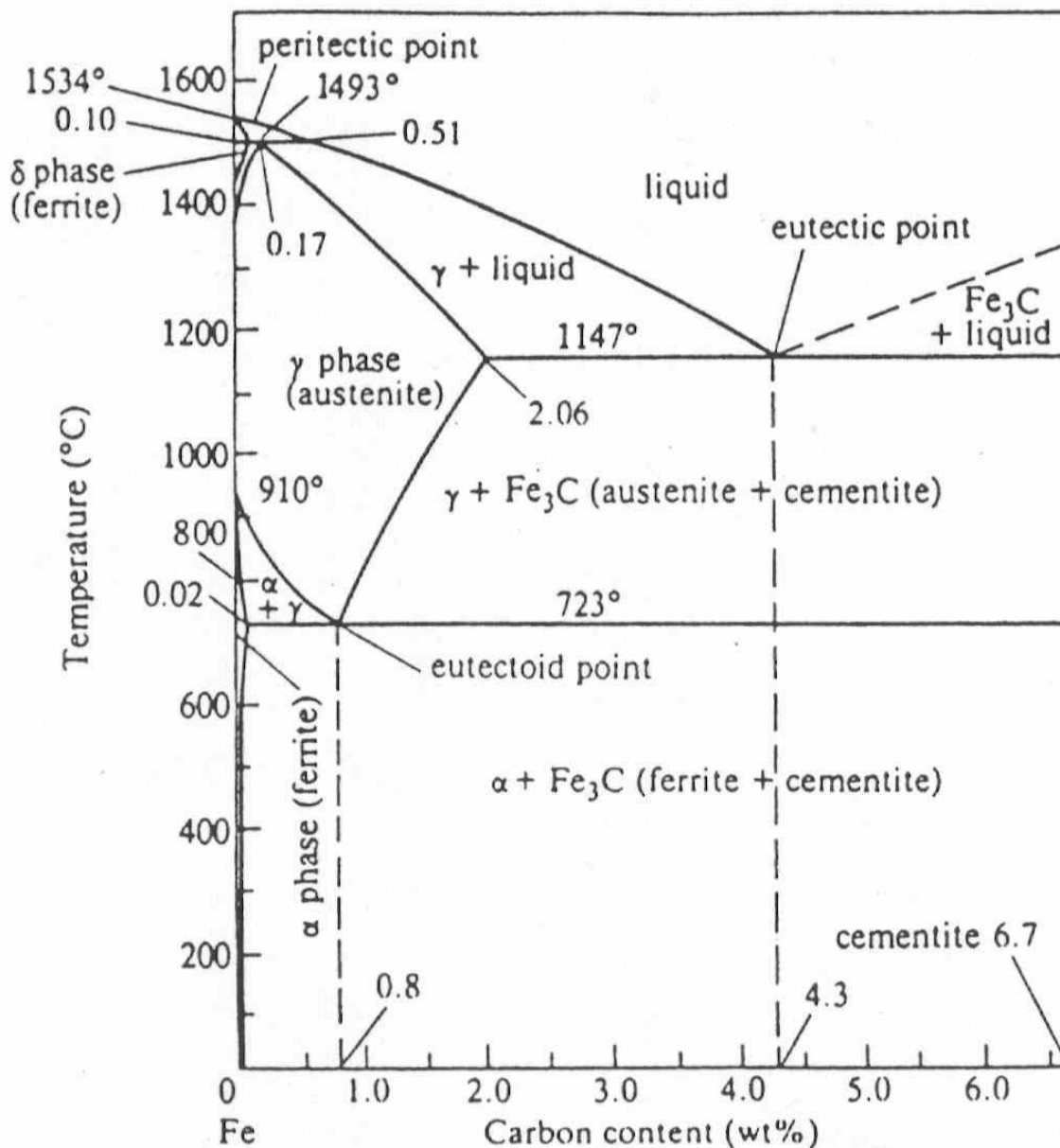
2. In many cases some materials are too hard for any practical applications. The specimens can be tempered to relieve some hardness at the expense of hardness and tensile strength. Using the Iron-Iron Carbide Phase Equilibrium Diagram, select a tempering temperature and perform a tempering.

Repeat step (a) for the microstructural analysis and hardness tests.

3. The original microstructure and properties of the specimens can be restored by annealing. With reference to the Iron-Iron Carbide Phase Equilibrium Diagram determine the annealing temperature for each specimen. Contact the Technician and heat the specimen in the furnace to the required temperature. Allow the specimen/s to soak at that temperature for some time. Then cool the specimen very slowly to room temperature.

Repeat step (a) for the microstructural analysis and hardness tests.

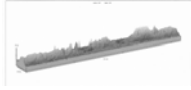
Comment on the different results obtained.



Iron-Iron Carbide Phase Equilibrium Diagram (Reproduced from Engineering Metallurgy Pt. I by Higgins).

**REFERENCE:**

- Material Science and Engineering- An introduction by W. D. Callister
- Engineering Metallurgy- Part I by R. A. Higgins
- Materials Science and Engg. By V. Raghavan
- Manufacturing Engineering and Technology by S. Kalpakjian



Mauritius

All pictures obtained from the Metallurgy Lab of the University of