ME 212 LABORATORY EXPERIMENT #1

MICROSCOPIC EXAMINATION OF METALS

1. OBJECTIVE

To study the microscopic strucures of metals.

2. INTRODUCTION & THEORY

The properties of metals highly depend on their structures. The internal structures determine how materials perform under a given application. The effects of most industrial processes applied to metals to control their properties can be explained by studying their rnicrostructures. The branch of materials science dealing with microscopic examination of metals is called Metallography.

The most common method used to examine the structures of materials is optical technique. A specimen about 20 mm on an edge is cut from the metal to be examined. In some cases where the subject is small or unhandy like razor blade, it is embedded in a plastic case which is called "mounting". A mirror polish is produced on one face of the specimen by grinding on successively fine emery (sand) papers and polishing on revolving cloth wheels with fine abrasives such as diamond or alumina powder. To reveal the structural details such as grain boundaries, phases and inclusions, this polished surface is etched with chemical solutions. The etchant attacks various parts of the specimen at different rates and reveals the structure. A metallographic microscope is used to examine the microstructure.

Additional Info about Optical Microscopy:

In order to be able to use metallographic methods effectively, a clear understanding of principles of construction of the optical microscope is necessary. First of all, light coming from the light source must be directed to the same side as that from which the specimen is viewed. In conventional optical microscopes, light is directed through proper prisms and passes through the objective. The objective itself is a one sided convex lens with its flat side directed to the specimen. The characteristic properties of an objective are its focal length and the numerical aperture marked on the mounting. The numerical aperture (N.A) can be calculated by taking the product of the index of refraction of glass and the sine of the half angle under which light enters the lens from the point in focus.

The oculars used in optical microscopes are basically double-sided convex lenses. The image coming from the focal point of the objective is directed through the ocular and is thus magnified. The magnification capability of the oculars is usually marked on the eyepieces. The three properties of an optical microscope which exhibit great importance are the following: Magnification, Resolution and Flatness of Field.

The resultant magnification of the optical microscope can be calculated by taking the product of the magnification powers coming from the objective and the ocular. The resolution "r" can be calculated as:

$$r = \frac{wavelength}{2 * N.A.}$$

The resolution value calculated in this way designates the minimum distance between two points which can just be distinguished as being seperate by looking at the image produced by the microscope.

3. PREPARATION OF SPECIMENS FOR MICROSCOPIC EXAMINATION

In order to examine a metal microscopically, a small specimen must be cut and then mounted first.

MOUNTING

Here are the steps for “hot mounting”:

1. Select a flat, smooth face for mounting (debur this face if necessary).
2. Select a matched die set for use(usually the one having 30 mm diameter is used in ME 212 Lab applications. If it is already placed, skip this part).
3. Switch on the compressor. When it stops operating, open the small black valve to provide compressed air to the mounting press. Adjust the pressure by turning the pressure button on press such that it does not exceed 300 bars.
4. Press the button: Rem upward, until the lid can be taken out.
5. If there are any remainings of Bakelite powder from previous applications, clean them. Then, spray “Mounting Release” inside the ram and under the lid to prevent tight adhesion of the two parts.
6. Place the sample in the die, desired face downward, resting on the polished face of the base. Center the sample as well as possible and press the button: Rem downward
7. Pour “phenolic resin” (Bakelite) into the die cavity until the latter is filled to within about 1-1/2 inches of the top of the die. Close the lid and tighten it slightly.

Figure 1. Bakelite Powder used for Hot Mounting (*Spi-Supplies – www.2spi.com*)

8. Press the “T_h” button. Set the heating temperature(usually 190 Celsius in ME 212 Lab applications)
7. Press the “T_c” button. Set the heating temperature(usually 40 Celsius in ME 212 Lab applications)
8. Press the “TIME” button. Set the heating time(usually 7 minutes in ME 212 Lab applications)
9. Press the “START” button. Heating will start and continue until the temperature becomes T_h.
10. When the total process ends, the device gives a warning sound signal. Press the “STOP” button to cut off the signal.

Figure 2. Conventional Hot Mounting Press in ME 212 Course Laboratory

11. Loose the lid. Press the button: Rem upward, until the lid can be taken out. The mounted specimen moves upward, which is ready to be used for further applications.
12. Press the button: Rem downward. Close the lid. Bring the pressure back to 0 using the pressure button. Evacuate the air inside the compressor.

When samples are considerably sensitive to heat and/or pressure, the method of "cold mounting" is used to mount them. Additionally, cold mounting does not require an investment in a mounting press and is therefore good for infrequent mounting tasks. The resin, a mixture of two or three components, is poured over the sample after it has been placed in a mounting cup. After curing (solidification of the resin), the sample can be taken out of the cup and processed. Three different types of cold mounting resins are available: Epoxy resins, Acrylic resins and Polyester resins. Acrylic Resins have very rapid cooling times and are thus preferred when the number of samples to be mounted is relatively high. Epoxy resins have perfect penetration and flow characteristics and are used to achieve the optimum mounting quality. Polyester resins are very economic and have relatively short curing times.

Figure 3. Various Resins Used for Cold Mounting (*www.struers.com*)

After mounting, a plane surface free from any irregularities must be prepared by grinding, which is performed by starting with emery paper no.1 and going on successively finer grades such as No. l/0, 2/0, 3/0 and 4/0 (or 240, 320, 400 and 600). It is important that the specimen must be cleaned during transfer to successively finer abrasive papers to prevent carry-over of coarse abrasive particles. The removal of striations from coarser abrasive is achieved more readily if the specimen is rotated 90° during transfer. Washing the specimen prior to polishing also contributes to successful preparation. Final removal of irregularities is accomplished by polishing the specimen on a rotating cloth which is covered with extremely fine abrasive usually applied as a liquid suspension (Al_2O_3-water) until mirrorlike surface is obtained. Polishing is performed by starting with powder having largest size and progressing with powders having smaller size. At this stage microscopic examination will reveal the presence of any cracks, seams, nonmetallic inclusions and similar scale inhomogenities.

ETCHING

In order to reveal the crystalline structure(grains and grain boundaries) of the specimen, the polished surface is etched by a proper etchant. For steels Nitol (3% HNO_3-ethyl alcohol) is used. This step is represented by differential chemical attack depending upon chemical composition, energy content, and grain orientation. Thus the grain boundaries are attacked at a greater rate than the proper grain due to higher energy content of the grain boundaries. The result is a depression of the grain boundaries. In addition, the presence of chemically different phases results in variations in the rate of chemical attack. These changes in the rate of chemical attack produce deviations both in angle and depth of certain portions of the surface. Thus the light is reflected in varying amounts depending on the angle and depressions of the portion of the surfaces resulting in light and dark regions. In this manner the crystalline microstructure of the specimens are revealed.

Etching the Specimen

Etching may be performed by immersion or swabbing the etchant on the specimen. The mount is removed from the solution when a bloom appears. A bloom is a slight haze that appears and is evidence of the first appearance of the microstructure. If necessary, further etching may be performed after examination under a microscope to strengthen any details. After etching, the mount is throughly rinsed in running water. Then alcohol is sprayed over the surface and the mount is finally dried in a stream of hot air.

Etchants and Etching Procedure in Our Examples

- Plain Carbon Steel: 1% Nital, immersion for 25 sec.
- Tool Steel: 1% Nital, immersion for 25 sec.
- Stainless Steel: 10% aqueous oxalic acid solution, electrolytic etching for 90 sec. at 6V dc.
- Aluminum: Keller’s reagent, immersion for 10-20 sec.

A more detailed list of etchants used for various metals/alloys can be found below:

List of Etchants Used for Various Metals/Alloys :

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 micro-constituents.
General Purpose In,Sn,Pb,Cu,Ni,Al,Mg,W,Mo and their Alloys	Solution A: $K_2Cr_2O_7$ - 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.

4. TASKS :

Please perform the following tasks in the "Results&Discussion" part of your laboratory reports (Refer to your textbook as neccessary):

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- Draw the microstructure you see under microscope by hand and identify the different phases and features.

REFERENCES

- Van Vlack, L. H., *Elements of Materials Science and Engineering*, Addison-Wesley Pub. Co., (Mass:1994)
- Guy, A. G., *Elements of Physical Metallurgy*, Addison-Wesley Pub. Co., (Mass:1959)
- Kehl, G. L., *Principles of Metallographic Laboratory Practice*, McGraw Hill Book Co. , (New York:1949).