Laboratory 8

Metallographic Analysis

1. Objectives:

Students are required to understand the principles of microscopy analysis, to learn and to gain experience in the preparation of metallographic specimens. Adding to this, examine and analyze the microstructures of metallic alloys.

2. Introduction:

Microscopy analysis is an essential tool in understanding the microstructure or nanostructure of materials, chemicals or products. Data from microscopy analysis is important to progress in any product development program, or conduct a failure analysis where your product or material has failed.

In order to conduct an effective digital microscopy analysis, a rigid step-by-step process is followed. The results and images captured must be assessed to gain the valuable insight that you will need to understand your materials.

In sequence, the steps include sectioning, mounting, grinding and polishing, etching and microscopic examination. Specimens must be kept clean and preparation procedure carefully followed in order to reveal accurate microstructures.

This practical work will take you through the metallographic sample preparation process step-by-step with demonstrations and explanations of sectioning, mounting, grinding and polishing, etching and microscopic examination.

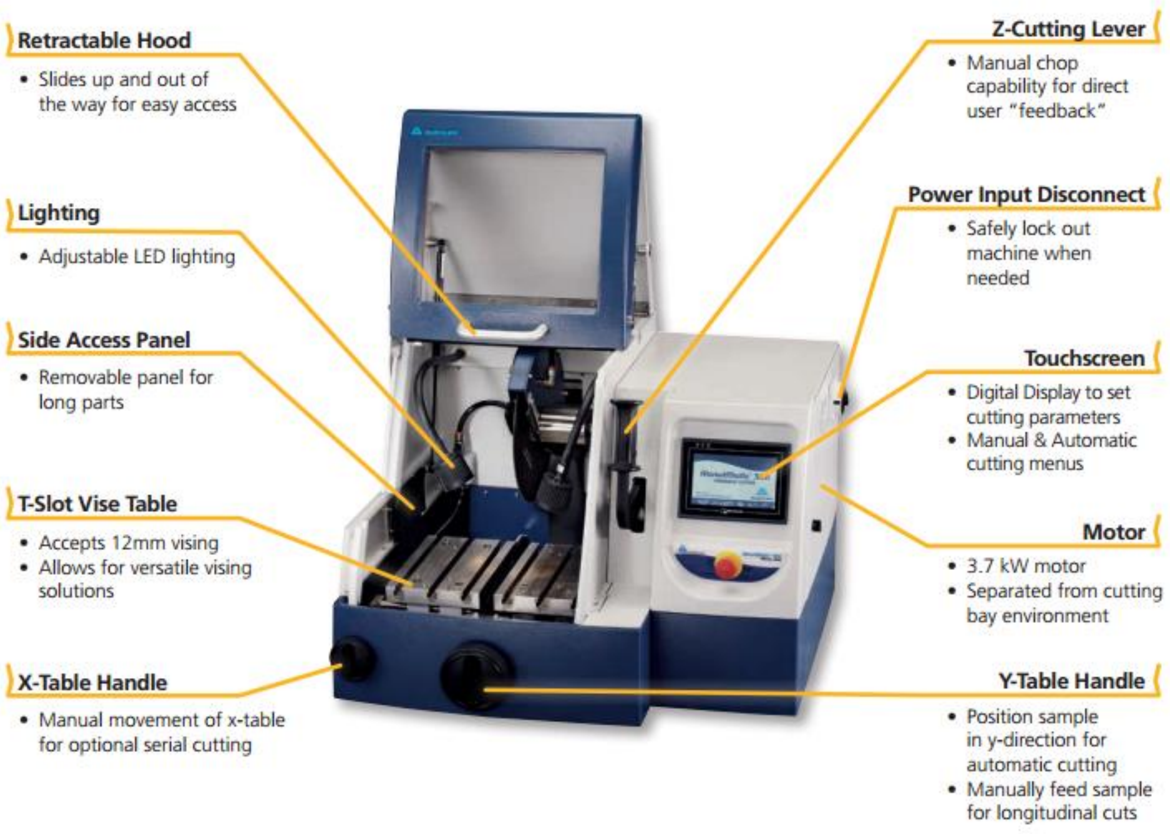
3. Equipment description:

In order to view a metal specimen under an optical microscope, the sample must be prepared following these steps:

- Sectioning
- Mounting
- Grinding and polishing
- Etching

The used equipments for each step are described below:

- ⇨ **Step 1:** The AbrasiMatic 300 Abrasive Cutter is a bench-top cutter featuring manual cutting action in 3 directions or automated cutting in 1 direction. This gives the user the maximum versatility to section a wide variety of sample materials, sizes and geometries.



⇨ **Step 2:** Mounting machine

Buehler metallurgical mounting press for the production of thermosetting and thermo plastic moulds using an automatic cycle. The system is powered using single phase input and using electro hydraulic pump for pressure on the sample.

Technical details of the machine:

-Automatic Metallurgical mounting press

- 30 mm mould assembly

- Easy to use control panel.

- Simple bayonet closure

-Cycle (preheating cycle + cooling) completion: alarm notification

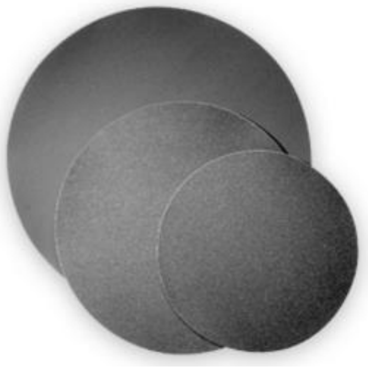
The most important features include its maximum heating temperature and how intimately the heater and water cooler are connected to the mold assembly.

➔ **Step 3:** Grinding and polishing

Metallographic Grinding and Polishing Machine is a double disc table type machine, suitable to grind and Polish the metallographic sample, with a rotate speed range 50-1000rpm, it’s a necessary equipment for sample preparation. It's equipped with cooling device, used to cooling the sample when grinding, to prevent the sample overheating and destroy metallographic organization.

- It's easy to operate, safe and reliable
- The working disk can be changed and replaced swiftly.

<u>Supplies for grinding</u>:



For metallographic preparation, silicon carbide (SiC) abrasives are used in coated abrasive grinding papers ranging from very coarse grit size to very fine grit sizes. Initial grinding is recommended with 80 grit (P80) paper, followed by P180, P240, P320, P600, P1000, and P1200.

<u>Supplies for polishing:</u>

Final polishing abrasives are selected based upon specimen hardness and chemical reactivity. The most common polishing abrasive is alumina. Alumina abrasives are primarily used as mechanical abrasives because of their high hardness and durability.

Etching:

The metallographic etchants used for pure Aluminum and Aluminum alloys is Sodium hydroxide (NaOH), also known as lye and caustic soda. It is an inorganic compound which is composed of: 1-2g NaOH and 100 ml of water.

For steels, the most common etchant is Nital, it is composed of 1-10mL HNO_3 and 90-99 mL methanol or ethanol. This compound reveals grain boundaries and constituents. The 2% Solution is most common. The samples are immersed for up to about 10s.

For copper, the used etchant is HNO_3. The sample is immersed for a few minutes (Between 3 and 5 minutes). ***see Metallography, Principles and practice "George F.Vander Voort"**

4. Safety Instructions

ATTENTION	ATTENTION Proper cutting procedures must be followed; abrasive cut-off wheels will crack and become flying projectiles when used improperly.
ATTENTION	The sample MUST be washed thoroughly before proceeding from one grinding stage to the next!!

ATTENTION	After etching: do not touch, wipe or swab the specimen following etching; dry off the rinsing alcohol on the specimen with the air blast
ATTENTION	During mounting, you must pay attention to the hot water release at the end pre-heating step
ATTENTION	During grinding, use both hands to hold the specimen; unsecured specimens can "Catch an Edge and FLY"!
ATTENTION	AT the end of polishing, DO NOT TOUCH THE SPECIMEN SURFACE!!!

5. Basic principles

Metallography is the study of the structure of metals and alloys. Metallographic analysis can be used as a tool to help identify a metal or alloy, to determine whether an alloy was processed correctly, to examine multiple phases within a material, to locate and characterize imperfections such as voids or impurities, or to observe damaged or degraded areas in failure analysis investigations.

Optical microscopy is sufficient for general purpose examination; advanced examination and research laboratories often contain electron microscopes (SEM and TEM), X-ray and electron diffractometers and possibly other scanning devices.

Incorrect preparation of samples may result in altering the true microstructure and will lead to erroneous conclusions. Adding to this, if the metallurgist is not careful hot or cold working can take place during the preparation of the samples.

a. Sectioning and cutting:

Most metallographic samples need to be sectioned to the area of interest. Depending upon the material, the sectioning operation can be obtained by abrasive cutting (metals and metal matrix composites), diamond wafer cutting (ceramics, electronics, biomaterials, minerals), or thin sectioning with a microtome (plastics).

In this case, abrasive cutting is the adopted method. Sometimes, operations such us shearing produce severe cold work.

Abrasive cutting is a way that offers the best conditions to eliminate these undesirable features.

Since heat created by abrasive cutters must be avoided during this operation, low speed cut off wheels are used. Adding to this, effective sample cooling is a key factor in producing high quality burn-free cuts.

Abrasive cut-off wheels are designed to give the user a fast, comfortable cut while providing long life and performance advantages over conventional products. They consist of abrasive grains bonded together with rubber or other materials in the form of a thin wheel. These latest are indexed from hard to soft and are referred by the grade. As a rule; a hard bonded wheel should be selected when sectioning soft stock, and a soft wheel for sectioning a harder material.

b. Mounting:

After cutting the specimen, the next step is mounting; this latest refers to the process of compressing and heating Bakelite powder (Bakelite is a low cost, relatively hard thermosetting polymer that is commonly utilized) around a piece of metal in order to form a solid disk or puck that can be used to handle the sample easier. When transparency is required in this process, expensive transoptic thermoplastics are utilized.

Mounting produces specimens with uniform size so that it is easier to handle in automatic holders for further preparation steps.

Basically, there are two methods: Hot Mounting, Cold Mounting

In hot mounting process, the specimen is mounted under heat and pressure with a hot mounting press.

On the other side, cold mounting is preferred for samples which are sensitive to damage from heat and pressure (like coatings…). Cold mounting resins are easy to use and require mixing which is then poured into a mould and allowed to set.

Within this practical work, hot mounting is used to prepare the specimen.

During mounting, the pressure and molding temperature remain constants, thus time is the only variable. The metallic sample is placed in the mounting cylinder which is in turn encased in a pre-measured amount of polymer. The mounting cylinder is then sealed, pressurized and heated to complete the polymerization process necessary to solidly encase the metal sample.

c. Grinding and polishing:

In order to obtain a highly reflective surface that is free from marks and cold working due to specimen cutting, the specimens must be carefully grinded and polished before they can be examined under the microscope.

Grinding process can be accomplished either wet or dry using 80 to 1200 abrasive papers. The main idea of this step is to carefully move from one stage to the next where the abrasives become finer at each successive stage. Movement from one stage to the next should only proceed when all of the scratches from the preceding stage are completely removed. In general, successive steps are 240, 320, 400 and 600 grit SiC and the grinding rate should steadily decrease from one stage to the next.

Adding to this, this process involves rotation of the sample by 90° between stages while the grinding angle must be held constant during the grinding at any one stage.

Polishing involves the use of diamond abrasives. These latest provide the best, and most expensive, compounds utilized in polishing. The specimen is held at one position on the wheel, without rotation, until most of the previous grinding marks are removed. It can be rotated slowly, until only scratches from the 25-micron aluminum oxide are visible. During this polishing stage, moderate pressure can be applied to the specimen and the entire stage should generally take 1 or 2 minutes.

If you fail in following these rules, you will have abrasive particles between the different stages of grinding and this is time consuming … you will have problems in removing the resulted scratches …!

Standard grit	120	180	240	320	360	400	600	800	1200
European (P-grade)	P120	P180	P220	P320	P500	P800	P1200	P2400	P4000
Nomial micron size	106	75	58.5	40.5	30.2	21.8	15.3	6.5	2.5

Table: Comparison of standard US grit size to European grit and normal micron size

d. Etching:

While examining a none properly etched specimen, the microscopic observation will not reveal much information; it will give a few structural features such as inclusions, cracks or other imperfections. Etching process is used to reveal microstrutural features or present phases.

Etchants are usually dilute acid or dilute alkalis in water, alcohol or some other solvent. The etching process is usually accomplished by merely applying the appropriate solution to the specimen surface for several seconds.

Do not touch, wipe or swab the specimen following etching; dry off the rinsing alcohol on the specimen with the air blast and then move on to the microscopic examination stage!

6. Experimental Procedure

In this practical work, use the samples studied in the tensile testing part, especially the part near the rupture region.

a. Sectioning:

1. Place the sample on the slotted table so that it is at the center of the swing of the head.
2. Place the Locking Lever in the forward position to allow the Control Rod to be moved to the furthest position.
3. Move the Rear Vise section to contact the rear face of the sample. Move the Front Section to a position near the front face of the sample.
4. Tighten the Nuts that secure the Vise sections to the Table.
5. Push the Control Rod in to make firm contact with the sample and pull back the Locking Level to secure the sample.
6. Turn on the coolant; it should flow readily to both cool and clean the cutting area.
7. Apply steady moderate pressure until the specimen is sectioned; avoid jerking motions that may cause the blade to splinter.

b. Mounting:

Supplies for mounting:

Bakelite: thermosetting phenol formaldehyde (formed from a condensation reaction of phenol with formaldehyde)

1. Clean the specimen to remove cutting and handling residues
2. Remove debris from mold assembly
3. Lower ram assembly by clicking on: 
4. Place the specimen in the center/bottom of the plastic mounting ring base with the examination surface facedown.
5. Mix the appropriate quantity of polymer (2 spoons of Bakelite) and add it to the sample
6. Attach the mounting ring cylinder to the base by simple pressing firmly in place.
7. Start the mounting process
8. The pre-heating step begins; it refers to the polymerization of the Bakelite powder and takes 3 min. At the end of this step, you must pay attention to the hot water release as will be mentioned by your teaching assistant
9. Then the machine goes through the cooling of the specimen for 10 min.
10. After 10 min, an alarm notification is triggered.
11. Press the bottom to open the mounting ring, then remove the mounted specimen
12. Clean mold and ram assembly
13. Now, you can proceed with grinding and polishing

c. Grinding and polishing:

⇨ **Grinding:**

1. Label the specimen with your name so that it can be easily identified.
2. Turn the Motor On, then the water, adjust the flow to obtain a good film of water. Too much water will cause a spray when it contacts the sample.
3. Begin with the P80 grade and use both hands to hold the specimen, carefully place the sample face onto the exposed area of the belt, be careful not to contact the rotating surface with a sharp edge of the specimen or your hand.

4. During grinding, apply moderate pressure evenly, move the sample left-and-right across the belt surface to obtain uniform grinding. Use both hands to hold the specimen; unsecured specimens can "Catch an Edge and FLY"!
5. Lift the sample from the wheel periodically to determine the progress of grinding but do not rotate the sample. The P80 grade stage is complete when all the lines scratched in by the grinder are parallel on the specimen surface. If any line or scratch is not in the same direction as the other lines, continue grinding until all of the lines are parallel.
6. When all of the sample's scratches are parallel, proceed to the P180 grade stage with the scratches oriented approximately perpendicular to the intended grinding direction and repeat steps 3 through 6.
7. When the P180 grade stage is complete, you're ready to move on to the following abrasive paper (P240 grade) and proceed the same steps till you arrive to the highest grade (P2000).
8. You should continue grinding until the previous stage's scratches are gone
9. Sufficient water must be applied to provide lubrication and flush away the removal products.

⇨ **Polishing:**

1. Put the polishing cloth on the rotating table and turn the water on
2. Apply a small amount of the aluminum oxide abrasive solution (9µm) to the polishing cloth
3. When polishing the specimen, hold it with both hands, apply a moderate amount of pressure, and don't let it go
4. At the final stage, you must have a mirror surface on your specimen. Before proceeding to Etching, wash and dry both the specimen and your hands thoroughly. DO NOT TOUCH THE SPECIMEN SURFACE!!!

d. Etching:

1. Place the specimen on the table
2. Without touching the specimen surface, apply a few drops of Etchant to the specimen surface covering the entire metallic surface.
   For Steel, use the Nital etchant, for aluminum use NaOH etchant and for Copper use HNO_3
3. After about 5 to 10 seconds, rinse the Etchant into the sink with water and quickly rinse the specimen with alcohol, but do not touch the surface!

4. Use the Hot Air Gun to dry the sample.
5. Proceed to Microscopic Examination; if further etching is required you may return and proceed through steps 1 to 4 varying the time depending on the results.
6. If the specimen has many scratches and marks or the microstructure cannot be seen after several etches, return to grinding and go back through the necessary steps.

e. Microscopic analysis:

1. Use the metallurgical microscope to view the specimen at various magnifications and note the microstructural aspects of the material.
2. Take a photograph of each specimen at the optimal magnification.
3. Make sure that the specimen that you prepared is clearly labeled and the specimen surface is in its final condition; it must be submitted with your lab write-up to the laboratory instructor.

7. Questions

1- Do the experiment as described in paragraph 6 for the samples used in the tensile testing.

2- Discuss the obtained experimental results and compare the microstructure of the samples: steel and aluminum.

8. Results

Take a snapshot of the Micrographic analysis of the studied specimens: Steel Aluminum and Copper, and give an analysis regarding their microstructures, (type of grains, problems in etching or polishing …).

Make a comparison between the microstructures that you find and examples of microstructures found in literature.