



**U**niversity of  
**Jordan**

# Microscopic Examination



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Note: All the drawings in this report are drawn using AutoCAD

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# Safety

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**S**afety is a state in which hazards and conditions leading to physical, psychological, or material harm are controlled to preserve the health and well-being of individuals and the community.

**Before starting the experiment in laboratory, we must follow safety rules:**

- 1.** Put on protective clothing like lab coat, a pair of safety glasses and a pair of disposable gloves.
- 2.** Do not hold a workpiece by hand. Workpiece will become very hot while being cut.
- 3.** To prevent damage to the microscope and to protect yourself from injury. always carry the microscope with two hands. Place one hand on the arm of the microscope and place the other hand underneath the base of the microscope.
- 4.** Do not taste or sniff chemicals.
- 5.** Rings and jewellery must not be worn.
- 6.** Long and loose hair must be contained.
- 7.** Close fitting / protective clothing must be worn.

# Objectives

One of several methods that may be used to evaluate the grain size of a specific metal, the location of flaws, and the placement of non-metallic inclusions inside the metal, among other uses, is microscopic examination. In our work, we estimated the grain size and used a lab microscope to study the metal's structure on steel.

## MATERIALS :

- Rod of iron
- Rod of aluminium
- $H_2O$
- saws
- Rubber Gloves
- Mask
- Flask
- Sandpaper (silicon carbide paper 120, 180, 240, 400, 600, 800, 1200)
- Safety glasses
- Lab coat
- Ethanol
- Alcohol
- Resin
- Aluminium oxide

## Materials and Apparatus

## APPARATUS :

- Cutting machine
- Grinding machine
- Fume hood
- Mounting machine
- (computer) microscope





# Experimental procedure

**In metallography there are two types of examination: microscopic & macroscopic.**

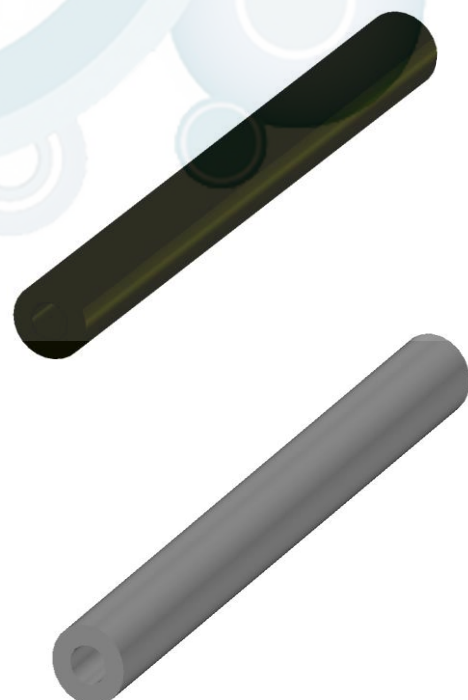
- ✓ **Macroscopic examination:** in this process the internal structure reveals by naked eye or low magnifying Lens.
- ✓ **Microscopic examination:** in this process the internal structure reveals by high magnifying Lens.

Anyway, in this experiment we will examine and analyze the specimen microscopically.

**But before we start examining the specimen, we must prepare the specimen for examination through the following steps:**

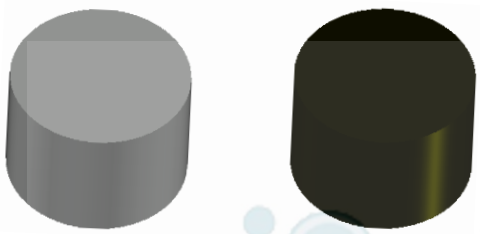
## 1) Specimen selection:

- We need to choose the material that the sample consists of, which in this experiment is **steel** and **aluminium**.
- The number, location, orientation of the sample examined are an important parameter.



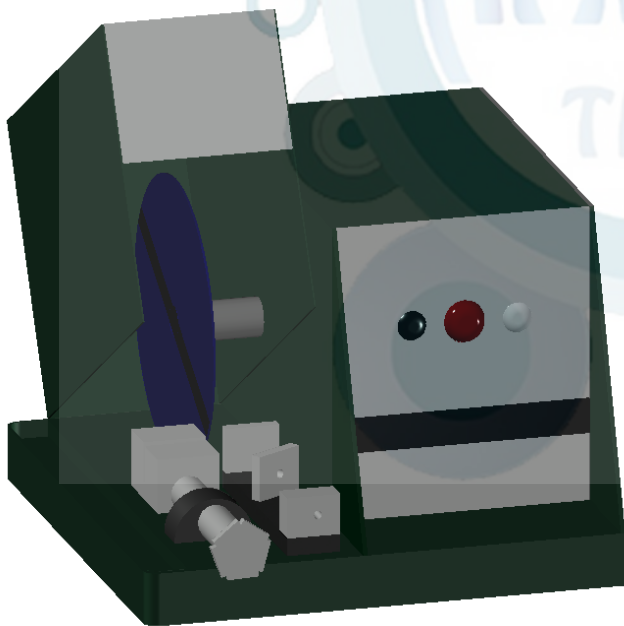
## 2) Sizing:

- **Cube:** each side (12-25 mm)
- **cylinder:** the height and the diameter (12-25 mm).



## 3) Sectioning or Cutting:

- It is performed carefully to avoid altering the structure of the material.
- Abrasive cutting is the most common cutting method and that what we were used in our experiment.

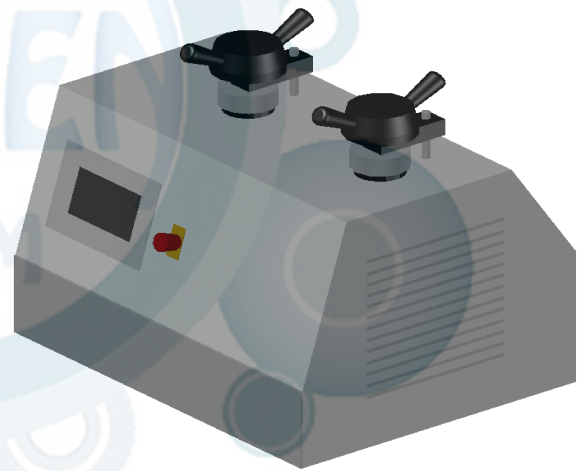


- the cutting tool is made of silicon carbide (Sic) or diamond particles.

- we used a coolant fluid to avoid overheating of specimen & possible change in material structure.

## 4) Mounting:

Small samples can be difficult to hold safely during grinding & polishing operations, and their shape may not be suitable for observation on a flat surface. They are therefore mounted in **powdered or granular of thermoset resins** such as [ **diallyl phthalates, phenolics, melamine, epoxies**] or some **thermoplastic resins** such as [ **acrylics, Bakelite**]



There are three types of mounting:

- **Thermal** mounting
- **Cold** mounting
- **Mechanical** mounting

However, in our experiment we use the **thermal mounting**.

Then *mechanical preparation* starts, which is divided into two operations: grinding & polishing.

## 5.) Grinding:

- Grinding is done **using rotating discs covered with silicon carbide** paper and water.
- There are several grades of paper, with [120,180, 240, 400, 600, 800, 1200] Grains of silicon carbide per square inch or cm.
- 180 grade therefore represents the coarsest particles & this is the best grade to begin the grinding operation with.
- Light pressure applied at the center of the sample.**

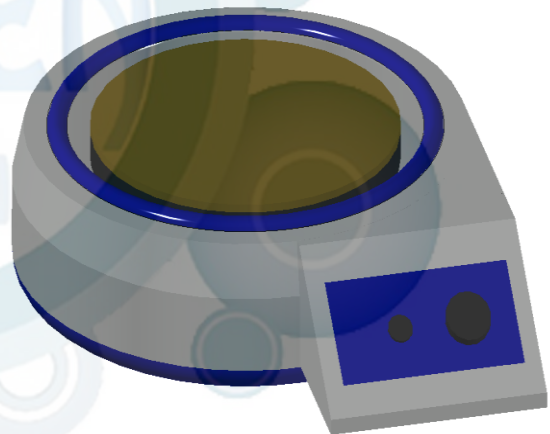


**the rotation direction.** This makes it easy to see when the coarser scratches have all been removed.

- After the final grinding operation done, we wash the sample in water followed by alcohol & dry it before moving to the polishing.

## 6) Polishing:

- The polishers consist of rotating discs covered with soft cloth impregnated with a pre-prepared slurry of hard powdery *alumina* “Aluminium *Oxide*” particles ( $\text{Al}_2\text{O}_3$ , the size ranges from (0.5-0.03  $\mu\text{m}$ ).



- We wash the sample in water & move to the next grade, **orienting the scratches from the previous grade normal (perpendicular) to**

- Begin with the coarse slurry and continue polishing until the grinding scratches have been removed. It is of vital importance that the sample is thoroughly cleaned using **soapy water**, followed by **alcohol**, and dried

before moving onto the final stage. Any contamination of the final polishing disc will make it impossible to achieve a satisfactory polish

- examining the specimen in the microscope after polishing should reveal mirror like surface.

{Note: from now on the sample is called *metallographic specimen* or *microsection*.}

## 7) Etching:

Is the process of clarifying the crystalline boundaries (in technical terms grains boundaries) by using a chemical solution, depending on the type of metal (we can know which solution “etchant” to use from particular tables) where:

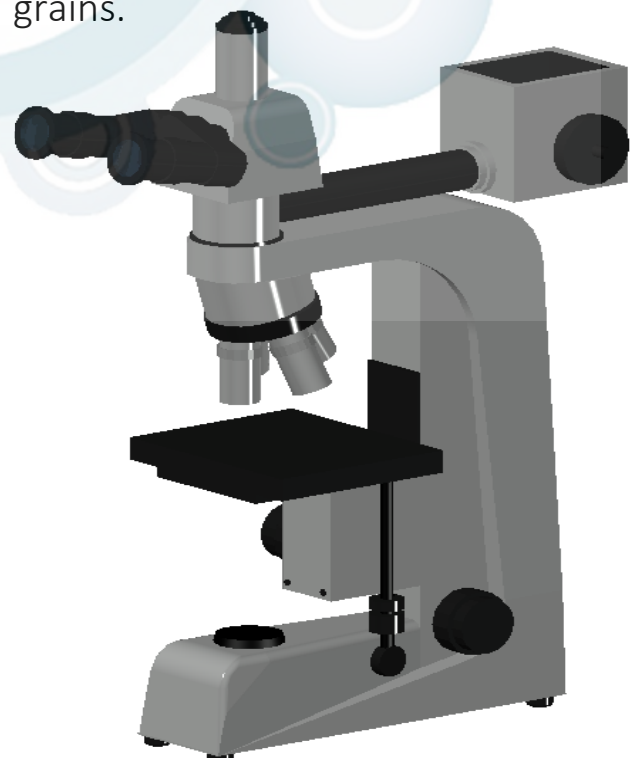
- The sample is immersed in this solution using tongs.
- the solution that steels immersed in consists of: [25%  $\text{HNO}_3$ , 75%  $\text{H}_2\text{O}$ ]
- the solution that aluminium immersed in consists of: [15%  $\text{HF}$ , 35%  $\text{HNO}_3$ , 25%  $\text{HCl}$ , 25%  $\text{H}_2\text{O}$ ]
- Then the sample washed with water & methyl alcohol.

- Finally, sample is dried under a hairdryer.



## 8) Using Metallurgical Microscope

Finally, we put the specimen on microscope {as shown below} to clearly see the shape and size of the grains.





- Turn on microscope lamp power.
- Carefully mount the metallographic specimen on top of the stage, with the polished surface facing up.
- Adjust the distance between the stage & the objective by turning the stage height adjustment knob.
- While looking through the eyepieces focus on sample by turning the coarse & fine focus adjustment knobs accordingly.

The sample shouldn't hit the objective lens.

## Results and discussion

- The first step done in our experiment, was to select a specimen either from aluminum (al) or iron (Fe), but before that, we had to cut the rods by using the cutting machine in the lab, because it didn't have the desired size we wanted.
- The next step was grinding, where took turns to do it, the process of grinding required us to place the specimen on the grinding machine with papers of sizes (120,180,240,400,600,800,1200) & while the machine was on , the instructor told us to open the machine's water tap in order to prevent the specimen from having fractions that cause deformation due to high temperature, also we had to rotate the specimen 90 degrees when changing the griding paper then we make polishing after polishing we saw the specimen by microscope, we were able to see the structure of the specimen clearly , and based on that we were able to calculate the average grain size by applying these steps:

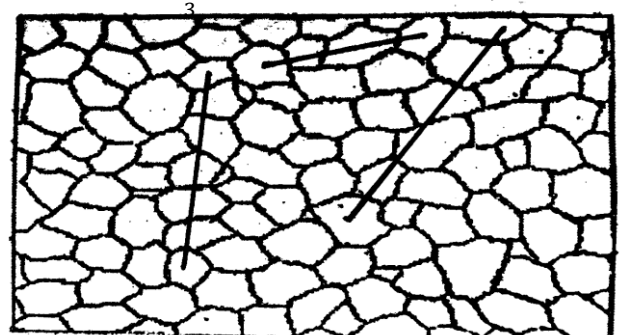
$$1) \text{ Grain size} = \frac{\left( \frac{L(\text{cm})}{\text{number of grains (cross)}} \cdot 10^4 \right)}{\text{magnification}}$$

$$2) \text{ Average Grain size} = \frac{(\text{Grain size 1} + \text{Grain Size 2} + \text{Grain Size 3})}{3}$$

And that's result we get in our paper:

$$\text{Grain size 1} = \frac{\left( \frac{2.8}{6} \cdot 10^4 \right)}{100} = 46.6 \frac{\text{Mm}}{\text{grains}}$$

$$\text{Grain size 2} = \frac{\left( \frac{1.6}{4} \cdot 10^4 \right)}{100} = 40 \frac{\text{Mm}}{\text{grains}}$$



❖ The figure above is our sample

$$\text{Grain size 3} = \frac{\left(\frac{2.4}{7} \cdot 10^4\right)}{100} = 34.28 \frac{\text{Mm}}{\text{grains}}$$

$$\text{Average grain size} = \frac{(46.6 + 40 + 34.28)}{3} = 40.3 \frac{\text{Mm}}{\text{grains}}$$

So how we do that firstly we draw 3 lines without intersection each another and the lines do not pass through the same grain

Convert cm to micrometer = 1 Cm =  $10^4$  Micrometer.

# Conclusion

This experiment's goal is to determine the average grain size and examine specimens under a microscope in order to identify which material is best for material engineers to use and to identify the characteristics of a polycrystalline material in to improve a material's tensile strength, thermal stability, and corrosion resistance. We scanned the specimen's structure under the microscope, then used that image to determine the grain size a specific formula.

# References

## ❖ References

- YouTube videos
- The manual
- Notes during the lab
- Wikipedia

## ❖ Technology

- AutoCAD
- Word