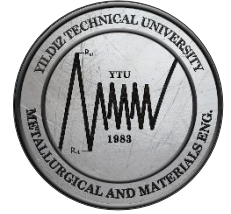


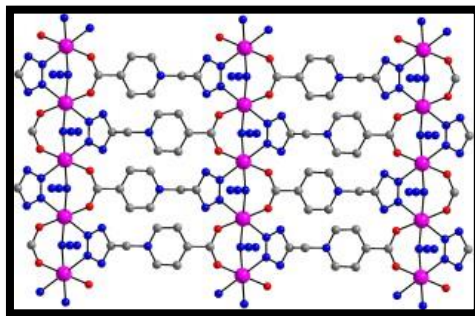
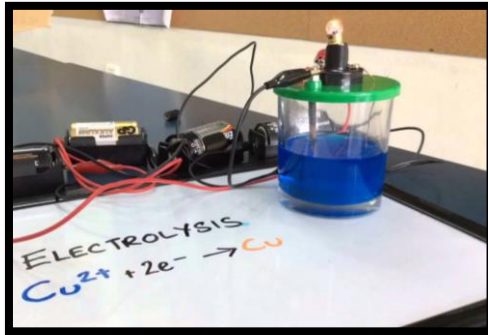


T.C.
YILDIZ TECHNICAL UNIVERSITY



FACULTY OF CHEMICAL AND METALLURGICAL
ENGINEERING

METALLURGICAL AND MATERIALS ENGINEERING



**MSE3961 LABORATORY-1
EXPERIMENT BOOKLET
2020-2021 FALL**

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LABORATORY SAFETY INSTRUCTIONS



LABORATORY SAFETY INSTRUCTIONS

- 1.** Handbags, coats, etc. should not be brought to the laboratory if possible. If these materials are brought to the laboratory, the materials should be kept at locations determined by the laboratory administrators.
- 2.** During the application of laboratories, long hair should be tied up and pendant earrings and bracelets should be removed. If there are cuts, wounds, etc. on the hands, working in the laboratory is possible only after they have been covered with waterproof tape, peep toe shoes should not be worn in the laboratory.
- 4.** Personal protective equipment (gloves, masks, eye / face protectors, hearing protection, work clothing) should be used to protect the eyes and the skin as long as worked in the laboratory.
- 5.** No food and beverage should not be consumed in the laboratory, and the face should not be touched while working
- 6.** It is necessary to know what is available in the first-aid chest and how the fire extinguisher works. Information about this must be obtained from the staff.
- 7.** In the laboratory, movements that distract others should be avoided. Gaming or joking is strictly dangerous and forbidden in the laboratory.
- 8.** Laboratory notebook should be kept for the studies conducted in the laboratory. Studies and observations must be recorded in this notebook.
- 9.** Taps, gas valves and switches, should be kept closed when not in use.
- 10.** Metallic laboratory tools should not be kept wet to avoid rusting.
- 11.** The solid materials to be dispensed should be dropped into the designated waste bin in the laboratory.
- 12.** Any incident occurred in the laboratory should be immediately notified to the administrators of the laboratory.
- 13.** No substance or material should be taken out of the laboratory without the permission of the laboratory's administrators.
- 14.** After use, each item should be cleaned according to the method of the appliance. Hands should be washed after leaving the laboratory.
- 15.** Before using the microscope, the objective and ocular should be carefully cleaned with a cloth before and after each use without damaging the lens.
- 16.** While diluting the acids and alkalis, they should always be poured slowly over the water, never the opposite.

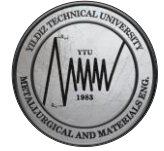


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17. If corrosive caustic substances such as acids or bases drip or splash to the naked skin, it should immediately be washed with plenty of water. The laboratory administrator must be informed about the incident.

18. When dealing with electricity, hands and electric switches and sockets must be dry. Electrical plugs should not be pulled out from the cord.



EXPERIMENT 1: WEAR TEST

1. WEAR TEST

1.1 AIM

Purpose of the experiment is determination of the wear behavior and wear resistance of metallic materials.

1.2 THEORETICAL INFORMATION

Wear is the material loss which occurs by the result of material transfer from one surface to another or removing of material from surface. At the DIN 50320, wear is described as the changes, which take place result of the removing particles by mechanical reasons, in the surface of used materials.

Material loss can occur in three ways in contact with the solid surfaces. These are local melting, chemical dissolution and breaking of particles from surface.

Main material (wearing material), counter material (abrasive), spacing material, load and motion are the main elements of wear system. This system is called a “Tribological System” in technique.

Environmental condition is another important parameter in tribological system. Wear accelerates with the effect of humidity and corrosive environment.

1. Factors that affects wear can be categorized in four main groups: I- Factors which are related to main material,
 - Crystal structure of material,
 - Hardness of material,
 - Elastic modulus of material,
 - Deformation behavior of material,
 - Surface roughness,
 - Size of the material.
2. Factors which related to the counter material and the effect of abrasive.
3. Environmental conditions,
 - Temperature,
 - Humidity,
4. Service conditions,
 - Pressure,
 - Velocity,
 - Sliding length.



EXPERIMENT 1: WEAR TEST

A. Wear Mechanisms:

There are different types of wear mechanism. These can be explained by the headlines are given below.

a. Classification in Terms of Mechanisms

Adhesive Wear:

This mechanism is the result of adhesion between couple of metal during sliding on each other. Stresses on the surfaces sliding on each other reach to or exceed the yield stress with small installations. As a result of this, adhesion forces occur between contact metal parts. Because of the adhesion forces, material transfer, cold welding and breaking of small parts take place between metal parts.

However adhesive wear is the most common wear mechanism, it generally does not accelerate the failure. Adhesive wear occurs by the breaking of adhered or welded particles during the relative motion of metal parts in contact.

Adhesive wear can be reduced by reduction of force which is applied on the surface, increasing of hardness of material and very well lubrication between metal parts. As a result, adhesive wear is proportional to the normal force which is applied on the surface, sliding length and surface hardness of wearing material.

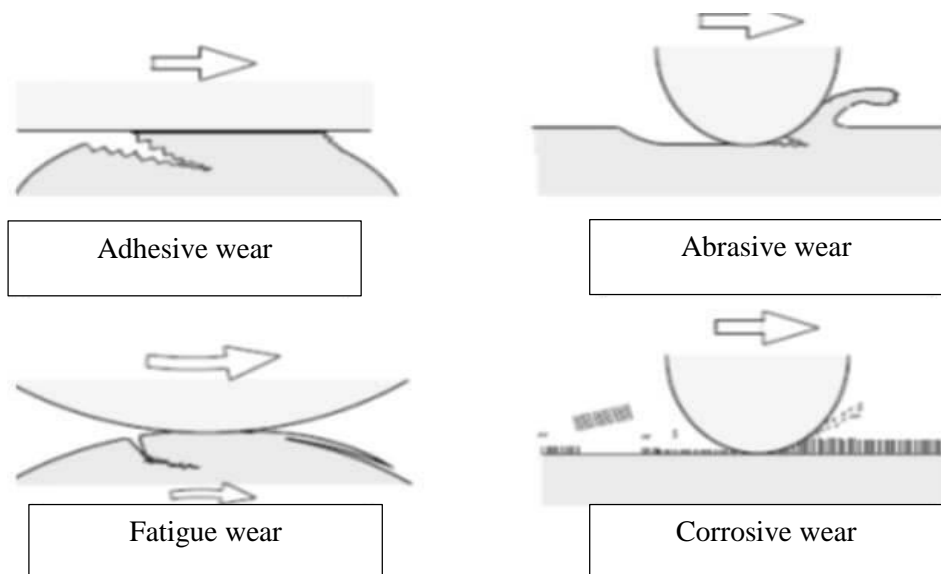


Figure 1.1 Wear mechanisms

Abrasive Wear:

Abrasive wear occurs during the sliding of two metal parts of contact which have different hardness values.

Non-metallic materials generally cause the wear in the form of scratches.



EXPERIMENT 1: WEAR TEST

Abrasive wear rate can be decreased by reducing of load applied to the surface of material. By this way, particles submerge to the surface of materials less and during removal of burrs less trace is leaved by particles. To reduce the abrasive wear:

- Use a harder alloy,
- Apply heat treatment to increase the hardness,
- Coating of hard layer on the surface of the material can be used.

If there are free abrasive particles between two metals or fix/free abrasive particles exist on a metal, these conditions will be grouped as:

Third body abrasive wear and
Two body abrasive wear.

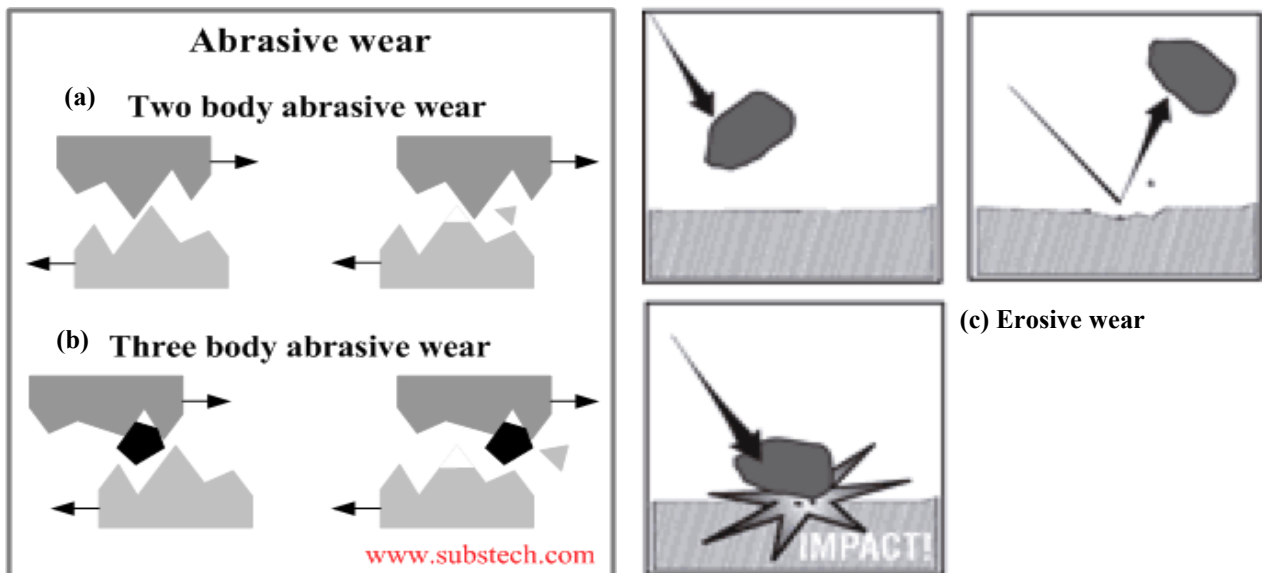


Figure 1.2 Schematic representation of a) Two body abrasive wear, b) Third body abrasive wear, c) Erosive wear

b. Classification in Terms of Physical Appearance of Damage

Sliding Wear:

- Created by grained minerals;

Sliding wear created by grained minerals is determined with respect to the hardness of mineral.

For the non-metallic hard materials, wear increases with increasing of hardness of mineral as well as metallic materials, but brittle fractures occur on the wear surface of hard material. Hardness of material is an important parameter to determine the wear resistance.

- Wear between metal-metal

Wear between the surfaces without lubrication depends on the surface conditions of a pair of material. Also, machining of sliding surfaces (surface roughness, direction of process) significantly affect the wear of materials.



EXPERIMENT 1: WEAR TEST

Erosion Wear:

Erosion (hydro-abrasive) wear is created by the fluids. During the flowing of liquids, gases, they remove the particles from the surface of materials by the effect of explosion or stroke at the surface of materials.

Corrosive Wear:

When wear and corrosion affect the material simultaneously, it is called corrosive wear. Chemical corrosion can occur together with the types of wear. Films are formed as a result of chemical reactions strongly attach to the surface inhibit the surface wear, but if films are brittle, they will increase the wear rate significantly because films are broken during the friction.

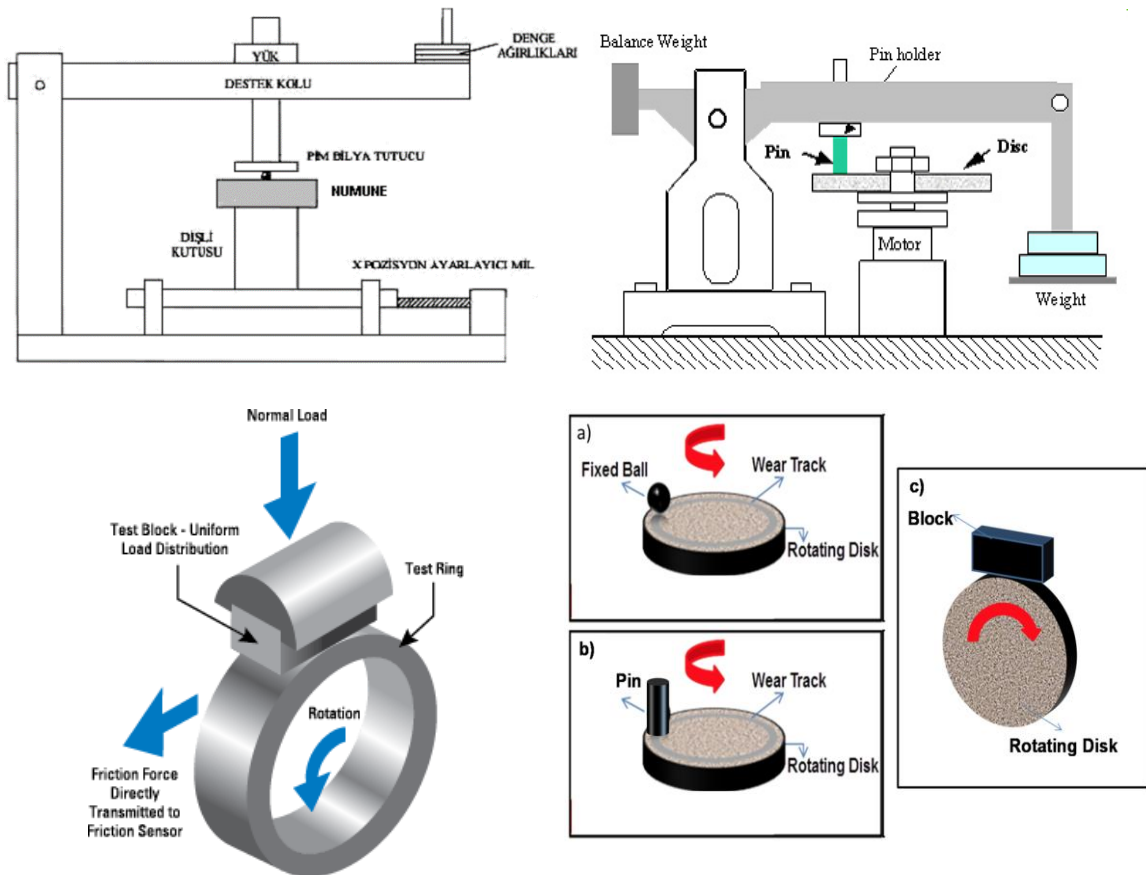


Figure 1.3 Experimental setups for wear test, a) Ball on plate, b) Pin on plate, c) Pin on roller, d) Abrasion test for rubber ring.

1.3 EQUIPMENTS AND MATERIALS

- Wear test sample
- Wear machine (Pin on Disks)
- Abrasive pin
- Computer
- Precision balance
- Experimental Parameters



EXPERIMENT 1: WEAR TEST

1.4 EXPERIMENTAL PROCEDURE

- 1) The weight of the wear sample should be measured before the test,
- 2) Set the sample on the wear machine
- 3) Check the surface of the pin
- 4) Put the pin on the sample gently.
- 5) Decide applied load, time, and number of turn.
- 6) Start the test,
- 7) Calculate the weight loss of the wear sample after the test

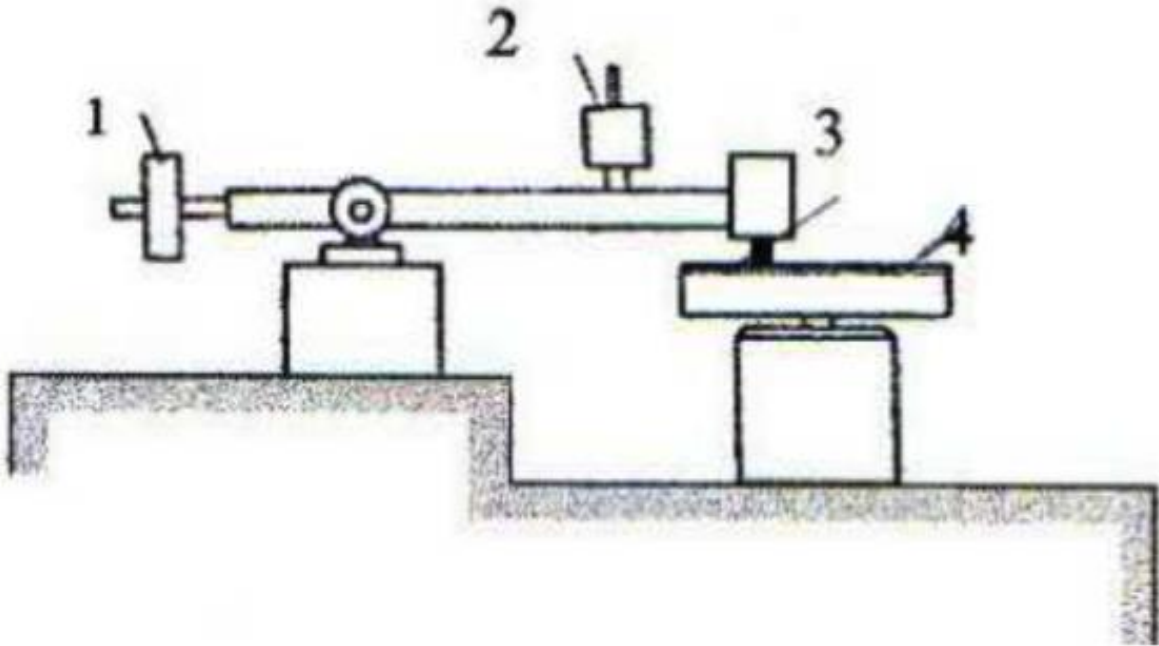


Figure 1.4 Pin abraded on sandpaper (Pin on Disk): 1. Counter load, 2. Load, 3. Sample, 4. Disk

1.5. REQUIREMENTS

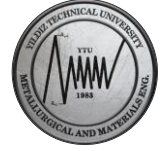
- Explain the type of wear
- Explain the factors affecting tribologic system.
- Draw the friction coefficient – time plot.

1.6 REFERENCES

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EXPERIMENT 1: WEAR TEST

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Metallurgical and Materials Engineering
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EXPERIMENT 2: LEACH AND CEMENTATION



2. LEACH AND CEMENTATION

2.1 OBJECTIVE OF THE EXPERIMENT

Investigation of copper solubility and parameters affect this solubility in acidic solutions of copper sulphate and chalcopyrite based copper ores. Precipitation of copper in the solution by using cementation process and determination of parameters which affect the efficiency.

2.2 THEORETICAL INFORMATION

Solutioning Process (Leaching)

Generally hydrometallurgical processes are carried out in two stages. First stage is the leaching process and in this stage is selectively solutioning of metals by interaction of ore minerals with an appropriate solvent. Second stage is regaining of metals from solutions.

Leaching is a wide spread process it includes sectors from mining to metal production. The solvent in the leach process can be water, acid, alkaline, salt based or combination of them. An the dissolving material can be ore, concentrated ore, free metal, compounds like sulphur, carbonate, silicate and sulphate or flue dust, intermediate product and any waste includes metal.

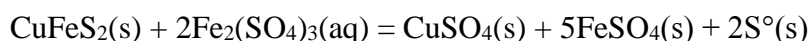
Ores can be subject to some preparation treatments between furnace productions and leach process. Pretreatments before the leaching are crushing, grinding, beneficiation and roasting.

Purpose of the leach process is increase selective dissolution efficiency, rapid dissolution efficiency and total dissolution efficiency. Beneficial minerals should be selectively dissolved in leach process. Because with selective leach, reactant consumption is minimized and clean solutions are obtained for metal regaining sequence. Solvent composition and concentration, dissolution temperature, time and solid in pulp ratios are main variables of the leach process that need to be controlled.

Copper ores usually are processed by pyrometallurgical routes for a long time. However pyrometallurgical techniques are recently replaced with hydrometallurgical ones. Sulphur dioxide (SO₂) emission, requirement of ore beneficiation, loss of other metals and high costs are disadvantages of pyrometallurgical techniques. In hydrometallurgical processes, main principal is dissolving of valuable elements by using selective reactants.

In hydrometallurgical technique, copper ore is dissolved with an appropriate leach solution after crushing and grinding processes. Then metallic copper is produced from this solution. Sulphuric acid, ammonia, ammonium salts, chloride-sulphate solutions, nitrate solutions and solutions include microorganisms can be used for leaching process of copper ores.

Chalcopyrite is the most common copper ore so hydrometallurgical researches and efforts are focused on this kind ores. Reactions of chalcopyrite mineral in the sulphuric acid leach solution are given below.





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EXPERIMENT 2: LEACH AND CEMENTATION



Roasting process before leaching can have some economic and environmental problems like pyrometallurgical techniques. These kinds of problems can be eliminated by direct leaching of sulphur based ores that grinded into right dimensions. Direct leach is easily applied to oxide based ores. However for sulphur based ores in atmospheric conditions highly oxidant environments ore in autoclaves high temperature and high oxygen pressure are needed.

Dissolution Mechanisms

Reaction rate in leaching; shows how the reaction product or reactant concentrations changes over time in the reaction. As it is known, the dissolution rate in leaching generally decreases with time. This situation; as the reaction time progresses, the surface area of the reaction becomes smaller due to the reduction of the solvent concentration or the formation of a protective layer on the surface (passivation). The dissolution rate is also directly dependent on the activation energy. The magnitude of the activation energy in leaching processes indicates the difficulty of dissolution. In other words, hardly soluble minerals have high activation energy.

Factors Affecting Leaching

Most hydrometallurgical processes involve heterogeneous reactions (the type of reaction between multiple phases). The rate of these reactions depends primarily on the hydrodynamics of the leaching system (such as mixing and diffusion) and the chemical interactions of the components present in the system. Clearly, leaching of any ore in an aqueous environment is influenced by the following factors:

1. Grain size of the ore,
2. Composition of the ore,
3. Composition and concentration of the solvent,
4. Contact time of the ore with the solvent,
5. Leaching temperature

Cementation

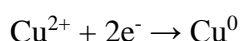
Any metal in the solution dissolves another metal (granule, plate, powder, scrap) that has a more negative electrode potential than it in the EMF series of metals is called “cementation”. The cementation process is applied either to the production of a metal or to the removal of impurities from the electrolyte. When copper production is taken as an example, cementation becomes a matter of evaluation of poor solutions.

Reactions occurring in copper cementation process:

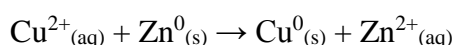
1. Formation of Zn^{2+} ions by anodic half-cell reaction:



2. Formation of metallic copper by cathodic half-cell reaction:



3. Total cell reaction:

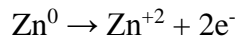


The pH value of the solution applied to the cementation is extremely important. Hydrogen ion concentration in acidic media is high:

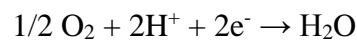


$2\text{H}^+ + 2\text{e}^- \rightarrow \text{H}_2$ the secondary reaction evolving according to the reaction is activated. Thus, there is unnecessary increase of cementator consumption. When cementation is being done, the solutions are either not mixed or very slowly mixed that not allowed to aeration. In case of excessive oxygen dissolves in the solution because of severe mixing metallic Zn and Cu move into ionic state as a result of oxygen reduction.

Anodic reaction:



Cathodic reaction:



Cementation Chemistry: If an iron component is immersed in an aqueous solution containing copper ions metallic copper tends to precipitate, while iron tends to go through the solution. Electrochemical potential is the driving force of this reaction. This potential is calculated with Nernst equation given in (2) below.



$$E = E^0 + (RT / nF) \times \ln(a_{\text{Me}}) \quad (2)$$

In this equation,

E = Real potential (V)

E^0 = Standard potential (V)

R = Gas constant

T = Temperature (K)

n = Oxidation level

F = Faraday constant ($\text{C} \cdot \text{mol}^{-1}$)

a = Activity

If the Nernst equation would apply to the copper iron couple;

$$E_{\text{Cu}} = E_{\text{Fe}} \quad (3)$$

$$E^0_{\text{Cu}} + (RT/nF) \times \ln(a_{\text{Cu}^{2+}}) = E^0_{\text{Fe}} + (RT/nF) \times \ln(a_{\text{Fe}^{2+}}) \quad (4)$$

$$0.34 + (0.059/2) \times \log(C_{\text{Cu}^{2+}}) = -0.444 + (0.059/2) \log(C_{\text{Fe}^{2+}}) \quad (5)$$

$$\log(C_{\text{Fe}^{2+}} / C_{\text{Cu}^{2+}}) = 26.57 \quad (6)$$

$$(C_{\text{Fe}^{2+}} / C_{\text{Cu}^{2+}}) = 10^{27} \quad (7)$$

This result theoretically means that there is only 1 copper ion (Cu^{2+}) per 10^{27} iron ions (Fe^{2+}) in solution after cementation. This very large ratio means that the reaction (1) completely moves to right side. According to reaction (1) 1 molar iron (55.85 g) cementite 1 molar copper (63.54



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EXPERIMENT 2: LEACH AND CEMENTATION



g). This value is determined as 1 kg iron per 1 kg of copper. In industrial applications 1.5-2.5 kg iron is required due to the following side reactions.



The oxygen of the air is directly oxidized +2 valence iron causing iron consumption.

The selection of metals in the cementation process is made according to the EMF series.

EMF Series

Reaction	$E^0, \text{V(SHE)}$	
$\text{Au}^{+++} + 3e = \text{Au}$	+1.42	Noble ↑
$\text{Pt}^{++} + 2e = \text{Pt}$	+1.2	
$\text{O}_2 + 4\text{H}^+ + 4e = 2\text{H}_2\text{O}$	+1.23	
$\text{Pd}^{++} + 2e = \text{Pd}$	+0.83	
$\text{Ag}^+ + e = \text{Ag}$	+0.799	
$\text{O}_2 + 2\text{H}_2\text{O} + 4e = 4\text{OH}^-$	+0.401	Reference ↓
$\text{Cu}^{++} + 2e = \text{Cu}$	+0.34	
$\text{Sn}^{+++} + 2e = \text{Sn}^{++}$	+0.154	
$2\text{H}^+ + 2e = \text{H}_2$	0.00	
$\text{Pb}^{++} + 2e = \text{Pb}$	-0.126	
$\text{Sn}^{++} + 2e = \text{Sn}$	-0.140	
$\text{Ni}^{++} + 2e = \text{Ni}$	-0.23	
$\text{Co}^{++} + 2e = \text{Co}$	-0.27	
$\text{Cd}^{++} + 2e = \text{Cd}$	-0.402	
$\text{Fe}^{++} + 2e = \text{Fe}$	-0.44	
$\text{Cr}^{+++} + 3e = \text{Cr}$	-0.71	
$\text{Zn}^{++} + 2e = \text{Zn}$	-0.763	
$\text{Al}^{+++} + 3e = \text{Al}$	-1.66	Active ↓
$\text{Mg}^{++} + 2e = \text{Mg}$	-2.38	
$\text{Na}^+ + e = \text{Na}$	-2.71	
$\text{K}^+ + e = \text{K}$	-2.92	

2.3 EQUIPMENT AND MATERIALS

1. Chalcopyrite ore
2. Copper sulphate
3. Sulphuric acid
4. Purified water
5. Precise scale
6. Heated Magnetic Mixer
7. Mixed water bath
8. Pipette, washing bottle, beaker, funnel, filter paper



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Element (wt %)							
	Cu	Fe	Co	Zn	Ni	Au (ppm)	Ag (ppm)
Küre ore	1.5-2	47-49	0.08-0.12	0.1-0.5	0.3-0.4	0-0,5	-
Küre) concentrated ore	18-20	47-49	0.08-0.12	0.1-0.5	0.3-0.4	4-4.5	20-22

2.4 EXPERIMENTAL PROCEDURE

1. The chalcopyrite concentrate which have been prepared for leaching after size reduction and drying are dissolved in sulfuric acid at 50 °C with 1/50 solid liquid ratio.
2. The amount of dissolved in solution is analyzed by Atomic Absorption Spectrometer.
3. The reason for the high or low dissolution efficiency value is discussed.
4. Prepare a CuSO₄ solution containing 5 g/200 ml. Cementation is carried out using Fe and Zn metal powders at different pH values (pH = 3, pH = 5) in order to observe the cementation process and to determine optimum metal precipitation conditions. The precipitate is separated from the solution by filtration.
5. Copper recovery rate is calculated for each test group.
6. All the results obtained are interpreted.

Calculations

Calculations for Leaching Process:

Leaching efficiency % = [Amount of Cu passing through the solution (mg / l) / Amount of Cu in the sample (mg / l)] x100

Calculation of Cementation Process:

% Cementation efficiency % = [amount of precipitated copper (g / l) / amount of copper in solution (g / l)] x 100.

2.5 ASSIGNMENTS AND REQUESTS

1. Calculate precipitated copper efficiency using the results of Cu analysis after precipitation and filtration of the precipitated parts obtained in the test results with CuSO₄ solution containing 5 g / 200 ml Cu with Fe and Zn powder.
2. Explain the relationship between precipitation efficiencies and pH values.
3. Which sementator provided higher efficiency? (Zn or Fe) Explain in detail.
4. Calculate the leaching efficiency of chalcopyrite concentrate. Is the efficiency value high or low? What is the reason of this situation?

HOMEWORK: Explain the working principle of the Atomic Absorption Spectrometer.

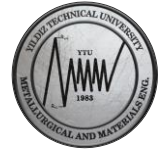


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EXPERIMENT 2: LEACH AND CEMENTATION



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3. TENSILE, IMPACT AND HARDNESS TESTING

PART 3.1: HARDNESS TESTING

3.1.1 AIM

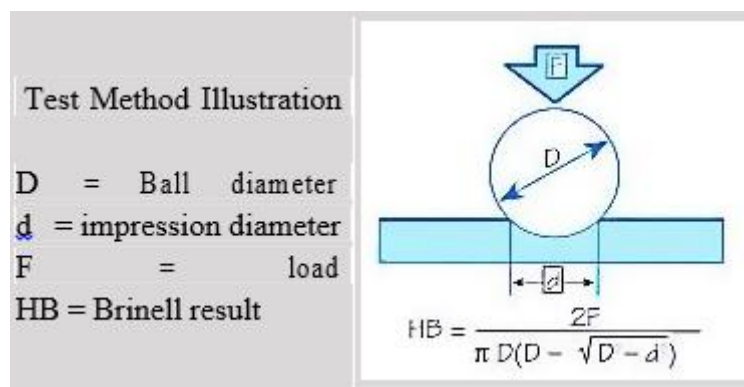
To introduce the principles of indentation hardness testing, emphasizing the limitations and significance of the results.

3.1.2 INTRODUCTION

Hardness is generally considered as resistance to penetration. The harder the materials, the greater the resistance to penetration. Hardness is directly related to the mechanical properties of the material. Factors influencing hardness include microstructure, grain size, strain hardening, etc. Generally, as hardness increases so does yield strength and ultimate tensile strength (UTS), thus specifications often require the results of hardness tests rather than tensile tests. The most popular methods are Brinell, Vickers and Rockwell hardness tests for metals and alloys.

BRINELL TEST

In a standard Brinell test 10 mm diameter hardened steel ball is forced to penetrate the material by 3000 kgf for steels and cast irons. The load and ball diameter selection is important depending on the hardness of materials and 500 kgf is used for softer materials with the same ball diameter. Keeping the ratio of load P to the square of diameter D^2 constant (30 for steels and cast irons and 5 for soft metals and alloys), different load and ball diameter combinations can be selected and used in Brinell hardness testing. The Brinell Hardness Number (BHN) is obtained by dividing the applied force P , in kgf; by the curved surface area of the indentation, which is actually a segment of sphere. The geometry of indentation is given in Figure-1, and the hardness is determined according to the relationship,



where D is the diameter of the indenter ball and d is the average diameter of the indentation, both in mm.

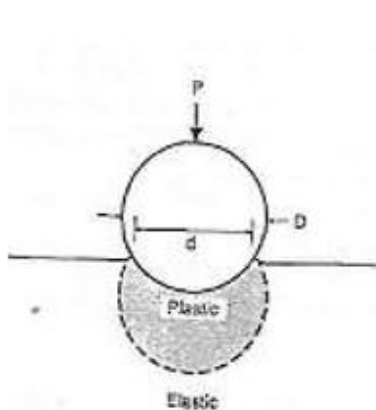
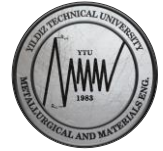


Figure-1: Geometry of deformation under a Brinell hardness indenter.

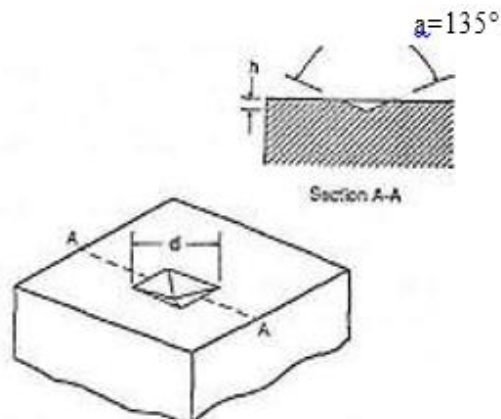


Figure-2: Vickers hardness indentation.

VICKERS TEST

The Vickers hardness test is based on the same principle as the Brinell test, except the indenter is a diamond pyramid with square base. The angle between the faces of pyramid is 136° as shown in Figure-2. The Vickers Hardness Number (VHN) of materials is obtained by dividing the applied force P , in kgf, by the surface of the pyramidal depression yielding the relationship

$$\text{VHN} = \frac{1.8544P}{d^2} \quad (\text{in some sources VHN is cited as DPH})$$

where d is the average length of diagonals in mm. Due to the shape and hardness of indenter the method is applicable to metals and alloys with wide variety of hardness. Test load is selected between 1 and 120 kgf depending on the hardness of materials. It is also possible to apply micro hardness testing by keeping the force between 5 grf and 2 kgf in Vickers scale.

ROCKWELL TEST

In the **Rockwell** test, a diamond cone or a hard steel ball is employed as the indenter depending on the hardness of materials. Diamond cone or *Brale* indenter with cone angle of 120° is used to test hard materials and the balls of sizes between 1.6 mm (1/16") and 12.7 mm (1/2") are used in testing softer materials. Rockwell tests differ from other indentation hardness tests in that the depth of indentation determines the hardness rather than the indentation size (see Figure3). Therefore, surface condition of specimens is very important in Rockwell testing because of its high dependency on the accuracy in indentation depth measurements. In order to establish a reference position a *minor load* of 10 kgf. is first applied, and the major load is then applied. Additional penetration due to *major load* is measured and readings are obtained from a calibrated scale (dial) directly, which has a maximum value of 100, depending on the depth of penetration. Most commonly used Rockwell hardness scales are given in Table-1 with typical applications. The hardness numbers are designated HR_X , where X indicates the scale used (i.e.



50 HRC for 50 points on the C scale of dial). It should be noted that a Rockwell hardness number is meaningless unless the scale is not specified.

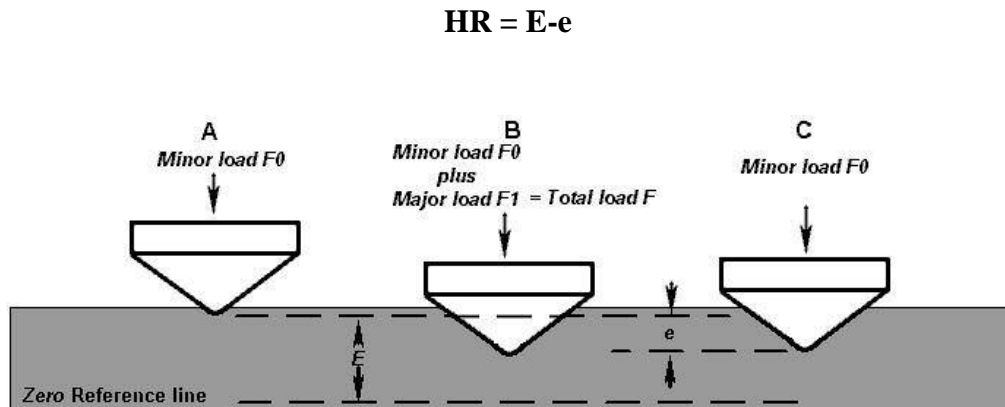


Figure 3.3: Increasing depth of penetration in the Rockwell test

Table 3.1: Commonly used Rockwell hardness scales.

Scale	Indenter Type	I Major Load (kgf)	Typical Applications
X			
A	Diamond Brale	60	Tool Materials
D	Diamond Brale	100	Cast Irons, Sheet Steels
C	Diamond Brale	150	Hardened steels and cast irons, Ti alloys
B	1/16" Diameter Ball	100	Annealed steels, Cu and Al alloys
E	1/8" Diameter Ball	100	Al and Mg alloys, reinforced polymers
F	1/16" Diameter Ball	60	Soft sintered products
M	1/4" Diameter Ball	100	Very soft metals, polymers
R	1/2" Diameter Ball	60	Very soft metals, polymers

Since the deformations caused by an indenter are of similar magnitude to those occurring at the ultimate tensile strength in a tension test, some empirical relationships have been established between hardness and engineering ultimate tensile strength of metals and alloys. For example, for steels UTS can be **roughly estimated** from Brinell hardness as follows:

$$UTS \text{ (in MPa)} = 3.45 \times \text{BHN}$$



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EXPERIMENT 3: TENSILE, IMPACT AND HARDNESS TESTING



3.1.3 EQUIPMENT

Rockwell hardness tester

Brinell hardness tester

Microhardness (Vickers) tester

Different test specimens

3.1.4 PROCEDURE

1. Determine the proper Rockwell hardness scale,
2. Calibrate the Rockwell hardness tester,
3. Take five readings per specimen,
4. Perform Brinell hardness measurements on designated specimens (3 readings)
5. Using microhardness tester, determine the VHN at 0.20 mm distances from the surface to the center of case carburized specimen, until the hardness remains unchanged.
6. Determine the core hardness of the carburized steel

3.1.5 ANALYSIS

Calculate the range and mean in hardness values for each test specimen which will be considered in lab report.

3.1.6 LAB. REPORT REQUIREMENTS

1. Results

List hardness values of Rockwell and Brinell for each specimen. List VHN versus distance from surface for carburized specimen.

Determine the effective carburizing depth that corresponds to 400 VHN.

2. Discussion (*only those indicated will be answered in the lab report*)

2.1. How do the Rockwell and Brinell tests actually measure hardness? Give any appropriate sketches and formulae. Are there any units involved? Describe the procedure for the Rockwell test, explaining the reason for the pre-load.

2.2. What is the limitation on the thickness of specimens for a hardness test? Explain. Calculate the minimum thickness for one specimen for the Rockwell test and one for the Brinell test.

3. What are the limitations for distance from specimen edge to indentation and distance between indentations? Explain why these limitations exist in both cases.

4. What surface condition is necessary for Brinell, Rockwell and Vickers?

5. Why is the mean pressure (stress) under the indenter much greater than the yield stress? How much greater is it?

6. What are the advantages of Vickers test against Brinell test?



3.1.7 REFERENCES

Metals Handbook, 9th ed., *Mechanical Testing*, Vol. 8, 1990.

G. Dieter, *Mechanical Metallurgy*, S1 ed., Mc Graw Hill, 1986.

N. Dowling, *Mechanical Behavior of Materials*, Prentice Hall, 1993.

"An Evaluation of the Impression Test for Estimating the Tensile Properties of Metallic Materials," H.N. Jones, *Journal of Testing and Evaluation*, Vol. 20, pp. 403-407, Nov. 1992.

PART 3.2: TENSILE TEST AND ANALYSIS OF MECHANICAL PROPERTIES OF A METAL

3.2.1 SUBJECT: Tensile testing and evaluation of tensile properties of materials.

3.2.2 OBJECTIVE:

- a) To become familiar with the principles and capabilities of mechanical testing machines.
- b) To carry out a tensile test on the given metal alloy and polymer specimens.
- c) To understand how to evaluate.
 - (i) Yield strength
 - (ii) Ultimate tensile strength
 - (iii) Fracture stress (normal and actual)
 - (iv) Percentage elongation
 - (v) Percent reduction in area
 - (vi) Modulus of Elasticity strength constant (K)
 - (vii) Strain hardening coefficient (n)

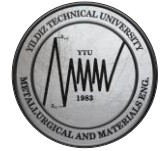
3.2.3 PROCEDURE:

The student will observe the testing of the various samples as the mechanical testing machine is operated by the instructor and/or Teaching Assistant.

1. Measure the specimen dimensions
2. The specimen is attached the grips of the testing machine (Figure 2).
3. An extensometer used to measure elongation, is attached to the gage-length of the specimen.
4. While the specimen is pulled in tension, the data acquisition software collects the load and extension data from the testing machine.
5. The specimen is pulled to failure.
6. The final dimensions of the specimen are measured.



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EXPERIMENT 3: TENSILE, IMPACT AND HARDNESS TESTING



Primary calculations made:

Note down the initial and final dimensions of the sample:

Initial length = L_0

Initial width = W_0

Initial thickness = T_0

Final length = L_f

Final width = W_f

Final thickness = T_f

Initial cross sectional area, $A_0 = W_0 T_0$

Initial volume, $V_0 = A_0 L_0$

Prep Work

Measure the initial diameters and cross-section areas of your specimen.

Mark a gauge length.

Record the direct results from the test.

Elastic Modulus = _____

Ultimate stress = _____

1. Define engineering stress, engineering strain, true stress, and true strain.
2. Check out the cross-sectional area of the test section. Is it your standard ASTM tensile specimen? Check the width and thickness at three different places for the appropriate cross-section dimensions.
3. Discuss the difference between elastic and plastic deformation.
4. Make a copy of the data files from the data acquisition software onto a memory stick.
5. On your engineering stress strain curves (see 'Results'), discuss and indicate elastic limit, upper yield point, lower yield point, ultimate stress, proportional limit, and fracture stress. Discuss what kind of properties would be observed if the material had a brittle fracture.
6. Discuss the classical 'cup and cone' fracture (ductile fracture) and type of fracture of cast irons.



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EXPERIMENT 3: TENSILE, IMPACT AND HARDNESS TESTING



7. List any possible sources of error in your testing and evaluation of engineering stress-strain curve.

RESULTS: Use a spreadsheet program (Microsoft Excel, etc.) to calculate and plot true stress-strain curves from the load-extension data collected from the data acquisition software.

Afterwards, calculate:

- i. Modulus of Elasticity, E , from the elastic region of stress-strain curves.
- ii. 0.2% offset yield strength.
- iii. Fracture strength. (normal and actual)
- iv. % Elongation at Fracture = $\frac{L_f - L_o}{L_o} \times 100$
- v. % Reduction of area = $\frac{A_o - A_f}{A_o} \times 100$
- vi. Value of the strength constant, K , and the strain hardening coefficient, n , in the plastic region using:

$$s_T = K \varepsilon^n$$

Actual strain = Elongation / gage length (gage length = 1 inch)

$$E = \Delta L / L_0$$

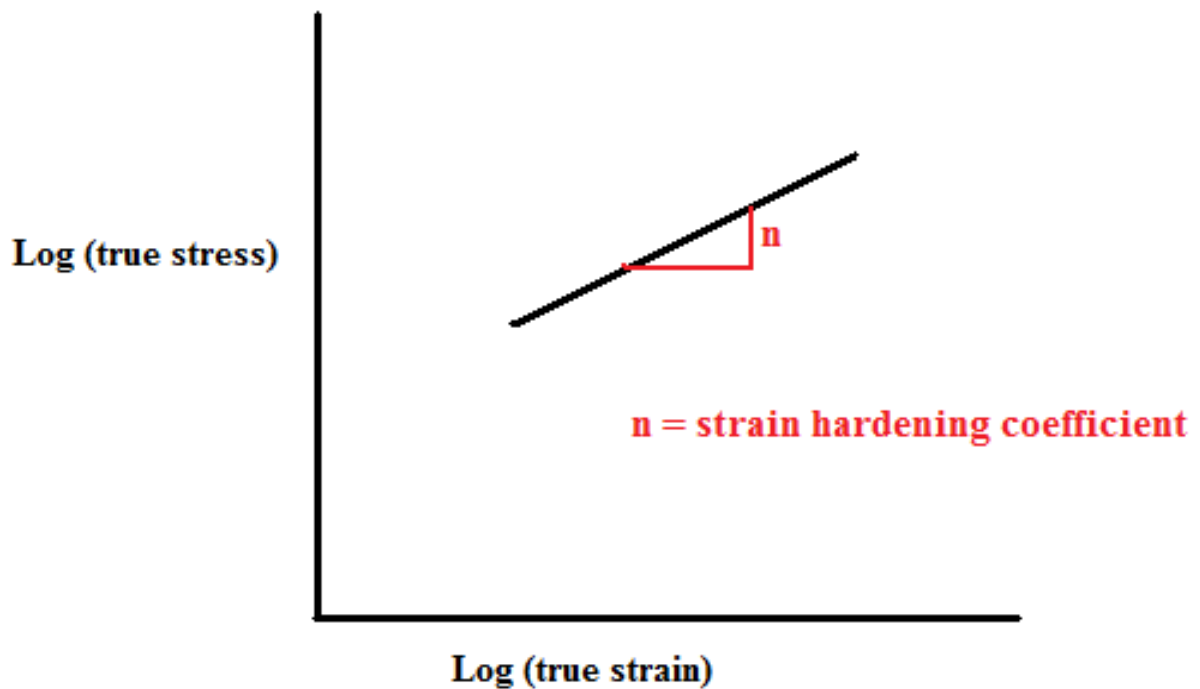
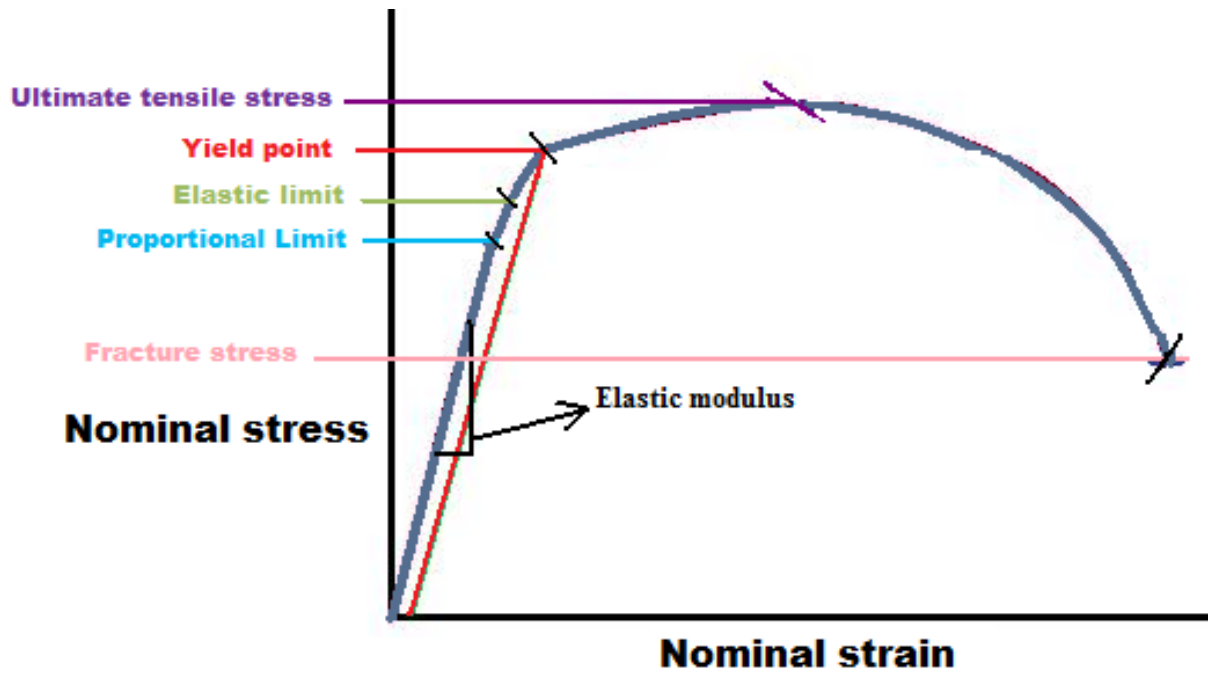
Actual Area = $V_f / (L_f + \text{Elongation})$

Nominal stress = Load / A_0

Nominal strain = Elongation / L_0

True stress = Load / Actual area = F/A_f

4. Draw a graph between Nominal stress (y-axis) and Nominal strain (x-axis); Log (True stress) (y-axis) and Log (True strain) (x-axis), from which the following can be calculated. Sample graphs are shown below:



Note: Actual stress and true stress are the same; actual strain and true strain are the same.

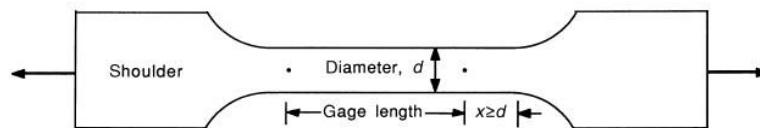


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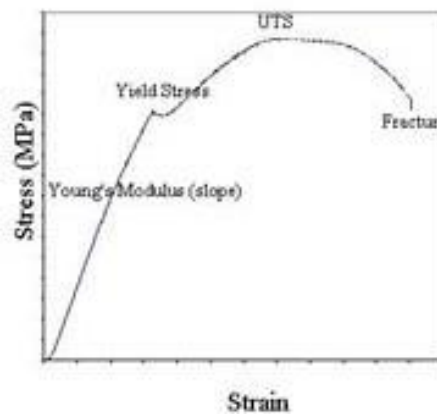
EXPERIMENT 3: TENSILE, IMPACT AND HARDNESS TESTING



A picture of Instron® universal testing machine in the Laboratory. More information about the tensile testing machines could be found from the references at the end of the guideline.

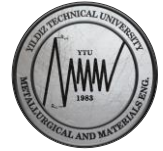


Typical tensile specimen



3.2.4 REFERENCES:

1. Annual Book of ASTM Standards, Vol. 03.01, Metals Tests methods and procedures, designation E8, 1995 (or the recent edition) It should be available in the Research Park Reference Library.
2. ASM Handbook, Mechanical Testing, Vol. 8, ASM-Intl., Materials Park, Ohio (available in the Research Park Reference Library).



PART 3.3: IMPACT TESTING

3.3.1 OBJECTIVE

To conduct Charpy V-notch impact test and determine the ductile-brittle transition temperature of steels.

3.3.2 EQUIPMENT

Coolants

Standard Charpy V-Notched Test specimens

Impact tester

3.3.3 INTRODUCTION

Notched-bar impact test of metals provides information on failure mode under high velocity loading conditions leading sudden fracture where a sharp stress raiser (notch) is present. The energy absorbed at fracture is generally related to the area under the stress-strain curve which is termed as toughness in some references. Brittle materials have a small area under the stress-strain curve (due to its limited toughness) and as a result, little energy is absorbed during impact failure. As plastic deformation capability of the materials (ductility) increases, the area under the curve also increases and absorbed energy and respectively toughness increase. Similar characteristics can be seen on the fracture surfaces of broken specimens. The fracture surfaces for low energy impact failures, indicating brittle behavior, are relatively smooth and have crystalline appearance in the metals. On the contrary, those for high energy fractures have regions of shear where the fracture surface is inclined about 45° to the tensile stress, and have rougher and more highly deformed appearance, called fibrous fracture.

Although two standardized tests, the Charpy and Izod, were designed and used extensively to measure the impact energy, Charpy v-notched impact tests are more common in practice. The apparatus for performing impact tests is illustrated schematically in Figure-I. The load is applied as an impact blow from a weighted pendulum hammer that is released from a position at a fixed height h . The specimen is positioned at the base and with the release of pendulum, which has a knife edge, strikes and fractures the specimen at the notch. The pendulum continues its swing, rising a maximum height h' which should be lower than h naturally. The energy absorbed at fracture E can be obtained by simply calculating the difference in potential energy of the pendulum before and after the test such as,

$$E = m.g.(h-h')$$

EXPERIMENT 3: TENSILE, IMPACT AND HARDNESS TESTING

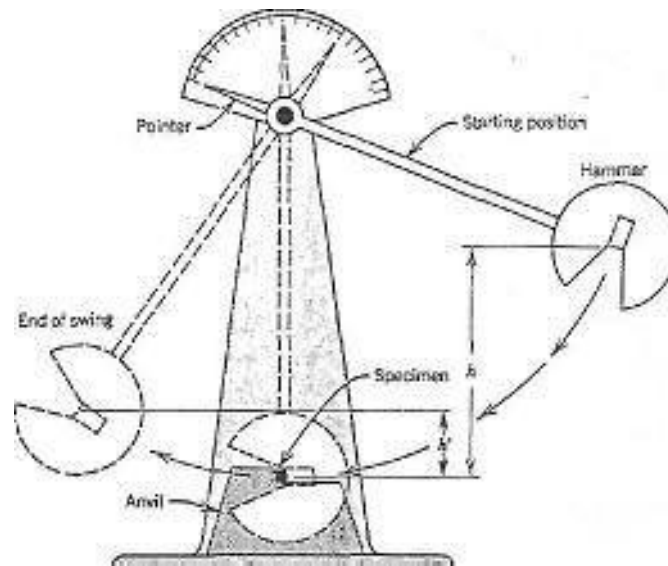


Fig. 1. Apparatus for impact testing of materials.

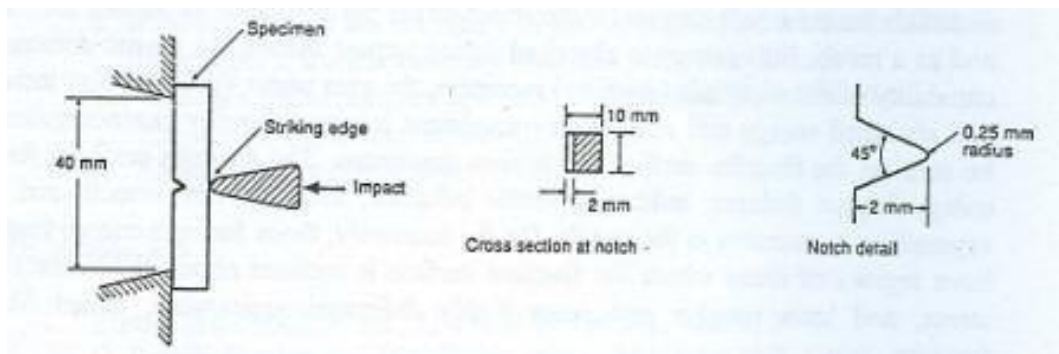


Fig. 2. Specimen and loading configuration for Charpy V-notched impact test.

Body centered cubic (bcc) metals, particularly steels, often exhibit a decrease in impact energy as the temperature is lowered. The temperature at which a sharp decrease in impact energy occurs is called the ductile-brittle transition temperature (DBTT) as shown in Figure-3 schematically. This transition temperature is generally chosen as a lower limit for the application of such metals.

Some steels may show transition characteristics in their failure mode from ductile to brittle gradually as temperature is decreased, which is given in Figure-4 schematically. In this case, different approaches may be used in determining transition temperature but the average energy concept is the most popular one. Determination of transition temperature can also be done by examining the fracture surfaces of specimens tested at different temperatures. For example, the temperature, at which the fracture surface consists 50 percent cleavage (crystalline) and 50 percent ductile (fibrous) types of fracture, is called fracture appearance transition temperature (FATT). Another common criterion is to determine the transition temperature on the basis of

an arbitrary energy absorbed. For example, *20 J transition temperature* is an accepted criterion for low-strength ship steels.

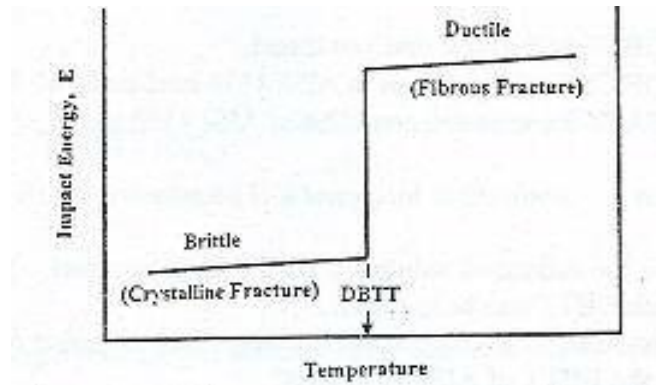


Fig. 3. Typical ductile-brittle transition curve for annealed low carbon steel.

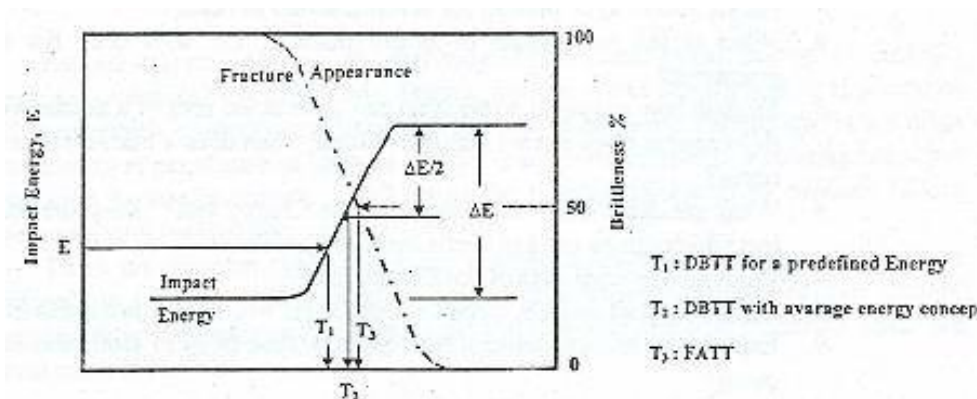


Figure-4: Various criteria of transition temperature obtained from Charpy tests.

3.3.4 PROCEDURE

Note: Test one material with two specimens for each temperature,

1. Check the zero calibration of the impact tester.
2. First, test a specimen at room temperature.
3. Based on the room temperature result, decide whether to concentrate on higher or lower temperature.
4. Test specimens over a selected range of temperatures, attempting to establish fully ductile test, a fully (or nearly) brittle test, and as many temperatures in between as possible.
5. Note fracture energy and estimate the % brittleness from the appearances of fracture surfaces of the specimens.



3.3.5 LAB REPORT REQUIREMENTS

1. Results

Display the test data in a Table.

Plot impact energy versus temperature and % brittleness versus temperature.

Find DBTT of the steel that you tested.

2. Discussion (*the questions will be answered in the lab report*)

Give the estimated values of DBTT for your steel. Suggest 2 ways in which DBTT can be lowered.

What are the 3 basic factors which contribute to brittle fracture of steels? Do all 3 have to be present for brittle fracture to occur?

Explain how a triaxial stress state can arise at the root of a notch. Would this occur in thick or thin material? Thus, when does a biaxial stress state occur.

What are the main uses of the Charpy test?

List the ASTM and TS specifications for the two impact tests with titles.

Explain the relation between fracture toughness (K_{IC}) of steels and impact energy.

Explain the effect of carbon content on transition behavior of plain carbon steels in annealed condition.

Explain the effect of manganese on DBTT of steels.

REFERENCES

1. Metals Handbook, 9th ed., *Mechanical Testing*, Vol. 8, 1990.
2. G. Dieter, *Mechanical Metallurgy*, SI ed., Mc Graw Hill, 1986.
3. N. Dowling, *Mechanical Behavior of Materials*, Prentice Hall, 1993.
4. ASTM and TS Standards.
ASM Metals Handbook, 9th ed. Vol. 12.



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EXPERIMENT 4: POWDER METALLURGY



4. POWDER METALLURGY

Purpose of Powder Metallurgy: Metal and metal alloys are made a durable body with the help of pressure and temperature without melting their powder. This heat treatment, called sintering, replaces melting and is carried out at a temperature below the melting point of the metal powder used. In the sintering of single component powders, the sintering temperature can be lower than the melting temperature of the material (approx. 80% of the melting temperature). In multi-component systems, the sintering temperature can be selected from the components just below the melting temperature of the lowest melting temperature. This type of sintering is called solid phase sintering. In addition, sintering temperature in multi-component systems can be taken above the melting temperature of at least one of the components, such sintering processes are also called liquid phase sintering.

Why Powder Metallurgy Manufacturing Method?

The parts manufactured with T / M can be examined in two main groups depending on the preferred reason of this method.

Group 1: Parts of which the T / M method is more economical, although they can be manufactured by other methods.

Group 2: T / M's only alternative parts

Powder Metallurgy Processes

Powder metallurgy technique consists of 3 stages.

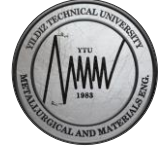
1. Powder production,
2. Pressing (powder particles into a single part with various operations),
3. Sintering and final parts production with secondary operations if necessary.

PART 4.1: APPARENT DENSITY (WET DENSITY)

4.1.1 PURPOSE OF EXPERIMENT: Determining the loose, uncompressed density of the powder and determining the conformity to the standards.

4.1.2 TOOL, DEVICE AND MATERIALS USED: Scale, metal powder, standard test apparatus

4.1.3 THEORETICAL KNOWLEDGE AND EXPERIMENT: The bulk density is the loose (uncompressed) density of the powder (g / cm^3). In order to determine this density, the size and shape of the powder is poured from a funnel specified in the standards and is provided to fill the cylindrical container with a free fall. The height between the funnel outlet and the upper surface of the cylindrical container is a constant value. Carefully peel off the excess powder in the cylindrical container which is filled and weigh the powder inside. Since the test container volume is known (25 cm^3), the bulk density is easily calculated as (g / cm^3). The ISO standard number of this experiment is 3923. All conditions and experiments were carried out. In Figure 1, the dimensions of the equipment used for this experiment are given schematically.



The apparent density of stainless steel powders produced by water atomization varies between 2.80-3.20 g / cm³. This value can be up to 5 g / cm³ for dusts produced by gas atomization. This value varies between 2.80-3.00 g / cm³ for copper powders and 3.00-3.25 g / cm³ for bronze powders. The bulk density is a very important factor in filling the mold of the powder during the pressing step. The bulk density is closely related to the shape, size and distribution of the powder grain. As the grain shape moves away from the sphere, the intergranular space rate decreases.

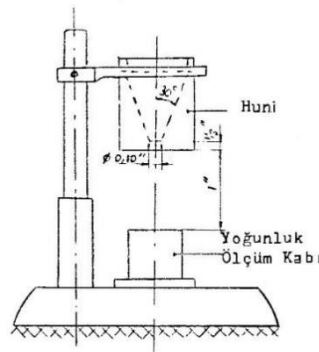


Figure 4.1 Measurement of Masonry Density

PART 4.2: FLUIDITY TEST

4.2.1 PURPOSE OF EXPERIMENT: Determination of the desired flow time from the powders and their compliance with the standards

4.2.2 TOOL, DEVICE AND MATERIALS USED: Scale, metal powder, standard test equipment, stopwatch

4.2.3 THEORETICAL KNOWLEDGE AND EXPERIMENT

The purpose of this test is to determine the fluency values of raw materials from X company. This rate is defined as the time that 50 grams of powder must pass through a 2.54 mm funnel. It has a flow time of 15 seconds for spherical stainless steel. This ratio varies from 25 to 30 seconds for irregularly shaped powders.

Fluency; the size and shape of a given amount of a powder type or mixture thereof is the ability to flow from a predetermined funnel. In these measurements, the flow time of the powder sample of 50 g of powder is generally determined and this value is considered as the flux of the powder. Funnel dimensions and shape with flow hole dimensions (hole diameter and length) are specified in the standards (ISO 4490). The experiment is usually repeated three times and the mean value is calculated. Its fluency also depends on the powder size, grain size, specific surface size and powder grain shape. Fluency increases as the grain size decreases.

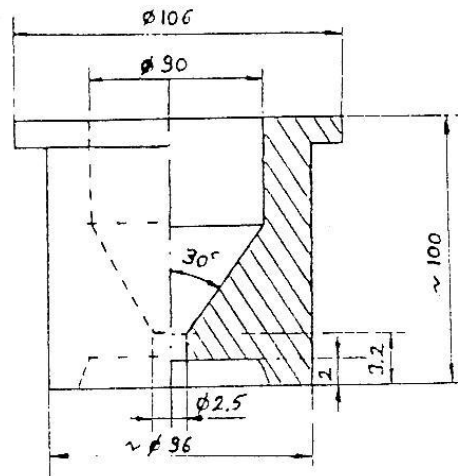


Figure 4.2 An Experimental Funnel for Determining Fluency is Shown Schematically.

PART 4.3: COMPRESSIBILITY (GREEN DENSITY)

4.3.1 PURPOSE OF EXPERIMENT: Determination of the theoretical density of the powder after pressing.

4.3.2 TOOL, DEVICE AND MATERIALS USED: Scales, metal powder, press, caliper, mold.

4.3.3. THEORETICAL KNOWLEDGE AND EXPERIMENT

This property corresponds to the density obtained when the powder is under pressure. This is the density reached after pressing. Austenitic stainless steel powders have better compressibility than martensite. compressibility; The yield limit is a function of the sample's hardness, the softness of the particles, the void ratio of the sample, the shape and size of the grains.

If the metal powders are formed in the mold, it is provided that the powder free to fill the mold cavity very well and fully (high fluency capability) as close to the theoretical density as when it is pressed into the mold cold (when stapled).

The softer the material, the higher its compressibility. The compressibility is also closely related to the friction between the beads and the molds in the pressing of the powder grains. The theoretical density of the component: for example, the green density obtained for Fe is 6.65 g / cm³, Fe: 7.87 g / cm³: $(6.65: 7.87) * 100 = 84.5\% = \% \text{ theoretical density.}$

- 84,5% Filled
- 15.5% Pore



PART 4.4: SIEVE ANALYSIS, DIMENSIONAL DISTRIBUTION AND CALCULATION OF AVERAGE PARTICLE SIZE

4.4.1 PURPOSE OF EXPERIMENT: Classification of powder particles of different sizes according to their size and calculation of average powder particle size.

4.4.2 TOOL, DEVICE AND MATERIALS USED: Metal powder, sieves, scales with different apertures.

4.4.3 THEORETICAL KNOWLEDGE AND EXPERIMENT

The calculation of the distribution of certain particles of powder according to the shape and weight of the grain particles. In this method, the top layer of the sieves, which are lined up from the top to the top of the bottom, are put on dry weight (weighing 100 or 50 grams) of metal powder and vibrated for a period of time on each sieve. The grains are weighed precisely. A table is organized as follows. The screens must be anti-magnetic.

Table 4.1 Sieve Analysis Chart (example)

Aperture size d (µm)	Oversize ΔR (% volume)	Residue on sieve R (%)	Undersize powder amount D(%)	Average grain size for each sieve da (µm)
0.400	0	0	100	0.45
0.315	5.5	5.5	94.5	0.36
0.200	20.5	26.0	74.0	0.26
0.100	42.0	68.0	32.0	0.15
0.063	23.0	91.0	9.0	0.08
0.040	6.6	97.6	2.4	0.05
0.040	2.4	100.0	0.0	0.02

The values in this table: $d_o = \sum \Delta R * d_a / 100$

d_o : Arithmetic mean grain diameter

%R: Quantity on each sieve, %

R: Sum of sieve size, %

d_a : average grain size for each sieve, mm

D: Amount passed through the screen, %

Example:

$$d_o = (0 * 0.045) + (5.5 * 0.36) + (20.5 * 0.26) + (42 * 0.15) + (23 * 0.08) + (6.6 * 0.05) + (2.2 * 0.02) / 100 = 0.136 \mu\text{m}.$$



PART 4.5: COMPRESSING

4.5.1 PURPOSE OF EXPERIMENT: Determination of pressure-density relationship for different pressures by shaping powders in molds

4.5.2 TOOL, DEVICE AND MATERIALS USED: Metal powder, mold, press

4.5.3 THEORETICAL KNOWLEDGE AND EXPERIMENT

Densification in Powder Metallurgy is the second important process. Pressing is that prepared metal powder mixture is compressed to reach the predetermined density in the appropriate press. It forms the metal powder filled into the press mold into a solid object. In order to achieve a high degree of pressing, a well-formed powder and high pressing pressures are required. The density achieved by pressing affects the mechanical properties of

The parts produced by powder metallurgy in the first degree.

Pressing is mostly cold (room temperature), but in special cases it is also hot. By pressing, the metal powder is given a desired level of density and mechanical strength according to the size and shape of the piece to be produced.

There are three basic steps in the mold condensation process.

1. Fill the mold cavity with the specified amount of metal powder,
2. Compression of the powder by means of staples,
3. Removing the shaped part from the mold.

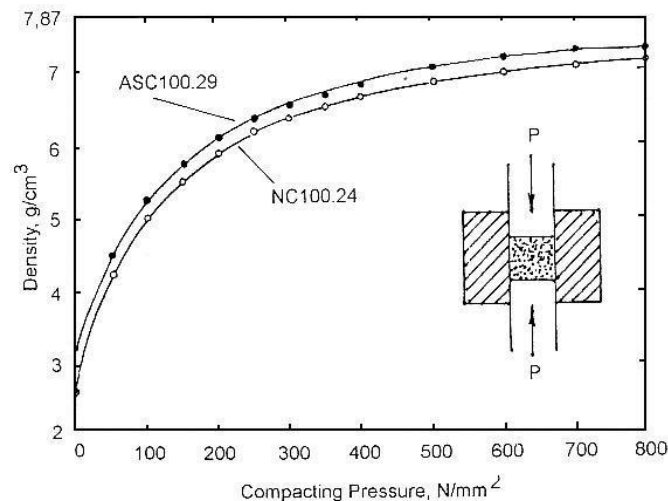


Figure 4.3 Density-pressure relationship for two commercial iron powders

- * pressure \uparrow , density \uparrow , pore \downarrow
- * pore \uparrow , density \downarrow
- * pore \uparrow , strength \downarrow
- * pore \uparrow , lubricant ability \uparrow



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Porosity: Sintered bushing, oil pump rotor, gears, bearing material etc. used in places. (After the production of the parts is completed, the powders in the pore are vacuumed, oil is given under vacuum, air is taken from the pores and oil is pumped into the cavities).

PART 4.6: SINTERING PROCESSES APPLIED TO METAL POWDERS

4.6.1 PURPOSE OF EXPERIMENT: Effect of sintering process conditions on structure and properties

4.6.2 TOOL, DEVICE AND MATERIALS USED: T / M sample, atmosphere controlled oven.

4.6.3 THEORETICAL KNOWLEDGE AND EXPERIMENT

Sintering is the heat treatment applied on the powder without disrupting the shape of material. Prestige shaped powder is not suitable for use in this form. However, the required strength increases with sintering. In this process, diffusion occurs between the powder grains at certain temperature and time, and weak mechanical bonds become strong mechanical bonds. Sintering temperature and time; A suitable sintering temperature and time should be determined according to properties desired for the parts. The main reason for the low density and strength in the parts is the very low sintering temperature and time. The sintering temperature should be as close as possible to the melting temperature of the powder.

Table 4.2 Sintering temperature and time of some metals and alloys

Powder materials	Sintering temperaturee (°C)	Sintering Temperature Standby Time (min)
bronzes	760-871-820	10-20
Brasses	843-898	10-45
copper	843-898	12-45
Steel, C' steel	1010-1148	8-45
Stainless steels	1033-1287	30-60
Fe (ferrit)	1204-1482	10-600
nickel	1010-1148	30-45
Alnico magnets	1204-1301	120-150
Tungsten Carbides	1426-1482	20-30
Molybdenum	2054	120
Tungsten	2343	480

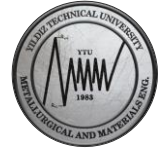


Metallurgical and Materials Engineering
MSE3961 Laboratory-1 Experiment Booklet
EXPERIMENT 4: POWDER METALLURGY



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5. RECYCLING

5.1. OBJECTIVE OF THE EXPERIMENT

The objective of this experiment is to inform the students about secondary aluminum production processes from the scrap.

5.2. THEORETICAL INFORMATION

FUNDAMENTAL DEFINITIONS

Waste: Materials and items that need to be disposed or already disposed.

Reuse: Repeatedly using of wastes until they reach the end of their economic lifetime, without any treatment other than collecting and cleaning.

Recycling: Operation of bringing materials back to manufacturing processes as a raw material after their lifetime has ended.

Recovery: Physical, chemical or biochemical methods to transform the components of the waste into other products or different forms of energy depending on the characteristics of the waste. Definition of recovery also includes both reuse and recycling phenomena.

Recycling Rate: Ratio of the total amount waste recycled to the total amount of waste generated.

The main scraps used for the aluminum recycling are;

- Beverage cans,
- Aluminum foil wastes, cake pans, and some other kitchenware,
- Plates,
- Window profiles made of aluminum,
- Outdoor and patio furniture,
- Cables,
- Aviation scraps,
- Automotive scraps,
- Extrusion, casting and machining processes sourced scraps.



Figure 5.1 Images of some aluminum scraps



EXPERIMENT 5: RECYCLING

At the first step, soiled and used aluminum pieces which may be painted or coated are delivered to the conveyor with other packaging wastes. Throughout the conveyor, waste pile is passed through some large magnets in order to remove iron-based and some other metals. Separated aluminum wastes are crushed and baled via scrap baling machine thereafter to be delivered to production facility. Main reason of baling is minimizing the logistic expenses. Baled aluminum may contain excessive moisture which may cause burst in furnace at high temperatures. In order to prevent this incident bales are crushed first. Afterward, crushed pieces are placed into hot furnace for removal of present paint, moisture and other pollutions at working temperature. After removal stage, aluminum is molten for forming processes at 720 °C in a rotary furnace with accompanying stirring to attain complete melting. Process gases need to be removed with powerful fans then to be cooled and treated in industrial filter bags for removal of residual particles before releasing to atmosphere through high chimneys. Molten aluminum accumulated at the bottom of the furnace is collected to cast into desired moulds. After several steps they can be used for diverse applications again.

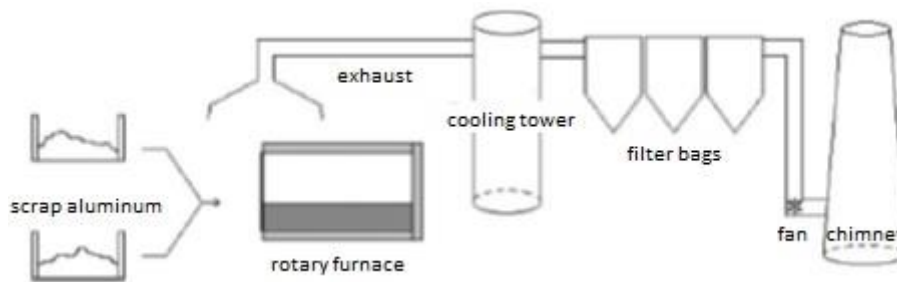


Figure 5.2 Image of aluminum recycling unit

Recycling of used aluminum not only creates more space for wastes, it also reduces the consumption of bauxite ore and energy.

When one ton of aluminum is produced from used aluminum;

- 1.300 kg bauxite,
- 15.000 liter cooling water,
- and 860 liter process water are consumed less.
- 2.000 kg CO₂ and 11 kg SO₂ are less emitted.

Required energy for producing aluminum from scrap is less almost twenty times than required for producing from bauxite ore. Also it reduces the emission of pollutant gases by %99, greenhouse gas emission by %95 and waste water pollution by %97 rate so recycling contributes energy-saving furthermore it is environment friendly.

IRON AND STEEL RECYCLING

Iron is third most abundant metal in the Earth's crust. The metal iron can be obtained from iron ores and it rarely exists as the element iron. Haematite, limonite, goethite, magnetite, siderite and pyrite can be listed as the well-known iron ores. To obtain metallic iron, the impurities in the ore must be removed by chemical reduction and it is mainly used in steel making which can be regarded as a carbon alloy. When iron is heated with carbon to a temperature of 1150-1200 ° C, the resulting melt is an alloy containing 96.5% Fe - 3.5% C



EXPERIMENT 5: RECYCLING

and called cast iron or pig iron. This product can be cast in fine details, although it remains extremely fragile to be formed as long as it is not decarburized to remove the most of contained carbon.

Classification of steel scraps by source:

- Circulating scrap; scraps that are acquired from units like steelworks, foundry and rolling plants with well-known chemical analysis and high quality.
- Old Scrap; especially automobile bodies sourced scraps which containing slight amount of phosphorus and sulfur.

For iron and steel recycling, foreign substances in the scrap which have lower oxygen affinity than iron like copper and tin should be taken care of during the process.



Figure 5.3. Images of iron and steel scraps

Recycling in iron and steel industry is mainly carried out in electric arc furnaces which is a good option for iron and steel making. Unlike the basic oxygen steelmaking method, cold metal (scrap) is used in electric arc furnaces instead of hot metal. The scrap steel is poured into an electric arc furnace with a crane from the top and furnace is closed with a cover subsequently. This cover carries three electrodes those dip into the furnace. The electricity passing through the electrodes creates an arc, and the heat that comes out melts the scrap. The amount of electricity used in this process is high enough to meet the energy needs of a city with 100,000 people. Other metal alloys can be added to obtain the required chemical composition in the smelting process. The oxygen is also blown to purify the desired steel iron.



Figure 5.4 Image of operating electric arc furnace

EXPERIMENT 5: RECYCLING

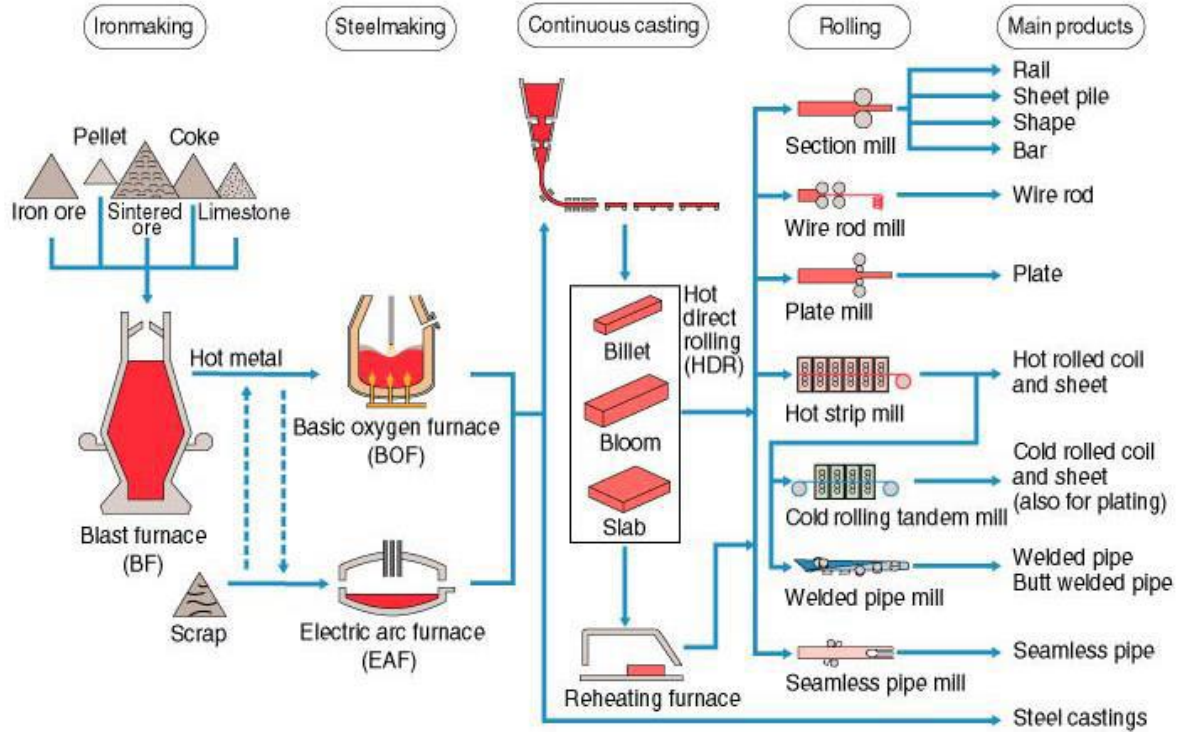


Figure 5.5 General flow scheme of iron and steel making

RECYCLING OF COPPER

Following scraps are generally used for secondary copper production;

- Wire rods
- Heat exchangers
- Copper tubes
- Cables and electrical connectors
- Electromotors, generators, transformer coils
- Miscellaneous copper plates and sheets.



Figure 5.6 Images of copper scraps



EXPERIMENT 5: RECYCLING

Copper production from scrap is usually made in two ways;

1. Melting at a copper smelter along with copper ore
2. Melting along with a proportion of copper ingots at a dedicated copper recycling plant

At a copper smelter, the scrap copper and alloy components are loaded into the furnace which is then fired up. The copper ore is fed into the furnace along with the required amount of limestone and sand. Oxygen and air are supplied and when the mix has become molten, it is then tapped into rectangular molds. The resultant rectangular plates are then purified to 99.9% pure copper using electrolysis processing.

At a copper recycling plant, the bales of compacted copper along with the required content of alloys are loaded into a furnace along with a proportion of pure copper ingots.

The furnace can either be of an electric induction type or a reverberatory one. The electric furnace melts all the contents and tips to pour the molten copper through a spout into molds for either further processing or they are shipped to a copper smelter.

A reverberatory furnace is a square box structure lined with firebricks and is usually gas fired, having a loading door at the front which can be lowered or raised as required. Once the copper is molten, samples are taken, and when approved, the molten copper is poured into molds and left to cool. The resultant copper ingots can be sent for further refining or dispatched to a copper smelter for further processing.

- The cost of copper from scrap is lower than from ore. The recycling of 1 ton of copper from the waste/scrap saves 85% of the total energy consuming comparing to primary production.
- Considering that 12-13% of the reserves are used in the world, it seems that their life span is limited, via recycling the existence of raw material resources is extended.
- The storage and removal of municipal solid waste are highly expensive processes. With recycling operations this expenses can be lowered and those waste product can be reused as raw materials.
- Recycling is more environment friendly process due to less gas and dust releasing during the whole steps.

5.3 EQUIPMENT AND MATERIALS

Materials

- Aluminum scrap
 - Thick scrap (Excessive parts of cast products)
 - Fine scrap (Chips from machining process)
- Flux (composition will be given during the experiment.)

Devices/Equipment

- Melting crucible
- Melting furnace
- hydraulic press
- Cylindrical steel mould



EXPERIMENT 5: RECYCLING

- Oven
- Precision scale
- Mould for casting

5.4 EXPERIMENTAL PROCEDURE

On the first step, thick scrap at the half of the total charge is weighed and placed in the furnace with melting crucible at 730 °C for melting. Liquid metal forms in the crucible at the end of this process. During the melting of thick scrap, fine scrap chips are briquetted using a hydraulic press in a cylindrical steel mold. At this step, compressive pressure of the press and fill factor of the briquettes will be measured. Before being placed in the melting crucible, briquettes are preheated for some time. Preheating time and temperature will be determined during the experiment. Subsequently, the briquettes are added to the liquid metal pool in the crucible. During the melting process of the briquettes, the powdered chemicals that will form the flux are weighed on a precision scale and mixed in a container. The mixture is added into the crucible and mixed in liquid metal with a metal rod. After standing in a furnace for about ten minutes, liquid aluminum is cast into a metal mould and solidified. On the last step cast metal will be weighed to calculate total melt and recycling efficiency.

5.5 ASSIGNMENTS AND REQUESTS

Whole necessary calculations will be made by students and further assignments will be given during the experiment.

5.6 REFERENCES

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6. CARBOTHERMIC REDUCTION

6.1 OBJECTIVE OF THE EXPERIMENT

The objective of this experiment is to investigate the thermodynamic and kinetic aspects of the solid state reduction of ferrous raw materials using carbon (carbothermic reduction).

6.2 THEORETICAL INFORMATION

The iron and steel industry is a sector that produces products with the desired chemical and physical properties by melting iron ore in blast furnaces or scrap in arc furnaces. The finished steel products have numerous types and uses. Changing consumer needs, developments in technology and competition are further increasing the product diversity in the iron and steel industry. The most important heavy industry, iron and steel industry, provides raw materials to many significant industries like construction, infrastructure, automotive, white goods and industrial machinery industries. Therefore, the industrialization of a country is directly related to its strong iron-steel industry and consumption [1-3].

Two different methods are used for production of iron and steel products. These are production in integrated plants from ore and production in electric arc furnace plants from scrap. In integrated plants, the iron and steel production process begins with the preparation of iron ore by sintering or pelletizing, or charging the iron ore directly to the blast furnace. In blast furnaces, the ore is reduced with the aid of coke, and liquid iron is obtained. The resulting liquid pig iron is converted to steel in a basic oxygen converters. In the electric arc furnace, the steel scrap is melted in the arc furnaces and the desired chemical composition is obtained [4-6].

China, Japan and U.S.A. are the iron and steel top producers around the world. Nearly half of the world's iron and steel production is carried out by China. Turkey ranks 8th in the world with a share of 2.08% in iron and steel production and second in Europe. According to 2014 data, 10,283 million tons of 34,035 million tons of crude steel produced in Turkey is equivalent to 30,21% of the total amount of raw steel produced in our country, while 23,752 million tons corresponding to 69,79% are produced by 28 electric arc furnaces [1-3].

Iron is most abundant in the form of oxides as hematite (Fe_2O_3) and magnetite (Fe_3O_4). Removal of oxygen from the structure of oxides and transforming it to their lower oxides and/or metallic phase by using reductants (CO , H_2 , C), which have higher affinity to oxygen, is called reduction.

Reduction of iron oxides is conducted technologically by two different methods. The first method is the indirect reduction with CO gas resulting from the reaction of coke combustion (CO_2 formation), and reduction of CO_2 by carbon in accordance with Boudouard Reaction, in the blast furnace, which is the first stage of steel production in integrated plants and where liquid pig iron production is performed (indirect reduction). A part of the reduction takes place in the lower zones of the blast furnace directly by solid carbon (direct reduction). The temperature in the blast furnace tuyere zone is up to $2000\text{ }^\circ\text{C}$ and the resulting product is liquid crude iron (pig iron). A modern blast furnace produces over 10,000 tons of liquid crude iron per day [4-6].



EXPERIMENT 6: CARBOTHERMIC REDUCTION

The other reduction method is a process in which iron oxides are converted to solid metallic phase without melting by solid or gas reducing agents. This method, known as direct reduction, produces sponge iron (Direct Reduced Iron) with high metallization ratio. Due to its low trace element and composition stability, sponge iron is an alternative to high quality steel in electric arc furnaces for high value added products. This method is not as widespread as blast furnaces, but has industrial importance. In 2014, 74.6 million tons of sponge iron were produced in the world. Today, MIDREX and HYL / Energiron processes, that use gas reducers, are the most common sponge iron production methods. The SL / RN process is the most common of all coal-using plants [7,8].

Sponge iron is the name of the product, which is produced by reduction of granulated, lump or pelletized iron ore with using gas or solid reducing agents under its melting temperature (950 °C - 1100 °C). The obtained product contains high metallic iron as well as gangue components from ore, unreduced iron oxides and some carbon. The sponge iron has the following properties;

- total iron content generally over 85%,
- the degree of metallization varies between 90% and 95%,
- carbon content is between 1 and 2.5%,
- gang content varies between 2-4%,
- the sulfur content is less than 0.005% for sulfur-free gas processing and 0.02% for processes using sulfur-containing coal and limestone,
- apparent density ≤ 4 g/cm³.

To allow the sponge iron to be stored under ambient conditions and to be easily charged to the arc furnace, HBI (Hot Briquetted Iron) is produced by compressing pellet and lump of sponge iron under high pressure at a temperature of more than 650 °C [9].

The fact that most of the production in our country is conducted by electric arc furnace plants which use high amount of scrap as raw material and the scrap amount in our country is much lower than the usage, makes the country dependent on foreign supplies. The Turkish iron and steel industry supplies 30% of the scrap used as raw material in the arc furnaces from domestic sources and 70% from imports. Also 40% of the iron ore, which is the raw material needed by the integrated facilities, is supplied by domestic and 60% by import. In 2013, 19.7 million tons of iron and steel scrap were imported. According to ITC-Trade Map statistics; Turkey, which is the world's largest iron and steel scrap importer in 2010, achieves 16% of world scrap imports [8].

In order to eliminate the dependence on external scrap, examination of alternative technologies which produces steel from iron ore is required. Some other factors in the emergence of alternative iron and steel technologies are:

- reduction of raw material cost,
- increasing of productivity by reducing product cost,



- enhancing of product quality,
- new product development,
- energy-saving,
- Increasing of recycling,
- solution of environmental problems.

The basis of alternative iron steel production is the production of sponge iron from iron ore by solid pre-reduction, which consists of low-temperature reaction at the top of the blast furnace [9].

Iron ore pellets are frequently used as raw material in sponge iron production. Iron ore pellets are industrial charging materials obtained as fine-grained minerals rich in iron by agglomeration and induration. The iron pellet has higher cost than other charge elements (lump iron ore and sinter). Despite this disadvantage, it is one of the indispensable inputs of the blast furnace thanks to its features. The reasons why the pellets are near ideal for the blast furnace can be summarized as follows:

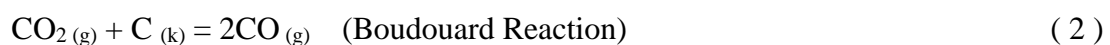
- high iron content (65-67% Fe),
- superior resistance to crumbling and dusting due to its high strength and transport properties,
- good gas permeability due to their close size and uniform gas distribution,
- good reducibility properties due to high porosity.

Providing the desired characteristics ensures that positive results such as the use of high-temperature pellets, reduction of coke consumption, reduction of the amount of slag, increase of production speed are achieved [9].

Thermodynamic Investigation of Reduction Reaction

The carbothermic reduction of iron oxides to metallic iron, is accomplished by removal of the oxygen contained in the raw materials by deterioration of the iron-oxygen-carbon thermodynamic equilibrium to the reduction condition. In this condition, iron oxides reduce to metallic iron by the result of series reactions [4-6].

The reduction of the iron oxide in blast furnace is carried out by reductive CO gas, which is obtained by re-reacting of CO₂ gas with carbon, due to the fact that CO₂ gas, produced by combustion of carbon, is unstable at high temperatures (see Ellingham Diagram).





The reduction of hematite to iron with CO/CO₂ gas mixture, takes place in three stages. The temperature-dependent empirical expressions (3), (4), and (5) of the standard free energy change (ΔG°_T) for these reactions and reactions are given below.



In order to determine the reduction conditions of iron oxides, firstly the equilibrium conditions must be determined with the help of the following relations.

$$\Delta G_T = 0 \text{ (Equilibrium Condition)} \quad (6)$$

$$\Delta G^{\circ}_T = -RT \ln K_p \quad (7)$$

$$K_p = \frac{a^2_{\text{Fe}_3\text{O}_4} \cdot P_{\text{CO}_2}}{a^3_{\text{Fe}_2\text{O}_3} \cdot P_{\text{CO}}} \quad (8)$$

When Fe₃O₄ and Fe₂O₃ are assumed to be pure, their activities are equal to "1". In this case;

$$\Delta G^{\circ}_T = -RT \ln \frac{P_{\text{CO}_2}}{P_{\text{CO}}} = RT \ln \frac{P_{\text{CO}}}{P_{\text{CO}_2}} \quad (9)$$

From this, the $\left(\frac{P_{\text{CO}}}{P_{\text{CO}_2}}\right)_D$ ratio which balances the reduction reactions at the T₁ temperature can be calculated. The basic thermodynamic condition for the reduction of iron oxides is as follows for each reaction.

$$\Delta G_T < 0 \quad (10)$$

$$\left(\frac{P_{\text{CO}}}{P_{\text{CO}_2}}\right)_{\text{Ortam}} > \left(\frac{P_{\text{CO}}}{P_{\text{CO}_2}}\right)_{\text{Denge}} \quad (11)$$

The Baur-Glaessner diagram and Boudouard curves drawn using reactions (2) and (9) are shown in Figure 1.



EXPERIMENT 6: CARBOTHERMIC REDUCTION

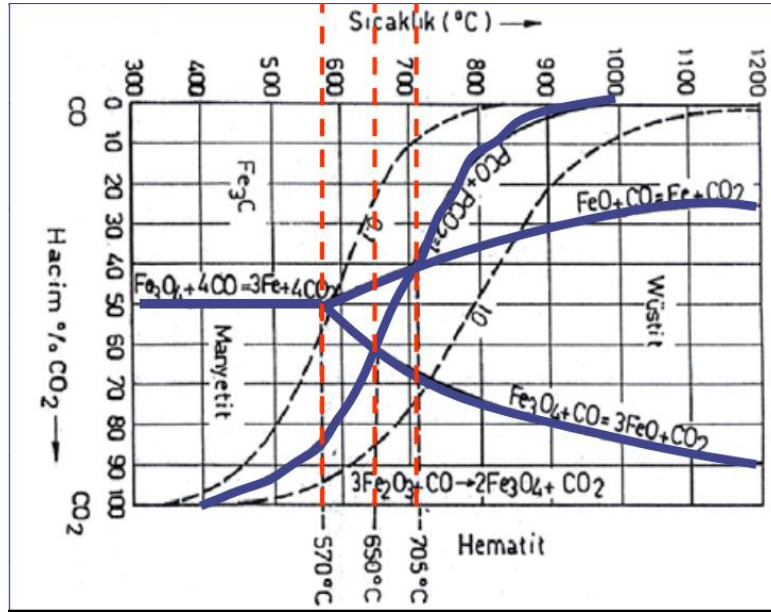


Figure 6.1 Bauer-Glaessner diagram and Boudouard curves

According to Figure 1, the metallic iron reduction of iron oxides at a total pressure of $P_{CO} + P_{CO_2} = 1$ atm is only possible above 705 °C, under 705 °C, hematite to magnetite and magnetite to wüstite transformation is possible. It can be seen from Figure 1 that a magnetite-wüstite transformation occurs between 650 and 705 °C and a hematite-magnetite transformation occurs below 650 °C. It can be seen that as the total pressure of $P_{CO} + P_{CO_2}$ increases, the reduction temperature of the iron oxides is forced to shift to higher temperatures [4-6].

Kinetic Analysis of Reduction Reaction

In a reaction, the reaction rate can be determined by the time-dependent change in the quantitative properties of a substance participating in the reaction. Accordingly, the reaction rate can be given by;

$$r = -\frac{dC}{dt} \quad (12)$$

The reaction rate can be calculated from the slope of the change in concentration plotted against time. The most important parameters affecting the rate of a homogeneous reaction are temperature and concentration.

k = reaction rate constant (temperature dependent)

$$r = -\frac{dC}{dt} = kC^n \quad (13)$$

n = reaction ratio



EXPERIMENT 6: CARBOTHERMIC REDUCTION

One of the most important parameters affecting the rate of chemical reactions is temperature. The equation for obtaining the relation between temperature and velocity constant in homogenous reactions is Arrhenius equation.

$$k=A.e^{-EA/RT} \quad (14)$$

In this equation, E_A is the activation energy. The rate constants k must be determined for at least two temperatures to determine the activation energy. A is the frequency (frequency) factor and R is gas constant ($R= 8.314 \text{ J/mol K}$). As seen from the Arrhenius equation, the reaction rate depends on the temperature as an exponential function. A small change in temperature increases the rate constant drastically. Taking the logarithm of both sides, the following relation equation is obtained.

$$\ln k = \ln A - \frac{E_A}{RT} \quad (15)$$

To calculate the activation energy, when the graph of $\ln k - 1/T$ is plotted, the angle of the line obtained is equal to the slope ($-E_A / R$) (Figure 2).

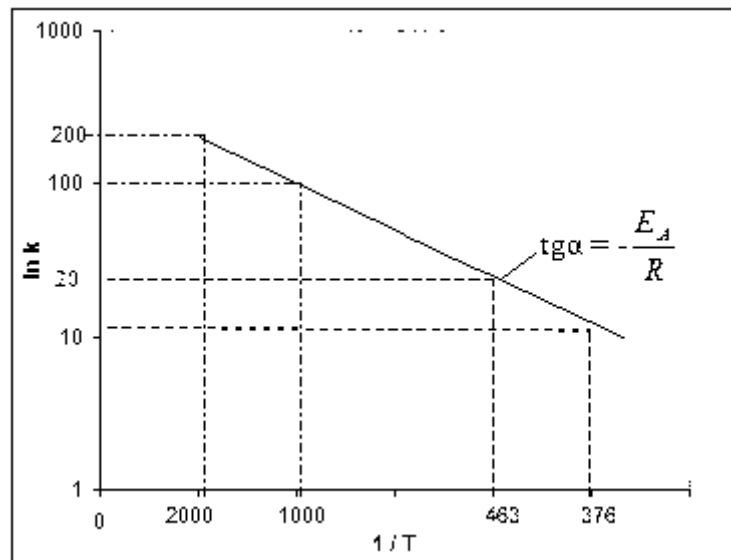


Figure 6.2 Change of reaction rate with temperature

Reactions are termed as homogeneous and heterogeneous reactions, respectively, according to their occurrence between one or more phases. The reduction of iron oxide raw materials is a heterogeneous reaction. Heterogeneous reactions occur between more than one phase and are characterized by the presence of an interface between the reactants. For example, in the case of a solid-gas reaction, the interface layer is the outer surface of solid, which is in contact with the gas. In this case, we can gather all heterogeneous reactions in five groups depending on their interface: Solid-Gas, Solid-Liquid, Solid-Solid, Gas-Liquid, Liquid-Liquid [10-13].



The occurrence of heterogeneous reactions between more than one phase is as follows [10,11]:

1. Transport of the reducing gas to the reaction interface (diffusion)
2. Interfacial reactions.
 - a) Adsorption of the reducing gas to the reaction interface.
 - b) Chemical reaction at the interface.
 - c) Desorption of the products occurred after the reaction.
3. Interfacial transport of reaction products resulting from reaction (diffusion)

Generally, the rate constant of one of the steps listed above is very low compared to the others. In this case, this step is the step controlling the rate, and the rate of this step determines the total reaction rate. Heterogeneous reactions develop, depending on the slowest step:

- **Diffusion Controlled**
- **Chemical Reaction Controlled**

If the rate constants of these two steps are equal;

- **Mixed Controlled**

The interfacial area in heterogeneous reactions is of great importance as the amount of material carried in transferring the reactants from one phase to the other depends on the interface area. In reactions with solids, small grained solids react faster than large grains due to their larger surface area [10-13].

The geometric shape of the solid material entering the reaction with liquid or gas plays an important role in determining the rate of the relevant reactions. If the solid is disc or plate-shaped, it is assumed that the surface area remains constant throughout the reaction. Because the reaction on the disc and the plate proceeds from surface towards the interior by protecting the surface area of the surface. However, if the solid is spherical or pellet-shaped, the reaction proceeds inward from the outer surface of the sphere. In this case, as the diameter of the reaction surface is reduced, the surface area changes continuously throughout the reaction and therefore the reaction rate decreases. Since the solids reacting is generally regarded as a sphere, the relationship between the rate constant for the chemical reaction in the heterogeneous reaction and the realization rate of the reaction is given as:

$$1 - (1 - R)^{1/3} = kt \quad (16)$$

R = Realization rate of reaction)can be indicated by letters n, x, F, L)

W_o = Initial weight

$$R = \frac{W - W_o}{W_o} \quad (17)$$

W = Final weight

EXPERIMENT 6: CARBOTHERMIC REDUCTION

The reaction interface of spherical solid materials is quite evident. As the reaction progresses, the reaction interface travels from the outer surface to the center of the particle, and there is a definite boundary between the unreacted area and the reaction surface. This model reaction is called the topochemical model or the shrinking core (Figure 3).

If the reaction products formed are dissolved in the environmental phase, the surface area will decrease over time. Typical examples of this are combustion of coke or dissolution of a solid in a liquid. In this reaction model, the step controlling the reaction rate will be chemical reaction or diffusion.

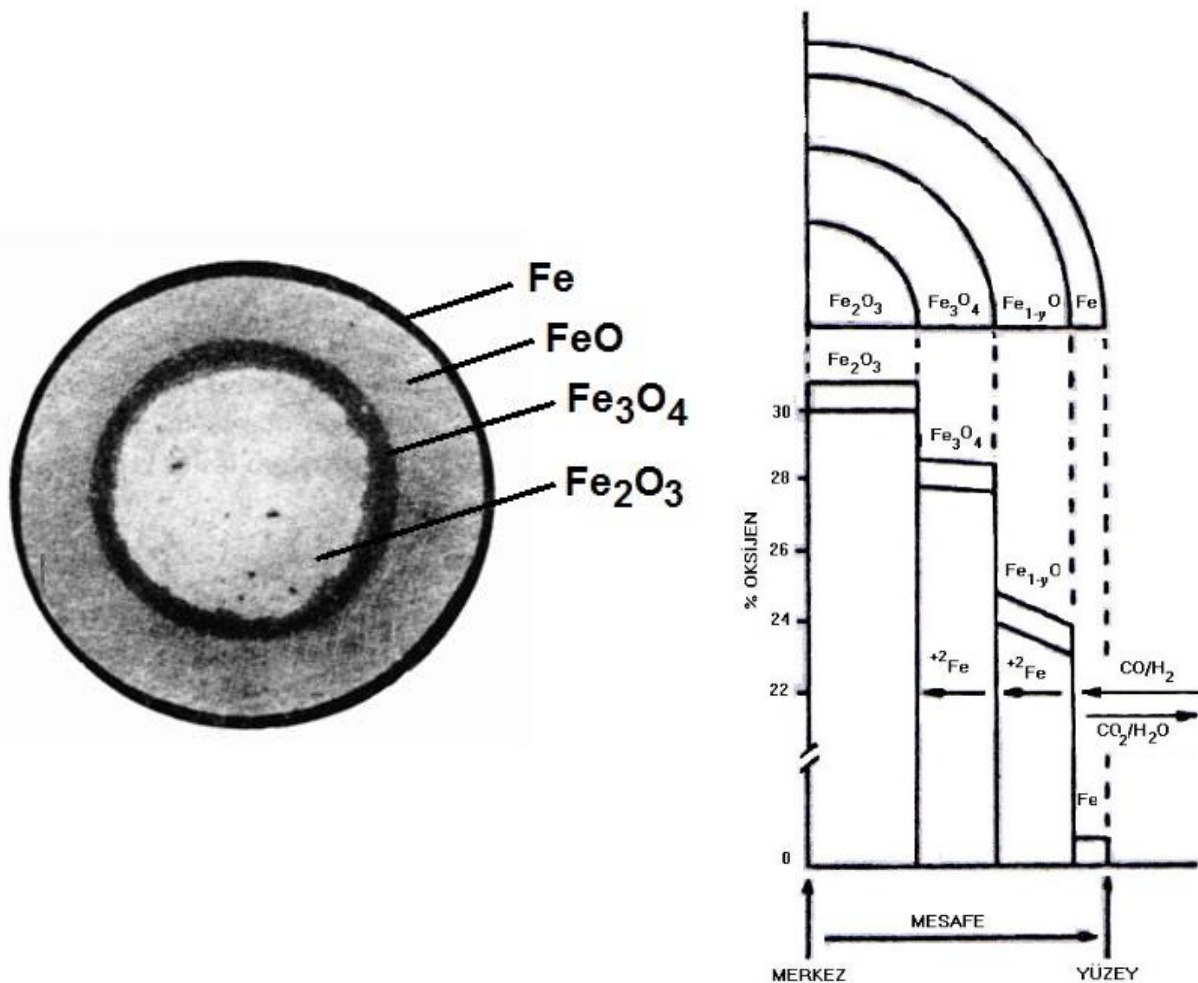


Figure 6.3 Schematic representation of the reduction of the hematite particle and the topochemicality in reduction [5, 6, 8].

If the reaction products form a layer between the original solids and the gases which react with the gases, such as the oxidation of the metals or the metal sulfides or the reduction of the oxides with gases, the molecules must be diffused from this layer so that the reaction can proceed. Due to the porous or non-porous formation of the reaction product formed by the reactions occurring in the solid state, the kinetics of the reaction will also be different [10-13].



6.3. EQUIPMENT AND MATERIALS

- 1) Iron ore pellets and coal (coke or lignite dust)
- 2) Tube furnace
- 3) Precise scale
- 4) Graphite crucible
- 5) Pens and tongs

6.4 EXPERIMENTAL PROCEDURE

- 1) In order to observe the reduction behavior, identical pellets are selected from the pellets produced from the obtained from iron concentrate and weighed to determine their weights.
- 2) Using the total reduction reaction, the amount of carbon required for the reduction of these pellets and the amount of coal to be used are calculated accordingly. 150% of the theoretical amount of coal calculated on the basis of total pellet weight is weighed and used.
- 3) When the tube furnace reaches a temperature of 800 and 1000 ° C, the pellets are buried in the coke bed prepared in the graphite crucible and charged into the furnace with the charging rod.
- 4) Pellets are taken from the furnace on the 5th, 10th and 20th minutes respectively, and be cooled to room temperature and weighed after reduction.

The chemical composition of the pellet and coke used in the experiments are given below.

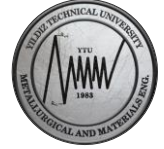
	Fe ₃ O ₄	SiO ₂	Al ₂ O ₃	S	Mn	CaO	MgO	K ₂ O	Na ₂ O	P	C
Pellet	94,60	2,20	0,75	0,40	0,10	0,60	0,58	0,07	0,04	-	-
Coke	-	8,26	4,30	0,54	-	1,24	0,35	0,29	0,08	0,14	80,30

Reduction ratios of pellets are calculated using the formula below:

$$\text{Reduction Ratio (\%)} = \frac{\text{Removed Oxygen (wt.)}}{\text{Removable Oxygen (\%)}} \quad (18)$$

6.5 ASSIGNMENTS AND REQUESTS

- 1) Write down the aim and procedure of the experiment.
- 2) Calculate the required amount of theoretical coal.
- 3) Draw the variation of the reduction ratio with time and evaluate the graph.
- 4) Calculate the instantaneous reduction rate (dR / dt) at 5th, 10th and 20th minutes.

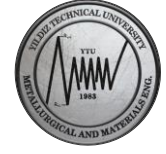


EXPERIMENT 6: CARBOTHERMIC REDUCTION

- 5) For the reduction of ferrous raw materials, under what conditions is the reduction kinetics controlled by chemical reaction, diffusion or mixed controlled? How do you see this difference in the partially reduced iron oxide particle? Schematically draw.
- 6) Using the reactions (4) and (5), plot the % CO - temperature graphs for 700, 800, 900 and 1000 °C temperatures in the total $P_{CO} + P_{CO_2} = 1$ atm environment and name the zones. (Bauer-Glaesner diagram plotted against % CO amount).

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**EXPERIMENT 7: GLASS PRODUCTION AND OPTICAL
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7. GLASS PRODUCTION AND OPTICAL PROPERTIES OF MATERIALS

PART 7.1: GLASS PRODUCTION

7.1.1 OBJECTIVE OF THE EXPERIMENT

The objectives of this experiment are to prepare the glass batch, synthesize the glass by the melt-quenching method, and shape the glass by fiber drawing and mold casting methods.

7.1.2 THEORETICAL INFORMATION

Glasses are any of the highly viscous amorphous materials formed from a melt by cooling to rigidity without crystallization. Their main characteristics are transparency, hardness, fragility, and chemical stability [3, 4]. Contrary to crystalline materials, the glasses do not have regularly repeating long-range periodic order and they possess short to medium range order (Figure 1) [2-5].

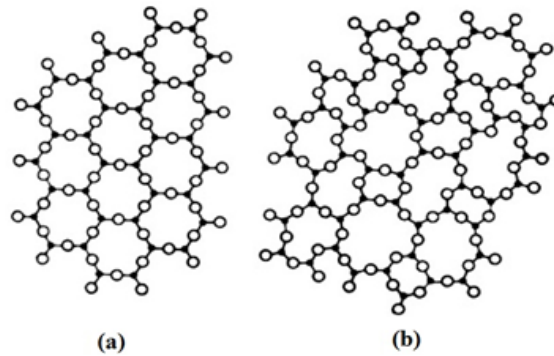


Figure 7.1 Atomic arrangement of a) a crystalline, b) an amorphous structure.

When heated to an appropriate temperature, many inorganic elements and compounds form liquids having viscosities close to water (10^{-4} - 10^{-3} Pa·s) [6]. Those liquids solidify rapidly when cooled to their solidification points. If the crystallization speed is slow enough, it may be possible to cool the solution below the solidification point without crystallization. This material is named as glass. The mechanical properties of this material are similar to those of an elastic solid material. In order to form vitreous structure, it must be cooled rapidly below the melting temperature to prevent the crystallization of the supercooled liquid. The crystallization rate is the factor controlling the glass formation. The mechanical properties of the glass resemble an elastic solid and its structural properties are like a viscous liquid [2, 7].

When a liquid is cooled, space for the atoms to move around decreases. On further cooling below the glass transition temperature, the atoms can no longer move around with respect to each other so the material becomes a solid. A measure of this is the specific volume, which can be defined as the difference between the density of the crystal and of the liquid. When a glass-forming material cools, the excess volume decreases and finally the density of the glass approaches that of the crystal, as illustrated in Figure 2. In practice, the formation of an amorphous or crystalline solid depends on how rapidly the liquid is cooled through the glass transition temperature. Upon cooling the liquid, if there is a discontinuity in volume change or



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in rate of cooling, the liquid crystallizes. However, if the liquid passes into a supercooled state, the volume decreases and no crystallization occurs [3, 4, 8].

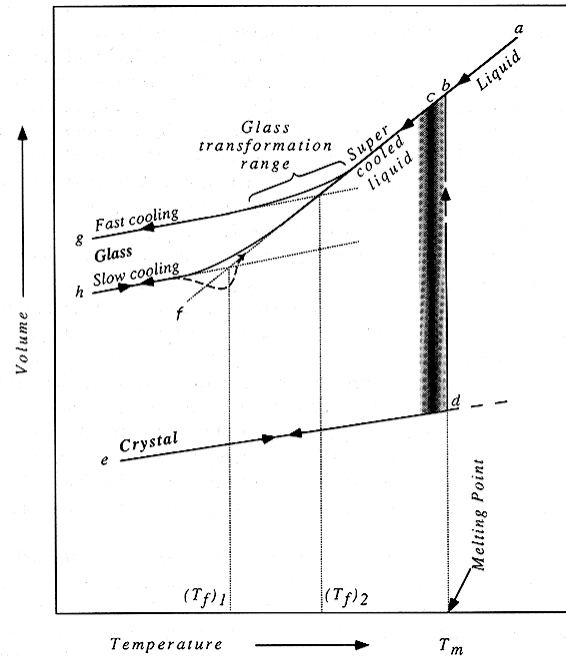


Figure 7.2 Specific volume – temperature change of crystalline and amorphous solids.

There is a decrease in the volume of glass held at a temperature below the glass transition temperature. This region where the free molecule movement in the glass stops and the glass stabilization takes place is called the glass transition region. Along the glass transition region where the glass is trying to become more stable, the properties of the glasses depend on the cooling rate in a certain area. The glass transition temperature is proportional to the cooling rate; it will be low at low cooling rates [6, 9].

In general, glasses are either produced from high quality, chemically pure components or from a mixture of far less pure minerals. Research specimens, optical glasses, and many glasses used for low volume, high technology applications are produced using those chemicals we might routinely encounter in any chemical laboratory. Bulk commercial products are produced from minerals.

Process of glass manufacturing includes six basic steps: raw materials selection, batch preparation (calculation of the relative proportions, weighing and mixing of raw materials), melting and refining (removal of any unmelted batch remnants, impurities, and bubbles), conditioning, forming, post-processing (heat treatments to remove stresses, thermal tempering).

Sugar glass

Common table sugar is called sucrose. The sucrose molecule is essentially a glucose molecule joined to a fructose molecule. The glucose molecule, which makes up the corn (glucose) syrup, bonds well with the sucrose in the solution seeing as they are similar molecules. The extra



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glucose molecules actually inhibit crystallization of the sugar upon cooling because the added components make it harder for individual sugars to form crystals. On the other hand, water reduces the melting temperature of table sugar from 186 °C to ~130 °C, allows to obtain a melt at lower temperatures.

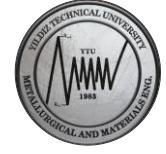
The temperature at which a given sugar solution can be cooled to form a glass at a room temperature varies on the percent composition of each material. For hundreds of years, candy makers have gone by a system of rating the candy on its hardness by dropping a spoonful into room temperature water. The candy can vary from soft-ball to hard-ball to hard-crack. It is simply based on what the candy feels like to touch. When the candy snaps or cracks when dropped into water, it is at hard crack, and that is the point where it will become a glass if cooled at room temperature. That is considered the pouring temperature.

7.1.3 EQUIPMENT AND MATERIALS

- Table sugar
- Corn (glucose) syrup
- Water
- Beakers with different volumes
- 5 ml syringe
- Spatulas in different sizes
- Analytical balance
- Heating plate
- Metal pot
- Silicone mold

7.1.4 EXPERIMENTAL PROCEDURE

1. In order to prepare a 50 g batch, 60% sugar, 30% corn syrup and 10% water in wt. are weighed with an analytical balance.
2. The weighed components are placed in a metal pot and the metal pot is placed on the heating plate.
3. The mixture is mixed at increasing temperature with a metal spatula to form a solution.
4. When a homogeneous melt is obtained, first a drop of the solution is dropped into water at room temperature to check whether it is ready for pouring.
5. The sugar melt is dropped onto the bench and with the help of a spatula, fibers are drawn from the molten sugar. The change in the elongation of the fibers is observed with dropping temperature.
6. Glass is casted by pouring the solution into a room temperature silicon mold. The glass is removed from the mold when it cools down to room temperature.
7. The properties of the obtained glass are examined physically.



**EXPERIMENT 7: GLASS PRODUCTION AND OPTICAL
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7.1.5. QUESTIONS

1. Calculate the required raw material (SiO_2 , Na_2CO_3 and CaCO_3) amount to synthesize a 50 g soda-lime silicate glass batch with the composition of 75 SiO_2 -15 Na_2O -10 CaO mol%.
2. Write the roles of the below given commercial glass components.
 SiO_2 , Na_2O , Li_2O , K_2O , CaO , MgO , Al_2O_3 , B_2O_3 , ZnO , Sb_2O_3 , SnO
3. Explain the crystalline and amorphous structure depending on cooling rate.

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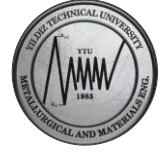
PART 7.2: INVESTIGATION OF OPTICAL PROPERTIES OF MATERIALS

7.2.1. OBJECTIVE

The objective of this experiment is to measure the transmittance of ultraviolet and visible radiation of a set of optical materials and evaluate differences between their transmittance behaviors.

7.2.2. THEORETICAL BACKGROUND:

Light is electromagnetic radiant energy. Depending upon the point of view, light can be thought of as having either wave properties or particle properties. According to the wave theory, electromagnetic radiation is composed of both electric and magnetic fields whose waves vibrate in mutually perpendicular planes. It is the electric field of light interacting with the electrons of



matter which produces observable properties of a substance. Spectroscopy is the study of objects based on the spectrum of color they emit or reflect as a result of that interaction.

Two fundamental characteristics are associated with any wave – the wavelength and the frequency. The wavelength, λ (Greek letter, lambda), is measured from the crest of a wave to the adjacent crest (see Fig. 1). Wavelengths range from very small (measured in nanometers, 10^{-9} m) to quite large, several meters. The frequency is the number of waves passing a point in unit time and is designated ν (Greek letter, nu). Frequency can be expressed in cycles (or vibrations) per second. Wavelength and frequency are inversely proportional to each other; the shorter the wavelength, the more waves pass a point in a given time and thus the higher the frequency. Similarly, the longer the wavelength, the lower the frequency.

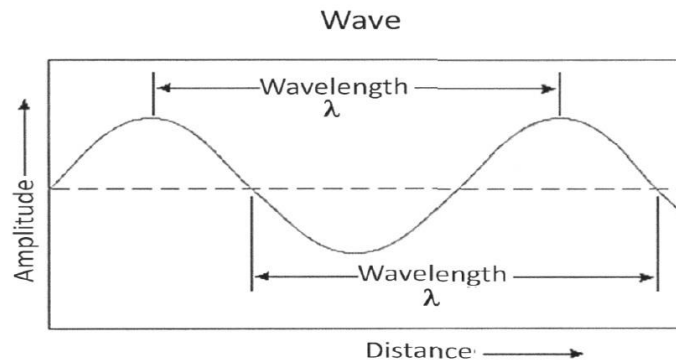


Figure 7.3 The description of wavelength

Wavelength and frequency are related by the speed of light ($c = 2.998 \times 10^{10}$ cm/s):

$$c = \lambda \cdot \nu$$

Another important relationship is that between these quantities and the energy of light (E):

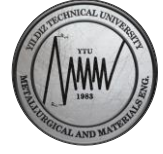
$$E = hc / \lambda$$

Where: h is Planck's constant (6.626×10^{-34} Joule·sec)

The greater the energy of the radiation, the shorter its wavelength and the higher its frequency; the smaller the energy of radiation, the longer its wavelength and the lower its frequency.

Light from the sun is composed of a continuum of energies and thus, of a continuum of wavelengths and frequencies. This continuum of wavelengths is called a spectrum. Fig. 2 shows the entire wavelength range of the electromagnetic spectrum arbitrarily divided into regions called bands and the portion occupied by visible light.

When light impinges on a substance, one or more combination of things can happen to the light. The light can be absorbed, transmitted, reflected, diffracted, or scattered by the substance. The absorbed light energy causes such changes as atomic and molecular rotation, vibrations, and electron transitions to higher energy levels. As a result of this absorption, our eyes or specially designed instruments may sense phenomena such as heat, fluorescence, phosphorescence, or color. Modern instrumentation can record these phenomena to a very high degree of precision.



EXPERIMENT 7: GLASS PRODUCTION AND OPTICAL PROPERTIES OF MATERIALS

Most of the electromagnetic radiation continuum is invisible to humans; the portion we perceive is referred to as visible light. Wavelengths of visible light extend from about 800 nm (8.0×10^{-5} cm) to about 400 nm (4.0×10^{-5} cm).

It is against the high energy Ultraviolet (UV) radiation that sun screen lotions are formulated to protect our skin in the outdoors. Our skin perceives the low energy infrared (IR) radiation only as heat. The lamps placed over food in cafeterias emit most of their energy in the red part of the Infrared region, keeping the food warm.

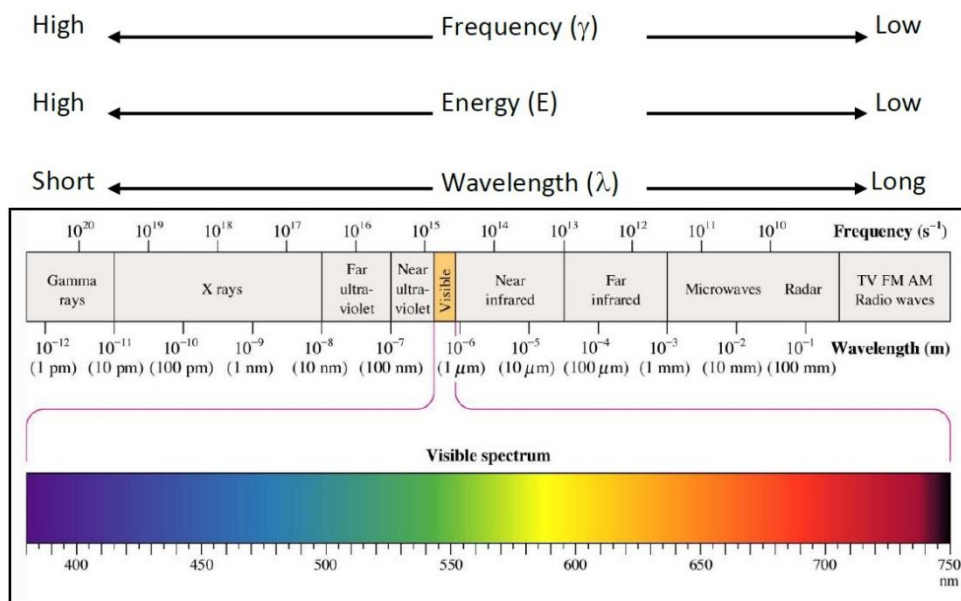


Figure 7.4 Electromagnetic Spectrum

If a substance absorbs all wavelengths in the visible range, none of the light is reflected back to our eyes and the substance appears black. As opposed to that if all of the visible light striking our eyes is reflected prior to striking the eye, the color appears white. If the substance transmits all of the incident visible light, it appears colorless (transparent). Colorless substances usually absorb in the UV or IR regions of the spectrum, on either side of the visible range.

If a substance absorbs light principally in one wavelength range (generally, a number of wavelengths on both sides of the principal absorption are also absorbed, so a broad absorption band results), the color perceived will be a mixture of all the wavelengths which are not absorbed. For example, the indigo dye in blue jeans has its maximum absorbance in the 500 – 650 nm range. Because this absorbance is in the red-to-green region, the wavelengths which are not absorbed are in the 400-500 nm range, thus, the color observed would be blue-violet.

Spectroscopy is a basic analytical technique and research tool that utilizes the interaction between matter and electromagnetic energy. A spectrometer is an instrument that separates electromagnetic radiation according into wavelengths, passes these separated wavelength bands through a sample, and detects the intensity of the transmitted light. In analyzing a new sample,



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a scientist first determines the sample's absorbance spectrum. The absorbance spectrum is a plot of absorbance vs. wavelength and shows how the absorbance of light depends upon the wavelength of the light. The absorption spectrum is characterized by the wavelength of maximum absorption (λ_{\max}) at which the absorbance is the greatest (see Fig. 3 below). It is characteristic of each unique compound providing information on the electronic structure of the compound.

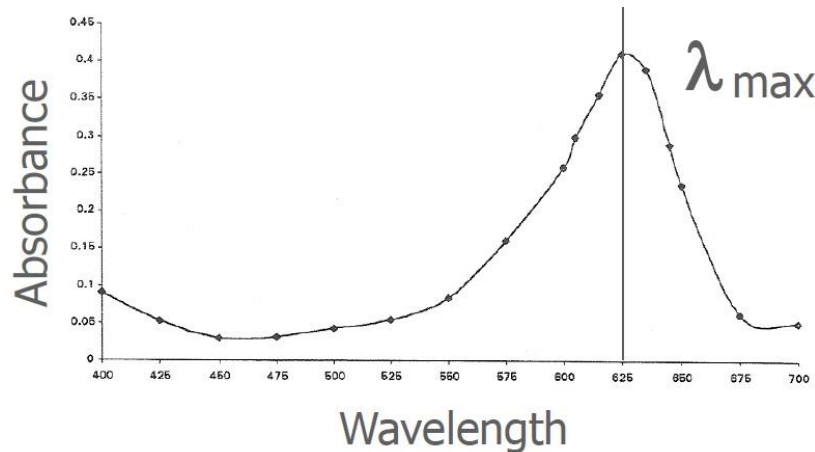


Figure 7.5 The example of an absorption spectrum

All spectrometers have the following fundamental parts: a source or radiant energy, a prism or grating to isolate radiant energy to narrow wavelength regions, a device for holding the sample, and a detector for measuring light intensity. Sophisticated instruments include automatic recorders, digital readouts, computer interfaces, and arrays of detectors that allow the user to analyze a wider range of wavelengths.

The Beer-Lambert Law:

The Beer-Lambert law defines the mathematics relating the absorption of light to the properties of the material through which the light is traveling. The derivation of the equations used with the law involves two principle variables, transmissivity (T) (or percent transmittance (%T) and absorbance (A).

The amount of light transmitted as it is passing through the substance. It is defined as the ratio of the light intensity leaving the sample (I) to the intensity entering the sample (I_0):

$$T = \frac{I}{I_0}$$

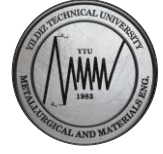
Spectrometers measure the percent transmittance (%T) of light passing through the sample.

$$\%T = \left(\frac{I}{I_0} \right) \cdot 100$$



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Absorbance is the amount of light absorbed expressed in logarithmic terms. It is defined as the negative logarithm (base 10) of the Transmittance.

$$A = -\log_{10} T = -\log_{10} \left(\frac{I}{I_0} \right)$$

The law states that there is a logarithmic dependence between the transmissivity and adsorption of the light passing through the sample.

Experimental Study:

In this experiment, the permeability properties of a standard window glass, sunglass lens and a special purpose glass containing cobalt will be examined. For this, percentage transmittance (% T) behaviors in visible and UV region will be determined by using UV-Visible spectrophotometer. The standard window glass appears completely transparent, while the sunglass lens appears black and the cobalt glass appears blue. It will be evaluated whether the results obtained are consistent with these differences in the appearance of the glasses. At the same time, it will be evaluated whether the permeability properties of glasses are suitable for their intended use.

7.2.3 MATERIALS AND EQUIPMENT:

- UV–Visible spectrophotometer
- Standard window glass
- Sunglass lens
- Cobalt glass

7.2.4 PROCEDURE

1. Turn on spectrophotometer from power button (at the back of the device). It will take approximately ten to fifteen minutes for the instrument to warm up.
2. Run “**UVWin Software**” from desktop and press “**OK**”.
3. From left frame select “**Spectrum**”.
4. From menu select “**Measure/Parameters Settings**” to enter measurement parameters.
5. From “**Measure**” tab select Absorbance (%Abs) or Transmittance (%T) mode to measure, from “**Scan Parameters**” tab adjust the starting and ending wavelength.
6. From “**Instrument**” tab select the lamps which will be used, turn off the D2 lamp if not necessary (if UV region will not be covered).
7. Press “**OK**” to save measurement settings.
8. Press “**Baseline**” to run the measurement without any sample to perform baseline correction. The screen will display 100 %T or 0 %Abs if correction is completed successfully.
9. Place the glass sample into the sample holder.
10. Replace the black cover and instrument chamber cover and press “**Start**”.
11. When scanning has finished, press “**Yes**” to save scanning results.



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7.2.5 QUESTIONS

1. Which kind of ultraviolet radiation contains the most energy? What is the range of wavelengths of this radiation?
2. Which form of ultraviolet radiation is most damaging to your eyes? What is the range of wavelengths for this radiation?
3. Which type of radiation should sunglasses block?
4. Describe the difference between absorbance and transmittance.
5. An absorbance spectrum shows a peak at 540 nm. What does this mean?
6. A transmittance spectrum shows a peak to 80% between 480 nm and 524 nm. What does this mean?

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8. REDUCTION ELECTROLYSIS

8.1 OBJECTIVE OF THE EXPERIMENT

The objectives of present experiment are to get information about kinds of electrometallurgy, carry out the electrolysis of copper sulphate solution and to link their findings with the industrial electrolytic refining of copper. The results of this experiment can lead to a discussion about electroplating and the electrolytic refining of copper.

8.2 THEORETICAL INFORMATION

8.2.1 WHAT IS ELECTROMETALLURGY?

Electrometallurgy is the recovery and purification of metals through electrolytic processes by using electrical energy.

Electrometallurgy makes use of electrothermal and electrochemical processes. Electrothermal processes are used to extract metals from ores and concentrates and to produce and refine ferrous and nonferrous metals and alloys based on the metals extracted. In such processes, electric energy is used as a source of heat.

Electrometallurgy deals with technical aspect of metal electrodeposition. The electrometallurgical processes can be categorized into four main groups: Electrowinning, Electrorefining, Electroplating, Electroforming. The schematic representation of electrometallurgical processes is shown in Fig. 1

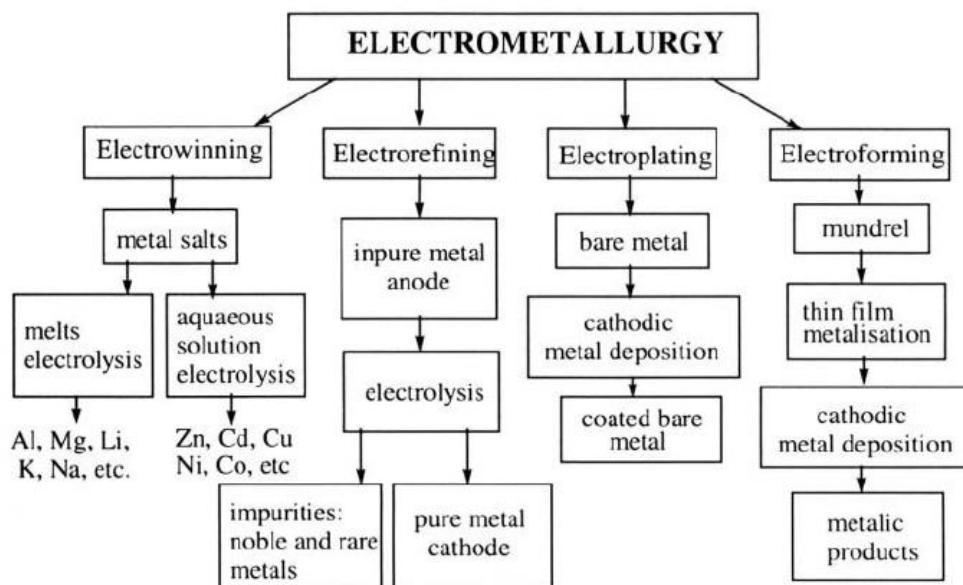


Figure 1. Schematic illustration of electrometallurgical processes.

Electrowinning is the extraction of metals by electrodeposition from aqueous solution or melts of their salts. On a large scale electrodeposition from molten salts is used for extraction of electronegative elements which cannot be electrodeposited from aqueous solutions, such as aluminum and magnesium, as well as very pure copper, zinc and cadmium by electrodeposition from an aqueous solutions of the metal salts.



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Electrorefining is the purification of metals by electrolysis. The impure metal is dissolved anodically and pure metal is deposited cathodically, while the impurities being left as anode sludge or as ions in the solution. Many metals are electrorefined such as copper because of conducting application and precious metals because of their cost. Electrorefining is also a part of processes in recycling of metals. It should be noted that large electrolytic plants for metal production are heavy consumers of electric energy¹. In the metal electrorefining and electrowinning the main requirements are to produce pure and compact deposits. This is done at lower current densities. From qualitatively the same, but less concentrated solutions at higher current densities metal deposits in form of powder are obtained. Powder electrodeposition can also be treated as kind of electrowinning or electrorefining, which produces the metal deposits in forms suitable for sintering and various different applications.

Electroplating can be defined as a treatment that modifies the surface of a metal or occasionally a nonmetal, without changing its bulk properties, in order to improve the appearance of a surface, to increase the corrosion and abrasion resistivity, etc. The improving the appearance was the aim of electroplating earlier, now it is mainly the change of surface properties from those of substrate material to those of electroplated metal. Obviously, the coating can successfully change the surface properties of substrate only if it is compact and nonporous, as well as good adherent. Metal objects we meet in everyday life are often electroplated, but it seems that the most important application of electroplating technology is the manufacture of electronic components (circuit breakers and contacts). Electroplating can be performed from molten salts and aqueous and non-aqueous solutions, depending on the nature of electrodeposited metal, but most frequently from aqueous solutions.

Electroforming is the manufacture of articles by electrodeposition. If deposit is good from electroplating point of view except adhesion, and can be removed from the cathode as an entity in itself, it has been electroformed. Electroforming is a branch of electroplating technology, but involve some additional steps, as for example the production, preparation and extraction of the master.

Electroless metal deposition and anodic oxidation of metals can also be include in the field of electrometallurgy.

Empirically is known what type of deposit can be obtained under specific conditions, however how and why this can be achieved still remains a mystery in some cases.

8.2.2 BASIC FACTS

8.2.2.1 Electrodes and electrochemical reactions, cell and circuit

In a metallic conductor free conduction electrons transport the charge whereas in an electrolytic conductor it is ions. In order to include an electrolytic conductor in an electrochemical circuit it is necessary to make electrical contacts to and from the electrolyte by metallic conductors. A metallic conductor immersed in an electrolyte solution is an electrode, and two electrodes connected electrolytically represent an electrochemical cell¹. The simplest electrochemical circuit is shown in Fig.2



The electrochemical circuit consists of a current source, metallic connecting wires, an electrochemical cell, ohmic resistance, current and voltage measuring instruments and a circuit breaker. In technical practice more complicated circuits are used, but in principle all of them are the same as the one shown in Fig. 2.

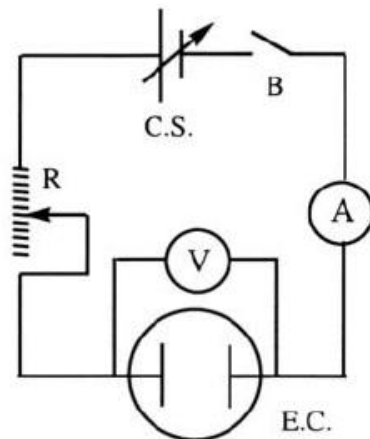


Figure 2. An electrochemical circuit. **C.S.**-current source, **R** -ohmic resistance, **V** - voltmeter, **A**-ammeter, **E.C**- electrochemical cell, **B** - circuit breaker.

The electrochemical circuit consists of a current source, metallic connecting wires, an electrochemical cell, ohmic resistance, current and voltage measuring instruments and a circuit breaker. In technical practice more complicated circuits are used, but in principle all of them are the same as the one shown in Fig. 2.

Obviously, a steady current flow in the circuit from Fig. 2. can only be maintained if there is a change of charge carrier at the metal-electrolyte interface by a chemical transformation involving the transfer of electrons across the interface, i.e., by an electrochemical reaction. It constitutes the bridge between the current of electrons in the metallic part of the electrochemical circuit and the current of ions in the electrolytic part of the circuit.

8.2.2.2 The electrochemical double layer and possible electrochemical reactions

If the metal ions in the solution are the same as in the electrode metal lattice, or if the same substance is present in the electrolyte in two oxidation states, an electron transfer reaction can occur at the metal-electrolyte interface and lead to the development of a potential difference. Such an interface behaves like an electrical circuit consisting of a resistor and a capacitor in parallel. The electron transfer takes place until a dynamic equilibrium is reached. In the case of metal electrodes, depending on the system, this process begins with either the deposition of ions from solution onto the metal electrode or with the dissolution of the metal electrode. In equilibrium the electrode is more positive than the solution in the first case and more negative in the second one. A number of electrochemical reactions are possible at such an interface, as for example



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1. The reduction of metal cations to the metal and vice versa



2. The reduction of hydrogen cations to gaseous hydrogen and vice versa



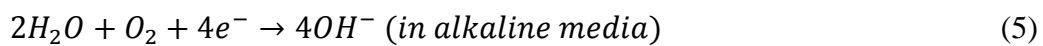
3. The decrease of the oxidation state of the cations and vice versa



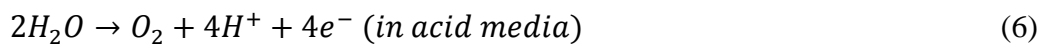
4. The reduction of anions to metal and vice versa



5. The reduction of molecules to anions and vice versa



6. The oxidation of molecules to cations and vice versa



8.2.3 SELF DRIVING CELLS

8.2.3.1 The Nernst equation and energy producing cells

For the electrochemical reaction



where O is the oxidized state which accepts n electrons and R is the reduction state or the donor of electrons, in equilibrium, the Nernst equation is written in the form

$$E_{O/R} = E_{O/R}^{\theta} + \frac{RT}{nF} \ln \frac{a_O}{a_R} \quad (8)$$

Where $E_{O/R}$ is the equilibrium electrode potential, $E_{O/R}^{\theta}$ is the standard electrode potential and a_O and a_R are the activities of the electron acceptor and donor, respectively 1,2.

In Table 1. the standard potentials for some electrode reactions are given.

Table 1. Standard potentials of metal deposition and dissolution reactions.

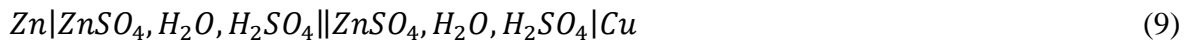
Electrode reaction	Standard potentials, V	Electrode reaction	Standard potentials, V
$Li^{+} + e^{-} = Li$	-3.045	$Co^{2+} + 2e^{-} = Co$	-0.277
$K^{+} + e^{-} = K$	-2.925	$Ni^{2+} + 2e^{-} = Ni$	-0.250
$Na^{+} + e^{-} = Na$	-2.714	$Sn^{2+} + 2e^{-} = Sn$	-0.163
$Mg^{2+} + 2e^{-} = Mg$	-2.363	$Pb^{2+} + 2e^{-} = Pb$	-0.125
$Al^{3+} + 3e^{-} = Al$	-1.662	$Cu^{2+} + 2e^{-} = Cu$	0.337
$Zn^{2+} + 2e^{-} = Zn$	-0.763	$Cu^{+} + e^{-} = Cu$	0.521
$Cr^{3+} + 3e^{-} = Cr$	-0.744	$Ag^{+} + e^{-} = Ag$	0.799
$Cr^{3+} + 3e^{-} = Cr$	-0.744	$Ag^{+} + e^{-} = Ag$	0.799
$Fe^{2+} + 2e^{-} = Fe$	-0.440	$Au^{3+} + 3e^{-} = Au$	1.498
$Cd^{2+} + 2e^{-} = Cd$	-0.403	$Au^{+} + e^{-} = Au$	1.691



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The signs of two electrodes connected to a cell can be determined by using the values of standard electrode potentials. Only, if they are close to each other the Nernst equation should be used to determine the polarity of them. The equilibrium potential difference for the cell



which is illustrated in Fig. 2.2, can be evaluated as follows.

Obviously, the equilibrium concentration of electrons in the more negative electrode will be larger than in the more positive one. According to Table 2.1, the possible reactions on the electrodes are:

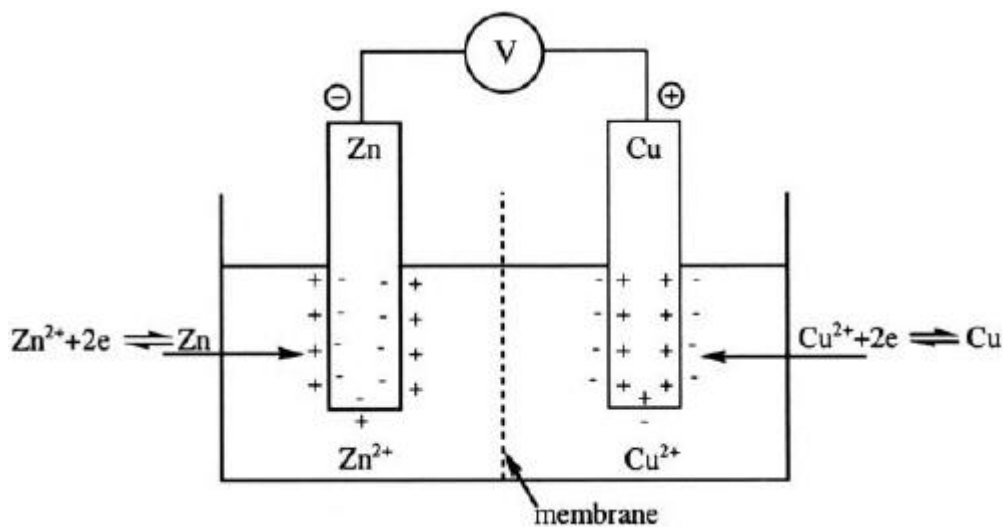
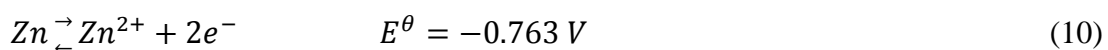
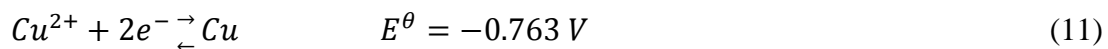


Figure 2. Schematic representation of a cell $\text{Zn}|\text{ZnSO}_4, \text{H}_2\text{O}, \text{H}_2\text{SO}_4||\text{ZnSO}_4, \text{H}_2\text{O}, \text{H}_2\text{SO}_4|\text{Cu}$ in equilibrium. V is a high impedance voltmeter.



and



The equilibrium potential difference E is given by

$$E = \left(E_{\text{Cu}^{2+}/\text{Cu}}^\theta + \frac{RT}{2F} \ln a_{\text{Cu}^{2+}/\text{Cu}} \right) - \left(E_{\text{Zn}^{2+}/\text{Zn}}^\theta + \frac{RT}{2F} \ln a_{\text{Zn}^{2+}/\text{Zn}} \right) \quad (12)$$

If the electrodes are connected as in Fig. 3, the reaction on the electrodes are:



and



The overall reaction is then:





Obviously, oxidation will take place on the more negative electrode, making it an electron sink, and reduction will occur on the more positive electrode, making it an electron source.

Such electrochemical transformations at the two interfaces provides a stream of electrons available for external use, which is the essence of energy producing cells (electrochemical power sources).

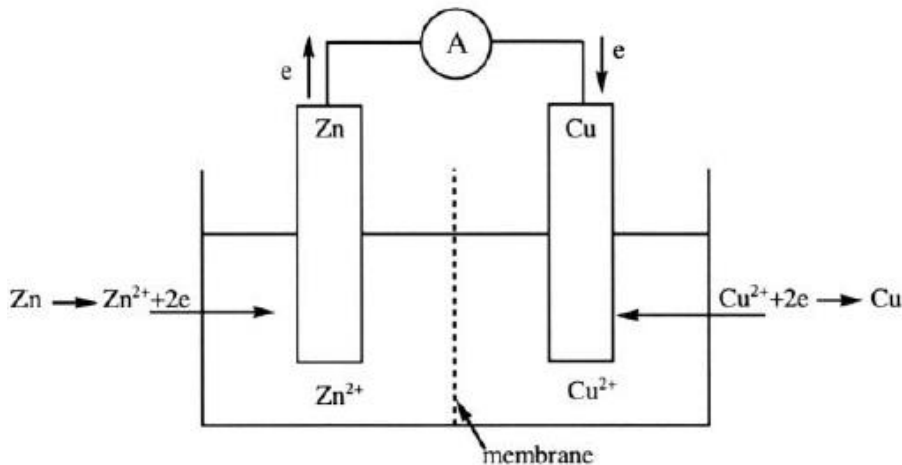


Figure 3. The cell $Zn|ZnSO_4, H_2O, H_2SO_4||ZnSO_4, H_2O, H_2SO_4|Cu$ as an energy producer. A is the low impedance ampermeter.

Reaction, given by Eq. 15 will go from left to right until the potentials of the electrodes become equal, which corresponds to the zero cell voltage and equilibrium activities $a_{e,Zn^{2+}}$ and $a_{e,Cu^{2+}}$ of zinc and copper ions, respectively. It follows then from Eq. 12 that if $E = 0$

$$E_{Cu^{2+}/Cu}^{\theta} - E_{Zn^{2+}/Zn}^{\theta} = \frac{RT}{2F} \ln K^{\theta} \quad (16)$$

where

$$K^{\theta} = \frac{a_{e,Zn^{2+}}}{a_{e,Cu^{2+}}} \quad (17)$$

represents the equilibrium constant for reaction 15. The value of $K^{\theta} \sim 10^{37}$ means that the Cu^{2+} ions can be completely removed from the electrolytic solution by reaction 15.

8.2.3.2 Cementation

If a piece of zinc is immersed in a copper sulfate solution the reaction 13 occur at one local area of the electronic conductor and reaction 14 at another local area. Reaction 13 and 14 occur spontaneously, and the overall reaction is given by Eq. 15. The system is self-driven and produces power, but the corresponding electrochemical energy is unavailable because the two interfaces are short-circuited. This reaction is used in purification of zinc sulphate solution from more positive metallic ion impurities in zinc electrowinning process.



8.2.3 ELECTROLYSIS

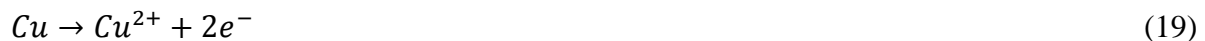
8.2.3.1 Decomposition voltage

The self-driving cell from Fig. 3 can be rearranged to a driven cell by connecting the power supply in the circuit as shown in Fig. 4.

The reactions on the electrodes will be



and



and the electron stream will flow in the opposite direction to that in the same cell working as a self driven one only if the power supply voltage is larger than the equilibrium potential difference of the same cell, working as a self driven one.

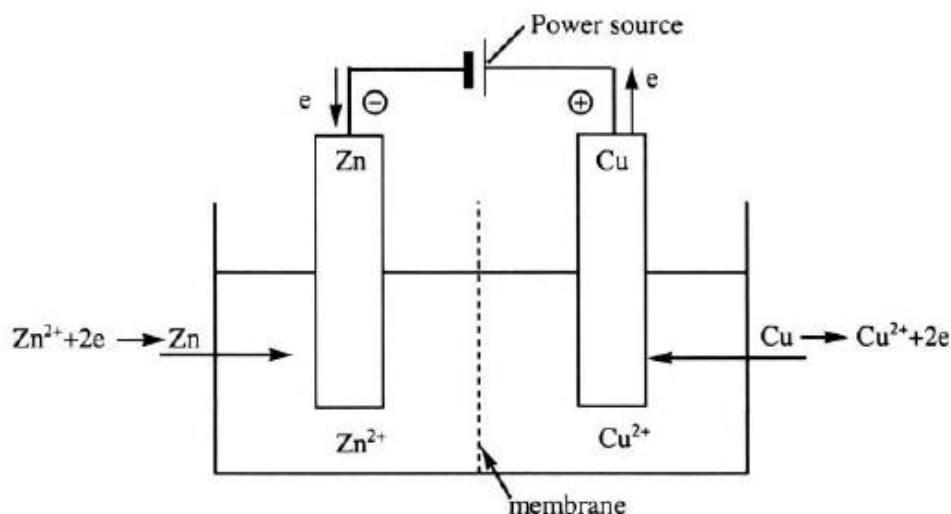


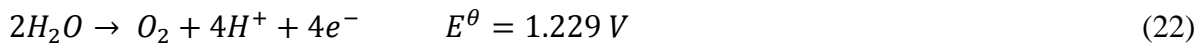
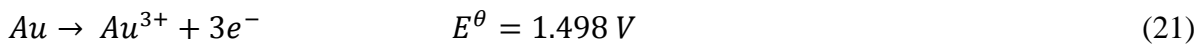
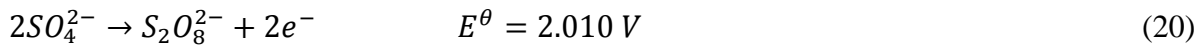
Figure 4. Schematic representation of the cell $\text{Zn}|\text{ZnSO}_4, \text{H}_2\text{O}, \text{H}_2\text{SO}_4||\text{ZnSO}_4, \text{H}_2\text{O}, \text{H}_2\text{SO}_4|\text{Cu}$ as a driven cell.

If two or more anodic and cathodic reactions are possible in some driven cell, the reactions with the lowest equilibrium potential difference will take place on the electrodes first. This means that the reaction with the most positive equilibrium potential will take place first on the cathode (the electrode connected with the negative terminal of power supply, at which reduction occurs), and the reaction with the most negative equilibrium potential on the anode (the electrode connected with the positive terminal of the power supply, at which oxidation occurs). It is important to remember that the terms anode and cathode are connected with the nature of the reaction (oxidation or reduction) at the electrode and not with their polarity. Thus, in a self driven cell, the anode is the negative terminal and the cathode is the positive terminal of the cell, a situation which is precisely the opposite of that which exists in an externally driven cell.

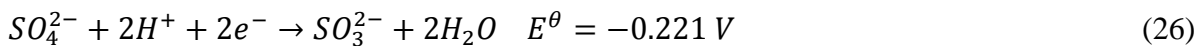
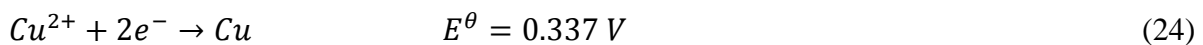
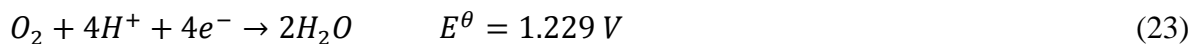


8.2.3.2 A cell with an insoluble anode

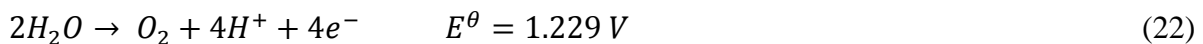
In the driven cell: $(-)\text{Cu}|\text{CuSO}_4, \text{H}_2\text{SO}_4, \text{H}_2\text{O}|\text{Au}(+)$ the following reactions on **the anode (Au)** are possible:



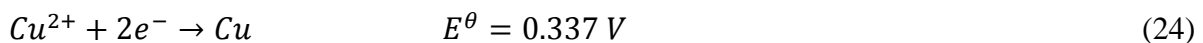
and **on the cathode (Cu)**



According to the rule derived in the conclusion from section 2.3.1, on the anode the oxygen evolution reaction will occur, and on the cathode copper ions will be reduced to the metal phase, because the most positive cathodic reaction can be neglected due to the low oxygen concentration in the electrolyte solution. Hence the reaction:



will take place at the anode and the reaction



at the cathode.

Obviously, the minimum external cell voltage for electrolysis to occur in this case is 0.893 V.

The overall reaction in the cell is

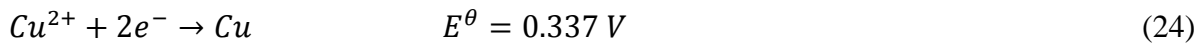
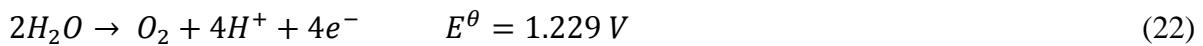
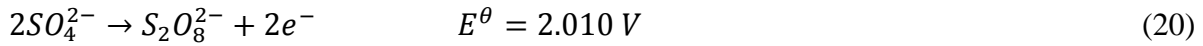


It follows from the Eq. 25 that in a cell with an insoluble anode the concentration of depositing ions decreases and the hydrogen ion concentration increases during electrolysis. The mechanism of the extraction of metals from ionic solutions and the essence of the electrowinning process are well explained by Eq. 25.

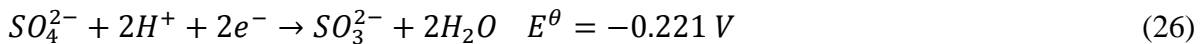
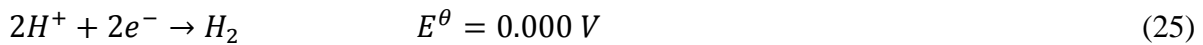
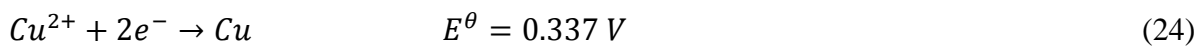
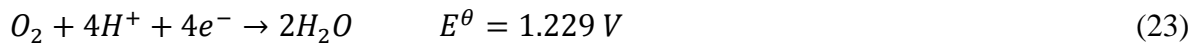


8.2.3.3 A cell with a soluble anode

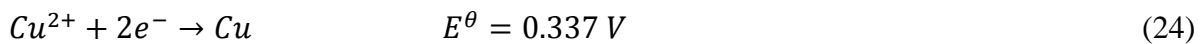
In the cell: $(-)\text{Au}|\text{CuSO}_4, \text{H}_2\text{SO}_4, \text{H}_2\text{O}|\text{Cu}(+)$ the following reactions are possible on the anode (**Cu**):



and on the cathode (**Au**):



Hence, the reaction



occurs on the anode, and



at the cathode, and so the composition of the electrolyte solution remains constant if the anode is made of pure copper and oxygen is removed from the solution. The lowest cell voltage at which electrolysis can start in this cell is zero.

It is obvious that electrorefining processes are based on electrolysis in cells with soluble anodes.

8.2.3.4 Current efficiency

Metal deposition can be accompanied by any other cathodic reaction, most frequently hydrogen evolution. This leads to the situation in which metal is deposited but metal deposition uses only a part, I_{Me} of the total current, I , through the cell. The current efficiency

$$\eta = \frac{I_{Me}}{I} \quad (27)$$

indicates which part of the total current is used for the deposition of metals. It is a very important parameter of an electrodeposition process.

8.2.3.5 Faraday's law

Faraday's law relates the quantity of electricity passed through the cell and the quantity of chemical substances which react on the electrodes. It states that the mass of metal, m , electrodeposited on the cathode is given by

$$m = \frac{ItM}{nF} \eta_I \quad (28)$$



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where I is the total current, t is the deposition time, M is the molar mass of the deposited metal, η_I is the current efficiency and nF is the number of Faradays per mole of consumed ions. It follows from Eq. 28 that can be easily determined by measuring the electrodeposited mass of metals and supplied quantity of electricity.

8.3 EQUIPMENT AND MATERIALS

Apparatus: Eye protection, Beaker (250 cm³), Stainless steel plate cathodes and one hard lead plate anode electrodes (about 5 mm diameter), 2 Retort stand and clamp to hold electrodes, DC power supply (6 volt), Leads and crocodile clips 4.

Chemicals: Aqueous copper(II) sulphate, about 0.5 M, 200 cm³

8.4 EXPERIMENTAL PROCEDURE

Electrolysis is the chemical process of using an electrical current to stimulate non-spontaneous reactions. A **non-spontaneous reaction** is one that needs energy to work while it proceeds. In other words, the process would not happen on its own, as it goes in an unfavourable, or a reversed, direction.

Important uses include its role in separating metals from a natural source, such as our friend the copper ore. Other useful benefits for electrolysis include the purification of metals and decomposition of compounds like water.

The site where electrolysis occurs is in an **electrolytic cell**, which is a type of electrochemical cell that drives an electrical current using a non-spontaneous reaction. So, not like a cell in your body, but a container. Thus, these cells must have an energy source to drive the reaction in the reverse or opposite direction, like a battery. Be careful not to confuse an electrolytic cell with electrolysis. Always remember that the electrolysis process is performed using an electrolytic cell.

How Does Electrolysis Work?

We can understand the process of electrolysis by looking at an example involving the purification of a copper metal. This process will require an **anode**, a positively charged electrode, and a **cathode**, a negatively charged electrode. In this case, an impure copper metal is the anode, while the pure copper metal is the cathode. The goal is to use electrolysis for the purpose of recycling a dirty old piece of copper pipe, or impure copper, to a beautiful, purified copper pipe.



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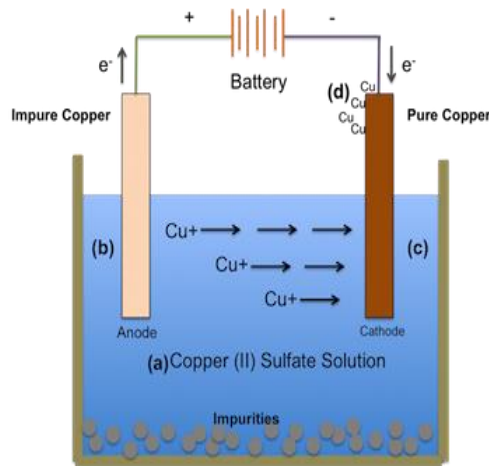


Figure 5. Schematic representation of copper (II) sulphate solution electrolysis cell.

The copper sulfate **electrolyte**, or solution, ensures there is a liquid medium conducive for ion movement inside the cell (a). When copper ions leave the impure copper pipe through the help of an energy supply, they migrate from the anode (b) to the cathode (c). After reaching the cathode, they collect the electrons needed to form a neutral, or no charge, copper metal. These neutral copper metal atoms gather on the pure copper cathode (d). The leftover impurities from the anode, or the ones not traveling to the cathode to be purified, collect at the bottom of the cell.

In the experiment copper sulfate solution is prepared at a concentration of 25 g / ml to be used instead of the copper leaching solution. The prepared solution is divided into equal amounts in two separate beakers. Anode and cathode plates are prepared with two pre-cleaned and prepared stainless steel plate cathodes and one hard lead plate anode. Cathode plates are weighed with a precision scale before the experiment and their weights are noted. A cathode and an anode plate prepared for each experiment are immersed in solution parallel to each other and current is supplied to the prepared system from the power supply.

Two experiments are performed in which all parameters except the current intensity are kept constant (duration: 15 min, concentration: 25 g / ml Cu_2SO_4). At the beginning of the experiments, the system is applied in such a way that the amount of current is 0.1 A and the second is 0.2 A. Thus, it is tried to observe the effect of the applied current amount on the amount of copper obtained.

Both experiments are carried out for 15 minutes, then the copper deposited cathode plates are dried and finally weighed. As a result of the test, the amount of copper deposited is calculated from the difference between the initial and final weights of the cathode plates obtained as a result of each test.



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8.5 ASSIGNMENTS AND REQUESTS

Determine the amount of copper deposited at the cathode plates.

8.6 REFERENCES

1. POPOV K.I., DJOKIĆ S.S., GRGUR B.N., “Fundamental Aspects of Electrometallurgy”, 2002
2. YTÜ Metalurji Müh., “Demir Dışı Metal Üretimi Laboratuvar Kitapçığı”, 1997
3. BOR, F.Y., “Ekstraktif Metalurji Prensipleri Kısım II”, Birinci Baskı, 1989



9. PRECIPITATION HARDENING

9.1 AIM:

The objective of this laboratory is for you to explore the strengthening of Al by precipitation hardening.

9.2 THEORY:

Pure fcc metals such as Al have low yield strengths because the stress required to move a dislocation is small. One method of strengthening an alloy is through precipitation reactions. Precipitation hardening is the process of hardening or strengthening of an alloy by precipitating finely dispersed precipitates of the solute in a supersaturated matrix. This process involves the following three basic steps:

1. The first step is the solution heat treatment or homogenization. Figure 1 illustrates this phenomenon. During this step, an alloy of composition X_1 is heated to a temperature T_1 , between the solvus and solidus temperatures and soaked there until all of the solute dissolves into the α phase and a uniform solid-solution structure is produced.

2. The second step is quenching. Quenching is simply cooling the sample rapidly to a lower temperature, T_3 , usually room temperature, and the cooling medium is usually water at room temperature. During quenching, the solute is not immediately able to diffuse out of a phase and the alloy is said to be supersaturated. The rationale behind the quenching process is to preserve the uniform solid solution structure of the alloy below the homogenization temperature.

3. The final step in the precipitation hardening process is aging. It is the process of precipitating incoherent precipitates from a supersaturated solid solution. When aging occurs at room temperature, it is called natural aging. Aging above room temperature is called artificial aging.

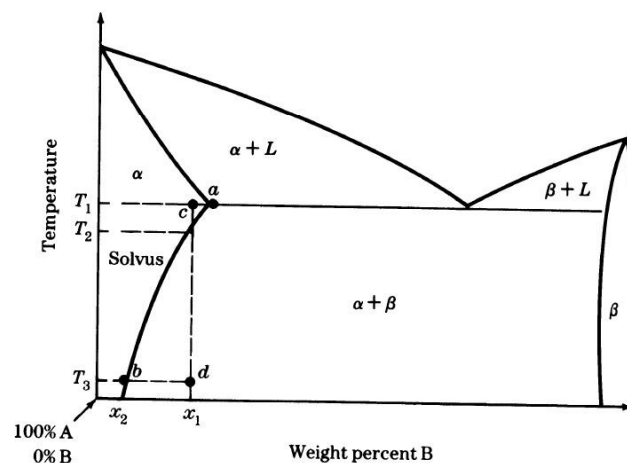


Figure 9.1 Binary phase diagram for two metals A and B having a terminal solid solution which has a decreasing solid solubility of B in A with decreasing temperature



The phase diagram for Al-Cu is illustrated in Fig. 2. By solution annealing an Al alloy (2024) containing ≈ 4.4 Wt% Cu, and then quenching, i.e. cooling so fast that little diffusion is possible, the Cu can remain dissolved in Al, but it is not in equilibrium. It was mentioned above that solute hardening is not an effective hardening mechanism, and so the quenched alloy must be slowly annealed to precipitate incoherent Al_2Cu (θ phase) particles. Since the number density of precipitates controls hardness, not the size of the individual particles, the trick is to nucleate as many particles as possible but keep them from growing. If the anneal is too long or at too high a temperature, some precipitates will grow while others shrink and disappear, lowering the number of precipitates and the strength. This condition is called overaging.

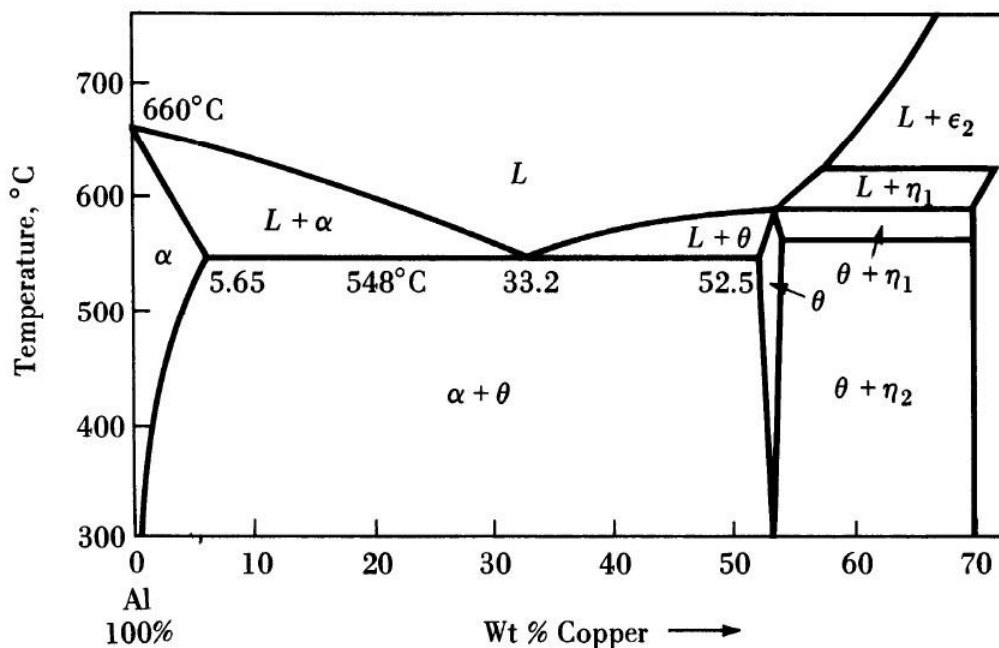


Figure 9.2 Phase diagram for Al-Cu

As it turns out, Cu in aluminum do not simply precipitate out according to the phase diagram, but rather it forms metastable precipitate phase, before finally forming the equilibrium phase, such as Al_2Cu . For Al-Cu, the first phase to form is a fully coherent precipitate called a GP (Guinier Preston) zone. This is followed by intermediate θ'' and θ' phases before the stable θ (Al_2Cu) phase forms. As these reactions occur, the hardness changes, at first increasing as the precipitates nucleate and grow (or age), and changing from coherent to incoherent phases. Eventually the strength decreases as the incoherent precipitates grow in size but decrease in number (i.e., overage), see Fig. 3.

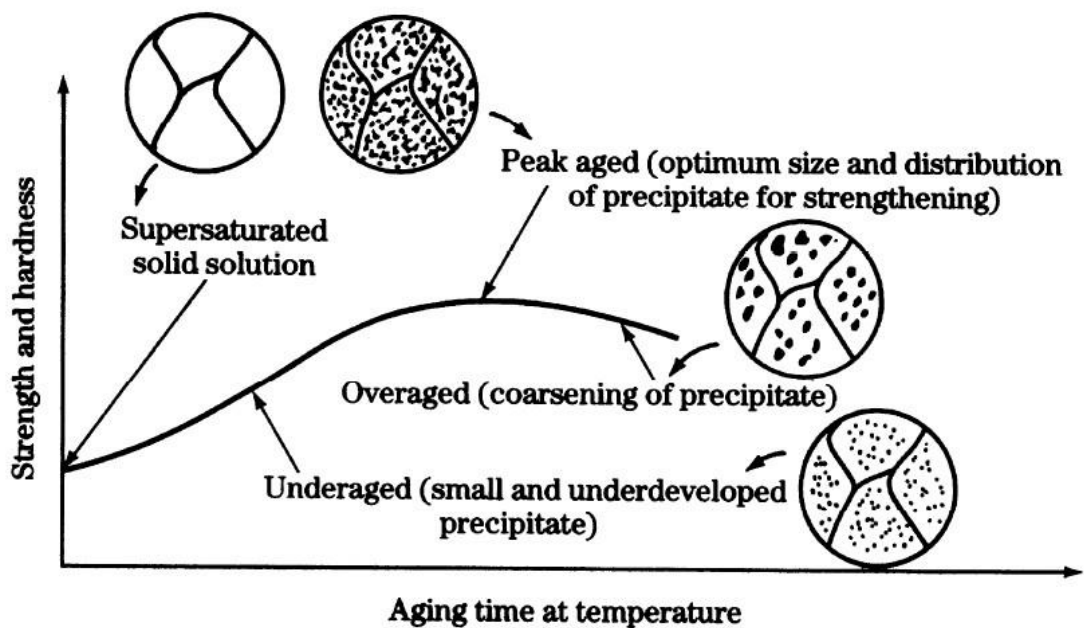
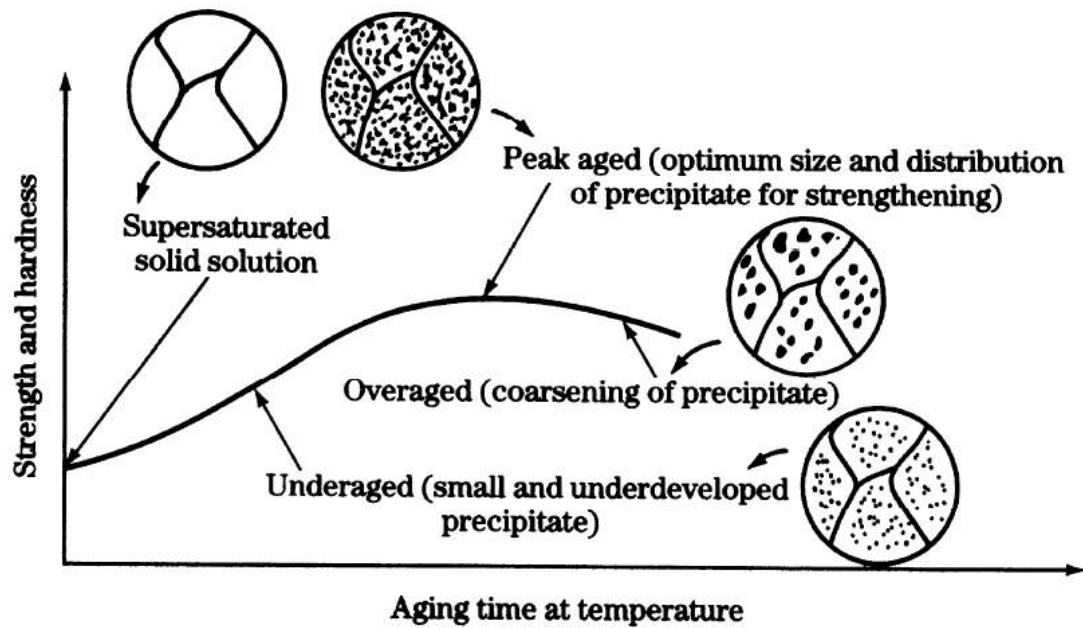


Figure 9.3 Microstructural development with aging time.



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EXPERIMENT 9: PRECIPITATION HARDENING



9.3 STANDARDS

ASTM: B918/B918M

MATERIALS AND APPARATUS

- Heat treatable Al samples
- Heat treatment furnace
- Quenching gadget
- Etuv
- Hardness test machine

9.4 PROCEDURE:

Two types of heat treatments will be conducted:

1. Temper T4: Solution treatment and water quench and natural aging.
2. Temper T6: Solution treatment, water quench, and artificial aging.

In this experiment heat treatable Al alloy such as AlMgSi or AlCuMg will be used. All specimens must have at least 2 mm thickness. Commercially purchased Al alloys should be solutionized and quenched into cold water. In this lab, we will use a common solutionizing time of 30 min. and a solutionizing temperature of 520-530 C. For temper T4 hardness measurement must be made immediately cause precipitation process begins at the time. For temper T6, the bars should be aged at 150 C for ½, 1, 2, 3, 4, 5, 6, 8, 10 h and 200 C and 250 C degrees for ¼, ¾, 1,5, 2,5, 3,5, 5 h, following the solutionizing and water quench. After the aging, the samples should be removed and allowed to cool in air.

9.5 PRECAUTIONS

- Proper solution time must be given to the specimens.
- Proper care should be taken while hardening the specimens.
- The specimens must be as per IS and BS specifications.

9.6 STUDENTS PREPARATION PRE- AND AFTER EXPERIMENT

Pre-experiment; Students are to be research and study about chemical composition of Al alloys, and its effect on the hardening mechanism, Precipitation Hardening mechanism and heat treatment conditions of given materials.



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EXPERIMENT 9: PRECIPITATION HARDENING



After experiment; Students are to do hardness test after tempering on the Al samples. All hardness values versus time are to be shown as graphic style and the reason of differences between these values are to be explained.

Experiment report shall include;

- Description Precipitation Hardening of metallic materials
- Main theme of test
- Used materials, equipments and apparatus of experiment,
- Test conditions
- Hardness values obtained from experiment are shown as graphical style
- Detailed analysis of differences between samples
- References



10. CORROSION EXPERIMENTS

PART 10.1: CORROSION OF ZINC IN ACIDIC SOLUTION

10.1.1 AIM: Investigation of the electrochemical behavior of zinc in acidic solution.

10.1.2 THEORIC INFORMATION

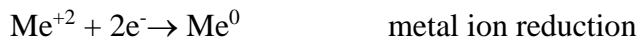
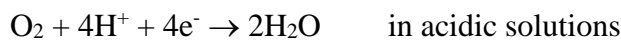
Metals undergo electrochemical corrosion degradation in aqueous environments. Electrochemical corrosion occurs spontaneously with the electrochemical cell formation in the metal-environment system. There is no need to external energy.

5 elements must be present in electrochemical corrosion system. These are anode, cathode, interface, electronic conductor and electrolytic conductor. While oxidation reactions occur on the anode, reduction reactions occur on the cathode.

On anode;



On cathode;



reactions occur. The reactions on the cathode are evaluated according to the environmental conditions. Corrosion occurs on the anode regions. Cathode reactions occur on the cathode, the metal does not degrade.

There are various methods for the quantitative evaluation of the corrosion experiments. In the method of calculating the weight loss, the weight loss of the test sample weighed before and after the experiment is used.

$$H = K \cdot \Delta G / A \cdot d \cdot t$$

H: Corrosion rate

G: Weight loss (gr) ($\Delta G = G_1 - G_2$)

A: Surface area exposed to the solution (cm^2)

d: Metal density (gr/cm^3)

t: Duration of experiment (sn)

K: Constant for the desired corrosion rate unit (3.45×10^6 for mpy)



The fact that the cathodic reactions in electrochemical corrosion phenomena is hydrogen and oxygen reduction, in some cases, allows the determination of the amount of gas released during the experiment and the determination of the corrosion rate. At very low rates of corrosion, calculating the increase of the metal ion concentration in the corrosive solution gives more accurate results than calculating the weight loss in the metal sample. In determining the corrosion rate, a suitable method, that will give an accurate result, should be chosen.

10.1.3 EXPERIMENTAL PROCEDURES

Equipment and Materials: Beaker, glass funnel, burette, pipette pump, drying machine, scales, waterproof polishing papers, HCl solution, zinc, rope.

Experimental:

Zinc sample is cut in a certain size from the zinc plate and make a hole to hang. And then the surface and edges of the sample are polished using water proof abrasive papers having various grit numbers following by cleaned from the oil and dirt on the surface in the ultrasonic bath. The sample, whose dimensions and weight are measured, is fixed to the bottom of the funnel using the line rope. The funnel is placed through a burette and is put it upside down in a beaker filled with the HCl solution of pH 1, as shown in Figure 1. And the the solution is drawn into the burette at a certain level. The drop in the solution level is recorded every 3 minutes. Also, the time, when the solution level reaches zero in the burette, is recorded. At the end of the experiment, the sample is removed from the solution, washed, dried and weighed again.

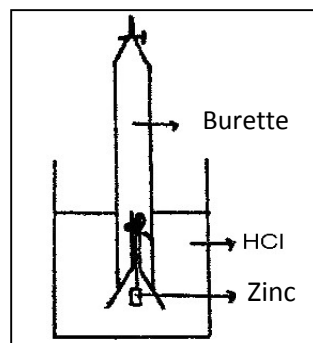


Figure 10.1 Experimental set-up

10.1.4 REQUIREMENTS:

1. Write cathodic and anodic reactions.
2. Calculate the total corrosion rate (mpy) using the method of the calculating the mass loss.
3. Calculate the total corrosion rate (mpy) using the method of the determining the amount of the evolved gas during the test. Plot the change in corrosion rate depending on the time, using the dissolved amount of zinc (mgr) -time (min) axes and examine this change and find the corrosion rate. Use the recorded your gas volume readings in the burette during experiments.



EXPERIMENT 10: CORROSION EXPERIMENTS

4. Compare the corrosion rates calculated using both the methods of the calculating the mass loss of the samples and the determining the amount of the evolved gas during the test. Is the corrosion rate determined by the weight loss the same as the rate of corrosion determined by the loss of hydrogen gas? If not, indicate the reasons.

PART 10.2: GALVANIC CORROSION

10.2.1 AIM: Determination of the effect of cathode / anode surface areas and stirring of solution on corrosion rate in galvanic corrosion.

10.2.2 THEORIC INFORMATION

The potential difference in any system can be caused by many reasons:

- Contact of different metals,
- Difference in ion concentration on the metal surface in the solution,
- Different stress zones in metal,
- Internal structure differences in metal,
- Temperature differences.

Galvanic corrosion is a type of corrosion that occurs when different types of metals and alloys in contact with each other are placed to the same atmosphere. While the metal, which has a more negative electrode potential than the other metal which is into contact with each other, is acting as an anode and degrades, the other metal acting as a cathode and protected from corrosion. As the potential difference between the metals increases, the probability of galvanic corrosion increases. The environmental mobility and the surface size of the metals are also important among the other factors affecting the galvanic corrosion. In case of the active metal has a smaller surface area than the noble metal, the corrosion is faster because a small anode area raises electrons to a larger cathode area.

10.2.3 EXPERIMENTAL PROCEDURES

Equipment and Materials: Beaker, magnetic stirrer, ultrasonic cleaner, 1Ω resistor, 2 electrode holders, ammeter, steel and copper plates, various waterproof abrasive paper, 3.5% NaCl solution, methyl alcohol.

Experimental:

In this experiment, copper and steel pairs are used. First, the surfaces of the samples are cleaned, washed and removed oil. Then the parts, which will be dipped into the solution, are marked on the surfaces. First, copper and steel sheets having equal size are placed in electrode holders and immersed in 3.5% NaCl solution (Figure 2). The current flowing is recorded through a resistor connected between the electrodes through the system after waiting for 3 minutes. And then the solution is started to stir and the current is again recorded after 3 minutes. The abovementioned procedures are repeated using large copper-small steel and small copper-large steel plates.



EXPERIMENT 10: CORROSION EXPERIMENTS

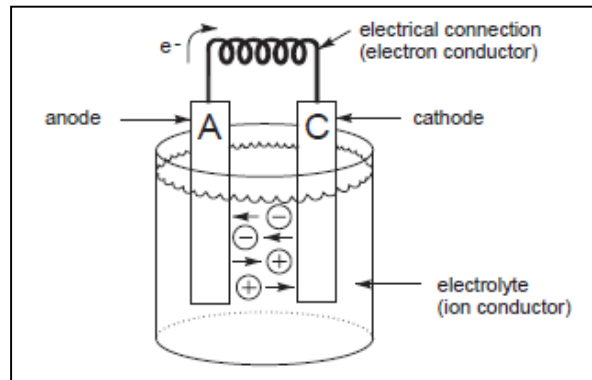
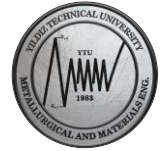


Figure 10.2 Experimental set-up in galvanic corrosion

10.2.4 REQUIREMENTS:

1. Define and write each anodic and cathodic reactions.
2. Draw the relation of $A_{\text{anode}} / A_{\text{cathode}}$ as a graph for all measurements (steady and stirred state) and explain.



11. PRODUCTION OF COMPOSITE MATERIALS

11.1 OBJECTIVE

Production of E-glass reinforced plastic matrix composite plates and making fracture analysis.

11.2 THEORY

The new material group formed by at least two different materials via forming an interface in between without creating chemical bond is called composite.

In the composites the load-bearing main element is the reinforcement fibers. The most commonly used and cost-effective reinforcement type is the glass fiber. Aramid and carbon fibers are both reinforcement types with high mechanical properties and expensive. Reinforcements are also available in different ways. Figure 1 shows the classification of composites according to their reinforcement shape and distribution. Figure 2 illustrates the conditions of these species within the structure. Figure 3 shows different weaving types.

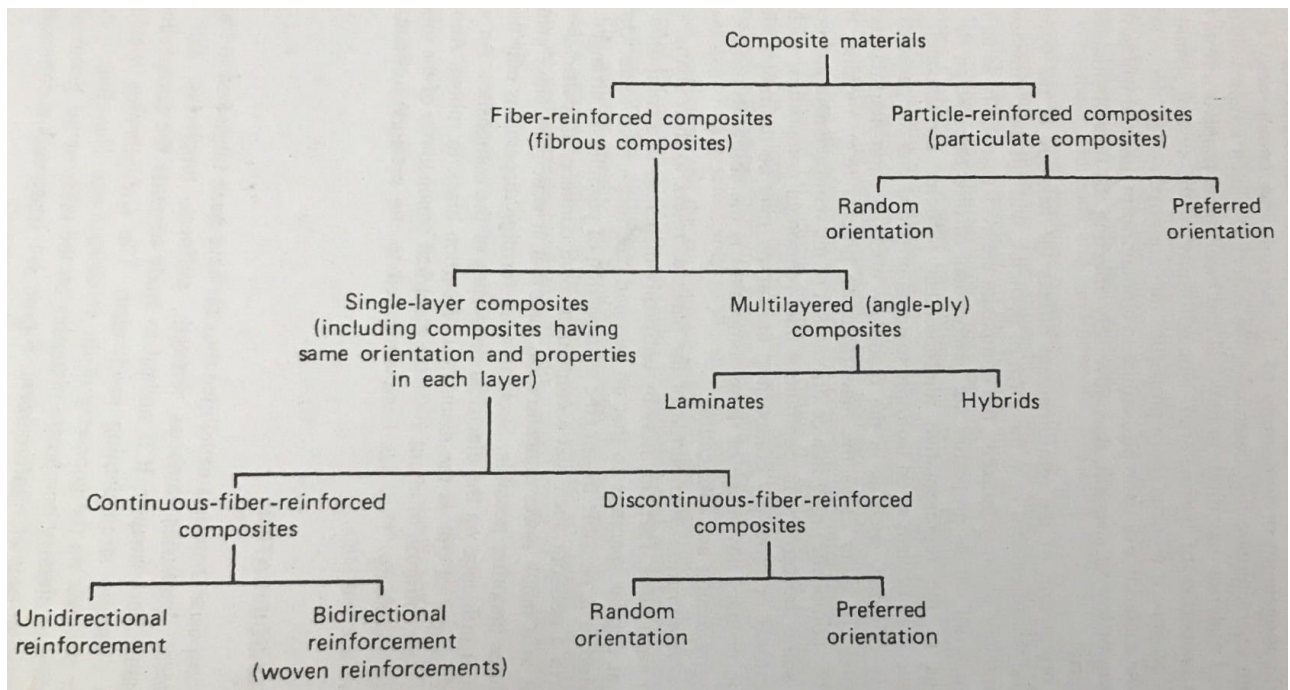


Figure 11.1 Classification of composites according to reinforcement shapes and distributions

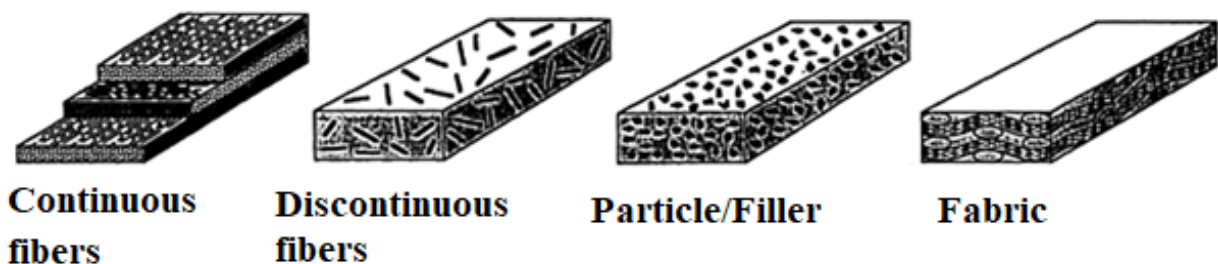


Figure 11.2 Various reinforcement types

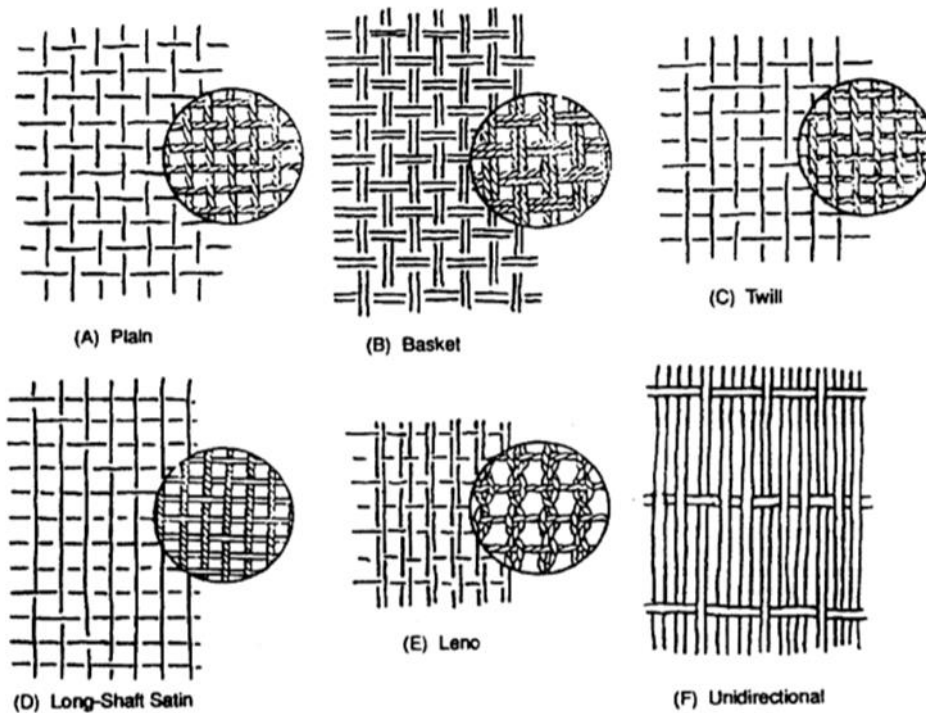


Figure 11.3 Various woven fabric types

11.2.1 Grouping of Composite Materials by Matrices

- **Plastic Matrix Composites (PMCs):** It is the most widely used composite type today. It has approximately 80% utilization rate in all composites. They can be reinforced with continuous or discontinuous fibers, or they can be reinforced with particles.
- **Metal Matrix Composites (MMCs):** It is the most used group after plastic matrix composites. Metal matrices, which are generally used as particle-reinforced, have applications in where wear resistance is required. In addition, honeycomb structure produced from aluminum is included in this group.
- **Ceramic Matrix Composites (CMCs):** They are not widely used. Unlike other groups, it has a brittle matrix. Composites in this group are reinforced with ductile fibers to increase the toughness of the brittle matrix.

11.2.2 Plastic Matrix Composite

Plastic matrix is the most widely used composite type with many advantages. Therefore, it is important to know the plastic to learn the properties of the composite. Plastics consists of repeating of molecules of simple molecules formed by carbon, hydrogen, oxygen, nitrogen and other organic or inorganic elements. The plastic matrix is generally divided into two groups:

- a) **Thermosets:** Epoxy, polyester, vinylester, phenolic resins
- b) **Thermoplastics:** PE (polyethylene), PP (polypropylene), PS (polystyrene), PET (polyethylene terephthalate), PBT (polybutylene terephthalate), PA (polyamide), PPS (polyphenylene sulfide), PEEK (polyether ether ketone)...



According to the reinforcement, a classification can be made as follows:

- Plastic-Plastic Composites
- Plastic-Metal Composites
- Plastic-Ceramic Composites
- Plastic-Foam Composites

11.2.3 Production Methods of Plastic Matrix Composites

- Hand lay-up
- Spray-up
- Vacuum bagging and autoclave
- Vacuum infusion
- RTM (Resin Transfer Molding)
- SMC/BMC (Sheet Mold Compound/Bulk Mold Compound)
- Filament winding
- Centrifugal casting
- Pultrusion
- Thermoplastic injection/extrusion

11.2.4 Glass Fiber Production

Glass fiber is produced from conventional glass production raw materials such as silica, colemanite, aluminum oxide, soda, magnesium oxide. The raw material is finely ground and mixed to obtain a homogeneous mixture and fed to a melting furnace operating at about 1600 ° C. Here, the mixture slowly becomes liquid. With a winding system suitably placed in the process, a high velocity of 50-70 m/s and a 5-20 micron diameter glass fiber are collected on a bobbin by winding on the mandrel, depending on the type of application.

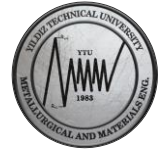
The glass fibers are coated with a chemical composition, called a binder, before being bundled. The type of binder is one of the most important factors affecting the performance of glass fiber in the composite material. The strength of the composite is proportional to the strength of the resin-glass bond. The strength of this bond depends on the type of binding groups in the binder used. The binder consists of a mixture of "film-forming", "binding groups", "antistatic additive", "plasticizer" "lubricant" materials.

11.3 USED STANDARDS

TS EN ISO 179-1: Plastics - Determination of Charpy impact properties - Part 1: Impedance impact test

11.4 USED DEVICES AND MATERIALS

- Fiber reinforced composite samples according to test standards
- Digital caliper
- Impact device



11.5 EXPERIMENT

11.5.1 Materials

- Reinforcement element: E-glass weave or mat fabric
- Matrix material: Orthophthalic based unsaturated polyester resin
- Resin accelerating agent: Cobalt octoate
- Resin initiating agent: Methyl Ethyl Ketone (MEK) Peroxide
- Dosing pipettes
- Resin Mixing Bowl
- Mixer Mold
- Mold release agent
- Brush
- Acetone or thinner
- Plastic glove

11.5.2 Application

1. The mold release agent is applied to mold and left to dry.
2. The reinforcing material to be used is cut into appropriate sizes and its weight is measured.
3. Twice amount of resin is prepared in a container by mixing with accelerator and the initiating agent at a required ratio. (The accelerator and initiator should not be placed at the same time because they react violently with each other!)
4. Apply a small amount of resin with a brush onto the dried mold release agent.
5. A layer of reinforcing fabric is placed on it.
6. Resin is applied by light pressure on the reinforcing fabric with brush.
7. Put the second layer of reinforcing fabric and repeat the process until required thickness is obtained.
8. Wait until the resin is cured.
9. Clean the brushes, containers and pipettes with acetone.

11.6 RECEIVING RESULTS

The dimensions of the samples in different layer thicknesses will be measured and the cross-sectional area will be calculated by taking minimum values from three measurements. Each student will do this by using a digital caliper. The test shall be carried out by placing the sample on impact device. Impact energy will be measured, samples will be compared and the fracture type will be examined.

11.7 CALCULATION

Impact energy will be measured in joules (J) and divided by the cross-sectional area of the sample.



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EXPERIMENT 11: PRODUCTION OF COMPOSITE MATERIALS



11.8 CONCLUSION

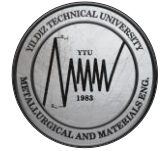
The production steps of the composite parts will be observed in details. Materials with various layers thicknesses will be compared. Fracture analysis will be done. fiber pull-out and/or delamination problems will be observed in fractured sections.

11.9 INFORMATION REQUIRED IN THE EXPERIMENT REPORT

1. Definition, classification, types and examples of composite materials.
2. General information about plastic materials used as matrix materials, types, examples.
3. Short information about the production methods of plastic matrix composite materials.
4. General information about glass fibers.
5. Identification of materials used.
6. Application of composite production.
7. Calculation of the impact strength.
8. Comparison of the results of the samples.
9. Fracture analysis.
10. Specifying the sources used at the end of the report.

11.10 REFERENCES

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12. NON-DESTRUCTIVE TESTING

12.1 AIM

The aim of this laboratory is to determine the internal and external faults found in materials and to gain experience with and understanding of the types, advantages and applications of various NDT methods. To be able to choose the best NDT method for a given part.

12.2 EQUIPMENT AND MATERIALS USED

Ultrasonic testing apparatus, ultrasonic probes, metallic reference (calibration) blocks, ultrasonic gel, x-ray tube, reference radiographs, penetrometer, radiography device, magnetic particles, fluorescence powder, ultraviolet lamp, penetration fluid, developer fluid, Eddy current test device, various surface cleaners.

12.3 BACKGROUND

Up to this point we have learnt various testing methods that somehow destruct the test specimens. These were, tensile testing, hardness testing, etc. In certain applications, the evaluation of engineering materials or structures without impairing their properties is very important, such as the quality control of the products, failure analysis or prevention of the engineered systems in service.

This kind of evaluations can be carried out with Non-destructive test (NDT) methods. It is possible to inspect and/or measure the materials or structures without destroying their surface texture, product integrity and future usefulness.

The field of NDT is a very broad, interdisciplinary field that plays a critical role in inspecting that structural component and systems perform their function in a reliable fashion. Certain standards have been also implemented to assure the reliability of the NDT tests and prevent certain errors due to either the fault in the equipment used, the miss-application of the methods or the skill and the knowledge of the inspectors.

Successful NDT tests allow locating and characterizing material conditions and flaws that might otherwise cause planes to crash, reactors to fail, trains to derail, pipelines to burst, and variety of less visible, but equally troubling events. However, these techniques generally require considerable operator skill and interpreting test results accurately may be difficult because the results can be subjective.

These methods can be performed on metals, plastics, ceramics, composites, cermets, and coatings in order to detect cracks, internal voids, surface cavities, delamination, incomplete defective welds and any type of flaw that could lead to premature failure. Commonly used NDT test methods can be seen in table 1. These are universal NDT methods; however, very special tests have been developed for specific applications.



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EXPERIMENT 12: NON-DESTRUCTIVE TESTING



Table 12.1 Commonly used NDT techniques

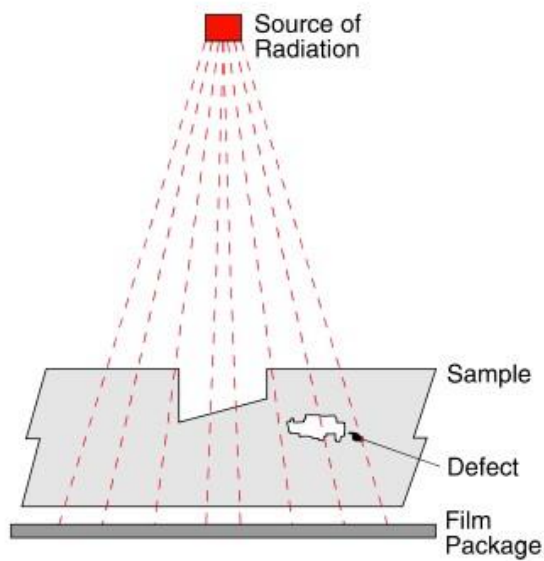
Technique	Capabilities	Limitations
Visual Inspection	Macroscopic surface flaws	Small flaws are difficult to detect, no subsurface flaws.
Microscopy	Small surface flaws	Not applicable to larger structures; no subsurface flaws.
Radiography	Subsurface flaws	Smallest defect detectable is 2% of the thickness; radiation protection. No subsurface flaws not for porous materials
Dye penetrate	Surface flaws	No subsurface flaws not for porous materials
Ultrasonic	Subsurface flaws	Material must be good conductor of sound.
Magnetic Particle	Surface / near surface and layer flaws	Limited subsurface capability, only for ferromagnetic materials.
Eddy Current	Surface and near surface flaws	Difficult to interpret in some applications; only for metals.
Acoustic emission	Can analyze entire structure	Difficult to interpret, expensive equipments.

Visual inspection:

VI is particularly effective detecting macroscopic flaws, such as poor welds. Many welding flaws are macroscopic: crater cracking, undercutting, slag inclusion, incomplete penetration welds, and the like. Likewise, VI is also suitable for detecting flaws in composite structures and piping of all types. Essentially, visual inspection should be performed the way that one

would inspect a new car prior to delivery, etc. Bad welds or joints, missing fasteners or components, poor fits, wrong dimensions, improper surface finish, delamination in coatings, large cracks, cavities, dents, inadequate size, wrong parts, lack of code approval stamps and similar proofs of testing.

Radiography has an advantage over some of the other processes in that the radiography provides a permanent reference for the internal soundness of the object that is radiographed. The x-ray emitted from a source has an ability to penetrate metals as a function of the accelerating voltage in the x-ray emitting tube. If a void present in the object being radiographed, more x-rays will pass in that area and the film under the part in turn will have more exposure than in the non-void areas. The sensitivity of x-rays is nominally 2% of the materials thickness. Thus, for a piece of steel with a 25mm thickness, the smallest void that could be detected would be 0.5mm in dimension. For this reason, parts are often radiographed in different planes. A thin crack does not show up unless the x-rays ran parallel to the plane of the crack. Gamma radiography is identical to x-ray radiography in function. The difference is the source of the penetrating electromagnetic radiation which is a radioactive material such as Co 60. However, this method is less popular because of the hazards of handling radioactive materials.



Schematic illustration of a typical exposure arrangement for radiography. The source of radiation can be either an X-ray tube or a radioactive isotope.



The resultant radiograph shows the subject as seen from the source.

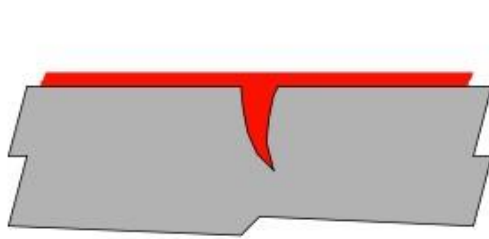
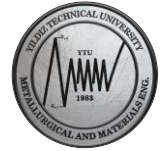
Liquid (Dye) penetrant method:

Liquid penetrant inspection (LPI) is one of the most widely used nondestructive evaluation (NDE) methods. Its popularity can be attributed to two main factors, which are its relative ease of use and its flexibility. The technique is based on the ability of a liquid to be drawn into a "clean" surface breaking flaw by capillary action.

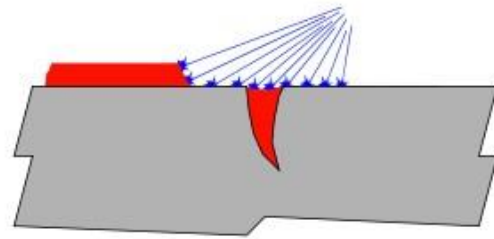
This method is an inexpensive and convenient technique for surface defect inspection. The limitations of the liquid penetrant technique include the inability to inspect subsurface flaws and a loss of resolution on porous materials. Liquid penetrant testing is largely used on nonmagnetic materials for which magnetic particle inspection is not possible.

Materials that are commonly inspected using LPI include the following; metals (aluminum, copper, steel, titanium, etc.), glass, many ceramic materials, rubber, plastics.

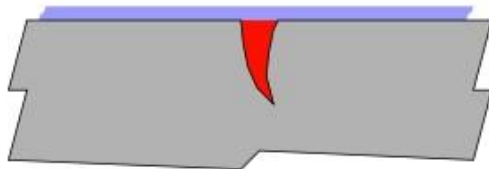
Liquid penetrant inspection is used to inspect of flaws that break the surface of the sample. Some of these flaws are listed below; fatigue cracks, quench cracks grinding cracks, overload and impact fractures, porosity, laps seams, pin holes in welds, lack of fusion or braising along the edge of the bond line.



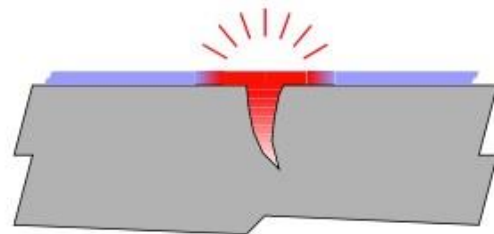
A. Penetrant applied to the surface and enters defect



B. Excess penetrant removed from surface



C. Developer powder applied to draw penetrant out of crack.

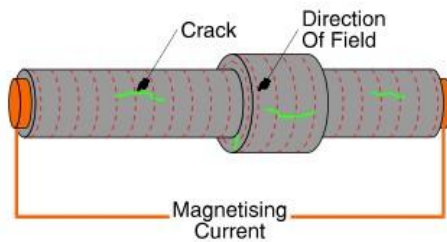


D. Accentuated indication of crack as penetrant spreads around the opening.

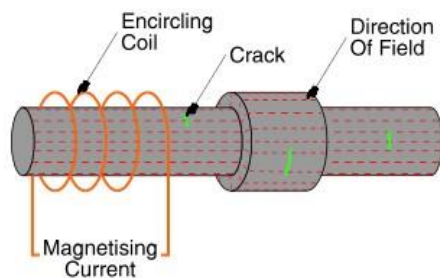
Magnetic particles:

Magnetic particle inspection is one of the simple, fast and traditional nondestructive testing methods widely used because of its convenience and low cost. This method uses magnetic fields and small magnetic particles, such as iron filings to detect flaws in components. The only requirement from an inspect ability standpoint is that the component being inspected must be made of a ferromagnetic material such iron, nickel, cobalt, or some of their alloys, since these materials are materials that can be magnetized to a level that will allow the inspection to be effective. On the other hand, an enormous volume of structural steels used in engineering is magnetic. In its simplest application, an electromagnet yoke is placed on the surface of the part to be examined, a kerosene-iron filling suspension is poured on the surface and the electromagnet is energized. If there is a discontinuity such as a crack or a flaw on the

surface of the part, magnetic flux will be broken and a new south and north pole will form at each edge of the discontinuity. Then just like if iron particles are scattered on a cracked magnet, the particles will be attracted to and cluster at the pole ends of the magnet, the iron particles will also be attracted at the edges of the crack behaving poles of the magnet. This cluster of particles is much easier to see than the actual crack and this is the basis for magnetic particle inspection. For the best sensitivity, the lines of magnetic force should be perpendicular to the defect.



Schematic arrangement for detecting longitudinal defects using current flow through the subject. The source of the current can be a portable / mobile power pack or, in the case of a bench unit, from the built-in power pack.



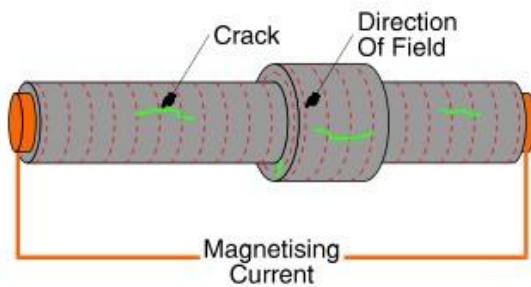
Schematic arrangement of an encircling coil for the detection of circumferential and transverse defects. The coil may be cable wrapped round loosely or wound on a former, as in a bench unit.

Eddy current testing:

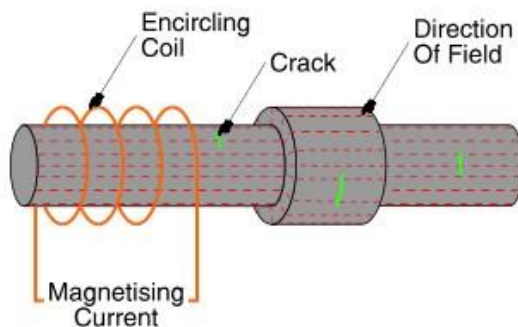
Eddy currents are created through a process called electromagnetic induction. When an alternating current is applied to the conductor, such as copper wire, a magnetic field develops in and around the conductor. This magnetic field expands as the alternating current rises to maximum and collapses as the current is reduced to zero. If another electrical conductor is brought into the close proximity to this changing magnetic field, current will be induced in this second conductor. These currents are influenced by the nature of the material such as voids, cracks, changes in grain size, as well as physical distance between coil and material. These currents form an impedance on a second coil which is used to as a sensor. In practice a probe is placed on the surface of the part to be inspected, and electronic equipment monitors the eddy current in the work piece through the same probe. The sensing circuit is a part of the sending coil.

Eddy currents can be used for crack detection, material thickness measurements, coating thickness measurements, conductivity measurements for material identification, heat damage detection, case depth determination, heat treatment monitoring.

Some of the advantages of eddy current inspection include; sensitivity to small cracks and other defects, ability to detect surface and near surface defects, immediate results, portable equipment, suitability for many different applications, minimum part preparation, no necessity to contact the part under inspection, ability to inspect complex shapes and sizes of conductive materials. Some limitation of eddy current inspection; applicability just on conductive materials, necessity for an accessible surface to the probe, skillful and trained personal, possible interference of surface finish and roughness, necessity for reference standards for setup, limited depth of penetration, inability to detect of the flaws lying parallel to the probe coil winding and probe scan direction.



Schematic arrangement for detecting longitudinal defects using current flow through the subject. The source of the current can be a portable / mobile power pack or, in the case of a bench unit, from the built-in power pack.



Schematic arrangement of an encircling coil for the detection of circumferential and transverse defects. The coil may be cable wrapped round loosely or wound on a former, as in a bench unit.

Ultrasonic Inspection:

Ultrasonic Testing (UT) uses a high frequency sound energy to conduct examinations and make measurements. Ultrasonic inspection can be used for flaw detection I evaluation, dimensional measurements, material characterization, and more. A typical UT inspection system consists of several functional units, such as the pulser/receiver, transducer, and display devices. A pulser/receiver is an electronic device that can produce high voltage electrical pulse. Driven by the pulser, the transducer of various types and shapes generates high frequency ultrasonic energy operating based on the piezoelectricity technology with using quartz, lithium sulfate, or various ceramics. Most inspections are carried out in the frequency range of 1 to 25MHz. Couplants are used to transmit the ultrasonic waves from the transducer to the test piece; typical couplants are water, oil, glycerin and grease.

The sound energy is introduced and propagates through the materials in the form of waves and reflected from the opposing surface. An internal defect such as crack or void interrupts the waves' propagation and reflects back a portion of the ultrasonic wave. The amplitude of

the energy and the time required for return indicate the presence and location of any flaws in the work-piece.

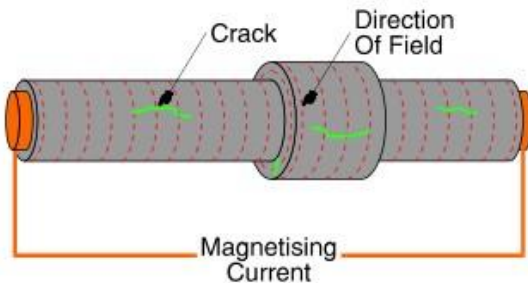
The ultrasonic inspection method has high penetrating power and sensitivity. It can be used from various directions to inspect flaws in large parts, such as rail road wheels pressure vessels and die blocks. This method requires experienced personnel to properly conduct the inspection and to correctly interpret the results.

As a very useful and versatile NDT method, ultrasonic inspection method has the following advantages; sensitivity to both surface and subsurface discontinuities, superior depth of

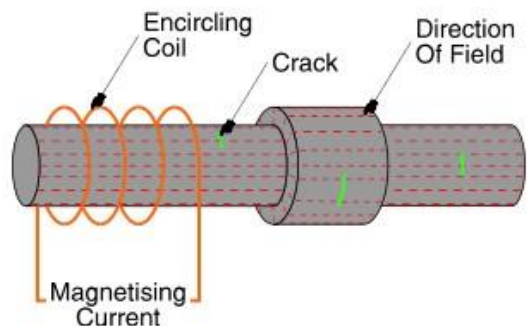


penetration for flaw detection or measurement, ability to single-sided access for pulse-echo technique, high accuracy in determining reflector position and estimating size and shape, minimal part preparation, instantaneous results with electronic equipment, detailed imaging with automated systems, possibility for other uses such as thickness measurements.

Its limitations; necessity for an accessible surface to transmit ultrasound, extensive skill and training, requirement for a coupling medium to promote transfer of sound energy into test specimen, limits for roughness, shape irregularity, smallness, thickness or not homogeneity, difficulty to inspect of coarse grained materials due to low sound transmission and high signal noise, necessity for the linear defects to be oriented parallel to the sound beam, necessity for reference standards for both equipment calibration, and characterization of flaws.



Schematic arrangement for detecting longitudinal defects using current flow through the subject. The source of the current can be a portable / mobile power pack or, in the case of a bench unit, from the built-in power pack.



Schematic arrangement of an encircling coil for the detection of circumferential and transverse defects. The coil may be cable wrapped round loosely or wound on a former, as in a bench unit.

Acoustic Method:

There are two different kind of acoustic methods: (a) acoustic emission; (b) acoustic impact technique.

Acoustic emission:

This technique is typically performed by elastically stressing the part or structure, for example, bending a beam, applying torque to a shaft, or pressurizing a vessel and monitoring the acoustic responses emitted from the material. During the structural changes the material such as plastic deformation, crack initiation, and propagation, phase transformation, abrupt reorientation of grain boundaries, bubble formation during boiling in cavitation, friction and wear of sliding interfaces, are the source of acoustic signals. Acoustic emissions are detected with sensors consisting of piezoelectric ceramic elements. This method is particularly effective for continuous surveillance of load-bearing structures.



Acoustic impact technique:

This technique consists of tapping the surface of an object and listening to and analyzing the signals to detect discontinuities and flaws. The principle is basically the same as when one taps walls, desktops or countertops in various locations with a finger or a hammer and listens to the sound emitted. Vitrified grinding wheels are tested in a similar manner to detect cracks in the wheel that may not be visible to the naked eye. This technique is easy to perform and can be instrumented and automated. However, the results depend on the geometry and mass of the part so a reference standard is necessary for identifying flaws.

12.4 PROCEDURES

Liquid penetrant method:

In this method, the surfaces to be inspected should be free from any coatings, paint, grease, dirt, dust, etc., therefore, should be cleaned with an appropriate way. Special care should be taken not to give additional damage to the surface to be inspected during the cleaning process. Otherwise, the original nature of surface could be disturbed and the results could be erroneous with the additional interferences of the surface features formed during the cleaning process. Surface cleaning can be performed with alcohol. Special chemicals like cleaner-remover can also be applied if needed. In the experiment, only cleaner-remover will be sufficient. Subsequent to surface cleaning, the surface is left to dry for 2 minutes.

Commercially available cans of liquid penetrant dyes with different colors are used to reveal the surface defects.

Steps used in the experiment:

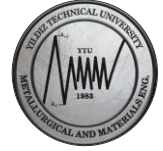
- Clean the surface with alcohol and let surface dry for 5 min.
- Apply the liquid penetrant spray (red can) to the surface and brush for further penetration. Then, wait for 20 min.
- Wipe the surface with a clean textile and subsequently apply remover spray (blue can) to remove excess residues on the surface and wait for a few min.
- Apply the developer spray (yellow can) at a distance of about 30cm from the surface. The developer will absorb the penetrant that infiltrated to the surface features such as cracks, splits, etc., and then reacted with it to form a geometric shape which is the negative of the geometry of the surface features from which the penetrant is sucked.
- The polymerized material may be collected on a sticky paper for future evaluation and related documentation, if needed.

Magnetic particle:

In this experiment, commercially available magnetic powder manufactured for NDT inspection will be used. A strong U shape magnet will be used to provide the necessary magnetic field at the inspected area.



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The following steps are applied during the experiment:

1. The surface of the specimen will be roughly cleaned wiping with a piece of textile. The fluorescent magnetic spray will be applied on the surface being inspected.
2. Magnetic field will be applied with a strong magnet to the location of interest.
3. The spots where the fluorescent magnetic particles accumulated will be inspected under UV light.

Eddy current inspection:

For this experiment, Magnefest ED-51 0 type unit will be used. A pencil type prop will be used for the inspections. The inspection is performed with 2 MHz frequency and at the related calibration settings. The test blocks were previously prepared for this experiment. Any coatings or paints on the surface of inspected specimens should be treated with special procedures.

The following steps should be applied during the experiment:

1. Inspection area should be clean, smooth, free from any irregular or uneven paint, dirt, grease, etc.
2. There shouldn't be any visible damage or discontinuity.
3. During the inspection procedure, the probe will be positioned near the inspection area, on the compensation point and lift off and zero will be adjusted if necessary.
4. The inspection will be carried out by using probe scans. The probe tip will be always at a right angle the inspection surface.
5. Any indication with indicator deflection to the right should be evaluated. All evaluated indications should be measured.
6. After this procedure, all evaluated indications with indicator deflections, will be classified as cracks and be recorded.

Ultrasonic inspection:

For this experiment, USM-2 type ultrasonic unit will be used. The props used supports to work at frequency of 5 MHz. Echo techniques will be employed to find the cracks.

Instrument will be tuned to a frequency of 5 MHz. An appropriate couplant used should not cause corrosion or other damage. During the inspection, the calibration will be done on the reference standard, if needed. Two different test blocks will be employed in this test, sufficient amount of couplant will be applied to the transducer scan areas on the forward and after sides of the support fitting. The display will be monitored for crack indications. A crack signal will be similar to the following:



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The following steps should be applied during the experiment:

1. The couplant should be applied on the inspected area.
2. For the circular test specimen, the prop will be placed in the corresponding space in the supporting fitting tool. Enough couplant should be used between the probe and tool.
3. For the flat specimen, no tool is needed, couplant only applied between the inspected surface and the probe.
4. Special attention should be paid on the location where possible cracks exist. A discontinuity like a crack produces a peak on the screen.
5. Attention should also be given to the movement of the possible peak caused by the cracks on the specimen.

12.5 REPORT

You are supposed to prepare a test report for this experiment obeying the report preparation rules. **So, your report should contain abstract, introduction, experimental procedures, results, discussion, conclusion and references. The advantages and disadvantages of each NDT method must be stated precisely in your reports.** You should also answer the questions asked to you at the end of the experiment installing the related parts of your report. You must return your report on time