Chemical Technology Lab. I

(10626478)

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Part Two

Material and Corrosion Lab

Contents

Experiment M1: Specimen Preparation for Metallographic Study and Microscopic Examination

Objectives:

- Learning and practicing the procedure used for preparing metallic specimens for metallographic studies.
- To study the microstructure of the given ferrous and non-ferrous alloys
- To calculate the grain size and an ASTM grain size number of these alloys
- To estimate the carbon content of the steel specimen

Theory

To estimate the grain size, ASTM grain size number and the carbon content of the steel specimen, using equations below:

$$N(4000) = \frac{Length\ (10\ cm) \times 1(\#of\ lines)}{\#of\ grains \times 4000} \qquad (1)$$

The number of grains were found by dividing the micrograph for each alloy into small squares.

Then:

$$N(100) \times 100^2 = N(4000) \times 4000^2 \qquad (2)$$

Also,

$$N(100) = 2^{n-1} \qquad (3)$$

Where:

N: number of grains per $(\text{inch})^2$ measures at (100x)

n: grain size number

$$\%\ Pearlite\ =\ black\ square\ /\ overall\ square$$

$$\%\ Ferrite\ =\ 100\ -\ \%Pearlite$$

$$\%Ferrite\ =\ (0.76 - X)/\ (0.76 - 0.02)$$

X: Carbon percentage

Apparatus and Materials:

- Polisher-Grinder machine
- Sand papers with grit numbers (320, 400, 500, 600, and 1200).
- Drier
- Optical Microscope attached with PC or monitor
- Ethanol
- Etching reagents
- Polishing (rubbing) compound
- Steel and Aluminum alloy specimens

Procedure:

1) Grind one surface of the given specimen using 320 grit sand paper attached to the grinding wheel with the continuous flow of water.
2) Continue the grinding process using sand papers of higher grit numbers.
3) Polish the specimen using the polishing cloth attached to the polishing wheel by using the rubbing compound until the surface becomes shiny as a mirror.
4) Wash the specimen with water.
5) Clean the specimen's polished surface with boiled ethanol at 70°C and dry it.
6) Use the proper etchant to etch the polished surface of the specimen until the surface becomes dull. Choose the suitable etchant from Table (M1.1).
7) Dry the specimen and use the microscope to observe the internal structure at different magnifications.
8) Save the best micrograph you have chosen.
9) The technician will provide you with a printout copy of the micrograph

Table (M1.1): Etchant solutions for different metals and alloys

Material	Composition of etchant	Etching procedure
Iron & steel	1-5 Parts Nitric Acid 100 Parts Alcohol	Immerse or Swab
Copper & brass	1 Part Ammonium Hydroxide 1 Part 3% Hydrogen Peroxide 1 Part Water	Swab
	5 g Ferric Chloride, 10 ml Hydrochloric Acid 100 ml Water	Immerse
Aluminum	5-10 g Ammonium Persulphate 1 ml Hydrofluoric Acid 99 ml Water	Immerse
	10 g Sodium Hydroxide, 100 ml Water	Immerse
Stainless steel	10 g Oxalic Acid 100 ml Water	Use Electrolytically
	5 ml Sulfuric Acid 100 ml Water	Use Electrolytically

Results and Calculations:

1) Record the magnification of the microscope used in producing the micrographs
2) Measure the grain size of each specimen by using the intercept method.
3) Find the ASTM grain size number
4) Find the approximate carbon content of the steel specimen

Discussion and Questions:

1) Comment on the resulted surface of both specimens (steel and aluminum alloy)
2) What do we mean by the grit number of sand papers (i.e. 320)
3) Why low grit number sand paper must be used before higher one?
4) What is the role of etchant? How?
5) Discuss the effect of grain size and shape on the mechanical properties of the specimen material. Also, explain how smaller grain size gives higher strength
6) What are the phases observed for the steel specimen?
7) Draw the development of microstructure for steel specimen when it is cooled from austenitic region to room temperature
8) What is the difference between eutectoid, hypo-eutectoid and hypereutectoid steel?

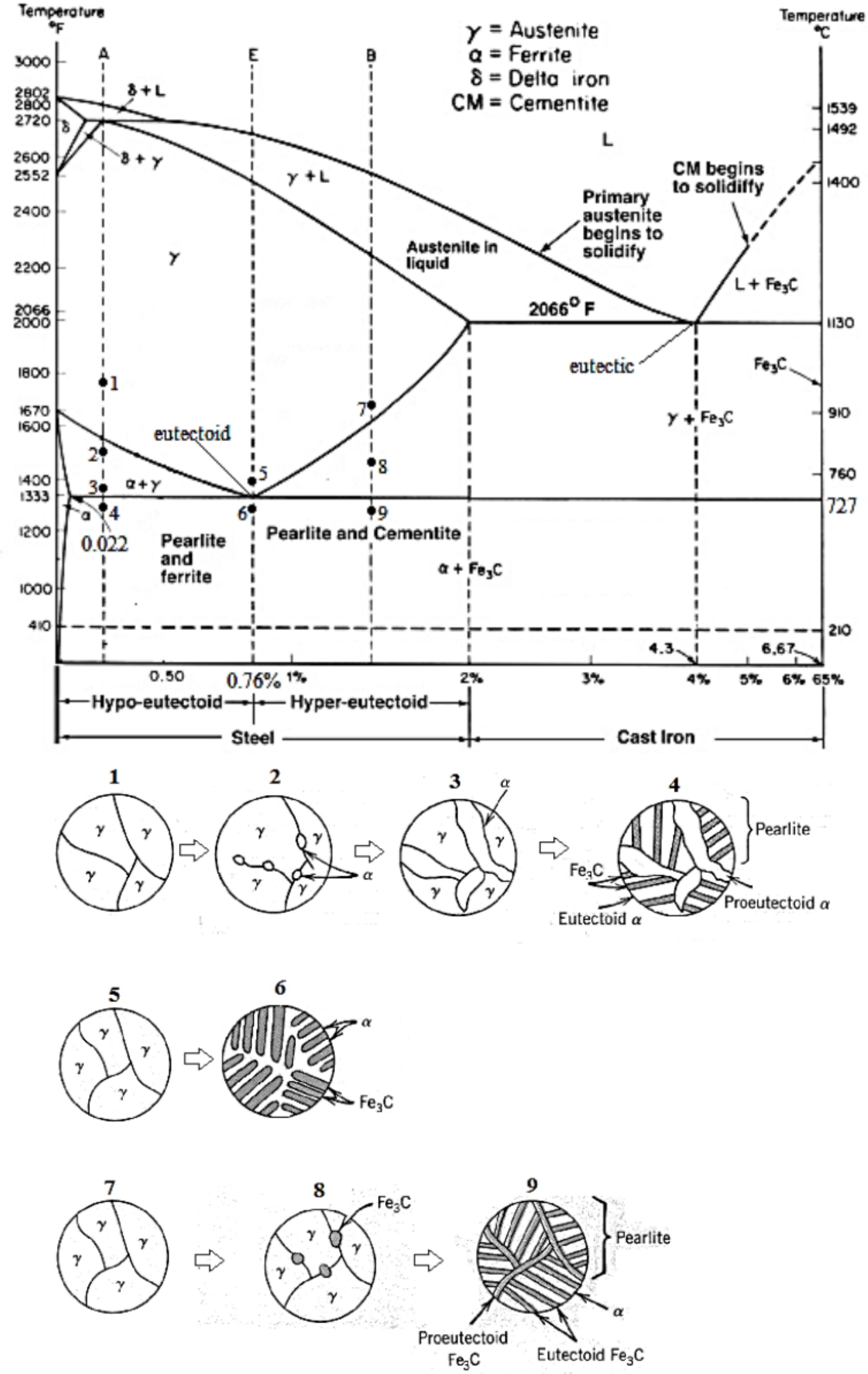

Figure M1.1: Iron-Iron Carbide phase diagram

Experiment M2: Hardness Testing

Objectives:

- Find the Vickers hardness number for steel specimen and aluminum specimen
- Observe the relation between load and hardness number

Apparatus and Materials:

- Micro-hardness tester machine (Vickers hardness)
- Grinded and polished steel specimen

Vickers Hardness Testing:

A diamond pyramid with an apex angle o136° is used to indent the surface of the test maerial under an indentation force (P). The average diagonal length of the impression (d) is measured and the Vickers hardness number is calculated: $VHN = \frac{2P*sin(\frac{136}{2})}{d^2} = 1.854\frac{P}{d^2}\ \ kgf/mm^2$

Where the value of d is calculated as follows:

$$d = \frac{d_1 + d_2}{8}$$

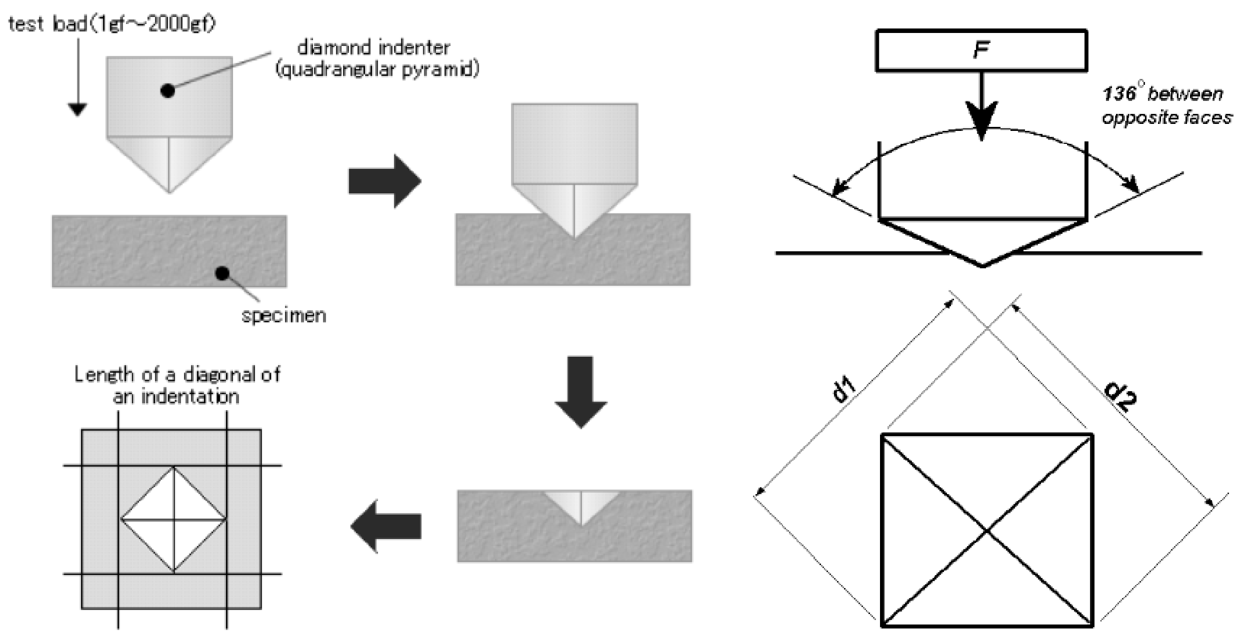

Figure M2.1: Vickers hardness test

Procedure:

1) Specimens tested by micro-hardness machine must be grinded and polished.
2) Place the specimen on the stage where the tested surface perpendicular to the indenter.
3) Turn on the tester.
4) Rotate the objective indenter turret to the scanning objective (40× or 10×).
5) The specimen can be brought into focus by the stage-elevating handle located on the right side of the tester.
6) Set the dwell time at 10 to 15 seconds.
7) Select the weight for the load application using the Dial-A-weight selector knob on the upper right side of the tester.
8) Rotate the objective-indenter turret to the indenter.
9) Press start and wait for the indentation to be completed.
10) Rotate the objective-indenter turret to the scanning objective.
11) Advance the left filar line so that the left inner edge of the line just touches the left most point of the impression.
12) Advance the right filar line so that the right inner edge of the line just touches the right most point of the impression.
13) Record the value of the filar micrometer.
14) Repeat steps 11 to 13 with the eyepiece rotated 90 degrees to measure the height of the impression.
15) Average the two readings if different.
16) Repeat steps 8 to 15 for different loads.
17) This procedure will be done for steel and aluminum specimen.

Observations:

Complete Table M2.1 with the required data and information

Table M2.1: Vickers hardness testing data

Specimen's material:				
Total magnification:				
Load (kgf)	**d_1 (μm)**	**d_2 (μm)**	**$d_{average}$ (μm)**	**VNH (kgf/mm^2)**
50				
100				
200				
500				
1000				

Results and Calculations:

1) Calculate the Vickers hardness number at each load for both steel and aluminum specimens.
2) Find the average Vickers hardness number.
3) Plot the relation between VHN and the corresponding load.

Discussion and Questions:

1) Comment on your results
2) What are the main differences between Brinell and Vickers hardness tests?
3) What do you conclude from the values of average VHN for aluminum specimen?
4) What are the other hardness tests? How hardness number can be converted from one scale to another?

Experiment M3: Impact Test

Objectives:

- Measuring the resistance of material to impact loading.
- Determining the material's toughness (Impact Energy).
- Determining whether the material is ductile or brittle.

Apparatus and Materials:

The apparatus used in this experiment is the Brook's Pendulum Tester (Figure M3.1).

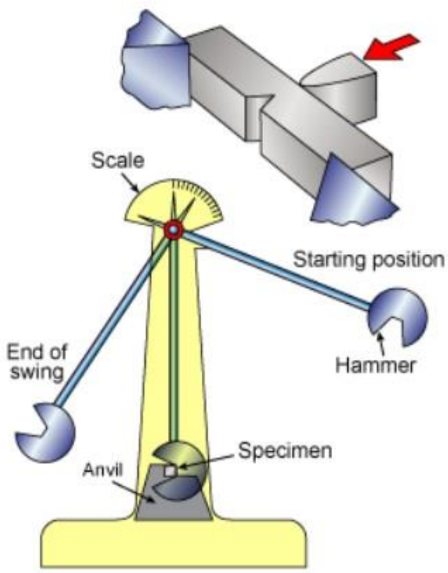

Figure M3.1: The Brook's Pendulum Tester

The test specimens are steel and aluminum alloys with the dimensions shown in Figure M3.2:

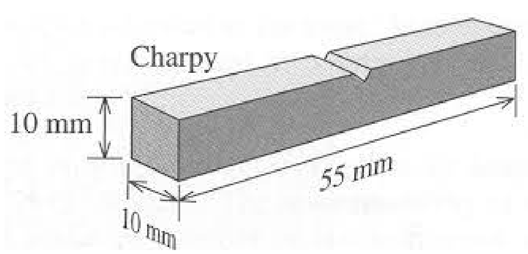

Figure M3.2: Impact test specimen

Procedure:

1. Record the dimensions of the specimen that are needed.
2. Insert the specimen in the specimen block such that the striker will strike the specimen at the back of the notch.
3. While the pendulum is at its elevated position, adjust the indicator to the zero angle position.
4. Release the pendulum by releasing simultaneously the safety knob and the release knob.
5. Read the energy absorbed in rupturing the specimen directly from the dial scale which is calibrated in Joules.

Results and Calculations:

Calculate the resistance to the impact which is defined as:

$$a_k = \frac{A_k}{b.h_k}$$

Where : A_k is the absorbed striking energy of the notched specimen in J
b is the width of the specimen in mm
h_k is the thickness of the specimen at the center of the notch in mm

Discussion and Questions:

1. Comment on your results
2. What is the primary difference between Charpy and Izod impact tests?
3. Discuss the effect of temperature on impact resistance
4. What is the relationship between the impact strength and microstructure of the test material?

Experiment M4: Heat Treatment: (Quenching, Normalizing and Annealing)

Objectives:

- To investigate the conventional heat treatment procedures, such as quenching, normalizing and annealing, used to alter the properties of steels.
- To study the effects of heat treatment on the microstructure and mechanical properties of steels; Vickers hardness will be measured for heat-treated specimens.

Apparatus and Materials:

- High temperature furnace
- Vickers micro-hardness testing apparatus
- Optical microscope attached with PC.
- Four plain carbon steel specimens

Procedure:

1) Determine Vickers hardness number of the samples.
2) Heat the steel samples in furnace up to 800 °C (25 – 50 °C above the upper critical line on the iron-iron carbide phase diagram).
3) Quench one specimen rapidly in water (Quenching).
4) Quench another specimen in oil.
5) Leave one specimen in still air to cool relatively slowly (Normalizing).
6) Leave the other specimen in furnace after turning the furnace off to cool slowly (Annealing).
7) Measure the hardness of the specimens at 1000 *gf*
8) Observe the microstructure of the specimens under the microscope at an appropriate magnification.

Observations:

Complete Table M4.1 with required data and information

Table M4.1: Heat treatment experiment's results

Total magnification of scanning objective lens				
Total magnification of microscope				
Cooling Media	**d_1 (μm)**	**d_2 (μm)**	**$d_{average}$ (μm)**	**VNH (kgf/mm^2)**
Original Specimen				
Water				
Oil				
Air				
Furnace				

Results and Calculations:

1) Calculate the Vickers hardness number for the test specimens.
2) Draw the hardness number versus heat treatment method.
3) Attach the micrograph of the test specimens.

Discussion and Questions:

1) Comment on your results
2) What microstructure is expected to result in each sample when it is cooled to room temperature using different cooling rates
3) What factors affect the cooling rate?
4) How can water quenched specimen be softened?

Experiment M5: Precipitation Hardening (Age Hardening)

Objectives:

- To enhance the strength and hardness of non-ferrous alloys (aluminum alloy) by means of precipitation hardening/age hardening where fine precipitates formed and block the motion of dislocations.

Apparatus and Materials:

- High temperature furnace
- Vickers hardness testing machine
- Optical microscope
- Commercial aluminum specimens

Procedure:

1) **Solution treatment:** Insert the specimens into the furnace and heat above a solvus line (550°C) and leave for 30 minutes until a homogeneous α phase is produced.
2) **Quenching**: cool the heated specimen in water to produce a supersaturated solid solution.
3) **Aging**: heat cooled specimens to temperature below the solvus line about 180°C.
4) Take specimens out the furnace one by one in 10-minute increment between every two successive specimens
5) Cool each specimen separately in water or in air.
6) Test each specimen by Vickers testing machine (the specimen needs grinding and polishing)
7) Observe the microstructure of each specimen under the microscope (specimen needs etching).

Steps from 1 to 3 are summarized in Figure M5.1

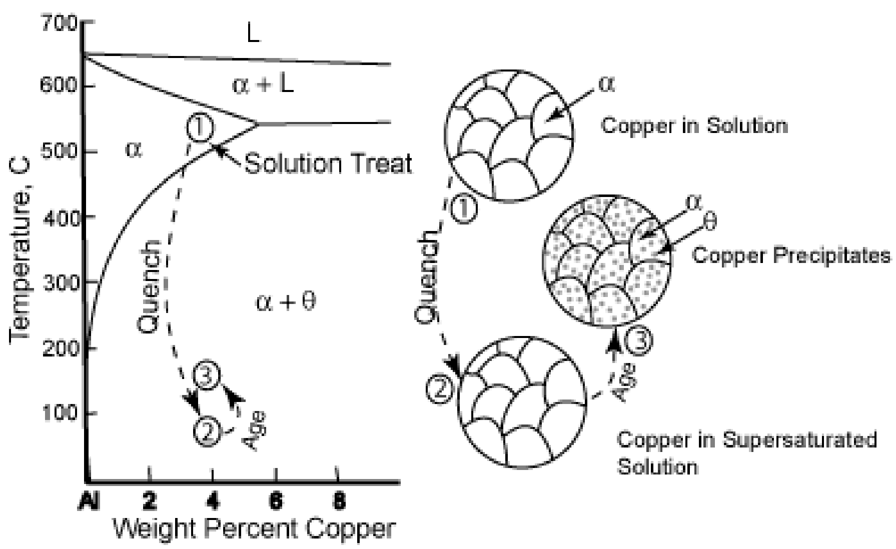

Figure M5.1: Precipitation hardening steps (solution treatment, quenching and aging)

Observations:

Complete Table M5.1 with the required data and information

Table M5.1: Precipitation hardening experiment results

Total magnification of scanning objective lens				
Total magnification of the microscope				
Time (min)	**d_1 (μm)**	**d_2 (μm)**	**$d_{average}$ (μm)**	**VNH (kgf/mm^2)**
0				
10				
20				
30				
40				
50				
60				
70				
80				
90				
100				

Results and Calculations:

1) Calculate Vickers hardness for each specimen
2) Plot hardness number versus aging time
3) Plot hardness number versus logarithmic aging time
4) Attach the micrograph of each specimen

Discussion and Questions:

1) Comment on your results
2) Briefly describe the three steps involved in the Precipitation Hardening process
3) What are the main differences between artificial and natural aging?
4) Which composition of non-ferrous alloys can be age hardened?

Experiment M6: Corrosion Test

Objectives:

- Study the effect of acid concentration on corrosion rate of a certain metallic alloy.
- Study the effect of fixed acid concentration on corrosion rate of different metallic alloys.
- Study the effect of different chemical solutions (corrosive media) on the corrosion rate of certain metallic alloy.

Apparatus and materials:

- Graduated cylinder (100 ml, 250 ml).
- Beakers (600 ml).
- Accurate balance
- Caliper
- Drier
- Sand papers
- Sulfuric acid of different concentrations.
- Different chemical solutions have the same concentration
- Specimens of different materials (Stainless Steel, Steel, Galvanized steel Brass, and Aluminum).

Procedure:

1) Each specimen will be cleaned with sand papers.
2) Dimensions (length, width, and thickness) and initial weight of each specimen must be measured.
3) Specimens of the same materials are immersed into a solution of (H_2SO_4 of different concentrations) for the same period of 30 minutes.
4) Specimens of different metals were immersed into a solution of (H_2SO_4 of the same concentration) for the same period.
5) Specimens of the same metals were immersed in different environments of the same concentrations for the same period.
6) All the specimens were removed at the same time, cleaned by using water and then dried.
7) The specimens were weighted again using a sensitive balance, and the weight was recorded (final weight).

Results and Calculations:

Corrosion penetration rate (CPR) is calculated as follows:

$$CPR = \frac{w}{\rho A t} \left(\frac{length}{time}\right)$$

Where: *w*: weight loss, *ρ*: metal density, *A*: surface area exposed to corrosive media, and *t*: time of exposure

1) Complete the tables (Table M6.1, Table M6.2, Table M6.3 and Table M6.4) with required data and calculate corrosion penetration rate in *mm/year*.
2) Draw corrosion rate versus acid concentration, corrosive media and metal type

Discussion and Questions:

1) Comment on your results
2) Define corrosion
3) What are the main methods of corrosion control?
4) What factors do influence corrosion rate?

Table M6.1: The effect of acid solution on different metals

Type of metals	***Steel***	***Aluminum***	***Stainless Steel***	***Galvanized Steel***	***Brass***
Symbol	A1	A2	A3	A4	A5
Density, ρ (gm/cm³)					
Exposure time, t (hr)	0.5	0.5	0.5	0.5	0.5
Corrosive media	H_2SO_4	H_2SO_4	H_2SO_4	H_2SO_4	H_2SO_4
Corrosive concentration (%)	10	10	10	10	10
Length (cm)					
Width (cm)					
Thickness (cm)					
Surface area (cm²)					
Initial weight (gm)					
Final weight (gm)					
Weight losses (gm)					
CPR (mm/year)					

Table M6.2: The effect of corrosive medium on steel corrosion rate

Type of metals	**Steel**	**Steel**	**Steel**	**Steel**
Symbol	B1	B2	B3	B4
Density, ρ (gm/cm^3)				
Exposure time, t (hr)	0.5	0.5	0.5	0.5
Corrosive media	H_2SO_4	HNO_3	HCl	NaOH
Corrosive concentration (%)	10	10	10	10
Length (cm)				
Width (cm)				
Thickness (cm)				
Surface area (cm^2)				
Initial weight (gm)				
Final weight (gm)				
Weight losses (gm)				
CPR (mm/year)				

Table M6.3: The effect of acid concentration on steel corrosion rate

Type of metals	**Steel**	**Steel**	**Steel**	**Steel**	**Steel**	**Steel**
Symbol	C1	C2	C3	C4	C5	C6
Density, ρ (gm/cm^3)						
Exposure time, t (hr)	0.75	0.75	0.75	0.75	0.75	0.75
Corrosive media	H_2SO_4	H_2SO_4	H_2SO_4	H_2SO_4	H_2SO_4	H_2SO_4
Corrosive concentration (%)	10	20	30	40	50	97
Length (cm)						
Width (cm)						
Thickness (cm)						
Surface area (cm^2)						
Initial weight (gm)						
Final weight (gm)						
Weight losses (gm)						
CPR (mm/year)						

Table M6.4: The effect of exposure time on steel corrosion rate

Type of metals	**Steel**	**Steel**	**Steel**	**Steel**	**Steel**
Symbol	D1	D2	D3	D4	D5
Density, ρ (gm/cm^3)					
Exposure time, t (hr)	0.25	0.417	0.583	0.75	1
Corrosive media	H_2SO_4	H_2SO_4	H_2SO_4	H_2SO_4	H_2SO_4
Corrosive concentration (%)	10	10	10	10	10
Length (cm)					
Width (cm)					
Thickness (cm)					
Surface area (cm^2)					
Initial weight (gm)					
Final weight (gm)					
Weight losses (gm)					
CPR (mm/year)					