YILDIZ TECHNICAL UNIVERSITY

METALLURGICAL AND MATERIALS ENGINEERING DEPARTMENT

MSE4121 PRODUCTION LABORATORY

EXPERIMENTAL BOOKLET

2025-2026

FALL

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IMPORTANT INFORMATION

1. There will be 10 experiments during the term.
2. Only in case of not being able to participate in the experiment for a week, it will be possible to participate in the make-up experiment. A student who does not attend the trial for more than two weeks will be considered absent.
3. There will be no experiment during the midterm week.
4. Experiments not entered will be evaluated as **zero**.
5. Different experiments will be carried out for designated groups every week, and all students are required to participate in group experiments with their student numbers in the laboratory program shared on the department website.
6. Experiments will be carried out face to face and in the laboratory where each experimental subject is relevant.
7. There will be **a short exam (Quiz)** before the experiment. For this reason, you are expected to look at the purpose of the experiment, test process, equipment and chemicals to be used in the experiment in your experimental booklet.
8. Students who **cannot get a score of 50 or above from the quiz before the experiment will not be allowed into the experiment and will be marked as absent.** For this reason, before coming to the experiment, you are expected to have information about the experiment by looking at the relevant experiment sheet.
9. After the experiment, you must write **an experiment report in the new format** specified on the subject observed, discussed and learned during the experiment. Your test report format should be as published on the website. The experiment report must be delivered to the instructor to whom the experiment relates on time.
10. The report of the experiments will be prepared individually and according to the format shared with you. Reports that are not prepared in accordance with the format will not be evaluated. Similar reports will be evaluated as **"ZERO".**
11. You must participate in the experiments. Your participation in class and experiments will determine your performance.
12. You must bring **your own laboratory coat, latex gloves, laboratory glasses and mask** when coming to the experiments. You must have laboratory safety equipment with you to participate in the experiment.

Students who do not have a laboratory coat will not be allowed into the experiment and will be marked absent.

EXPERIMENT NAME: PRECIPITATION HARDENING

1. PURPOSE OF THE EXPERIMENT

The primary purpose of the precipitation hardening experiment is to examine the effect of the applied process on the mechanical properties (strength, hardness, etc.) of materials that can respond to heat treatment (such as 2xxx, 6xxx, and 7xxx series aluminum alloys).

Improving the mechanical properties of the material with thermal methods without changing the chemical composition

2. THEORETICAL INFORMATION

The mechanical properties of materials are largely dependent on their internal structure. Since the internal structure is related to the chemical composition and the mechanical or heat treatments applied to the material, it can be said that the mechanical properties of the materials also depend on these factors. In this context, strength is one of the most important mechanical properties of a material and is expressed as "resistance to plastic deformation". Plastic deformation of metals is mainly caused by the progression of linear defects called dislocations in the crystal. Therefore, mechanical properties such as strength, hardness, and ductility are explained by both the density of the dislocations in the internal structures of metals and their interactions with other components and defects. Any factor that will complicate or prevent the movement of dislocations in the internal structure of metals will lead to an increase in the strength of the material. Conversely, any factor that will facilitate dislocation movements will enable plastic deformation to occur more easily.

There are various methods used to strengthen metallic materials. The main ones can be classified as; Strain hardening, Alloying, Grain size reduction, Martensitic transformation hardening, Precipitation hardening, etc. With the development of technology and due to its technical features, aluminum, which is one of the youngest members of the global metal world, is widely used in many areas of the industry. In practice, aluminum, which has a very high strength-to-weight ratio and is quite light compared to steel; has become especially attractive for industries such as automotive, and aerospace industries, since its mechanical properties can be increased to a level comparable to steel when it is doped by the addition of alloying elements [1].

Heat treatment generally includes heating and cooling processes applied to metallic materials to change their mechanical properties. In this context, precipitation hardening/aging heat treatment can be applied to suitable aluminum alloys. Aging can only occur in alloys with a solvus curve in the equilibrium diagram, and solid solution compositions are limited only by the solvus curve. Elements such as Cu, Zn, Mg, and Si added into Al alloys make this alloy applicable for precipitation hardening with the intermetallic structures they form in the primary phase [2]. Therefore, precipitation hardening heat treatment can be applied to 2xxx (Al-Cu), 6xxx (Al-Mg-Si), and 7xxx (Al-Zn-Mg) series aluminum alloys. The phase diagram of the 2xxx series Al alloy, which can be applied to precipitation hardening heat treatment, is given in Figure 1. Thanks to the precipitation hardening process, it is possible to develop light and high strength-to-weight Al alloys suitable for use in the aerospace industry.

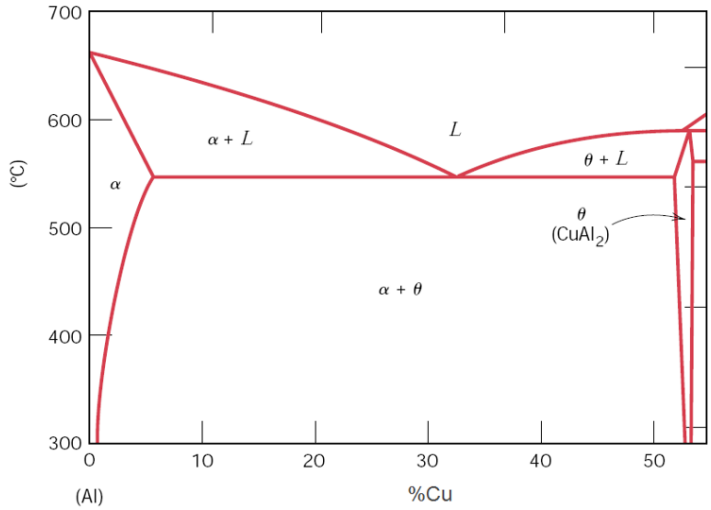
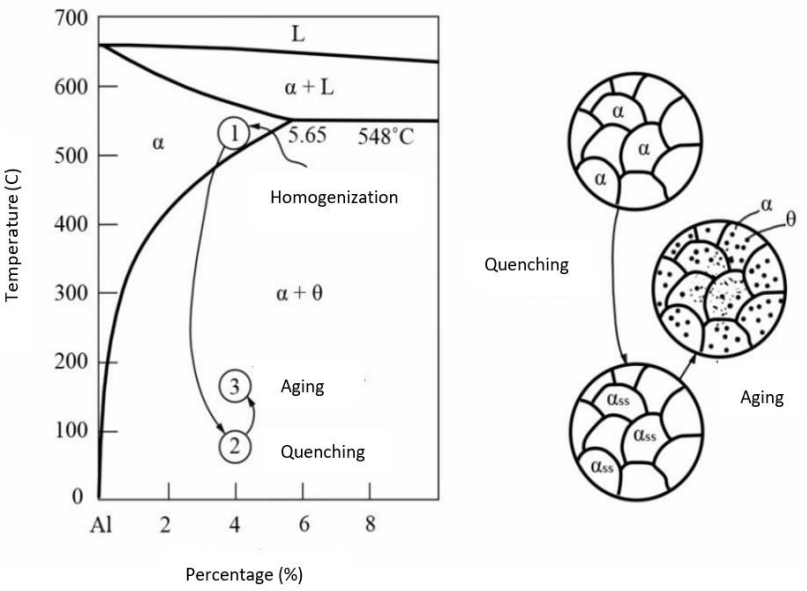


Figure 1. Phase diagram of 2xxx series Al alloy that can be precipitation hardening heat treatable

The first precipitation hardening application for an increase in strength and hardness of Al alloys was examined by Alfred Wilm in the early 20th century (1906). Alfred Wilm, in his studies on aluminum alloys, observed that duraluminum gained hardness as a result of sudden cooling from high temperature (quenching) and waiting at room temperature, just like steel. The increase in hardness of the aluminum alloy known as duraluminium (Al-4.4%Cu-1.5% Mg-0.5%Mn) as a result of quenching and waiting at room temperature is called natural aging. Waiting for a certain period of time at a temperature between 100°C-300°C after quenching is called artificial aging. It has been revealed in later studies that the aging time is shortened by increasing the aging temperature. However, if the aging temperature is too high or the aging time is too long, in this case, the so-called over-aging phenomena occur and a decrease in material strength is observed again [3]. On the other hand, the type, distribution, amount, average diameter, and number of the precipitated second phases affect the strength of the material.

The precipitation hardening heat treatment is conducted in three stages:



1. Solution Treating: High-temperature annealing (Homogenization process) in order to homogenize the alloy

2. Quenching: Obtaining supersaturated structure with sudden cooling in a water environment (α_{ss})

3. Aging (Natural/Artificial): Aging for a certain period of time at a temperature below the homogenization temperature (100-300°C)

Figure 2. Precipitation hardening process steps and the changes in microstructure [4]

The precipitation hardening process steps and changes in the microstructure are given in Figure 2 [4].

3. TOOLS, DEVICES and MATERIALS

- Samples of 6xxx series aluminum alloy,
- Oven that can reach an elevated temperature for solution treating,
- Sudden cooling (Quenching) environment,
- An oven that can be heated to 300ºC for artificial aging,
- Brinell hardness device.

4. EXPERIMENTAL STUDIES

As the test sample, 6xxx series Al alloy with a thickness of at least two mm and containing Al-Mg-Si was preferred. Within the scope of the experiment, four samples are used to determine the effect of aging time on the hardness of the material.

In the first stage of the experiment, all samples are placed in the oven at 530-550ºC for approximately 30 minutes to apply "Homogenization annealing". After the period specified, as the second stage, the samples are taken out of the furnace as quickly as possible and instantly cooled in a water environment to ensure “sudden cooling”.

One of the samples is separated so that the hardness can be measured without applying artificial aging, and the other three samples are subjected to artificial aging for the specified periods in an oven at approximately 200ºC. For measuring the hardness, the samples are taken out of the oven at the end of the applied aging period and cooled rapidly. Hardness measurement should be done quickly, as the time spent outside the oven will affect the result. Hardness measurements are carried out by using a Brinell hardness measuring device.

5. RESULTS and DISCUSSION

The formula to be used in the determination of the Brinell hardness values is given in Equation 1.

$$HB = \frac{2xF}{\pi \times D \times (D - \sqrt{D^2 - d^2})} \quad (1)$$

F: Force (N,kg), D: Ball diameter (mm), d: Mark diameter (mm)

In order to make comparisons by using the data obtained as a result of the experiment, a hardness-aging time graph will be created.

6. INFORMATION REQUIRED IN THE EXPERIMENT REPORT

- **Cover Page:** In accordance with the format; Emblem, Course Information, Supervisor of the Experiment, Student Name-Surname/Student Number, and Group Number
- **Theoretical Information:** An original summary of the knowledge conveyed on the relevant subject during the experiment, prepared by the student
- **Experimental Studies:** Materials and equipment used in the experiment; The procedure steps explained in accordance with the application conditions and the order of the experiment (with cause and results); Brinell hardness calculations (with units)
- **Evaluation:** Drawing a Brinell Hardness-Time graph by using Excel graphics, researching the data in the existing literature, comparing it with the results obtained within the scope of the experiment, and interpretation by the student.

7. REFERENCES

[1] GÜVEN, Ş. ve DELİKANLI, Y., (2012). "AA 2024 Alüminyum Alaşımında Çökelme Sertleşmesinin Mekanik Özelliklere Etkisi", Teknik Bilimler Dergisi, 2: 13-20.

[2] YAMAN, M.B. KOCAMAN, E. ve Barış, A., "Al7075 Alaşımına İlave Edilen Al-5Ti-1B Tane İncelticinin Yaşlanma, Mikroyapı, Sertlik ve Korozif Özellikleri Üzerindeki Etkisi", Gazi University Journal of Science Part C: Design and Technology, 10: 870-883.

[3] YAŞAR, A.C. ESER, A.A. ÖZCAN, A. ve ACARER, M., "ALÜMİNYUM DÖVME VE EKSTRÜZYON ALAŞIMLARINDA AŞIRI YAŞLANDIRMANIN MİKROYAPI ve MEKANİK ÖZELLİKLERE ETKİSİ".

[4] AL-SAADI, H.I.A. ve TUNAY, R.F., (2017). "Suni Yaşlandirma İşleminin Alüminyum Alaşiminin Sertliği Üzerine Etkisi", Mühendislik Bilimleri ve Tasarım Dergisi, 5: 525-532.

EXPERIMENT NAME:CERAMIC PRODUCTION VIA CASTING SLURRY INTO PLASTER MOLD

1. Aim of the Experiment

The objective of this experiment is to examine the fundamentals of ceramic production and the preparation of a ceramic slurry, as well as to learn how to shape the prepared ceramic slurry by slip casting into a plaster mold. The preparation of raw materials, grinding kinetics, the rheological behavior of the ceramic slurry, and its formability will be examined in detail.

2. Theoretical Part

2.1. Preparation and Rheology of Ceramic Slurry

A ceramic slurry contains ceramic raw materials, water, and additives such as binders and deflocculants. To prepare an optimal composition, special tables known as Seger Tables or Seger Ratios are used. These Seger tables show the ratios of oxides present in ceramic raw materials. When developing new compositions, it is essential to keep the ratios constant, as each oxide has a different effect on rheology. Seger tables vary according to the final product and the desired properties of the final product.

Rheology is the science of fluid flow. Fluidity can be described using two main values: viscosity and thixotropy. Viscosity is the value of a material's fluidity. Fluidity and viscosity have an inverse relationship. Thixotropy refers to the change in viscosity over time.

There are two main groups of fluids: Newtonian and non-Newtonian fluids. Newtonian fluids, like water, have constant viscosity over time. However, for non-Newtonian fluids, viscosity changes over time. The viscosity of non-Newtonian fluids decreases with the time of measurement.

Ceramic slurry is a non-Newtonian fluid and exhibits thixotropic properties. In studying the rheology of ceramic slurry, terms such as flocculation, deflocculation, and deflocculants should be explained.

For ceramics, if the viscosity is too low, the surface quality of the products will be poor, and cracks may form on the surface. If the viscosity is too high, it can lead to pinhole defects and difficulties in transporting (mobility) the slurry.

Thixotropy is as important as viscosity. If the thixotropy is too high, the drying time of the product will be extended. If thixotropy is too low, it can make the product brittle.

2.1.1. Flocculation, Deflocculation, Deflocculant

The particles of a clay material suspended in water exhibit behavior in two completely different mechanisms. This is caused by the electrostatic charges on the particle surfaces, which induce both attraction and repulsion. Regularly, in an acidic environment, the particles attract each other, a

phenomenon known as "flocculation." In an alkaline environment, the particles repel each other, which is referred to as "deflocculation."

In a deflocculated state, the surface charges of the particles are neutralized, allowing these particles to remain as single, separate units in suspension. Without charges and attraction, there is no force holding the particles together. Therefore, in a deflocculated state, there is a decrease in viscosity. In a flocculated state, three-dimensional structures are formed due to the electrostatic attraction between the particles, leading to an increase in viscosity.

Defloculants

The term deflocculant refers to an additive that, when added, causes a decrease in viscosity. Deflocculants increase the zeta potential between particles, in other words, they enhance the repulsive forces between particles, preventing flocculation.

There are various mechanisms through which deflocculants operate in suspension. These include:

- Raising the pH to basic values through the addition of a base or hydrolysis.
- Replacing flocculant cations with alkali cations in double-layer clays.
- Adsorbing anions under an electric field to achieve a negative charge on the particles.
- Adding a protective colloid.
- Eliminating flocculating ions through precipitation or the formation of coordination complexes.

Typically, the effects of deflocculants occur through the mechanisms mentioned above. These mechanisms do not depend on whether the deflocculant is organic or inorganic in nature.

A good casting slurry should have the following properties:

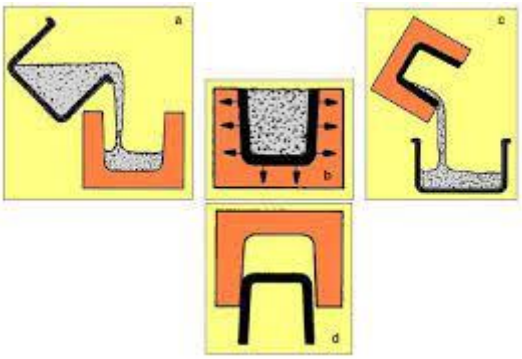
- It should have low viscosity for easy spreading within the plaster mold.
- Solids should not settle.
- It should be easily removable from the mold after casting.

The plaster mold casting method is generally preferred for the production of large-volume and complex-shaped products. This method is used for shaping asymmetric products, certain special firebricks, tableware, and sanitary ware such as sinks, toilets, and bathtubs, as well as in the production of advanced technology ceramics. Plaster mold casting is preferred in the production of sanitary ware due to the large volume and complex shapes of the products.

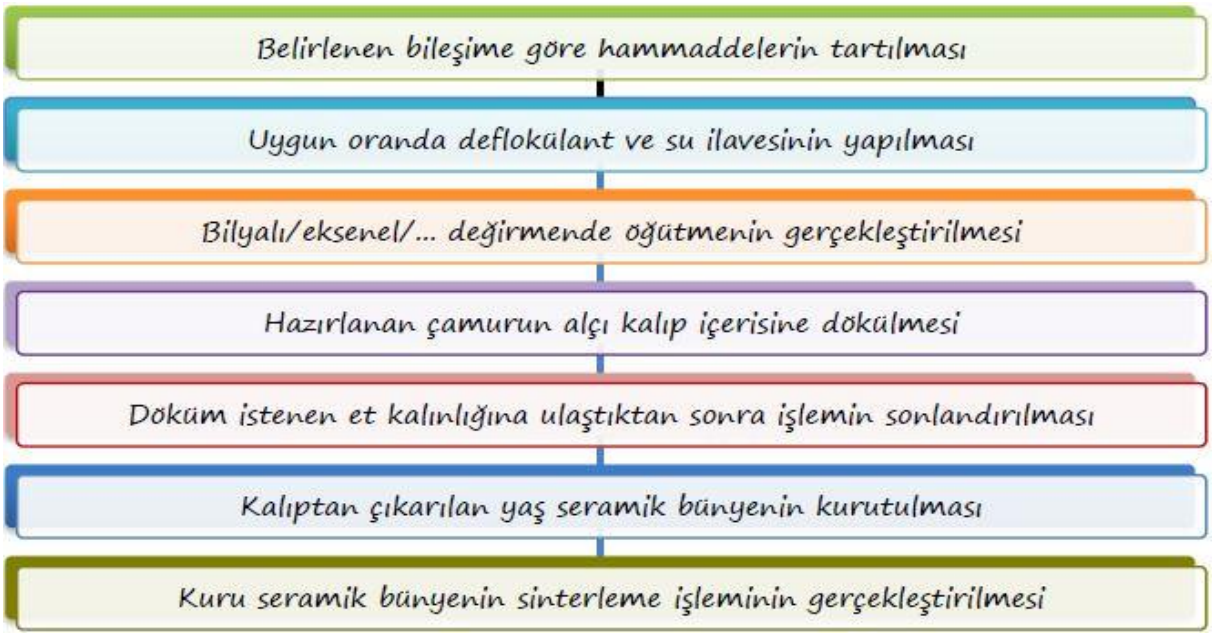
The molds used in casting are porous and water-absorbing plaster molds. Additionally, porous synthetic materials are also used. To ensure healthy production through casting, the plaster mold must first be suitable for the molding technique, meaning it should not stick when opened, be impact-resistant, have

uniform thickness, appropriate porosity, and be sufficiently dried. Furthermore, it is crucial that the mold is clean and that the joining surfaces are smooth when preparing for casting.

Plaster molds absorb water very quickly. The water in the slurry poured into the mold is rapidly drawn out by the mold, starting from the surface of the mold. As the water layer between the particles decreases, the repulsive forces between the particles are overcome by the attractive forces, allowing the particles to come closer together and adhere. Over time, the number of stacked particles increases, forming a solid-liquid transition layer starting from the inner surface of the mold. The thickness of this layer increases over time and is known as the uptake rate. This rate decreases as time goes on.



Şekil 1: Schematic Representation of Shaping by Slip Casting Method



Şekil 2. General Flow Diagram for the Production of Ceramic Products by Casting Method

3. Experiment

3.1. Required Tools

Mill, Viscometer, Mixer, Pycnometer, Sieve (90 μm), Scale, Plaster Mold, Caliper

3.2. Application of the Experiment

First, the raw materials are weighed and mixed according to the composition. Then, the required amount of water and deflocculant is added to the mixture. The prepared slurry is milled in a planetary ball mill. After the milling process, the density of the slurry is determined using a pycnometer. The measurement is taken by weighing the slurry corresponding to its density value. Next, the prepared slurry is mixed with a mixer at a speed of 700 rpm, and the viscosity is measured using an analog viscometer with a rotation speed of 20 rpm. The viscosity should be in the range of 4-6 Poise (the required value for floor tiles). To determine thixotropy, the slurry is allowed to rest for 5 minutes after each viscosity measurement. Then, the viscosity is measured again, and the difference between these two viscosity values provides the thixotropy. When the slurry reaches its final viscosity, the added deflocculant does not affect the viscosity until more deflocculant is added. This high amount of added deflocculant does not behave as expected and increases the viscosity of the slurry. Thus, the proof of the viscosity value achieved is the increase in viscosity observed with the added deflocculant during stability and monitoring.

After the slurry preparation process, slip casting will be performed into the plaster mold. The viscosity value and liter weight of the previously prepared ceramic slurry will be measured. These analyses will allow for the rheological analysis of the slurry and determine its suitability for casting. Subsequently, the plaster molds will be cleaned to be ready for casting, and the ceramic slurry will be poured into the mold. At this point, to ensure the experiment proceeds efficiently and allow for comparisons, the relevant experimental group will be divided into pairs or groups of three to allow the ceramic slurry to rest in the plaster mold for different durations. It is expected that the ceramic, which rests for different durations, will have different thicknesses when removed from the mold. Therefore, each group will sequentially allow their ceramic molds to rest for 3 minutes, 6 minutes, and 9 minutes, draining excess slurry from the molds. The mold will be placed upside down on the table, and a waiting period will be allowed for the ceramic to easily come out of the mold. Afterward, the ceramic will be carefully removed from the mold, and the relevant product will be allowed to rest for 10 minutes in the open air.

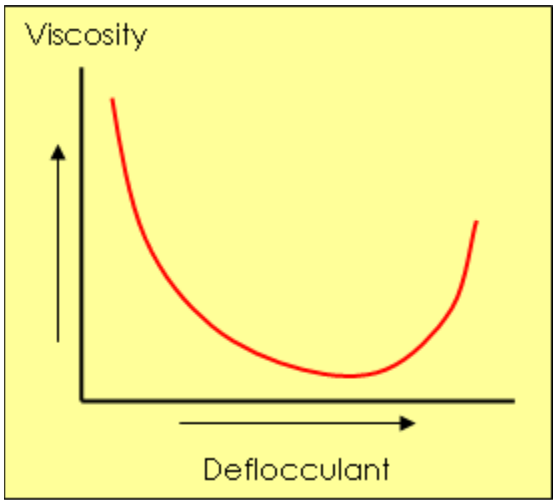


Figure 1. Viscosity - Added Deflocculant Diagram

4. Results

As a result, this experiment explained and demonstrated the preparation of raw materials, grinding kinetics, the rheological behavior of a pourable ceramic slurry, and the shaping of the product using the plaster mold slip casting method.

5.Resources

(1) RAHAMAN M. N., Ceramic Processing, 2006
(2) BARNES H. A., Handbook of Elementary Rheology, 2000
(3) WORRAL W. E., Ceramic Raw Materials, 1982
(4) CICEK B., Ceramic Processing Methods Lecture Notes, 2015
(5) "Seramik", TMMOB Kimya Mühendisleri Odası, 1980, Ankara
(6) Ates Arcasoy, Seramik Teknolojisi, M.Ü. Güzel Sanatlar Fakültesi, Seramik Bölümü Yay.1983

Requirements for the Report:

- Objective of the experiment
- Brief theoretical information about the experiment
- The application of the experiment will be described in order with original language.
- The percentages of materials used in theory and practice will be presented in a table (EXCEL).
- Comments will be made on the results obtained in the experiment, and graphs will be drawn (EXCEL).

EXPERIMENT NAME: 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 multicomponent 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 park with various operations),

3. Sintering and final parts production with secondary operations if necessary.

PART 1.1: APPARENT DENSITY (WET DENSITY)

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

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

1.1.3 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). All conditions related to this test and the conduct of the test are specified in the TS EN ISO 3923-1 standard. 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 / cm^3. This value can be up to 5 g / cm^3 for dusts produced by gas atomization. This value varies between 2.80-3.00 g / cm^3 for copper powders and 3.00-3.25 g / cm^3 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.

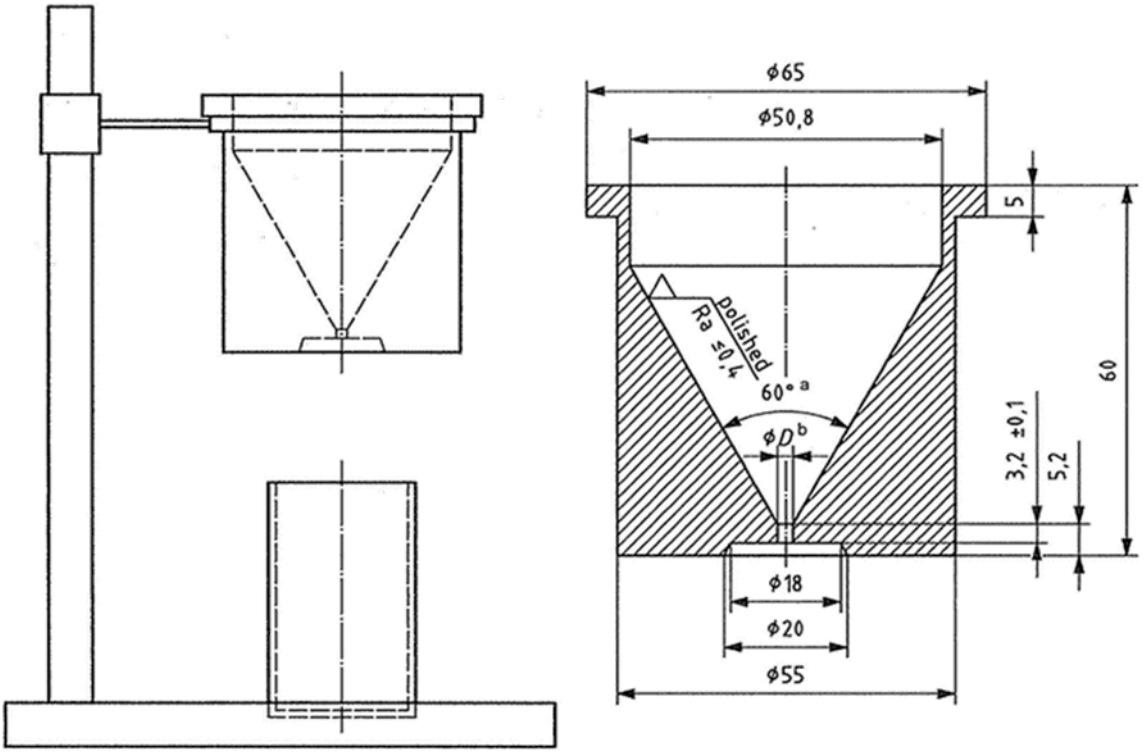


Figure 1.1 Measurement of Masonry Density

PART 1.2: FLUIDITY TEST

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

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

1.2.3 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 (TS EN 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.

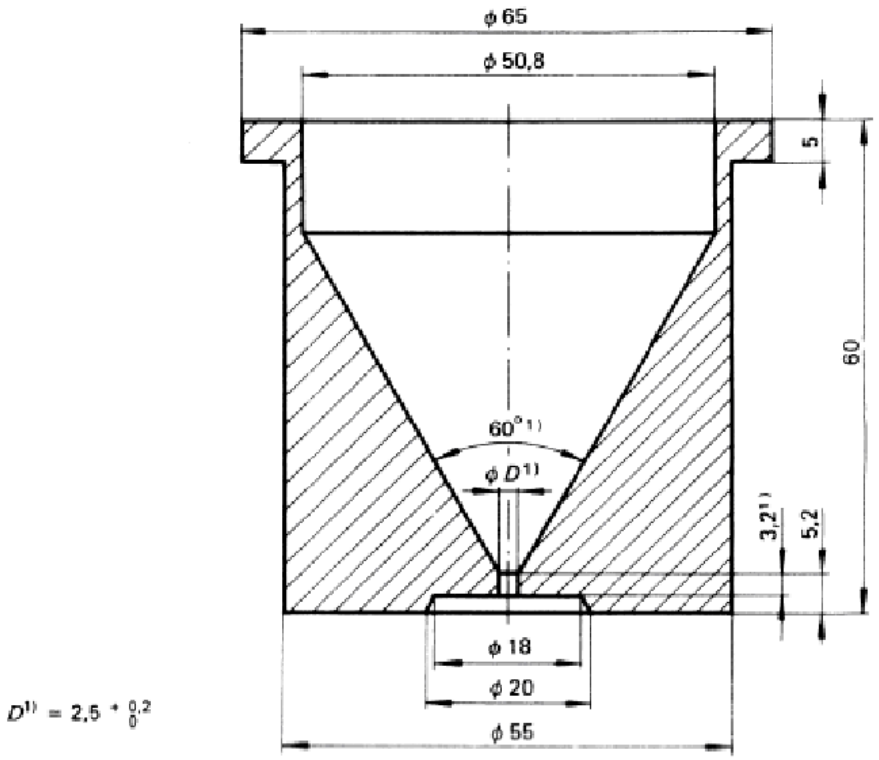


Figure 1.2 An Experimental Funnel for Determining Fluency is Shown Schematically.

PART 1.3: COMPRESSING

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

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

1.3.3 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.

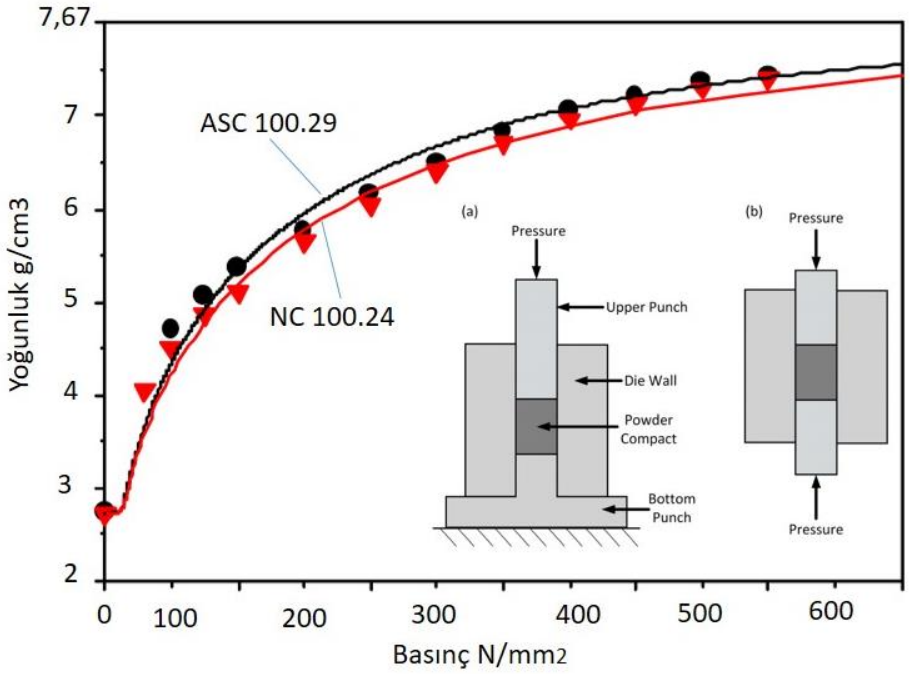


Figure 1.3 Density-pressure relationship for two commercial iron powders

* pressure ↑ , density ↑ , pore ↓

* pore ↑ , density ↓

* pore ↑ , strength ↓

* pore ↑ , lubricant ability ↑

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 1.4: COMPRESSIBILITY (GREEN DENSITY)

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

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

1.4.3. 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/cm^3, Fe: 7.87 g/cm^3: (6.65: 7.87) * 100 = 84.5% = % theoretical density.

- 84,5% Filled

- 15.5% Pore

PART 1.5: SINTERING PROCESSES APPLIED TO METAL POWDERS

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

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

1.5.3 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.

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

Table 1 Sintering temperature and time of some metals and alloys

REFERENCES

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[2] Toz Metalürjisi ve Parçacıklı Malzeme İşlemleri, Randall M. GERMAN, 2007

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EXPERIMENT NAME: The Sol-Gel Technique

1. PURPOSE OF THE EXPERIMENT

This experiment aims to provide professional knowledge about the sol-gel technique and gain hands-on experience in manufacturing materials with the sol-gel technique. Within the scope of this experiment, the sol-gel technique will be introduced. Then ceramic-based nanopowder synthesis and the production of thin-film coatings with different techniques will be carried out using the sol-gel technique.

2. THEORETICAL INFORMATION

The Sol gel method is a wet chemical process used in ceramic production. It was first discovered in the 1800s by Ebelman and Graham. From the 1930s on, Sol-gel was widely studied, and in 1938, the first patent on the sol-gel process in Germany was obtained. The sol-gel method comprises all systems where a suspension can be a gel. The sol-gel method can produce nano-dimensional ceramic powders, thin-film ceramic coatings, ceramic-based materials, and fibers [1-3].

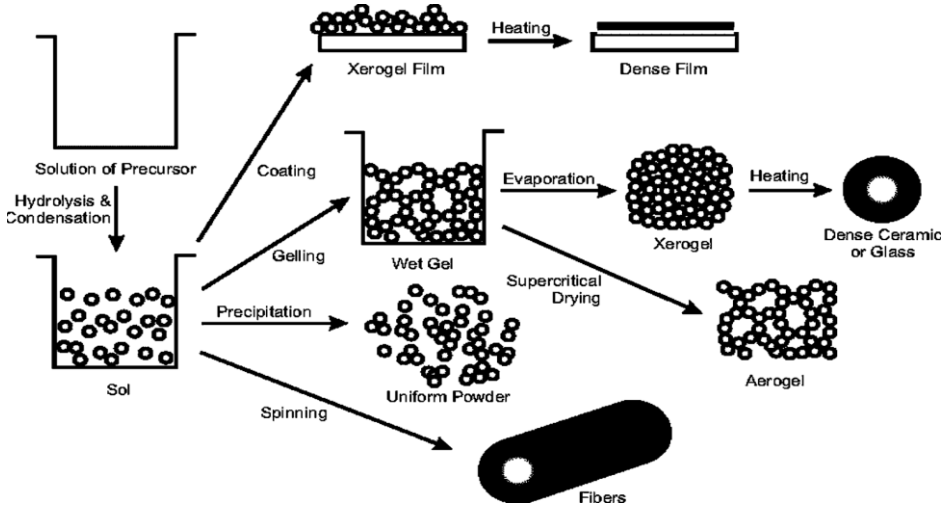


Figure 1. The Sol-Gel technology and products [2].

What is Sol-Gel?

The term sol gel describes the process of agglomeration of nano-sized solid particles (sol) dispersed in a liquid and forming a three-dimensional and continuous network structure (gel) in the liquid [1- 3].

What is sol?

The sol is the structure consisting of the liquid's continuous phase and the solid's dispersed phase. If the nano-sized solid particles are dispersed in the liquid phase and if they do not precipitate immediately, the structure is defined as the sol. It is possible to distribute the solid phase in a homogeneous manner by external forces such as centrifugation.

What is Gel?

The gel is a solid-like and wet structure in which the nanoparticles forming the solid network structure are placed in 3D. The continuous phase in a gel is the solid network formed by the nanoparticles, and the dispersed phase is the liquid phase. Gels have both solid and liquid properties. While their density is close to liquids, certain relationships exist between atoms, such as in solids.

Sol Production:

There are generally two methods of preparing the sol:

- Direct formation of nanoparticles in liquid: Molecules dissolved in the liquid are converted into larger molecules at the end of mixing liquids. The resulting macromolecules then become solid particles in nano-blocks. Example SiO_2 (silica) based nano-sol.
- Nanoparticles (such as carbon nanotubes and quantum points) are generated using specific production methods. The nanoparticles are then dissolved in the liquid phase. Surface modifiers (polymers, soaps, etc.) called surfactants ensure homogeneous distribution.

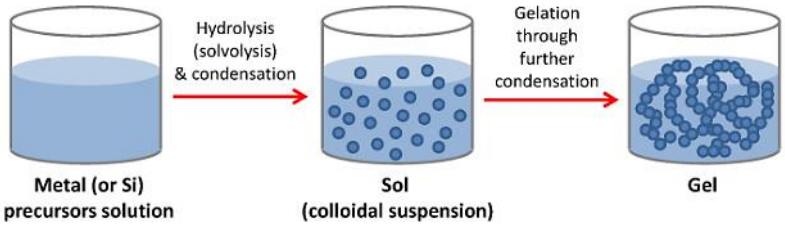


Figure 2. Schematic representation of the sol and the gel states [3].

Sol to Gel Transformation:

The solid nanoparticles dispersed in the solution must form a network structure to convert a solution to a gel. To form the network structure, the solid particles in the solution (called *Brownian Motion* in the liquid) must collide and stick together at the end of the collision. The bonding process is much easier for solid particles with reactive groups on their surface. Because after the collision, reactive groups can form a bond. Since adhesion will not occur for solid particles that do not have reactive groups on their surfaces, the surfaces of such nanoparticles must be reactive with additives or by peeling the surface. As a result, the non-reactive particles are brought together by the bond structure or electrostatic forces that will occur at the end of the collision.

As the solution turns into a gel, the viscosity of the structure increases, and the structure becomes non-flowable at the gel point. At the gelation point, the flow of the liquid has ended because the gel network formed by the particles is dispersed within the entire volume of the liquid. The time taken until the end of gel formation after the gelling agent is mixed into the sol is called the "*gelling time*". [2].

Factors Affecting Sol-Gel Chemistry:

- **pH:** pH is very important in colloid systems involving water. In the formation of silica gels, silanol groups are formed as a result of the hydrolysis of silica. The formation of silanol groups is affected by pH. The silanol groups then form the silica nanoparticles. Silanol groups then lead to the formation of silica nanoparticles and the development of the network.
- **Solvent liquid (solvent):** It is very important that the nanoparticles do not precipitate during the formation of the gel. Therefore, the solvent must be capable of dissolving the nanoparticles. In addition, the solvent also helps the liquid nanoparticles to form the network structure, thereby guaranteeing gelation.
- **Temperature:** The kinetics of the formation of the nanoparticles and the network structure formation is activated by the temperature. When the temperature is too low, the gelling time increases and causes too high agglomerates to over-grow and precipitate without forming the network structure.

- **Heat** Formed by the Reaction: The chemical reactions that occur during the formation of nanoparticles in the sol and the formation of the network structure cause heat release. This heat causes the reactions to accelerate.
- **Time:** Depending on the type of gel produced, gelation steps occur at different times. The product's properties resulting from the slower-formed solution are superior. The slower reaction results in a particularly uniform network in the gelation stage. This allows higher strength and more transparent (transparent) gels (if desired). The more transparent gel structure appears less bluish because it causes less Rayleigh scattering.
- **Catalyst:** In the sol-gel technique, acids (H+) and bases (OH-) are used as catalysts. The sol-gel method is sensitive to pH, as the catalytic effect is achieved through different mechanisms for acids and bases. Although the catalyst material is used in very small amounts (mg/mL), it reduces the gelation time from weeks to minutes.
- **Mixing:** Mixing the sol in the sol-gel technique is important for the chemical reaction to occur uniformly. However, continued mixing after the gelation phase has begun may result in fragmentation of the semi-gelled web at micro and macro levels. Even if the gelation of the entire structure occurs at the end, the gelation time will be prolonged.

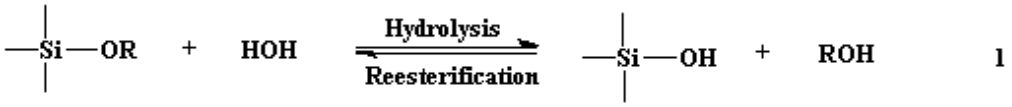
In sol-gel application, inorganic compounds such as metal alkoxide solutions or metal powders, nitrates, hydroxide, oxides, etc., are combined with a certain proportion of water and acid to form a solution. By mixing the solution at certain temperatures, a series of chemical reactions occur in succession in the solution. The electrochemical interactions of the particles form a network, and this process is called gelation. This network is growing and reaching all points in the system to create a complete structure of the gel is obtained.[1-5]

Steps of Sol-Gel Method: The first step in the sol-gel synthesis is solution formation. Various starting materials are mixed with the appropriate solvents in this step to prepare homogeneous solutions. The sol-gel process contains multiple steps; *alkoxide hydrolysis, polymerization (peptide), gelation, and calcination/sintering.*

I. Alkoxide Hydrolysis

Alkoxides are used as starting material to form a solution. M (OR) n. **M**; metal material to be coated, **R**; CH_3 (methyl), C_2H_5 (ethyl) alkyl group, **n**; shows the values of the metal that vary according to the value.Due to their high electronegative OR group, metal alkoxides exhibit high reactive properties. Physical properties are controlled by changing the alkali groups in OR. The amount of water, catalyst type, solvent concentration, and temperature factors affect the rate of hydrolysis. Normally alkoxides are soluble in alcohol and hydrolyzed with water under acidic, basic, or neutral conditions. The optimum molar water/alkoxide ratio is 100. The distance between alkoxide and water molecules increases when this ratio is obtained. Acid catalysts bind polymers with weak bonds, while base (alkali) catalysts bind with strong bonds. When working in a hot environment with distilled water (> 80 0C), a more stable colloid structure is formed [1, 5]. During hydrolysis, the OH-ion in the water replaces the OR-ion in alkoxide (Reaction 1).

Reaction 1. *Hydrolysis of alkoxide [7-8]*

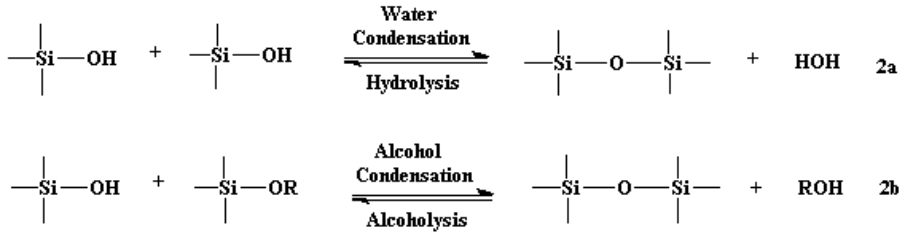


$$M(OR)_4 + H_2O \longrightarrow HO\text{–}M(OR)_3 + ROH$$

II. Polymerization (peptide):

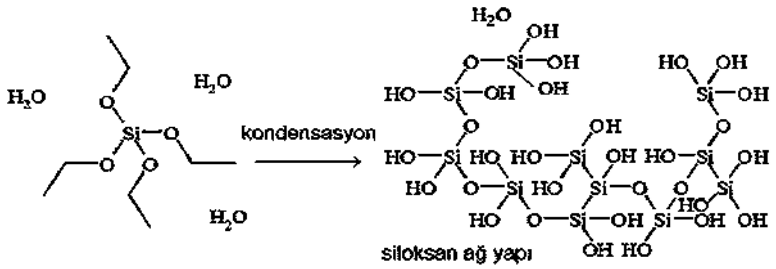
The hydrolyzed Si-OH molecules during the polymerization step form Si-O-Si (siloxane monomer) bonds with two different reactions (Reactions 2a and 2b). This process is defined as asyon condensation Condensation can occur in two ways: water condensation and alcohol condensation. In water condensation, water is released by converting Si-OH molecules to Si-O-Si molecules (Reaction 2a), while alcohol condensation produces alcohol (Reaction 2b). The polymeric oxide structure is formed by hydrolysis and condensation reactions. The polymers in the solution grow with a condensation reaction. This is the transition point from the solution to the gel and is determined by the increase in the viscosity of the solution.

Reaction 2. *Condensation reactions [7-8]*



$(OR)_3M–OH + HO–M(OR)_3 \rightarrow (OR)_3M–O–M(OR)_3 + H_2O$ (Reaksiyon 2a)

$(OR)_3M–OH + RO–M(OR)_3 \rightarrow (OR)_3M–O–M(OR)_3 + ROH$ (Reaksiyon 2b)



In the polymerization process, the solution is prepared by dispersing the precipitates through a solvent action. The electrolytes used in the polymerization give the particles a certain charge. The reason for the loading is that the colloidal particles are stable only when they are loaded. The amount of acid to be used is adjusted by the pH of the medium [6]. Polymerization is a de-coagulation event. Coagulation is the collapse of the colloidal particles due to the zeroing of the electric charge. If a solution forms a negatively charged colloidal solution, it forms a positively charged one with OH ions (bases) and is polymerized with H + ions (acids). Peptidization does not occur if the electrolyte supplied to the solution is more or less than necessary. The high-concentration electrolyte prevents the peptidization by leaving the grains unloaded. When it is used in small amounts, the sediment condition continues as the load is insufficient [5-6]. The selected acid type is one of the important factors affecting peptidization. When the acid concentration is too low, the effect of the electric charge cannot be achieved. This condition makes it impossible to use almost all other organic acids in the solution gel process except for a few strong acids.

III. Gelation:

The monomers formed by the polymerization in the solution come together to form the nanoparticles [6]. The gelation event is closely related to the shape of the colloidal particles. The gel-forming molecules bind to each other with weak or strong bonds, forming skeletal tissues with liquid in the spaces between them. These tissues form the gel structure. The gel formation constitutes sufficient small sol particles for the prepared solution. These particles are formed by agglomerates (agglomerates) with

the electrochemical interaction of the surface charges or by forming gels of precipitated solid particles. The gels in this web structure are then spread over the entire structure and expand triple in volume.

IV. Calcination and Sintering

After drying, the gel is heat treated for the production of dense ceramic material. According to the structure of the gel and the conditions of formation, the following reactions occur during the heat treatment; *Decomposition of salts, Carbonization or organic waste combustion, Chemical water removal, Loss of micropores, condensation.*

The gelling material is usually calcined by heating it to a temperature lower than its melting temperature. At the end of the process, the porosity of the material decreases. With calcination and sintering, the mechanical properties of the material also increase.

Advantages and Disadvantages of the Sol-Gel Method

Advantages	Disadvantages
It enables solids with high surface area and free energy to be sintered at lower temperatures.	The cost of the produced powders is high.
Fiber production is achieved by using metal-alkoxide solutions.	The process is long and the amount of shrinkage during the process is high.
The production of amorphous solid glass materials, which cannot be obtained by cooling from the liquid phase, is realized.	Fine pores may be present in the structure.
Thin SiO_2 and TiO_2 ($\leq 1\mu$) coatings are produced on glass.	Residual hydroxide and residual carbon may form in the structure [3].
Powder production occurs in controllable shapes and sizes.	
The second phases are ensured to be distributed homogeneously within the main phase.	

3. EXPERIMENTAL STUDIES

3.1 Tools, Devices, and Materials

• Tetra Ethyl Ortho Silicate (TEOS, $Si(OC_2H_5)_4$)
• Ammonium Fluoride (NH_4F)
• Ammonium Hydroxide (ammonia, NH_4OH)
• Ethanol (C_2H_5OH)
•Pure water
• Fume hood
• Precision scales, magnetic stirrer and magnetic fish
• Pipette pump, pipette, dropper, and beakers
• Materials for underlaying, turntable and strip casting equipments
•Power source and furnace

3.2 Experimental Procedure

For the experiment, firstly, sol production is performed. In the preparation of the sol, 3 mL of TEOS and 5 mL of ethanol (in a fume hood) are mixed in a beaker using a magnetic stirrer (Solution I). (Since the silicon-based alkoxide TEOS can hydrolyze in the lungs if inhaled, it is recommended to mix it under air flow.) Then, 3 mL of water is dissolved in 5 mL of ethanol (Solution II). A solution consisting of two different catalysts (Stock Solution) is used to form the sol. To prepare the stock solution, 1.9 g of ammonium fluoride salt (NH_4F) and 23 mL of ammonium hydroxide (NH_4OH) solution are dissolved in 100 mL of distilled water. Add 10 drops of the stock solution to Solution 2. Finally, the second Solution is slowly added to Solution 1, which continues to mix. During the addition of Solution 2, the

transparent Solution 1 gradually becomes whitish and opaque. This is due to the polymerization of the left molecule growing as a result of the formation of nanoparticles. After the polymerization process, the structure appears as opaque (milky) as some of the light held in the solution is reflected by the nanoparticles (***Tyndall Effect***). The SiO_2 (silica) source used in the preparation of the sol provides TEOS water loss, while ethanol is the solvent that helps TEOS and water mix. During the hydrolysis reaction, base (alkali) based ammonium hydroxide (NH_4OH) acts as a catalyst, accelerating the formation of Si-OH bonds. Although hydrolysis takes place if the ammonium fluoride salt is not used, the reaction rate is further increased with the fluorite ion present in the ammonium fluoride. As the amount of polymerization increases, the viscosity of the solution increases. Before gelation, spin coating process is performed on the substrates on the turntable by taking them from the left with a dropper, or dip coating process is performed by immersing and pulling the substrate into the left. In addition, the thickness of coated lamel is determined by adding the produced powder into the polymer for tape casting.

Powder Production via sol-gel technique:

Finally, the sol-gel poured into the mold is completely gelled at the end of 15 minutes. Drying of the gel (removal of water or alcohol as a by-product) and calcination produce nano-spheric SiO_2 powder. At the end of the calcination process, the powder obtained from the oven is ground in a mortar, and the final powder product is obtained.

Dipping Coating:

The dip coating method is carried out by dipping a substrate material into a solution for coating and withdrawing it at a *constant speed, controlled temperature, and atmospheric conditions*. **Coating thickness depends on the following parameters;** substrate retraction speed, surface tension of the substrate, depends on the density and viscosity of the solution. **The steps of the coating by dipping method;** *dipping the substrate into the solution, removing the substrate from the solution and evaporation of the solvent from the solution on the surface of the substrate.*

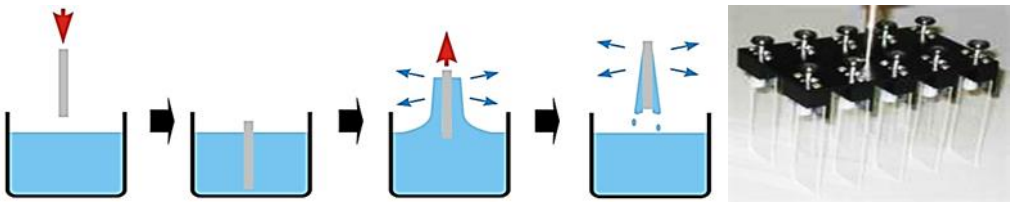


Figure 3. Dip coating steps [9].

Spin Coating:

Spin Coating is used in the production of thin films. Typically, the process is based on dropping a drop of solution into the center of a pad and then rotating the pad at high rotational speeds (typically 3000 rpm). Central acceleration causes the excess solution to be removed and the remaining solution to spread as a thin film on the substrate surface. Final film thickness and coating quality depends on the following parameters.

Solution properties such as; viscosity, drying rate, solid rate and surface tensions. **Process conditions such as;** speed, acceleration etc. Typically, the coating process consists of three steps.

1. Dropping solution on the prepared substrate
2. With high-speed rotation, the removal and spread of excess solvent and
3. With drying, the solvent is evaporated and the coating process is completed with gelling.

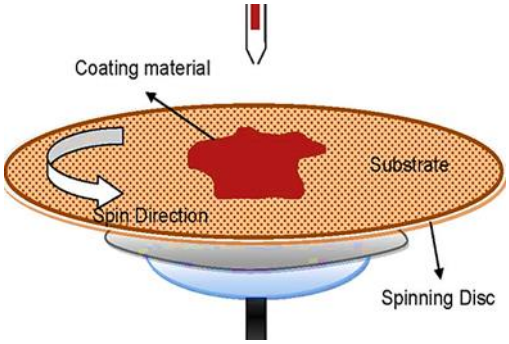


Figure 4. Spin coating turntable and method [9].

Tape Casting

The tape casting method is a technique that allows for the production of various materials, primarily ceramics, as well as metals, metal alloys, and plastics. In a method where casting is possible under ambient conditions, the casting slurry prepared with solid-phase powder material and other components is poured onto a substrate using a system referred to as a Doctor Blade. Drying, the separation of auxiliary components, and if necessary, thermal and mechanical processes will result in the final product. The tape casting method, classified as a liquid phase production technique, has advantages such as minimal material loss compared to traditional casting methods, low cost, ease of production, absence of post-production processing costs, and the ability to produce fine structures ranging from millimeters to nanosize. The tape casting method can be used not only to produce thin film but also to coat the surface of a material [10]. The most commonly used **solvents** in tape casting technology include water, methanol, and acetone. To avoid sedimentation and agglomeration processes that may occur in the liquid, **dispersants** such as phosphate esters, Menhaden fish oil, and glyceryl trioleate are used. Typical organic compounds used as **binders** include polyvinyl alcohol (PVA), polyvinyl butyral, polymethyl methacrylates, and others. Typical **plasticizers** include polyethylene glycol (PEG), dioctyl phthalate, triethylene glycol, diethyl oxalate, and others. The organic components of the casting suspension burn completely during the sintering process [10].

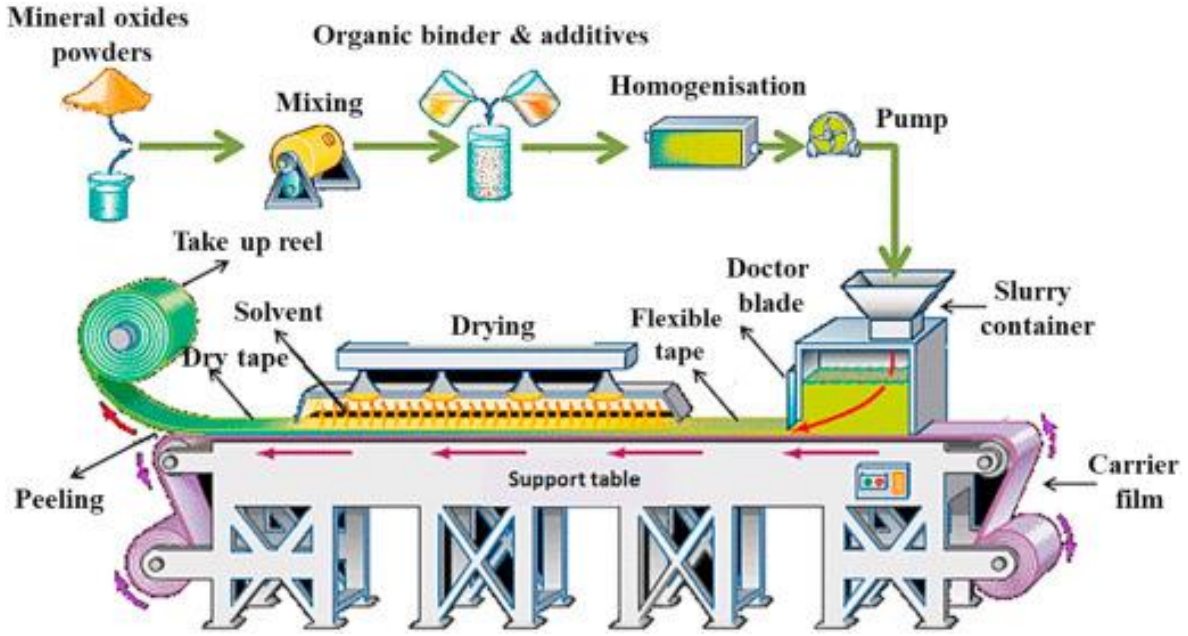


Figure 5. Tape casting unit and steps [10].

4. RESULTS AND DISCUSSION

Weigh the gel product before drying and calcination. Re-weigh after drying and calcination. Thus, the *weight loss in the sample is calculated as %.*

At the end of the experiment, gel production should be ensured starting from the sol. Then, oxide-based nanopowder should be obtained by drying and calcining the obtained gel. SiO_2 film should be formed on the substrates by using dip coating and spin coating techniques of the prepared sol. During coating, the effective parameters (solution viscosity, coating speed, coating time etc.) for different methods should be changed, and the effect of these parameters on the coating quality should be observed. At the end of the experiment, students will have learned the following practical and theoretical outcomes:

- Colloidal systems
- Basic principles of the sol-gel technique
- The Sol-Gel coatings
- Characterization of coating layers
- Nanopowder synthesis with sol-gel technique
- Preparation of the final report (theoretical knowledge, experimental study, results, references)

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EXPERIMENT NAME: GLASS PRODUCTION

1. OBJECTIVE OF THE EXPERIMENT

The aim of the experiment is to prepare a glass batch, synthesis of glass sample using traditional melt-quenching method, and shaping glass using molding method.

2. THEORETICAL INFORMATION

2.1. Glass Industry

The glass industry, which provides inputs to many sectors such as construction, automotive, x"energy, white goods, food, beverages, pharmaceuticals, cosmetics, tourism, furniture, pipes, electrical, and electronics, is one of the fundamental industrial sectors; it holds great importance for national economies. [1] Parallel to the global economy, the glass industry, which is growing at an average rate of 2-4% annually, has an annual production quantity of approximately 175 million tons; the quantitative distribution of total production according to regions and product types can be seen in Table 1. [2]

Table 1: Distribution of world glass production by product type

	Capacity (Million Tons)	Share(%)
Flat glass	82	47
Glass Packaging	80	46
Glassware	6	3
Glass Fiber	6	4
Total	**175**	**100**

Table 2 shows the ranking some of the leading countries in the world glass sector based on their export data.

Table 2: Export data of glass sector's some of the leading countries in the world Million Tons)

Countries	2017	2018	2019	2020	2021
China	15.9	16.9	17.9	18.3	21.6
Germany	6.9	7.8	7.1	6.6	7.8
USA	5.8	5.8	5.5	4.9	5.6
France	3.3	3.5	3.4	3.0	3.5
Hong Kong	2.7	3.1	3.1	3.3	3.3
World	**72.1**	**77.6**	**76.8**	**73.8**	**86.8**

In Turkey, the glass sector is an important industry that contributes to the country's leading sectors, creates net added value for the economy, and demonstrates continuous growth and development. The main production areas of the glass industry in Turkey are as follows:

- Flat Glass:

-Float Glass

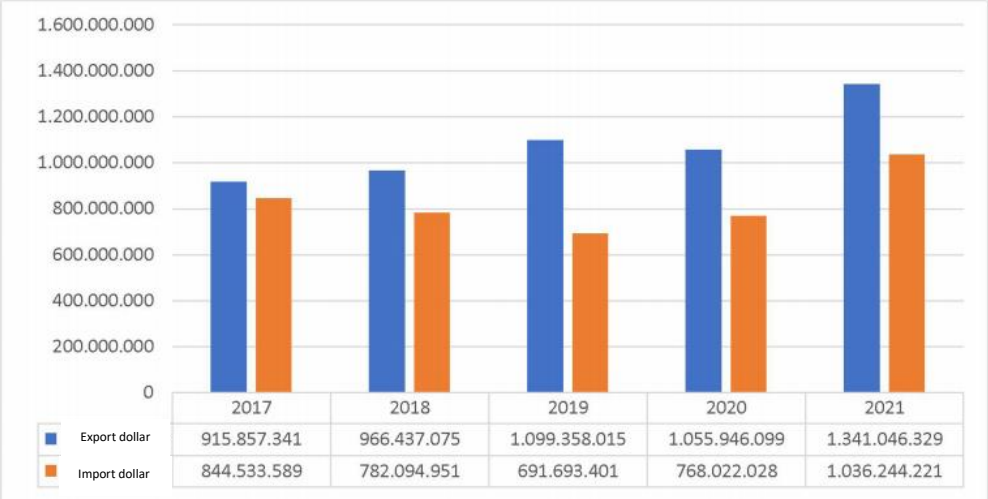
-Processed Glass (tempered, laminated, bulletproof, mirrors, coated, and tempered glass for white goods, energy, and decoration purposes)

- Household Glassware
- Glass Packaging
- Glass Fiber (glass wool, felt, tape, yarn, chopped strand, etc.)
- Others (broken glass, glass beads, glass bulbs, electric lamps, cathode ray tubes, glass inner cores, signaling glasses and glass optical elements, watch and eyeglass lenses, glass bricks, tiles, roofing tiles, mosaics, glass laboratory and pharmacy equipment, glass beads, etc.).

The total production and national income contribution of the Turkish Glass Sector constitute 0.44% of the total production value of the Turkish Industrial Sector. The foreign trade volume of glass products is also at the level of 2.3 billion dollars.

The number of people employed in the primary glass products manufacturing is approximately 12.000, and together with employment in secondary processes and unregistered activities, this figure is estimated to be around 50.000. This accounts for approximately 0.001% of the 29 million workforce. With its high domestic production rate, the sector has generated 7.6 billion dollars of domestic added value in Turkey over the last decade (see Table 3).

Table 3: Foreign Trade Values of Glass and Glass Products [2]



	2017	2018	2019	2020	2021
Export dollar	915.857.341	966.437.075	1.099.358.015	1.055.946.099	1.341.046.329
Import dollar	844.533.589	782.094.951	691.693.401	768.022.028	1.036.244.221

In our country, 90% of the production capacity of the glass industry is met by Şişecam. Turkish Glass Industry began with the establishment of the first glass facility, the Paşabahçe Factory, with a capacity of 3.000 tons in 1935, in line with Atatürk's instructions to İş Bankası to establish and develop the glass industry in Turkey. Having a history of 80 years, the Turkish Glass Industry has continued its development and growth during this period. The major producers in Turkey, apart from Şişecam, include Güral Cam, Marmara Cam, İzocam, Toprak Cam, Schott Orim, Yıldız Cam, Star Grup, Kutaş, Hatipoğlu Cam, Gürsan Cam, Olimpia, Başkent, and Dora Cam [1].

2.2. Theoretical Information About Glass Material

Glasses are any of highly viscous amorphous materials formed from a melt by cooling to rigidity without crystallization. Their characteristics are: transparency, hardness, fragility and chemical stability [4,5]. 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) [3-6].

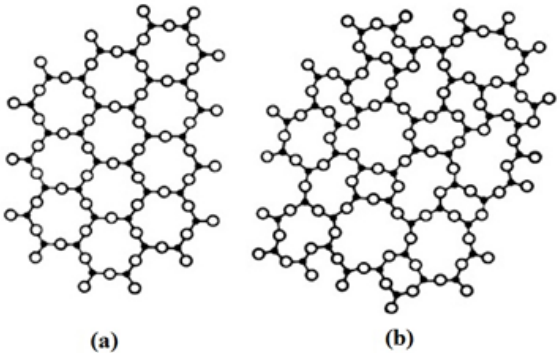


Figure 1: Atomic arrangement of a) crystalline, b) amourphous structure.

When heated to the appropriate temperature, many inorganic elements and compounds form liquids having close to viscosities to water (10^{-4} – 10^{-3} Pa.s) [7]. These liquids solidify rapidly when cooled to the solidification points. If the crystallization speed is slow enough, it may be possible to cool the solution below the solidification point without crystallization. The mechanical properties of this material, namely glass, are similar to those of an elastic solid material. In order to form vitrous strucure, 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 to an elastic solid and its structural properties are like a viscous liquid [3,8].

When a liquid is cooled, the space for the atoms to move around decreases and on further cooling below the glass transition temperature the atoms can no longer move around with respect to each other and so the material becomes a solid. A measure of this is the specific volume, which can be measured 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 in rate of cooling the liquid crystallizes, however if the liquid passes into a supercooled state the volume decreases and no crystallization occurs [4,5,9].

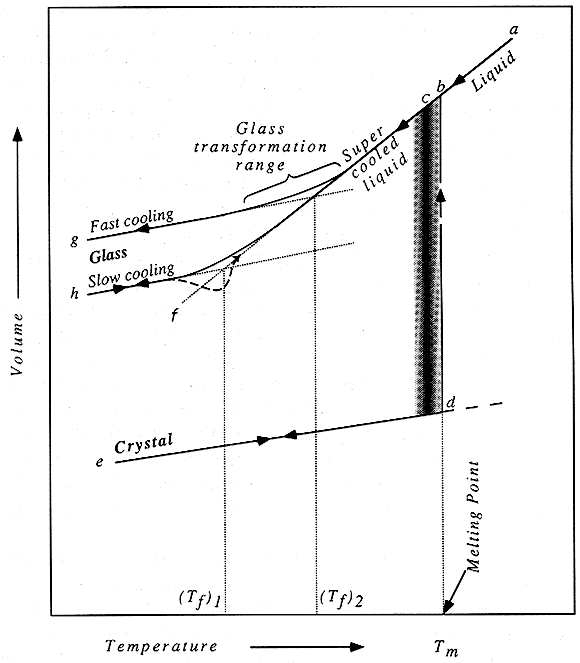


Figure 2: Specific volume – temperature change of crystalline and amourphous solids.

There is a decrease in the volume of glass held in 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 [7,10].

Crystallization of a liquid or an amorphous solid is a complex process involving simultaneous nucleation and growth of crystallites. Crystallization is initiated by crystal nucleation. Nucleation may occur spontaneously or it may be induced artificially. Homogeneous nucleation occurs in the interior of the parent phase without the involvement of a foreign substance. At temperatures below a material's melting point, the driving force for solidification is the difference in Gibbs free energy (ΔG) between the liquid and the solid [9,10].

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).

3.MATERIALS AND EQUIPMENT USED IN THE EXPERIMENT

Quartz sand (SiO_2), Soda (Na_2CO_3), Limestone ($CaCO_3$), Agate mortar, Spatula, High-temperature gloves, Alumina crucible, Precision balance, Drying oven, Glass casting mold, Glass melting furnace, Annealing furnace

4.EXPERIMENTAL PROCEDURE

In experimental studies aimed at soda-lime glass production, technical grade quartz sand (SiO_2), soda ash (Na_2CO_3), and limestone ($CaCO_3$) are used. Carbonates used to obtain Na_2O and CaO undergo calcination during melting, converting into oxides. Table 4 summarizes the general properties of the oxide components used in experimental studies.

Table 4: General properties of oxide components used in experimental studies.

	Molar Mass(g)	**Density (g/cm^3)**	**Melting Temperature (°C)**
SiO_2	60,08	2,648	1713
Na_2O	61,98	2,27	1132
CaO	56,07	3,34	2613

For the preparation of the blend of glass with a composition of $75SiO_2$-$15Na_2O$-10CaO in mol%, powders are weighed on a precision balance (with a sensitivity of 10^{-4} g). After weighing, they are homogeneously mixed in an agate mortar. Then, the mixture is placed in an alumina crucible and held in a furnace at 1400°C for 30 minutes. The molten glass is poured into a preheated stainless steel mold in an oven at 200°C for 30 minutes. For homogenization, this melting/casting step can be repeated two or three times depending on the glass composition. To relieve internal stresses that could cause cracking in the produced glass, the stainless steel mold containing the poured glass is held in a tempering furnace preheated to 450°C for 30 minutes before being gradually cooled to room temperature inside the furnace.

5.POST-LAB QUESTIONS

1. Calculate the weights of the raw materials required to prepare a blend of soda-lime glass with a composition of $75SiO_2$-$15Na_2O$-10CaO in mol% using SiO_2, Na_2CO_3, and $CaCO_3$ as raw materials (MW CO_2: 44.01 g).
2. Write down the functions of the components of commercial soda-lime glass in glass structure.

SiO_2, Na_2O, Li_2O, K_2O, CaO, MgO, PbO, Al_2O_3, $B2O_3$, ZnO, Fe_2O_3, CaF_2, Sb_2O_3, SnO

3. Provide information about the basic properties and usage areas of the type of glass specified by the instructor.

4. Explain the formation of amorphous and crystalline structures depending on the cooling rate.

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EXPERIMENT NAME: POLYMER MATRIX COMPOSITE MATERIAL PRODUCTION

1. THE PURPOSE OF THE EXPERIMENT

Production of thermoset polymer matrix and E-Glass fibre reinforced composite plates by hand lay-up method.

2. THEORETICAL INFORMATION

2.1. Theoretical Background

Polymers; carbon, hydrogen, oxygen, nitrogen and other organic or inorganic elements formed by monomers, called monomers, are materials obtained by breaking the weak bond in the groups of the molecule in the simple structure and transforming it into a long and chain structure called polymer.

Polymers are mainly divided into two groups, thermoplastics and thermosets, based on their behaviour to heat and solvents.

Thermoplastics are linear and/or branched chain polymers with no cross-links between the chains. Due to these properties, they are soluble in suitable solvents, melt when heated and harden when cooled. These plastics can be repeatedly heated and cooled without significant changes in their properties. They soften and flow under heat and pressure and thus can be shaped in various forms. They can also dissolve in suitable solvents and can be moulded into various shapes. Polyethylene (PE), polypropylene (PP), poly(vinyl chloride) (PVC) and polystyrene (PS) are some of the commonly used thermoplastic polymers.

Thermosets are polymers with dense cross-links (network-structure) between their chains. Due to the cross-links, they are insoluble in any solvent, do not melt when heated, and degrade when heated to sufficiently high temperatures. After thermosets are permanently shaped and hardened by a chemical reaction, they cannot be softened again by heating and cannot be put into another shape. For this reason, they are moulded in a partially polymerised state by the action of heat or by the combined action of heat and pressure. In addition to heat treatment, there are also many polymers that harden or mature only by chemical reaction at room temperature. During moulding, polymerisation progresses, the plastic cross-links and the polymer loses its flow properties. Therefore, thermosets do not enter the remanufacturing process like thermoplastics, i.e. they are not recyclable. Phenol-formaldehyde, melamine-formaldehyde, epoxy resins, vinyl ester resins and unsaturated polyester resins are examples of the most widely used thermoset polymers.

2.2. Principles of Experiment

The new material group formed by at least two different materials by forming an interface between them without chemical bonding is called composite. Basically, composite materials have two main components.

The matrix phase forms the main component of the composite material. Its main task is to hold the structure together stably by wrapping around the reinforcement (fiber) component. Another task is to transmit and distribute any applied force to the reinforcement phase through the interface bond without being destroyed. An ideal matrix material should be able to coat the fibers very well by surrounding them properly.

The main load-bearing element in composites is reinforcing fibers. Glass fibers are the most widely used and the cheapest type of reinforcement. Aramid and carbon fibers are reinforcement types with high mechanical properties and high cost. Reinforcements can also be available in different forms. Figure 1 shows the classification of composites according to their reinforcement shape and distribution. In Figure 2, the situations of these species in the structure are exemplified. Finally, different weaving types are shown in Figure 3.

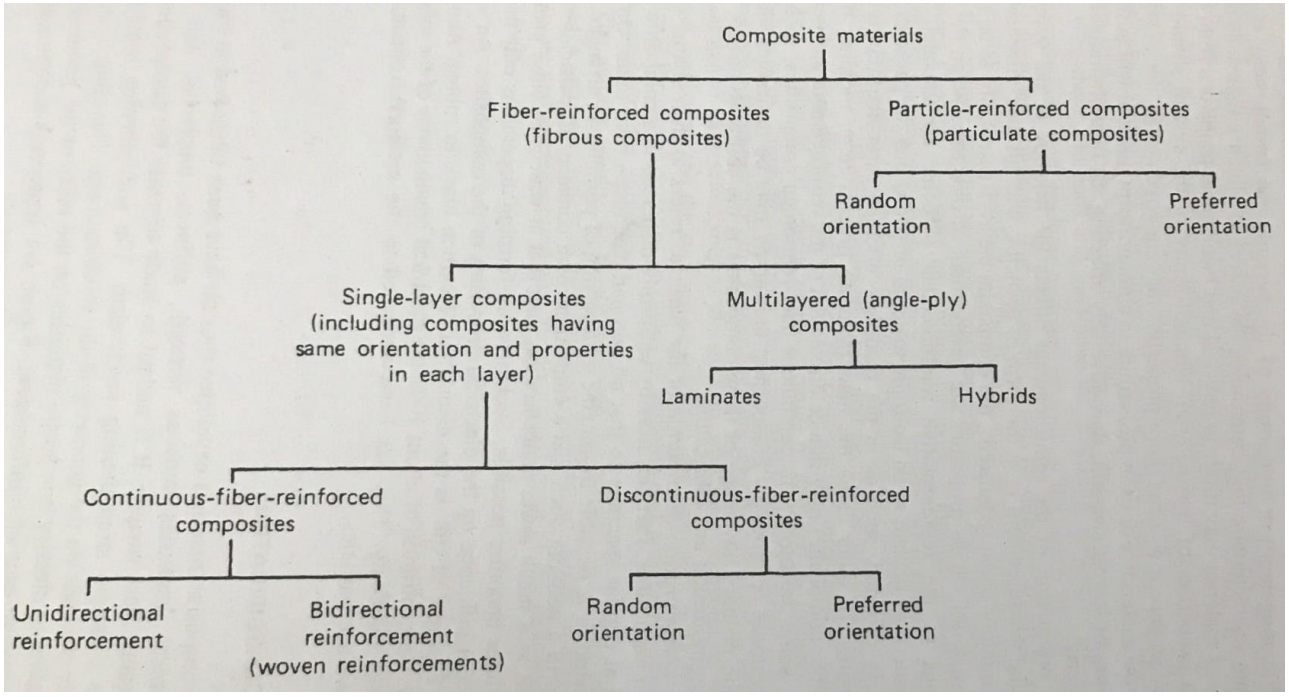

Figure 1. Classification of composites according to reinforcement shapes and distributions

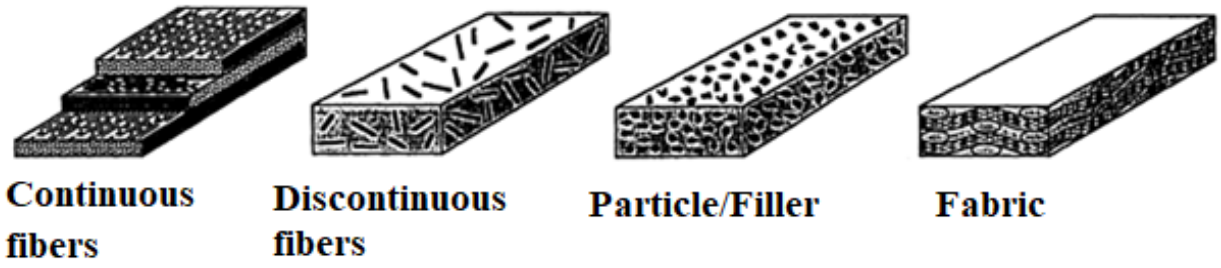

Figure 2. Various reinforcement types

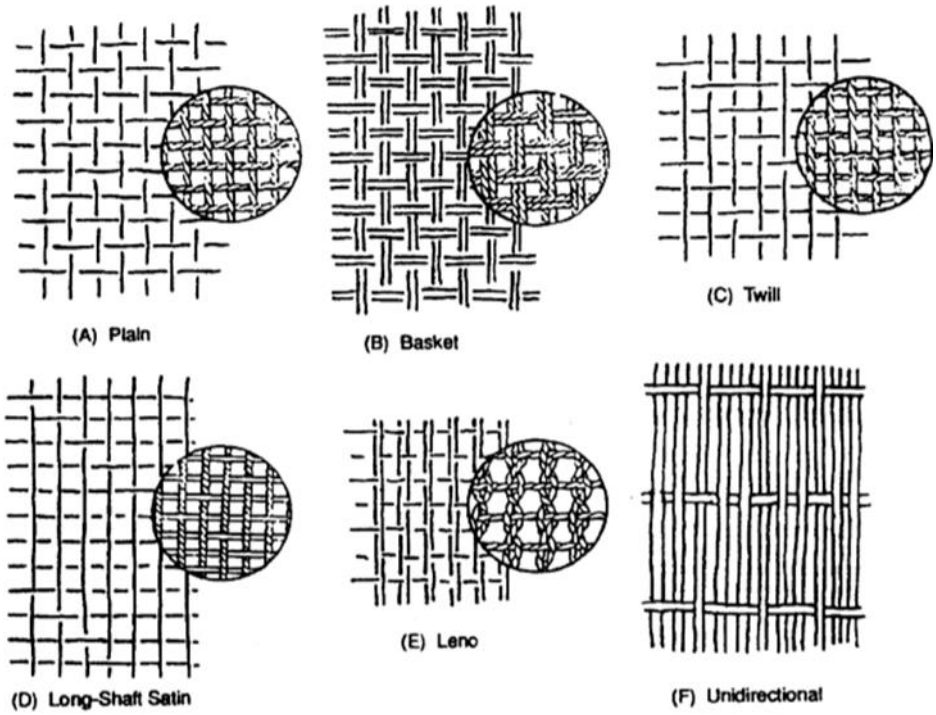

Figure 3. Various woven fabric types

2.2. Classification of Composite Materials According to Their Matrices

- ***Polymer Matrix Composites (PMCs):*** It is the most widely used composite type today. It has approximately 97 % 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.

2.3. Polymer Matrix Composites

Polymer 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)…

Glass fibre reinforced unsaturated polyester resins (composites) are the most widely used type of reinforced unsaturated polyester resin. Before curing, the product consists of a mixture of a linear polymer and a liquid monomer. Being a low viscosity liquid, it can be

mixed with large amounts of fillers and thoroughly wet the glass fibre. Unsaturated polyesters can be reinforced with up to 80% glass fibre. These reinforced, unsaturated polyesters have a strength of 172-344 MPa, good impact strength and chemical resistance when hardened (crosslinked). Glass fibre reinforced unsaturated polyesters are used in the construction of automobile panels and body parts as well as small boat hulls, building panels, bathroom parts, pipes, water tanks and fuel tanks where high abrasion resistance is required.

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

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

2.4. Production Methods of Plastic Matrix Composites

Hand Lay-Up Method: It is the most widely used method for the production of large-area thermoset matrix composites. After the release agent is applied, the gelcoat is applied. After the gelcoat layer has hardened, chopped-strand mat fabrics or woven fiber fabrics and thermosetting resin are applied with a brush or roller. Reinforced plastic product can be obtained at a rate of 25 - 35% by hand lay-up, which is a molding method that requires low fixed capital investment. Since it is a labor-intensive production method, capacity depends on the number of labor and molds. An average of 2 products can be taken per day from a mold.

Spray-Up Method: It is a molding method that enables the hand lay-up method to be applied more rapidly. During production, resin and fiber are sprayed on the mold with the help of a special machine. In the spraying method, the continuous fiber bundle is used by chopping 17-50 mm in length during the spraying process. It provides mass production opportunity and labor saving in large surface products.

Vacuum Bagging and Autoclave: These are the methods applied to increase the properties of the composite after hand laying or spraying. A vacuum blanket is placed on various separation fabrics to absorb excess resin on resin composite products that have not been cured in vacuum bagging. A heat-resistant film called vacuum bag is placed at the top by attaching a vacuum nozzle and is closed with a special paste to ensure impermeability. The system is put into vacuum and vacuum continues to be applied until the curing process is complete. Excess resin is passed through the product, increasing the fiber volume ratio, thinning the section and decreasing the weight, eliminating the voids and increasing the mechanical properties as a result. Similarly, in the autoclave, uncured products are vacuumed by taking the vacuum bagging system. Then, curing is achieved by giving heat and pressure in autoclave ovens.

Vacuum Infusion: Chopped-strand mat fabrics or woven fiber fabrics are placed on top of each other and taken to the vacuum bagging system. However, in the vacuum bag, in addition to the vacuum opening, the openings through which the resin will enter are opened. As the system is put into vacuum, the resin starts to fill into the inside of the bag and wets the fibers to form the composite.

Resin Transfer Molding (RTM): In this production method, two-sided products are obtained by using two molds, male and female. Fiber fabrics are placed on the mold and the molds are closed. Thermoset resin is injected into the mold under pressure from a pre-prepared resin injection point. With the resin injection method, a higher quality product is obtained more quickly and economically than hand lay-up method.

Sheet Molding Compound / Bulk Molding Compound (SMC/BMC): It is a method of forming fiber, resin and filling material mixtures in hot press molds at 150 - 170°C and 50 - 120 kgf/cm^2 pressure. Complex shaped products can be obtained. It is a fast and rapid method of 3 - 6 minutes. In SMC, long chopped by pre - combining long-cut (25 - 50 mm) fibers with filling and resin is used, while in BMC, short - chopped (3 - 12 mm) fiber, filling and resin combination is used.

Filament Winding: It is a molding method used especially for the production of pipes and tanks. It is in the form of winding continuous fiber bundles on a rotating mold at certain angles after wetting from the thermoset resin bath.

Centrifugal Casting: It is used in the production of cylindrical products such as pipes, tanks, poles. The chopped fiber and thermosetting resin are sprayed together into a rotary die. The centrifugal force resulting from the rotation of the mold ensures that the laminate adheres to the mold surface and obtains a smooth product on both sides.

Pultrusion: It is based on the principle that continuous fiber bundles are hardened while being drawn through a hot mold in the desired profile after passing through a thermoset resin bath. In the direction of fiber reinforcement, very durable profile products with a very high glass fiber content are obtained.

Thermoplastic Injection / Extrusion Method: While extrusion machines are used for molding profile type products, injection machines are used for molding complex shaped products. In injection and extrusion machines working with the same principle, the granular thermoplastic raw material supplied from the feeding chamber is heated in the heating zone, making it fluid, and short-cropped fiber is fed on the one hand. Then, a homogeneous mixture is provided with the auger grooves on the one hand, and it is carried towards the outlet end on the other hand. In extruders, profiles are drawn in accordance with the shape of the mold with the help of pressure effect and pulling apparatus through the mold placed at the exit end, while in injection machines, short clipped fiber mixed with fluidized thermoplastic is injected into the closed mold located right next to the exit nozzle, and it is cooled and hardened in a closed mold.

2.5. 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.

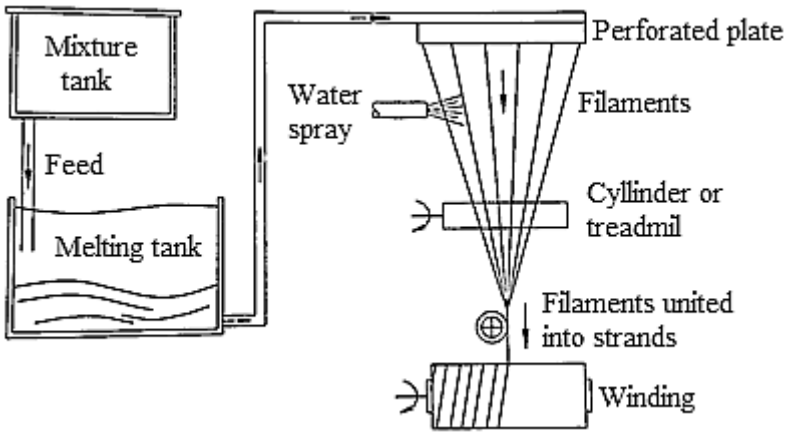

Figure 4. Schematic drawing of glass fiber production

3. EXPERIMENTAL STUDIES

3.1. Materials Used

- Reinforcing element: E-glass woven and mat fabric
- Matrix material: Orthophthalic based unsaturated polyester resin
- Resin accelerating agent: Cobalt octoate
- Resin initiating agent: Methyl Ethyl Ketone (MEK) Peroxide
- Dosing pipettes
- Plastic cup
- Stirring stick
- Mold release agent
- Brush
- Acetone or thinner
- Plastic gloves

3.2. Experimental Procedure

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.

4. RESULTS AND DISCUSSION

4.1. Experimental Results and Discussion (Information Required In the Experiment Report)

1. Please give general information about the theoretical part of the experiment (Definition, classification, types and examples of composite materials, general information about polymer materials used as matrix materials, types, examples, general information about glass fibres used as fibre materials, etc.). ***[10 Points]***
2. Please give detailed information about the materials used in the experiment (Unsaturated polyester resin, cobalt octoate and MEK (MethylEthylKetone) Peroxide, E-Glass). ***[10 Points]***
3. Please describe the experiment in detail (in chronological order and explaining the reason for each procedure). ***[40 Points]***
4. Please write down the difference(s) you observe between the ideal application of the hand lay-up method and the experimental application. Discuss what kind of negative effects these differences may cause in the composite material produced. ***[30 Points]***
5. Please write down what you have learnt from this experiment and what the experiment has contributed to you in accordance with the experiment format. ***[10 Points]***

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EXPERIMENT NAME: REDUCTION ELECTROLYSIS

1. AIM OF THE EXPERIMENT

In this experiment; It is aimed to learn the electrolysis phenomenon, to learn the refining (purification) and reduction (recovery) processes of copper and to obtain electrolytic copper by using copper in solution.

2. THEORETICAL INFORMATION

Electrometallurgy

It is called "Electrometallurgy" to produce metals in all kinds of raw materials containing ore or metal by using electrical energy. In reality, electrometallurgy is an application of electrochemistry. Here, electrochemistry methods are applied to metals.

Electrolysis

It is the phenomenon of the liberation of metal ions at the cathode and non-metallic ions at the anode as a result of chemical decomposition by passing an electric current through aqueous or molten electrolytes. Ions are discharged at the electrodes; It can be liberated, collected as an element, and react with the electrode.

Electrode: Conductor that conducts electric current to liquid, solid and gas phases. In an electrochemical sense, the electrode is the system that combines the electrode conductor and the ionic conductor.

Electrolyte: It is the environment that contains (+) and (-) charged free ions. It is the liquid that conducts the electric current used in electrolysis. Molten salts and solutions of acids, bases and salts are used as electrolytes.

Electrolysis cell: Cells in which electrical work is converted into chemical work by applying external voltage.

Generator: It is a direct current source that provides electrical energy in the external circuit. The electrode connected to the positive pole of the generator is the anode, and the electrode connected to the negative pole is the cathode.

In electrolysis; cations (+ charged ions) are reduced to the cathode, and anions (- charged ions) are oxidized to the anode. If there is more than one type of cation in the electrolysis vessel, the one with the greatest reduction potential (potential) is reduced first. Then the reduction continues sequentially. If there is more than one type of anion in the electrolysis vessel, the anion with the greatest oxidation tendency is collected at the anode first. The less active anion is first released at the anode, and the less active cation is first released at the cathode. In electrolysis, compounds can be separated into their elements. If a voltage higher than the battery voltage is applied to the electrochemical cell, the reaction in the battery is reversed. Electrolysis occurs. This phenomenon is called charging the battery.

The electrolysis process is carried out in a cell called an electrolysis vessel or tank. This cell consists of two electrodes immersed in a compound that dissolves into positively and negatively charged ions, and these electrodes are arranged so that they do not touch each other (usually 5- 20 cm between the two electrodes). In order to carry out the electrolysis process, these electrodes are connected to a direct current source and the voltage (electric field) between the electrodes moves the ions towards the oppositely charged electrode (pole). Therefore, (+) charged ions go to the cathode, while (-) charged ions flow towards the anode. Atoms or

molecules that balance their charge at the opposite pole precipitate on the electrode or enter into new reactions with the molecules in the electrolyte.

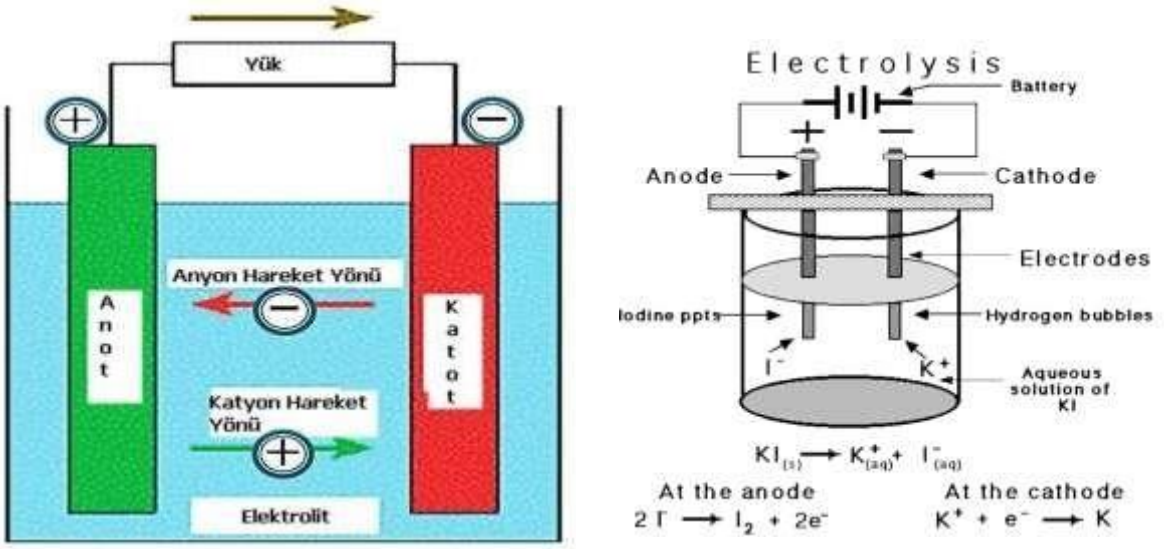

Figure 1. Electrolysis Method

Electrolysis of copper (Cu) in industry is used for two purposes. One of them is the purification of copper, which is carried out in order to collect the impure copper metal from the impurities in it and collect it on the cathode; The other is the copper recovery process to recover the copper metal from aqueous solutions.

Application Areas of Electrolysis

Electrolysis processes, as mentioned earlier, are used in the field of metallurgy by electrolysis, in the preparation (method using insoluble anode-recovery electrolysis) or refining (method using soluble anode-purification electrolysis) of metals. It is also a method of protecting against corrosion by an electrolytic metal deposit and coating metals with a metal deposit (for example, nickel plating, zinc plating, cadmium plating, chrome plating, silver or gold plating). Pure hydrogen is also obtained, in particular, by the electrolysis of water. Application areas include gas production (chlorine), obtaining anode layers with protective oxides on metal (the process of anodizing aluminum by means of alumina), electropolishing, degreasing of metals as cathode or anode. Electrolysis also allows the measurement of current intensities, especially current amounts in voltmeters. Treatment electrolysis, which is based on the separation of organic tissues with the help of continuous current, is also used in medical applications such as the destruction of nerve endings (neurons), hardened tumors, polyps in the nostrils, and the treatment of urethra or esophagus narrowing.

Refining (Purification) Electrolysis of Copper

The refining electrolysis of copper is an electrolysis process that enables to obtain refined copper of sufficient purity, and this process is carried out with soluble copper anodes. Copper sulfate ($CuSO_4$) and H_2SO_4 solution are used as electrolytes. One of the copper electrodes immersed in the solution is pure copper, while the other is impure copper. As the (+) charged anode, copper (blister copper) electrodes obtained by purification at high temperatures (98-99% purity) are used. These electrodes contain O, S, Au, Ag, Pt and various amounts of As, Sb, Bi,

Sn, Se, Te, Pb, Zn, Fe as impurities in their composition. As the (-) charged cathode, titanium plates coated with electrolytic copper or copper plates (99.5% purity) coated with oil to prevent sticking are used. While electrons flow from the anode to the cathode out of the electrolyte; They flow from the cathode to the anode in the electrolyte. Under suitable conditions, the anode oxidizes and goes into solution and is reduced at the cathode. Some of the other impurities in the anode do not dissolve, they break off from the anode and collect at the bottom of the cell. This residual material is called "anode mud". Some impurities dissolve into the electrolyte. Since the anode and cathode are of the same composition, the separation voltage is theoretically zero and the cell voltage is slightly above the electrolyte resistance.

Electrolytic refining is done for two reasons. While the main purpose is to get rid of impurities that cause a decrease in conductivity, noble metals and semi-metallic metals (Se,Te) that pass into the anode sludge during the refining process add value to the business as they cover the cost in the refinery.

When current is applied between the anode and the cathode, the following occurs in the $CuSO_4$- H_2SO_4-H_2O type electrolyte:

The operating conditions of classical copper purification electrolysis are 0.2-0.35 V cell voltage. The electrolysis conditions are as follows:

- 35-50 g/l Cu and 140-220 g/l H_2SO_4 in electrolyte
- Temperature 50-65 ºC
- Cathode current density 150-250 A/m2 (15-25 mA/cm2)

Reactions occurring in the purification electrolysis of copper;

Anode reaction: $Cu^0 \rightarrow Cu^{+2} + 2e^-$ (Here, copper dissolves as Cu+2 ions as a result of anodic oxidation.)

Dissolved (+) charged copper ions (Cu^{+2}) reach the cathode surface.

Cathode reaction: $Cu^{+2} + 2e^- \rightarrow Cu^0$ (Here Cu^{+2} copper ions are reduced to metal state by recombining with electrons transferred from the anode.)

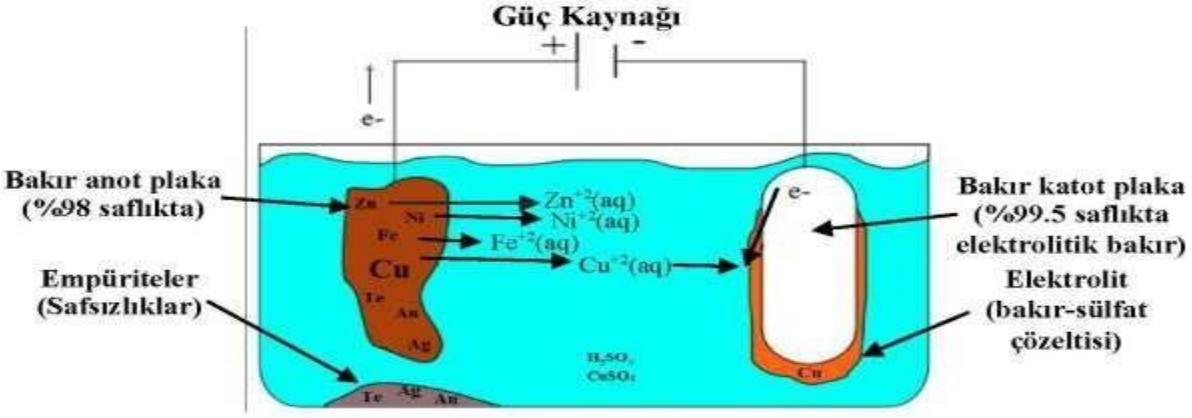

Figure 2. Purification electrolysis of copper

Among these options, it is seen that there is no energy consumption in copper refining electrolysis. However, since the anodic and cathodic reactions are kinetic impediments, excessive voltages must be applied, and the voltage drop due to the resistors at the junctions must also be considered.

Reduction (Reduction) Electrolysis of Copper

Recovery electrolysis of copper is an electrolysis process that provides copper from solutions, and insoluble anode and cathode electrodes are used during this process. While a hard lead alloy containing 4-6% Sb is used as the anode, stainless steel and recently titanium plates are used as the cathode. For the reduction process, a temperature of 40-60 ºC and a current density of 70- 150 A/m2 are selected. During electrolysis, (-) charged ions in the solution go to the anode, and (+) charged ions go to the cathode. Anions are oxidized at the anode surface, while cations are reduced at the cathode surface. Thermodynamically, copper reduction electrolysis takes place at 0.89 V.

Reactions during reduction electrolysis of copper; $CuSO_4 + H_2O \rightarrow Cu + H_2SO_4 + ½O_2$ Anode reaction: $SO_4^{-2} + H+ + 2e- \rightarrow H_2SO_4 + ½O_2$

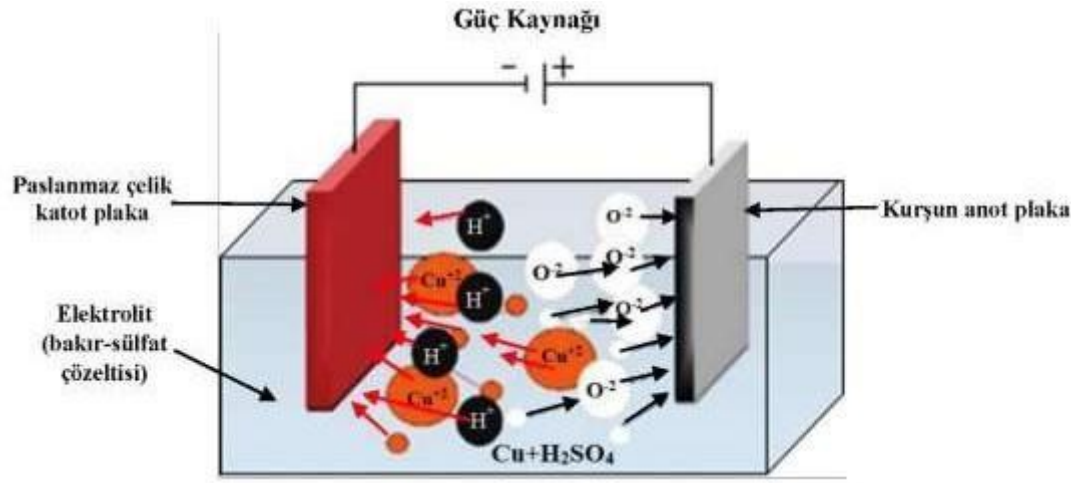

Figure 3. Recovery electrolysis of copper

One of the differences between the purification and recovery electrolysis of copper is that the 'cathode' reactions are the same but the 'anode' reactions are different. In addition, in the electrolysis of copper purification, metal ions dissolved from the anode are reduced at the cathode; In the recovery electrolysis of copper (as an insoluble lead alloy containing 4-6% Sb is used), the anode reaction takes place by oxidation of an anion or molecule in the electrolyte.

Electrolysis Conditions and Additives

Colloid is a homogeneous mixture. This homogeneous mixture contains gelatin and thiourea. Gelatin provides homogeneous and uniform bonding of copper to the cathode surface and creates a fine crystalline hard copper structure. Thiourea is used to eliminate the side effects of gelatin. Another task of the thiourea is to isolate the spikes and pits on the cathode surface. Another additive, mersolate, acts as an antioxidant and delays sulfation on the cathode surface. The purpose of adding HCl to the homogeneous mixture is to precipitate silver ions (Ag+) in the electrolyte as silver chloride (AgCl).

Table 1. Electrolysis conditions and additives

Parameters	**Conditions**
The copper concentration in the electrolyte	35-50 gr/l
H_2SO_4 concentration in the electrolyte	140-220 gr/l
Temperature	50-65 ºC
Cathode Current Density	150-250 A/m^2
Cell Voltage	0,2-0,35 V
Circulation Rate	0,02 m^3/dak.
Colloids Gelatine	0,0001-0,001 kg/m^3
Thiourea	0,0001-0,001 kg/m^3
HCl or NaCl	0,035 kg/m^3

3. CALCULATIONS

In an electrolysis circuit, the amount of product that should be theoretically obtained at the end of the process by the passage of the 'I' current for 't' time is calculated by the Faraday equation:

MT = (I . t . Ma) / (Z . F) * ;

In this equation,

MT = The amount of product obtained (accumulated at the cathode), I = The current intensity (A) passed through the circuit, t =

Time (sec),

Ma = Atomic weight of the substance deposited at the cathode (for Cu: 63 gr/mol), Z = Valence of the substance deposited at the cathode in the compound (for Cu: 2),

F = Faraday's constant (96500 Coulomb).

In electrolysis processes, the current density is calculated by the ratio of the current flowing through the circuit to the area of the cathode electrode immersed in the electrolyte:

Cathode Current Density = Current Through Circuit / Area of the Cathode Remaining in Electrolysis

Since some of the current flowing through the circuit is used to overcome the resistances during the experiments, the product obtained by electrolysis is less than the theoretical product. As a result of multiplying the ratio of the obtained product (Mg) to the theoretical product (MT) by 100, ** β = [(MG / MT)×100], % current efficiency (efficiency) is obtained. If the amount of

material collected at the cathode or separated from the anode is equal to the theoretical value calculated by the Faraday equation, it is understood that the current efficiency is 100%, and if not, it is below 100%. In addition, the value obtained as a result of [(MT-MG)/MT)×100] gives the experimental % error margin.

Case Study;

a- Refining Electrolysis

Before starting the experiment, the electrolyte liquid was prepared using the data in Table 1. The prepared electrolyte was placed in the beaker. Then the cathode plate was cleaned with alcohol and dried and its weight was measured on a precision balance. The temperature of the prepared electrolyte was increased to 65 ^{0}C and meanwhile magnetic stirrer was started. The current density was calculated by measuring the surface area of the cathode plate in the electrolyte. The prepared anode and cathode plates were connected to the power source and the current was given according to the calculated current density. After the electrolysis process took 15 minutes, the power supply was turned off just in time. The cathode plate was removed from the electrolyte, cleaned and dried with alcohol, and its final weight was measured on a precision balance. Then, the theoretical copper amount to be obtained during the experiment was calculated using the (*) equation. The current efficiency was calculated using the equation (**) to calculate the electrolysis efficiency.

b- Reduction Electrolysis

The electrolyte used in refining electrolysis was used in reduction electrolysis. Lead plate was used as the anode plate and steel plate was used as the cathode plate. After the cathode plate was cleaned with alcohol and dried, its weight was measured on a precision balance. Then, the surface dimensions of the part that will remain in the electrolyte were measured and connected to the power supply with the anode positive and the cathode negative. The voltage was increased by keeping the current applied to the refining electrolysis constant. After the electrolysis process took 15 minutes, the power supply was turned off just in time. After the cathode plate was removed, cleaned with alcohol and dried, the final weight was weighed. Then, the amount of copper to be collected during the electrolysis was calculated according to the (*) equation. To calculate the electrolysis efficiency, the current efficiency was calculated using the equation (**)

c- Example Interpretation

The yield (99.2%) we obtained as a result of refining electrolysis showed that the experiment was carried out correctly. However, in reduction electrolysis, more copper was collected than the theoretically required amount. The reasons for this may be: the current density cannot be adjusted exactly or the anode and cathode plates are closer to each other than they should be.

The copper mined in the refining electrolysis was obtained by dissolving the fire-refined anode plate with a purity of 98-99%. On the other hand, in reduction electrolysis, the copper in the electrolyte is recovered. One of the differences between refining electrolysis and reduction electrolysis is the anode plate used. Lead plate was used in reduction electrolysis and there was no dissolution of the used plate.

The reason for using a lead plate is that the lead remains on the anode surface directly forming insoluble $PbSO_4$. If the anode copper contains too much lead, the resulting PbSO4 completely covers the surface and causes the anode to passivation.

Another difference is the anode reactions that occur. While metal ions dissolved from the anode are reduced at the cathode in refining electrolysis, since insoluble anodes (usually lead alloys containing 4 – 6% Sb) are used in reduction electrolysis, the anode reaction must be achieved by oxidation of an anion or molecule in the electrolyte. The oxidation process is a task undertaken by the OH- ion, which is formed as a result of the dissociation of water in aqueous solutions. The refining and reduction electrolysis anode reactions are given below.

Although lead is a basic metal; Explain the reason and/or reasons why copper is used as an anode material in reduction electrolysis, accompanied by related reactions.

When we look at the EMF series, we can see that lead is more basic than hydrogen, and copper is more noble. With this simple approach, we can say that lead will dissolve if it is used as an anode during copper reduction electrolysis. However, in lead and copper reduction electrolysis, PbO and PbO formed by the breakdown of water, and this PbO makes the H_2SO_4 solution and $PbSO_4$ compound. These compounds coat the lead anode surface and form a stable anode surface. As a result, lead is insoluble and is therefore used as an anode material.

$2H_2O \rightarrow O_2 + 4H^+ + 4e^-$ (Anode Reaction), $Pb + \frac{1}{2} O_2 \rightarrow PbO$, PbO +

$H_2SO_4 \rightarrow PbSO_4 + H_2O$

4. MATERIALS AND DEVICES USED IN THE EXPERIMENT

- Electrolysis cell,
- Direct current power supply (voltmeter, ammeter and connection cables),
- Copper-sulphate and sulfuric acid solution (35-50 g/L Cu-containing solution in Electrolyte, 140-220 g/L H_2SO_4), alcohol (for cleaning), gelatin powder and thiourea (0.001 g) as additives
- Precision scales,
- Beakers,
- Cathode electrolytic copper and anode blister copper (purified at high temperatures) plates (for purification electrolysis),
- Cathode stainless steel and anode lead plates (for recovery electrolysis),
- Heater, magnetic stirrer, caliper. Duties of additives:

Gelatin: It creates a fine crystalline hard copper structure by ensuring that the copper deposited on the cathode is properly and homogeneously bonded to the surface.

Thiourea: Eliminates the side effects of gelatin and isolates the spikes and pits on the surface of the cathode material.

5. CONSTRUCTION OF THE EXPERIMENT

The electrolyte solution is prepared by weighing the specified amounts of copper-sulphate ($CuSO_4$) and H_2SO_4 and put into the beaker. Specified amounts of gelatin powder and thiourea are added to the solution and mixed. The material from which the metal will be collected (cathode plate) is cleaned with alcohol and dried, and the result is recorded by weighing it on a precision scale. The anode and cathode electrodes are placed in the electrolyte solution. The connection cable of the anode plate to the (+) pole of the DC power supply; The connection

cable of the cathode plate is attached to the (-) pole. Considering 150 A/m^2 in both copper purification and recovery electrolysis experiments as current density, the area of the part of the cathode plate immersed in the electrolyte is calculated, and after determining the amount of current to be switched, the DC power supply is operated and the circuit is energized. The electrolysis process continues for 15 minutes and the power supply is turned off according to the exact time, and the current supplied to the circuit is cut off. The cathode plate is cleaned with alcohol as in the beginning, dried and its final weight is re-weighed. The amount of material collected at the cathode is determined and compared with the amount that should theoretically accumulate, the current efficiency and experimental error margin are calculated with the help of the equations given above. The execution of both experiments is carried out in the same way.

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EXPERIMENT NAME:ORE DRESSING

1. OBJECTIVE OF THE EXPERIMENT

In this experiment, it is aimed to investigate the crushing, grinding and separation steps according to the size of the ore preparation, the determination of the process parameters and also the flotation process which is one of the ore enrichment processes.

2.THEORETICAL INFORMATION

2.1 GENERAL TERMINOLOGY ON ORE DRESSING

Ore: The rock that is made up of one or more minerals, which are economically valuable and can be consumed directly or after some beneficiation operations, in industry. Ore is the raw material of metal production.

Mineral: A mineral is a naturally formed solid and inorganically crystallized structure that has a homogeneous, specific chemical composition and a specific crystal structure.

Concentrate: It is a product obtained by beneficiation of minerals which are formed as a result of ore dressing or beneficiation processes and which are aimed to be separated from raw ore.

Tenor: Percentage of metal or economically valuable minerals in an ore.

2.2 ORE DRESSING AND BENEFICIATION

Increasing the proportion of base metal minerals in naturally low-grade ores is achieved through ore dressing or beneficiation processes. In these processes, the minerals are partially separated by exploiting the different physical and chemical properties of the base metal and gangue minerals **(Figure 1)**. Ore dressing is a process implemented for economic and technological reasons.

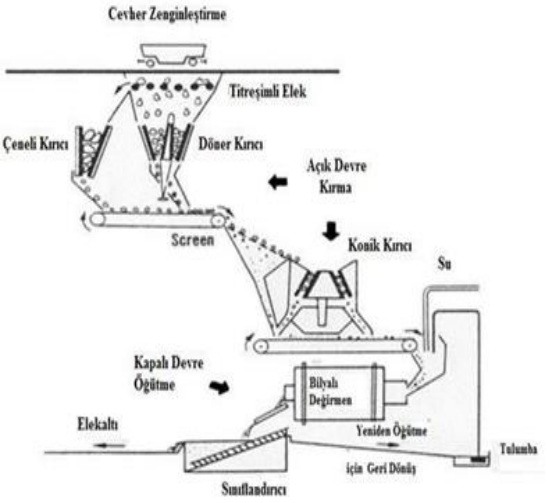


Figure 1. Ore dressing processes.

a) Technological Reasons for Ore Dressing

Some ores need to provide certain conditions (grain size, grade and element content) in order to be technologically produced. For example, for iron ore used in pig iron production; 10 mm < Grain Size < 100 mm is required. For this purpose, crushing, grinding and sintering-pelletizing is applied.

In addition, both P % and the total alkali oxide content ($Na_2O\%+ K_2O\%$) must be < 0.1%.

b) Economic Reasons for Ore Dressing

An ore that is uneconomical as mined can be made economical through ore processing. For example, producing lead metal directly from an ore containing 5% Pb is never economical; however, by increasing the lead grade to 60% through ore processing methods, production can be made economical. Similarly, an ore already economically viable when mined can be enriched in terms of grade and content through appropriate processes, thus further increasing its economic value. For example, it is economical to produce pig iron directly from a 50 % Fe-containing ore, but increasing the iron tenor to over 50 % further increases the economics.

2.2.1 ORE DRESSING PROCESSES

Crushing: It is the coarse size reduction carried out with the help of crushers. It is applied in two stages; coarse crushing (average 100 mm grain size) and fine crushing (1-10 mm grain size).

Grinding: It is the fine size reduction carried out with the help of mills (below 0,1 mm grain size).

Classification: It is the stage in which ore particles liberated by crushing and grinding are separated into fractions according to their sizes or settling velocities

Enrichment: Minerals are separated from each other partially by using density, magnetic, electrical and surface properties.

2.2.1. CRUSHING

Crushing is the first stage of the size reduction process. It is carried out to free the different minerals contained in the ore from each other, providing a size and surface area suitable for the process or intended use. The forces applied in crushing include impact, compression, shear, and friction. The equipment used for crushing is called a crusher. Machines of various shapes, designs, and sizes are used in ore processing. Crushing is applied to grain sizes between 200 and 0.5 cm. Crushing between 200 and 10 cm is called coarse crushing, while crushing between 10 and 0.5 cm is called fine crushing. Jaw, cone, and hammer crushers are the most commonly used crusher types in ore processing plants.

2.2.1.2 GRINDING

Grinding is the final stage of size reduction after crushing. The process is conducted with the aim of freeing one of the various minerals from others in the ore, providing suitable size or surface area or requested size for the purpose of use. The forces applied in grinding are; impact, compression, shear and friction forces. The devices used for grinding are called mills. The mills are selected according to the type of the ore, size of the desired product in the grinding cycles or after grinding. Ball and rod mills are the most commonly used in ore dressing plants. Grinding is carried out as wet or dry depending on the flow of the process and the state of the ore. According to the grinding scheme, the classifier and other process machines in the system are selected. Dry grinding requires about 1.3 times more power than wet grinding.

2.2.1.3 CLASSIFICATION

For removal from grinding circuits or classification according to the size of the material; different classifiers are used according to the applied process, structure of the ore, size, physical and chemical properties. These are known as; sieves, hydrocyclones, mechanical classifiers (spiral classifiers, notched classifiers, solid centrifugal classifiers) and air classifiers. Screening is the process of separating a solid material mixture into components of different dimensions using screens. According to the screen size used in the sieving classifying "mesh number" concept is used. Mesh number indicates the number of holes per unit area (in^2 or mm^2) of a screen. These are classified as; according to the structure of the sieve surface sheet, parallel bar screens, and wire mesh according to their working stationary (fixed grid and stationary arched sieve) and moving (traveling grate, rotary screen, shaking screens and vibrating screens). By sieving, two types of products are obtained, one screen underflow (subsieve) and oversize (oversieve). Industrial sieves are divided into two main divisions: "fixed sieves" and "moving sieves", depending on whether the surface of the workpiece is fixed or movable. The simplest forms of fixed screens are grids. Grids are the most suitable

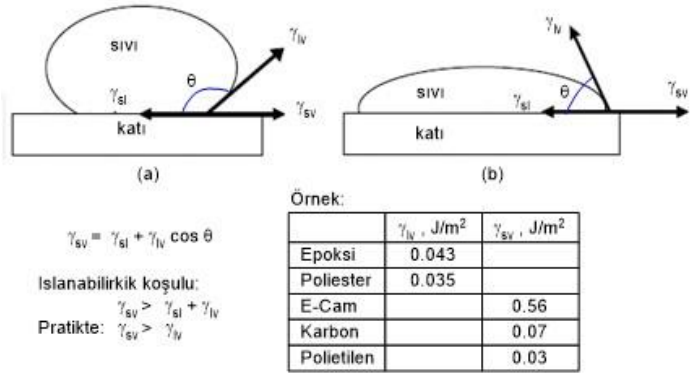
type for large sized items. They are usually made oblique and allow the pieces falling from the grid spacing to separate from the grid as the material on them moves down. Various motions are given to the grid to reduce clogging in the moving screen. With these movements, material is pushed in one direction and sieving is facilitated.

2.2.1.4 FLOTATION

Flotation is derived from the word float. In the ore beneficiation processes, flotation is a method of separating some minerals in an ore from other minerals sunk in the water by floating and removing some of them from the water. In this process, the separation is made by using the differences in the surface properties of the minerals.

Wetting of particles is known to be one of the important parameters affecting many technological processes such as wetting, flotation, agglomeration, solid-liquid separation and dust suppression. In the flotation system consisting of solid, liquid and gas phases, if the solid phase prefers the gas phase relative to the liquid, it is called hydrophobic, if liquid phase is preferred to gas phase, it is called hydrophilic. Hydrophobic minerals are low surface-energy minerals (coal, graphite, sulfur, talc, etc.). The wettability / hydrophobicity and buoyancy properties of the solids were investigated in terms of solid-water and solid-water vapor interfaces, chemical bonds, bulk properties, crystal structure of the solids and reactivity of the solids with water.

The high contact angle (θ) in the solid, liquid and air triple system means that the wetting of the liquid by the liquid is minimal. The forces in the solid, liquid, air triple system are as shown in **Figure 2**. The case where the triple phase is balanced is expressed by Young Equation.



	γ_{lv} , J/m²	γ_{sv} , J/m²
Epoksi	0.043	
Poliester	0.035	
E-Cam		0.56
Karbon		0.07
Polietilen		0.03

Figure 2. The forces in solid, liquid, air triple system and Young Equation

It is possible to determine the wetting and buoyancy characteristics of minerals or associations by several methods, empirical and empirical. The numerical value parameter obtained from these techniques is the critical wetting surface tension, γ_c. At low liquid surface stresses lower than this γc, the mineral loses its hydrophobicity or buoyancy property by being completely wetted by this solution. The surface tension (γ_{SH}) of the liquid used for a good contact angle (between solid-liquid-air interfaces), ie θ> 0, must be greater than the γ_c value of the mineral. This is the first of the conditions required for the successful flotation.

Low surface energetic minerals (γ_c <72 dyn / cm) are wetted by surface-energized fluids lower than the Critical Wetting Surface Energy (γ_c). Selective separation of the two layers in the flotation system is based on whether one of the solids is partially wetted by the flotation solution or completely wetted by the solution while the other layer is not wetted (θ = 0 state). Partially wetted solids clinging to the floating air bubbles.

Two of the most commonly used techniques for measuring the hydrophobicity of minerals or solids, and therefore the wetting of the γ_c value that determines good flotation, are the "contact angle measurement method" and the "flotation method".
Application areas of flotation for ore dressing are; flotation of metallic ores, flotation of non-metallic ores and the cleaning of the solid fuels.
The advantages of flotation are; beneficiation of very fine grained ores, beneficiation of the complex ores, control of the product tenor as desired and the insignificance of specific weight difference of minerals. On the other hand, the disadvantages of the flotation are; the higher cost compared to gravity and magnetic separation methods, loss of metal is high and the grinding costs increase because of the excessive grinding of the ore, and the environmental pollution.

2.2.2.1. Reagents Used in Flotation

Various reagents are added to the flotation medium in order to float or suppress the desired mineral(s) in the flotation. It is possible to sort these reagents as follows.

Collectors: It is a chemical substance that imparts hydrophobicity to surfaces by modifying surface properties through adsorbing to the surfaces of mineral(s).
Frothers: These are foam forming chemicals in flotation circuits. The main goal of the foaming agents is to be able to form a foam of sufficient volume and strength. Foams should be able to explode easily after exiting the flotation cell.
Control Reagents: Reagents that are used to adjust the flotation conditions.
i) Suppressor Reagents: These are the flotation reagents are used to suppress unwanted mineral(s). These reagents reduce collector adsorption on the mineral surface.
ii) Activating Reagents: Reagents that increase collector adsorption to the surface of mineral(s).
iii) Other Control Reagents: Reagents in this group provide; regulation water hardness, bind the harmful ions for the flotation, flocculation or dispersion of some minerals in the pulp.

2.2.2.2.Flotation Machines

Flotation machines are usually composed of successive cells. the residue of previous cells is subjected to flotation in each cell. There is a connection between each cell, or a residual flow plate between cells. Air inlet and mixing operations to the pulp inside the cell are conducted by three types of methods;

- Self-aeriated mechanical cell (Agitation)
- Air blown mechanically agitated cells (Sub-aeration)
- Air blown, air mixed cell (Pneumatic)

These properties are taken into consideration for the construction of various types of cells. Currently the most used cell types in the industry are self-aerated mechanical cell types manufactured by companies such as Denver **(Figure 3)**, Fagergren, Humbold, Massco.

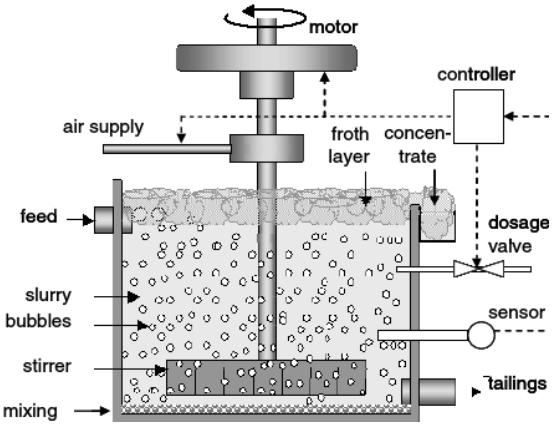


Figure 3. Denver flotation machine

3. EQUIPMENT AND MATERIALS

- Various Crushers and Mills
- Sieves and Screening Device
- Denver Flotation Machine
- Pipette, Washing Bottle, Enamel Containers
- Precise Balance
- Reagents (Collector, Frother)
- Ground Galena Ore

4. EXPERIMENTAL PROCEDURE

4.1 Ore Dressing Experiment

- The galena ore whose particle size distribution is to be examined is weighed and its weight recorded. The weighed mixture is placed in a planetary mill and subjected to high-energy grinding for 10 minutes. After removing the ground particles from the mill, they are weighed again and their weight recorded.
- The sieves are stacked from largest to smallest according to their mesh numbers and placed in the screening device. The ground ore is then fed to the sieve at the top of the sieve set.
- The screws of the sieve set are tightened and the sifting process is carried out by running the device for ten minutes.
- The sieve set is removed from the machine and the amount of material remaining in each sieve and the total amount of sieved material are recorded in Table 1.

4.2 Flotation Experiment

- 200 g of galena ore with a grain size of 200 μm is weighed and adjusted to a 20% solids content based on the volume of the flotation cell. The cell is placed in the flotation machine, the machine is started, and the mixing process is continued for 5 minutes.1-2 drop of the appropriate collector used for galena ore, is added to the pulp and mixing is continued for 5 minutes.
- The appropriate collector used for galena ore is added 1-2 drops to the pulp and the mixing process is continued for another 5 minutes.
- For the flotation of minerals whose surfaces have become hydrophobic, one drop of a frother is added to the medium to generate froth. After the addition of the frother, mixing is continued for another 1–2 minutes.
- Minerals with hydrophobic surfaces accumulate on the surface as foam. The foam is skimmed off and transferred to a separate container to obtain the concentrate.
- The resulting concentrate is dried in an oven at 105°C. The weight of the dried concentrate is determined using a precision scale.

5. ASSIGNMENTS

1. Write the objective and procedure of the experiment. **(10 points)**.
2. Record the results of each sieve analysis in **Table 1 (25 points)**.
3. By using the sieve analyzes obtained from the experimental procedure, generate the sieve analysis charts of the input and output products and draw the total subsieve

and oversieve curves. Determine the average grain size from the intersection of the two drawn lines **(25 points)**.

4. Find the theoretical average grain size by using the **Equation 1** and compare the theoretical grain sizes found at the intersection of the straight lines **(20 points)**.
5. Determine the enrichment ratio (Z) by weighing the concentrate and the residue after flotation (Z=Ore Fed/Concentrate) **(20 points)**.

$$\frac{\Sigma(X.M)}{100} \quad (1)$$

(X= Sieve interval or diameter (same as grain size), M=% grain class weight)

Table 1 Sieve analysis data and calculations

Sieve Radius (mm)	**Weight**		**Cumulative oversieve (%)**	**Cumulative subsieve (%)**
	g	**%**		
Total				

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EXPERIMENT NAME:PYROMETALLURGY

1. PURPOSE 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 (direct reduction).

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 iron and steel sector provides raw materials to many important industries including construction, infrastructure, automotive, white goods and machinery industries. Therefore, a strong iron and steel sector is directly associated with the level of industrialization of a country [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 modern blast furnace produces over 10,000 tons of liquid crude iron per day [4-6]. The other reduction method is the process in which iron oxides are converted into metallic state in the solid phase with solid or gas reductants without melting. Part of the reduction in the blast furnace occurs with solid carbon in the lower regions of the furnace. In this method, known as direct reduction, the final product is sponge iron with a high metallization rate. Sponge iron is obtained by reducing iron ore in powder, piece or pellet form below the melting temperature (at 950°C - 1100°C) using a gas or solid reducer. The total iron content of sponge iron is generally over 85% and the degree of metalization of sponge iron is between 90-95%. The carbon content of sponge iron varies between 1-2.5% and the gangue content varies between 2-4%. Apparent density of the sponge iron is ≤4 g/cm3 [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%) superior resistance to crumbling, uniform gas distribution and excellent reducibility of the pellets, make them one of the blast furnace's essential inputs, despite their higher cost relative to other charge elements. Furthermore, utilization of pellets in the blast furnace provides positive results including reduced coke consumption, decreased amount of slag, and increased production rate [9].

Thermodynamic Investigation of the 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 CO_2 gas with carbon, due to the fact that CO2 gas, produced by combustion of carbon, is unstable at high temperatures (see Ellingham Diagram).

$C\,(k) + O_2\,(g) = CO_2\,(g)$ (1)

$CO_2\,(g) + C\,(k) = 2CO\,(g)$ (Boudouard Reaksiyonu) (2)

The reduction of hematite to iron with CO/CO_2 gas mixture, takes place in three stages. The temperature-dependent empirical expressions (3), (4), and (5) of the standard free energy change (ΔG^o_T) for these reactions and reactions are given below.

$3Fe_2O_3 + CO = 2Fe_3O_4 + CO_2$ $\quad \Delta G^oT = 4.376.000 + 1454{,}91\ T$ (3)

$Fe_3O_4 + CO = 3FeO + CO_2$ $\quad \Delta G^oT = 1.610.900 - 27.54\ T$ (4)

$FeO + CO = Fe + CO_2$ $\quad \Delta G^oT = -18.700 + 22{,}46\ T$ (5)

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

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

$$\Delta G_T^0 = -RT\ln K_p \quad (7)$$

$$K_p = \frac{a^2_{Fe_3O_4}.P_{CO_2}}{a^3_{Fe_2O_3}.P_{CO}} \quad (8)$$

When Fe_3O_4 and Fe_2O_3 are assumed to be pure, their activities are equal to "1". In this case;

$$\Delta G_T^0 = -RT\ln\frac{P_{CO_2}}{P_{CO}} = RT\ln\frac{P_{CO}}{P_{CO_2}} \quad (9)$$

From this, the $\left(\frac{P_{CO}}{P_{CO_2}}\right)$ ratio which balances the reduction reactions at the T_1 temperature can be calculated. The basic thermodynamic conditions for the reduction of iron oxides are $\Delta G_T < 0$ and the $\left(\frac{P_{CO}}{P_{CO_2}}\right)$ ratio of the environment is greater than the equilbirium $\left(\frac{P_{CO}}{P_{CO_2}}\right)$ ratio. The Baur-Glaessner diagram and Boudouard curves drawn using reactions (2) and (9) are shown in **Figure 2.1**.

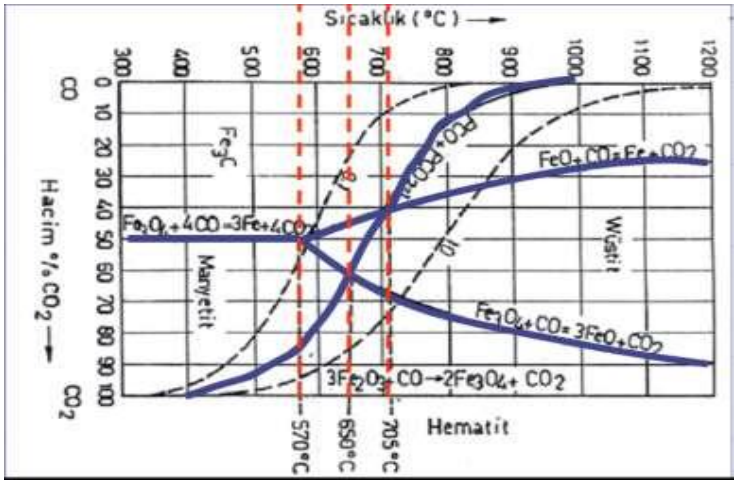

Figure 2.1 Baur-Glaessner Diagram and Boudouard curves.

According to **Figure 2.1**, the metallic iron reduction of iron oxides at a total pressure of Pco + Pco2 = 1 atm is only possible above 705 °C. At temperatures below 705 °C Fe_2O_3 may transform to Fe_3O_4 and Fe_3O_4 may transform to FeO. Additionally as the total pressure of $P_{CO} + P_{CO2}$ increases, the reduction temperature of the iron oxides is forced to shift to higher temperatures [4-6].

Kinetic Investigation of the Reduction Reaction

The reaction rate in a reaction can be determined by the change in the quantitative properties of a substance participating in the reaction over time. 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. Heterogeneous reactions may be gathered in five groups depending on their interface: solid-gas, solid-liquid, solid-solid, gas-liquid, liquid-liquid [9-12]. The occurrence of heterogeneous reactions between more than one phase is as follows [9,10]:

1. Transport of the reducing gas to the reaction interface (diffusion)
2. Interfacial reactions.
3. Adsorption of the reducing gas to the reaction interface.
4. Chemical reaction at the interface.
5. Desorption of the products occurred after the reaction.
6. 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 as “diffusion controlled”, “chemical reaction controlled” and “mixed controlled” if the rate constants of these two steps are equal. 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 [9-12]. The geometric shape of the solid reacting with the liquid or gas plays an important role in determining the rate of the relevant reactions. In reactions carried out with spherical-shaped solids, the reaction interface is quite evident. As the reaction progresses, the reaction interface moves from the outer surface to the center of the part and a clear boundary is formed between the reacted and unreacted part. This model is called the reaction topochemical model or shrinking core. **(Figure 2.2)**.

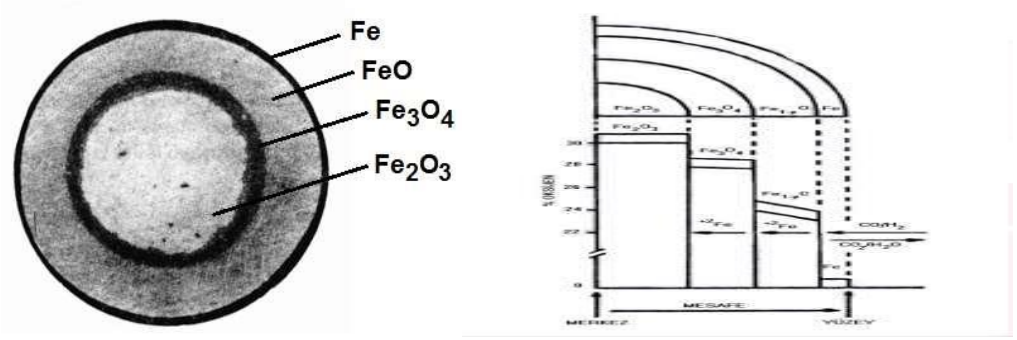

Figure 2.2 Schematic representation of the reduction of the hematite particle and the topochemicality in reduction [5-7].

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. 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 [9-12].

3. EXPERIMENTAL STUDIES

Tools, Devices and Materials

1. Iron ore pellets and coal (coke or lignite dust)
2. Furnace
3. Precise scale
4. Graphite crucible and charging rod
5. Pens and tongs

Experimental Procedure

- Among the pellets produced from iron ore concentrates, identical pellets are selected and weighed.
- Using the total reduction reaction, the amount of carbon required for the reduction of these pellets and, accordingly, the amount of coal to be used are calculated. 150% of the theoretical amount of coal calculated on the basis of total pellet weight is weighed and used.
- When the tube reaches 1000°C, the pellets are buried in the coke bed prepared in the crucible and charged to the furnace with the charging rod.
- At the 10th, 20th and 30th minutes respectively, one pellet is taken from the furnace, cooled to room temperature and weighed with a precise scale.
- Reduction percentages of pellets are calculated with the formula given below.

$$\text{Reduction \%} = \frac{\text{Removed Oxygen}}{\text{Removable Oxygen}} * 100$$

The chemical compositions of the pellets and coke used in the experiments are given below.

	Fe_3O_4	SiO_2	Al_2O_3	S	Mn	CaO	MgO	K_2O	Na_2O	P	C
Pelet	94,60	2,20	0,75	0,40	0,10	0,60	0,58	0,07	0,04	-	-
Kok	-	8,26	4,30	0,54	-	1,24	0,35	0,29	0,08	0,14	80,30

4. ASSIGNMENTS

- Write the purpose and procedure of the experiment. (15 points)
- Calculate the theoretical amount of coal required for the reduction of each pellet and the % reduction ratios obtained. (20 points)
- Draw and evaluate the % reduction rates over time graph. (15 points)
- Calculate the reduction rate to be achieved at the 15th and 45th minutes. (15 points)
- 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 observe that difference in the partially reduced iron oxide particle? Schematically draw. (15 points)

- Using the reactions (4) and (5), plot the % CO - temperature graphs for 700, 800, 900 and 1000 °C temperatures in the total PCO + PCO2 = 1 atm environment and name the zones. (20 points)

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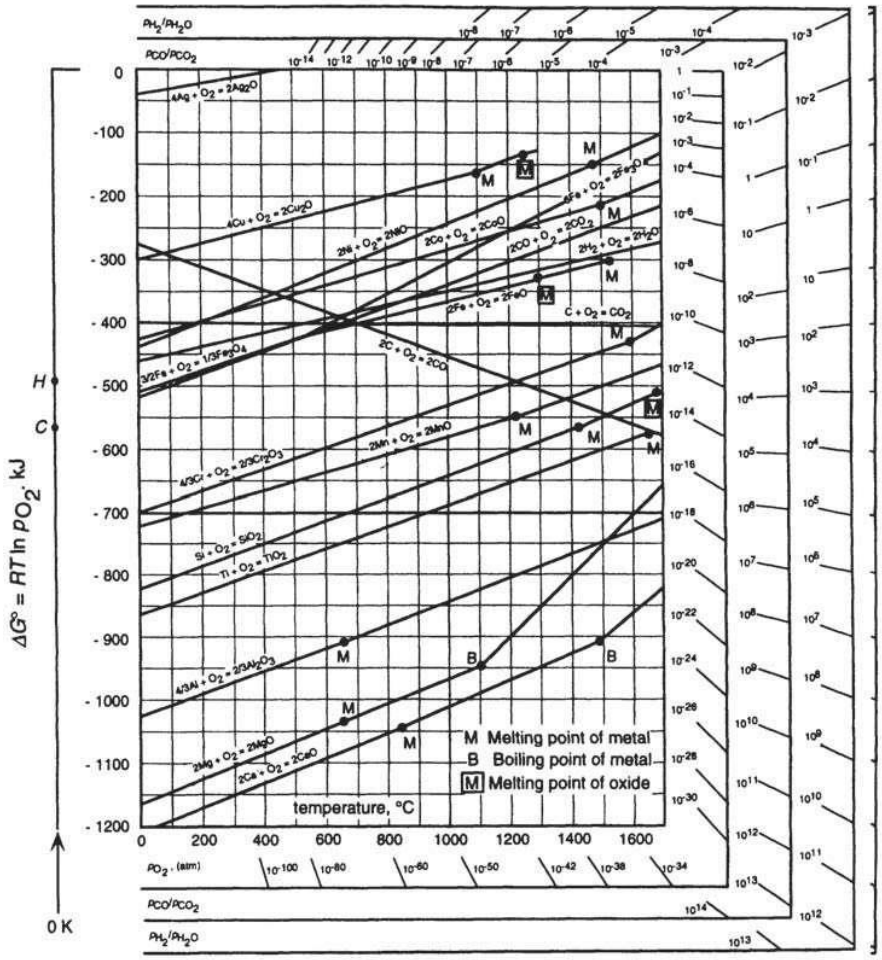
Supplementary: Ellingham Diagram



EXPERIMENT NAME: Waste Metal Recycling and Casting

1.Objective of the Experiment

To study the manufacturing of a part using the sand casting method and the recycling processes of scrap aluminum metal.

2.Introduction

In the manufacturing of metal parts, fundamental methods such as casting, plastic forming, machining, welding, and powder metallurgy are used. Casting is the process of melting metals or alloys, pouring them into a mold, and allowing them to solidify. This process not only shapes the part but also determines the material's microstructure and mechanical properties. Casting offers high efficiency, especially in the production of complex parts, as the liquid metal can be molded into a form close to the final product.

Casting is widely used in the automotive industry (engine blocks, pistons), aerospace (turbine blades), machine manufacturing (pump housings, gears), and the energy sector (generator, turbine parts). Additionally, cast parts have extensive applications in many other fields such as construction, medicine, and agriculture. Due to this versatility, casting is considered one of the fundamental pillars of modern industry.

All casting processes fundamentally involve five main steps:

- **Melting:** The solid metal or alloy is heated in various furnaces, such as induction, arc, or crucible furnaces, to a temperature above its melting point to achieve the necessary fluidity for casting.
- **Molding:** A mold, which is the negative form of the part, is prepared using a pattern designed by considering the geometry of the part to be produced, shrinkage after solidification, and necessary allowances for subsequent operations (e.g., machining).
- **Pouring:** The molten liquid metal is carefully poured into the mold using ladles or crucibles. Controlling the metal's temperature, flow rate, and turbulence is crucial at this stage.
- **Solidification and Cooling:** The liquid metal poured into the mold begins to solidify by losing heat and takes its final shape. This is the most critical stage, directly affecting the material's microstructure and mechanical properties.
- **Finishing (Post-Processing):** The cooled part is removed from the mold. Elements of the casting system such as the gating system, riser, and vent are separated from the part. Surface roughness is cleaned using methods like sandblasting or tumbling. If necessary, additional processes such as heat treatment to improve mechanical properties or machining to ensure dimensional accuracy are applied.

2.1 General Advantages and Limitations

Advantages:

- **Design Flexibility:** Allows for the production of parts with almost unlimited geometric complexity.
- **Material Variety:** Nearly all metals and alloys, such as cast iron, steel, aluminum, and copper alloys, can be cast (casting is also a common manufacturing method for glass, ceramic, and polymer materials).
- **Size Range:** Production is possible in a very wide range of sizes and weights, from small jewelry pieces of a few grams to ship engine blocks weighing hundreds of tons.
- **Cost-Effectiveness:** It is more economical than other methods, especially for mass production and complex parts.
- **Isotropic Properties:** Cast parts generally exhibit non-directional (isotropic) mechanical properties, which means the part shows similar strength in all directions.

Limitations:

- **Dimensional Accuracy and Surface Finish:** Especially in methods like sand casting, dimensional tolerances are wider, and the surface quality is lower. This often requires additional machining.
- **Casting Defects:** Due to the nature of the solidification process, there is a risk of defects such as gas porosity and shrinkage cavities, and controlling these defects requires experience and process control.
- **Mold Cost:** The initial investment cost of permanent metal molds, in particular, is high, making it uneconomical for low-volume production.
- **Thin Sections:** In some casting methods, it can be difficult to fill very thin sections without defects.

2.1.1 Sand Casting Process and Principles

The casting process is divided into two main groups based on the structure of the mold used: **expendable molds** and **permanent molds**.

- **Expendable Molds:** These molds are single-use and are broken and destroyed to remove the product after each casting operation. Sand casting, shell molding, ceramic molding, plaster molding, and investment casting are methods that fall into this category.
- **Permanent Molds:** As the name suggests, these molds can be used repeatedly. Typically made of metal, these molds are ideal for high-volume production. Permanent mold casting, die casting, centrifugal casting, and continuous casting are examples of this group.

The sand-casting process consists of a series of precise and interrelated steps:

Step 1: Pattern and Core Design and Preparation

- **Pattern:** This is a positive replica of the part to be produced. During its design, allowances such as "shrinkage allowance" are added to compensate for the volumetric contraction of the metal during solidification, and "machining allowance" is added because surfaces may need to be finished later. The pattern is usually made from materials like wood, wax, plastic, or metal.
- **Core:** It is used to create internal cavities, channels, or complex external undercuts within the part. Cores are generally made from special sands reinforced with binders like resin. The essential properties a core must have are sufficient mechanical strength, high gas permeability to allow gases formed during casting to escape, and collapsibility (the ability to crumble) for easy removal from inside the part after casting is complete.

Step 2: Molding

- The molding process is carried out in a two-part metal frame called a "**flask**".
- One half of the pattern is placed in the lower flask (**drag**), and specially prepared molding sand is filled around it and compacted.
- The same process is repeated for the other half of the pattern in the upper flask (**cope**).
- When the pattern is carefully removed from the sand, a mold cavity with the negative geometry of the part is formed. At this stage, channels such as the "**gating system**" for the liquid metal to enter the mold, the "**riser**" which acts as a reservoir to compensate for metal shrinkage, and the "**vent**" to allow gases to escape are also formed in the sand.
- If the part has internal cavities, the prepared cores are placed in their designated seats within the mold cavity at this stage. Finally, the lower and upper flasks are assembled, making the mold ready for pouring.
- **Figure 1** shows a basic illustration of a sand mold.

Step 3: Pouring and Shakeout

- The molten metal is poured into the prepared mold through the sprue of the gating system.
- After the metal solidifies in the mold and cools to near room temperature, the sand mold is broken, typically on vibrating shakers, to remove the part. This process is called "**shakeout**". Sand molds are single-use, but a large portion of the sand can be reclaimed by sieving and adding new binders.

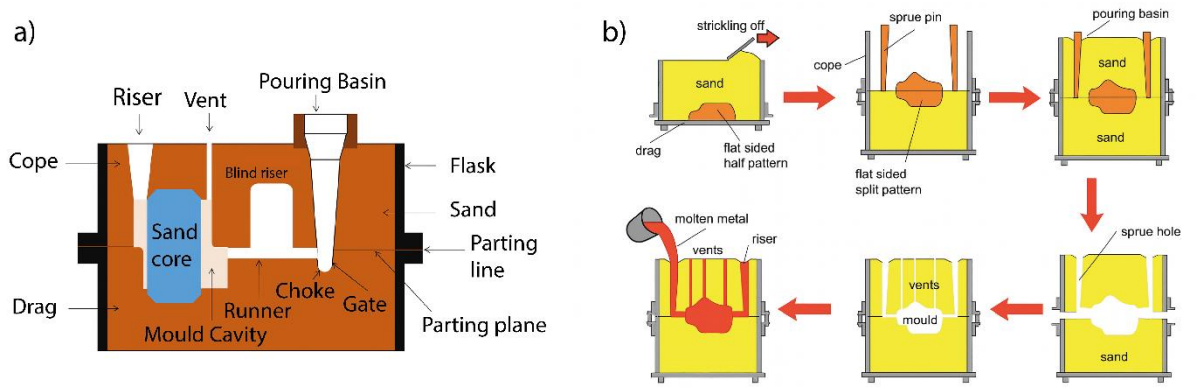


Figure 1 a) A schematic of a fundemental sand mold. b) The process of sand mold preperation.

The mold consists of three basic components:

- **Sand (Aggregate):** Forms the main skeleton of the mold and must be resistant to high temperatures.
    - **Silica Sand (SiO_2):** The most common type of sand used in foundries. Its main advantages are availability, low cost, and high melting point (refractoriness). However, its significant expansion at high temperatures can negatively affect dimensional accuracy.
    - **Other Sands:** Special sands like zircon sand offer superior properties such as higher thermal conductivity and lower thermal expansion, but they are more expensive and preferred for special applications.
- **Binders:** These are materials that bind the sand grains together, giving the mold its shape and strength.
    - **Clay (Usually Bentonite):** The most traditional and economical binder, which, when activated with water, coats the sand grains and provides them with plasticity and green strength.
    - **Chemical Binders:** Materials like resins (furan, phenolic) or sodium silicate harden through chemical reactions, creating much stronger and dimensionally stable molds.
- **Additives:** These are materials added in small quantities to improve the properties of the molding sand. For example, coal dust improves the casting surface finish, while graphite powder prevents the liquid metal from sticking to the sand.

One of the most important properties of sand molds is their "breathing" capability, also known as **gas permeability**. This property allows the gases generated during the casting process to escape easily from the mold. The high temperature of the liquid metal causes a significant amount of gas to form by vaporizing moisture in the mold and burning organic binders. If these gases cannot escape through the voids between the sand grains, they become trapped in the liquid metal. This leads to serious defects in the cast part, such as "**gas porosity**" or "**pinholes**".

Coarse-grained sands with high permeability allow gases to escape easily but result in a rougher surface finish. Conversely, fine-grained sands used for a smoother surface finish have lower gas permeability. This engineering trade-off requires determining the optimal sand mold formulation based on the specific requirements of the part being produced.

Sand casting is divided into different methods based on the binder and process used in molding. The most common are:

- **Green sand molding:** In this method, the molding sand, prepared with a mixture of **water and clay (bentonite)**, is used for casting directly without any drying process. It is the most economical and fastest method. However, the moisture in the mold increases the risk of gas defects due to vaporization, and the mold strength is lower compared to other methods.
- **Dry sand molding:** Molds prepared with the green sand method are dried in an oven at approximately 150-350 °C before pouring. This process eliminates gas defects caused by steam. Dry molds offer a better surface finish as they have higher strength and erosion resistance. The disadvantage is the increased cost and production time due to the additional drying process.
- **CO_2 process (chemically bonded):** In this method, the mold is hardened instantly by passing carbon dioxide (CO_2) gas through a sand mixture containing sodium silicate as a binder. This method produces very high-strength and dimensionally accurate molds without the need for oven drying. On the other hand, the working life of the prepared sand is short, and cleaning the mold after casting is more difficult than with other methods.

An analysis of the advantages and disadvantages of the sand-casting method:

Advantages:

- **Low Cost:** Mold materials such as sand and clay are inexpensive, and the sand itself can be reused, making it one of the most economical casting methods.
- **Material and Size Flexibility:** It is suitable for almost all castable alloys such as steel, cast iron, and aluminum. It allows for the production of parts in a very wide size range, from a few grams to hundreds of tons.
- **Complex Geometries:** The production of complex-shaped parts with internal cavities is possible through the use of cores.

Disadvantages:

- **Low Surface Quality and Dimensional Accuracy:** Due to the size of the sand grains, the surface roughness is greater compared to other methods. Dimensional tolerances are wider, so additional machining is often required for precise dimensions.

- **Low Production Rate:** The molding, pouring, and cooling processes are time-consuming, making it less efficient for high-volume mass production compared to methods like die casting.
- **Thin Section Limitation:** Due to the fluidity of the liquid metal and the cooling effect of the mold, casting very thin sections (usually less than 3 mm) is difficult.

2.1.2 Liquid-Solid Phase Transformations and Their Relation to Casting

Casting is essentially a liquid-to-solid phase transformation. This transformation determines the final part's microstructure and, consequently, its mechanical properties such as strength, ductility, toughness, and hardness. Therefore, understanding the principles of solidification is fundamental to controlling the casting process and identifying potential defects.

In the liquid state, metal atoms are in an amorphous structure and move randomly. As the metal loses heat, these disordered atoms begin to transition into a regular and repeating structure, the crystal structure, where they are energetically more stable. This transition occurs in two fundamental stages that form the grain structure of the cast part: (1) **nucleation** and (2) **growth**.

Nucleation, the first step of solidification in casting, is the formation of very small and stable solid particles within the liquid metal. This process occurs through two basic mechanisms. **Homogeneous nucleation** is the formation of stable nuclei by the random coming together of atoms in completely pure and homogeneous liquid metals. However, this is a very rare occurrence and is only possible with significant cooling of the liquid below its solidification temperature (**undercooling**). Therefore, it is mostly observed in laboratory settings. In industrial casting applications, **heterogeneous nucleation** almost always occurs. Here, foreign surfaces such as the mold wall, slag particles, or specially added inoculation materials provide a starting point for nucleus formation. These surfaces lower the energy barrier by allowing atoms to use an existing surface instead of forming a nucleus among themselves, requiring less undercooling.

To improve the mechanical properties of metal parts, a fine and homogeneous grain structure is generally preferred. For this purpose, particles that increase the nucleation rate, such as Al-Ti-B, are added to the liquid metal. This process, called **inoculation**, promotes dense nucleation and significantly improves the quality of cast parts.

After stable nuclei are formed, the solidification process continues with the **growth** of these nuclei. The crystal growth rate varies depending on the direction of heat transfer and the chemical composition of the alloy. Especially in alloys, the growth rate is not equal in all directions; structures that grow faster in specific crystallographic directions form branched crystals resembling tree branches. These structures, called "**dendrites**," merge to form the basic grain structure of the cast part. The regions between the dendrite arms are the last areas to solidify, rich in alloying elements, and are prone to the formation of defects like micro-shrinkage.

The grain structures formed during solidification are observed in two main forms, depending on the heat flow:

- **Equiaxed Grains:** These grains form in situations where the liquid cools at an equal rate from all directions and heat transfer is not directional (typically in regions like the center of the casting). They have a roughly spherical or polyhedral structure.
- **Columnar Grains:** These are long, thin grains that grow opposite to the direction of heat flow, occurring when heat is extracted in a single direction (e.g., from the cold mold wall towards the center). They are generally seen near the mold walls.

The **cooling rate** is the most important parameter controlling the solidification kinetics and the resulting microstructure. The solidification process can be thought of as a competition between the "nucleation rate" and the "growth rate". The cooling rate changes the balance of this competition, determining the final grain size.

- **Fast Cooling** (e.g., Metal Mold / Permanent Mold Casting): A high cooling rate creates a large undercooling in the system. This triggers the simultaneous formation of a large number of stable nuclei (nucleation rate increases). However, since the system solidifies rapidly, these numerous nuclei do not have time to grow (growth rate is limited). As a result, a "**fine-grained**" microstructure is obtained. Fine-grained structures generally offer superior mechanical properties, such as higher strength and toughness, because the grain boundaries hinder dislocation movement.
- **Slow Cooling** (e.g., Sand Mold): A low cooling rate causes only a small amount of undercooling in the system. This leads to the formation of fewer nuclei (nucleation rate is low). These few nuclei have ample time to grow by collecting atoms from the surrounding liquid during the slow cooling period (growth rate is dominant). Consequently, a "**coarse-grained**" microstructure is formed.

This relationship allows the casting engineer to control the cooling rate by adjusting parameters such as the mold material (heat-insulating sand or heat-conducting metal), part section thickness, and pouring temperature, thereby achieving the desired microstructure and mechanical properties. This is the most concrete and important example of the fundamental "process-structure-property" relationship of materials science in casting.

Chvorinov's Rule is an empirical relation stating that the total solidification time (t_s) of a casting is proportional to the square of the ratio of the casting's volume (V) to its surface area (A): $t_s = B(V/A)^2$. Here, B is the mold constant. This rule indicates that among parts with the same volume, those with a smaller surface area (more compact, spherical shapes) will cool and solidify more slowly. This principle plays a fundamental role in the design of risers used to prevent shrinkage defects.

2.2 Common Casting Defects and Prevention Strategies (with a focus on Aluminum)

Non-ideal casting conditions can lead to various defects in cast parts, causing problems with performance, sealing, and aesthetics. This section will discuss the formation mechanisms of defects frequently encountered (see **Figure 2**), particularly in aluminum casting, and methods

to prevent them. The fundamental principles presented here will also be useful for understanding defects encountered in the casting of other metals.

Gas Porosity

Liquid aluminum has a high capacity for dissolving hydrogen. This hydrogen typically originates from water vapor, combustion products, or oils on the charge materials. As aluminum begins to solidify, its hydrogen solubility decreases by about 20 times. This sudden drop causes the hydrogen gas, which is rejected by the dendrites, to become trapped in the liquid and form bubbles when it reaches a critical concentration. These bubbles become entrapped within the solidifying metal, creating defects known as gas pores, which are usually spherical in shape.

The main methods to prevent this are:

- **Degassing:** This is the most effective way to remove hydrogen from the molten metal. Typically, inert gases (e.g., Argon or Nitrogen) are injected into the liquid metal using a graphite rotor. The rotating rotor breaks the inert gas into thousands of small bubbles. As these small bubbles rise to the surface through the liquid metal, they attract and carry the dissolved hydrogen to the surface. The hydrogen is then released into the atmosphere, reducing the gas content in the metal to safe levels.
- **Raw Material Control:** To prevent gas defects at the source, it is essential to pay attention to the quality of the raw materials used. Charge materials like scrap and ingots should be clean, dry, and free of oil. This is because moisture or oils on the surface of these materials vaporize and turn into hydrogen during melting. Materials can be preheated before being charged into the furnace to completely remove any moisture.
- **Melting Practice:** The solubility of hydrogen in liquid metal is directly proportional to temperature. Therefore, the melting and holding temperatures of the metal should not be unnecessarily high. Keeping temperatures as low as possible and under control reduces hydrogen absorption. Additionally, minimizing the metal's contact with the atmosphere and avoiding turbulent motion during pouring helps prevent hydrogen from mixing into the liquid.

Shrinkage Porosity

Like almost all metals, aluminum shrinks in volume as it transitions from a liquid to a solid state because its density increases. This volumetric shrinkage occurs in three stages: shrinkage during the cooling of the liquid, shrinkage during solidification, and contraction as the solid cools to room temperature. The volume loss that occurs during solidification will cause a cavity to form in that region if additional liquid metal cannot be supplied to the solidifying area. These cavities, characterized by their rough, dendritic internal surfaces, typically occur in the last sections to solidify, such as thick sections or "hot spots".

The main methods to prevent this are:

- **Riser Design and Use:** Risers are extra liquid metal reservoirs placed on top of or next to the casting. According to Chvorinov's rule, the riser must be designed to solidify later

than the section of the part it feeds. This way, the volume loss occurring as the part solidifies is compensated by the liquid metal in the riser, ensuring the shrinkage cavity forms within the riser instead of the part.

- **Directional Solidification:** This involves optimizing the design of the casting and the gating/riser system so that solidification begins at the point farthest from the riser and progresses gradually towards it. This ensures that the solidification front is continuously fed with liquid metal.
- **Use of Chills:** To accelerate the cooling of thick sections or hot spots, metal parts with high thermal conductivity (chills) are placed in those areas of the mold. This causes that region to solidify earlier, reducing the risk of shrinkage.

Gas porosity and shrinkage porosity, the two most stubborn defects in aluminum casting, are fundamentally two different outcomes of the same physical principle: the phase change during solidification. Hydrogen porosity occurs when a "solute" (hydrogen) is rejected from the metal due to a drop in solubility, whereas a shrinkage cavity forms because the "solvent" (aluminum) itself increases in density. It is no coincidence that these two defects often occur together. The hot spots most prone to shrinkage are also the last regions to solidify in the casting. Hydrogen, rejected by the solidifying dendrites, also accumulates in this last remaining pool of liquid metal. Therefore, since the same region suffers from both a deficit of liquid metal needed to compensate for volumetric shrinkage and the highest concentration of hydrogen, these two defect mechanisms often combine in the same location to form larger and more complex voids.

Hot Tearing

This is a type of crack that occurs towards the end of solidification, in a "mushy" zone where dendrites are in contact, but a thin film of liquid still exists between them, and the material exhibits both solid and liquid properties. At this stage, if the tensile stresses from thermal contraction of the solidified skeleton exceed the material's very low strength and ductility at that moment, tears occur in the intergranular regions. Sharp internal corners, abrupt changes in cross-section, and mold hindrance of contraction can trigger this defect. The main measures to prevent this defect are:

- **Part and Mold Design:** Sharp corners should be avoided in the part design, replaced with generous radii, and abrupt section changes should be made with smooth transitions.
- **Alloy Composition:** Alloys with a wide solidification range are more prone to hot tearing. Optimizing the alloy composition and using grain-refining elements (to create more grain boundaries and distribute stress) can reduce the tendency.
- **Casting Parameters:** Keeping the pouring temperature as low as possible can help reduce thermal gradients and, consequently, thermal stresses.

Other Defects

- **Inclusions:** These are foreign materials present in the liquid metal that become trapped in the part's structure after solidification. The most common in aluminum are aluminum

oxide (Al_2O_3) films that form instantly on the metal's surface and are folded into the metal by turbulence. Slag, refractory particles, or sand grains breaking off from the mold can also form inclusions. To prevent this, clean melting practices, slag removal, and filtering the liquid metal through ceramic foam filters before pouring are necessary.

- **Cold Shut:** This is a seam-like surface defect that occurs when two or more streams of liquid metal flowing from different directions fail to fuse completely due to an oxide film on their surfaces or low temperature. Low pouring temperature, slow mold filling rate, or improper gating design can cause this defect.

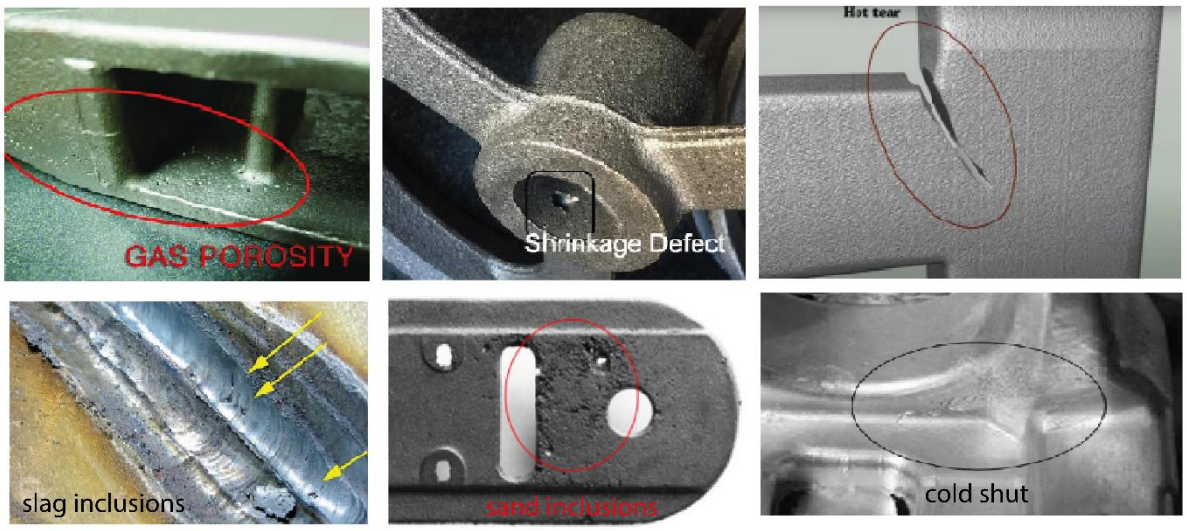


Figure 2 Defects frequently encountered in casting.

2.3 **Scrap Aluminum Recycling**

The most striking and important aspect of aluminum recycling is the immense economic and environmental benefits it provides:

- **Energy and Cost Advantages:** Recycling aluminum requires up to 95% less energy compared to producing primary aluminum from bauxite ore. Primary production is extremely energy-intensive due to the electrolysis process, whereas recycling is fundamentally a melting process. This massive energy difference makes recycling one of the most efficient industrial processes, not only for environmental sustainability but also in terms of cost.

- **Reduction of Greenhouse Gas Emissions:** The 95% reduction in energy consumption translates to a similar decrease in greenhouse gas emissions. This allows the aluminum industry to directly contribute to combating climate change by shrinking its carbon footprint.

- **Conservation of Natural Resources:** Bauxite ore, the raw material for aluminum, is a finite natural resource obtained through mining. Mining activities can cause significant environmental damage, such as deforestation, soil erosion, and loss of biodiversity. Recycling helps conserve these natural resources and ecosystems by reducing the need for new bauxite mining.

- **Circular Economy:** One of aluminum's most remarkable properties is that it can theoretically be recycled infinitely without any loss of quality or metallurgical properties. This feature makes it a perfect candidate for transitioning from a linear "take-make-dispose" economy to a "circular economy" model where resources are continuously kept in the loop.

Aluminum recycling can be viewed not just as a simple waste management activity, but as a strategic "**energy banking**" system. The vast amount of energy loaded into aluminum during primary production essentially remains "embedded" or "stored" in the metal itself. The recycling process repeatedly releases this embedded energy for a very low cost, only about 5% of the original cost. From this perspective, a piece of aluminum scrap is not just waste, but an energy reserve and an economic asset for future production.

The recycling process, also known as secondary aluminum production, consists of a series of physical and metallurgical operations:

1. **Collection and Sorting:** The process begins with the collection of aluminum scrap from end-of-life products (beverage cans, automotive parts, window profiles) or manufacturing processes (chips, cutting scrap). The collected scrap is sorted at recovery facilities according to alloy types (casting, wrought, etc.) and forms.
2. **Preparation (Pre-treatment):** The sorted scrap is prepared for the melting process. This stage includes shredding or grinding the scrap into smaller pieces, removing organic contaminants like paint, plastic coatings, and oil through thermal methods, and separating other metallic impurities like iron using powerful magnets.
3. **Melting and Refining:** The cleaned and prepared scrap is melted in large-capacity furnaces such as reverberatory or rotary furnaces. During melting, the chemical composition of the liquid metal is analyzed. A refining process is carried out by adding salt-based chemicals called flux to remove unwanted elements (impurities) and dissolved gases, and to float oxide films.
4. **Ingot Casting:** The refined liquid aluminum, with its chemical composition brought to the desired standards, is typically cast into ingots using continuous casting machines. These ingots constitute the secondary raw material for foundries, ready for use and of the same quality as primary aluminum.

Although the infinite recyclability of aluminum is a theoretical ideal, practical applications present some metallurgical challenges. Variations in the composition of scrap entering the recycling loop and the impurities they contain can negatively affect the quality and mechanical properties of the cast part.

"Tramp elements" or **impurities** are undesirable elements that accumulate in recycled aluminum alloys, which are not present in the original specification or exceed permissible limits. These elements typically enter the system when different aluminum alloys (e.g., cast and wrought alloys) are collected and melted together, or when foreign materials within the scrap (steel bolts, copper wire, etc.) are not completely separated.

One of the most common and harmful impurities in secondary aluminum is **iron**. Iron, which has extremely low solubility in aluminum, forms brittle, needle- or plate-shaped Al-Fe-Si intermetallic compounds in the microstructure during solidification. Similarly, the accumulation of elements like copper (Cu), zinc (Zn), and manganese (Mn) above normal levels can lead to the formation of undesirable phases, a decrease in corrosion resistance, and changes in heat treatment behavior.

Specifically, the needle-like and plate-shaped intermetallic phases formed by iron act as discontinuities within the aluminum matrix, causing stress concentrations under mechanical load. These regions become initiation and propagation points for cracks. As a result, the material's plastic deformation capacity, i.e., its **ductility** (elongation at break) and **impact strength (toughness)**, is significantly reduced; the material exhibits more brittle behavior. Furthermore, these intermetallic phases act as crack initiation sites under cyclic loads, severely reducing fatigue resistance. Some impurities can also increase the tendency for casting defects like hot tearing by altering the metal's fluidity or solidification mechanism.

To manage these challenges associated with using recycled scrap, various metallurgical strategies are applied during the melting and refining stages:

- **Use of Flux:** Special salt mixtures (fluxes) added to the liquid metal during melting both clean the metal surface by dissolving oxide films and facilitate the separation of some solid inclusions by collecting them in the slag.
- **Alloying and Correction:** Based on chemical analyses performed after melting, the alloy's composition is adjusted to meet standards. More importantly, "corrective" elements can be added to neutralize the effects of harmful elements like iron. For example, adding a controlled amount of Manganese (Mn) to the liquid metal promotes the transformation of harmful needle-like Al-Fe-Si phases into a less detrimental compact or "Chinese script" morphology.
- **Filtration:** Passing the liquid metal through porous ceramic foam filters just before pouring it into the mold significantly increases the cleanliness of the metal by physically trapping solid oxide films and other small inclusions.

In conclusion, producing high-quality cast parts from scrap aluminum depends on careful control of the entire process:

- **Input Material Control:** The most critical step in the process is to use the cleanest and best-sorted scrap possible. A well-managed stock of scrap procured from reliable sources simplifies all subsequent refining operations and reduces costs.
- **Precise Process Control:** Precise monitoring and control of melting and pouring temperatures, the effectiveness of the degassing process, and alloying steps are mandatory to achieve a consistent level of quality.
- **Continuous Quality Assurance:** Performing chemical analysis with a spectrometer, microstructural examinations, and mechanical tests at every stage of the production

process (scrap acceptance, post-melting, final product) allows for the early detection of potential deviations and the implementation of corrective measures.

The concept of aluminum's "infinite recyclability" is practically limited by the thermodynamic inevitability of impurity accumulation. Each recycling cycle has the potential to introduce a certain degree of disorder, i.e., impurities, into the system. This fact reveals that the circular economy is not just a logistical issue (collection and sorting) but also a fundamental materials science and metallurgy problem (refining and purification). Therefore, the future of a sustainable aluminum cycle depends not only on the development of more efficient collection systems but also on the implementation of innovative refining technologies that can economically remove accumulated impurities and the design of next-generation alloys that can tolerate higher impurity levels.

3 Experimental Procedure

In this experiment, a green sand mold will be prepared using a loose pattern, and scrap aluminum melted in an electric resistance furnace open to the atmosphere will be poured into the mold. After casting, the resulting part will be examined, defects identified during a preliminary inspection will be determined, and potential solutions will be discussed. *Each student is expected to create a flowchart of the experiment during the procedure.*

4 About the Report

All terms and concepts in this manual form the foundation of metallurgy and materials engineering principles, as well as the field of manufacturing engineering. The required content for the report will be determined weekly and announced to the students. It is of great importance for your personal and professional development that the assignments, which will mostly be in a question-and-answer format, are completed thoroughly. Therefore, I recommend that you conduct in-depth research on the topics using both traditional sources and AI-based tools. Remember, the work that seems like a burden today will make significant contributions to your professional success tomorrow.

References

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3)Groover, M. P. (2010). Fundamentals of modern manufacturing: Materials, processes, and systems (4th ed.). John Wiley & Sons.