



**YILDIZ TECHNICAL UNIVERSITY**

**METALLURGICAL AND MATERIALS  
ENGINEERING DEPARTMENT**

**MSE3952 LABORATORY II**

**EXPERIMENTAL BOOKLET**

**2023-2024**

**SPRING**

## Table of Contents

Important Information .....	3
Experiment 1: The Sol-Gel Technique.....	4
Experiment 2: Jominy Hardenability Test .....	10
Experiment 3: Metallography .....	13
Experiment 4: Part Production with Powder Metallurgy and Density Measurement with Archimedes' Principle.....	17
Experiment 5: Polymer Matrix Composite Production.....	21
Experiment 6: Mineral Processing (Ore Dressing) .....	27
Experiment 7: Recrystallization .....	33
Experiment 8: Glass Experiment.....	36
Experiment 9: Casting and Recycling .....	40
Experiment 10: Experimental Determination of Casting Ceramic Slurry into Plaster Mold and Determination of % Shrinkage Due to Drying, Firing, and Total Shrinkage.....	48

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

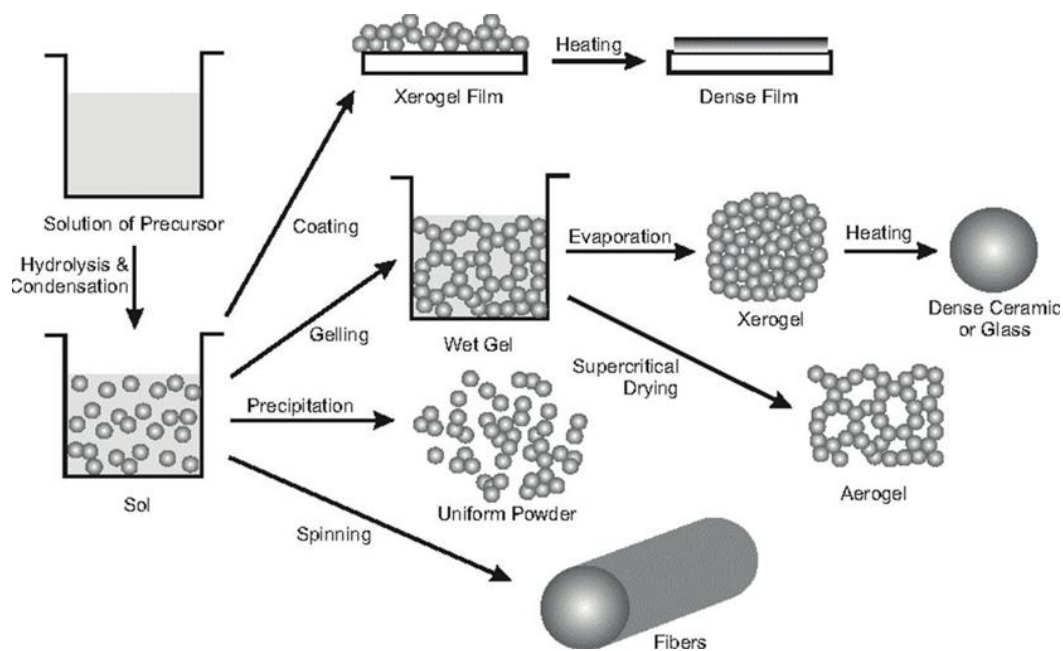
## EXPERIMENT 1: THE SOL-GEL TECHNIQUE

### 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 nano powder synthesis and the powder produced will be analyzed.

### 2. THEORETICAL INFORMATION

The Sol-gel method is a wet chemical process used in ceramic production. 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 in *Figure 1* [1-3].



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

#### ***What is Sol-Gel?***

The term sol gel describes the process of forming a three-dimensional and continuous network structure in the liquid (gel) after the agglomeration of nanosized solid particles (colloidal-sol) dispersed 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.

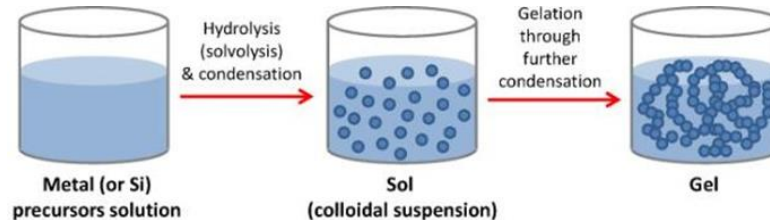
#### ***What is Gel?***

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 for preparing the sol:

- Molecules dissolved in the liquid turn into larger molecules as a result of mixing the liquids. The resulting macromolecules then form nanosized becomes solid particles. Example  $\text{SiO}_2$  (silica) based nanosol.
- Nanoparticles (such as carbon nanotubes and quantum dots) are created using special methods. The nanoparticles are then dissolved in the liquid phase. Surface modifiers (polymers, soaps, etc.) called surfactants are used to ensure homogeneous distribution.



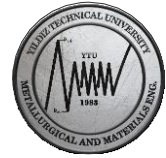
**Figure 2.** Schematic representation of Sol and Gel [3].

### **Gelation:**

In order for the sol to transform into gel, the solid nanoparticles dispersed in the sol must come together and form a network structure. In order for the network structure to form, the solid particles in the structure are mixed into liquid. It is necessary for the molecules to collide as a result of their random movements under the influence of temperature, called "**Brownian Motion**", and to stick together at the end of the collision. The adhesion process is much easier for solid particles that have reactive groups on their surfaces. Because after the collision, reactive groups can come together and form bonds. Since adhesion will not occur for solid particles that do not have reactive groups on their surfaces, the surfaces of such nanoparticles must be made reactive by adding additives or by stripping the surface. As a result, non-reactive particles are brought together (reactive) by the bond structure or electrostatic forces that will form at the end of the collision. As the sol turns into gel, the viscosity of the structure increases and the structure becomes unable to flow at the gelation point. At the gelation point, the reticulated structure formed by the particles has spread throughout the entire volume of the liquid, and the ability of the liquid to flow ceases. The time between mixing the gelling agent into the solution and the end of gel formation is called "**gelation time**" [2].

### **Factors Affecting Sol Gel Chemistry:**

- ❖ **pH:** The pH of the sol affects the rate of hydrolysis of the precursor. In the formation of silica gels, silanol groups are formed as a result of the hydrolysis of silica. Silanol groups then enable the formation of silica nanoparticles and the development of the network. Different pH conditions lead to changes in the size, porosity, and crystallinity of the final ceramic nanoparticles.
- ❖ **Solvent:** Plays a role in controlling the precursor solubility, gelation kinetics and the final structure of the gel. Solvents with different polarities and boiling points affect reaction rates and distribution of particles in the gel.
- ❖ **Temperature:** Higher temperatures generally promote faster reactions but also lead to undesirable side reactions or phase transformations. Too low a temperature increases the gelation time, while too high a temperature causes the nanoparticles to agglomerate. It causes them to grow excessively and precipitate before they can form a network structure.
- ❖ **Reaction Type:** The heat released during the chemical reactions of the formation of nanoparticles in the sol and the formation of the network structure causes the reactions to accelerate.
- ❖ **Time:** Gelation steps occur at different times. The properties of the product resulting from the slower sol-gel reaction are superior. Especially during the gelation phase, the slower the reaction, the more uniform the internal structure of the gel formed. This allows obtaining a higher strength and transparent gel.
- ❖ **Additives, Catalyst and Stabilizers:** Additional chemicals can be added to the sol to change particle size, increase stability or control the gelation process. These additives affect the morphology and properties of the resulting ceramic material. Acid in sol gel technique ( $\text{H}^+$ ) and bases ( $\text{OH}^-$ ) are used as catalysts. Since the catalytic effect is achieved through different mechanisms for acids and bases,



the sol gel method is sensitive to pH. Although the catalyst material is used in very small amounts (mg/mL), it reduces the gelation time from weeks to minutes.

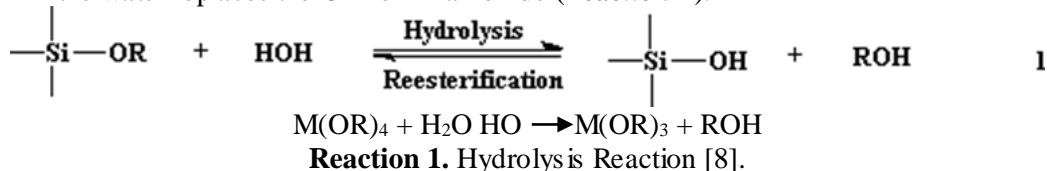
- ❖ **Mixing:** In the sol gel technique, mixing the sol is important so that the chemical reaction occurs uniformly and each molecule in the sol has access to the chemical required for the reaction. However, continued mixing after the gelation phase has begun may cause disintegration of the semi-gelled network at micro and macro levels. Even if the gelation of the entire structure eventually occurs, the gelation time is prolonged.

In sol gel application; Metal alkoxide solutions or metal powders, inorganic compounds such as nitrate, hydroxide, oxide are combined with water and acid in certain proportions to form a solution. By mixing the solution at certain temperatures, a series of successive chemical reactions occur in the solution. A network is formed by the electrochemical interactions of the surface charges of the particles, and this process is called gelation. Gel is obtained as this network grows and reaches all points in the system, forming a complete structure [1-5].

**Steps of the sol gel method:** Alkoxide hydrolysis, Polymerization (Peptitization), Gel formation, Calcination/Sintering.

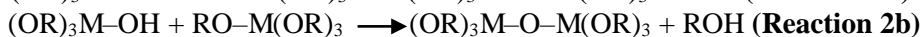
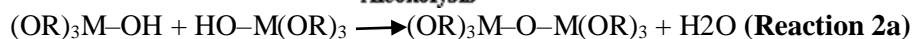
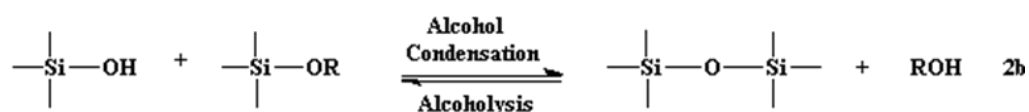
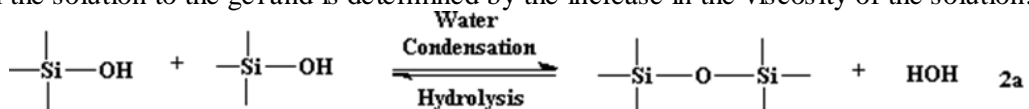
### I. Alkoxide hydrolysis

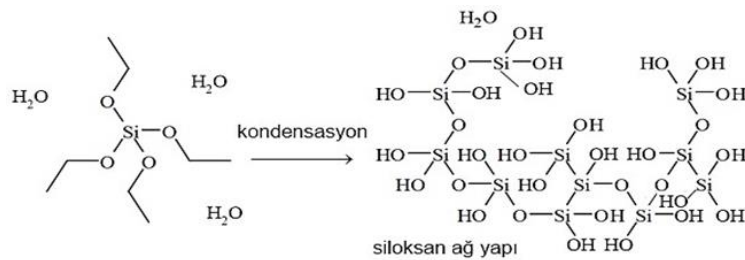
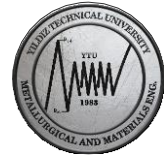
Alkoxides are used as starting material to form a solution.  $M(OR)_n$ . M; metal material to be coated, R;  $CH_3$  (methyl),  $C_2H_5$  (ethyl) alkyl group, and n; shows the values of the metal that vary according to the value. Due to their high electronegative OR group, metal alkoxides exhibit high 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 ( $> 80^\circ C$ ), 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**).



### 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 asycondensation. 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 [8].

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<sup>+</sup> 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, and 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 produced powders is high.
Fiber production occurs with the use of metal-alkoxide solutions.	The process is long and the amount of shrinkage during the process is high.
Solid glass materials with amorphous structure, which cannot be obtained by cooling from the liquid phase, are produced.	Fine pores may be present in the structure.
Thin SiO <sub>2</sub> and TiO <sub>2</sub> (≤1μ) coatings are produced on glass.	Residual hydroxide and residual carbon may occur in the structure [3].
Powder is produced in controllable shapes and sizes.	
Homogeneous distribution of the second phases within the main phase is ensured.	





### 3. EXPERIMENTAL STUDIES

#### 3.1 Tools, Devices, and Materials

- ✓ Tetra Ethyl Ortho Silicate (TEOS,  $\text{Si}(\text{OC}_2\text{H}_5)_4$ )
- ✓ Ammonium Fluoride ( $\text{NH}_4\text{F}$ )
- ✓ Ammonium Hydroxide (ammonia,  $\text{NH}_4\text{OH}$ )
- ✓ Ethanol ( $\text{C}_2\text{H}_5\text{OH}$ )
- ✓ Pure water
- ✓ Fume hood
- ✓ Precision scales
- ✓ Magnetic stirrer, Magnetic fish
- ✓ Pipette pump, pipette, and dropper
- ✓ Beakers
- ✓ 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 ( $\text{NH}_4\text{F}$ ) and 23 mL of ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) 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  $\text{SiO}_2$  (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 ( $\text{NH}_4\text{OH}$ ) 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 fluoride ion present in the ammonium fluoride. As the amount of polymerization increases, the viscosity of the solution increases.

#### *Nanosized Powder Production by Drying and Calcination of Gel:*

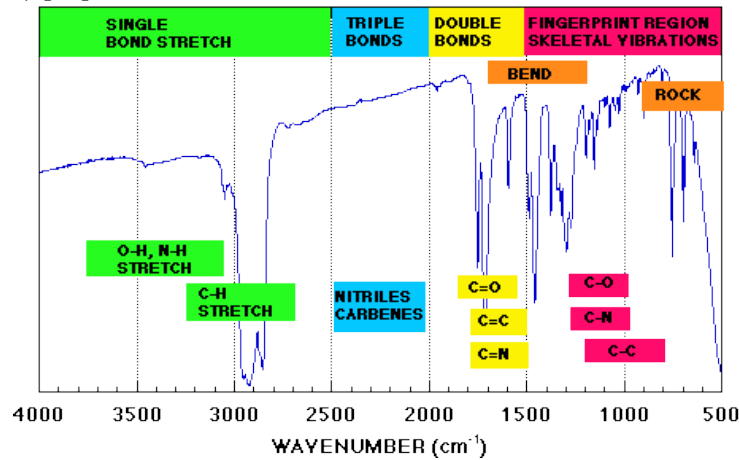
Finally, the sol/gel poured into the mold completely gels after 15 minutes. Nanosized  $\text{SiO}_2$  powder is obtained by drying (removing by-product water or alcohol) and calcination of the gel. At the end of the calcination process, the powder taken from the oven is ground in a mortar to obtain the final powder product.

#### *Fourier Transform Infrared (FTIR) spectroscopy*

The purpose of absorption spectroscopy techniques (FTIR, ultraviolet-visible ("UV-vis") spectroscopy, etc.) is to measure how much light a sample absorbs at each wavelength. Because chemical bonds absorb infrared (IR) energy at specific frequencies (wavelengths), the fundamental structure of compounds can be determined based on the spectral positions of their IR absorption. The plot of a compound's IR transmission versus frequency is the "fingerprint" that identifies the material when compared to reference spectra (Figure 3) [9]. IR spectroscopy is the study of the interaction of IR light with matter. IR light is absorbed by molecules at specific frequencies depending on the molecular bonds between atoms and the types of atoms found at the end of the bonds. Photon energies in the IR region cause vibrational excitation of covalently bonded atoms. High-energy mid-IR radiation excites fundamental vibrations when the energy is absorbed by molecules, elevating them from the ground state to the first vibrational state. The difference between IR and FTIR is that



the latter is constructed from an interferogram as the raw signal. This represents the light intensity not as a function of wavelength (as in dispersive devices), but as a function of the position of the mirror within the interferometer. This is “FT”. To produce intensity as a function of wavenumber, the signal must first be Fourier transformed (FT) [10].



**Figure 3.** Specific peak ranges of organic and inorganic chemical bonds in the sample according to wave lengths [9].

#### 4. RESULTS AND DISCUSSION

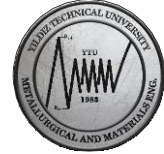
The gel product is weighed before drying and calcination. At the end of drying and calcination, it is weighed again. Thus, the weight loss occurring in the sample is calculated as % Weight Loss. At the end of the experiment, gel production should be ensured starting from the left. Then, oxide-based nanopowder should be obtained by drying and calcining the resulting gel. **Fourier Transform Infrared (FTIR) spectroscopy** analysis of the raw materials, gelling solution and nano powder used in the experiment will be performed and their interpretation will be learned.

At the end of the experiment, students will have learned the practical and theoretical outcomes listed below:

- Colloidal systems
- Basic principles of Sol Gel technique
- Nanopowder synthesis with Sol Gel technique
- Academic analysis and interpretation of the final product obtained in the Sol Gel method
- Preparation of the test result report (theoretical information, experimental study, results, references)

#### 5. REFERENCES

- [1] Internet source: URL: <http://www.aerogel.org>
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- [10] Internet sources: URL: <https://photometrics.net/fourier-transform-infrared-ftir-spectroscopy/>



## EXPERIMENT 2: JOMINY HARDENABILITY TEST

### 1. PURPOSE OF THE EXPERIMENT

- Distinguish between hardness and hardenability.
- Perform Jominy test as a method to indicate hardenability.

To increase the hardness of the material by the thermal process without changing the chemical composition.

### 2. THEORETICAL INFORMATION

#### *The Jominy Test*

Hardenability is the ability of an alloy to be hardened by the formation of martensite as a result of a given heat treatment. It is a qualitative measure of the rate at which hardness drops off with distance into the interior of a specimen as a result of diminished martensite content. With the Jominy end-quench test, except for alloy composition, a cylindrical specimen is austenitized and upon removal from the furnace, the lower end is quenched by a jet of water. The cooling rate is a maximum at the quenched end and diminishes with position from this point along the length of the specimen. With diminishing cooling rate more time is allowed for carbon diffusion and the formation of a greater portion of the softer pearlite or bainite. A steel alloy that is highly hardenable will retain large hardness values for relatively long distances. The presence of nickel, chromium, and molybdenum in the alloy steels delay the austenite-to-pearlite and/or bainite reactions, thus permitting more martensite to form for a particular cooling rate.

Factors Affecting Hardenability:

- Carbon Content
- Alloying elements
- Grain size
- Cooling rates

TTT diagrams for (a) hypoeutectoid, (b) eutectoid and (c) hypereutectoid steels are given in *Figure 1*.

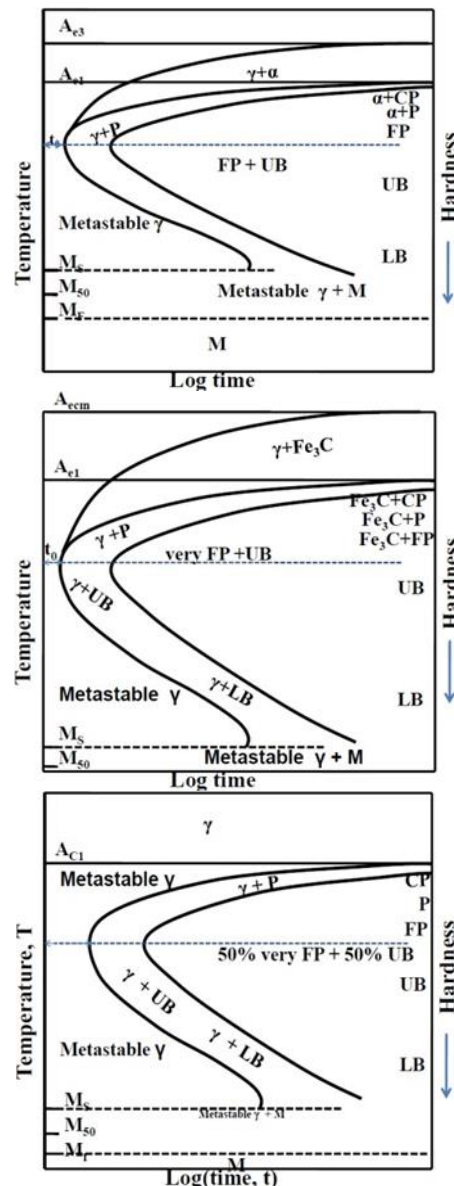
As the carbon percentage increases  $A_3$  decreases, similar is the case for  $A_r3$ , i.e. austenite stabilises. So the incubation period for the austenite to pearlite increases i.e. the C curve moves to right. However after 0,8 wt% C any increase in C,  $A_{cm}$  line goes up, i.e. austenite become less stable with respect to cementite precipitation. So transformation to pearlite becomes faster. Therefore C curve moves towards left after 0,8% C.

Almost all alloying elements (except, Al, Co) increases the stability of supercooled austenite and retard both proeutectoid and the pearlitic reaction and then shift TTT curves of start to finish to right or higher timing. This is due to i) low rate of diffusion of alloying elements in austenite as they are substitutional elements, ii) reduced rate of diffusion of carbon as carbide forming elements strongly hold them.

However Al, and Co increase rate of nucleation and growth of both ferrite or pearlite and therefore shift TTT diagram to left. In addition under the complex diffusional effect of various alloying element.

The simple C shape behaviour of TTT diagram get modified and various regions of transformation get clearly separated. There are separate pearlitic C curves, ferritic and bainitic C curves and shape of each of them are distinct and different.

Fine grain size shifts S curve towards left side because it helps for nucleation of ferrite, cementite and bainite.



**Figure 1.** TTT diagrams for (a) hypoeutectoid, (b) eutectoid and (c) hypereutectoid steels

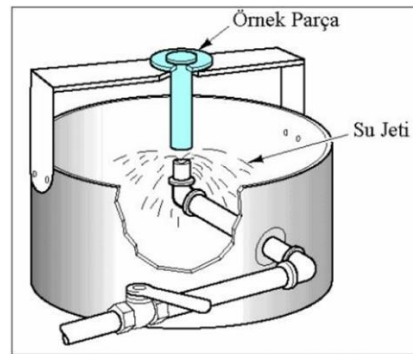
ASTM A 255 measures hardenability of steels. Hardenability is a measure of the capacity of a steel to be hardened in depth when quenched from its austenitizing temperature. Hardenability of a steel should not be confused with the hardness of a steel. The Hardness of a steel refers to its ability to resist deformation when a load is applied, whereas hardenability refers to its ability to be hardened to a particular depth under a particular set of conditions. Information gained from this test is necessary in selecting the proper combination of alloy steel and heat treatment to minimize thermal stresses and distortion when manufacturing components of various sizes.

### 3. EXPERIMENTAL STUDIES

First, a sample specimen cylinder either 100mm in length and 25mm in diameter, or alternatively, 101.6 mm by 25.4 mm is obtained. Second, the steel sample is austenitised. This is usually at a temperature of 800 to 900°C. Next, the specimen is rapidly transferred to the test machine (Fig. 2), where it is held vertically and sprayed with a controlled flow of water onto one end of the sample. This cools the specimen from one end, simulating the effect of quenching a larger steel component in water. Because the cooling rate decreases as one moves further from the quenched end, you can measure the effects of a wide range of cooling rates from very rapid at the quenched end to air cooled at the far end.



2023-2024 Spring MSE3952 Laboratory II  
Experiment 2: Jominy Hardenability Test



**Figure 2.** Schematic view of Jominy test.

The hardness is measured at intervals along its length beginning at the quenched end. For alloyed steels an interval of 1.5mm is commonly used whereas with carbon steels an interval of 0.75mm is typically employed. And finally, the Rockwell hardness values are plotted versus distance from the quenched end.

The Jominy Test data illustrates the effect of alloying and microstructure on the hardenability of steels. Commonly used elements that affect the hardenability of steel are carbon, boron, Chromium, Manganese, Molybdenum, Silicon, and Nickel.

Carbon is primarily a hardening agent in steel, although to a small degree it also increases hardenability by slowing the formation of pearlite and ferrite. But this effect is too small to be used as a control factor for hardenability.

Boron can be an effective alloy for improving hardenability at levels as low as .0005%. Boron is most effective in steels of 0.25% Carbon or less. Boron combines readily with both Nitrogen and Oxygen and in so doing its effect on hardenability is sacrificed. Therefore, Boron must remain in solution in order to be effective. Aluminum and Titanium are commonly added as "gettering" agents to react with the Oxygen and Nitrogen in preference to the Boron.

Slowing the phase transformation of austenite to ferrite and pearlite increases the hardenability of steels. Chromium, Molybdenum, Manganese, Silicon, Nickel and Vanadium all affect the hardenability of steels in this manner. Chromium, Molybdenum and Manganese being used most often.

#### 4. INFORMATION REQUIRED IN THE EXPERIMENT REPORT

- Draw Jominy curve for 1040, 1060, 4140 and 4340 steels depending on the distance from the end.
- Which mechanisms reduce the hardenability of steel when Al and Co are introduced? Explain.
- Which mechanisms increase the hardenability of steel when the other alloying elements except Al and Co are introduced? Explain.
- Why is the Jominy test not applied to high alloy steels? Explain.

#### 5. REFERENCES

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- 2) Thelning, Karl-Erik, Steel and its Heat Treatment, Butterworths, 1975.
- 3) Llewellyn D T and Hudd R C, Steels: Metallurgy and Applications, 3rd Edition, Reed Educational and Professional Publishing, 1998.
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## EXPERIMENT 3: METALLOGRAPHY

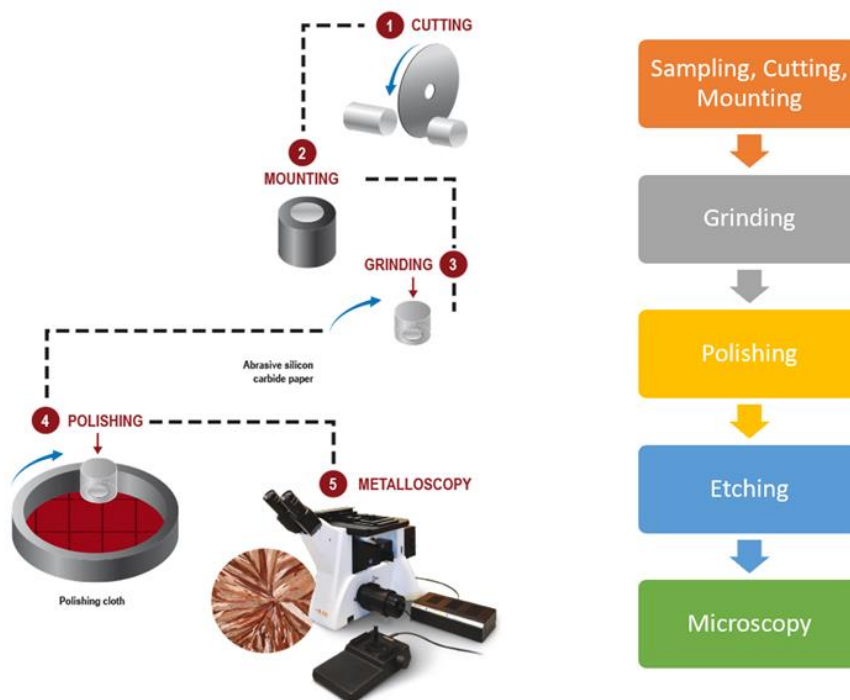
### 1. PURPOSE OF THE EXPERIMENT

Metallography is a branch of science that investigates the microstructure of metals and alloys. The fundamental aim of metallography is to comprehensively examine the internal structure of a metal or alloy under a microscope, with the goal of understanding its mechanical properties, durability, thermal behavior, and machinability. The metallographic experiment aims to prepare samples for microscopic examination, involving the learning of techniques such as grinding, polishing, and etching [1].

Metallographic examination is employed to observe the internal structure of a material at various stages (e.g., casting, heat treatment, deformation) in order to understand factors influencing the material's performance. This experiment involves steps such as plotting the microstructure graph, determining grain size, and examining phase composition. The information obtained is utilized to optimize the material's production process, ensure quality control, and enhance its service life [2].

### 2. THEORETICAL INFORMATION

Prior to microscopic examination in the study of surface topography or internal structure of materials, it is necessary for samples to undergo preparatory procedures. During metallographic investigations, from sample preparation to microscopic examination, the following stages are followed (*Figure 1*).

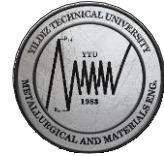


**Figure 1.** Metallographic process steps applied to samples.

#### I. SAMPLING, CUTTING, AND MOUNTING

Sampling in metallography is the process of selecting and extracting a sample that will be used to examine the internal structure of a material. The chosen sample should represent a whole entity from which information is sought. Care must be taken during the cutting of the sample to prevent overheating and excessive deformation. In principle, the cutting process should induce minimal heating, deformation, and material loss (especially for small pieces). The occurrence of these conditions can lead to changes in the microstructure. Cutting can be performed using methods such as hand saw, lathe-powered saw, or, ideally, using a water-cooled rotating disc. The critical considerations during sampling and cutting stages are outlined below.

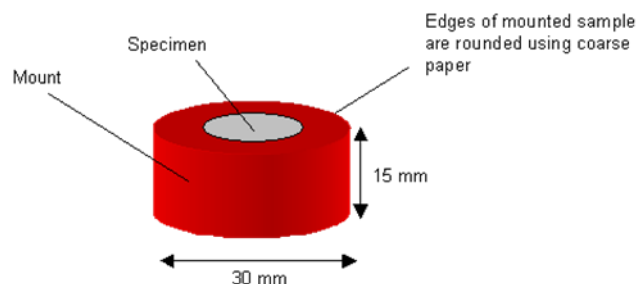




- **Sample Selection:** The sample should be chosen to represent the material adequately. Representing a homogeneous structure enhances the reliability of the analysis results. The location from which the sample is taken should be the region that best reflects the material's properties.
- **Sample Size and Shape:** The size of the sample should be chosen appropriately based on the equipment and analysis methods to be used. The shape of the sample should be determined according to the analysis objectives.
- **Cutting Process:** The cutting process should be carried out carefully to preserve the internal structure of the sample. The material used during cutting should not affect the sample's properties or cause damage.
- **Cooling Method:** The cooling method used during the cutting process is important to prevent thermal deformation of the sample. Cooling liquids or cold air can be employed.
- **Sample Edges:** The edges of the sample should be processed smoothly after cutting to minimize deformations that may occur during cutting.
- **Cleanliness and Contamination:** The sample should be clean and protected from external factors. Additionally, the cleanliness of the sample is crucial for interpreting analysis results accurately.

### **Mounting (Hot Mounting/Cold Mounting)**

Mounting is applied in the steps of metallographic sample preparation (such as grinding and polishing) to facilitate the handling of samples that cannot be held manually. For hot mounting, the sample placed in a mold, along with bakelite powders, is heated to approximately 150-190 °C under a specific pressure for a set duration, then cooled and removed. Epoxy, acrylic, and bakelite resins can be used for hot mounting, but Bakelite is most commonly used, leading to the process being known as "Bakelite mounting." The representation of a sample mounted using the hot mounting process is presented in *Figure 2*. Cold mounting is performed at room temperature without applying external pressure or temperature. Resin and hardener are mixed and poured over the inverted sample in the mold. Acrylic, polyester, and epoxy resins can be used. After a specified duration, the resin solidifies, and the sample is removed from the mold.



**Figure 2.** The representation of a sample mounted using the hot mounting process.

## **II. GRINDING**

Grinding is the process of obtaining the surface roughness of a mounted or directly taken sample, transforming it into a piece with a flat, homogeneous surface using abrasive papers with SiC abrasive powders. Abrasives are classified based on the number of abrasive particles per 1 square inch (2.54 cm<sup>2</sup>) of grind paper. As the number of particles per square inch increases, the grind paper number grows. Samples can be ground with grind papers of different grit sizes sequentially, such as 60, 120, 180, 320, 400, 600, 800, 1000, 1200, and 2000. Grinding is an abrasive process, and the grit size should be chosen in each grinding step to eliminate abrasive traces left on the surface from the previous step, gradually reducing surface roughness. Water is used during the grinding process to remove particles worn and detached from the sample surface. Without this, detached particles can cause damage to the surface. Additionally, wet grinding prevents the sample surface from heating.





### III. POLISHING

Polishing is the final stage to achieve a smooth and scratch-free surface. After fine grinding, the polishing process is performed to obtain a flat surface with a mirror-like finish that is free of scratches. The polishing process typically occurs on rotating disc-like devices. Solutions called pastes, consisting of suspensions of hard particles (such as diamond suspension), are used in the polishing process. The abrasive particles used can be diamond, aluminum oxide, or metal oxide-based, depending on the material's hardness. Different abrasive powders should be used while transitioning from one disc to another to avoid carrying foreign materials, necessitating the washing of both the sample and hands. To determine if polishing is sufficient, the surface is examined visually and under an optical microscope at 100X magnification. The surface should exhibit a mirror-like finish when viewed with the naked eye, and no scratches should be visible under the microscope.

### IV. ETCHING

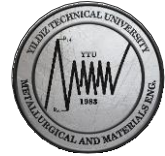
The surface obtained after the polishing process is suitable for examining certain material parameters. Some types of examinations that can be performed on polished surfaces include residue analysis, determination and classification of the type and graphite density of cast irons, crack examinations, porosity examinations, examinations of the distribution of certain secondary phases, examinations of certain coatings, etc. However, polished surfaces reflect light evenly, making it challenging to observe structural details. To address this, etching is performed to create contrast in the structure. The etchants used in etching cause the metal to dissolve anodically. The goal is to create the necessary optical contrast for light metal microscopy by taking advantage of different dissolution rates between phases or different phases. Etchants are organic or inorganic acids, alkalis, or their combinations prepared in liquids such as alcohol, pure water, glycerin, or their mixtures. Each metal structure or composition should be etched with an appropriate etchant; otherwise, sufficient contrast will not be achieved, and the desired result in imaging will not be obtained [3], [4]. (For example, Concentrated Nitric Acid: Nital 2 (a mixture of 2 ml nitric acid + 98 ml alcohol) etchant for steel samples or hydrofluoric acid (HF) for aluminum).

#### 3. TOOLS, DEVICES and MATERIALS

- ✓ Samples to be examined
- ✓ Hot mounting device / Bakelite powder
- ✓ Grinding device / Abrasive grind paper with different grit sizes
- ✓ Polishing machine / Polishing cloth / Polishing solution
- ✓ Etchant suitable for the sample to be examined for etching
- ✓ Light metal microscope for imaging

#### 4. EXPERIMENTAL STUDIES

The sample to be examined will undergo a hot mounting process (Bakelite mounting) to facilitate ease of handling. Subsequently, the sample will be grinded progressively from coarse to fine grits. During each change of grind paper, the sample will be rotated by 90 degrees to gradually reduce surface roughness. Following the grinding process, a polishing procedure will be applied to achieve a mirror-like finish on the sample surface. Subsequently, a suitable etchant, prepared according to the characteristics of the sample under investigation, will be applied to the sample surface. In the final stage, the prepared sample, tailored for microscopy, will be subjected to microscopic examination under a light metal microscope for detailed analysis.



## 5. INFORMATION REQUIRED IN THE EXPERIMENT REPORT

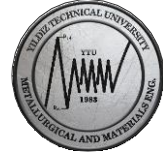
- **Cover Page:** In accordance with the format; Emblem, Course Information, Supervisor of the Experiment, Student Name-Surname/Student Number, and Group Number
- **Theoretical Information:** An original summary of the knowledge conveyed on the relevant subject during the experiment, prepared by the student
- **Experimental Studies:** Materials and equipment used in the experiment; application conditions of the experiment, and step-by-step procedures explained in accordance with the conditions of the experiment, including reasons and results
- **Evaluation:** The interpretation of the obtained sample image at the end of the experiment by the student, comparing the results obtained within the scope of the experiment with existing data in the literature

## 6. REFERENCES

- [1] Di Gianfrancesco, A. (2017). Technologies for chemical analyses, microstructural and inspection investigations. In *Materials for ultra-supercritical and advanced ultra-supercritical power plants* (pp. 197-245). Woodhead Publishing.
- [2] Song, B., Wen, S., Yan, C., Wei, Q., & Shi, Y. (2020). *Selective Laser Melting for Metal and Metal Matrix Composites-Materials Characterization*. Academic Press.
- [3] Mandal, M., Aashranth, B., Samantaray, D., & Vasudevan, M. (2023). Improvements in the Metallography of Ferritic–Martensitic Steels Through a Color Etching Procedure. *Metallography, Microstructure, and Analysis*, 12(1), 49-61.
- [4] Ashkenazi, D. (2023). Short Discussion Concerning the Metallography, Microstructure, and Analysis (MMA) 2023 Special Issue on Archaeometallurgy. *Metallography, Microstructure, and Analysis*, 12(5), 73



2023-2024 Spring MSE3952 Laboratory II  
Experiment 4: Part Production with Powder Metallurgy  
and Density Measurement with Archimedes'  
Principle



## EXPERIMENT 4: PART PRODUCTION WITH POWDER METALLURGY AND DENSITY MEASUREMENT WITH ARCHIMEDES' PRINCIPLE

### 1. PURPOSE OF THE EXPERIMENT

It is the transformation of metals and metal alloys into durable objects with the help of pressure and temperature, without melting their powders. This heat treatment, called sintering, replaces melting and is performed at a temperature below the melting point of the metal powder used. In the sintering of single-component powders, the sintering temperature can be taken below the melting temperature of the material (approximately 80% of the melting temperature). In multi-component systems, the sintering temperature can be selected just below the melting temperature of the component with the lowest melting temperature. This type of sintering processes is called solid phase sintering. Additionally, in multi-component systems, the sintering temperature can be taken above the melting temperature of at least one of the components, and such sintering processes can also be carried out. It is called liquid phase sintering.

### 2. THEORETICAL INFORMATION

#### *Why Powder Metallurgy Manufacturing Method?*

Parts manufactured by T/M can be examined in two main groups depending on the reason why this method is preferred.

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

**Group 2:** Parts that cannot be manufactured by other methods and where T/M is the only alternative.

#### *Powder Metallurgy Processes*

Powder metallurgy technique basically consists of 3 stages.

1. Powder production,
2. Pressing (turning dust particles into a single component through various processes),
3. Final part production by sintering and secondary processes if necessary

### SECTION 1.1 APPARENT DENSITY (WET DENSITY)

**1.1.1 PURPOSE OF THE TEST:** Determining the density of the powder in its loose, uncompressed state and determining its compliance with the standards.

**1.1.2 TOOLS, DEVICES AND MATERIALS USED:** Scale, metal powder, standard test equipment.

#### **1.1.3 THEORETICAL INFORMATION AND EXPERIMENT PERFORMANCE:**

Bulk density is the density of the powder in its loose (uncompressed) state ( $\text{g/cm}^3$ ). To determine this density, the powder is poured through a funnel whose dimensions and shape are determined by standards, and it is allowed to fill the cylindrical container underneath with a free fall. The height between the funnel outlet and the upper surface of the cylindrical container is also a constant value. The excess powder in the overfilled cylindrical container is carefully scraped off and the powder inside is weighed. Since the volume of the test container is known ( $25 \text{ cm}^3$ ), the bulk density is easily calculated as ( $\text{g/cm}^3$ ). All conditions related to this test and the conduct of the test are determined in the TS EN ISO 3923-1 standard. The equipment dimensions used for this experiment are given schematically in *Figure 1.1*. The apparent density of stainless steel powders produced by water atomization varies between  $2.80\text{-}3.20 \text{ g/cm}^3$ . This value can go up to  $5 \text{ g/cm}^3$  for powders produced by gas atomization. This value varies between  $2.80\text{-}3.00 \text{ g/cm}^3$  for copper powders and  $3.00\text{-}3.25 \text{ g/cm}^3$  for bronze powders. Agglomeration density is a very important factor in filling the mold with the powder during the pressing (densification) stage. Agglomeration density depends closely on powder grain shape, size and distribution. As the powder grain shape moves away from spherical, the intergranular void ratio decreases.



2023-2024 Spring MSE3952 Laboratory II  
Experiment 4: Part Production with Powder Metallurgy  
and Density Measurement with Archimedes'  
Principle

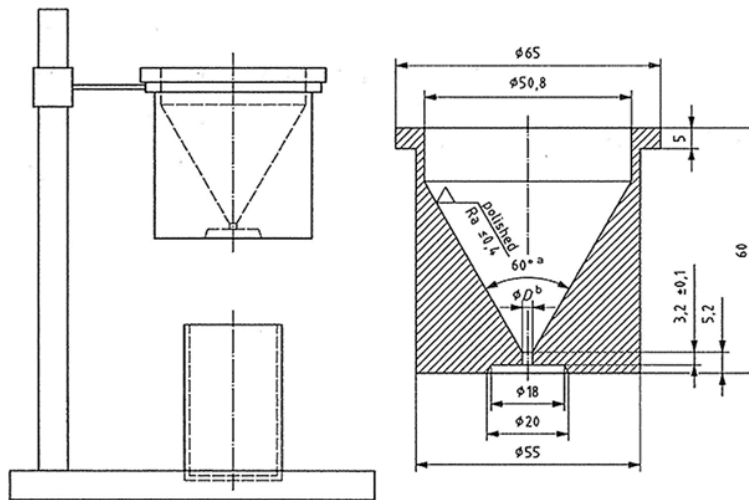


Figure 1.1 Measurement of Bulk Density.

## CHAPTER 1.2: FLUENCE TEST

**1.2.1 PURPOSE OF THE TEST:** To determine the compliance with the standards by determining the flow time required from the powders.

**1.2.2 TOOLS, DEVICES AND MATERIALS USED:** Scale, metal powder, standard test equipment, stopwatch.

### 1.2.3 THEORETICAL INFORMATION AND EXPERIMENT PERFORMANCE:

The purpose of this test is to determine the desired fluidity values from powder raw materials. This ratio is defined as the time it takes for 50 grams of powder to pass through the 2.54 mm funnel. It has a flow time of 15 seconds for spherical shaped stainless steel. This rate varies between 25-30 seconds for irregularly shaped powders. Fluency; It is the ability of a certain amount of a powder type or mixture to flow through a funnel of predetermined size and shape. In these measurements, the flow time of a 50 g powder sample from the funnel is generally determined and this value is considered as the fluidity of the powder. Flow with funnel dimensions and shape Hole dimensions (hole diameter and length) are specified in the standards (TS EN ISO 4490). The experiment is usually repeated three times and the average value is calculated. Flowability also depends on the grain size, specific surface size and powder grain shape of the powder. As the powder grain size decreases, the fluidity increases.

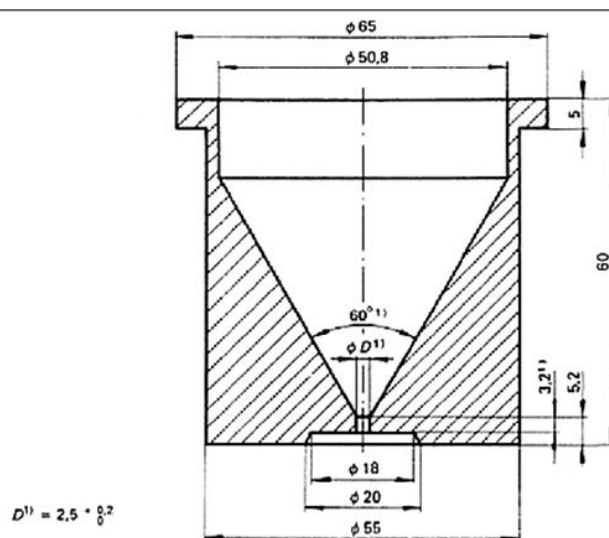


Figure 1.2 An Experimental Funnel to Determine Fluency is Given Schematically.

### SECTION 1.3: PRESSING

**1.3.1 PURPOSE OF THE EXPERIMENT:** Determining the pressure density relationship for different pressures by shaping the powders in molds.

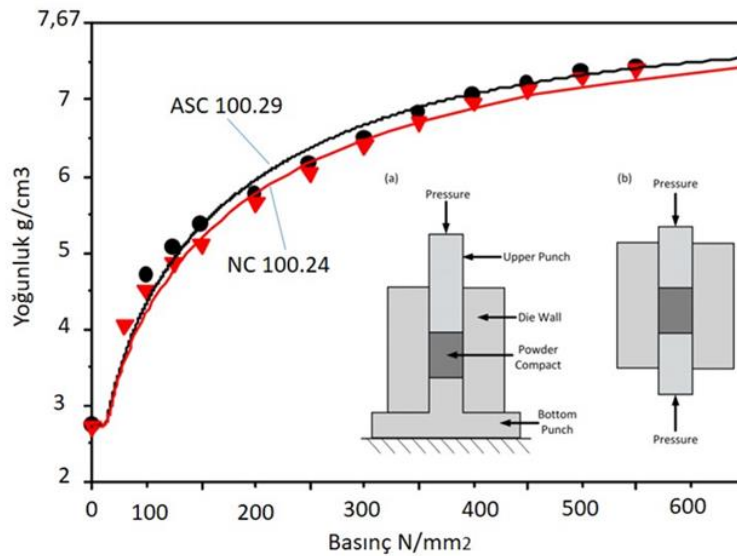
**1.3.2 TOOLS, DEVICES AND MATERIALS USED:** Metal powder, mould, press.

#### **1.3.3 THEORETICAL INFORMATION AND EXPERIMENT PERFORMANCE:**

Densification is the second important process in Powder Metallurgy. Pressing; It is the compression of the prepared metal powder mixture in appropriate presses with the help of molds and punches in order to reach the predetermined density. It turns the metal powder filled into the press mold into a solid object. In order to achieve a high pressing degree, a well-formable powder and high pressing pressures are required. The density reached during pressing primarily affects the mechanical properties of parts manufactured with powder metallurgy. Pressing is usually done cold (room temperature), but in special cases it can also be done hot. By pressing, the metal powder is given the desired density and mechanical strength according to the size and shape of the part to be manufactured.

There are three basic stages of the densification process in the mold.

1. Filling the specified amount of metal powder into the mold cavity,
2. Compressing the powder with pressure using punches,
3. Removing the shaped part from the mold.



**Figure 1.3** Density-pressure relationship for two commercial iron powders.

- \* Pressure ↑ , Density ↑ , Porosity ↓
- \* Porosity ↑ , Density ↓
- \* Porosity ↑ , Strength ↓
- \* Porosity ↑ , Lubrication ability ↑

Porosity: Sintered bushing, oil pump rotor, gears, bearing material, etc. It is requested in places. (After the part manufacturing is completed, the dust inside the pore is vacuumed, oil is given under vacuum, oil is pumped into the spaces by removing the air inside the pores)

### SECTION 1.4: PRESSIBILITY (GREEN DENSITY)

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

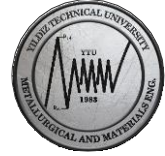
**1.4.2 TOOLS, DEVICES AND MATERIALS USED:** Scale, metal powder, press, caliper, mould.

#### **1.4.3 THEORETICAL INFORMATION AND EXPERIMENT PERFORMANCE:**





2023-2024 Spring MSE3952 Laboratory II  
Experiment 4: Part Production with Powder Metallurgy  
and Density Measurement with Archimedes'  
Principle



This property corresponds to the density achieved when the powder is under pressure. It is the density reached after pressing. Austenitic stainless steel powders have better pressability than martensite ones. Pressability; The yield limit is a function of the hardness of the sample, the softness of the grains, the void ratio of the sample, and the shape and size of the grains. If metal powders are shaped in the mold, the powder fills the mold cavity very well and completely (high fluidity ability). As desired, when cold pressed (punched) in the mold, it is ensured that it reaches as close to the theoretical density as possible. The softer the material, the higher its pressability. Pressability is closely related to the friction between the powder grains during pressing and between the grains and the mold. Theoretical density of the part:

for example, the green density achieved for Fe

If  $6.65 \text{ g/cm}^3$ , Fe:  $7.87 \text{ g/cm}^3$

$(6.65:7.87) \times 100 = 84.5\% \rightarrow$  % Gives the theoretical density.

84.5% Full

15.5% Porosity

### **SECTION 1.5: SINTERING PROCESSES APPLIED TO METAL POWDERS**

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

**1.5.2 TOOLS, DEVICES AND MATERIALS USED:** Sample produced by T/M, atmosphere-controlled oven.

#### **1.5.3 THEORETICAL INFORMATION AND EXPERIMENT PERFORMANCE:**

Sintering; It is a heat treatment performed without damaging the shape of the pressed powder material. The powder shaped by the press is not suitable for use in its current state. However, the necessary strength increase is achieved by sintering. In this process, diffusion occurs between powder grains at a certain temperature and time and weak mechanical bonds turn into strong mechanical bonds. Sintering temperature and time; In sintering, a sintering temperature and time suitable for the desired properties of the parts must be determined. The main reason for the low density and strength in the parts is the very low sintering temperature and time. The sintering temperature should be as close as possible to the melting temperature of the powder.

**Table 1.1** Sintering temperatures and times of some metals and alloys.

Toz Malzemesi	Sinterleme Sıcaklığı (°C)	Sinterleme Sıcaklığında Bekleme Süresi (dk)
Bronzlar	760-871-820	10-20
Pirinçler	843-898	10-45
Bakır	843-898	12-45
Çelik, C'lu çelikler	1010-1148	8-45
Paslanmaz çelikler	1033-1287	30-60
Fe (ferrit)	1204-1482	10-600
Nikel	1010-1148	30-45
Alnico magnetler	1204-1301	120-150
Tungsten karbürler	1426-1482	20-30
Molibden	2054	120
Tungsten	2343	480

### **3. RESOURCES**

- [1] Powder Metallurgy Lecture Notes, Adem BAKKALOĞLU, 2015.
- [2] Powder Metallurgy and Particulate Material Processing, Randall M. GERMAN, 2007
- [3] Höganäs Handbook For Sintered Components, 1997.
- [4] Powder Metallurgy Science, Randall M. GERMAN, 1994.
- [5] ASM Handbook Volume 7, Powder Metallurgy, 1993.
- [6] TS EN ISO 3923-1 Metal powders - Apparent density determination - Part 1: Funnel method
- [7] TS EN ISO 4490 Metallic powders - Determination of flow time through a calibrated funnel (hall flowmeter) measurement method



## EXPERIMENT 5: POLYMER MATRIX COMPOSITE PRODUCTION

### 1. PURPOSE OF THE EXPERIMENT

Production of E-Glass reinforced composite plates with plastic matrix and fracture analysis is by making impact test on samples taken from these plates.

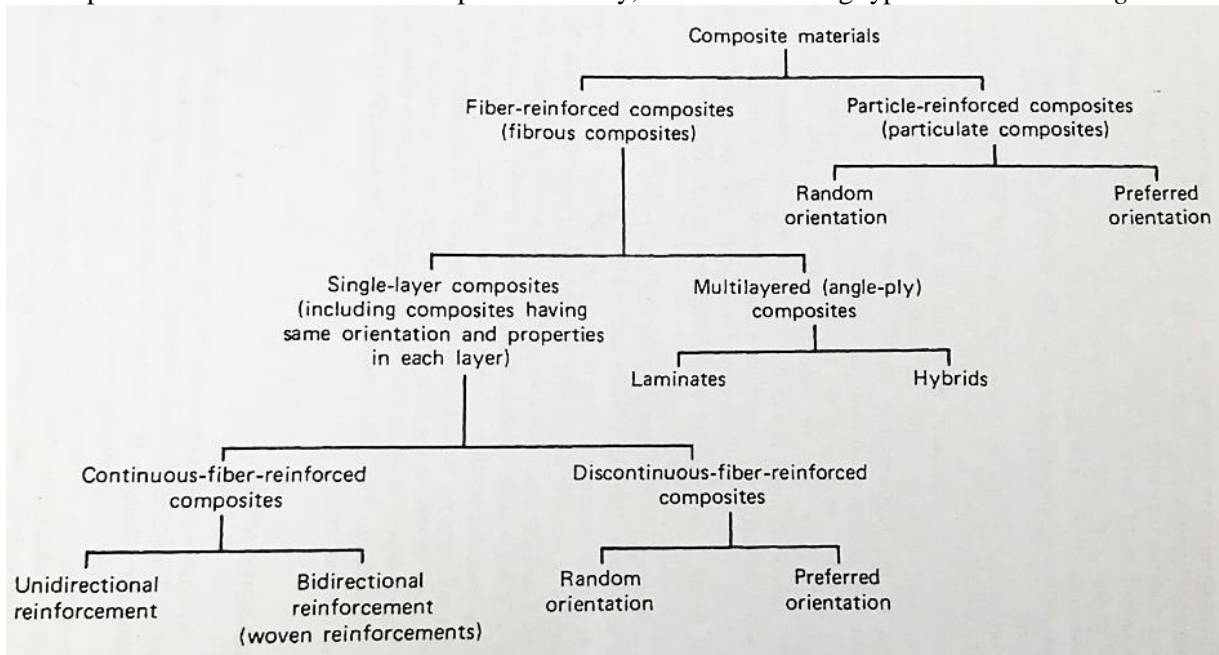
### 2. THEORETICAL INFORMATION

#### 2.1. Principles of Experiment

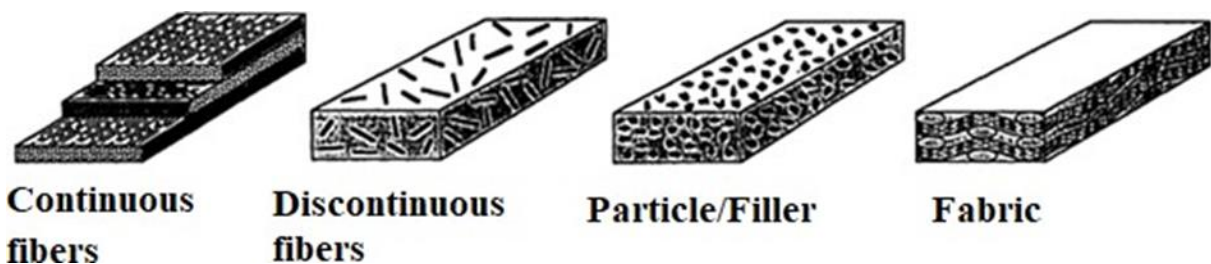
The new material group formed by at least two different materials by forming an interface between them without chemical bonding is called composite. Basically, composite materials have two main components.

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.

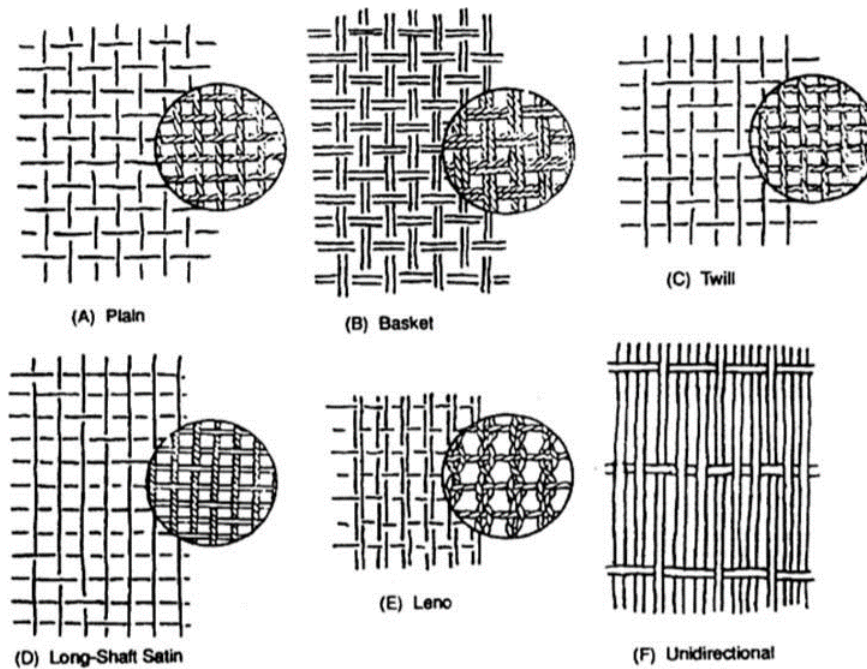
The main load-bearing element in composites is reinforcing fibers. Glass fibers are the most widely used and the cheapest type of reinforcement. Aramid and carbon fibers are reinforcement types with high mechanical properties and high cost. Reinforcements can also be available in different forms. *Figure 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

- **Plastic 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. Plastic Matrix Composites

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

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

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

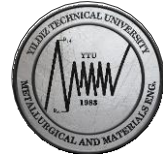
- Plastic-Plastic Composites
- Plastic-Metal Composites
- Plastic-Ceramic Composites
- Plastic-Foam Composites

## 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,



2023-2024 Spring MSE3952 Laboratory II  
Experiment 5: Polymer Matrix Composite  
Production



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<sup>2</sup> 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.

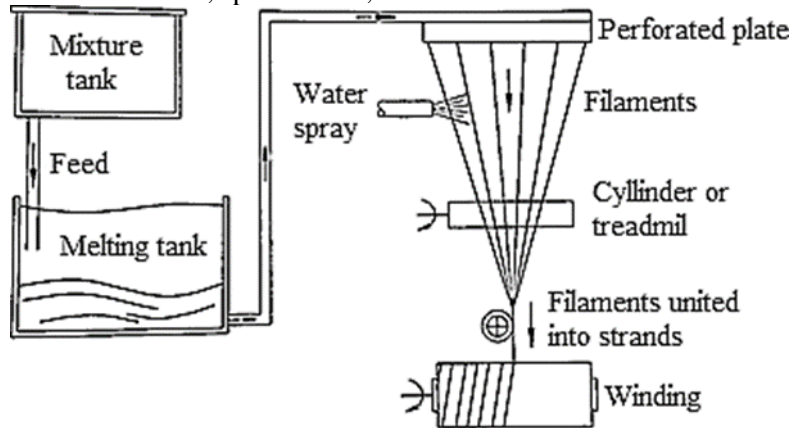


Figure 4. Schematic drawing of glass fiber production.

## 3. EXPERIMENTAL STUDIES

### 3.1. Tools and Devices Used

- Fiber reinforced composite samples in accordance with test standards
- Digital Caliper
- Impact Device

### 3.2. Kullanılan Standartlar ve Metotlar

- TS EN ISO 527-4: Plastics - Determination of Charpy impact properties - Part 1: Impedance impact test
- TS EN ISO 14125: Plastic composites - fiber reinforced - determination of bending properties
- TS EN ISO 179-1: Plastics - Determination of Charpy impact properties - Part 1: Toolless impact testing

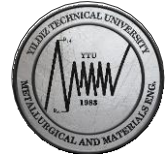
### 3.3 Materials Used

- Reinforcement element: E-glass weave or mat fabric
- Matrix material: Orthophthalic based unsaturated polyester resin





2023-2024 Spring MSE3952 Laboratory II  
Experiment 5: Polymer Matrix Composite  
Production



- Resin accelerating agent: Cobalt octoate
- Resin initiating agent: Methyl Ethyl Ketone (MEK) Peroxide
- Dosing pipettes
- Resin Mixing Bowl
- Mixer Mold
- Mold release agent
- Brush
- Acetone or thinner
- Plastic glove

### ***3.4. 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.

### ***3.5. Sample Preparation and Impact Test***

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

## **4. RESULTS AND DISCUSSION**

### ***4.1. Experimental Results and Discussion***

Composite part production stages will be observed in detail. Materials with different layer thicknesses will be compared. As a result of the experiments, fracture analyzes will be made on the samples. As a result of the examinations made on the sample fracture sections, fiber pull-out and delamination problems will be observed, which the main damage causes of the composites are.

### ***4.2. Calculations and Assignments***

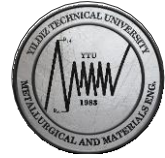
The impact energy will be read on the device in Joules (J), and the impact strength will be found by dividing the sample cross-sectional area.

### ***4.3. Information Required In the Experiment Report***

- Definition, classification, types and examples of composite materials.
- General information about plastic materials used as matrix materials, types, examples.



2023-2024 Spring MSE3952 Laboratory II  
Experiment 5: Polymer Matrix Composite  
Production

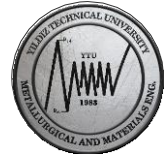


- Short information about the production methods of plastic matrix composite materials.
- General information about glass fibers.
- Identification of materials used.
- Application of composite production.
- Calculation of the impact strength.
- Comparison of the results of the samples.
- Fracture analysis.
- Specifying the sources used at the end of the report.

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## EXPERIMENT 6: MINERAL PROCESSING (ORE DRESSING)

### 1. PURPOSE 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. If valuable minerals do not contain metallic elements, it is called industrial raw material instead of ore dressing.

**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

The process of raising the percentage of the base metal mineral in the low grade ore occurring in nature. By taking advantage of the different properties of the gangue minerals and the base metal mineral, minerals are partially separated from each other by ore dressing or beneficiation process (*Figure 1*). Ore dressing is applied due to economic and technological reasons.

##### 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;  $10 \text{ mm} < \text{Grain Size} < 100 \text{ mm}$  is required. For this purpose, crushing, grinding and sintering-pelletizing is applied. In addition, both % P and %  $\text{Na}_2\text{O} + \text{K}_2\text{O}$  must be  $< 0.1\%$ .

##### b) Economic Reasons for Ore Dressing

- Using an uneconomic ore directly as is produced from the quarries (eg, producing lead metal directly from a 5 % Pb-containing ore) is never economical. With the ore dressing process, the lead percentage is increased to 60% and economic efficiency is ensured.
- To further increase the economics of an economic ore as produced from the quarries. 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.

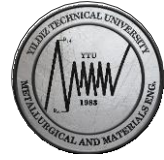
#### 2.2.1 ORE DRESSING PROCESSES

**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).

**Screening:** It is the dimensioning done with the help of screens.

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



### **2.2.1. CRUSHING**

Crushing is the first step of size reduction. It is conducted to make one of the different minerals freed from others, the process is done with the aim of providing suitable size or surface area or suitable size for the purpose of use.

The forces applied in the field; impact, compression or crushing, cutting and friction forces. The devices used for crushing are called crushers. They are mechanical tools that apply pressure, impact and shear force to the grain to bring them to a smaller grain size. Crushing machines are developed especially in terms of design features such as product characteristics, machine costs and energy use. Thus, ore dressing machines in a variety of shapes, structures and sizes are used. Crushing is applied to grain sizes between 200 and 0.5 cm. Crushing between 200-10 cm is named coarse crushing; and crushing between 10-0.5 cm, it is called fine crushing. Jaw, cone and hammer crushers are the most widely used types of crushers in ore dressing plants.

Removal of the crushed material in the desired size from the crushing cycles or classification of the material according to the size; different sieves are used according to the applied process, structure of the ore, size, physical and chemical properties. 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).

#### **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 or crushing, cutting 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. 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; hydrocyclones, mechanical classifiers (spiral classifiers, notched classifiers, solid centrifugal classifiers) and air classifiers.

#### **2.2.1.3 SCREENING**

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 ( $\text{in}^2$  or  $\text{mm}^2$ ) of a screen. 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.

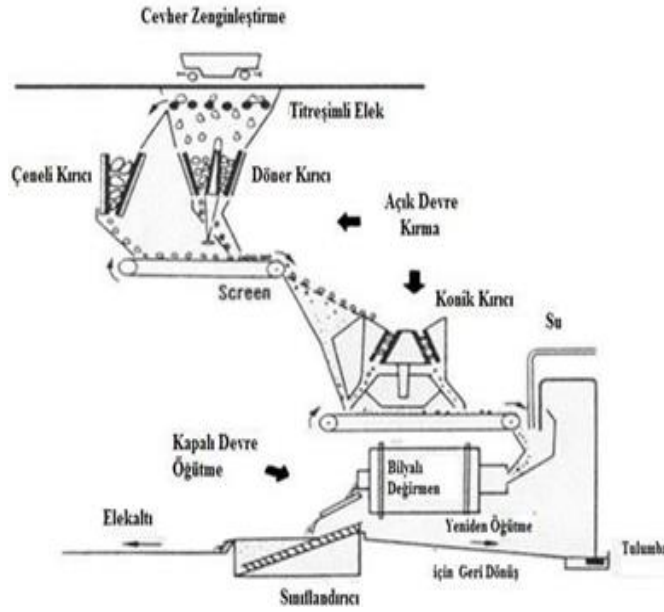


Figure 1. Ore dressing processes.

### 2.2.2 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.

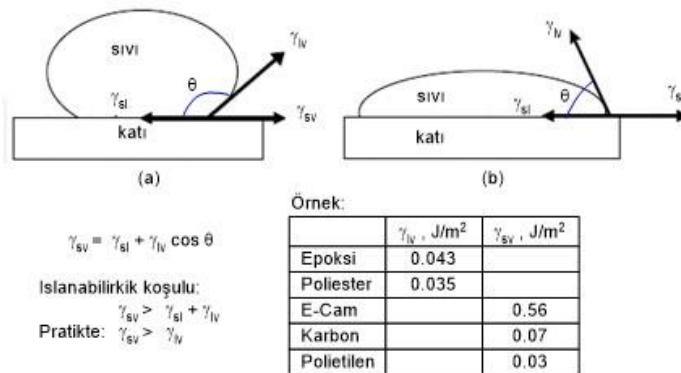


Figure 2. The forces in solid, liquid, air triple system and Young Equation.

The high contact angle ( $\theta$ ) 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. 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,  $\gamma_c$ . At low liquid surface stresses lower than this  $\gamma_c$ , the mineral loses its hydrophobicity or buoyancy property by being



completely wetted by this solution. The surface tension ( $\gamma_{SH}$ ) of the liquid used for a good contact angle (between solid-liquid-air interfaces), i.e.  $\theta > 0$ , must be greater than the  $\gamma_c$  value of the mineral. This is the first of the conditions required for the successful flotation.

Low surface energetic minerals ( $\gamma_c < 72$  dyn / cm) are wetted by surface-energized fluids lower than the Critical Wetting Surface Energy ( $\gamma_c$ ). 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 ( $\theta = 0$  state). Partially wetted solids cling 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  $\gamma_c$  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-aerated mechanical cell (Agitation)
- Air blown mechanically agitated cells (Sub-aeration)
- Air blown, air mixed cell (Pneumatic)

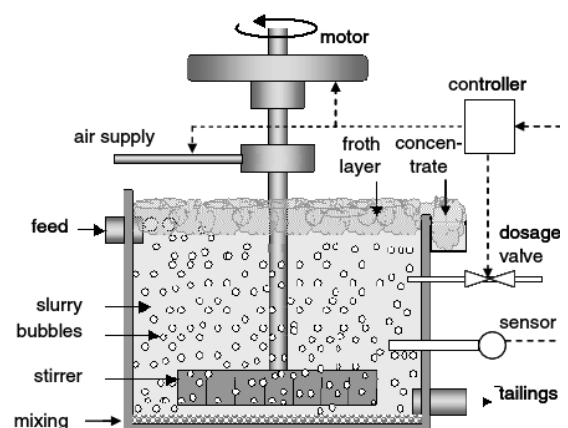
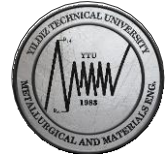


Figure 3. Denver flotation machine.



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.

### 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 STUDIES

#### 4.1 Ore Dressing Experiment

- The weight of the mixture to be examined for particle size distribution is weighed and recorded. The weighed mixture is fed to the crusher and crushing is performed by operating the crusher. After the crushed particles are removed from the crusher, they are weighed again and recorded.
- The sieves are arranged on the sieving device in the order of their interval, according to their mesh numbers. Then the crushed ore at the top of the sieve set is fed.
- The screws of the sieve set are squeezed and sieving is carried out by operating the device for ten minutes.
- By taking the sieve set from the machine, the amount of material left in each sieve and the total amount of sieved material are recorded in Table 1.1.

#### 4.2 Flotation Experiment

- 200 g. of galena ore with -200  $\mu\text{m}$  grain size is weighed and adjusted to 20% solids ratio depending on the volume of flotation cell. The cell is placed in the flotation machine and the pulp is mixed by starting the machine. The pulp is conditioned by stirring 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.
- One drop of the frother, which provides for the formation of an aerosol foam for floatation of the hydrophobicized minerals, is added. After the addition of frother the pulp is conditioned for another 1-2 mins.
- After the end of the last conditioning period, the air inlet tap of the flotation device is opened and the pulp is aerated. Mineral(s), whose surface has become hydrophobic, adhere to the air bubble and accumulate on the surface as foam.
- Foams are removed from the surface and concentrated in a separate container, and the process is terminated after the extent of the mineral to be floated has finished.

### 5. INFORMATION REQUIRED IN THE EXPERIMENT REPORT

- Write the objective and procedure of the experiment. (10 points).
- The results of each sieve analysis shall be recorded in the chart given in Table 1.1 (25 points).
- 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



2023-2024 Spring MSE3952 Laboratory II  
Experiment 6: Mineral Processing (Ore Dressing)



the average grain size from the intersection of the two drawn lines (25 points).

- 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).
- Determine the enrichment ratio (Z) by weighing the concentrate and the residue after flotation ( $Z = \text{Ore Fed} / \text{Concentrate}$ ) (20 points).

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

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

**Table 1.1** Sieve analysis data and calculations

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

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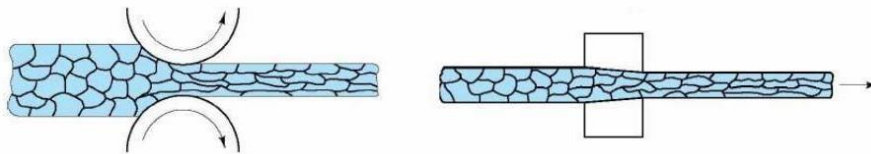
## EXPERIMENT 7: RECRYSTALLIZATION

### 1. PURPOSE OF THE EXPERIMENT

To observe the alterations that the recrystallization annealing may provide in the grain structure and the mechanical properties of the cold worked materials. To investigate the effects of the deformation amount, annealing time and temperature on the recrystallization process.

### 2. THEORETICAL INFORMATION

Cold working, which is also known as work hardening, is the process of strengthening metals through plastic deformation. This is made possible through the dislocation movements that are produced within the crystal structure of the material. Many metal fabrication processes involve cold-working, such as cold rolling sheet and plate, wire drawing, and deep drawing. The crystalline grain structure of cold worked material is distorted. Grains elongate along the direction of the deformation (*Figure 1*).



**Figure 1.** Distortion of the grain structure during the plastic deformation [1]

As a result of the cold working, the density of dislocations and subsequently hardness and strength of the metallic material increase, while its ductility and toughness decrease. This phenomenon is called the strain hardening. During the plastic deformation, the formation of atomic gaps and the increase in the density of these gaps with the increasing deformation amount, causes cracks and ruptures in the material. Therefore, the amount of deformation that can be applied to the material with strain hardening is limited. Furthermore, while the enhanced hardness and strength that the working treatment provides may be useful in specific applications, it is frequently essential to revert the metal to its original condition in order to perform further forming operations. The array of dislocations generated by cold working increases the amount of strain energy stored in the lattice. As a result, compared to the undeformed condition, the cold-worked condition is thermodynamically unstable, and the cold-worked metal will attempt to recover to a state with lower free energy.

Recrystallization refers to the rearrangement and reorientation of the elongated grains to refined equiaxed grains when a metal is heated at its recrystallization temperature. Metallic materials must be deformed with a specific amount to recrystallize at a certain temperature, which is called the critical deformation rate. With increasing amount of deformation, the energy stored in the material increases which favors the nucleation. Hence, when annealed at the same temperature, the metal with the greater amount of deformation recrystallizes more rapidly. Additionally, the presence of the alloying elements and their distribution (solid solution or secondary phase) and the grain size of the material after cold work are the parameters effects the recrystallization characteristics. The removal of the cold-worked condition occurs by a combination of three processes, namely, recovery, recrystallization and grain growth [2-3].

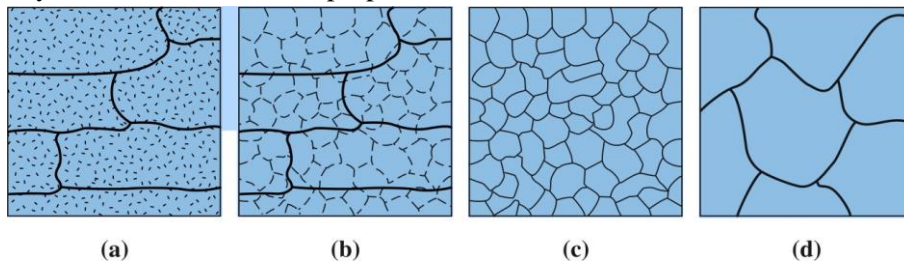
**Recovery:** The initial cold-worked microstructure is made up of distorted grains with a large number of tangled dislocations. With the increasing temperature, the extra thermal energy allows the dislocations to move and form the boundaries of a polygonized subgrain structure. (*Figure 2b*). This low-temperature treatment, known as recovery, reduces residual stresses caused by cold working without causing an alteration in dislocation density. The mechanical properties of the metallic material remain relatively unchanged since the number of dislocations is not reduced during recovery. Yet, because residual strains are decreased or eliminated when dislocations are repositioned, recovery is commonly referred to as a stress-relief anneal. Additionally, recovery restores the electrical conductivity and improves the corrosion resistance of the material.

**Recrystallization:** When a metallic material that has been cold worked is heated above a particular temperature, quick recovery reduces residual stresses and generates the polygonized dislocation structure. The nucleation of new small grains at the cell boundaries of the polygonized structure eliminates the majority of dislocations. (*Figure 2c*). Since the number of dislocations is drastically decreased, the strength of the recrystallized metal reduces while its ductility increases. The formation of new grains in a material that has been cold-worked by heating is referred to as recrystallization.

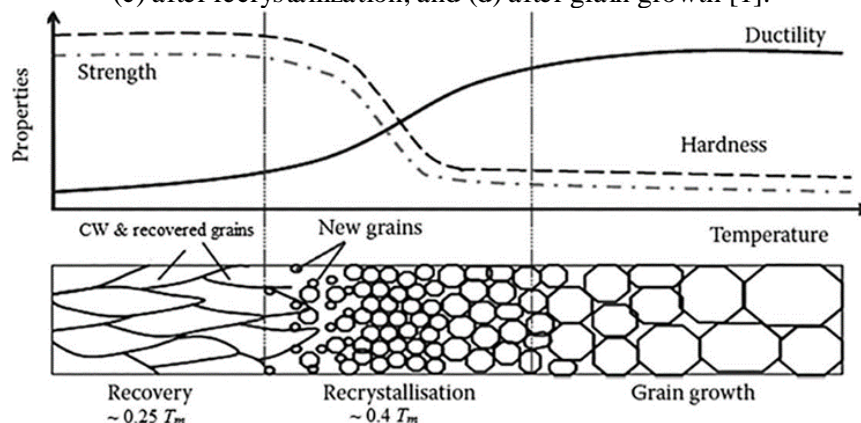
The driving force for recrystallization is the difference between the internal energy between a cold worked material and that of a recrystallized material. The recrystallization temperature is the temperature at which 50% of the recrystallization of a cold-worked material is accomplished within one hour. Typically, this temperature corresponds to 40% melting temperature of the material. However, the recrystallization temperature is not a fixed temperature and is influenced by a variety of processing variables which are given below.

- Recrystallization temperature decreases with the increasing deformation amount. Greater amounts of deformation decreases the stability of the material and favours the nucleation of recrystallized grains.
- A smaller original cold-worked grain size reduces the recrystallization temperature by providing more sites—the former grain boundaries—at which new grains can nucleate.
- Pure metals recrystallize at lower temperatures than alloys.
- Increasing the annealing period lowers the recrystallization temperature, since more time is available for the nucleation and growth of the recrystallized grains.

**Grain Growth:** Heating the material at a temperature above the recrystallization temperature causes the growth of newly formed grains. (*Figure 2d*). This phenomenon, called grain growth, is driven by the reduction in grain boundary area to reduce the internal energy. Grain growth is generally undesirable since it adversely affects the mechanical properties of the material [4].



**Figure 2.** The microstructure of a metallic material, (a) after cold-worked, (b) after recovery, (c) after recrystallization, and (d) after grain growth [1].



**Figure 3.** Schematical representation of the alterations in the structure and the properties of the material as a result of cold working and annealing [4]



### **3. EXPERIMENTAL STUDIES**

#### **3.1 Tools, Devices and Materials**

- Rolled Al samples (1xxx series)
- Furnace
- Optical microscope
- Vickers hardness measurement device

#### **3.2 Experimental Procedure**

- 1) Samples that are cold worked by the rolling process with different deformation amounts are prepared metallographically.
- 2) Microstructures of the samples are examined and their grain size and hardness values are determined.
- 3) Recrystallization annealing is applied to samples at the temperatures of 100 °C and 150 °C for 60 and 90 minutes.
- 4) Microstructures of the samples that are metallographically prepared after the recrystallization are examined and their grain size and hardness values are determined.

### **4. RESULTS AND DISCUSSION**

As a result of the experiments, the alterations that the recrystallization annealing may provide in the grain structure and the hardness of the cold worked materials may be observed. Also the effects of the deformation amount, annealing time and temperature on the recrystallization process are determined.

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## EXPERIMENT 8: GLASS EXPERIMENT

### 1.PURPOSE 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
<b>Total</b>	<b>175</b>	<b>100</b>

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
<b>World</b>	<b>72.1</b>	<b>77.6</b>	<b>76.8</b>	<b>73.8</b>	<b>86.8</b>

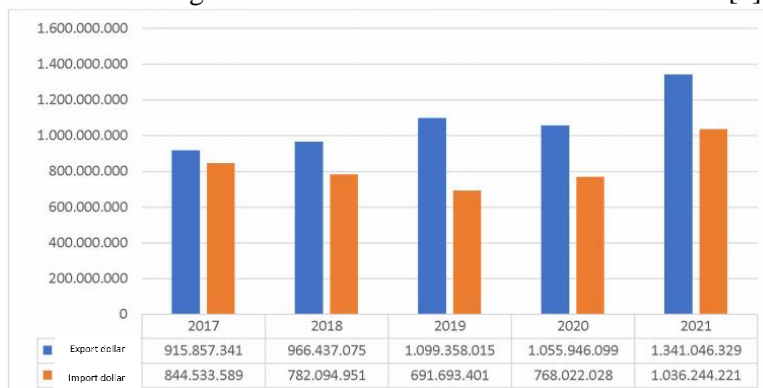
In Turkey, the glass sector is an important industry that contributes to the country's leading sectors, creates net added value for the economy, and demonstrates continuous growth and development. The main production areas of the glass industry in Turkey are as follows:

- 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*).

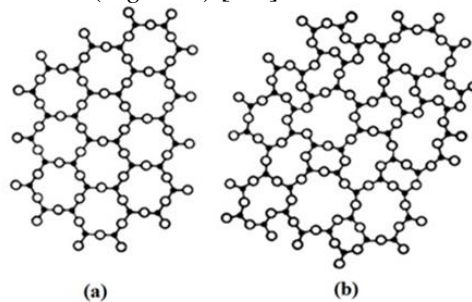
**Table 3.** Foreign Trade Values of Glass and Glass Products [2].



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 Gural 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].



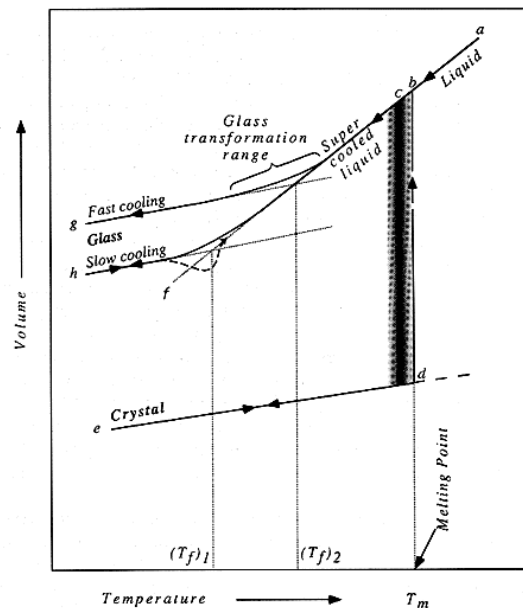
**Figure 1.** Atomic arrangement of a) crystalline, b) amorphous structure.

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 vitreous structure, it must be cooled rapidly below



the melting temperature to prevent the crystallization of the supercooled liquid. The crystallization rate is the factor controlling the glass formation. The mechanical properties of the glass resemble 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].



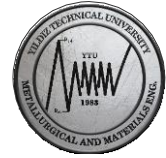
**Figure 2:** Specific volume – temperature change of crystalline and amorphous solids.

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 ( $\Delta G$ ) 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 ( $\text{SiO}_2$ ), Soda ( $\text{Na}_2\text{CO}_3$ ), Limestone ( $\text{CaCO}_3$ ), Agate mortar, Spatula, High-temperature gloves, Alumina crucible, Precision balance, Drying oven, Glass casting mold, Glass melting furnace, Annealing furnace.

#### 3.1 EXPERIMENTAL PROCEDURE:

In experimental studies aimed at soda-lime glass production, technical grade quartz sand ( $\text{SiO}_2$ ), soda ash ( $\text{Na}_2\text{CO}_3$ ), and limestone ( $\text{CaCO}_3$ ) are used. Carbonates used to obtain  $\text{Na}_2\text{O}$  and  $\text{CaO}$  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 ( $\text{g/cm}^3$ )	Melting Temperature ( $^\circ\text{C}$ )
<b><math>\text{SiO}_2</math></b>	60,08	2,648	1713
<b><math>\text{Na}_2\text{O}</math></b>	61,98	2,27	1132
<b><math>\text{CaO}</math></b>	56,07	3,34	2613

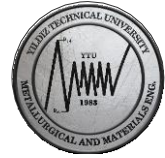
For the preparation of the blend of glass with a composition of 75 $\text{SiO}_2$ -15 $\text{Na}_2\text{O}$ -10 $\text{CaO}$  in mol%, powders are weighed on a precision balance (with a sensitivity of 10-4 g). After weighing, they are homogeneously mixed in an agate mortar. Then, the mixture is placed in an alumina crucible and held in a furnace at 1400 $^\circ\text{C}$  for 30 minutes. The molten glass is poured into a preheated stainless steel mold in an oven at 200 $^\circ\text{C}$  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 $^\circ\text{C}$  for 30 minutes before being gradually cooled to room temperature inside the furnace.

#### 4. POST-LAB QUESTIONS

- Calculate the weights of the raw materials required to prepare a blend of soda-lime glass with a composition of 75 $\text{SiO}_2$ -15 $\text{Na}_2\text{O}$ -10 $\text{CaO}$  in mol% using  $\text{SiO}_2$ ,  $\text{Na}_2\text{CO}_3$ , and  $\text{CaCO}_3$  as raw materials (MW  $\text{CO}_2$ : 44.01 g).
- Write down the functions of the components of commercial soda-lime glass in glass structure.  $\text{SiO}_2$ ,  $\text{Na}_2\text{O}$ ,  $\text{Li}_2\text{O}$ ,  $\text{K}_2\text{O}$ ,  $\text{CaO}$ ,  $\text{MgO}$ ,  $\text{PbO}$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{B}_2\text{O}_3$ ,  $\text{ZnO}$ ,  $\text{Fe}_2\text{O}_3$ ,  $\text{CaF}_2$ ,  $\text{Sb}_2\text{O}_3$ ,  $\text{SnO}$
- Provide information about the basic properties and usage areas of the type of glass specified by the instructor.
- Explain the formation of amorphous and crystalline structures depending on the cooling rate.

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## EXPERIMENT 9: CASTING AND RECYCLING

### 1. PURPOSE OF THE EXPERIMENT

The purpose of the experiment is the production of liquid metals which contain alumina with sand molding process, getting information about recycling and finding efficiency of the experiment by calculating ratio of recycling.

### 2. THEORETICAL KNOWLEDGE

#### FUNDAMENTAL DEFINITIONS

Casting: It is the method of production solidified molten metal whose metal's shape will be determined with respect to mold cavity.

Mold: It is the container which has capability of forming metal and solidifying liquid part of metal.

Core sand: It is a sand mass prepared in appropriate dimensions and placed in the mold for the parts of the part to be cast that are desired to be empty.

Runner: Special channel that sends the molten material to the mold cavity.

Riser: A column of molten metal placed in the mold to feed the castings as it shrinks and solidifies and it can be also called as "feed head".

Vent: Small opening in the mold to facilitate escape of air and gases.

#### INTRODUCTION

Five basic manufacturing methods are used in the manufacturing of metal-based parts; These are casting, plastic forming, machining, welding and powder metallurgy. Sometimes one of these, and often more than one, is used in part production sequentially. Casting is advantageous because liquid metal can take shape in fluidity in mold cavity, and casting parts can be produced in many different ways. These are large parts (engine blocks, cylinder heads, pistons, rims, etc. for automotive vehicles, wood burning furnaces, machine bodies, wagon wheels, pipes, large sculptures, pump bodies, radiators) and small parts (tooth coverings, jewelry, small sculptures, frying pans, valves, etc.) and all types of metals, ferrous and non-ferrous.

#### BASIC CASTING STEPS

1. Melting the metal.
2. Pouring into the mold.
3. Waiting to solidify for a while.
4. Breaking the mold and removing the part.

#### CASTING METHODS

A-Methods using expendable mold: A mold that must be distributed to remove wooden casting part after solidification

Mold materials: sand, plaster, similar materials, and binders

Casting types: sand mold casting, shell mold casting, ceramic mold casting, plaster mold casting and precision casting.

B-Methods using permanent mold: uses a permanent mold that can be used repeatedly to produce large numbers of castings.

Mold materials: Made of metal or a ceramic refractory material.

Casting types: metal mold casting, pressure casting, centrifugal casting and continuous casting.

Waste casting methods are more advantageous in terms of producing more complex, larger geometry-shaped parts, being easier to design and produce, being cheaper, and being able to cast materials with higher melting temperatures. On the other hand, the disadvantages of waste casting methods compared to methods using permanent molds are that it is difficult to produce very fine-grained casting parts and that they are more expensive for high-speed production.

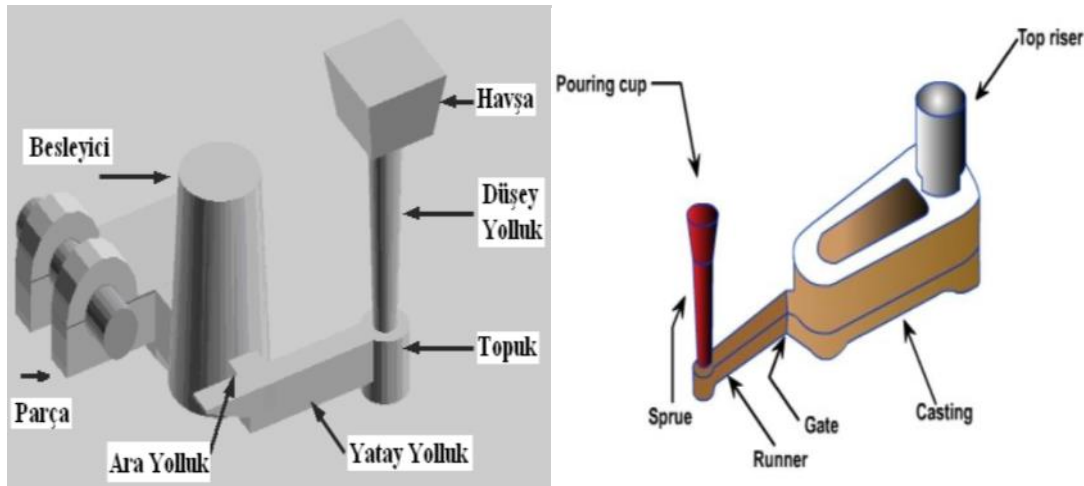
Part production with casting methods using spent molds consists of five basic steps performed sequentially. These are i) model design and manufacturing, ii) mold and core preparation, iii) melting and liquid metal processes, iv) mold filling, v) mold breaking and cleaning processes, respectively.

### MODEL DESIGN

The model is a design of the part to be cast, and its dimensions are determined by calculating shrinkage and machining allowances to the dimensions of the part to be cast. Models are made of wood, metal, wax/plastic and plaster. Models are divided into four: a) Single or free models, b) Free models with runners, c) models attached to the plate (plate model) and special models. In the first stage, the shape of the part is designed by taking into account the function that the part will see and the design criteria in casting. Then, the runner and feeder design is made. After the model design is perfected as much as possible through mold filling and solidification simulations, model manufacturing is carried out.

As explained above, a similar piece that shows the shape of the part required for the preparation of the mold is called a "model". The places where these model parts meet are called "divided surfaces". In mixed-shaped parts, it is desired that the model be easy to process, and if the number of parts to be cast is large, the model should be durable, and the model material is selected. The most commonly used model material is wood, although metal and plastic are also used in models.

Dimensions, divided surfaces, core heads and runner system adjustments must be made very carefully while making the models. In particular, the runner system is critical because the runner must be placed in the most appropriate place, therefore at the lowest point of the mold cavity, to prevent the liquid material from solidifying during the flow time and to prevent the mold from deteriorating during flow. Runners, vents and risers are the elements of this system. While the vents should be in the highest regions, the runners are placed in the lowest region of the mold cavity. Before casting, a melted metal bath is created, this bath serves as a riser and is called countersink. The liquid metal reaches the heel before the vertical runner. The heel is lower than the level of the horizontal runners and goes, preventing the liquid metal from scattering and creating turbulence. The basic elements of the runner system are shown in *Figure 1* below.



**Figure 1.** Runner System.

In addition, the composition of the alloy used in the sand mold has an effect on the preparation of the mold and the shaping of the part. In general, the sand mold material must contain sand grains that provide the necessary refractory properties and a water alloy composition that allows binding sand grains that are naturally present in the sand or can be added later to hold together, thus making the sand a suitable mold material. The manual molding process can be done on the scale with the model or on the ground with the help of the model plate. Plate models are widely preferred because they provide ease of molding. Frames called degrees are used to prevent the molds from falling apart due to compression pressure, turning and transporting. Degrees are made of wood or metal. Sand molding can be seen schematically in *Figure 2* and the side view of the mold in *Figure 3*.



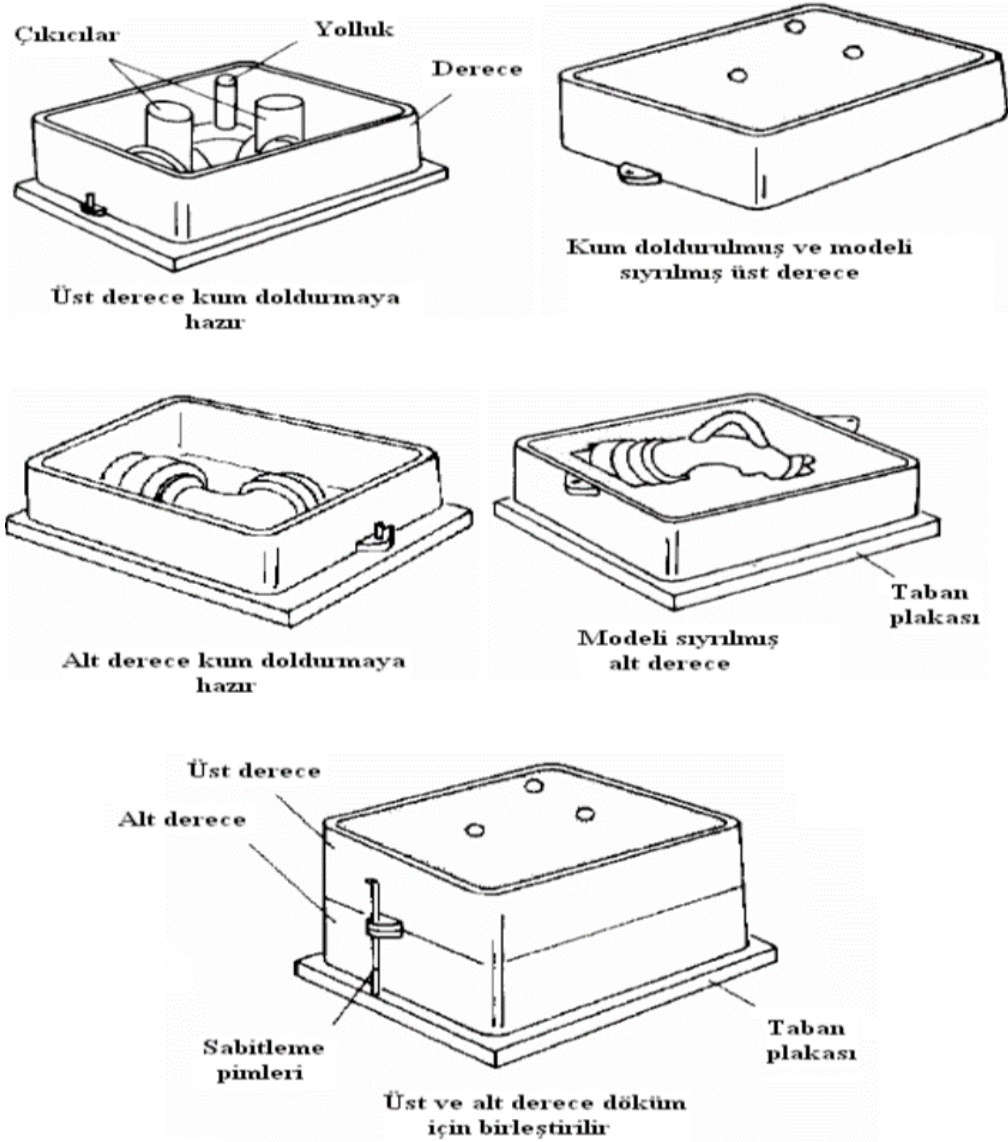
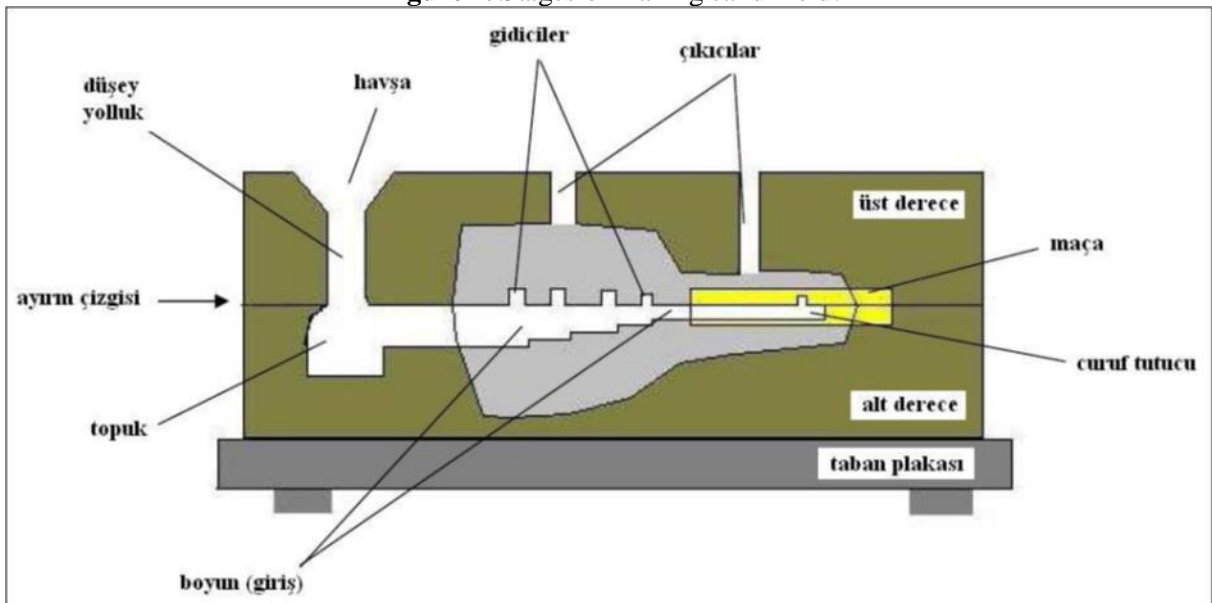
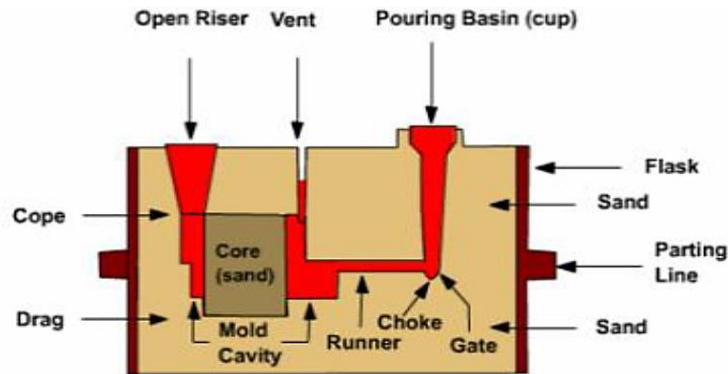


Figure 2. Stages of making sand mold.







**Figure 3.** One of the views for sand mold.

Sand cores are internal molds that are placed in the mold cavity and ensure that the parts they cover come out empty after casting. Sand cores are mostly made of sand. Metal cores can be used in casting methods using permanent molds. Sand cores are prepared in wooden or metal boxes called core boxes or core boxes. Organic binder and self-hardening special core sand mixtures are mostly used in core preparation. Once prepared for the match, it must have various features. It must be able to take shape and maintain its shape, be gas permeable, be heat resistant, have wet strength, dry strength, be able to withstand liquid metal pressure, and be flexible.

### **MOLD MATERIALS**

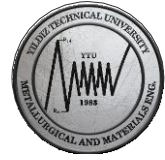
Alloy element composition was mentioned briefly in the mold and sand core preparation section. Silica sand is widely used in mold and sand core making. Silica sand is a cheap mold material that is easily obtained. Other types of sand are used in special applications requiring high refractoriness, high thermal conductivity and low thermal expansion. Silica sands must contain a minimum of 95-96% SiO<sub>2</sub>. As the SiO<sub>2</sub> content of the sand decreases, its refractoriness decreases. The grain size distribution of sand affects the quality of cast parts. While coarse-sized sands provide high permeability, facilitating gas release, they reduce the surface quality of the parts. The use of fine-grained sand increases the surface quality and reduces permeability. Additionally, fine-grained sands require more binder. The grain shape of the sand is as important as its grain size distribution. Angular sand grains have low packing density, poor mold ability and high binder requirements. For this reason, round shaped sands are preferred in mold making. Today, natural sands are rarely used in foundry. Synthetic sand mixtures, whose grain shape and distribution have been determined and the residues have been washed and removed, are preferred. The first method of binding sand grains together was the use of clay and water. Clay swells when combined with water and holds the sand grains together. This method, also called green sand molding, is now being replaced by chemical binder methods.

Clay bound mold sands can be used over and over again by re-introducing lost water and lost clay. Due to the high temperature in casting, some of the clay changes and loses its binding properties. The best binding clays are bentonites. Two types of bentonite are used: sodium and calcium based. Another mold additive used especially in the casting of iron-based alloys is pulverized coal powder. It has the effect of increasing surface smoothness and making peeling easier.

A wide variety of chemical binders are available for mold and core making. The majority of these are based on organic resin or sodium silicate, but were also used with inorganic binders such as cement in the early applications of chemical binders.

Chemical binders are used in two ways. In self-setting mixtures (also known as cold-setting), sand, binder and hardening chemical are mixed together. Hardening begins immediately but is slow enough to allow the sand to be shaped into a mold or sand core. After shaping, hardening continues and the mold or sand core is ready for casting. This method is mostly used in the production of large molds and is suitable for mass production.

In process hardening, sand and binder are mixed and shaped, no hardening reaction occurs during this time, hardening occurs in a short time with the application of heat or catalyst gas. This method is often used in making cores or molding small parts.



The most commonly used self-hardening resins are furan type resins. Acid catalyst is used as a hardener. Furan resins are obtained by adding furfury alcohol to urea formaldehyde, phenol formaldehyde, or a mixture of both. The hardening rate can be adjusted with respect to the amount and effectiveness of the acid catalyst. Some other self-curing resins are phenolic isocyanates (phenolic urethanes) and alkaline phenolic resins.

Sodium silicate, also known as water glass, is water-soluble glass, there are many types according to the silica ( $\text{SiO}_2$ ) and soda ( $\text{Na}_2\text{O}$ ) content. Sodium silicate can be hardened by a variety of methods, the addition of  $\text{CO}_2$  gas or liquid ester being the commonly used methods. Silicate type binders are odorless and not harmful to health, but the bond they form is not as strong as resin binders. Additionally, since it is an inorganic binder, it is not affected by heat and may cause distortion problems after casting. Therefore, various organic additions can be made directly to the liquid silicate or during sand mixing.

### ***Melting, Liquid Metal Processes Mold Filling, Mold Breaking and Cleaning***

The ingot, scrap and metal or alloy in work form to be used in the casting of the part are charged in a suitable melting furnace and the melting process is carried out. After the melting process, faxing, degassing, modification, grain reduction and grafting operations are performed depending on the type of metal or alloy. The chemical composition of the alloy is checked and adjusted. Following the liquid metal processes, after the temperature is brought to the desired values, the liquid metal is filled into the mold cavity appropriately. After solidification is completed and the part has cooled for a while, the mold is broken, the part is removed, cleaned, the runner and feeder are broken and made ready for finishing operations.

## **ALUMINUM CASTING**

Aluminum casting is the most used casting method after iron and steel. Aluminum casting can be cast with various alloys in different areas (table 1). One of the reasons why the aluminum casting method is used in many areas is that the aluminum element has many properties. It is preferred due to its many features such as being lightweight, resistant to corrosion, easy to shape, durable and recyclable, and having high conductivity, and the casting process is generally carried out by gravity mold casting, metal injection mold casting and sand mold casting methods. Aluminum sand mold casting method is widely used in the aluminum industry. In the aluminum sand mold casting method, a wooden model is first needed to produce the aluminum part. Aluminum is then cast into the sand molds prepared with this model. This method is generally preferred for parts that cannot be cast with other aluminum casting methods. Since the aluminum part to be produced has a very complex shape, the aluminum sand mold casting method can be used. In addition, in cases where a small number of aluminum parts are to be cast, the aluminum sand mold method is preferred because the mold cost is high in other casting methods. In other words, the aluminum sand mold casting method has advantages and other disadvantages, such as the production of very complex parts and low cost.

**Table 1.** Aluminum alloys classification.

<b>Al alařımı</b>	<b>Elementi</b>
1xxx	-
2xxx	Cu
3xxx	Mn
4xxx	Si
5xxx	Mg
6xxx	Mg ve Si
7xxx	Zn
8xxx	Li
9xxx	Yeni nesil

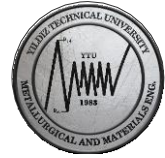
Tablo 1: Alüminyum döküm alařımının sınıflandırılması ve içerdđđ alařım elementi

### ***Advantages of Sand Mold Casting Method***

- It is suitable for casting ferrous and non-ferrous metal alloys.
- Sand molding is a suitable method for both small and very large parts.



## 2023-2024 Spring MSE3952 Laboratory II Experiment 9: Casting and Recycling



- Mold material is cheap and abundant.
- It is suitable for casting a small number of parts.
- It is the casting method with the lowest mold cost.

### ***Drawbacks of Sand Mold Casting Method***

- Surface and size precision is not good.
- Parts obtained from casting must be subjected to other processes.
- Molding causes a huge waste of time.
- Material consumption is high.

### ***FUNDAMENTAL DEFINITIONS OF RECYCLING***

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

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

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

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

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

The main scraps used for the aluminum recycling are;

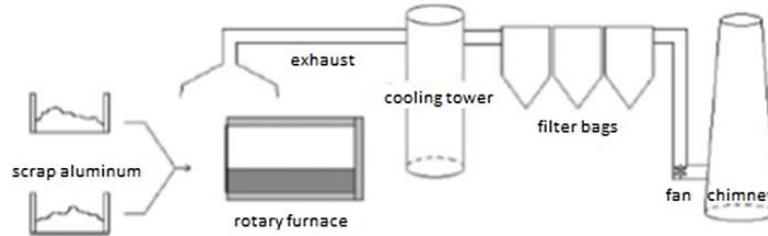
- Beverage cans,
- Aluminum foil wastes, cake pans, and some other kitchenware,
- Plates,
- Window profiles made of aluminum,
- Outdoor and patio furniture,
- Cables,
- Aviation scraps,
- Automotive scraps,
- Extrusion, casting and machining processes sourced scraps.



**Figure 1.** Images of some aluminum scraps.

At the first step, soiled and used aluminum pieces which may be painted or coated are delivered to the conveyor with other packaging wastes. Throughout the conveyor, waste pile is passed through some large magnets in order to remove iron-based and some other metals. Separated aluminum wastes are crushed and baled via scrap baling machine thereafter to be delivered to production facility. Main reason of baling is minimizing the logistic expenses. Baled aluminum may contain excessive moisture which may cause burst in furnace at high temperatures. In order to prevent this incident bales are crushed first. Afterward, crushed pieces

are placed into hot furnace for removal of present paint, moisture and other pollutions at working temperature. After removal stage, aluminum is molten for forming processes at 700 °C in a rotary furnace with accompanying stirring to attain complete melting. Process gases need to be removed with powerful fans then to be cooled and treated in industrial filter bags for removal of residual particles before releasing to atmosphere through high chimneys. Molten aluminum accumulated at the bottom of the furnace is collected to cast into desired molds. After several steps they can be used for diverse applications again.



**Figure 2.** Image of aluminum recycling unit

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

When one ton of aluminum is produced from used aluminum;

- 1 kg bauxite,
- 15.000 liter cooling water,
- and 860 liter process water are consumed less.
- 2.000 kg CO<sub>2</sub> and 11 kg SO<sub>2</sub> are less emitted.

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

### 3.EXPERIMENTAL STUDIES

In this experiment, a green sand mold will be prepared, thick scrap will be weighed before the experiment, and aluminum melted in an electrical resistance furnace will be poured into the prepared mold. The spilled aluminum material will be weighed and it will be determined how efficiently the experiment was carried out.

#### 3.1 Before the experiment

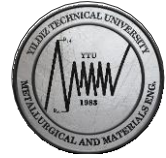
- ✓ Firstly, thick scrap, which will constitute the majority of the predetermined total melting amount, will be weighed.
- ✓ It is placed in the furnace in the melting crucible and kept for about 1 hour to melt at 720°C.

#### 3.2 During an experiment

- ✓ With this melting, a liquid metal bath is created in the crucible.
- ✓ During the melting of thick scraps, thin scrap chips are briquetted using a hydraulic press in a cylindrical steel mold.
- ✓ Before being placed in the furnace, the briquettes are preheated in the oven for a while.
- ✓ Preheating time and temperature will be determined during the experiment.
- ✓ Then, the briquettes are added to the liquid metal pool in the crucible.
- ✓ Powder chemicals that will form the flux during the melting process of the briquettes are weighed on a sensitive scale and mixed in a container.
- ✓ Afterwards, sand molding starts with a specified amount of sand and bentonite and water are loaded into the large sand mixer.



## 2023-2024 Spring MSE3952 Laboratory II Experiment 9: Casting and Recycling



- ✓ It is mixed until it becomes suitable for molding.
- ✓ The lower and upper degrees are placed on the molding plate.
- ✓ The plate model is placed between two degrees.
- ✓ It is turned so that the lower degree is on top.
- ✓ Graphite is sprinkled on the model.
- ✓ Sand is filled into the grade.
- ✓ The sand is gradually compacted using hand tools and a pneumatic rammer.
- ✓ The mouth of the runner is widened and countersunk.
- ✓ The skewer is pulled at 30-40 mm intervals.
- ✓ The model is created by separating the degrees from each other.
- ✓ If necessary, repairs are made within the mold.
- ✓ The grades are reassembled and the mold is made ready for casting.
- ✓ The prepared mold is brought to the front of the melting furnace.
- ✓ The created flux mixture is added into the crucible and mixed into the liquid metal by means of a rod.
- ✓ The crucible is removed from the furnace and the slag is removed.
- ✓ Liquid metal sand is filled into the mold.
- ✓ The remaining part is poured into the ready-made metal mold.
- ✓ Wait for solidification and cooling of the mold.
- ✓ The mold is broken and the casting piece is removed.

### 3.3 After the experiment

- ✓ Spilled metals (on the metal surface and in the sand mold) will be weighed and the total melting and recycling efficiency will be calculated.

## 4. INFORMATION THAT SHOULD BE INCLUDED IN THE REPORT

- a) Examples of parts produced by sand mold casting
- b) Casting errors caused by sand mold
- c) What are the controlled properties of molding sand and its mixture?
- d) Explain the grain reducers used in aluminum alloys and state in what proportions they are used.
- e) Give information about the modification agents used in aluminum alloys.
- f) Research the place of aluminum metal in recycling.
- g) Research how we can increase the efficiency of recycling achieved in the experiment.

## 5. REFERENCES

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2023-2024 Spring MSE3952 Laboratory II  
Experiment 10: Experimental Determination of  
Casting Ceramic Slurry into Plaster Mold and  
Determination of % Shrinkage Due to Drying, Firing,  
and Total Shrinkage



## EXPERIMENT 10: EXPERIMENTAL DETERMINATION OF CASTING CERAMIC SLURRY INTO PLASTER MOLD AND DETERMINATION OF % SHRINKAGE DUE TO DRYING, FIRING, AND TOTAL SHRINKAGE

### 1. PURPOSE OF THE EXPERIMENT

The aim of the experiment is to learn the slip casting method for ceramic slurries, to be able to shape them appropriately into plaster molds, and to determine the % shrinkage due to drying, firing, and total shrinkage of the cast ceramic molds.

### 2. THEORETICAL INFORMATION

Materials belonging to traditional ceramics can be categorized into two groups: clayey and non-clayey. Clayey materials include clay, kaolin, etc., while non-clayey materials include feldspar and quartz. These inorganic industrial raw materials are mixed in certain proportions, shaped, and then ceramic products are produced by firing and sintering.

*Clay:* It is a material composed mainly of finely grained minerals formed naturally, which generally becomes plastic when a sufficient amount of water is added and can harden with drying and firing. They are relatively soft and can be scratched with a fingernail. Clay facilitates shaping of ceramic products and provides the necessary raw strength.

*Quartz:* It is a non-plastic hard material. Its composition is  $\text{SiO}_2$ , showing the most pure chemical composition and physical properties among all minerals. It maintains the structure at high temperatures, reduces drying shrinkage, and allows gas to escape during firing without deformation.

*Feldspars:* They are alumina silicate minerals containing alkalis. They are not found in pure form. Depending on the alkali content, they facilitate sintering in the formation of ceramic products and, depending on the content, they form a glassy phase in the structure due to sintering and also exhibit a melting property.

*Kaolin:* These are aluminum silicates formed by the alteration of feldspathic rocks under natural conditions. Kaolins are generally white. They are the main raw materials of ceramics and give whiteness to the structure. As the percentage of  $\text{Al}_2\text{O}_3$  in kaolin increases, the firing temperature and strength also increase.

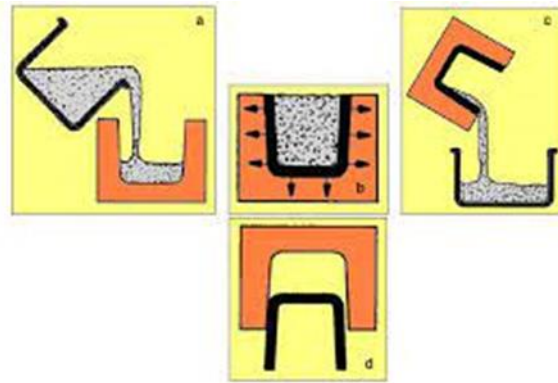
The slip casting method into plaster molds involves pouring the ceramic slurry (slip), which is prepared in the form of a solid-liquid mixture where the solid is homogeneously distributed in the liquid, held in suspension, and made flowable, into plaster molds. The water in the slurry is absorbed by the porous plaster mold. To obtain slip casting slurry, clayey (hydrophilic, water-loving) ceramic raw materials such as clay and kaolin are mixed with non-clayey (hydrophobic, water-repelling) ceramic raw materials such as feldspar and quartz by grinding them in ball mills to ensure homogenous mixing. One-third of the volume of the ball mill consists of solid raw materials, one-third of water, and the remaining one-third consists of grinding alumina balls. After sufficient ball milling, large particles are removed by sieving, and then the slurry (slip) ready for casting is mixed under mechanical mixers. The final slurry becomes ready for casting into plaster molds.

A good casting slurry should have the following properties:

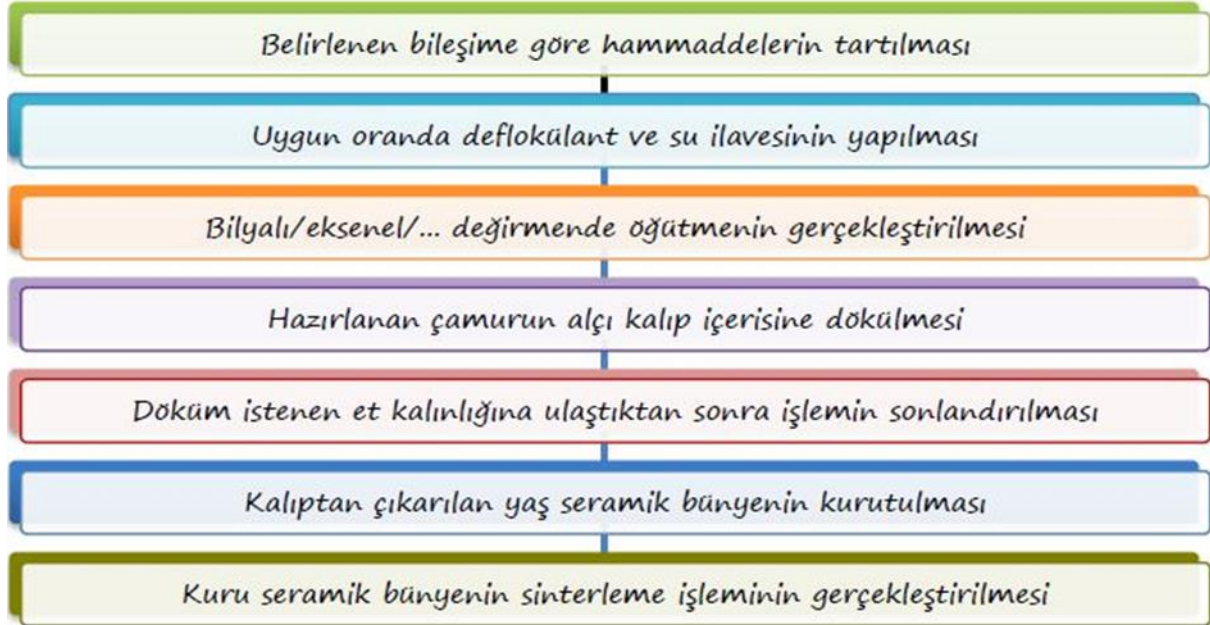
- ✓ It should have low viscosity to spread easily inside the plaster mold.
- ✓ Solid particles should not settle.
- ✓ It should be easily removable from the mold after casting.
- ✓ It should have high strength after casting.
- ✓ It should have low drying shrinkage.

The plaster casting method is preferred for the production of large-volume and complex-shaped products. This

method is used in shaping asymmetrical products, some special refractory bricks, tableware, and sanitary ware such as sinks, toilets, and bathtubs, as well as in the production of advanced ceramics. Plaster molds used in casting are porous and have the ability to absorb water. However, porous synthetic materials are also used. For healthy production through casting, it is necessary for the mold to be suitable for the molding technique, meaning it should have continuous joints, no sticking during opening, be resistant to impact, have uniform thickness, suitable porosity, and be adequately dried. Additionally, it is crucial for the mold to be clean and have smooth joint surfaces during preparation. Plaster molds absorb water very quickly. The water in the ceramic slurry poured into the plaster mold is rapidly drawn by the mold surface. As the layer of water between them decreases, the pushing forces between the particles, which approach each other, are overcome by the capillary forces of the particles. Thus, the particles begin to accumulate on top of each other and adhere to each other. As time passes, the number of accumulated particles increases, and a solid-liquid interface layer forms starting from the inner surface of the mold. The thickness of this layer increases over time, and the rate at which it thickens is known as the rate of thickness gain. This rate decreases over time.



**Figure 1.** Schematic representation of shaping with slip casting method.



**Figure 2.** General flow diagram for the production of ceramic products through casting method.

An important issue with ceramic products is that the dimensions may not remain constant from one part to another; different shrinkages may be observed from one to another. Therefore, shrinkage during drying or firing is one of the most important properties that need to be controlled in ceramic structures. High shrinkage can lead to deformation and damage to many ceramic products during drying or firing processes. Therefore, markings are made on the samples after shaping, and comparisons are made after drying and firing. Depending on the desired qualities of the product being formed, it is desired that the shrinkage does not exceed a certain percentage, for example, in porcelains, approximately 16% shrinkage is not desired.



2023-2024 Spring MSE3952 Laboratory II  
Experiment 10: Experimental Determination of  
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Determination of % Shrinkage Due to Drying,  
Firing, and Total Shrinkage



Formulas for calculating drying, firing, and total shrinkages are given below.

$$\% \text{ Yoğrulma Suyu} = \frac{(\text{Plastik Ağırlık} - \text{Kuru Ağırlık})}{\text{Kuru ağırlık}} \times 100$$

$$\% \text{ Kuruma Küçülmesi} = \frac{(\text{Plastik Uzunluk} - \text{Kuru Uzunluk})}{\text{Plastik uzunluk}} \times 100$$

$$\% \text{ Pişme Küçülmesi} = \frac{(\text{Kuru uzunluk} - \text{Pişme Uzunluğu})}{\text{Kuru uzunluk}} \times 100$$

$$\% \text{ Toplu Küçülme} = \frac{(\text{Plastik Uzunluk} - \text{Pişme Uzunluğu})}{\text{Plastik Uzunluk}} \times 100$$

### 3. EXPERIMENTAL STUDIES

#### 3.1. Devices and Materials Used

Plaster mold, Mechanical mixer, Viscometer, Scale, Casting slurry, Plastic beaker, Sponge, 1200°C Furnace, Caliper.

#### 3.2. Implementation of the Experiment

Firstly, the viscosity value and liter weight of the previously prepared ceramic slurry will be measured. With these analyses, the rheological analysis of the slurry is performed, and its suitability for casting is determined. Subsequently, plaster molds are prepared for casting, and the ceramic slurry is filled into the mold. At this point, to ensure the efficiency of the experiment and for comparability, the relevant experimental group will be divided into pairs or groups of three, and the ceramic slurry will be allowed to stand in the mold at different intervals within each group. It is expected that ceramic molds waiting for different minutes will have different wall thicknesses when removed from the mold. Therefore, each group will empty the excess slurry from their ceramic mold by waiting for 3 minutes, 6 minutes, and 9 minutes, respectively, and then the mold will be placed upside down on the table, and some time will be allowed for the ceramic to be easily removed from the mold. Subsequently, the ceramic will be carefully removed from the mold and left in the open air for 10 minutes. Then, each group will write their code carefully on the bottom of their product. Thus, confusion between ceramic products among groups is prevented.

After casting, initial measurements are taken. These include determining the wall thickness, determining the diameter of the product, and measuring the weight on a precision scale. After completing these procedures, each group leaves their products to dry. Since the necessary time for the product to dry completely will exceed the class hours, to ensure the continuity of the experiment and to develop production process awareness, the weight of the completely dried product will be measured again at the day and time provided by the experiment assistant in the following week. After the measurements taken on the same day, the ceramic products are placed in the furnace for firing. The firing temperature is 900°C, and the holding time is 2 hours. After this time, the weight of the fired product is measured, and the wall thickness and diameter are measured. Thus, the experiment is completed with the obtained data. To utilize these data, % plasticity water, % drying, % firing, and % total shrinkage values within the folio are calculated.

### 4. RESULTS

As a result, in this experiment, ceramic slurry is poured into plaster molds, and the process is continued with drying and firing processes. With the data obtained after these steps, it becomes possible to make comments about the relevant product and to prove that ceramic slurries waiting for different times after casting have different wall thicknesses.



2023-2024 Spring MSE3952 Laboratory II  
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## 5. REQUIRED IN THE REPORT

- 1- Show the relationship between Waiting Time - Wall Thickness and Waiting Time - % Total Shrinkage in graphics (prepared using Excel).
- 2- Explain the reasons for shrinkage during drying and firing.
- 3- What would you do to obtain thicker or thinner casting thicknesses in the same period?

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