Yıldız Technical University

Metallurgical and Materials Engineering Department

MSE3122 Material Characterization Laboratory

Experimental Booklet

2024-2025
Fall

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Lab Rules and Guidelines

1. A total of 10 experiments will be conducted throughout the semester.
2. Students who arrive more than 10 minutes late will not be allowed to participate in that week's experiment.
3. Missed experiments will be graded as zero.
4. You are allowed a maximum of 2 absences. A student who misses 3 weeks of experiments will be considered absent.
5. Experiment sheets and report formats will be uploaded to the department's webpage.
6. Reports of the experiments conducted must be submitted to the relevant research assistant no later than one week after the experiment, before the next session begins.
7. No experiments will be conducted during the midterm exam week.
8. Experiments will be conducted in person. Each student must attend the experiment with their assigned group. Experiment groups will be announced.
9. The final exam will contribute 40% to your overall grade.
10. In-term assessments will contribute 60% to your overall grade.
11. In-term assessments include quizzes, reports, and performance in experiments.
12. A short quiz will be conducted before each experiment.
13. It is your responsibility to attend the quiz at the scheduled date and time. No excuses will be accepted.
14. The average score of the pre-experiment quizzes will contribute 30% to your overall grade.
15. Students who score below 40 points on the quiz will be considered unsuccessful and will not be allowed to participate in the experiment. There will be no make-up sessions for failed experiments.
16. Experiment reports must be prepared individually and in the specified format.
17. Reports that do not comply with the required format will not be evaluated. Identical reports will be graded as "ZERO."
18. Active participation in experiments is mandatory. Your engagement will affect your performance evaluation.
19. Performance (10%) + Quiz (30%) + Report (20%) + Final Exam (40%)
20. A one-week make-up session will be scheduled at the end of the semester. Students who missed an experiment due to a valid medical excuse can apply for a make-up session by submitting a copy of their medical report and a petition to the department secretary. This can only be done once.
21. All students must adhere to occupational health and safety rules when attending experiments. For example, wearing short shorts or open-toe shoes is not allowed, and artificial nails should not be worn.
22. All students are required to bring and wear a white lab coat, laboratory goggles, and gloves during the experiment.

Experiment 1: X-Ray Diffraction (XRD)-Scanning Electron Microscope (SEM) Characterization Techniques and Sample Preparation

Res.Asst. Serra ERSOY

1. XRD Analysis

1.1. Aim of the Experiment

The XRD analysis, which provides information about the material's crystallinity, phase purity, phase identity, crystal structure, phase composition percentage, and crystallite size, will be explained, including how it works, the device's mechanism, sample preparation, and analysis interpretation. The drawing of XRD graphs and material identification, which are used in many fields ranging from rock identification to determining kidney stone types, detecting impurities in pharmaceuticals, and analyzing metals, alloys, and polymers, will be covered.

1.2.Theoretical Information

X-rays were discovered in 1895 by Conrad Rontgen. Since their physical properties were unknown at the time, they were named 'X,' and they are still referred to by this name today. X-rays are electromagnetic waves with wavelengths ranging from approximately 0.02 Å to 100 Å (1 Å = 10^-10 m) (radiation that has both electric and magnetic properties) and are invisible to the human eye. The position of X-rays in the electromagnetic spectrum is shown in ***Figure 1*** [1,2].

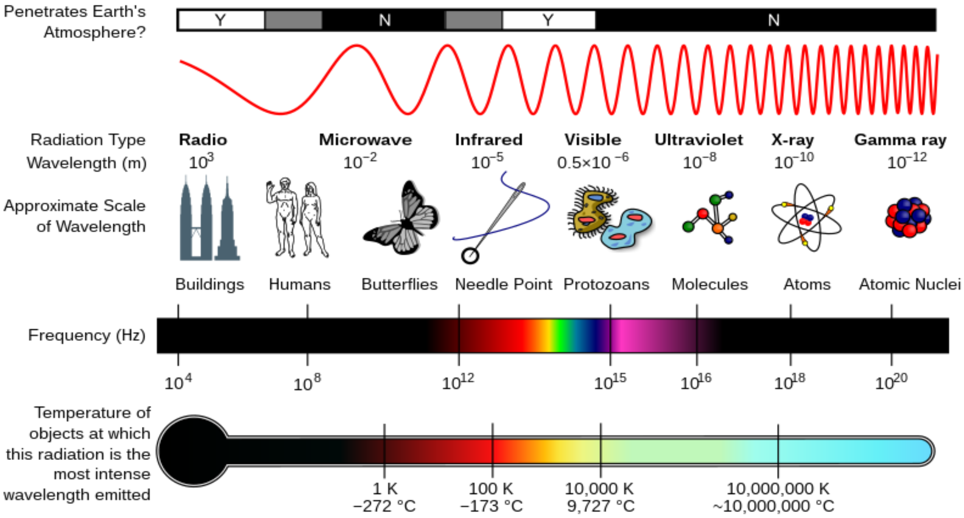


Figure 1. Electromagnetic spectrum and its scaling [2].

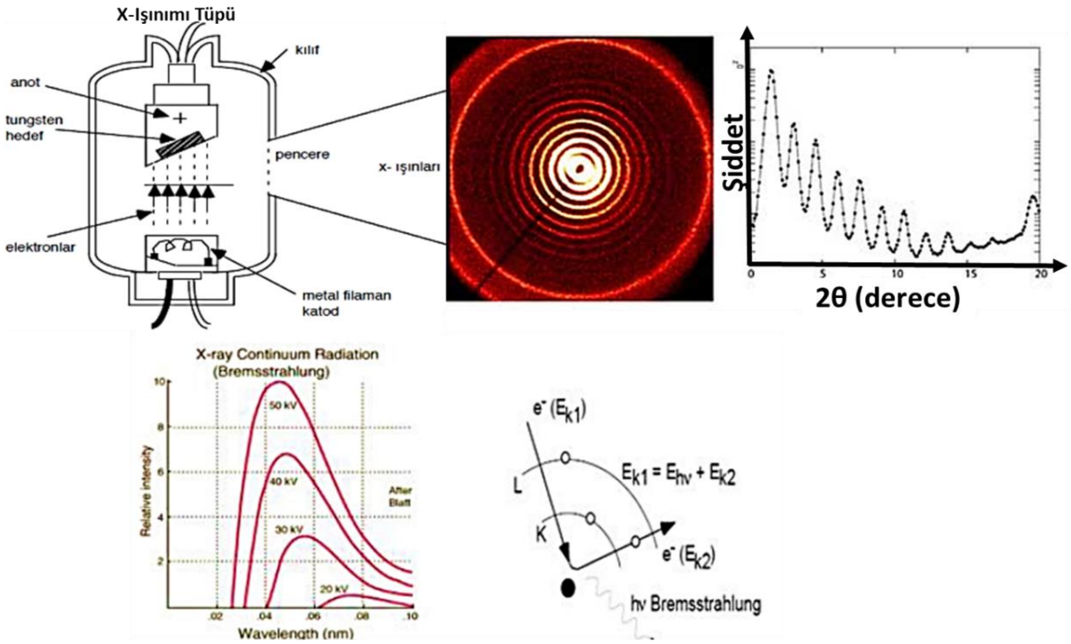
X-ray diffraction (XRD) is a technique used to determine the crystallographic structure and chemical composition of a material. XRD provides information about the material's chemical composition and crystal structure by irradiating the material with incoming X-rays and then measuring the intensity and scattering angles of the X-rays that leave the material. Qualitative and quantitative analyses of solid, powder, and thin film samples can be performed using an X-ray diffraction device [1,2].

The basis of this method is the analysis of a beam of X-rays, which consists of electromagnetic waves with very short wavelengths, directed onto the sample to be analyzed, and reflected after hitting the atoms of the crystals. Using X-ray techniques, the crystal class of a mineral, unit cell parameters, and the positions of various atoms in the crystal structure can be determined. In other words, crystallography studies the arrangement of atoms and molecules in three-dimensional (space) form using X-rays [1,2].

How Does an XRD Device Work?

X-rays occur in two forms: natural and artificial [3]:

- Natural X-Rays are produced by electron capture from the K energy shell by the atomic nucleus, alpha decay, internal conversion and beta decay.
- Artificial X-Rays are produced by the interaction of matter with accelerated particles such as electrons, protons, particles or ions, or by the interaction with photons from an X-ray tube or other suitable radioactive source. Characteristic (line) X-rays are produced by the interaction of matter with photons, and both characteristic and continuous X-rays are produced by the interaction of matter with charged particles. The rays in XRD analysis are produced by the X-ray tube.



X-Işını sürekli spektrumu

Bremstrahlung Işınımı

Figure 2. X-Ray Tube and X-Ray types according to interaction types [3].

The X-ray tube is a high-voltage cathode ray tube. The tube consists of a glass envelope from which the air has been evacuated under a high vacuum. One end contains the anode (positive electrode), and the other contains the cathode (negative electrode), both of which are tightly sealed with solder. When a high voltage is applied between the anode and cathode, electrons are emitted from the cathode. These electrons are accelerated towards the anode under high voltage and reach high speeds before striking the target. When the high-speed electrons collide with the metal target, they transfer their energy and emit a photon. These photons, whose energies are equal to the difference between energy levels, are called characteristic X-rays. A moving electron has kinetic energy. When a high-speed electron strikes the tungsten, it collides with a tungsten atom. The electron may collide with several atoms before being stopped. A small fraction (one percent or less) of the kinetic energy lost during the electron's deceleration is converted into X-ray radiation, while the remaining energy is transformed into heat. For this reason, the X-ray tube also has a cooling system (***Figure 2***) [3].

When X-rays are incident on a crystal structure, the rays are completely reflected from the solid surface with small angles of incidence and the rays are scattered by parallel planes of atoms in the crystal. This scattering in the crystal structure is called diffraction and diffraction occurs from scattering involving many atoms. The diffraction of X-rays in the crystal structure is explained by Bragg's Law and the simplest form of Bragg's law is given by the formula below (***Figure 3***) [3]:

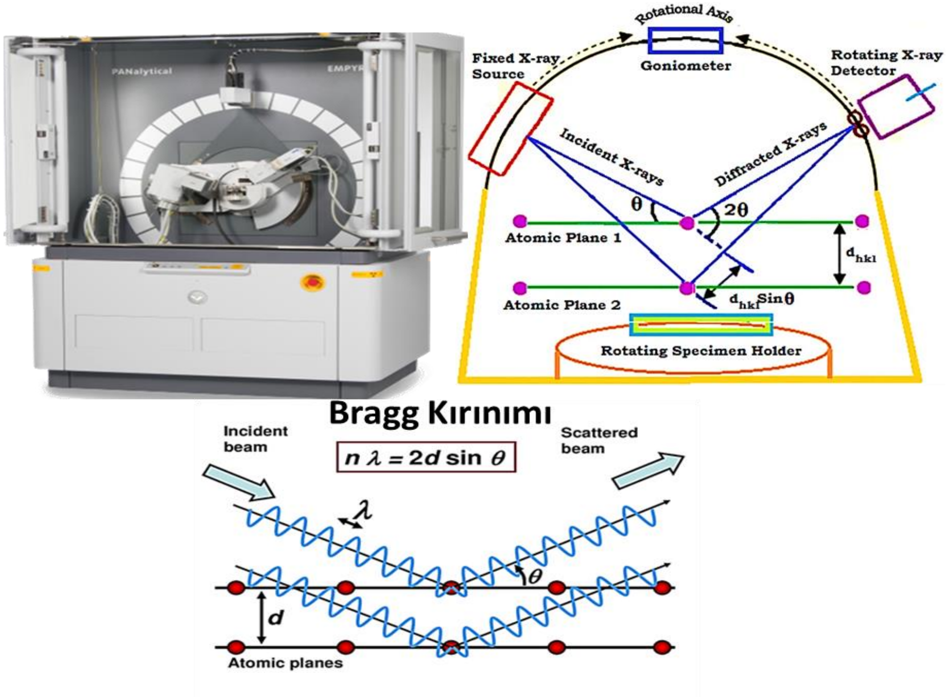


Figure 3. XRD device, working mechanism and Bragg diffraction formula [3].

Modern X-ray devices are equipped with counters that measure the diffraction angle and intensity of the diffracted radiation. In this way, a diffraction pattern is obtained, showing how the diffraction angle (2θ) changes with the intensity of the diffracted radiation. By examining the peak widths and background intensity on the pattern, information about the material's crystallization can be obtained [3]. The detector moves in a circular motion around the sample, and the detector position is recorded as the 2theta (2θ) angle. The detector records the number of X-rays observed at each 2θ angle, and the X-ray intensity is typically recorded as "counts" or "counts per second" [4,5]. In some devices, while the sample and detector rotate, the X-ray tube remains stationary. In others, the sample is held stationary while the detector and X-ray tube rotate (***Figure 5***).

Crystallography

Amorphous solid is a term used for solids that do not have a stable crystal structure. Materials like glass, polymers such as polystyrene, foods like cotton candy, and cosmetics like lipstick can be examples of amorphous solids. In amorphous (non-crystalline) crystals or minerals, atoms or molecules are arranged in a random order. The term 'crystal' refers to solids that show a regular internal structure, whether they have smooth external surfaces or not. Crystals, due to valence bonds, bring together a specific number and arrangement of neighboring atoms for each atom, creating structural compatibility within molecules. Thus, repetition can be observed along a linear polymer. Although XRD analysis is known to be applied to crystalline materials, amorphous materials can also be studied. In a crystalline material, when X-rays are diffracted by planes in the unit cell, detectors can detect which plane the diffraction came from. The diffraction peak positions from the published reference pattern can be used to determine the Miller indices (hkl) [4]. The (hkl) plane information is specific data known as the JCPDS card for materials, and through this, information on the chemical compositions, defects, impurities, and crystallinity within the material can be obtained. A phase is a specific chemical and atomic arrangement. For example, quartz, cristobalite, and glass are different phases of SiO_2. They are chemically identical but the atoms are arranged differently. As shown, the X-ray diffraction pattern is different for each phase. Amorphous materials like glass do not produce sharp diffraction peaks. The diffraction pattern of a mixture is simply the sum of the diffraction patterns of each phase (***Figure 4***) [4].

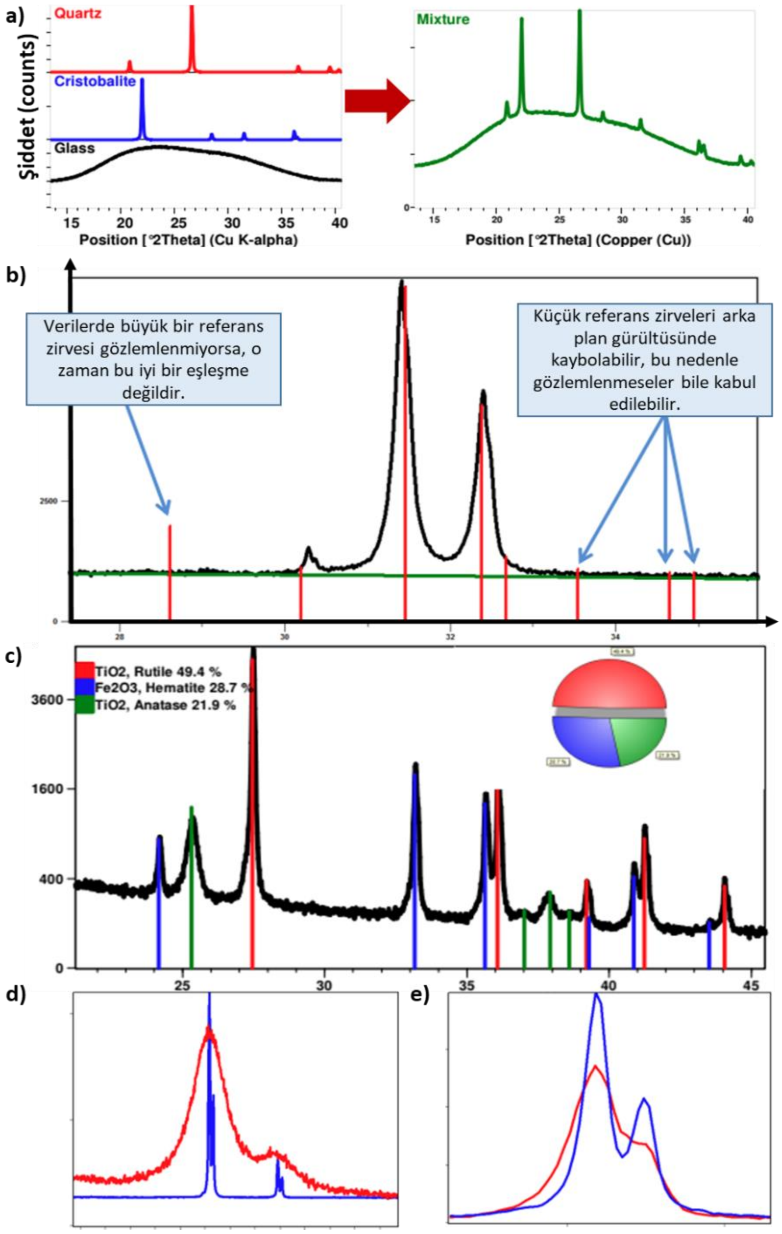
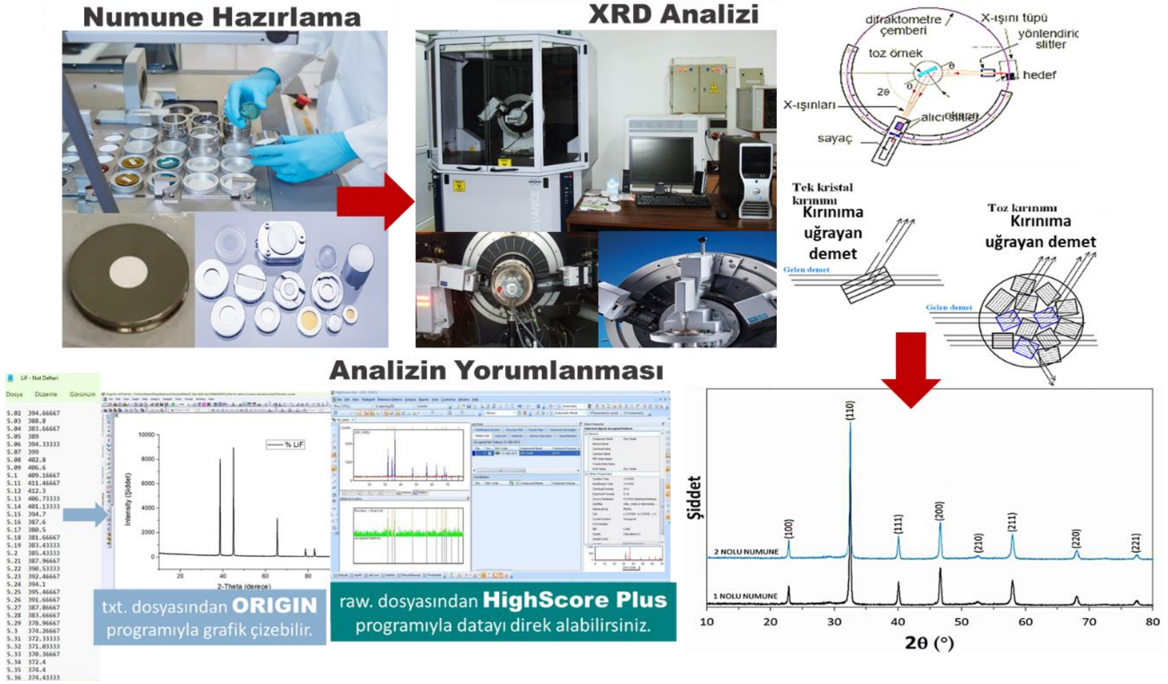


Figure 4. Examples of XRD graphs: **a)** Sum of peaks in a mixture of different phases of SiO_2, **b)** matching of XRD graph with reference peaks, **c)** XRD graph of a sample with mixed phases, **d)** solid (blue) and nanoscale (red) peaks of the same sample and **e)** graphs of the same sample taken on two different devices [4].

1.3. Sample Preparation and Interpretation of XRD Analysis



```
5.02  394.66667
5.03  388.8
5.04  383.66667
5.05  389
5.06  394.33333
5.07  399
5.08  402.8
5.09  406.6
5.1   409.16667
5.11  411.46667
5.12  412.3
5.13  406.73333
5.14  401.13333
5.15  394.7
5.16  387.6
5.17  380.5
5.18  381.66667
5.19  383.43333
5.2   385.43333
5.21  387.96667
5.22  390.53333
5.23  392.46667
5.24  394.1
5.25  395.46667
5.26  391.66667
5.27  387.86667
5.28  383.66667
5.29  378.96667
5.3   374.26667
5.31  372.33333
5.32  371.03333
5.33  370.36667
5.34  372.4
5.35  374.4
5.36  374.43333
```

Figure 5. Preparation of samples according to sample amount and type for XRD analysis, placing them in the XRD device with the sample holder and interpreting the data with Origin/HighScore Plus programs.

- <u>Powder samples:</u> Samples to be analyzed should be delivered to the technician in finely ground powder form. Sufficient information should be provided about samples that have undergone various physical and chemical processes. The number of samples to be delivered to the laboratory in powder form should be approximately 10 gr for rock samples; and at least 100-150 mg for samples synthesized in the laboratory and not available in large quantities [5].
- <u>Thin films:</u> Thin film samples to be delivered to the Central Laboratory should be prepared with a minimum size of 1x1 cm and should not be thicker than 7 mm [5].

Sample Preparation

- ✓ Depending on the type and amount of sample, the sample to be analyzed is poured into appropriate sample holders so that the surface is flat.
- ✓ After the sample is prepared, it is inserted into the sample section of the XRD analyzer, and the X-Rays are activated.
- ✓ A well-prepared sample should have <u>*crystallite and particle size of 1-5 μm, crystallites should be randomly oriented (no texture), should have a completely flat surface, sample height should be normal and should be able to be drilled short.*</u>
- ✓ The main problems that may arise from sample preparation are:
  - **Granularity:** If the sample is not ground well, single crystals will be present in the sample and granularity will occur. Therefore, if there are several large crystals in the sample and they are scanned, unexpected strong peaks may occur. To solve the problem, fine grinding, large sample holder and rotating sample holder devices can be preferred [6].
  - **Micro-absorption:** Large particles absorb a significant portion of the radiation. Absorption is less common in small particles. The problem is solved by fine grinding [6].

- **Preferential Orientation:** A good powder sample contains particles in all possible directions. However, some powder samples contain particles in only one direction. The problem can be solved by pressing the powder without rubbing it too much on the surface, using a reloading technique, and keeping the surface irregular in texture [6].
- **Crystallite size:** Crystallite size is calculated from the peak width. Good and correct grinding is important. Because in samples ground with high energy, the crystallite size is smaller than the ideal size. When it is smaller, peak broadening, lattice defects, stress in the crystal structure and amorphous cutting that is not visible to XRD occur. Therefore, ideal grinding is very important and there is an automatic sample grinder for this [6].
- **Sample height change:** If the sample height is too low or high, the peaks will shift and broaden. There will also be a loss of intensity [6].
- **Surface roughness:** If the sample surface is hard and rough, the peaks will shift and broaden [6].
- **Sample transparency**: If the sample surface is transparent, the peaks shift and broaden. For the sample transparency problem, it is necessary to use a very thin sample and allow the beam to pass through the sample. In other words, instead of the reflection method, the transmission method should be used [6].

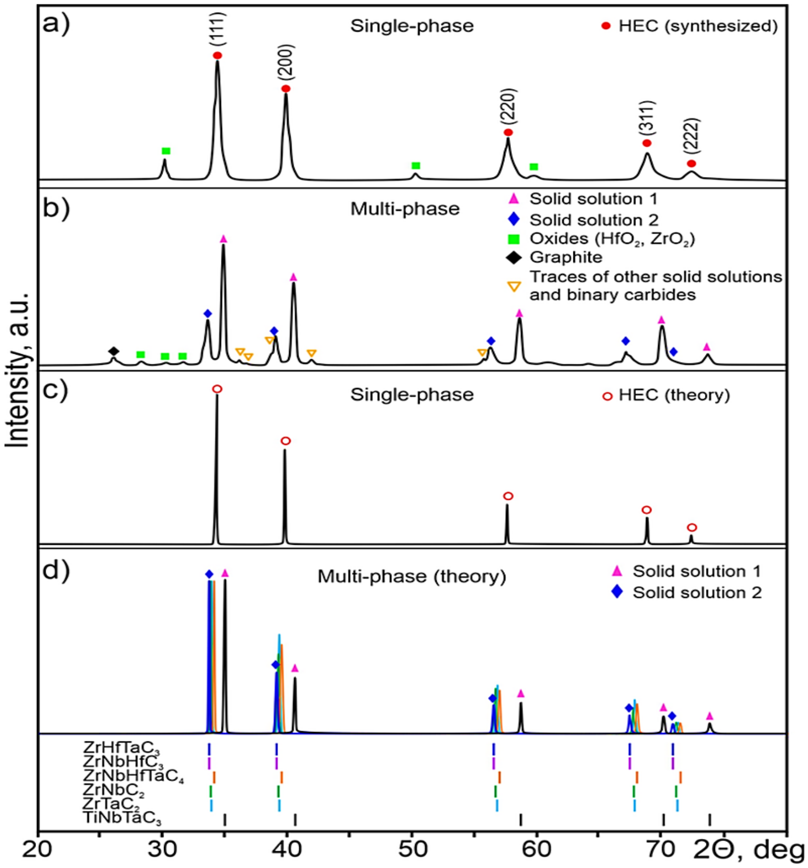


Figure 6. X-ray diffraction patterns of the synthesized **a)** single and **b)** multi-phase TiZrNbHfTaC5 samples and **c)** XRD patterns of the individual phases of ZrHfTaC3, ZrNbHfC3, ZrNbHfTaC4, ZrNbC2, ZrTaC2 and TiNbTaC3 found by analyzing the calculated Gibbs free energies of the simulated single-phase HEC and **d)** mixing of various multi-component carbides [7].

As seen in ***Figure 6***, interpretation can be made by using XRD analysis in the identification of single and multiple phases.

2. SEM Analysis

2.1.Aim of Experiment

Through SEM analysis, which is used for atomic-level examination of the sample’s topographical and two-dimensional images and elemental mapping for material identification, students will gain an understanding of the differences between this method and optical microscopy, the device's working mechanism, sample preparation, and analysis interpretation. It will be learned that various materials such as metals, textiles, fibers, plastics, polymers, and particles (such as sand, gravel, and pollen) can be studied. Additionally, engineering candidates will be able to make comments on the use of SEM characterization in a wide range of fields, from health sciences and forensic analysis of evidence to the study of rock types in archaeology and fundamental and engineering sciences.

2.2.Theoretical Information

Everything in nature is composed of building blocks too small for our eyes to see. To view these building blocks, equipment that can magnify the image is required. The optical microscope, first developed by Leeuwenhoek in the 1600s, could only achieve a 270x magnification. However, with the advancement of technology, by the early 1900s, optical microscopes capable of higher magnifications were developed, reaching a maximum magnification of 2000x with the use of 365 nm wavelength UV radiation and the resolution relation α λ (wavelength). Optical microscopes, using both the wave and particle properties of light, can assist in imaging down to the size of bacteria (1 μm) as shown in ***Figure 7***, but they cannot perform absolute focusing. The resolution (the smallest distance that can be seen) is related to the wavelength of the light used, and as the wavelength decreases, smaller objects can be seen. Despite having small wavelengths, X-rays and gamma rays in the electromagnetic spectrum can still increase magnification up to a certain point but cannot be focused or directed [8].

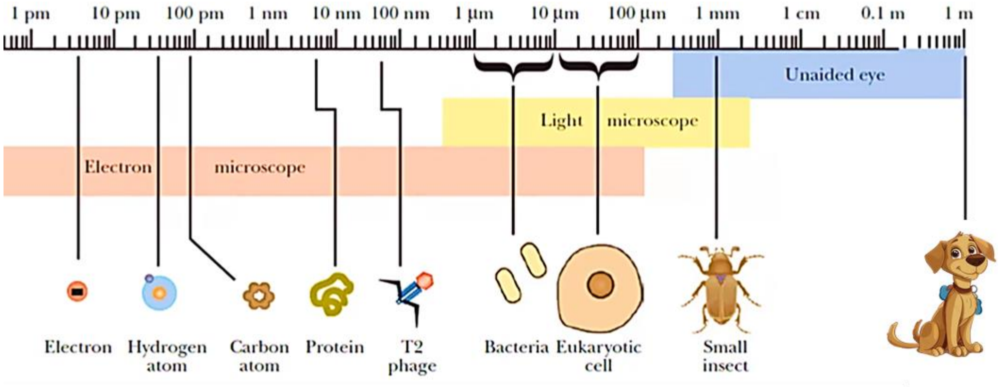


Figure 7. Size spectrum of objects that can be examined with the naked eye, light microscope, and electron microscope.

For this reason, electron microscopes were developed. When a stationary electron is placed in a potential difference of 1 volt, its energy value becomes 1 electron volt (eV), and this unit of energy is used to define many elements. The wavelength is related to the electron volt. For example, blue light has an energy of approximately 2.75 eV and a wavelength of 450 nm, while the energy of an electron in the power grid is 220 eV with a wavelength of 0.0825 nm. According to the relationship between resolution and wavelength, electron microscopes can be used to examine the atoms and elements in materials at higher magnifications. Additionally, since electrons are affected by both magnetic and electric fields, focusing is also possible [8].

There are two types of electron microscopes: ***Transmission Electron Microscope (TEM)*** and ***Scanning Electron Microscope (SEM)***. TEM and SEM are not interchangeable microscopes but are complementary to each other. In TEM, electrons generated by an electron gun are focused on a sample less than 100 nm thick, and the electrons pass through the sample, projecting an image of the sample's projection onto a screen or camera. 2D images are obtained, and high voltage is required with electromagnetic coils. TEM is used to visualize individual atoms, study atomic defects, and examine atomic arrangements. Although it can achieve very high magnifications (resolutions smaller than 50 pm), it is limited because the sample must be very thin. SEM, on the other hand, offers advantages such as roughness, contamination detection, obtaining topographical images, and easy sample preparation. 3D images can be obtained, and lower voltages are used compared to TEM. Its disadvantage, however, is that its resolution is limited to 0.5 nm [8].

How Does a SEM Device Work?

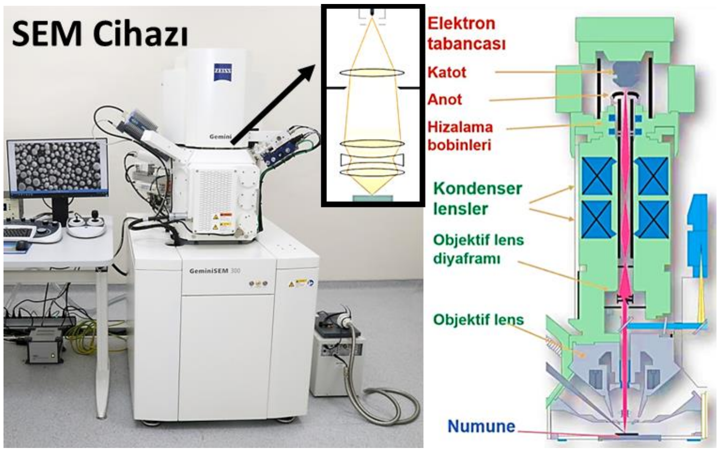


Figure 8. SEM device and its working mechanism [8].

Scanning Electron Microscope consists of three basic parts: “Optical Column”, “Sample Cell” and “Imaging System” [9].

In the optical column section:

- Electron Gun (Cathode Plate): It is the source of the electron beam.
- Anode Plate: This is the plate to which high voltage is applied to accelerate electrons towards the sample.
- Condenser Lenses: Used to obtain a thin beam of electrons to be incident on the sample surface.
- Objective Lens: Used to focus the light beam on the sample.
- Apertures and Coils: Apertures of various diameters attached to the lens allow the electron beam to be incident on the sample surface. Scanning coils used for scanning the surface are in this section.

Lens systems use electromagnetic fields to thin or focus the electron beam onto the sample. The entire optical column and sample are kept in a vacuum of 10^{-4} Pa [9].

In the sample cell section:

There is a sample table where the samples to be imaged will be placed. After the samples are placed and fixed on this table, the air in the sample cell is vacuumed to perform the imaging process. In this way, the electrons are directly incident on the sample in a vacuum environment without being directed towards any other deflector or direction [9].

In the imaging system:

There are detectors that collect various electrons and radiation generated because of the electron beam and sample interference, their signal multipliers and magnetic coils that scan the electron beam on the sample surface synchronously with the image screen [9].

Briefly, the electrons generated from the electron gun are focused on the smallest possible unit of the sample with the help of lenses (***Figure 8***). The electrons focused on the sample are scanned pixel by pixel by moving across the surface. As the electrons scatter, the detectors only detect intensity information and create bright, dark, or gray colors for specific pixels, thus scanning the material sequentially. For this reason, electron microscope images are black and white. In the interaction between electrons and matter, when an electron strikes the material, different secondary electrons are emitted [8]. SEM generates high-energy primary electrons and focuses them into a tight beam, which is then scanned across the surface of the material. The electrons penetrate the sample surface, with both elastic and inelastic random collisions causing the electrons to scatter within an interaction volume known as a teardrop shape. These interactions continue until the primary electron loses energy or is ejected from the material; the deflected electron path is random and can be modeled as shown in ***Figure 9***. These collisions produce secondary electrons, backscattered electrons, and specific signals called characteristic X-rays [9].

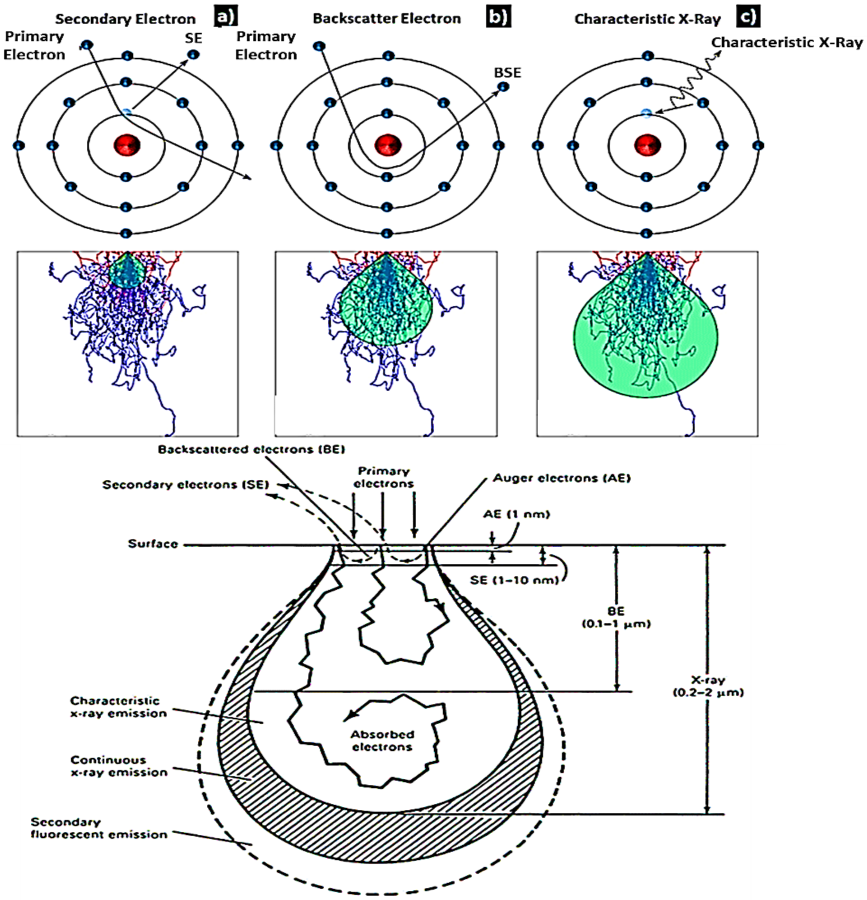


Figure 9. Schematics of signals generated from SEM: **a)** secondary electrons (SE), **b)** backscattered electrons (BSE), and **c)** characteristic X-rays. The blue paths end in the material and red paths are ejected as BSE [9].

- ***Secondary electrons (SE)*** result from inelastic collisions between primary electrons and atoms in the material, where the energy from primary electrons ejects a bound electron from the material (***Figure 9.a***). SEs have the smallest interaction volume because their low energy does not allow long-distance travel, and only SEs produced near the surface can be detected by SEM. SE micrographs are excellent at revealing surface topography and have the highest resolution [9]. (High resolution-topographic image)

- ***Backscattered Electrons (BSE)*** result from elastic collisions with atoms; they are deflected from the primary electron path, but energy is conserved (***Figure 9.b***). With sufficient collisions, the electrons are scattered from the material and return along their original path. BSEs originate from a deeper interaction volume because more collisions are needed to reverse their direction, and their higher energy allows for longer travel. Therefore, BSEs have lower resolution and do not reveal surface features. However, atoms with higher atomic numbers (Z) and densities are more efficient at producing BSEs compared to lighter elements, creating sharp contrast based on composition. BSE micrographs are useful for revealing phase properties [9]. (Atomic-elemental contrast)
- ***Characteristic X-rays*** are produced when a low-energy electron is ejected, leaving a vacancy that is later filled by a high-energy electron. When this occurs, a single photon with energy equal to the difference between the low and high orbital energy levels is emitted as an X-ray (***Figure 9.c***). The energy of the X-rays is characteristic of the atom from which they are ejected. Energy-dispersive X-ray spectroscopy (EDS) is a technique used to collect and analyze X-rays produced from a sample; it allows for chemical analysis of a single point or area. The X-ray interaction volume spreads over a deeper and wider area within the material. Therefore, since primary electrons penetrate without interaction, it is difficult to analyze fine drifted particles, surface films, or low-concentration contaminants with EDS [9]. (Elemental analysis)

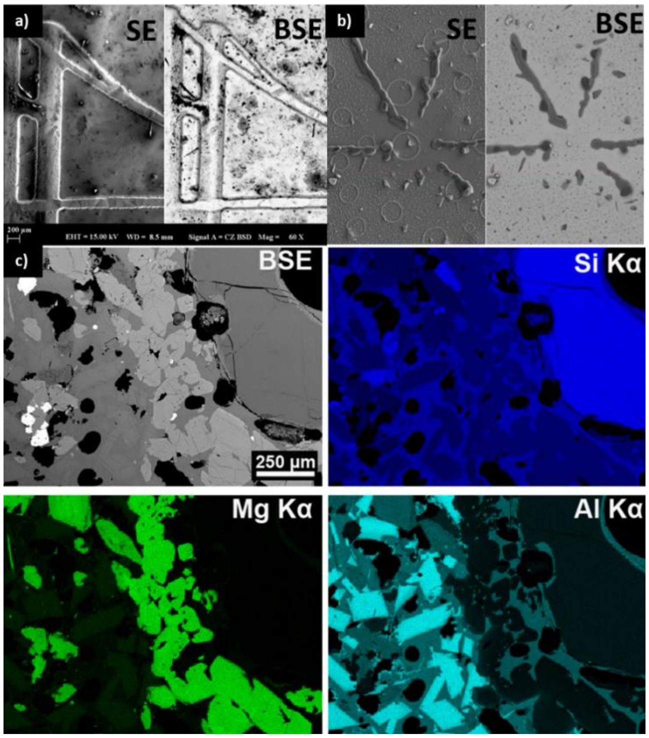


Figure 10. a) Examples of SEM images of a coin and **b)** rod-like object taken with SE and BSE, while **c)** EDS analysis images of Silicon, Magnesium and Aluminum elements with characteristic X-ray spectra of each element in the BSE image [8].

In the BSE image of the coin sample at approximately 60 magnifications shown in ***Figure 10.a***, the dark areas indicate low atomic number elements such as oxygen or carbon, while the bright areas indicate high atomic number elements such as nickel, copper or zinc. In ***Figure 10.b***, the SE of the rod-like sample and the BSE image of the same region are shown. In the SE, a concave image is shown in the topographic image.

In the BSE, there is brightness and darkness according to the atomic number difference. In ***Figure 10.c***, the EDS analysis in the BSE image is shown.

2.3. Sample Preparation and Interpretation of SEM Analysis

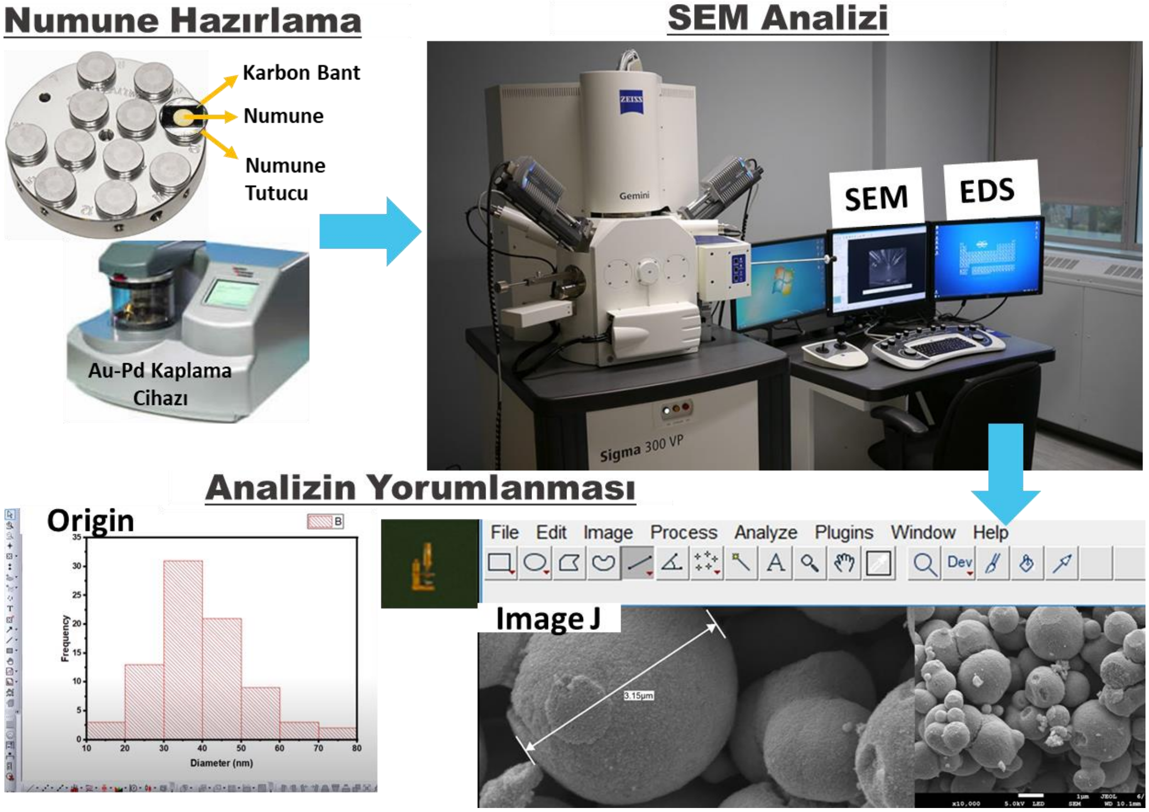


Figure 11. After the Au-Pd/C coating is applied to the insulating materials, the conductive materials are glued directly onto the carbon tapes on the sample holders and then placed in the SEM analyzer, and the particle size distribution is calculated using the Image J program from the SEM images obtained.

- ✓ All kinds of conductive samples that are not liquid or do not have liquid properties can be directly examined with SEM [9]. Solid or powder samples with diameters between 9-15 mm and thicknesses between 0.1-30 mm can be examined.
- ✓ For non-conductive or semiconductive samples, the sample is coated with a very thin (~ 3A/sec) conductive material using one of the Au-Pd Coating or C coating methods and is made ready for imaging [9].
- ✓ Hard biological samples (bone, teeth, wood, etc.) can be coated with the methods mentioned above and made ready for imaging [9].
- ✓ Since soft biological samples (plant or animal parts, tissues) contain liquid, the "Critical Point Dryer" method is used in the preparation of these samples. In this way, the sample is dried without losing its structure and shape and is made ready for imaging [9].

Au-Pd/C Coating

It enables the surfaces of semiconductor or non-conductor samples to be coated with a thin layer of Au & Pd/C to conduct imaging studies more accurately. Typical coating thickness is 20 – 30 nm. The coating process increases the conductivity of the sample, minimizes the sample charge that causes the deflection of the beam falling on the sample or distortion of the image, and increases the mechanical stability of the sample with heat transfer. It also increases the emission of primary and secondary electrons, helping detectors to collect more imaging data [9].

Sample Preparation:

a) To separate the powder sample to be examined into its particles, it is mixed in a beaker containing ethanol in an ultrasonic sonicator and the ethanol is evaporated.
b) Using double-sided carbon conductive tape (12 mm), carefully stick it onto the standard aluminum pin stud (12.7 mm x 8 mm) and lift the front surface of the tape.
c) Then, dry powder samples are poured onto the carbon tape and excess powder that does not adhere is cleaned from the pin.
d) The sample holder is placed on the sample table in the SEM device.
e) The images taken from the SEM device shown in ***Figure 11*** are measured with the help of the Image J program, taking particle sizes as micronbar reference, and the particle size distribution graph is drawn with the help of the Origin program.

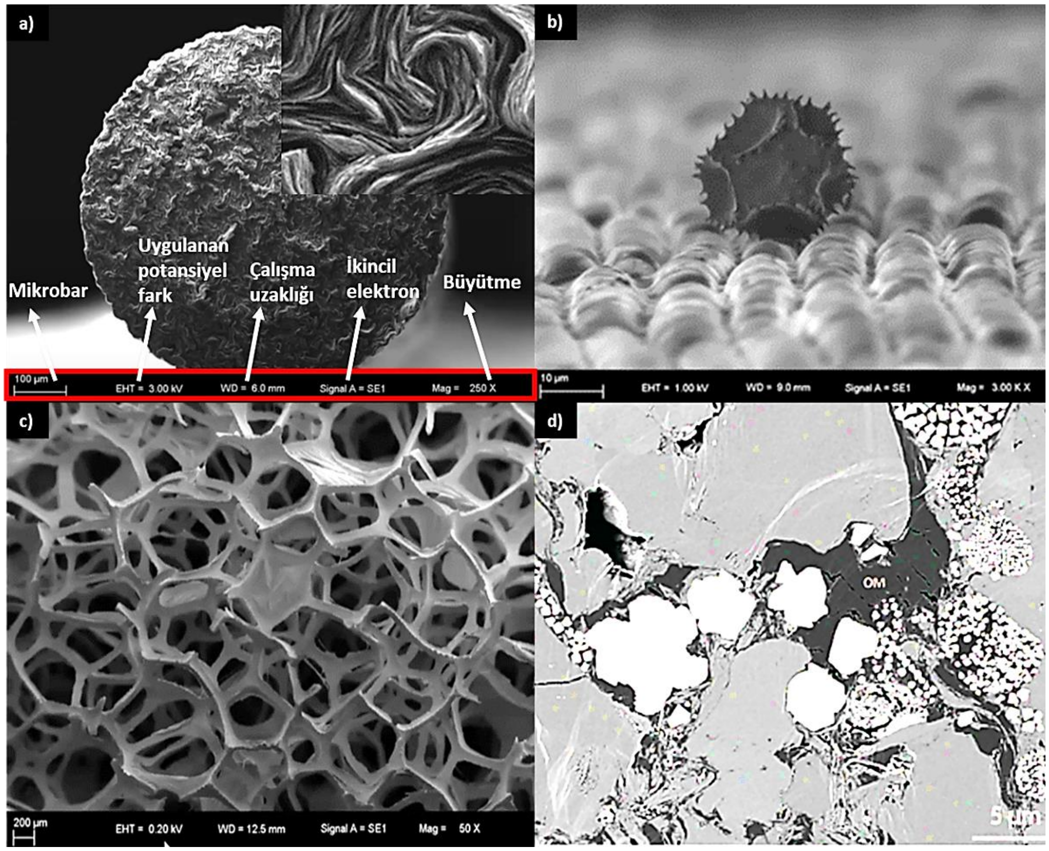


Figure 12. Sample SEM images **a)** Close-up and distant view of the pencil tip, **b)** SEM image of pollen, **c)** SEM image of a kitchen sponge, and **d)** BSE-SEM image of the milled surface showing pyrite framboids and crystals of different sizes (white), porosity (black), and phases (greys) [8].

Figure 12.a shows the SEM image of the pen tip taken with SE at different magnifications. The information at the bottom of the image is very important for interpretation. Each SEM image should include one micron bar, the applied potential difference, the working distance, the type of electron used, and the amount of magnification. The voltage applied in SEM can vary depending on the type of material. For example, since a biological sample is shown in ***Figure 12.b***, the voltage is kept low. When the energy of the electron is reduced, their resolution also decreases and their wavelengths increase. Since the magnification ratio can

vary depending on the brand of the device, the most reliable interpretation can be made according to the micron bar. SEM images taken from the same sample can also differ depending on the voltage and applied current (***Figure 13***). Additionally, the working distance and objective aperture also affect SEM images [10].

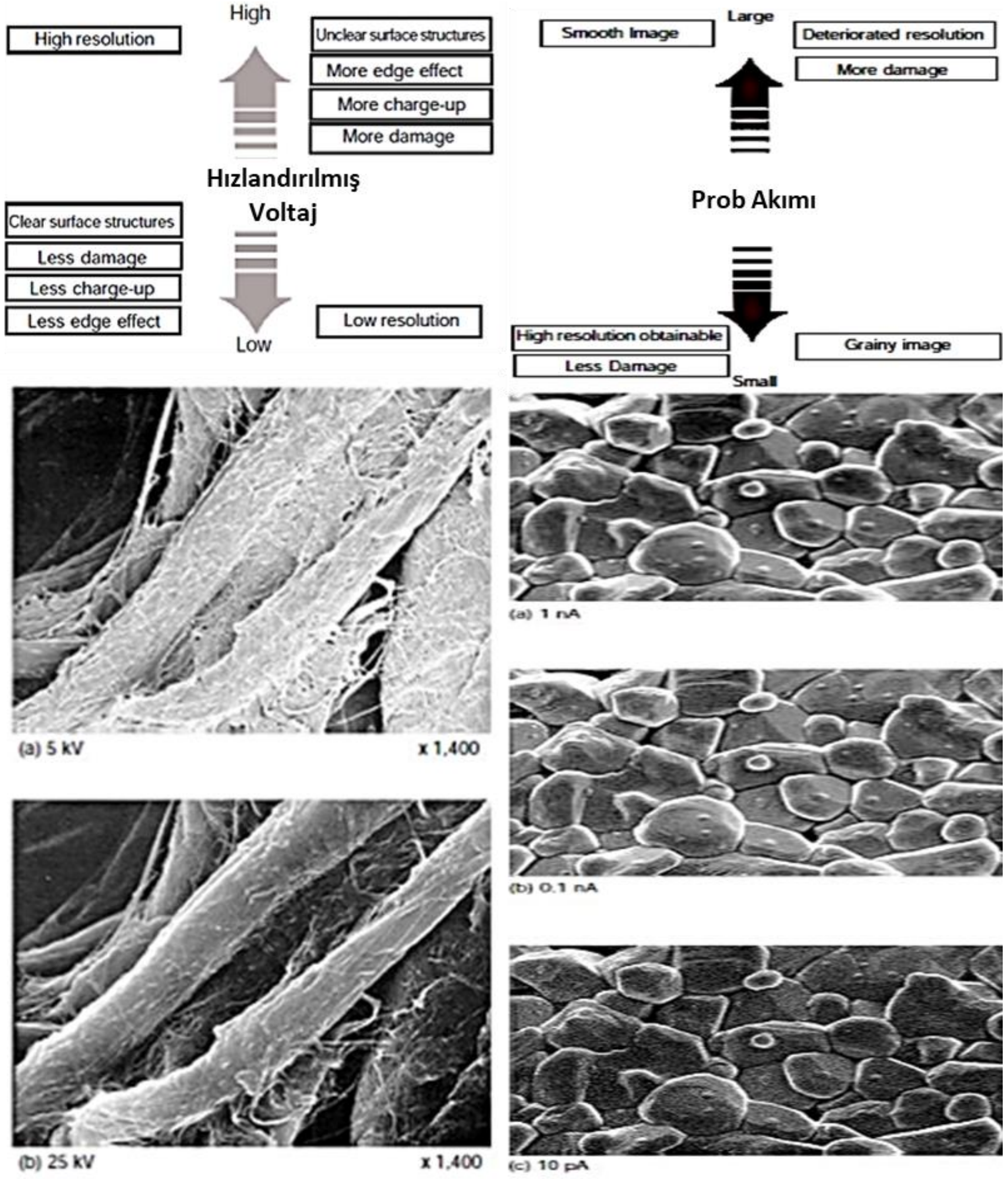


Figure 13. Effect of accelerated voltage and probe current on SEM images taken from the same material [10].

3. Results and Discussion

With this experiment, firstly, after gaining theoretical information about XRD analysis and SEM analysis, sample preparation studies will be carried out. After learning the importance of sample preparation and the problems that may be encountered, the XRD graph drawing of powder samples and the SEM analysis of powder and fiber samples will be interpreted.

- Graphic drawing from the txt format data of LiF compound with the Origin program and examination of the planes on the JCPDS card taken from the HighScore Plus application,

- SEM images of ZnO nanoparticles in different morphologies and particle size distribution graphs from the SEM image of PVA fibers will be drawn with Image J and Origin programs.

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

- ✓ Information about Origin, Image J and HighScore Plus programs
- ✓ XRD graph drawing from the txt. format file
- ✓ Commenting on the phases, crystallinity and planes in the drawn XRD graph
- ✓ Being able to distinguish the types of SEM images
- ✓ Commenting on the morphology and particle size of SEM images

4. References

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Experiment 2: Microstructural Examination of Materials

Res. Assist. Jülide Hazal ÖZDEMİR

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) 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).

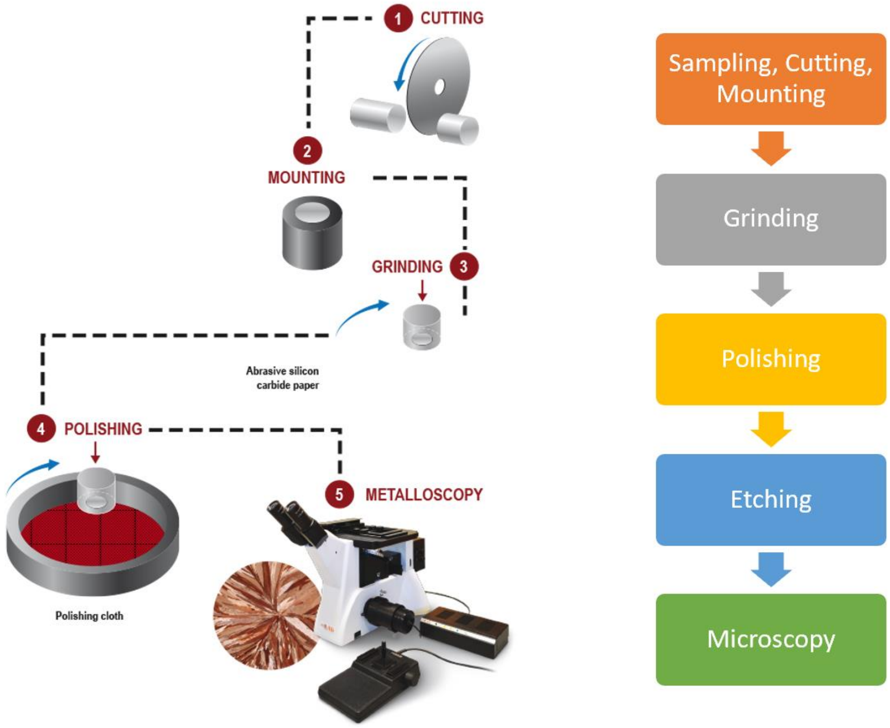

Figure 1. Metallographic process steps applied to samples

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.

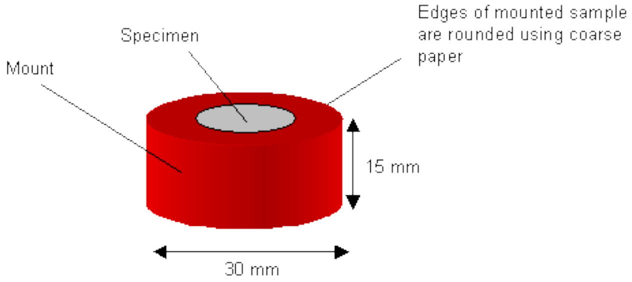

Figure 2. The representation of a sample mounted using the hot mounting process

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 ($2.54\ cm^2$) 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).

2. 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

3. 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.

4. 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

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Experiment 3: Analysis of the Rheological and Physical Properties of Casting Ceramic Slurry

Res. Asst. Mustafa BORUCU

1. Aim of the Experiment

The purpose of this experiment is to determine the rheological and physical properties of ceramic slurry and to examine the physical changes in samples kept in a plaster mold for specific periods. Within the scope of the experiment, the density of the slurry will be calculated using the pycnometer and liquid weight method, and viscosity changes will be compared based on variations in the amount of deflocculant. Additionally, parameters such as wet weight, dry weight percentage, wall thickness, and diameter of samples kept in the plaster mold for 3, 6, and 9 minutes will be calculated and analyzed. This will allow for a detailed evaluation of the casting suitability, rheological stability, and drying behavior of the ceramic slurry.

1.1. Preparation and Rheology of Ceramic Slurry

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

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

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

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

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

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

1.2 Flocculation, Deflocculation, Deflocculant

The particles of a clay material suspended in water exhibit behavior in two completely different mechanisms. This is caused by the electrostatic charges on the particle surfaces, which induce both attraction and repulsion. Regularly, in an acidic environment, the particles attract each other, a phenomenon known as "flocculation." In an alkaline environment, the particles repel each other, which is referred to as "deflocculation."

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

Defloculants

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

- There are various mechanisms through which deflocculants operate in suspension. These include:
- Raising the pH to basic values through the addition of a base or hydrolysis.
- Replacing flocculant cations with alkali cations in double-layer clays.
- Adsorbing anions under an electric field to achieve a negative charge on the particles.
- Adding a protective colloid.

Eliminating flocculating ions through precipitation or the formation of coordination complexes.

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

A good casting slurry should have the following properties:

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

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

The molds used in casting are porous and water-absorbing plaster molds. Additionally, porous synthetic materials are also used. To ensure healthy production through casting, the plaster mold must first be suitable for the molding technique, meaning it should not stick when opened, be impact-resistant, have uniform thickness, appropriate porosity, and be sufficiently dried. Furthermore, it is crucial that the mold is clean and that the joining surfaces are smooth when preparing for casting.

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

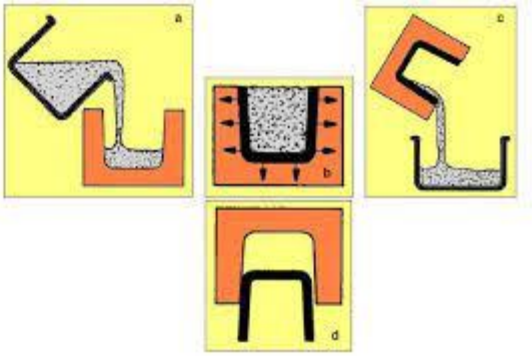

Figure 1: Schematic representation of shaping by slip casting method.

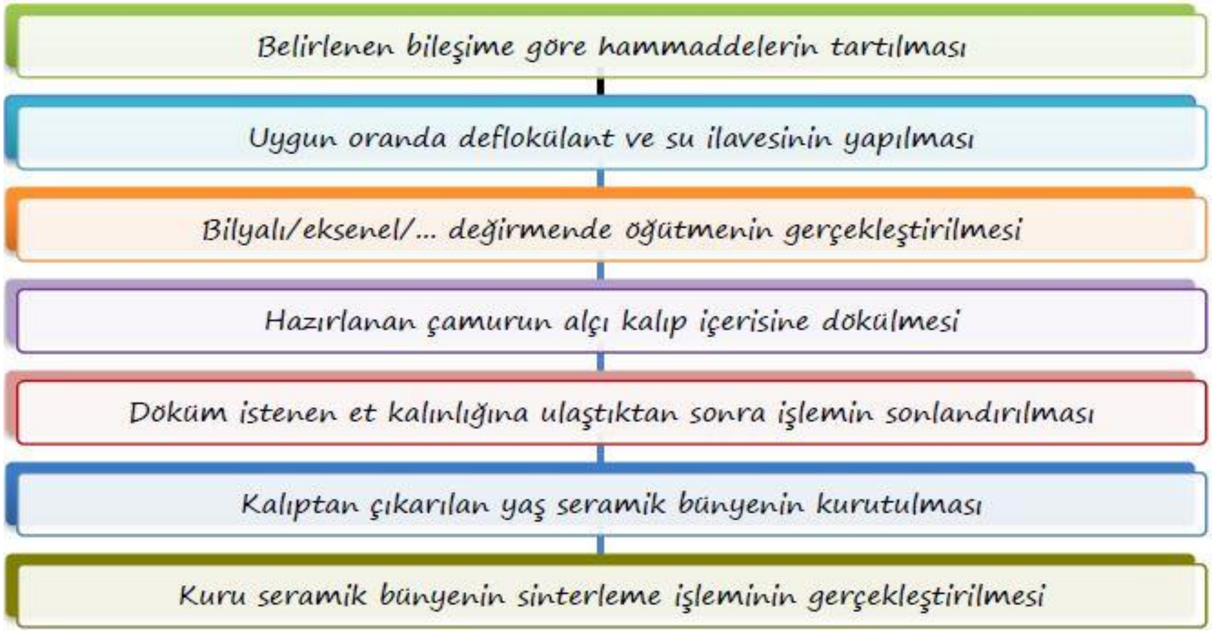

Figure 2. General flow diagram for the production of ceramic products by casting method.

2. Experiment

2.1. Required Tools

Viscometer, Mixer, Pycnometer, Scale, Plaster Mold, Caliper

2.2. Application of the Experiment

- Density Measurement of Ceramic Slurry:
    - The density of the prepared ceramic slurry is determined using the pycnometer and liquid weight method.
    - A specific volume of ceramic slurry is placed in the pycnometer, weighed, and its density is calculated using the density formula.
- Viscosity Measurement of Ceramic Slurry:
    - The viscosity of the slurry is determined using an analog viscometer.

  - First, a predetermined amount of ceramic slurry is stirred at 700 rpm, and its viscosity value is recorded.
  - Then, viscosity measurements are repeated by adding different amounts of deflocculant, and the viscosity-deflocculant relationship is analyzed.
- Plaster Mold Casting of Ceramic Slurry and Physical Analysis Based on Holding Time:
  - The plaster mold is prepared and cleaned for the experiment.
  - The ceramic slurry is poured into the mold and left to set for 3, 6, and 9 minutes.
  - The samples are measured in terms of wet weight, wall thickness, diameter, and dry weight for comparison.

To provide a more detailed process, the viscosity should be within the range of 4-6 Poise (required value for floor tiles). To determine thixotropy, the slurry is left to rest for 5 minutes after each viscosity measurement. Then, viscosity is measured again, and the difference between the two values gives the thixotropy. Once the slurry reaches its final viscosity, adding deflocculant does not affect the viscosity until an excessive amount is added. However, when too much deflocculant is added, it does not behave as expected and increases the viscosity. Therefore, reaching a stable viscosity and the subsequent increase with added deflocculant serves as proof of stability. After the rheological analyses, slip casting into plaster molds will be performed. The viscosity and liter weight of the pre-prepared ceramic slurry will be measured. These analyses will help determine the rheological properties and casting suitability of the slurry. Next, the plaster molds will be prepared for casting by cleaning their interiors, and the ceramic slurry will be poured into the molds. To ensure the experiment is efficient and provides a basis for comparison, the experimental group will be divided into subgroups of two or three, and each subgroup will leave the ceramic slurry in the plaster mold for different time durations. Since the ceramic slurry held for different durations is expected to have different wall thicknesses, each group will hold their ceramic slurry in the mold for 3, 6, and 9 minutes, then remove the excess slurry. The mold is then placed upside down on a table and left for a short time to allow easy removal of the ceramic piece. Finally, the ceramic product is carefully removed from the mold and left to air dry for 10 minutes.

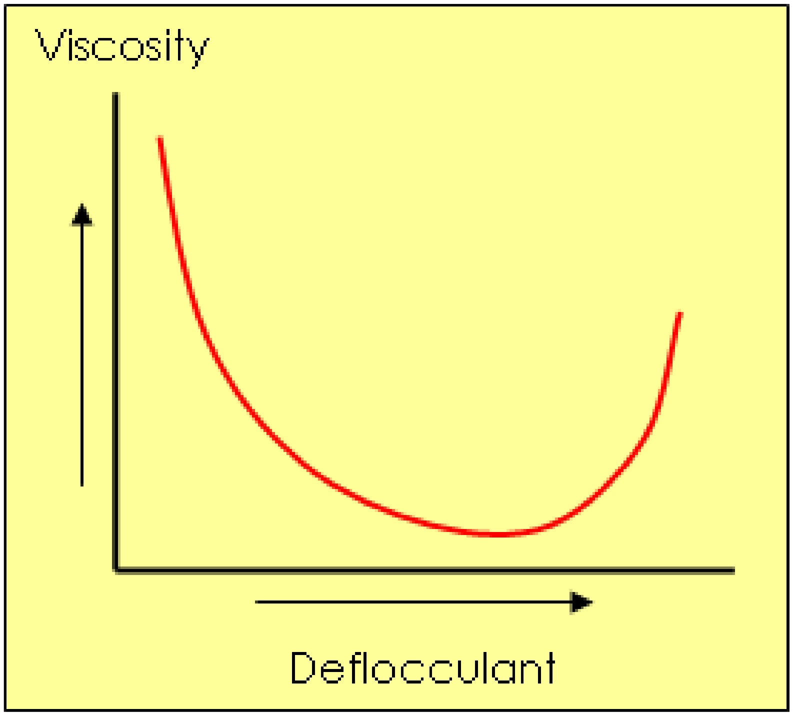

Figure 1. Viscosity - Added Deflocculant Diagram.

3. Results

As a result, this experiment demonstrated the rheological behavior of a castable ceramic slurry and analyzed the shaping process of the product using the slip casting method in a plaster mold through experimental analysis.

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Requirements for the Report:

1. Objective of the experiment
2. Brief theoretical information about the experiment
3. The application of the experiment will be described in order with original language.
4. Comments will be made on the results obtained in the experiment, and graphs will be drawn (EXCEL).

Experiment 4: Wear Test

Res. Asst. Caner KORKMAZ

1. Aim of the Experiment

Determination of wear behavior and resistance of metallic materials

2.Theoretical Information

The wear that occurs because of the friction of engineering materials in contact with each other causes great economic losses during the use of various machines and equipment. Wear is the material loss that occurs as a result of the transfer of material from one surface to another or the formation of wear parts. In DIN 50320, wear is defined as "the change that occurs by the separation of small parts from the surfaces of the material used due to mechanical reasons".

In solid surfaces in contact, material loss can occur in three ways. These are regional melting, chemical dissolution and physical separation from the surface. In practice, the damage caused by the material physically separated from the surface is included in the scope of wear.

In a wear system, main material (wearing), counter material (wearing), intermediate material, load and movement constitute the basic elements of wear. The system formed by all these elements is technically called "Tribological System".

One of the important factors in a wear system is environmental conditions. The exposure of system elements to moisture and corrosive effects accelerates wear.

Wear is generally a type of damage that is known in advance. Even if the material surfaces in contact with each other are protected by oxide films or lubricants, the deterioration of the oxide layer or lubrication under mechanical loads can cause the two surfaces to come into direct contact with each other. The friction that occurs because of this contact causes wear that limits the life and performance of the material under operating conditions. This damage can be minimized with factors such as proper lubrication, filtering, proper material selection and proper design, but it can never be prevented.

The factors affecting wear can be grouped into four main groups:

1. Factors related to the main material

1. Crystal structure of the material
2. Hardness of the material
3. Elasticity modulus
4. Deformation behavior
5. Surface roughness
6. Size of the material

2. Factors related to the opposite material and the effect of the abrasive

3. Ambient conditions

1. Temperature
2. Humidity
3. Atmosphere

4. Service conditions

1. Pressure
2. Speed

3. Sliding path

There are many wear mechanisms. It is possible to explain them under the following main headings.

2.1. Classification According to Mechanism System

Adhesive Wear: It is a result of the welding event that occurs in metal-metal wear pairs that make sliding friction with each other. The stresses on the surfaces sliding on each other reach or exceed the yield stress limit even with small loads. Thus, adhesion forces show themselves between the metals in contact. For this reason, material transfer from one part to another, cold welding and small parts breaking occur.

Adhesive wear, although the most common type of wear, generally does not accelerate damage. Adhesive wear occurs because of the breaking of the roughnesses on the surface that are welded or stuck to each other during the relative movement of a metal surface on another metal surface. If the two metals have the same hardness, wear occurs on both surfaces. Perfect lubrication between metals, reducing the load acting on the surface and increasing the hardness of the material reduce adhesive wear.

As a result, adhesive wear is proportional to the normal load acting on the surface, the sliding path and the surface hardness of the material being worn.

Abrasive Wear: Abrasive wear, also called tearing or scratching wear, is an important type of wear that causes rapid damage to the system. Abrasive wear occurs when metal surfaces, one harder and rougher than the other, slide in contact with each other.Wear caused by non-metallic materials is usually in the form of scratching.

Abrasive wear can be caused by hard particles penetrating soft metal. An example of this mechanism is the type of wear caused by dust particles entering the system from outside or combustion products formed in an engine.

The rate of abrasive wear can be reduced by reducing the load acting on the material surface. Thus, the particles are less likely to sink into the surface and leave fewer marks during burr removal. To reduce abrasive wear in terms of material.

- Using a harder alloy,
- Applying heat treatment to increase hardness,
- Covering the material surface with a hard layer is recommended. It is possible to reduce the abrasive wear rate with these measures.

Abrasive wear is the main reason for material losses in industrial devices. If the abrasive material is between two metals in a free state or if there are fixed or free grains that only wear one metal, then it is possible to group them as;

- Two-element abrasive wear,
- Three-element abrasive wear.

In metal-metal friction, wear starts as two-element abrasive or adhesive and continues as three-element abrasive. In this case, intervening dust, mineral grains, micro chips that become free because of scratching and fragmented oxide particles can form the third element (intermediate material). Since the micro chip particles that become free are generally harder than the main material (three element) abrasive wear accelerates wear. Abrasive wear, which is the most important type of wear in industrial machines, is generally encountered in the following places.

- In the blades and teeth of agricultural and construction machines such as tractors and graders,
- In ore processing and grinding facilities,

- In sieves, mills, and transport machines.

In such machines and machine parts, only abrasive wear can be mentioned, but other types of wear can also be seen together.

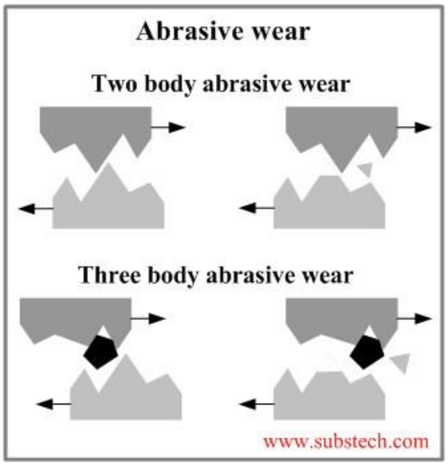


Figure 1 Upper part Two-dimensional abrasive wear, Lower part Three-dimensional abrasive wear.

2.2 Damage Classification with respect to Physical Appearance

Damage by Sliding Wear

a. Formed by granular minerals: Sliding wear caused by granular minerals is determined depending on the mineral hardness. In non-metallic hard materials, wear increases with the hardness of the mineral grains as in metal materials, but brittle fractures occur on the wear surface of the hard material. During wear, material hardness is an important factor affecting the magnitude of wear resistance.

b. Metal-metal wear: In hydrodynamic friction, which is one of the metal-metal friction types, there is generally no wear, and the material pair is not important. Because the materials do not touch each other at this moment. However, a minimum speed is required for full lubrication. In mixed lubrication, the force is provided partly by hydraulic and partly by the contact of solid objects. The properties of the materials and the sliding material pairs, and the lubricant influence these contact points. Especially in the friction of oil-free surfaces, the wear condition depends on the surface of the material pair. In addition, the processing of sliding surfaces (surface roughness, direction of processing) also greatly affects wear.

Erosion Wear

Erosion (hydro-abrasive) wear is the wear caused by fluid substances. Liquids and gases break off particles from the surface by causing an explosion or impact on the boundary surfaces of the part during flow and create a wavy surface with the effect of vortices. Thus, wear is accelerated even more. Corrosive Wear:

If the worn surfaces are also exposed to corrosive effects, this is called corrosive wear. Chemical corrosion can occur on its own or together with other types of wear. Chemical reactions that form films that adhere tightly to the surface prevent surface wear. However, if the film is brittle and loosely attached to the surface, wear is greatly accelerated. Because during the friction movement, the films crack and break off.

Rolling Wear

This type of wear is the wear that occurs in materials that move by rolling on each other. This wear is undoubtedly closely dependent on the properties of the materials. Wear during rolling can occur in a single material or in varying amounts in both materials. Whether or not the surfaces are lubricated is of great importance in rolling wear.

2.3 Classification of Wear by Special Names

Grinding Wear

Grinding wear occurs when particles under high pressure meet metal surfaces at low speeds, and particles are cut or broken off by opening many small scratches from the metal surface. Since this combination of high pressure and low speed occurs in the working conditions of heavy duty machines such as bulldozers and excavators, which are generally used in excavation work, this type of damage occurs on the cutting edge surfaces of these vehicles. The cutting and sinking tips used in excavators undergo deformation because of grinding wear and become dull.

Scraping Wear

Scraping wear is somewhat like adhesive wear, where micro-fusion occurs on the surfaces that meet. The difference between them is that adhesive wear occurs on surfaces that slide over each other, while scraping wear occurs on surfaces that do not move relative to each other. However, scraping wear occurs in systems where very low amplitude movements (vibration) occur, with the formation of micro-fusion. Scraping wear is the most common damage formation mechanism encountered in the joints of automobile shafts and bearings in systems connected with fasteners such as nuts and rivets operating in vibrating environments.

Wear tests are performed in different experimental setups (Figure 2). One of the most common test equipment in wear tests is a pin pressed on a disk (cylinder or rectangle). As seen in Figure 2, this pin is pressed onto the disk surface. There are other forms of the “pin on disk” method. However, the main idea is always the same. In such asymmetrical arrangements, the pin or block is often the sample, and the wear rate of this part is measured. The other part, the disk, is called the “outer surface”. Wear test setups have been standardized in accordance with international standards.

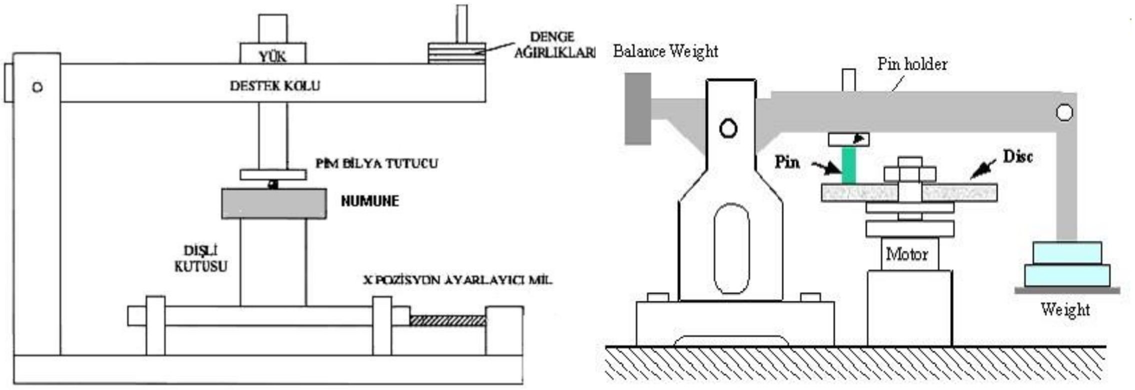
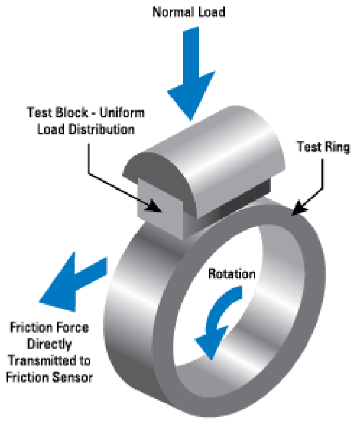


Figure 2: All the system of pin-on disk



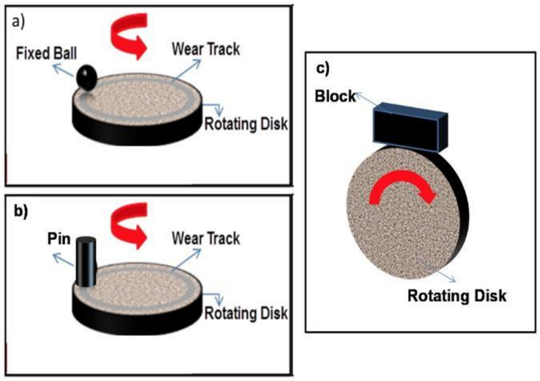


Figure 3: Wear test setups, a) Ball on disk, b) Pin on disk, c) Pin on roller

3. Devices and Materials Used

- The pin on disk wear test will be performed in our experiment.
- The pin on disk wear device
- Computer
- Wear Pin Sample
- Scale
- Optical Microscope

4. Experiment Procedure

- Sample weight will be measured before the experiment.
- The sample will be placed in the device.
- The pin will be checked and placed in the device.
- The pin will be slowly dropped on the sample.
- The load to be applied, the rotation speed and the path to be taken will be determined and adjusted on the device.
- The test will start
- After the test, the sample weight will be measured, and the weight loss will be calculated.
- The wear mark will be examined under an optical microscope and explained in detail.

5. Questions and Requests in the Report

- Explain the test procedure in detail.
- Draw the wear coefficient - time curve.
- Explain the type of wear
- Explain the factors affecting the tribological system

6. References

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Experiment 5: Corrosion and Prevention

Res. Asst. Berk ÇOBAN

PART 1: CORROSION OF LOW-ALLOY STEEL AND STAINLESS STEEL IN ACIDIC SOLUTION

1. Aim of The Experiment

Investigation of electrochemical behavior of steels in acidic solution and calculation of corrosion rate by two different methods.

2. Theoretical Information

Corrosion is the occurrence of undesirable changes in the physical, chemical and mechanical properties of materials in general, metals and alloys in particular, because of the environment they are in. Metals are found in their most stable form as ore in the earth's crust. They are normally extracted from their ores via certain processes that require energy. Metals are unstable in their pure metallic state because they are believed to be in the excited state. As a result, when metals are exposed to the environment in various forms, the exposed metal surface begins to degrade. Corroded metal is more thermodynamically stable than pure metal, but it loses important qualities, including malleability, ductility, hardness, conductivity etc. Electrochemical corrosion, a type of corrosion, is the degradation of metals in aqueous environments. There is no need for external energy in electrochemical corrosion that occurs spontaneously with the formation of electrochemical cells in the metal-media system.

The desire of a metal to become an ion determines its activity and shows the tendency of the metal to undergo corrosion. EMF (Electromotive force) series, which show the standard electrode potentials of metals, are thermodynamic series obtained by calculation and give an idea about the activity of metals. In addition, the galvanic series created by experiments in sea water can be more useful in comparing the activities of different metals and alloys.

When different metals and alloys meet each other in the same environment, the more active one is corroded by acting as an anode, while the other metal is protected from corrosion by acting as a cathode. On the other hand, two different metals do not necessarily have to be present in the environment for electrochemical corrosion to occur. Corrosion can also occur when a metal is alone in the electrolyte. Due to the different regions in the structure or surface of the metal, some parts of the metal can act as anodes and some parts as cathodes.

For electrochemical corrosion to occur, five elements must be present in the system. These are; anode, cathode, interface, electronic conductor and electrolytic conductor. During electrochemical corrosion, while oxidation reaction is observed at the anode, different reduction reactions occur at the cathode depending on the pH of the environment and the amount of dissolved oxygen in the electrolyte. The reactions taking place at the metal-electrolyte interface are given below:

On anode:

$Me^0 \rightarrow Me^{+2} + 2e^-$

On cathode:

$2H^+ + 2e^- \rightarrow H_2$ in acidic environment

$O_2 + 4H^+ + 4e^- \rightarrow 2H_2O$ in acidic environment (with air)

$O_2 + 2H_2O + 4e^- \rightarrow 4OH^-$ in alkaline or neutral environment

$Me^{+2} + 2e^- \rightarrow Me^0$ in any environment

Corrosion occurs at the anode sites. On the other hand, cathodic reactions take place at the cathode, and degradation does not occur in these regions of the metal. The corrosion rate is practically defined as the mass of metal that moves away from a unit surface area per unit time. Various methods are available for the measurement of corrosion rate. Of these, the weight loss method and the Tafel extrapolation and linear polarization methods, which are performed by drawing polarization curves, are the preferred methods. In addition, the fact that the cathodic reactions in electrochemical corrosion events are hydrogen or oxygen reduction allows, in some cases, to determine the rate of corrosion by determining the amount of gas released during the test. In determining the corrosion rate, the method that will give the most accurate result should be selected.

The corrosion rate can be expressed in various units. The most commonly used ones are mg/dm^2.day (mdd), mm/year, ipy (inch per year) and mpy (mile per year). These units are interchangeable (eg: 1 ipy = 1000 mpy = 25.4 mm/year).

When the weight loss in the test sample is determined, it is possible to calculate the corrosion rate with the help of the following formula.

$H = K \,.\, \Delta G / A \,.\, d \,.\, t$

H: corrosion rate **K**: constant for the desired corrosion rate unit (3.45×10^6 for 'mpy')

G: weight loss (g) **A**: surface area exposed to the solution (cm^2)

d: metal density (g/cm^3) **t**: duration of the experiment (s)

The units of the variables in this formula and the value of the constant number 'K' vary according to the corrosion rate unit to be calculated. In this experiment, the corrosion rate will be calculated in 'mpy'.

3. Experimental Procedures

Equipment and Materials: Steel, beaker, glass funnel, 2 M HNO_3 and HCl solutions, burette, pipette pump, drying oven, scales, polishing papers.

3.1 Experimental

Steel samples are cut in a certain size from the steel plates and made a hole to hang. And then the surface and edges of the sample are polished using water proof abrasive papers having various grit numbers following by cleaned from the oil and dirt on the surface in the ultrasonic bath. The sample, whose dimensions and weight are measured, is fixed to the bottom of the funnel using the line rope. The funnel is placed through a burette and is put it upside down in different beakers filled with the 2M HNO_3 and HCl solution, as shown in Figure 1. And the the solution is drawn into the burette at a certain level. The drop in the solution level is recorded every 3 minutes. When the solution level in the burette reaches zero, the experiment is terminated and the time is recorded. At the end of the experiment, the sample is removed from the solution, washed, dried and weighed again.

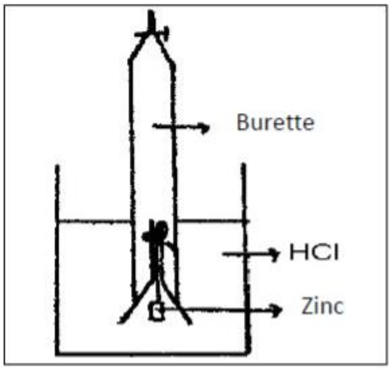

Figure 1. Experimental set-up

3.2 Requirements

1. Write cathodic and anodic reactions.
2. Calculate the total corrosion rate (mpy) using the method of the calculating the mass loss.
3. Calculate the total corrosion rate (mpy) using the method of the determining the amount of the evolved gas during the test. Plot the change in corrosion rate depending on the time, using the dissolved amount of steel (mg) -duration (min) axes and examine this change and find the corrosion rate. Use the recorded your gas volume readings in the burette during experiments.
4. Compare the corrosion rates calculated using both the methods of the calculating the mass loss of the samples and the determining the amount of the evolved gas during the test. Is the corrosion rate determined by the weight loss the same as the rate of corrosion determined by the loss of hydrogen gas? If not, indicate the reasons.

PART 2: CORROSION OF LEAD IN ACIDIC SOLUTION

1. Aim

Measuring the corrosion rate of lead in acidic solution by Tafel Extrapolation Method

2. Theoretical Information

In a corroded electrode, a cathodic reaction takes place simultaneously with the anodic reaction of the metal, depending on the environment. Thus, a mixed potential called 'corrosion potential (Ecorr)' is formed on the electrode surface as a result of the balance of two different reactions, one anodic and the other cathodic. Generally, Tafel kinetics are an accurate explanation of corrosion kinetics for conditions under which mass transport limitations are not considered, because corrosion conditions usually are removed from the reversible potentials for all the reactions

If an external current is applied to the electrode, the electrode potential takes on a value different from that at which no current is flowing. The change in electrode potential when current is applied is called 'polarization'. Experimentally obtained polarization curves can be used to determine the velocity of the metal in the electrolyte in question. For this purpose, two electrochemical methods are applied; Tafel extrapolation method and linear polarization method.

2.1 Tafel extrapolation method

Tafel extrapolation is one of the polarization methods widely utilized to measure corrosion rates, a faster experimental technique compared with the classical weight-loss estimation [36]. It is known that the corrosion rates obtained using Tafel extrapolation of polarization curves are not usually the same as those measured by the weight loss.

Polarization (or current-potential) curves are curves plotted on a semi-logarithmic scale. After the applied external current reaches a certain value, there is a significant break in these curves and after this point the logarithm of the applied external current (E - log i) shows a linear change. This region where the logarithm of the current changes linearly is called the 'Tafel Region'.

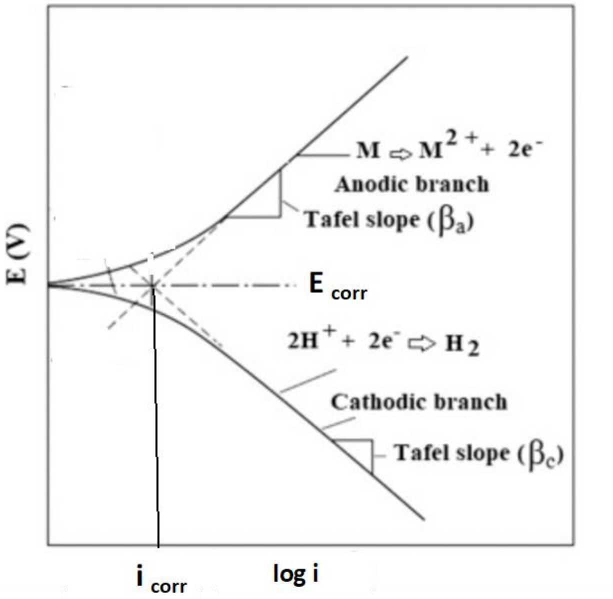

Figure 2. Tafel region of the polarization curve

If the lines in the Tafel region are extrapolated to the corrosion potential, the cutoff gives the 'corrosion current density (icorr)' value. By substituting the corrosion current density value in the formula below, the corrosion rate is calculated in mm/year.

$$Corrosion\ Rate = K\ x\ \frac{(i_{corr}\ x\ EW)}{d}$$

K= constant number, 3.27×10^{-3} mm.g/µA.cm.year,

i_{corr}= current density (A/cm^2),

EW = equivalent weight of the sample,

d= density of the sample, g/cm^3

3. Experimental Procedures

Equipment and Materials: Potentiostat, lead sample, 1 M H_2SO_4 solution, 5-necked cell, lead counter electrodes, saturated calomel reference electrode, crocodile cable, dryer machine, glue gun, silicone.

3.1. Experimental

Lead sample is washed and dried to remove dust and dirt on its surface. It is bonded with crocodile cable and covered with silicone except an active surface area of 1 cm^2. 1 M H_2SO_4 solution is filled into the 5-necked cell. Saturated calomel electrode as reference electrode, lead electrodes as counter electrode and lead sample as working electrode are placed in the cell. With the help of a potentiostat, first, the open circuit potential (OCP) value of the system is measured. Then, the sample is scanned within the determined potential range, including the OCP value, to obtain a semi-logarithmic current-potential curve. The i_{cor} value obtained from this curve is substituted in the formula given above and the corrosion rate is calculated in mm/year.

4. Requirements

- Write the anodic and cathodic reactions that took place in the experiment for the lead sample in H_2SO_4 solution.
- Calculate the corrosion rate of the sample in mm/year with the help of the i_{corr} value obtained from the Tafel curve.
- Convert the corrosion rate calculated in mm/year for lead into mpy and compare it with the corrosion rate calculated in mpy for the steel sample.
- Select 2 methods of protection from corrosion and explain these methods.

Bringing your calculators with you is recommended.

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Experiment 6: Hydrometallurgy

Res. Asst. Ozan AYDIN

1. Purpose of the Experiment

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

2. Theoretical Information

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

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

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

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

Copper ores usually are processed by pyrometallurgical routes for a long time. However, pyrometallurgical techniques have recently replaced hydrometallurgical ones. Sulphur dioxide (SO_2) emission, requirement of ore beneficiation, loss of other metals and high costs are disadvantages of pyrometallurgical techniques. In hydrometallurgical processes, the main principal is dissolving valuable elements by using selective reactants.

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

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

$$CuFeS_2(k) + 2H_2SO_4(s) = CuSO_4(s) + FeSO_4(s) + 2H_2S \qquad (1)$$

$$CuFeS_2(k) + 2Fe_2(SO_4)_3(s) = CuSO_4(k) + 5FeSO_4(k) + 2S°(k) \qquad (2)$$

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

2.1 Leaching Applications

In-situ Leaching: The leach solution is fed onto the ore which is crushed in situ using explosives. The advantages of this application are low investment and operating costs (e.g. extraction, transport and does not require solid-liquid separation). Despite these practical advantages, in-situ leaching requires favourable geological conditions, and the leach products are not selective, resulting in the consumption of large amounts of solvent. Reactions are slow and very low proportions of metallic values are dissolved,

Heap Leaching: The leaching solution is fed to the ore, which is broken and piled on a solid ground, and the solutions filtered through the heap are collected in ponds. In heap leaching, as in in-situ leaching, investment and operating costs are low. Solid-liquid separation is not necessary. In addition, a large proportion of useful minerals can be dissolved from metal-laden leach solutions due to their re-feeding into the circuit. On the other hand, if heap leaching is applied to metallurgical residues, the residues can only be utilised by subjecting them to preliminary ore beneficiation processes.

Tank Leaching: The raw material is subjected to leaching in the tank after preliminary preparations such as crushing and grinding. Preliminary preparations and raw material costs are high. In addition, investment and operating costs of leaching tanks are high compared to other leaching applications. However, the biggest advantage of tank leaching is that the reaction conditions can be kept under control.

Copper production from oxide copper ores is carried out by sulphuric acid leaching for acidic oxide copper ores and ammonium hydroxide + ammonium carbonate leaching for basic oxide copper ores. Copper is recovered from oxide-sulphide copper ores by flotation+leaching process. For example, malachite dissolves in sulphuric acid according to the following reaction:

$$Cu_2(OH)_2CO_3 + 2H_2SO_4 \longrightarrow CuSO_4 + CO_2 + H_2O \qquad (3)$$

In acid leaching applications, the copper taken into solution is evaluated by one of the cementation-electrolysis or solvent extraction-electrolysis methods.

Dissolution Mechanisms

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

Factors Affecting Leaching

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

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

Cementation

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

Reactions occurring in copper cementation process:

Formation of Zn^{2+} ions by anodic half-cell reaction: $Zn^{\circ} \rightarrow Zn^{2+} + 2e-$ (4)

Formation of metallic Cu by cathodic half-cell reaction: $Cu^{2+} + 2e- \rightarrow Cu^{\circ}$ (5)

Total cell reaction: $Cu^{2+} + Zn^{\circ} \rightarrow Cu^{\circ} + Zn^{2+}$ (6)

The pH value of the solution applied to the cementation is extremely important. Hydrogen ion concentration in acidic media is high: $2H^{+} + 2e^{-} \rightarrow H_2$ (7) the secondary reaction evolving according to the reaction is activated. Thus, there is an unnecessary increase of cementator consumption. When cementation is being done, the solutions are either not mixed or very slowly mixed that are not allowed to aeration. In case of excessive oxygen dissolves in the solution because of severe mixing metallic Zn and Cu move into an ionic state because of oxygen reduction.

Anodic reaction: $Zn^{\circ} \rightarrow Zn^{2+} + 2e-$ (8)

Cathodic reaction: $Cu^{\circ} \rightarrow Cu^{2+} + 2e-$ (9)

$1/2\ O^{2} + 2H^{+} + 2e- \rightarrow H_2O$ (10)

Cementation Chemistry: If an iron component is immersed in an aqueous solution containing copper ions metallic copper tends to precipitate, while iron tends to go through the solution. Electrochemical potential is the driving force of this reaction.

$$Fe^{\circ} + Cu^{2+} = Cu^{0} + Fe^{2+} \quad (11)$$

$$E = E_{Cu^{2+}/Cu} - E_{Fe^{2+}/Fe} \quad (12)$$

$$E_{Cu^{2+}/Cu^{0}} = E^{0}{}_{Cu^{2+}/Cu^{0}} + \frac{RT}{2F} \ln \frac{1}{a_{Cu^{2+}}} \quad (13)$$

$$E_{Fe^{2+}/Fe^{0}} = E^{0}{}_{Fe^{2+}/Fe} - \frac{RT}{2F} \ln \frac{1}{a_{Fe^{2+}}} \quad (14)$$

$$E = E^{0}{}_{Cu^{2+}/Cu^{0}} - E^{0}{}_{Fe^{2+}/Fe} - \frac{RT}{2F} \ln \frac{a_{Fe^{2+}}}{a_{Cu^{2+}}} \quad (15)$$

T: temperature (K), **F**: Faraday constant (96500 coulomb/g), **R:** Gas constant (8,31 Joule).

The standard reduction potential of copper is +0.34 and -0.44 volts for copper and iron, respectively. Substituting these values in (15) gives the following equation

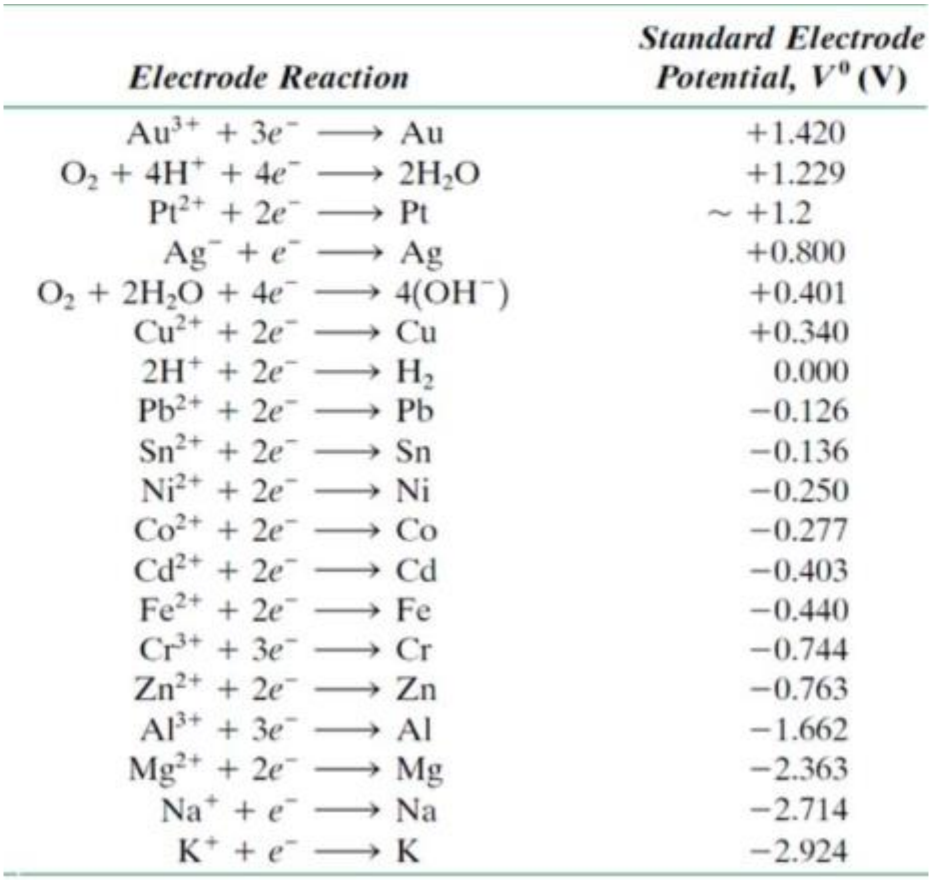
$$E = 0,78 - \frac{RT}{2F} \ln \frac{a_{Fe^{2+}}}{a_{Cu^{2+}}} \quad (16)$$

The reaction (10) will continue until E approaches zero. These conditions are the conditions where the ion concentrations reach equilibrium. The ionic equilibrium ratio a_{Fe2+} / a_{Cu2+} is 10^{25} at 25°C. This ratio, which is very large, shows that the reaction 11 will be completely to the right. According to equation 11, 1 mole of iron (55,85 g) cement 1 mole of copper (63,54 g), this value is determined as kg iron per 1 kg Cu. In industrial applications, 1.5-2.5 kg iron is required due to the following side reactions.

$Fe^\circ + 2Fe^{3+} = 3Fe^{2+}$ (17)

$Fe^\circ + 2H^+ = Fe^{2+} + H_2O$ (18)

Oxygen in the air oxidises the +2 valence iron and causes the metallic iron to be consumed according to reaction (14). The selection of metals in the carburising process is made according to the EMF series (Figure 1).



Electrode Reaction	Standard Electrode Potential, V^0 (V)
$Au^{3+} + 3e^- \longrightarrow Au$	+1.420
$O_2 + 4H^+ + 4e^- \longrightarrow 2H_2O$	+1.229
$Pt^{2+} + 2e^- \longrightarrow Pt$	~ +1.2
$Ag^- + e^- \longrightarrow Ag$	+0.800
$O_2 + 2H_2O + 4e^- \longrightarrow 4(OH^-)$	+0.401
$Cu^{2+} + 2e^- \longrightarrow Cu$	+0.340
$2H^+ + 2e^- \longrightarrow H_2$	0.000
$Pb^{2+} + 2e^- \longrightarrow Pb$	−0.126
$Sn^{2+} + 2e^- \longrightarrow Sn$	−0.136
$Ni^{2+} + 2e^- \longrightarrow Ni$	−0.250
$Co^{2+} + 2e^- \longrightarrow Co$	−0.277
$Cd^{2+} + 2e^- \longrightarrow Cd$	−0.403
$Fe^{2+} + 2e^- \longrightarrow Fe$	−0.440
$Cr^{3+} + 3e^- \longrightarrow Cr$	−0.744
$Zn^{2+} + 2e^- \longrightarrow Zn$	−0.763
$Al^{3+} + 3e^- \longrightarrow Al$	−1.662
$Mg^{2+} + 2e^- \longrightarrow Mg$	−2.363
$Na^+ + e^- \longrightarrow Na$	−2.714
$K^+ + e^- \longrightarrow K$	−2.924

Figure 1: EMF series.

3. Equipment and Materials

- Chalcopyrite ore
- Copper sulfate
- Sulfuric acid

- Distilled water
- Precise scale
- Magnetic stirrer
- Pipette, washing bottle, beaker, funnel, filter paper

4. Experimental Procedure

1 g/50 ml chalcopyrite concentrate is leached at 50°C. The amount of Cu in solution is analysed by Atomic Absorption Spectrometry. The reason for the high or low yield value is discussed. For the semantisation process, 5 g/200 ml of copper sulphate solution is prepared, pH=3 and pH=5 is adjusted and cementation is carried out with Zn and Fe cementators at 1.5 times the stoichiometry. Copper recovery efficiency is calculated for each experimental group. All results obtained are interpreted.

Calculations for the Leaching Process:

% Leaching efficiency = [Dissolved Cu (mg/L) / Amount of Cu in the sample (mg/L)] x100

Calculations for Cementation Process:

% Cementation efficiency = [Amount of precipitated Cu (mg/L) / Amount of Cu in the solution(mg/L)] x 100

Information to be Included in the Experiment Report

- Calculate precipitated copper efficiency using the results of Cu analysis after precipitation and filtration of the precipitated parts obtained in the test results with $CuSO_4$ solution containing 5 g / 200 ml Cu with Fe and Zn powder.
- Explain the relationship between precipitation efficiencies and pH values.
- Which cementator provided higher efficiency? (Zn or Fe) Explain in detail.
- Calculate the leaching efficiency of chalcopyrite concentrate. Is the efficiency value high or low? What is the reason of this situation?

! Homework: Explain the working principle of the Atomic Absorption Spectrometer

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Experiment 7: Investigation Of Optical Properties of Materials

Res. Asst. Utku EKİM

1. Objective

The objective of this experiment is to:

- measure the transmittance of ultraviolet and visible radiation by a set of standards
- optical materials.
- observe the transmittance of visible and ultraviolet radiation by optical materials and see the amount of radiation blocked out by these samples
- Measuring the refractive index of an optical material

2. Theoretical Background

Spectroscopy is the study of objects based on the spectrum of color they emit or reflect. Scientists spend much time studying the interactions between matter and energy. Because there are several forms of energy and a large supply of matter, these studies have provided many measurements and much knowledge about the universe and its behavior. A study of electromagnetic energy with matter provides valuable and important information about molecular structure and properties. In fact, some of the most convincing evidence about atomic and molecular structure, and the origin and makeup of the universe have been obtained this way.

Light is electromagnetic radiant energy. Depending upon the point of view, light can be thought of as having either wave properties or particle properties. The argument as to whether light behaves as waves or particles persisted for centuries. Wave–particle duality is a theory that proposes that all matter exhibits the properties of not only particles, which have mass, but also waves, which transfer energy. A central concept of quantum mechanics, this duality addresses the inability of classical concepts like "particle" and "wave" to fully describe the behavior of quantum-scale objects. The idea of duality originated in a debate over the nature of light and matter that dates to the 17th century, when Christiaan Huygens and Isaac Newton proposed competing theories of light: light was thought either to consist of waves (Huygens) or of particles (Newton). Through the work of Max Planck, Albert Einstein, Louis de Broglie, Arthur Compton, Niels Bohr, and many others, current scientific theory holds that all particles also have a wave nature (and vice versa). According to the wave theory, electromagnetic radiation is composed of both electric and magnetic fields whose waves vibrate in mutually perpendicular planes. It is the electric field of light interacting with the electrons of matter which produces observable properties of a substance.

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

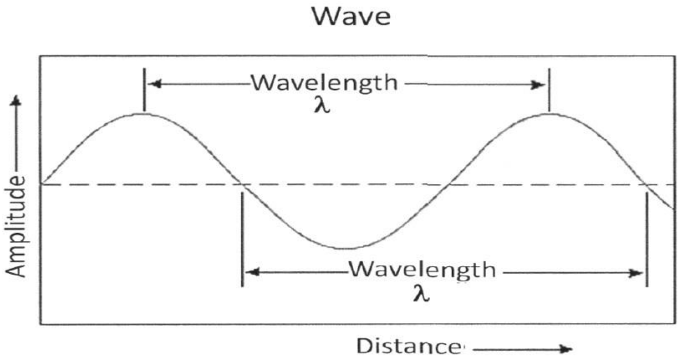

Fig. 1: The description of wavelength

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

$$\mathbf{c = \lambda \nu}$$

Another important relationship is that between these quantities and the energy of light.

$E = h\nu$ or the equivalent $E = hc / \lambda$

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

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

Relationship of Light Absorbed or Transmitted and Observed Color

Light from the sun is composed of a continuum of energy and thus, of a continuum of wavelengths and frequencies. Most of the electromagnetic radiation continuum is invisible to humans; the portion we perceive is referred to as visible light. Wavelengths of visible light extend from about 800 nm (8.0×10^{-5} cm) to about 400 nm (4.0×10^{-5} cm). Fig. 2 shows the entire wavelength range of the electromagnetic spectrum arbitrarily divided into regions called bands and the portion occupied by visible light.

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

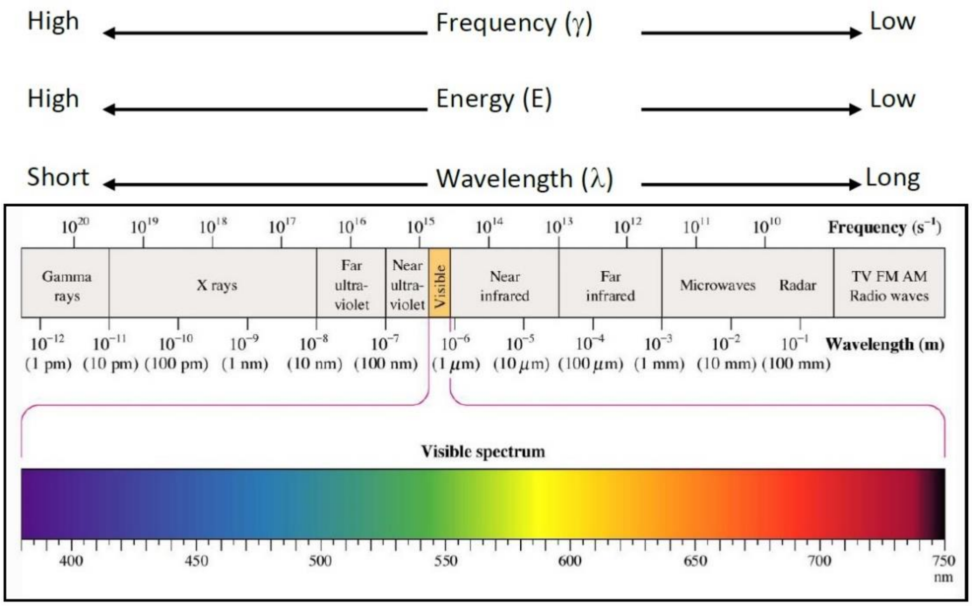

Fig. 2: Electromagnetic Spectrum

If little or none of the visible light striking our eyes is not absorbed prior to striking the eye, the color appears white. If this band of visible light is separated into narrow bands of wavelengths by a prism, we perceive the component colors. Isaac Newton divided the visible spectrum into seven color bands ranging in sequence starting with the shortest wavelengths (left to right in Fig. 2 above): violet, indigo, blue, green, yellow, orange, red.

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

The simplest cases are those where all incident light directed at a substance is either absorbed or transmitted. If a substance absorbs all wavelengths in the visible range, none of the light is reflected to our eyes and the substance appears black. If the substance absorbs none of the incident visible light, it appears white (all light reflected) or colorless (all light transmitted). Colorless substances usually absorb in the UV or IR regions of the spectrum, on either side of the visible range.

Spectroscopy

Spectroscopy is a basic analytical technique and research tool that utilizes the interaction between matter and electromagnetic energy. A spectrometer is an instrument that separates electromagnetic radiation according to wavelengths, passes these separated wavelength bands through a sample, and detects the intensity of the transmitted light. In analyzing a new sample, a scientist first determines the sample's absorbance spectrum. The absorbance spectrum is a plot of absorbance vs wavelength and shows how the absorbance of light depends upon the wavelength of the light.

The absorption spectrum is characterized by the wavelength of maximum absorption (λmax) at which the absorbance is the greatest (see Fig. 3 below. The value of λmax is important for several reasons. It is used to obtain the highest sensitivity and to minimize deviations from Beer's Law (see development below). It is characteristic of each unique compound providing information on the electronic structure of the compound.

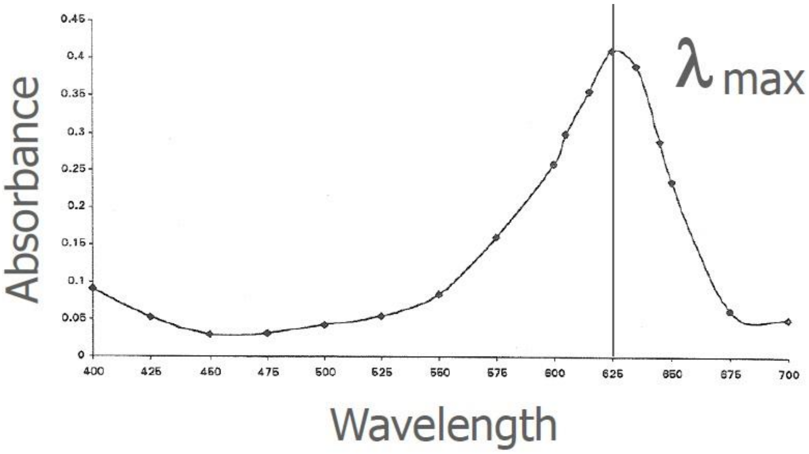

Fig. 3: The example of an absorption spectrum

The Beer-Lambert Law

The Beer-Lambert Law is a set of mathematical equations that demonstrate the connection between the properties of a material and the absorption of light. These equations are used to calculate two fundamental variables, namely transmittance (T), percent transmittance (%T), and absorbance (A).

$$T = I / I_0$$

$$\%T = I / I_0 \cdot 100$$

$$A = -\log T = -\log_{10} (I / I_0)$$

Looking at these equations, it is evident that there is a logarithmic relationship between the transmittance and absorption properties of a substance.

Refractometry

Refractometry is a branch of science that measures the change in speed of light as it passes from one medium to another, and consequently the change in the direction of light (refraction). This measurement, carried out with devices called refractometers, typically aims to determine the refractive index of liquids, gases, or solid substances. Refractometry enables qualitative analysis, purity control, and quantitative analysis.

Refractive Index and Measurement Methods

The refractive index is a property observed in transparent materials. It relates to the extent to which a substance deviates a beam of light from its original direction. This deviation occurs because the speed of light within the material differs from its speed in a vacuum. In other words, the lower the speed of light within a substance, the higher its refractive index. The refractive index is typically represented by the letter "n" and arises due to the change in the speed of light as it transitions from one medium to another, such as from air or vacuum into a different material. As one of the physical properties of a substance—alongside characteristics like solubility, melting point, boiling point, and density—the refractive index plays a crucial role in material identification. The refractive index of a medium is expressed as the ratio of the speed of light in a vacuum (c) to its speed in that medium (v):

$$\boldsymbol{n = \frac{c}{v}}$$

The refractive index of a transparent medium can be determined experimentally. For example, to measure the refractive index of water, a thin beam of light is directed at the surface of water contained in a glass vessel at an angle θ_1 to the normal. Due to the change in medium, the speed of light also changes, and the angle θ_2 that the refracted beam makes with the normal inside the water is measured (Figure 1.4). The ratio of the sinus of these angles is equal to the ratio of the speeds of light in the respective media.

$$\boldsymbol{\frac{\sin\theta 1}{\sin\theta 2} = \frac{v1}{v2}}$$

The refractive index varies depending on the material's chemical composition, structure, ambient temperature, pressure, and the wavelength of the incoming light.

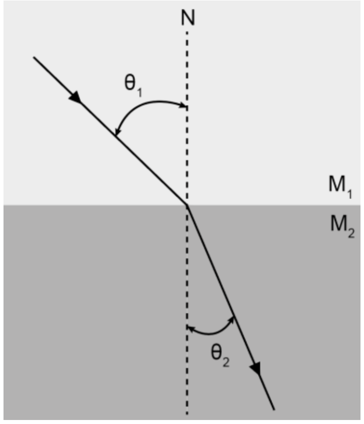

Figure 4: Light propagation in two media with different refractive indices

Abbe Refractometer

A device used to measure the refractive index is called a refractometer. Various methods are employed for refractive index measurement, and one widely used type is the *Abbe refractometer*, which operates based on the measurement of the critical angle. In this method, the sample is placed on a prism, and a light source is used to perform measurements at a specific wavelength. The measurement principle is based on directing light at various angles onto a prism in contact with the sample and observing the boundary between dark and bright regions on the prism. Since light cannot propagate within the prism at angles greater than the critical angle, the corresponding region appears dark, while areas corresponding to smaller angles appear bright. The boundary between these dark and bright regions corresponds to the critical angle. Using this critical angle, the refractive index is calculated and directly read from the instrument’s scale. The *Abbe refractometer* provides a highly precise and reliable measurement method, particularly for liquids and transparent solid materials. However, for solid samples, the surface must be optically prepared before measurement. This preparation includes grinding, polishing, and cleaning. These preparation steps are crucial for obtaining accurate and repeatable refractive index measurements. Additionally, proper calibration of the instrument and ensuring the sample has a homogeneous structure are essential for reliable results.

3. Materials And Equipment Used in the Experiment

UV-Vis spectrophotometer, Abbe refractometer, standard optical materials, alcohol, deionized water, drying paper.

4. Experimental Procedure

%Transmittance Measurement

1- The device is turned on, and the lamps are allowed to warm up.

2- Measurement parameters (wavelength range, sensitivity, lamps, etc.) are selected.

3- A baseline is taken for the specified measurement conditions before any sample is placed in the device.

4- The sample to be measured is placed in the sample holder.

5- Measurement is performed using the same measurement parameters.

6- The obtained spectrum is examined.

Refractive Index Measurement

1. Clean the sample surface before placing it in the Abbe refractometer.
2. Place the sample on the fixed prism of the refractometer and close the upper part with the eyepiece onto the sample.
3. Look through the eyepiece and adjust the measurement line so that half of the field appears bright and the other half dark.
4. Record the value read from the measurement scale.

4. Additional Equipment Required for the Experiment

- Graph paper (millimeter paper)
- Ruler

5. Post-Lab Questions

1. In the Marvel Universe, the *X-Men* character Cyclops' visor contains a **special ruby quartz (ruby crystal)** lens to control the destructive optic blasts emitted from his eyes. This lens acts as a filter, allowing certain wavelengths of light to pass through while absorbing others. It is known that the visor does not affect Cyclops' own vision. Which regions of the electromagnetic spectrum does this visor allow to pass through, and which regions does it absorb? Draw and explain the absorbance graph of this visor, including the relevant regions of the electromagnetic spectrum.

2. Using the measurements taken during the experiment, plot the absorption/transmission graphs of the given samples in Excel format, ensuring proper scaling. Analyze the absorption and transmission behavior of blue glass, sunglasses lenses, window glass, and the sample you prepared during the experiment. Your interpretation should include electromagnetic spectrum data. Compare the absorption and transmission properties of window glass with relevant literature data by finding absorption and transmission graphs from scientific sources. Then, compare these values with your own experimental results.

**** Please provide your sources at the end of your report in the references section.***

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Experiment 8: Mechanical Tests for Composite Materials

Res. Asst. Ali Rıfat ÜNAL

1. Tensile Test

1.1. Objective of the Experiment

The tensile test is carried out to determine the mechanical properties of materials and to classify them according to their mechanical behavior. The data obtained because of the tensile test are used directly in engineering calculations. For this reason, tensile testing is one of the most widely used destructive material inspection methods.

1.2. Conducting The Experiment

The tensile test is the pulling of the test specimen prepared according to the standards on a single axis, at a certain speed and at a constant temperature, until it is torn off. During the experiment, when a continuously increasing tensile force is applied to the standard specimen, the elongation of the specimen is recorded at the same time. Figure 1 shows schematic drawings of two specimens conforming to the test standard:

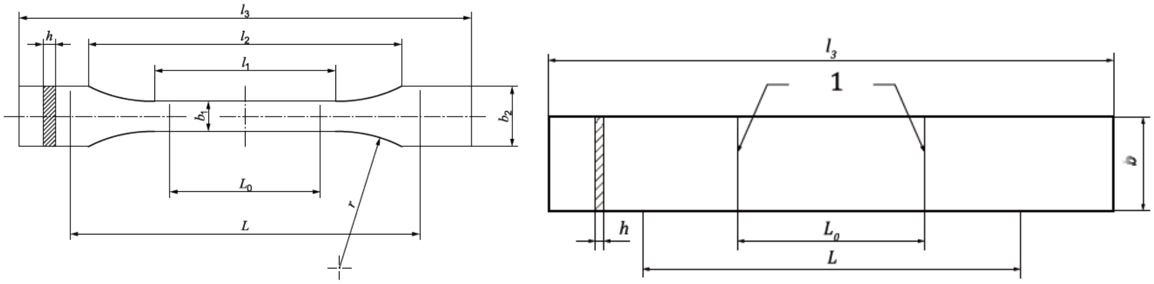

Figure 1. Schematic drawings of tensile specimens to be taken from extrusion or injection moulding (left side) and thin film or plate parts (right side), respectively, according to the BS EN ISO 179 standard

The prepared sample sample is placed between the jaws of the tensile device and compressed, and the experiment is started. Figure 2 shows a picture of a typical tensile device. During the test, the upper jaw moves upwards, and an increasing tensile load is applied to the tensile specimen continuously, and both the applied force and the elongation of the specimen are recorded until the moment of fracture.

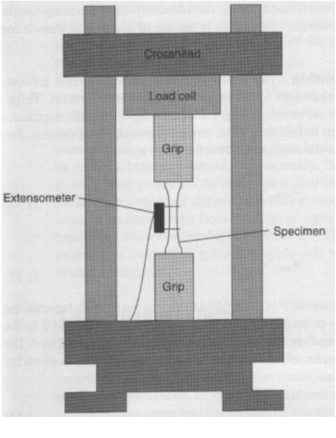

Figure 2. Schematic drawing of the application of the tensile test

As a result of this experiment, the Force (F) – Elongation (Δl) curve is obtained. However, it is necessary to give the dimensions of the sample used with this curve. Therefore, instead of this curve, the more functional stress-strain curve (σ – ε curve) is used. The stress-strain curve is called a tensile diagram.

In Figure 3, the characteristic stress-strain curves according to the BS EN ISO 527 standard are given schematically:

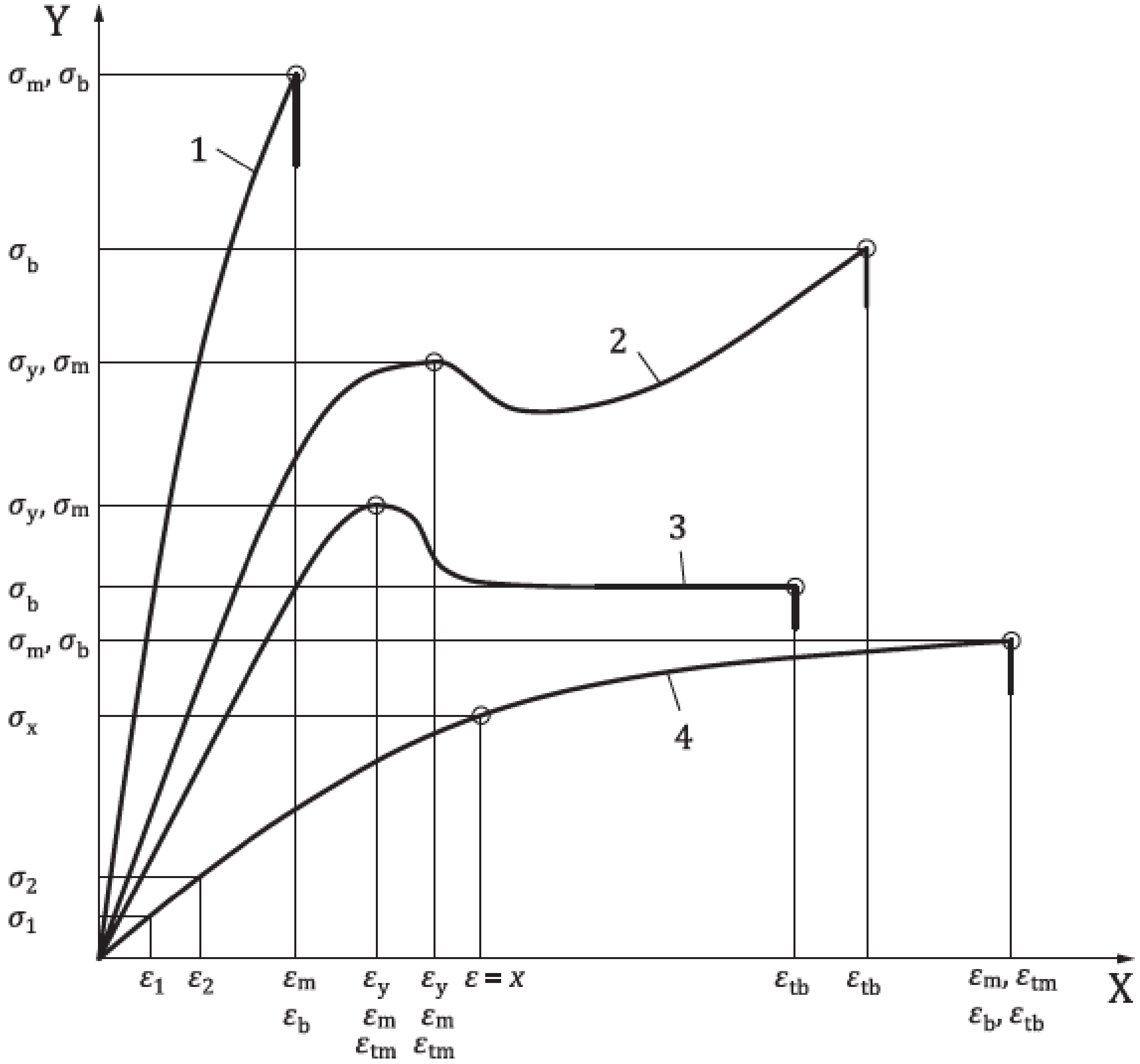

Figure 3. Characteristic stress-strain curves shown by materials with different mechanical properties

In the drawing given in Figure 3, curve 1 symbolizes brittle materials that do not show yield behavior even at low strain values, and curves 2 and 3 symbolize the increase (2) and non-increase (3) of the yield point with increasing stress after yield behavior. Curve 4 is the characteristic stress-strain curves of soft and rubbery specimens that break at high strain values (more than about 50%).

1.3. Theoretical Information

Definitions

As a result of the tensile test, the following mechanical properties of the material represented by the sample can be found:

Stress (σ): It means the force acting on the unit area. If the cross-sectional surface area (A_0)in the direction perpendicular to the tensile force (F) of the test specimen, the engineering stress (nominal stress) is calculated by the formula:

$$\sigma = \frac{F}{A_0}$$

During the experiment, with the elongation of the material, the cross-sectional area narrows according to the volume constancy rule, and in fact there is a higher stress value than the stress calculated in the part, this stress value is called the real stress and is calculated using the following equation:

$$\sigma = \frac{F}{A}$$

Engineering Strain (e): It is the first paint ratio of the length change that occurs when force is applied to the material before the force is applied. When the initial gauge size l_0 assumes the value l because of the charge P at any time in the experiment, the engineering strain (engineering elongation) in the direction of tensile is calculated using the following equation:

$$e = \frac{l - l_0}{l_0} = \frac{\Delta l}{l_0}$$

True Strain (ϵ_R) : During the experiment, the size of the measure is constantly changing. While the measure length is l at any time in the experiment, the increase in unit deformation at the end of an infinitesimal elongation, such as dl, is the true strain change:

$$\epsilon_R = \int_{l_0}^{l} \frac{dl}{l} = ln \frac{l}{l_0} \quad \text{(equation 1)}$$

In the engineering strain equation, $\frac{l}{l_0} = 1 + e$ and moved to equation 1,

$$\epsilon_R = ln\,(1 + \epsilon) \text{ (equation 2)}$$

is found.

For very small strains (I.e. elastic strain) $\boldsymbol{\epsilon}$ values can be taken as $\mathbf{ln}\,(\mathbf{1} + \mathbf{e}) \cong \boldsymbol{\epsilon}$ so $\boldsymbol{\epsilon_g} \cong \boldsymbol{\epsilon}$ is considered. $\boldsymbol{\epsilon}$ gets higher values where the difference between $\boldsymbol{\epsilon_g}$ and $\boldsymbol{\epsilon}$ rapidly increases.

Elastic Limit (σ_e) : It is the highest stress value at which plastic elongation is not seen, or only elastic shape changes occur when the force applied to the material is removed.

Elasticity Modulus (E): The amount of deformation or shape change that occurs in a structure depends on the magnitude of the applied stress. It is the result of the linear relationship between unit elongation and normal stress (tensile or compressive stress) and is defined as stress per unit elongation. The linear relationship between unit elongation and normal stress (tensile or compressive stress) can be defined as:

$$E = \frac{\sigma}{\varepsilon}$$

The proportionality constant E in this equation, known as Hooke's Law, is called the modulus of elasticity or Young's modulus.

The strain change that occurs in proportion to stress is called elastic shape change. In the tensile test, the slope of the linear elastic region, that is, the linear part of the unit shape changes by stress, corresponds to the elastic modulus E.

Modulus of elasticity can be thought of as stiffness, that is, the resistance of a material to elastic shape change. If the modulus of elasticity is high, it means that a material will be rigid, that is, the elastic shape changes after the applied force will be small. This value is an important design parameter in elastic deflection calculations. Elastic deformation is not permanent but means that the part will return to its original shape when the applied load is removed.

The modulus of elasticity is a characteristic property of the material.

Yield Strength (σ_{YS}): Most structures and materials are designed to change shape only elastic under stress. A plastic deformed (permanently deformed) part cannot fulfill the task expected of it. For this reason, it is necessary to know at what point the stress from which the plastic deformation begins, that is, the flow ends. In materials where the elastic-plastic transition is not evident, the yield point can be taken where the curve first diverges from linearity. This value, also called the proportionality limit, indicates the onset of plastic deformation on a micro scale:

$$\sigma_{YS} = \frac{F_{Yield}}{A_0}$$

Tensile Strength (σ_{TS}): The highest tensile stress value that a material can withstand until it breaks or breaks is defined as tensile stress. This stress is the highest stress value in the tensile diagram and is found by the following formula:

$$\sigma_{TS} = \frac{F_{Maximum}}{A_0}$$

Breaking Stress (σ_{BS}): It is the stress value at the time the specimen breaks. It is calculated by the formula:

$$\sigma_{BS} = \frac{F_{Break}}{A_0}$$

Elongation %: It is defined as the highest percentage plastic elongation rate that occurs in the length of the tensile specimen. The final length is measured by bringing together the broken parts of the sample subjected to the tensile test and the elongation in length:

$$\triangle l = l_b - l_i$$

is found by the equation. Here, l_i indicates the initial length of the specimen and l_b indicates the length of the specimen at break. Elongation at break is calculated by the following formula:

$$\%\, EB = \frac{\triangle l}{l_i} \cdot 100$$

This value indicates the ductility of the material.

Cross-sectional Contraction %: It is the largest percentage contraction or shrinkage rate that occurs in the cross-sectional area of the tensile specimen:

$$\%\, CSC = \left(\frac{A_{\dot{I}} - A_B}{A_{\dot{I}}}\right) \cdot 100$$

is found from this equation. Here, A_i denotes the initial cross-sectional area of the test specimen and A_B denotes the cross-sectional area at the time of fracture or the area of the fracture surface. The calculation of A_B can also be done using volume consistency.

$$V_{\dot{I}} = V_B \rightarrow A_{\dot{I}} L_{\dot{I}} = A_B L_B \rightarrow A_B = A_{\dot{I}} \frac{L_{\dot{I}}}{L_B}$$

Cross-sectional narrowing is an indicator of ductility, as is elongation at break. In ductile materials, a significant shrinkage or necking occurs, while brittle materials do not show shrinkage. In Figure 4, the fracture behavior of brittle and ductile materials is shown schematically.

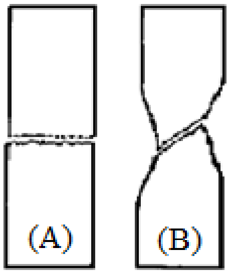

Figure 4. Brittle (A) and ductile (B) material fracture shape

Resilience: The property of the material to give back the energy spent only for elastic deformation, or the energy absorbed when it elastically changes shape by removing the force that makes the shape change is called resistance. This energy is determined by the area under the elastic part of the stress-unit elongation curve and is returned when the specimen breaks:

$$Resilience = \frac{\sigma_{el} \cdot \varepsilon_{el}}{2}$$

Toughness: It is defined as the plastic deformation energy per unit volume of the material and indicates the ability of the material to store or absorb energy until it breaks. Toughness is usually found by calculating the area under the σ – ε curve:

$$\left(\int_{0}^{\varepsilon_b} \sigma . d\varepsilon \right)$$

In this formula, ε_b is the highest or total unit amount of strain that occurs in the material until it breaks, and σ is the tensile strength. The determination of toughness with the help of the stress -strain curve is shown in Figure 5:

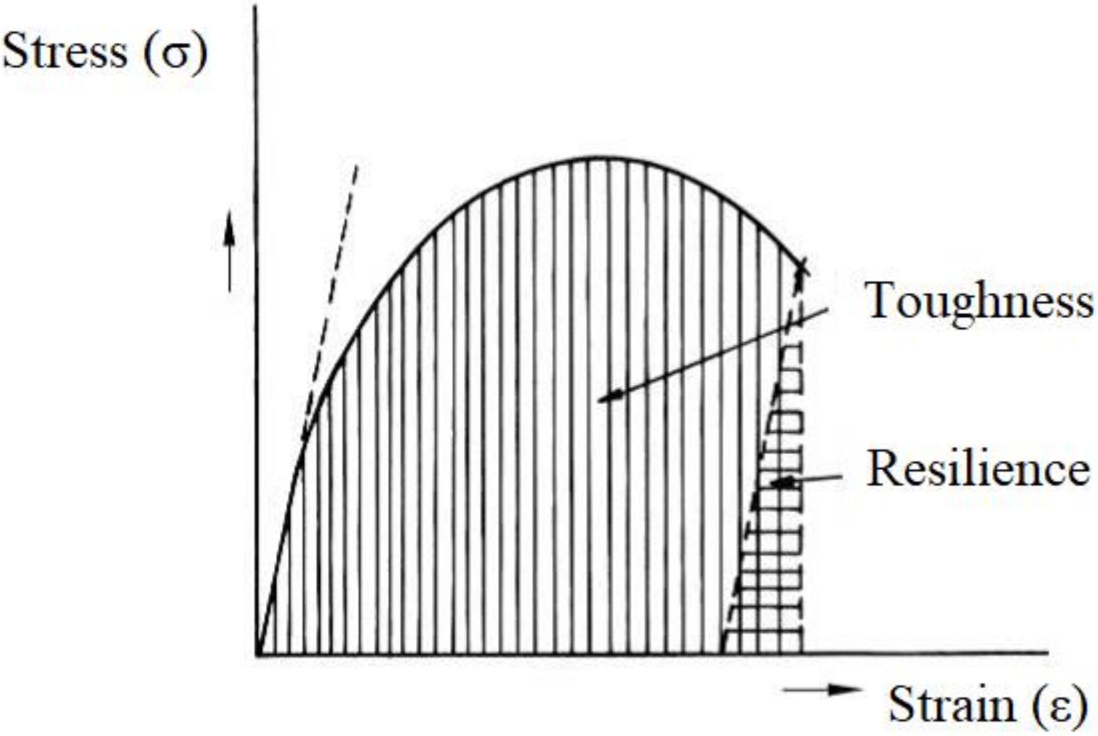

Figure 5. Determination of strain energies (resistance and toughness) with the help of stress - elongation (strain) curve

2. Impact Test

2.1. Objective of the Experiment

The purpose of the impact test in composite materials is to determine the strength of the material against dynamic stresses. The impact strength of the sample is calculated by measuring the amount of energy consumed while crushing the material during the impact test.

2.2. Theoretical Information

Impact tests are generally applied in two types.

a) ***Charpy Impact Test:*** It is the process of determining the energy spent for the fracture of the specimen by applying an impact load to the notch base of the specimen resting on two supports in the form of horizontal and simple beams with the hammer at the end of a pendulum and the effect of multi-axis stresses occurring at the base of the notch.
b) ***Izod Impact Test:*** It determines the energy spent for the fracture of the specimen by the hammer at the end of a pendulum at a certain height from a clutch jaw in the form of a vertical and cantilever beam and the effect of multiaxial stresses at the base of the notch.

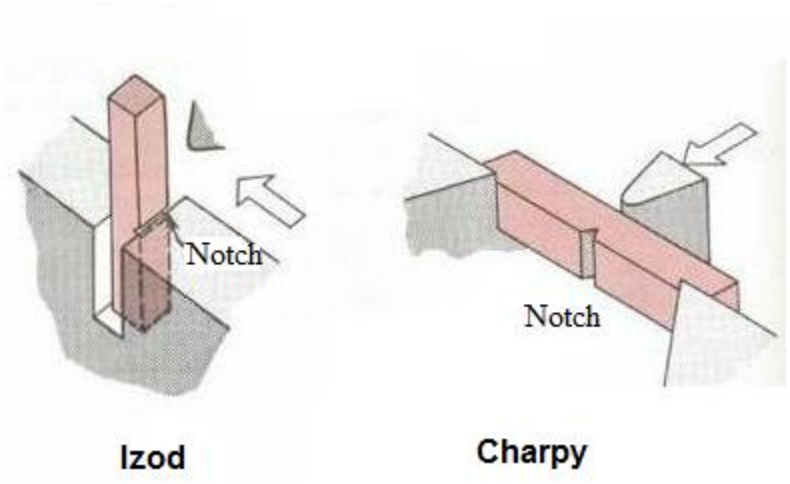

Figure 1. Izod and Charpy experimental setups

In the Charpy test, notches of certain dimensions should be opened, especially in specimens with high ductility and high impact strength. These notches are opened in the form of a V-notch, as shown in Figure 2, in accordance with the test standard. However, in impact specimens of hard and brittle materials, the notches are not opened. These notches are actually made to ensure that the fracture in ductile materials is in a single direction and to make it easier to calculate the fracture area at the end of the impact test.

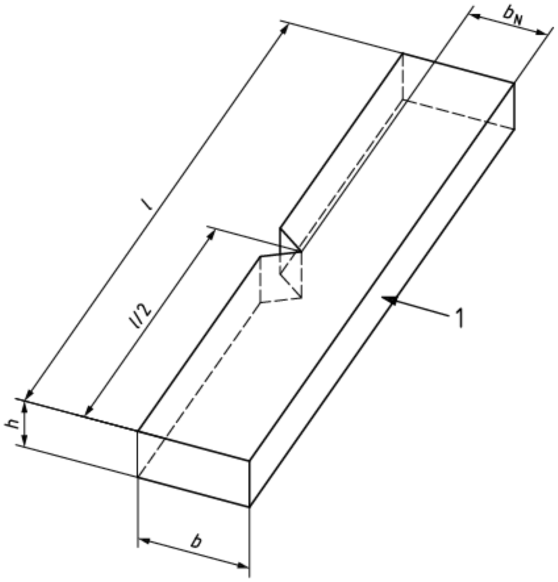

Figure 2. Schematic view of a Charpy specimen conforming to impact test standard

Seen in Figure 2; ℓ indicates the sample length, b indicates the sample width, h indicates the thickness of the sample, b_N indicates the width of the unnotched part of the sample, and 1 indicates the direction of the stroke to be applied by the pendulum..

2.3. The Principle of the Experiment

In the impact test, the amount of energy required to break the sample under dynamic stress is determined. The found value is defined as the energy absorbed by the material in Joules. If this value is divided by the fracture cross-sectional area, impact strength is obtained.

In these experiments, pendulum-type devices shown schematically in Figure 3 are used. The pendulum with mass m has a potential energy of the order of (m x g x h) when it is raised to the height h. When the pendulum is released from this height, it moves in a vertical plane, breaking the specimen and going up to a height of h_1 in the opposite direction. Thus, the potential energy remaining in the pendulum after the breakage of the sample is on the order of (m x g x h_1).

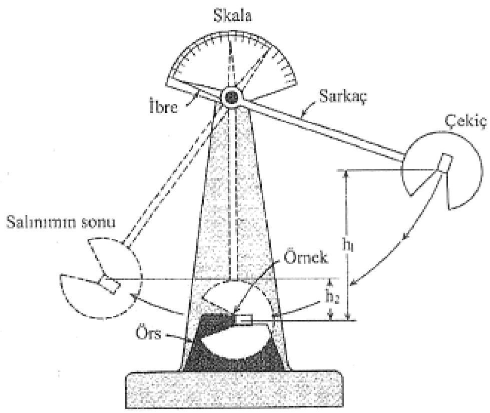

Figure 3. Schematic view of the Charpy impact device

The difference between the potential energy of the pendulum at the time it meets the sample and the potential energy remaining in the pendulum after the sample is broken gives the energy required to break that sample, in other words, the energy absorbed by the material.

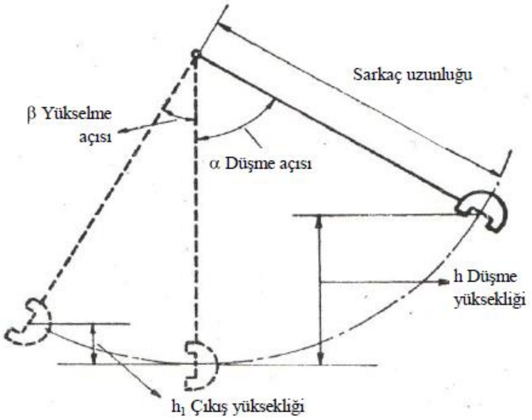

Figure 4. Schematic view of the working principle of the impact tester

Here the impact energy E_i can be calculated with the help of the following equation:

$$E_i = m \cdot g \cdot (h - h_1) - E_f = m \cdot g \cdot L\,(\cos\beta - \cos\alpha) - E_f$$

In this equation:

m : Mass of the pendulum (kg)

g : Acceleration due to gravity (m/s^2)

h : Initial height of the pendulum (m)

h_1 : Final height of the pendulum (m)

E_f : Energy lost due to friction (Joule)

L : Length of the pendulum (m)

2.4. Calculation Of Impact Strength

According to the BS EN ISO 179-1 standard, the impact strength of the composite specimen is calculated using the following equation:

$$\sigma_{cN} = \frac{W_c}{A_{CS}}$$

In this equation:

W_C : Energy absorbed by the sample during the impact test (Joule)

A_{CS} : Cross-sectional area (mm^2)

Also, the unit of strength (σ_{cN}) to be calculated according to the standard should be kJ/m^2.

3. Density Test

3.1. Objective of the Experiment

The objective of the experiment is to determine the density of composite materials with a polymer or polymer (plastic) matrix of unknown volume by immersion method.

3.2. Theoretical Information

Density refers to the amount of mass per unit volume of a material and gives important information about the properties of the material. Its formula is calculated as:

$$\rho = \frac{m}{V}$$

In this equation ρ is the density (g/cm^3), m is mass (g) and V is the volume (cm^3).

It is very difficult to obtain theoretical density values due to the fact that the materials contain voids and impurities in their volumes. Defects, inhomogeneities, impurity, porosity and purity of the material in the structure limit the achievement of the same theoretical density. For example, since the cavities remain closed in the structure, they are called closed porosity, while the cavities that appear on the surface and are in the form of indentations are called visible porosity (all open and semi-open porosities). Therefore, density calculations are made based on the apparent density. If the material is a material that can absorb water, the porosity present in the material can be calculated, but as mentioned, it is not possible to obtain a clear amount of porosity since the amount of impurity and impurity is not known exactly. For this reason, density measurements are made using the apparent density, that is, the current volume and mass of the material.

According to Archimedes' principle, thanks to the buoyant force, the density of the object can be calculated by switching from the weight to the volume of the object suspended in a liquid of known density. That is, the displaced mass of liquid is equal to the volume of that body.

A total of 3 samples, one pure polymer and two composite samples, will be used in the test. Since these samples are in solid form, BS EN ISO 1183-1 will be used as the test standard. All samples, the immersion liquid and density kit to be used in the test were selected and prepared in accordance with the said standard.

3.3. Preparation Of Test Specimens

- Samples should be cut to a size that will fit properly in the immersion container.
- Each sample should be prepared to be no smaller than 1 gram if possible.
- Samples should be cut with a suitable device so as not to lose their characteristics.
- There should be no open porosities in the prepared samples that could cause air bubbles. If there are any, they must be completely filled with the immersion liquid.
- If air bubbles form in the liquid due to the shape of the sample left in the immersion liquid, the air bubble must be removed with a suitable apparatus.
- If the sample is taken from the production site, it should be tested at least 2 hours after the production time. In other words, there should not be any wetness on the sample, it should be completely dry.

3.4. Devices, Tools and Consumables to be Used in the Test

- Precision scales
- Density kit
- An immersion liquid with a suitable density

3.5. Points to be Considered During the Test

The liquid or solution in which the test specimen is immersed and in contact during the test should not affect the specimen.

3.6. Conducting The Experiment

- Whether the prepared samples are less than 1 gram or not is verified by weighing one by one. Sample weights are recorded in the Experiment Record Form.
- The ambient temperature and humidity are measured and recorded in the Experiment Record Form.
- Determination of the density of the immersion liquid, the determination of the nun in which the experiment is carried out is not performed, at the beginning of each test series, the immersion liquid is used by transferring new liquid from the bottle and the density on the bottle is taken as the density of the immersion liquid, recorded in the Test Record Form. (ρ_{IL}) (TS EN ISO 1183-1 Article 5.1.2)
- The sample to be weighed should be placed approximately in the centre of the precision balance pan.
- The covers of the balance should be closed in every region while weighing is in progress.
- The prepared sample is weighed on the precision balance and the result is recorded in the Test Record Form. ($m_{S,A}$)
- The immersion container is filled with immersion liquid and placed into the balance. The balance weight value is zeroed.
- The sample weighed on the precision balance is thrown into the immersion liquid in the balance and the weight is measured. While the sample is in the immersion liquid, it is checked whether there are air bubbles, if any, the bubbles are removed with the help of a wire. Wait until the weight changing on the balance indicator remains constant and record the result on the Experiment Record Form. ($m_{S,IL}$)
- All measured weights are made with an accuracy of 0.1 mg.

Figure 1. Density measurement of a test specimen according to BS EN ISO 1183-1

The schematic figure above shows the density measurement of a test sample. In this method, from left to right, the weights of the sample in air and then in the immersion liquid are measured and recorded. Finally, the density is calculated from these values.

3.7. Calculation Of Density

According to BS EN ISO 1183-1 Standard, density is calculated according to the formula given below:

$$\rho_S = \frac{m_{S,A} \cdot \rho_{IL}}{m_{S,A} - m_{S,IL}}$$

were,

$m_{S,A}$: Weight of the sample in air (g)

$m_{S,IL}$: Weight of the sample in the immersion liquid (g)

ρ_{IL} : Density of immersion liquid (g/cm^3)

ρ_S : Density of the sample (g/cm^3)

4. Calcination Test

4.1. Objective Of The Test

The objective of this test is to determine the fiber volume ratios of polymer matrix composite materials.

4.2. Theoretical Information

In polymer matrix composites, the main load-bearing component is fibers. For this reason, knowing the fiber volume ratio in composite materials also provides important data about their mechanical strength. In general, it can be said that the higher the fiber volume ratio of a composite material, the higher its mechanical strength (such as tensile, bending, impact).

4.3. Conducting The Experiment

Fiber volume ratios in polymer matrix composites are determined by testing in accordance with the BS EN ISO 1172 standard. The performance of this test is as follows:

a) An empty crucible is weighed and its weight is written in grams. (m_1)
b) A sample that will represent the entire composite material is placed in this crucible and weighed in grams. (m_2)
c) This crucible is calcined in a calcination (muffle) furnace at a temperature of 650°C for 30 minutes according to the test standard.
d) After the crucible has cooled sufficiently, it is taken out of the oven and weighed in grams with the remaining fiber inside. (m_3)

4.4. Calculation

According to the BS EN ISO 1172 standard, the glass fiber content is calculated as a percentage of mass with the help of the following equation:

$$M_{Glass\ Fiber} = \frac{m_3 - m_1}{m_2 - m_1} \cdot 100$$

In this equation:

m_1 : Weight of empty crucible (g)

m_2 : Weight of the crucible with the composite specimen (g)

m_3 : Weight of the crucible together with the fiber after calcination (g)

If deemed necessary, the fiber volume ratio of the composite material can also be calculated by taking into account the matrix and glass fiber densities.

5. Requirements in the Report

a) In the part related to the Tensile Test, separately for all 3 samples,

b) Draw the Stress – Strain graph with the data obtained from the tensile test (raw data). ***(10 Points)***

c) Calculate Modulus of Elasticity (E-Modulus), Yield Strength (σ_{YS}), Tensile Strength (σ_{TS}) and toughness values. ***(20 Points)***

d) Interpret and evaluate the results obtained for 3 samples. ***(10 Points)***

e) Calculate the impact strength by giving information about all samples related to the impact test and interpret the results by comparing these strength values. ***(20 Points)***

f) Write down the conduct of the density experiment in such a way that each step is complete. Interpret the results by calculating the density value of each sample. ***(15 Points)***

g) Calculate the fiber volume ratio and porosity (void) ratio values of the two composite samples with the data you obtained at the end of the calcination and density tests. ***(20 Points)***

h) Write down what you have learned and what you have achieved at the end of the experiments. Write your report in the format of a standard test report and write the sources you use or use in the "References" section at the end of the report. ***(5 Points)***

6. References

[1] Autar K. Kaw, Mechanics of Composite Materials, Taylor & Francis Group, 2006.

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[6] A.R. Horrocks & S.C.Anand, Handbook of Technical Textiles, Woodhead Publishing Limited, 2000.

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Experiment 9: Mechanical Tests

Res. Asst. Zekeriya CÖMERT

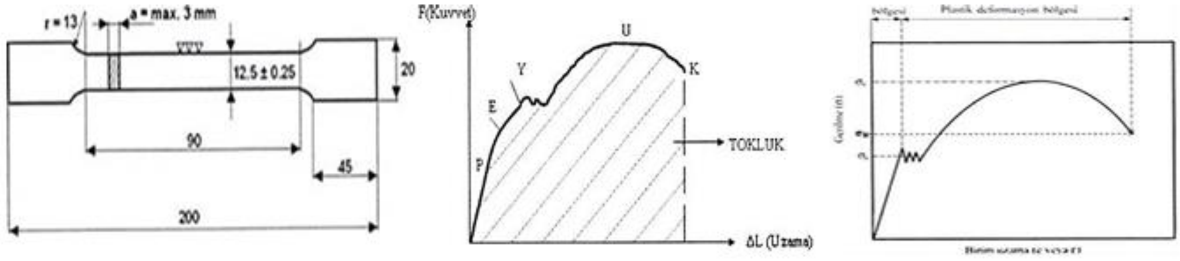
1. Tensile Test

1.1. Purpose of the Experiment

Tensile testing is performed to determine essential design information about the strength of materials and to classify materials according to their properties. Tensile test is the pulling of a test sample prepared according to standards on a single axis, at a certain pulling speed and at a constant temperature, until it breaks. During the test, the elongation values resulting from the force or tension applied to the tensile sample prepared according to the standards are recorded.

1.2 Therotical Information

Samples to be subjected to tensile testing must have all the properties of the material they represent. Therefore, the region where the sample is taken and the way it is collected and prepared are of great importance. While preparing the sample, care should be taken to avoid any changes in the material properties it represents. Excessive temperature formation should be prevented during production, and effects resulting from deformation should be eliminated. The shape and dimensions of the sample to be subjected to tensile testing are specified in the standards. For this reason, in order to ensure the reliability and comparability of the test results, the experiments should be carried out with samples prepared according to standards. Depending on the material to be taken, the cross-section of the tensile sample may be circular, square, rectangular, ring-shaped, and even in some cases special profile-shaped. Turkish standards have standardized tensile samples according to their shapes and sizes in TS EN ISO 6892-1.



Şekil 1. Dikdörtgen kesitli metal malzemeler için çekme numunesi ölçüleri, gerilme-gerinim diyagramı ve Düşük karbonlu yumuşak bir çeliğin çekme diyagramı.

The pulling device is mainly; It consists of two jaws that can move up and down relative to each other, to which the test piece is attached, and units that give movement or force to them and measure these two quantities. By moving one of the jaws at a constant speed, varying amounts of tensile force is applied to the test piece and the elongation corresponding to this force is recorded. At small force levels, the amount of extension is directly proportional to the force. The material is in elastic behavior; That is, when the force is removed, the elongation is reset. This character continues until point P. The proportionality limit changes the slope of the linear function after P. However, elastic behavior continues. Elastic behavior ends at point E “Elastic Limit”. Permanent after E; In other words, it can take too much force and plastic deformations begin. When the force is reduced, it follows a path parallel to the linear function. However, where the force is zero, the deformation is no longer zero, a certain plastic deformation remains. If the material continues to be loaded, it will flow at point Y. At the yield point, a large amount of plastic deformation occurs while the force is the same. The flowing material undergoes “work hardening” and becomes more durable. By increasing the force on this material, point U is reached. Point U is the "maximum stress" point, where local contractions begin in the material cross-section. This is called "necking" of the material. Necking also causes the material to undergo work hardening and the material can take on higher stresses; However, as the cross-sectional area narrows in the neck region, the net force it carries decreases. The sample usually moves uncontrollably to point K and breaks there. The area under the force-extension curve equals the energy required to degrade that sample; It is called satiety. The force extension curve is then rescaled. Elongations are converted to “unit-extension” by dividing by the initial length of the material. Likewise, the “tension” is calculated by dividing the force by the initial cross sectional area of the sample and the vertical axis is scaled again.

Definitions

Stress(σ): It means the load acting on unit area and is calculated by the formula:

$$\boldsymbol{\sigma = \frac{F}{A0}}$$

Unit Strain (ε): The ratio of the length change that occurs when a force is applied to the material to the initial length before the force is applied:

$$\boldsymbol{\varepsilon = \frac{\Delta L}{L0}}$$

Elasticity Modulus (E): It is the measure of the strength (strength) of the material. It is a result of the linear relationship between unit elongation and normal stress (tensile or compressive stress) and is defined as the stress per unit elongation. The linear relationship between unit strain and normal stress (tensile or compressive stress) can be defined as follows:

$$\boldsymbol{E = \frac{\sigma}{\varepsilon}}$$

When force is applied to the material, the elongations occurring in the material are proportional to the stresses within elastic limits. This is called "**Hooke's Law**". Elastic modulus is a characteristic feature of the material.

Yield Strength (σ_a): It is the stress value corresponding to the part where the applied tensile force remains approximately constant, but the plastic deformation increases significantly and the tensile diagram shows irregularity.

$$\boldsymbol{\sigma a = \frac{Fa}{A0}}$$

Tensile Strength ($\sigma_ç$): It is defined as the highest tensile stress that a material can withstand until it breaks or breaks. This stress is the highest stress value in the tensile diagram and is found by the formula below.

$$\boldsymbol{\sigma ç = \frac{Fmax}{A0}}$$

Breaking Stress (σ_K): It is the stress value when the sample breaks.

$$\boldsymbol{\sigma K = \frac{FK}{A0}}$$

Percent Elongation at Break (KU): It is defined as the highest percent plastic elongation rate occurring in the length of the tensile sample. The final length is measured by bringing together the broken parts of the sample subjected to tensile testing and the elongation in length is measured:

$$\mathbf{\Delta L = L_k - L_0}$$

It is found by the relation. Here, L0 indicates the initial measurement length of the sample, and Lk indicates the length of the sample at the moment of breaking. Elongation at break is; It is determined with the help of the relation. This value shows the ductility of the material.

$$KU(\%) = \frac{\Delta L}{L0} x100$$

Percent Section Shrinkage (KD): It is the largest percentage contraction or shrinkage rate occurring in the cross-sectional area of the tensile sample;

$$KD(\%) = \frac{A0 - AK}{A0} x100$$

It is calculated using the equation. Here, Ao indicates the initial cross-sectional area of the test sample, and AK indicates the cross-sectional area at the moment of fracture or the area of the fracture surface. To calculate EC, the expression that the volume will remain constant is used.

$$V_0 = V_K \qquad A_O.L_O = A_K.L_K \qquad A_K = A_0\ (L_o / L_K)$$

Section narrowing is an indicator of ductility, as is elongation at break. While a significant shrinkage or necking occurs in ductile materials, brittle materials do not show shrinkage. The fracture behavior of brittle and ductile materials is shown schematically in Figure 4.

Figure 2. (a) Fracture pattern of brittle material (b) fracture pattern of ductile material.

1.3. Experimental Procedure

For the tensile test, first a tensile sample in accordance with the standards is prepared from the material to be tested (Figure 2). Markings are placed on this sample. This sample, which is clamped properly and centrally between the jaws of the tensile testing machine, is pulled with an increasing load until it breaks. Meanwhile, the applied load F and the elongations (ΔL) shown by the material against it are measured by the device and video extensometer. Using the load (F) and elongation (ΔL) values obtained as a result of the experiment, the (F - ΔL) diagram is obtained. This diagram is also called a tension diagram.

1.4 Information Required in the Experiment Report

- Obtaining a stress-strain diagram separately for both force and elongation data obtained from the video extensometer.
- Calculation of elasticity modules from stress-strain diagrams.
- Calculation of yield stress, tensile strength and breaking stresses from stress-strain diagrams.
- Calculation of percent elongation at break and percent section reduction.

2. Hardness Test

2.1. Purpose of the Experiment

Measuring the hardness of materials and obtaining information about their strength.

2.2. Theoretical Background

Hardness is defined as the resistance of materials to plastic deformation. Hardness tests are one of the very important mechanical tests that enable quick and non-destructive control of materials and manufactured parts. Hardness measurement methods commonly used in technology are methods based on measuring the size of the permanent mark obtained on the sample. In all hardness methods, at least three hardness values are measured from each sample and the average value and standard deviation are calculated.

Rockwell Hardness: Rockwell hardness test principle is given in Figure 3. While performing the Rockwell hardness test, a preload of 10 kg (F0) is first applied to the sample. This preload is applied to ensure precise contact between the sample and the tip and to eliminate gaps in the measurement system. The depth to is reached by applying the preload F0. This location is taken as the reference plane for the hardness scale. After the preload is applied, a main load (Fana) is applied to the sample for approximately 10 seconds until the load amounts given above for different hardness scales are reached. The main load values are 90 kg for the Rockwell B experiment and 140 kg for the Rockwell C experiment. After applying and removing the fan test load, which must be at least four times greater than the blow dryer force, on the sample, a permanent sinking depth tb is obtained from the reference plane. In the Rockwell device where the processes in question are carried out, the hardness value is read directly from the indicator, instead of the measured permanent tb penetration depth.

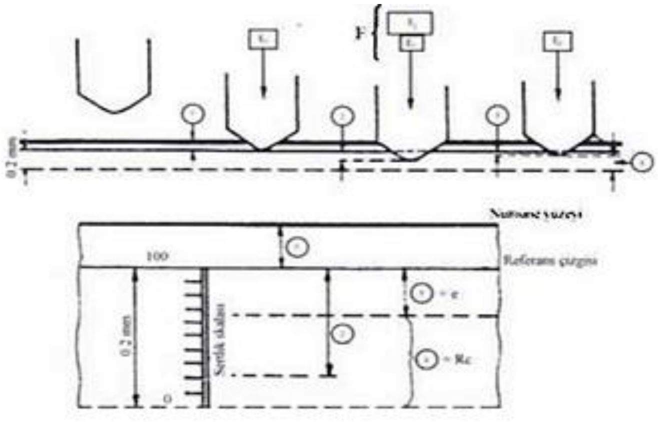


Figure 3. Rockwell Hardness Test Procedures. 1) The penetration depth of the tip at preload (10 kg), 2) Submersion depth of the tip at main loading (90 kg or 140 kg), 3) The penetration depth of the tip when the main load is removed (e), 4) Rockwell hardness (100-e).

Brinell Hardness: As shown in Figure 6.6, a permanent mark is created on the surface as a result of applying a certain load (F) to the surface of the material for a certain period of time with the help of a ball made of hard material of a certain diameter (D).

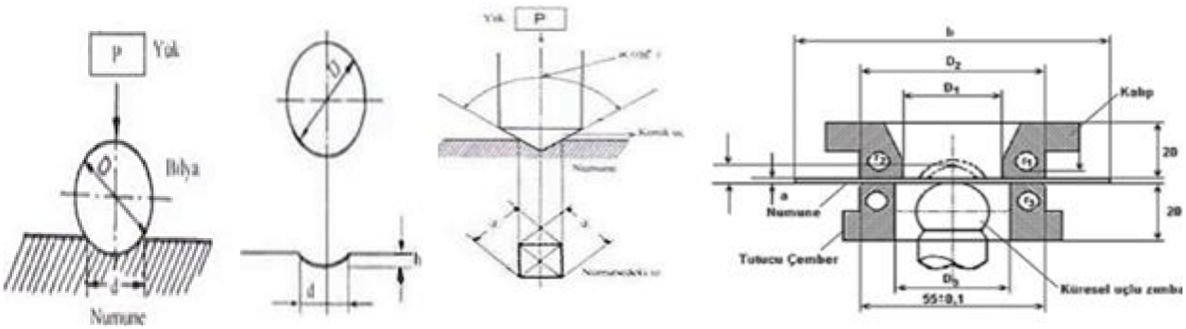


Figure 4 From left to right: Brinell Hardness, Vickers Hardness, and Erichsen cupping index

The spherical surface area of the resulting trace is determined by using the measured average diameter of the trace's circumference and the ball diameter. Accordingly, Brinell hardness value:

$$\mathbf{BSD} = \frac{2F}{\Pi D(D-\sqrt{(D^2-d^2)}}$$

It is calculated using F = Applied load (kg), D = Ball diameter (mm), d = Trace diameter (mm).

Vickers Hardness: As shown in Figure 6.7, the pyramid diamond tip creates a permanent square-based mark on the material to be measured for a certain time and load. By determining the diagonal average of the symmetrical trace formed, the Vickers hardness value of the material is calculated with the help of the equation given below.

$$\mathbf{VSD} = \frac{2F\,Sin(a/2)}{d2} = \frac{1{,}8544\ F}{d2}$$

F = Uygulanan deney yükü (kg), d = iz köşegenlerinin ortalaması (mm), a = Tepe açısı=136°.

3. Deep Tensile Test

3.1. Purpose of the Experiment

It is a test performed to determine the plastic deformation capability of metallic sheets and tapes. At the end of the test, the "Erichsen collapse value" is evaluated by measuring the depth in millimeters at the moment of tearin

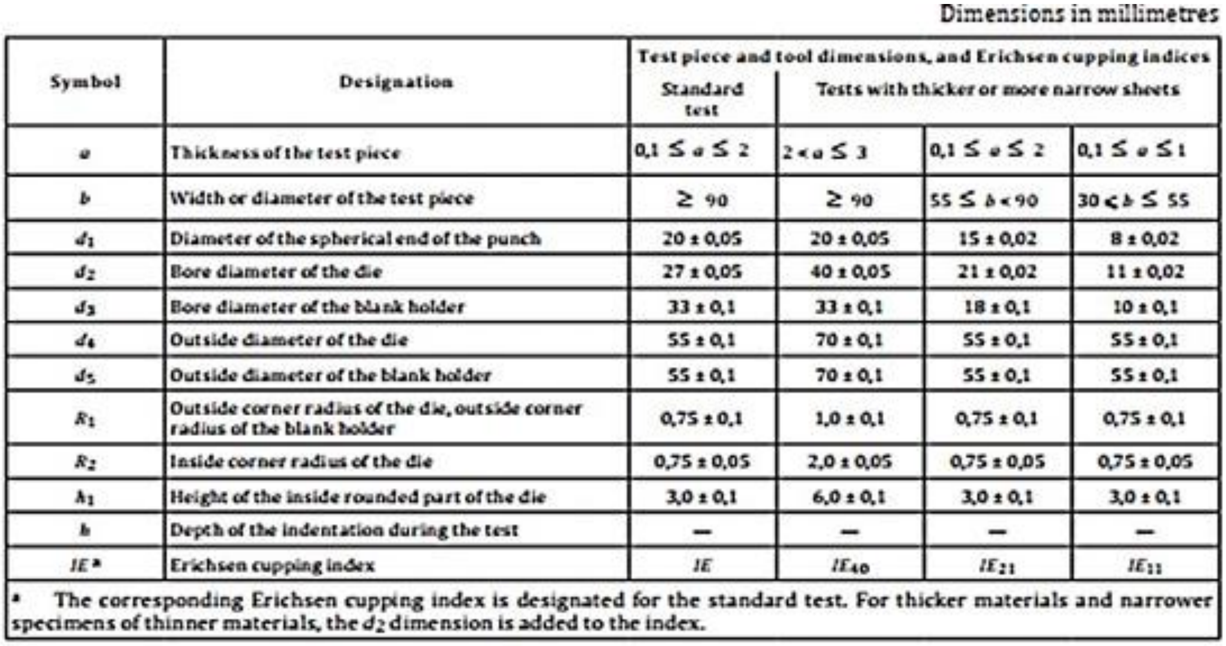
3.2. Theoretical Informations

The results of the Erichsen test are not used to standardize the materials, but rather they are examined as values that allow each material to be compared with the values of its own standard. The Erichsen test is especially applied to sheet metal and tapes used in processes such as press forming, plastering and deep drawing. The depth is the path taken from the first contact of the ball to the sheet until the moment the sheet cracks. With this test, the susceptibility of the material to plastic deformation and the macroscopic examination of the crack formed are examined. Depth depends on the sheet thickness. As the sheet thickness increases, the clamping ball is changed and more clamping force is applied. Depth values determined by curves for each sheet quality were determined. Residues such as phosphorus and sulfur that may be present in the material, as well as solidification rate, rolling method and heat treatments adversely affect the formability. The test can be performed until a crack is formed or until a certain deformation. In general, it is desired that the steel sheet does not tear. It is mostly applied on sheets used in automotive and white goods. The grain size of the material can be seen on the precipitation base. An orange-shaped surface (tattered edges) indicates coarse grain (an undesirable feature), while a shiny surface (circular crack) indicates fine grain (suitable). "limit shrinkage ratio" with the help of the largest diameter D that can be pulled without tearing at the base of the resulting overhang called the D/d ratio. If the pressure of the compression ring is too large, the frictional forces increase and the sheet will tear prematurely; conversely, if it is too small, the sheet will fold around the periphery under the influence of compressive stresses and tear again. The forming capability is evaluated according to the magnitude of the limit shrinkage ratio. The smaller the clamping diameter, the more easily tensile cracking occurs. The indentation test apparatus is generally composed of a holding hoop and die for fixing the specimen and a ball or spherical tipped punch that forces the specimen into the die. The test apparatus makes it possible to easily and accurately determine the moment when fracture begins in the specimen. For this purpose, there is an indicator that allows to measure the greatest depth of the collapsed part at the moment when the sample starts to break. The indicator can measure the depth of the collapsed portion at 0.01 mm intervals. There are also indicators to measure the force of the specimen breaking and the force of compression of the specimen before starting the experiment. The mold, retaining ring and spherical punch on the device are made of sufficiently strong materials so that they do not change shape during the experiment, which may affect the results.

3.3. Test Specimens

The specimens to be subjected to the test must be smooth and free of folds. Normally, specimens are prepared for at least 3 tests. Before the test, the specimen should not be subjected to hot or cold treatment in order to straighten it. There should be no defects such as burrs etc. on the edges of the specimen. The specimens used in the test are sized depending on the thickness and width of the sheets or bands they represent. These dimensions are given in the table. In the TS EN ISO 20482 Metallic materials - Sheet and strips - Erichsen precipitation test standard, the diameter of each steel is specified.

Table 1. Symbols and designations.



Dimensions in millimetres

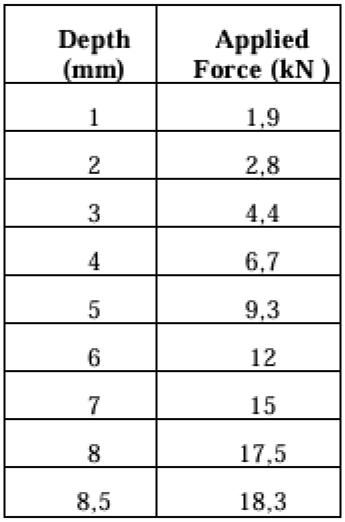
Symbol	Designation	Test piece and tool dimensions, and Erichsen cupping indices			
		Standard test	Tests with thicker or more narrow sheets		
a	Thickness of the test piece	$0,1 \leq a \leq 2$	$2 < a \leq 3$	$0,1 \leq a \leq 2$	$0,1 \leq a \leq 1$
b	Width or diameter of the test piece	≥ 90	≥ 90	$55 \leq b < 90$	$30 \leq b \leq 55$
d_1	Diameter of the spherical end of the punch	20 ± 0,05	20 ± 0,05	15 ± 0,02	8 ± 0,02
d_2	Bore diameter of the die	27 ± 0,05	40 ± 0,05	21 ± 0,02	11 ± 0,02
d_3	Bore diameter of the blank holder	33 ± 0,1	33 ± 0,1	18 ± 0,1	10 ± 0,1
d_4	Outside diameter of the die	55 ± 0,1	70 ± 0,1	55 ± 0,1	55 ± 0,1
d_5	Outside diameter of the blank holder	55 ± 0,1	70 ± 0,1	55 ± 0,1	55 ± 0,1
R_1	Outside corner radius of the die, outside corner radius of the blank holder	0,75 ± 0,1	1,0 ± 0,1	0,75 ± 0,1	0,75 ± 0,1
R_2	Inside corner radius of the die	0,75 ± 0,05	2,0 ± 0,05	0,75 ± 0,05	0,75 ± 0,05
h_1	Height of the inside rounded part of the die	3,0 ± 0,1	6,0 ± 0,1	3,0 ± 0,1	3,0 ± 0,1
h	Depth of the indentation during the test	—	—	—	—
IE [a]	Erichsen cupping index	IE	IE_{40}	IE_{21}	IE_{11}

[a] The corresponding Erichsen cupping index is designated for the standard test. For thicker materials and narrower specimens of thinner materials, the d_2 dimension is added to the index.

3.4 Experimental Procedure

The specimen is fixed with a precision of 0.01 mm. The specimen and spherical punch are lubricated with grease containing 23 - 28 % graphite. It is clamped between the mold and the holder ring with a load of 500 - 100 kg. When placing the specimen, make sure that the center of the collapse zone is equidistant from the edges. First, the sheet metal is compressed into the device with press load. According to the type and thickness of the steel, the speed of the plunging speed of the placed tamp is adjusted. Then, with the start of the load application, the bulge starts to form. The applied load is controlled from digital and analog indicators. It is observed that the load continues to increase for a while, but then the load value decreases. When the load starts to decrease, the load application is terminated and the device is stopped since tearing of the material is not desired. The amount of plunge, decrease in load and max. load are read from the indicators. The reason for having both analog and digital components in the device is that the variability of current and voltage does not affect the mechanical parts. Analog indicators with mechanical parts are known to be safer in case of excessive voltage fluctuations. A sample experiment data is given below. The values 8.5 mm and 18.3 kN are specimen cracking values.

Table 2. St13 Cold Rolled Steel Sheet Test Data.



Depth (mm)	Applied Force (kN)
1	1,9
2	2,8
3	4,4
4	6,7
5	9,3
6	12
7	15
8	17,5
8,5	18,3

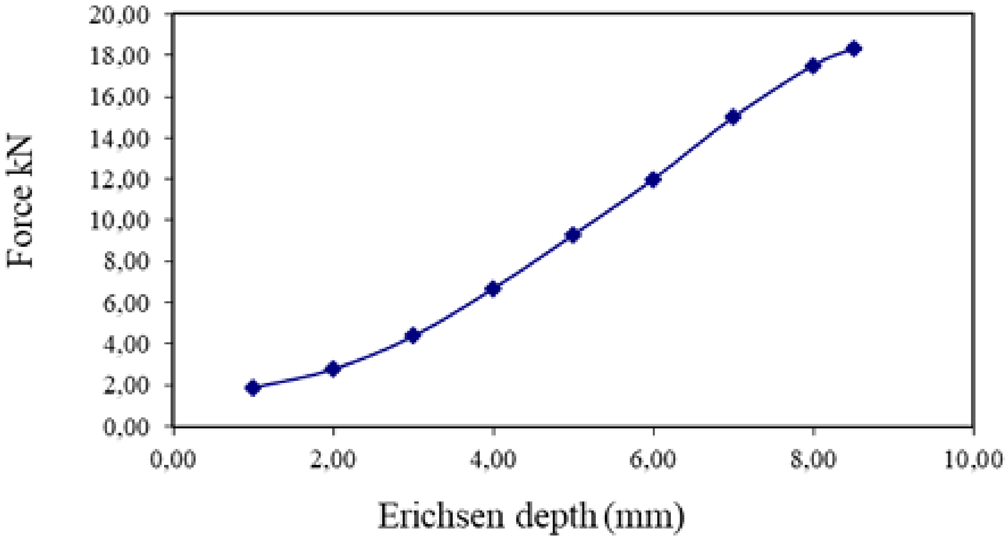


Figure 7. St13 Cold Rolled Steel Sheet Test Cracking curve.

3.5 Information Required in the Experimental Report

Draw and interpret the force depth curves of the tested specimens in scale.

3.6 References

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[2] William F. Smith 2006," Materials Science and Engineering" (Translated: Nihat G., Kınıkoğlu). Faculty of Chemistry-Metallurgy, Offset Workshop, Istanbul.

[3] TS EN ISO 6892-1 Metallic materials - Tensile test - Part 1: Test method at ambient temperature

[4] TS EN ISO 20482 Metallic materials - Sheet and strips - Erichsen precipitation test

[5] TS EN ISO 6506-1 Metallic materials - Brinell hardness test - Part 1: Test method

[6] TS EN ISO 6507-1 Metallic materials - Vickers hardness test - Part 1: Test method

[7] TS EN ISO 6508-1 Metallic materials - Rockwell hardness test - Part 1: Test method

Experiment 10: Jominiy Hardenability Test

Res. Asst. Oğuler SAZCI

1. Objective of the Experiment

- Distinguish between hardness and hardenability.
- Understand the concept of mass effect and ruling section.
- Perform Jominy test as a method to indicate hardenability.

2. Therotical Knowledge

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 Fig. 1.

As the carbon percentage increases A3 decreases, similar is the case for Ar3, 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, Acm 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.

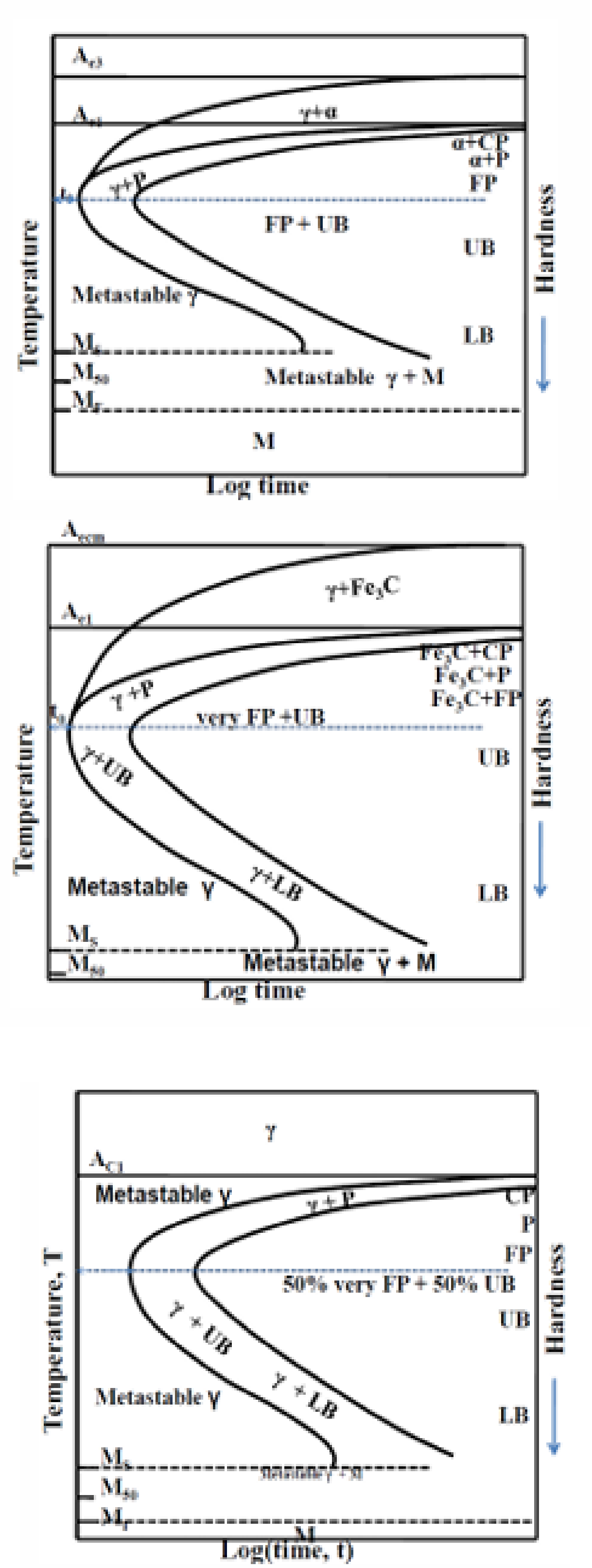

Figure 1 TTT diagrams for (a) hypoeutectoid, (b) eutectoid and (c) hypereutectoid steels (up to down,, respectively)

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 Procedure

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 vary rapid at the quenched end to air cooled at the far end.

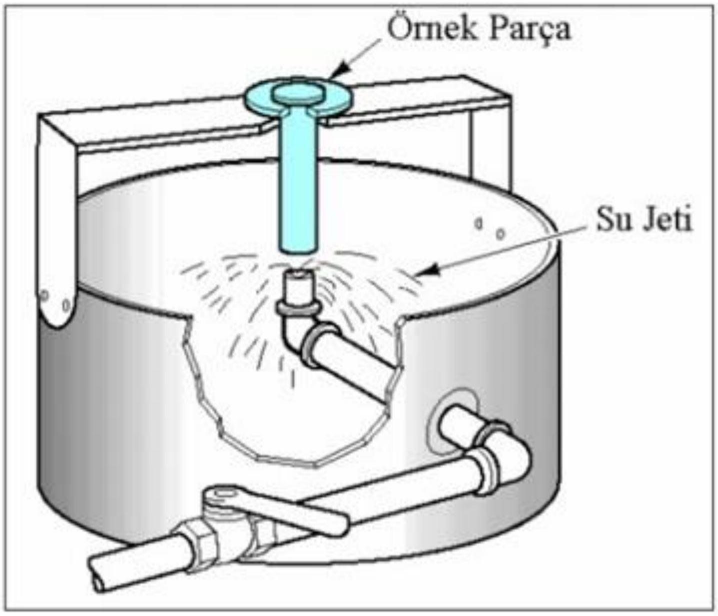

Figure 2. Schematic view of Jominy test

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 where as 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 affect 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 affective. 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 effect the hardenability of steels in this manner. Chromium, Molybdenum and Manganese being used most often.

4. Request in the Test Report

- Draw Jominy curve for 1040, 1060, 4140 and 4340 steels depending on the distance from the end.
- Explain how the Grossman hardenability test is performed and the advantages and weaknesses compared to the Jominy experiment.
- 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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3. Llewellyn D T and Hudd R C, Steels: Metallurgy and Applications, 3rd Edition, Reed Educational and Professional Publishing, 1998.

4. ASM Handbook, Volume 4: Heat Treating, ASM International, 1991.