

EXPERIMENT NO.-1

AIM: Determining the physical, chemical and mechanical properties of different class of materials

OBJECTIVES:

- To study the difference in thermal conductivity of different class of materials.
- To study the difference in electrical conductivity of different class of materials.
- Determine density of different class of materials.
- To study the difference in hardness of different class of materials.
- To study the chemical stability of different class of materials.

EXPERIMENTAL SET-UP:

The thermal conductivity experiment would be conducted on a hot plate attached with pyrometer for temperature measurement. Electrical conductivity measurements require digital multi-meter. Further, the hardness of the samples will be measured using simple scratch test. Chemical stability would be investigated using different reactive chemicals.

CONSUMABLE REQUIRED:

Acrylic (polymer), mild steel and glass slide

THEORY:

Materials are deeply rooted in our culture and contribute significantly in one form or other in our daily life. Understanding the different class of materials and their inherent properties is critical. Solid materials have been conveniently grouped into 3 basic categories: metals, ceramics and polymers, a scheme based primarily on chemical & atomic structure. Most materials fall into one distinct grouping or another. Metals are composed of one or more metallic elements (e.g., iron, aluminium, copper, titanium, gold, nickel) often containing some non-metallic elements (e.g., carbon, nitrogen, oxygen) in relatively small amounts. Atoms in metals and their alloys are arranged in a very orderly manner and are relatively dense in comparison to the ceramics and polymers. With regard to mechanical characteristics, these materials are relatively stiff and strong, yet are ductile (i.e. capable of large amounts of deformation without fracture) and are resistant to fracture. Ceramics are compounds between metallic & non-metallic elements; they are most frequently oxides, nitrides and carbides (E.g. aluminium oxide (or alumina, Al_2O_3), silicon dioxide (or silica, SiO_2), silicon carbide (SiC)). With regard to mechanical behaviour, ceramic materials are relatively stiff and strong. Further, they are typically very hard and exhibit extreme brittleness (lack of ductility) and are highly susceptible to fracture. They are also poor conductor of electricity and heat, however, possess high resistance to chemical attack and high temperature. Polymers include the familiar plastic

and rubber materials. Many of them are organic compounds that are chemically based on carbon, hydrogen and other non-metallic elements (i.e. O, N and Si). Furthermore, they have very large molecular structures, often chain-like in nature, that often have a backbone of carbon atoms. Some common and familiar polymers are polyethylene (PE), nylon, polyvinylchloride (PVC), polycarbonate (PC), polystyrene (PS), and silicone rubber. These materials typically have low densities and strength compared to metals and ceramics. In addition, many of the polymers are extremely ductile and pliable (i.e. plastic), which means they are easily formed into complex shapes. In general, they are relatively inert chemically and unreactive in a large number of environments. One major drawback to the polymers is their tendency to soften and/or decompose at modest temperatures, which, in some instances, limits their use. Furthermore, they have low electrical conductivities and are nonmagnetic.

PROCEDURE:

- Prepare samples of equal dimensions from glass, PS and aluminium.
- Measure the dimensions and weight of the samples for calculating the density.
- Further, place these samples over the hot plate.
- Set the temperature on the hot plate and turn it ON.
- Record the temperature on the top surface of the samples after different interval of time, and record in the

Material	Density (kg/m ³)	Electrical Resistance	Temperature (°C)			Scratch Resistance
			T ₁	T ₂	T ₃	
Glass	2.48×10^3	Overloading	43°C			High
Al plate	7.66×10^3	0.7Ω	22°C			High, except ceramic
Polystyrene	1.04×10^3	overloading	28°C			Least

Comment on Chemical Resistance:

	Reacts with Nitric Acid	Reacts with Acetone
Metal	✓	✗
Ceramic	✗	✗
Polyester	✗	✓

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Observation table.

- For checking electrical conductivity of the samples, place them on a non-conductive stand.
- Turn ON the multi-meter and check the electrical resistance after attaching probes on opposite ends.
- Further, following the Moh's hardness measurement process, try to scratch each sample with the other, starting with glass.
- Give ranking for scratch resistance to the samples starting from 1 for the material with lowest resistance.
- To check the chemical stability, place a drop of different chemicals on the samples and check reactivity with different materials. Comment on chemical stability of the materials to different reagents in the Observation section.

OBSERVATION:

Sample Dimension: Glass → length: 49.83 mm weight: 11.78 g
width: 20.17 mm thickness: 4.72 mm

Acrylic → length: 49.94 mm weight: 4.86 g
width: 19.75 mm thickness: 4.74 mm

Metal → length: 49.91 mm weight: 42.28 g
width: 19.77 mm thickness: 5.59 mm

Set temperature on hot plate: 90°C

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CONCLUSION:

- Al plate (metal) has highest density.
- The thermal conductivity of the glass (ceramic) is highest.
- The electrical conductivity of the metal is highest.
- The scratch resistance of Glass (ceramic) is highest and Polymer (acrylic) has lowest hardness.
- Glass (ceramic) has highest chemical resistance.

PRECAUTIONS:

- a. Do not touch the ~~hot~~ top surface of the hot plate
- b. Handle chemicals carefully and avoid spillage
- c. Samples should not be contaminated.
- d. The pyrometer should be handled carefully and not aimed towards eyes.

~~Nov 28/18/24~~

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EXPERIMENT NO.- 2

AIM: Determining the crystal structure & lattice parameters of the material from X-ray diffraction patterns

OBJECTIVES :

- a) Understand the diffraction phenomena and Bragg's law.
- b) Understand use of X-ray diffraction for material characterization.
- c) Understand the procedure for indexing the diffraction patterns & calculating the lattice parameters.

EXPERIMENTAL SETUP :

The diffraction patterns are obtained from X-ray diffractometer (make: Bruker; Model: Discover) using Cu-K α source. Prior to testing, samples are polished using the standard metallurgical sample preparation techniques.

CONSUMABLE REQUIRED:

Nickel & mild steel sample.

THEORY:

Behaviour of the material depends upon the crystal lattice defined by the arrangement of atoms in lattice structure. Therefore it is important to study and determine the shape and size of the lattice structure in order to understand the associated properties. X-ray diffraction (XRD) is a sophisticated technique which helps us

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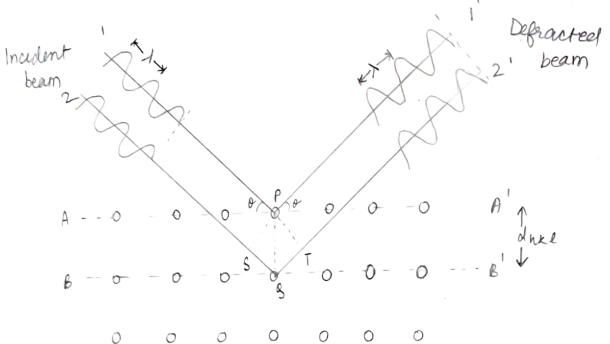


Figure 1: Diffraction of X-rays by planes of atoms (A-A' & B-B')

determine the crystal structure of the material in addition of details about the composition. Diffraction occurs when a wave encounters a series of regular spaced obstacles capable of scattering the wave. The spacing of the obstacles should be comparable to the wavelength. As such, X-rays are used for studying the material structure because of their high energy and wavelength of the order of the atomic spacing.

In order to obtain desired information, the X-rays should have constructive interference wherein each diffracted beam should be in phase with each other, through mutually reinforcement as shown in Fig. 2 parallel planes of atoms A-A' and B-B' shown in Fig 1 have the same h, k and l Miller indices, separated by the interplanar spacing d_{hkl} . A parallel, monochromatic, and coherent (in-phase) beam of X-rays of wavelength λ is incident on these two planes at an angle θ . Two rays in this beam, labelled 1 and 2, are scattered by atoms P and Q. Constructive interference of the scattered rays 1 and 2 occurs also at an angle θ to the planes if the path length difference between $1-P-1'$ and $2-Q-2'$ (i.e. $SQ+QT$) is equal to a whole number, n of wavelengths. Thus, the condition for diffraction is

$$n\lambda = SQ + QT \quad - (1)$$

On substituting the values for SQ and QT from Fig 1, it results in

$$n\lambda = d_{hkl} \sin \theta + d_{hkl} \sin \theta = 2d_{hkl} \sin \theta \quad - (2)$$

Eq. (3) is known as Bragg's law and n is the order of reflection which may be any integer ($1, 2, 3, \dots$) consistent with $\sin \theta$ not

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Table 1: Relation between crystal structure and the square sum of miller indices.

Crystal structure	Value of sum $s = h^2 + k^2 + l^2$
Simple Cubic	1, 2, 3, 4, 5, 6, 8, 9, 10, 11, 12, 13, 14, 16, ...
BCC	2, 4, 6, 8, 10, 12, 14, 16, ...
FCC	3, 4, 8, 11, 12, 16, ...
Diamond Cubic	3, 8, 11, 16, ...

exceeding unity. If Bragg's law is not satisfied, then the interference will be nonconstructive so as to yield a very low-intensity diffracted beam. The magnitude of the distance between 2 adjacent and parallel planes of atoms (i.e. the interplanar spacing d_{hkl}) is a $\frac{1}{s}$ of the Miller indices (h, k and l) as well as the lattice parameter (a). For crystal structures that have cubic symmetry

$$d_{hkl} = \frac{a}{\sqrt{h^2 + k^2 + l^2}}$$

in which a is the lattice parameter (unit cell edge length). Bragg's law, eqⁿ(3) is a necessary but not sufficient condition for diffraction by real crystals. It specifies when diffraction will occur for unit cell having atoms positioned only at cell corners. However, atoms situated at other sites (e.g., face and interior unit cell positions as with FCC and BCC) act as extra scattering centers, which can produce out-of-phase scattering at certain Bragg angles. The net result is the absence of some diffracted beams that, acc. to eqⁿ(3), should be present. Specific sets of crystallographic planes that do not give rise to diffracted beams depend on crystal structure. For the BCC crystal structure, $h+k+l$ must be even if diffraction is to occur, whereas for FCC, h, k and l must all be either odd or even; diffracted beams for all sets of crystallographic planes are present for the simple cubic crystal structure as shown in Table 1. These restrictions are called reflection rules.

For determining the crystal structure, the shape and size of the unit cell are deduced from the angular positions of the

diffraction line. An assumption is first made as to which of the seven crystal systems the unknown structure belongs to, and then, on the basis of this assumption, the current Miller indices are assigned to each reflection.

This step is called "indexing the pattern" and is possible only when the correct choice of crystal system has been made. Once this is done, the shape of the unit cell is known (from the crystal system), and its size is calculable from the positions and Miller indices of the diffraction lines. The number of atoms per unit cell is then computed from the shape & size of the unit cell, the chemical composition of the specimen and its measured density. Finally, the positions of the atoms within the unit cell are deduced from the relative intensities of the diffraction lines. The specimen preparation must ensure random orientation of the individual particles of powder, if the observed relative intensities of the diffraction lines are to have any meaning in terms of crystal structure.

A cubic crystal gives diffraction lines whose $\sin^2\theta$ values satisfy the following eqⁿ, obtained by combining the Bragg Law with the plane-spacing eqⁿ for the cubic system.

$$\frac{\sin^2\theta}{h^2+k^2+l^2} = \frac{\sin^2\theta}{s} = \frac{\lambda^2}{4a^2} \quad - \quad (4)$$

Since the sum $s = (h^2+k^2+l^2)$ is always integral and $\lambda^2/4a^2$ is a constant for any one pattern, the problem of indexing the pattern of a cubic substance is one of finding a set of integers s which will yield a constant quotient when divided one by one

OBSERVATION:

(For Chromium)

S.No	2θ	θ	$\sin^2\theta$	<u>BCC</u>		a	h, k, l
				$\frac{\sin^2\theta}{8}$	s		
1	44.17	22.085	0.141	$\frac{0.141}{2} = 0.07$	0.07	0.29	1, 1, 0
2	64.23	32.115	0.282	$\frac{0.282}{4} = 0.07$	"	0.29	0, 1, 0
3	81.58	40.79	0.426	$\frac{0.426}{6} = 0.07$	"	0.29	1, 1, 2
4	96.91	48.455	0.56	$\frac{0.56}{8} = 0.07$	"	0.29	2, 2, 0
5	113.84	56.92	0.702	$\frac{0.702}{10} = 0.07$	"	0.29	3, 0, 1
6	133.20	66.6	0.842	$\frac{0.842}{12} = 0.07$	"	0.29	2, 2, 2

 \therefore It is a BCC structure

$$\frac{\lambda^2}{4a^2} = 0.07 \quad \therefore a = 0.29 \text{ nm}$$

The crystal structure of Cr is BCC.The lattice parameter of Cr is 0.29 nm.

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CONCLUSION:

- The crystal structure of Nickel is FCC.
- The crystal structure of mild steel is BCC.
- The lattice parameter of Nickel is 0.356 nm and that of mild steel is 0.295 nm.

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EXPERIMENT - 3

AIM: Study the effect of plastic deformation on microstructure & mechanical properties.

OBJECTIVES:

- Perform plastic deformation of the material
- Understand effect of plastic deformation on grain size
- Study the influence of plastic deformation on hardness.

EXPERIMENTAL SET-UP :

Fresh bar of mild steel are cut using abrasive cut-off machine & machined on universal milling machine. Further, samples are sectioned using abrasive cut off machine, hot mounted & polished manually & using belt grinder and double disc polishing machine. Finally, sample are etched using recommended etchant and microstructure is studied using metallurgical microscope. Hardness of the processed & unprocessed mild steel will be performed using Microhardness tester.

CONSUMABLE REQUIRED:

Mild steel, tungsten carbide tool, abrasive paper of different grit sizes, velvet cloth, diamond/alumina paste, and chemicals.

THEORY:

The properties of some materials are significantly influenced by

the presence of imperfections. Permanent deformation of the materials (metals and alloys) require generation and presence of defects such as dislocations. A dislocation is a linear or one-dimensional defect around which some of the atoms are misaligned. Macroscopic plastic deformation simply corresponds to permanent deformation that results from the movement of dislocations, or slip, in response to an applied shear stress. The properties of the materials can be effectively tuned through effective modulation of the defects. During plastic deformation, the number of dislocations increases dramatically. Further, the size of the grains, or average grain diameter, in a polycrystalline materials also influences the properties. Adjacent grains normally have different crystallographic orientations and, of course, a common grain boundary. The grain boundary acts as a barrier to dislocation motion as it must change its direction of motion. This becomes more difficult as the crystallographic misorientation increases. As a result, dislocations tend to pile-up at grain boundaries.

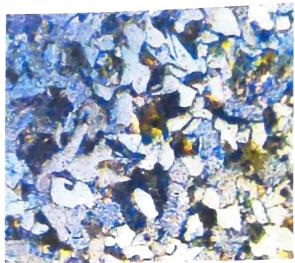
Plastic deformation through friction stir processing is one such processing technique through which properties of the materials can be effectively improved. In friction stir processing (FSP), material is deformed at very high strain rates, which leads to rise in temperature in the processed zone due to frictional heat. As the results, material is severely deformed at high temperature. Consequently, the microstructure of the processed material gets refined, improving its mechanical properties. In FSP, a tool having rotary motion under high axial load is

plunged into the material to be processed. This produces high strain rates and temperature which causes the material to flow around the rotating tool. Alongside, tool is also given a linear motion as a result of which a processed zone is formed having width equal to the diameter of the tool. This refinement can be restricted to the surface or could be extended to process whole of the bulk material depending upon the size & shape of the processing tool.

PROCEDURE:

- Carefully hold the metal bar on the abrasive cut-off machine and cut a sample of desired dimensions.
- Place the metal bar on milling machine table & firmly clamp it using the fixture.
- Machine the top surface using the required end-mill cutter & ensure flat surface is obtained.
- Hold the FSP tool in the adaptor of the milling machine & attached the same in the machine spindle.
- Set the spindle rotation & linear speed.
- Take the reference off-set from the sample surface.
- Turn on the machine and slowly plunge into the sample to the desired depth.
- Start the transverse motion once the required depth is achieved.
- Continue the transverse motion to the desired length.
- Lift the tool up away from the sample surface, and turn-off the machine.
- Unclamp the sample from the machine bed after it is cooled.

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Unprocessed



Processed

Microstructure of processed & unprocessed samples.

- Cut samples of required size from the processed region using abrasive cut-off machine.
- Mount the sample in the cross sectioned position using Bakelite powder in a hot mounting machine set at desired temperature and pressure.
- Polish the sample using standard metallurgical sample preparation technique.
- Clean the sample using distilled water & dry in the air.
- Measure the micro-hardness of the sample in the processed and unprocessed zones.
- Prepare suitable etchant as per the list provided.
- Use a cotton swab and/or a petri dish for the etching. Gently swab the surface of your sample with the etchant for limited time and quantity.
- Wash the sample again with distilled water & examine the sample under optical microscope. Several etching cycle might be required to reveal the microstructure.
- If the sample is over-etched, repeat the final polishing step & re-etch for a shorter time. Samples do be examined at high magnification generally require shorter etching times than those to be viewed at lower magnifications.

OBSERVATION :

The hardness of the unprocessed sample is 99.1 HV.

The hardness of the processed sample is 231.2 HV.

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CONCLUSION:

The hardness of the material is increased by 2.33 times after plastic deformation.

PRECAUTIONS:

- Don't wear loose clothes while operating the machine.
- Always wear shoes while operating the machine.
- Don't touch the rotating spindle.
- Wear goggles while operating the machine.
- Allow the tool & work piece to cool before removing them from the milling machine.
- Make sure that pressure in the hot mounting machine is maintained.
- Check for any leakage in the hot mounting press.
- Be careful to not hurt fingers while grinding & polishing.
- Do not operate the microscope and any machine with proper supervision.
- Keep the work areas clean.
- Turn the power off and cover the wheels when you are through. Clean up any spilled water & wipe up the countertop.
- Use goggles and gloves while handling chemicals.

Certificate

Name: ADYA SINGHAL.....

Roll No.: 2410110D27..... Class: P1 CSE..... Exam No.:

Institution:

This is certified to be the bonafide work of the student in
the Laboratory during the academic
year 20...../ 20.....

Number of practicals certified out of
in the subject of

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