

Manufacturing Processes Lab - II

(MMS751)

Laboratory Manual

B. Tech. in Metallurgical and Materials Engineering

Course Instructors

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SAFETY AND GENERAL INSTRUCTIONS

INSTRUCTIONS:

1. Wash your hands before entering the lab and wear protective clothing, such as lab coats or aprons, gloves, and eyewear. Be sure that your work area should be clean and dry.
2. Never attempt to operate any equipment without prior instruction.
3. Work in the laboratory only when a lab instructor is present and only on authorized experiments.
4. Do not bring any unnecessary items into the lab. Do not place any personal items (purses, book bags, coats, umbrellas, etc.) on the lab table or at your feet.
5. Make sure all apparatus is supported and squarely situated on the table.
6. Do not put anything in your mouth while in the lab. Never eat, chew gum, drink, taste chemicals, mouth pipette, lick labels, smoke, or store food or drink in the lab. DO NOT bring food and beverages into the laboratory.

POWDER HANDLING PRECAUTIONS:

- Certain powders of metals and metallic compounds can have harmful effects on users exposed to these powders. Powder handling requires proper safety precautions and cleanliness.
- Respiratory diseases or other dysfunctions can affect persons exposed to metallic dust. The particle size and the specific gravity of the material largely determine the deposition site for a respiration particle.
- Coarse particles are trapped on the precious membranes and do not reach the lungs; fine particles can reach the lungs and maybe dissolve into the body.
- However, special care is needed when using elemental powders as alloying elements (e.g., Cr, Ni, As, Cd). Contact with these metals in the powdered state should be minimized whenever possible.
- Another hazard with aluminum-based powders and several others is their thermal instability in the presence of oxygen. Aluminum powders in a finely divided state are pyrophoric (burn-in air) and potentially explosive. Aluminum powders require very little oxygen content in the atmosphere (less than 3 %), have a low ignition temperature (less than 600 °C), and very low explosive limit (20-50 g/m³).

Explosion and fire prevention is based on the evaluation of:

- Potential ignition sources (e.g., Electrostatic discharge);
- Dust cloud generators;
- Gaseous atmosphere composition.
- If a fire occurs in a metal powder, the fire should be approached with extreme caution. The best way to attack burning metal powder is with special dry-type fire extinguishing agents. Water should never be applied to burning metal powders.

POWDER COMPACTION PRECAUTIONS:

- Never place your hand or any part of your body in compaction press.
- Never operate the press when it is not working correctly. Stop working and immediately inform your instructor.
- Never approach press up to its limit or beyond. Always keep the load at least 5 tons less than its upper limit for safe operation.
- Clean pressing area after each job.
- Never try to change die or place your finger without first releasing the pressure.

SINTERING PRECAUTIONS:

- Immediately inform your instructor if atmospheric gas is found to be leaking.
- Never try to pick parts with bare hands when they are hot after sintering.
- Do not operate the sintering furnace once the sintering cycle has started.

EXPERIMENT NO 1

Demonstration of ball milling, compaction unit, dynamic light scattering technique and tube furnace

This laboratory aims to learn the science and technological aspects of powder metallurgy that involve synthesis and characterization of powder, compaction of powders, and sintering of powder compacts. Chemical reduction and ball milling will synthesize powder characterized under X-Ray Diffraction (XRD) and field emission scanning electron microscopy (FESEM). Compaction will be carried out using steel die and punch. Finally, sintering will be performed under a high-temperature tube furnace. Phase, microstructure, and hardness of the sintered products will be investigated by XRD, FESEM, and Vickers hardness, respectively.

Background

The technology of pressing metal powders into a specific shape is not new. Older civilizations practiced art in prehistoric times. As bear witness the iron pillar in Delhi, certain Egyptian implements and articles of precious metals made by the Incas. Modern powder metallurgy (P/M) technology commenced in the 1920s with the production of tungsten carbides and the mass production of porous bronze bushes for bearings. The modern era of powder metallurgy is traced to the development of tungsten filament.

Powder metallurgy is a process by which fine powdered materials are blended, pressed into the desired shape, and then heated to produce a solid object.

The powder is defined as a finely divided solid matter characterized by a small size less than 1 mm in size.

Advantages of Powder Metallurgy

The technical and commercial advantages of producing parts from powder can be summarized as below:

- Production to near net shape
- Few or no secondary operations
- High material utilisation from low levels of 'in process scrap'
- Homogeneous powder, and hence part, chemical composition due to absence of gross solidification segregation and uniform pre-alloyed powder particle composition
- Unique compositions and structures possible as there is no melting e.g. introduction of specific particles to give special properties such as silica and graphite in brake pads, and porosity in bearings for oil retention
- Non-equilibrium compositions possible e.g. copper-chromium alloys

Disadvantages and Limitations of Powder Metallurgy

Inevitably there are some limitations including:

- Costs of powder production
- Limitations on the shapes and features that can be generated, e.g., the process cannot produce re-entrant angles by fixed die pressing or radial holes in vertically pressed cylinders
- The size will always change on sintering. This can usually be predicted as it depends on several factors, including as pressed density that can be controlled.
- Potential workforce health problems from atmospheric contamination of the workplace.

Lab Equipment:

- Wet chemical powder synthesis apparatus
- Milling equipment: Planetary ball mill
- Compaction unit
- Sintering Furnace (Tube) with accessories
- Density measuring kit and electronic balance

Ball mill (Retsch PM 200 Planetary Ball mill)



Figure1: Planetary Ball Mill: The PM 200 is a convenient bench top model with two grinding stations.

Planetary Ball Mills are used wherever the highest degree of fineness is required. Apart from the classical mixing and size reduction processes, the mills also meet all the technical requirements for colloidal grinding and have the energy input necessary for mechanical alloying processes. The extremely high centrifugal forces of a planetary ball mill result in very high pulverization energy and short grinding times.

The grinding jars are arranged eccentrically on the sun wheel of the planetary ball mill. The direction of movement of the sun wheel is opposite to that of the grinding jars.

The grinding balls in the grinding jars are subjected to superimposed rotational movements, the so-called Coriolis forces. The difference in speeds between the balls and grinding jars produces an interaction between frictional and impact forces, releasing high dynamic energies. The interplay between these forces makes the high and very effective degree of size reduction of the planetary ball mill.

Dynamic light scattering (DLS)

Dynamic Light Scattering (sometimes referred to as Photon Correlation Spectroscopy or Quasi-Elastic Light Scattering) is a technique for measuring the size of particles typically in the sub micron region.

DLS measures Brownian motion and relates this to the size of the particles. Brownian motion is the random movement of particles due to the bombardment by the solvent molecules that surround them. Normally DLS is concerned with measurement of particles suspended within a liquid.

The velocity of the Brownian motion is defined by a property known as the translational diffusion coefficient (usually given the symbol, D). The size of a particle is calculated from the translational diffusion coefficient by using the Stokes- Einstein equation:

$$d(H) = \frac{kT}{3\pi\eta D}$$

where:-

$d(H)$ = hydrodynamic diameter

D = translational diffusion coefficient

k = Boltzmann's constant

T = absolute temperature

η = viscosity



Figure 2: Zetasizer Nano ZS90

In dynamic light scattering, the speed at which the particles are diffusing due to Brownian motion is measured. Measuring the rate at which the intensity of the scattered light fluctuates when detected using a suitable optical arrangement does this.

Powder compaction unit

Dies and punches are thoroughly cleaned with the help of tissue paper and Acetone to avoid the sticking of die at later stages of experiment. Powder sample of required amount is taken and put into die. Some drops of binder are added to the powder and the remaining parts of die are assembled. This die containing the sample is now placed over the manual hydraulic press for compaction of the sample. This sample is exposed to a pressure and held for around one minute. After a minute the pressure is removed and die is taken out. The base of the die is removed and is replaced with cotton. Then the die containing sample and cotton is again put in hydraulic press and is pressurised slowly and manually till the sample comes out of the die.

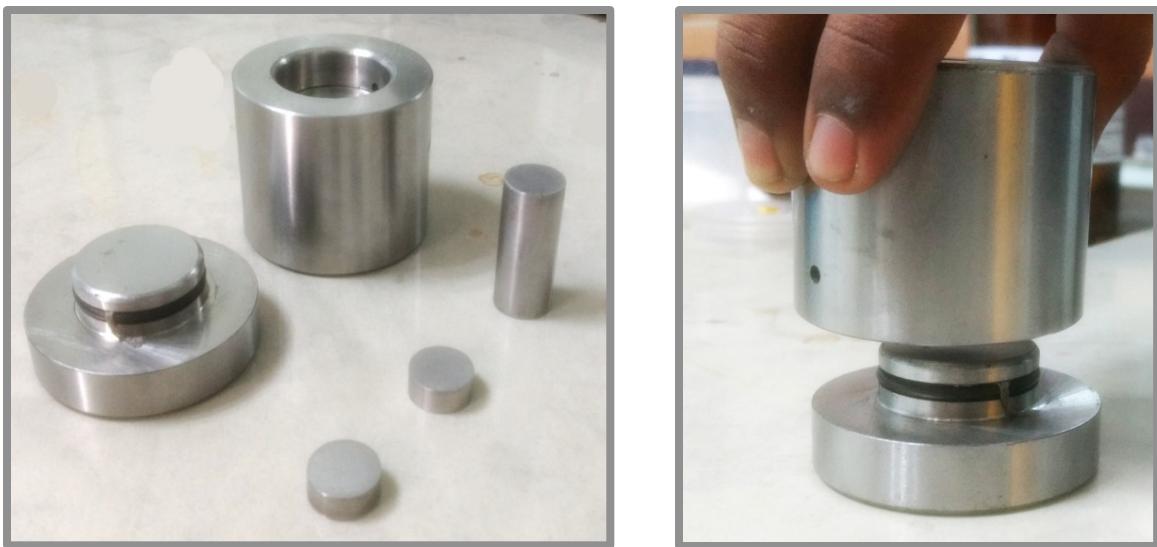


Figure 3: (a) Die, punch and support and (b) die-punch assembly



Figure 4: Manual hydraulic compaction machine

High temperature Tube Furnace



Figure 5: High temperature tube furnace

Samples are sintered in High Temperature Tube Furnace ('Resistance Heated, Maximum Teamperature: 1700⁰ C, Working Temperature: 1600 ⁰C, Heating Element: Super Kanthal, Power: 400W, Manufacturer: Nascar and Co., Model Number: EN170TF "Electroheat"'). Sample is placed inside the furnace and the furnace is turned on.

The furnace shows two temperatures: temperature of the sample and temperature of the furnace. Close monitoring of both temperatures was done to avoid the temperature difference more than 20 °C between the sample and furnace. After sintering the samples are taken out from the furnace.

Lab Deliverables:

1. List and briefly write about different powder production methods.
2. List and briefly write about different types of mills.
3. Briefly describe the basic steps involved in the powder metallurgy route to get the final component from powders.
4. What are the characteristics of powders through which we classify them?
5. State and explain the working principle of (i) die compaction and (ii) density measurement by Archimedes' principle.
6. List and briefly describe different types of heating elements and their use.

EXPERIMENT NO 2

Synthesis of nano powders by Chemical reduction

Objective: To synthesize cuprous oxide nanoparticles (NPs) through chemical reduction method

Basic Theory: Interest in cuprous oxide (Cu_2O) nanoparticles with defined size and shape arise from its useful optical and electronic properties. Several researchers have synthesized a variety of micro and nanostructures of Cu_2O , such as nanocubes, octahedra, nanocages, hollow spheres, nanowires, and other highly symmetrical structures. A number of documents and reports on the preparation methods of nano- Cu_2O exist due to its broad application prospects. Chemical reduction in aqueous media is a standard synthesis route for the production of silver, copper-silver core-shell or Cu_2O nanoparticle.

Raw Materials:

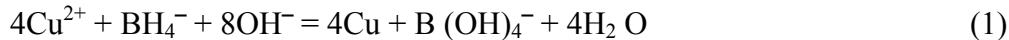
Reagents used for nanoparticle synthesis are copper sulfate pentahydrate, $CuSO_4 \cdot 5H_2O$, of >99% purity (Merck), polyethylene glycol 6000 (PEG 6000dMerck), sodium borohydride ($NaBH_4$ ->95%, Merck), ascorbic acid (>99%, Merck) and sodium hydroxide $NaOH$ (>97%, Merck). Chemicals are analytical grade with high purity. Polyethylene glycol (PEG) is used as a capping agent, whereas the primary reducing agent is sodium borohydride ($NaBH_4$). Ascorbic acid is added as an antioxidant, and the role of sodium hydroxide ($NaOH$) is to adjust the pH of the solution.

Procedure:

1. A blue solution is prepared by dissolving 1.25 g of $CuSO_4 \cdot 5H_2O$ in 50 ml deionized water, and the solution is stirred vigorously.
2. Then 6 g of PEG is dissolved in 50 ml deionized water, and the solution is added in the copper sulfate solution. As a result, a white color solution is obtained.
3. Next, ascorbic acid (0.9 g) and $NaOH$ (0.4 g) are dissolved in 100 ml deionized water, and the resulting solution is added to the previous solution.
4. Finally, an aqueous solution (50 ml) of $NaBH_4$ (0.4 g) is prepared and mixed to the solution under constant stirring. As a result an aqueous dark red solution is prepared.
5. The dark red solution is left to cool for the whole night. The next day, the color of the solution changes to yellow, and it is centrifuged.
6. Finally, the precipitate is used for further characterization.

Synthesis mechanism:

In aqueous solution, the reaction takes place as



- ◆ Initially, the Cu^{2+} have been compounded with PEG
- ◆ Then Cu^{2+} reacted with BH_4^- ion, and Cu particles have been generated.
- ◆ The solution has been kept under ambient atmosphere and the oxidation has been qualitatively monitored with time by observation its color change.
- ◆ Cu oxidized to Cu_2O by oxygen in air and colloidal suspension of Cu_2O nanoparticles form.

Lab Deliverables:

1. What are the advantages and disadvantages of wet-chemical methods?
2. List different methods to synthesize nano powders.
3. How to get different particle size through wet chemical methods?
4. Distinguish heterogeneous and homogeneous precipitation.

EXPERIMENT NO 3

Particle reduction by Ball milling

Objective: To achieve particle reduction of a ceramic powder using planetary ball mill.

Basic Theory:

Milling, mechanical impaction using hard balls, is a classic approach to fabricating powders from brittle materials. A jar mill, such as diagrammed in figure 3.1, consists of a cylindrical jar filled with balls and the material to be milled. As the jar rotates, the balls continuously collide with the material, crushing it into powder. The impact stress required to fracture a brittle material in milling relates to the defect structure and sensitivity to crack propagation. A balance between coalescence and fragmentation is achieved during milling, which leads to a rather stable average particle size.

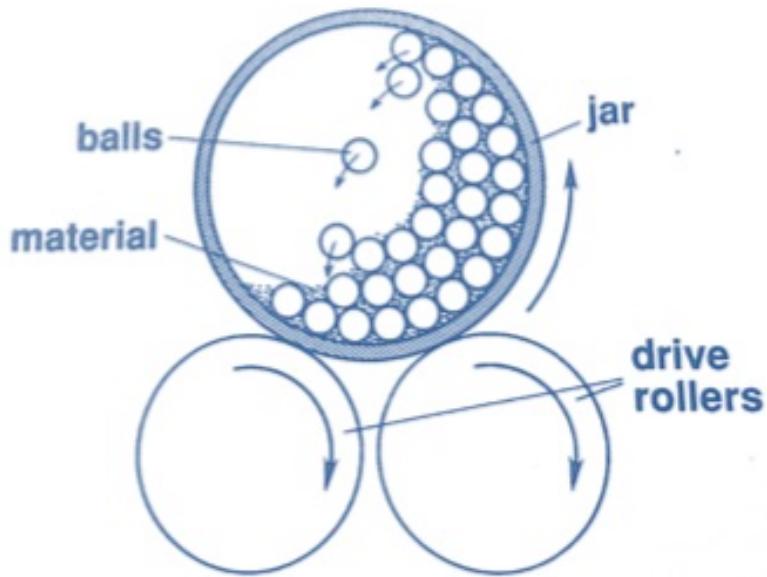


Figure 3.1: A view of the action in a jar mill. The jar is rotated on its side and the impact of the failing balls grinds the material into a powder.

Planetary Ball Mills are used wherever the highest degree of fineness is required. Apart from the classical mixing and size reduction processes, the mills also meet all the technical requirements for colloidal grinding and have the energy input necessary for mechanical alloying processes. The extremely high centrifugal forces of the Planetary Ball Mills result in very high pulverization energy and therefore short grinding times.

Planetary ball mill consists of at least one grinding jar, which is arranged eccentrically on a so-called sun wheel. The direction of movement of the sun wheel is opposite to that of

the grinding jars. The difference in speeds between the balls and grinding jars produces an interaction between frictional and impact forces, which releases high dynamic energies. The interplay between these forces produces the high and very effective degree of size reduction of the planetary ball mill.

Both wet and dry grinding is possible in planetary ball mill. For ultrafine milling wet grinding is preferred because in dry grinding there is a problem of agglomeration.

Precautions:

1. Make sure that milling cup is clamped before starting with the experiment.
2. All cups have same gross weight.
3. While feeding, opposite cups should be always fed in two stations planetary ball mill.
4. Precautionary measures should be employed while unloading the samples.

Apparatus used:

Planetary ball mill (PM 200), Al₂O₃ powder, grinding media (vials and balls), wetting agent (if required).

Procedure:

1. Fill the material and the grinding media into the jar
2. Insert the grinding jar into grinding jar holder
3. Insert the spider into the brackets and clamp it by turning the red sleeve up
4. Set the parameters viz. milling time, speed, interval time and pause time and start the milling process (The PM 200 is started with the preselected milling time, speed and reversal of direction of rotation, the machine rotates with set interval time in one direction, comes to stop. After pause time has elapsed the machine starts again.)
5. Run the machine for one minute and observe it for abnormal vibration and noise (If the loading is unbalanced the machine can generate undesirable sounds and vibrations.) if found, switch off the machine and check the arrangement (Gross weight of cups, any loosen part etc.
6. After milling time is completed allow sufficient time to reduce temperature to room temperature.
7. Open the milling chamber hood after pressing the open button and unlock.
8. Carefully take the grinding jars and take the powder out and clean it properly.

Lab Deliverables:

1. What is the effect of the rotational speed on grinding?
2. What is the effect of ball to charge ratio on grinding?
3. What is the function of a wetting agent on grinding?
4. List four types of grinding media.

EXPERIMENT NO 4

Characterization of nano and milled powders

Objective: To identify phases present and study the microstructural features of synthesized nano and milled ceramic powders.

Basic Theory:

The success of any powder metallurgical process depends to a great extent on the complete characterization and control of the metal powders. The method of powder production influences particle chemistry and structure, apart from the precise nature of particle size distribution. These properties also influence the behaviour of the powder during compaction and sintering, and the composition, structure and properties of the sintered material.

The larger depth of field in the scanning electron microscopy (SEM) is a distinct advantage, especially since it shows surface topography and can provide x-rays for compositional analysis. Important microstructure information can be gained by SEM analysis. Nucleation sites, contamination, cooling rate, grain size, and segregation can be assessed from SEM examinations.

X-ray diffraction (XRD) analysis

X-ray diffraction (XRD) studies are carried out for the purpose of phase identification, crystallite size and lattice strain of the powder samples. These studies are made by using Panalytical X'Pert PRO diffractometer as shown in this figure, operated with Cu-K_α radiation (having wavelength =1.54 Å) on X-ray diffractometer. The X-ray source is operated with Cu-K_α radiation at a voltage of 40 kV and current of 20 mA. The diffraction angle was varied in the range of 10-90° and the range of 2θ was selected such that all the major peaks of the expected phases to be present in the powder are covered. Subsequently, the XRD patterns were analyzed with the help of JCPDS (Joint Committee on the Powder Diffraction Standards) data file to identify the crystal structure of the constituent phases.

Electron microscopes have certain advantages over optical microscopes:

The biggest advantage is that they have a higher resolution and are therefore also able of a higher magnification (up to 2 million times). Light microscopes can show a useful magnification only up to 1000-2000 times. This is a physical limit imposed by the wavelength of the light. Electron microscopes therefore allow for the visualization of structures that would normally be not visible by optical microscopy.

Depending on the type of electron microscope, it is possible to view the three-dimensional external shape of an object (Scanning Electron Microscope, SEM).

In scanning electron microscopy (SEM), due to the nature of electrons, electron microscopes have a greater depth of field compared to light microscopes. The higher resolution may also give the human eye the subjective impression of a higher depth of field.

Electron microscopes have a range of disadvantages as well:

They are extremely expensive.

Sample preparation is often much more elaborate. It is often necessary to coat the specimen with a very thin layer of metal (such as gold). The metal is able to reflect the electrons.

The sample must be completely dry.

It is not possible to observe color. Electrons do not possess a color. The image is only black/white. Sometimes the image is colored artificially to give a better visual impression.

They require more training and experience in identifying artifacts that may have been introduced during the sample preparation process.

The energy of the electron beam is very high. The sample is therefore exposed to high radiation, and therefore not able to live.

Maintenance costs are high.

Equipment/ Raw Materials:

The raw materials consists of chemically synthesized Cu₂O nano particles and ball milled Al₂O₃ powders

Other accessories/chemicals/equipment:

- X-Ray Diffractometer (Panalytical X'Pert PRO)
- Field emission scanning electron microscope
- Pin Stubs
- Carbon tape

Procedure:

Powder sample preparation for scanning electron microscope:

1. Properly clean the pin stub sample holder using isopropyl alcohol or ethanol
2. Place a carbon tape on top of the pin stub
3. Spray very small amount powder gently on the carbon tape and ensure no agglomeration.

Lab Deliverables:

1. Draw an interaction volume along with different signals that are emitted when electron beam interacts with the specimen in a scanning electron microscope.
2. Discuss the origin of signals and their use in the following modes of operation in SEM:
 - a. Secondary Electron Imaging
 - b. Backscattered Electron Imaging
 - c. Energy Dispersive X-ray analysis
3. List various kinds of microstructural features observed in metals, alloys and composites. List the microstructural features that are observed in the current laboratory experiment.

EXPERIMENT NO 5

Particle size analysis

Objective: To identify phases present and study the microstructural features of synthesized nano and milled ceramic powders.

Basic Theory:

The success of any powder metallurgical process depends to a great extent on the complete characterization and control of the metal powders. The method of powder production influences particle chemistry and structure, apart from the precise nature of particle size distribution. These properties also influence the behaviour of the powder during compaction and sintering, and the composition, structure and properties of the sintered material.

The microstructure of the crystalline powder has a significant influence on the behaviour of powder during compaction and sintering and on the properties of the final product. Fine grain size is always desirable, as it improves the mechanical properties apart from the sinterability and the uniformity of dimensional changes.

The grain size can be dependent on the powder particle size. The particular powder production method, e.g. rapidly cooled powder, would naturally give rise to small particles and also small grain sizes.

It can be thus concluded that the particle size analysis is necessary for a complete characterization instead of an average value or even maximum or minimum values of size.

There are a number of particle size measurement techniques available in powder metallurgy, each having their own limitations. This table classifies some of the common methods of particle size determination and their limits of applicability.

X-ray diffraction (XRD) analysis

The X-ray analysis is used for estimating crystallite size of nanomaterials and therefore it is extensively employed in the present work.

Diffraction line broadening for crystalline materials has several causes, including strain and small crystal size.

The destructive interference from the crystal is not complete at angles slightly off Bragg condition

$$\lambda = 2d \sin \theta$$

Where λ is the x-ray wavelength, d is the interplanar spacing and θ is the diffraction angle.

The width of the diffraction peak increases as the thickness of the diffracting crystal decreases.

The most useful approach to particle size analysis using x-rays relies on the peak width at half of the maximum intensity as shown in this figure 1.

Peak broadening at that intensity depends, in part, on the number of diffracting planes in the crystal.

The Scherrer formula gives the crystal size D in terms of the broadening B , diffraction angle θ and x-ray wavelength λ as

$$D = \frac{0.9\lambda}{B \cos \theta}$$

The wider the diffraction peak, the smaller is the particle size.

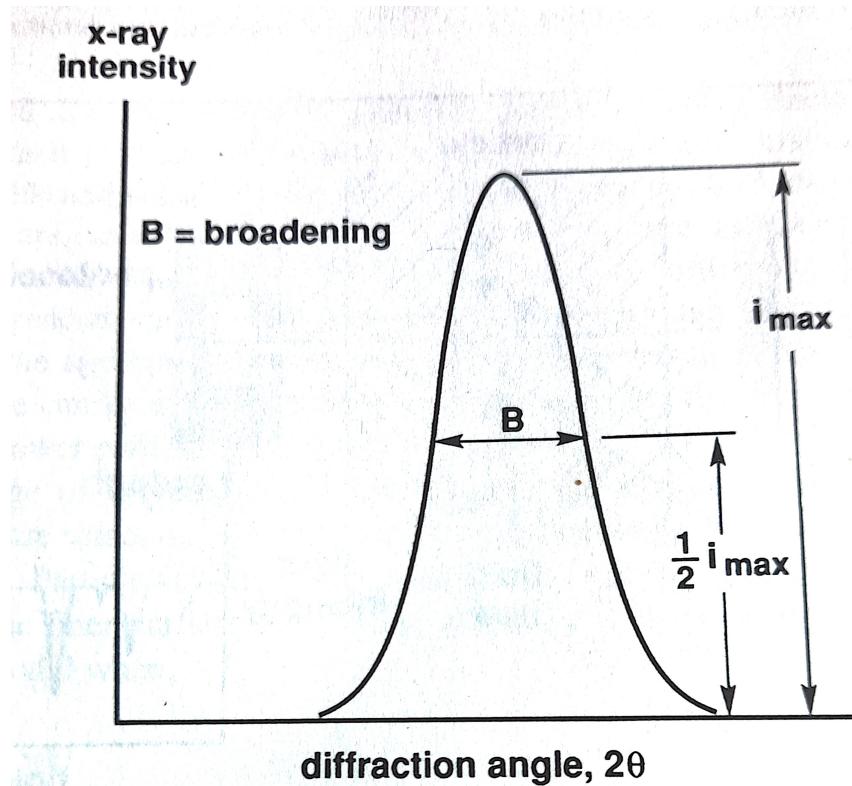


Figure1: X-Ray peak broadening is measured in terms of the width B at an intensity one half of the maximum.

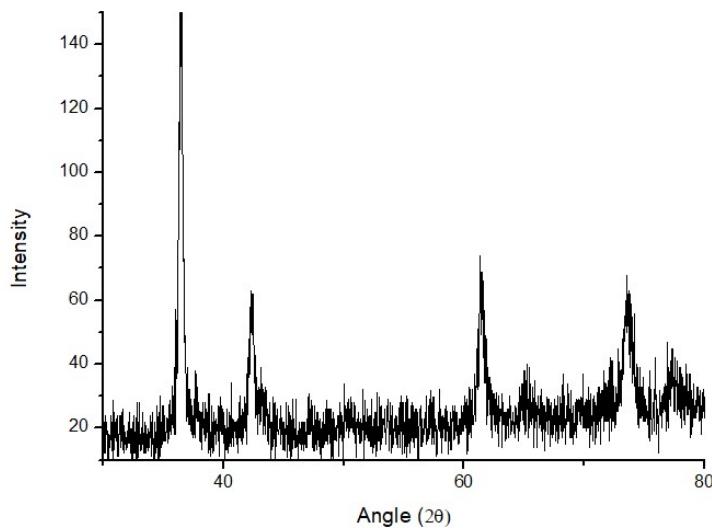


Figure 2: Typical XRD pattern of Cu₂O nano particle synthesized by chemical reduction method

Table1: The FWHM values of XRD pattern of Cu₂O nano particle

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
23.480850	14.143000	0.492000	3.78880	11.65
36.446790	121.376000	0.295200	2.46524	100.00
42.458020	39.839960	0.246000	2.12909	32.82
61.317050	43.526470	0.246000	1.51188	35.86
73.574810	30.597110	0.492000	1.28737	25.21
77.350730	10.451600	0.787200	1.23368	8.61

Electron microscopy analysis

A widely applied technique for particle sizing uses the ability of the eye to rapidly size dispersed particles in a microscope. Microscopy is reasonably accurate, the tedium of sizing statistically significant quantities of particles has led to use of authentic image analyzers. The image for analysis is generated by optical, scanning and transmission electron microscopes. The instrument choice depends on the particle size; however, the larger depth of field in the SEM is a distinct advantage, especially since it shows surface topography and can provide x-rays for compositional analysis.

The shape, size, morphology, distribution and microstructure of the powders are examined using a field emission scanning electron microscope (FESEM, Figure 3) coupled with energy dispersive X-ray (EDX) microanalyser. The SEM is operated at an acceleration voltage of 20 kV. The micrograph images were taken in the secondary (SE) and back scattered (BSE) modes as per the requirements.

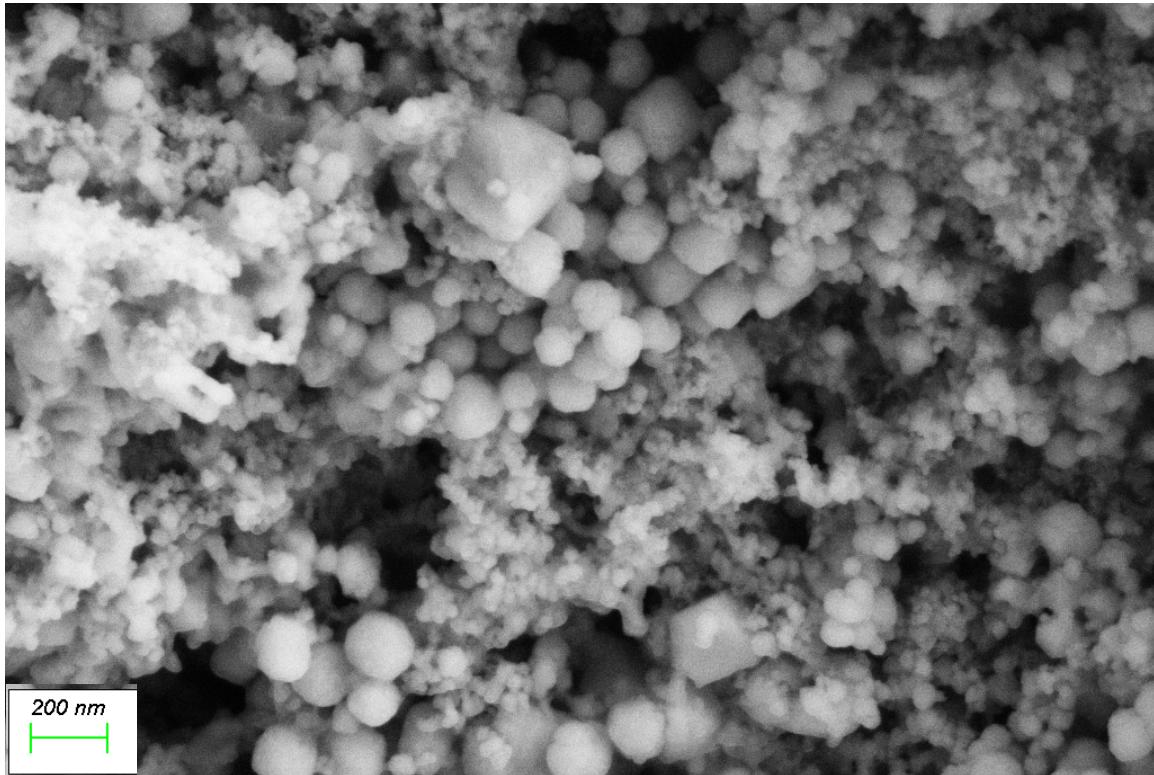


Figure 3: Typical FESEM image of the Cu₂O nano particles

Equipment/ Raw Materials:

The raw materials consists of chemically synthesized Cu₂O nano particles

Other accessories/chemicals/equipment:

- X-Ray Diffractometer (Panalytical X'Pert PRO)
- Field emission scanning electron microscope
- Pin Stubs
- Carbon tape

Procedure:

Powder sample preparation for scanning electron microscope:

1. Properly clean the pin stub sample holder using isopropyl alcohol or ethanol
2. Place a carbon tape on top of the pin stub
3. Spray very small amount powder gently on the carbon tape and ensure no agglomeration.

Lab Deliverables:

1. List and briefly write about different particle size analysis techniques.
2. What are the advantages and disadvantages of particle size analysis by microscopy?
3. Briefly explain particle size analysis by DLS measurement.
4. Determine the crystallite size and particle size of Cu₂O nano particle synthesized by chemical reduction method.

EXPERIMENT NO 6

Conventional die compaction of powders

Objective: To study the basic powder compaction technique to achieve compact properties with minimal wall friction.

Basic Theory:

Powder metallurgical processing is widely used for producing near-net shape engineering components. Most applications for powder metallurgy dictate that high densities can be attained in the final product. Powders that exhibit good sintering densification can be shaped using low pressures, often with the aid of an organic lubricant. Compaction relies on an external source for deforming the powders into a high density component that approaches the final geometry.

The compaction of metal powders has the following major functions:

- (a) to consolidate the powder into desired shape
- (b) to impart, to as high a degree as possible, the desired final dimensions with due consideration to any dimensional changes resulting from sintering.
- (c) to impart the desired level and type of porosity.
- (d) to impart adequate strength for subsequent handling.

Several approaches exist for achieving these goals. In general the techniques can be categorized as

- (a) continuous vs discontinuous process,
- (b) pressures – high vs low,
- (c) compaction velocity – high vs low
- (d) temperature -room to elevated temperature;
- (e) uniaxial vs hydrostatic pressures.

Die compaction represents the most widely used method and is considered as the conventional technique.

This involves rigid dies and special mechanical or hydraulic presses.

Densities of up to 90 % of full density can be achieved following the compaction cycle, the duration of which may be of the order of just a few seconds for very small parts.

The friction between the powder and die wall and between individual powder particles hinders the transmission of pressure.

A high uniformity in green parts can be achieved depending on:

- the kind of compacting technique
- the type of tools
- the materials to be pressed and the lubricant.

The compacting techniques used may be characterised by references to the movement of the individual tool elements – upper punch, lower punch and die relative to one another.

Pressing within fixed dies can be divided into:

- Single action pressing
- Double action pressing

In the **Single action pressing** the lower punch and the die are both stationary. The pressing operation is carried out solely by the upper punch as it moves into the fixed die. The die wall friction prevents uniform pressure distribution. The compact has a higher density on top than on the bottom.

In the **Double action pressing** type of pressing only the die is stationary in the press. Upper and lower punches advance simultaneously from above and below into the die. The consequence is high density at the top and undersides of the compact.

Compaction pressure can be generated in a simple uni-axial press using mechanical, pneumatic or hydraulic forces. As pressure increases, the particles are packed more tightly thus increasing the green density.

As shown in Figure 1, the initial rate of densification with the application of pressure is high. With continued deformation the slope of the density versus pressure curve decline reflecting particle work hardening.

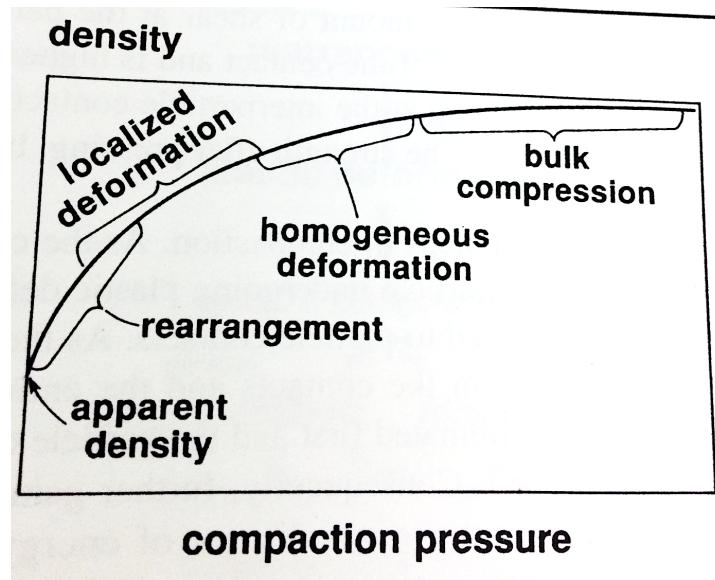


Figure 1: The phenology of powder compaction

The four major mechanisms controlling densification are rearrangement, localized deformation, homogeneous deformation and bulk compression.

As pressure is applied, the first response is rearrangement of the particles with filling of large pores, giving a higher packing coordination. Increasing pressure provides better packing and leads to decreasing porosity with the formation of new particle contacts. The point contacts undergo elastic deformation, and at all points in the compaction cycle a residual elastic energy is stored in the compact. High pressures increase density by

contact enlargement through plastic deformation. Thus, the pressure causes localized deformation at the contacts, giving work (strain) hardening and allowing new contacts to form as the gaps between particles collapse.

During deformation, cold welding at the interparticle contacts contributes to the development of strength in the compact. The strength after pressing, but before sintering, is termed the **green strength**.

Equipment/ Raw Materials:

Apparatus used in this experiment are powder, lubricant, cotton, steel die-punch set and powder compaction unit.

Lab Deliverables:

1. Write a full lab report based on your observations and results.
2. What are the practical precautions required before beginning powder compaction?
3. Differentiate between the functions of a lubricant and a binder.
4. What is the difference between die wall lubrication and powder lubrication?
5. Discuss advantages and disadvantages of conventional die compaction.
6. Define green density, green strength, compression ratio densification parameter and ejection pressure.
7. What is fill? How do you measure it?

EXPERIMENT NO 7

Solid state sintering

Objective: To study the sintering behavior of alumina powder compact in air environment.

Basic Theory:

Sintering is one of the most important steps in Powder Metallurgy processing. It is the process of consolidating either a loose aggregate of powder or a green compact of the desired composition under controlled conditions of temperature and time.

Sintering may involve:

- (1) single component system (e.g. pure metals and ceramics), where in shrinkage is major factor or
- (2) Multi component system, involving more than one phase, where several processes like solid solution formation and liquid phase formation may also occur in addition to densification.

Major variables in the sintering process are following:

- (1) Sintering Temperature
- (2) Sintering Time and
- (3) Sintering Atmosphere

- ❖ Sintering is the bonding together of particles at high temperature.
 - ❖ Particles are sintered by atomic motions, which eliminate the high surface energy associated with powders.
 - ❖ Smaller particles with high specific surface areas have more energy and sinter faster.
 - ❖ The sintering mechanism describes the path of atomic motion, which produces the mass flow.
 - ❖ For metal powders, the mechanisms are diffusion processes over the surfaces, along the grain boundaries, or through the crystalline lattice.
 - ❖ The structural changes associated with neck growth during sintering depend upon transport mechanisms, which is mainly diffusion process.
 - ❖ Diffusion process is thermally activated, meaning that specific energy is necessary for atomic movement.
-
- Sintering processes are broadly classified as **pressureless** sintering and **pressure assisted** sintering.
 - Pressurization during sintering is most useful in processing materials that are unresponsive to traditional sintering cycles: for example, composites and high temperature intermetallics.

- Most sintering processes, which are performed without an external pressure, are known as pressureless sintering. Pressureless sintering technique is further categorized into solid state sintering and liquid phase sintering.
- Sintering forms solid bonds between particles when they are heated.
- The bonds reduce the surface energy by removing free surfaces with the secondary elimination of grain boundary area via grain growth.
- With extended heating, it is possible to reduce the pore volume, leading to compact shrinkage.
- In many sintering systems dimensional change is undesirable. Structural materials such as silicon nitride, alumina, cemented carbide, silicon carbide and steels are processed to full density by sintering at relatively high temperatures.

Equipment/ Raw Materials:

Apparatus used in this experiment are powder, lubricant, cotton; steel die-punch set powder compaction unit and High Temperature Tube Furnace.

Lab Deliverables:

1. Write a full lab report based on your observations and results.
2. What are the practical precautions required before beginning powder sintering?
3. Weight the sintered compact and determine shrinkage.
4. Write down the name of most common sintering atmosphere.
5. Explain sintering mechanism.

EXPERIMENT NO 8

Liquid phase sintering

Objective: To study the sintering behavior of cobalt alloy powder compact in argon environment.

Basic Theory:

Liquid phase sintering method is getting more and more common, in which the presence of liquid phase during all or part of the sintering cycle of material is used for enhanced densification. There are two variations of the process:

(a) normal liquid phase sintering for which the formation of the liquid phase is associated with one or more components contained in the original green compact.

(b) infiltration of the original green compact with a liquid formed outside the compact during the very early period of sintering. Although simultaneous infiltration and sintering appears to be dominant, infiltration of a previously sintered part is also practised.

During liquid phase sintering three stages ‘rearrangement’ or ‘liquid flow’, ‘accommodation’ or ‘dissolution and reprecipitation’ and ‘coalescence’ or ‘solid phase sintering’ take place. These stages follow in the approximate order of their occurrence, but there may be significant overlapping for any specific system. Figure 6.1 shows the densification stages during liquid phase sintering. With progress in liquid phase sintering the densification kinetics is lowered. Increasing the liquid content up to approximately 35 volume percent aids initial densification. A coarse particle size and a high green density act to offset the favorable effects of the melt. In case the second stage is not effective, the melt will penetrate along the interparticle interfaces and cause particle separation. This will contribute in swelling.

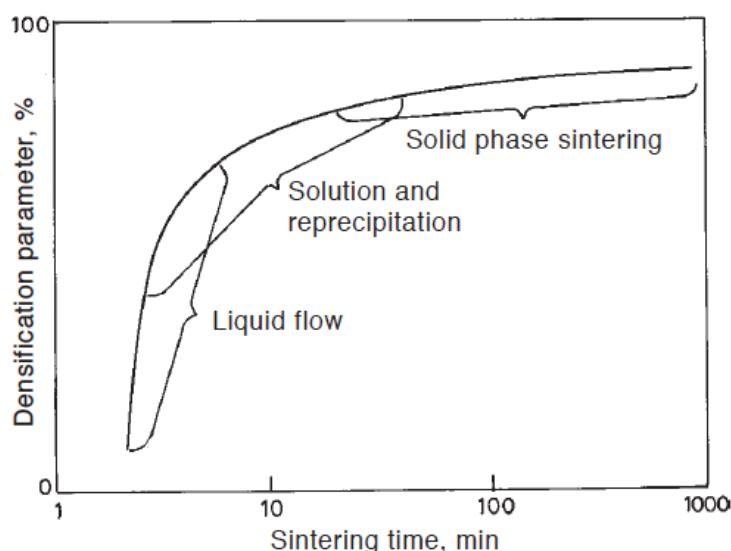


Figure 6.1: The densification stages during liquid phase sintering.

A small dihedral angle inhibits coalescence of neighbouring particles. Application of liquid phase sintering technology is widely made in the field of sintered steels, cemented carbides, heavy alloys, bronzes and silicon nitride systems.

Equipment/ Raw Materials:

Apparatus used in this experiment are powder, lubricant, cotton; steel die-punch set powder compaction unit, compacted sample, high temperature tube furnace, argon gas cylinder, and crucibles/boats.

Procedure:

1. Select high temperature tube furnace.
2. Place the sample in the middle of the furnace using suitable crucibles/boats
3. Switch on the furnace and programme as desired time temperature schedule.
4. Seal the tube furnace for Ar atmosphere
5. Run the programme and observe the furnace for leak proof and any abnormality during heating or cooling.
6. Switch off the furnace properly once the programme is over and take the sample out when cooled.

Lab Deliverables:

1. Write a full lab report based on your observations and results.
2. Weight the sintered compact and determine shrinkage
3. Explain mechanism of liquid phase sintering.
4. What is densification parameter? How do you measure it?

EXPERIMENT NO 9

Microstructural characterization and phase analysis of sintered products

Objective: Characterization of sintered alumina compact

Basic Theory:

When a green compact is sintered, the original particle boundaries can no longer be observed. Instead, the structure becomes similar to that of the metal in wrought and annealed conditions, except that it contains pores.

Pores are of two types open or closed. With progression of sintering, pores continue to shrink. At about 5% total porosity, the formation of closed pores commences. Many of the microstructures seen in P/M parts are caused by porosity and by the blends of elemental powders that constitute many alloys. These blends do not always result in homogeneous, well-diffused structures.

In general, the density achieved in sintered products is between 70 and 95 % of the fully dense wrought products, depending on the production technology in use and the type of application. Pores are of two types: (a) interconnected and (b) closed or isolated. In the first case the pores are connected with each other along the particle junctions. The pores are consequently irregular, unless the particles are initially spherical. Such pores can remain as low as 5% of total porosity. The latter types of pores, i.e. closed pores, are pronounced when total porosity is low (< 5%). They are often, but not necessarily, spherical.

X Ray Diffraction (XRD) is used to obtain structural information on an atomic scale from both crystalline and non-crystalline (amorphous) materials. XRD is a non-destructive technique and can be successfully applied to determine crystal structures of various types of materials such as metals, ceramics, alloys and inorganic compounds, in both the thin film/coating and bulk forms. XRD also can be applied to obtain structural information such as lattice strain, crystallite size and crystal orientation. The X-ray diffractometer is a versatile instrument for phase and structural analysis of metals, thin-films and powders.

Structure is general term, which is used to cover a wide range of structural features from visible to the naked eye down to those corresponding to the inter-atomic distances in the crystal lattice. Structure is classified into four categories: macrostructure, mesostructure, microstructure and nanostructure.

Microstructural analysis covers two aspects: qualitative and quantitative. The qualitative includes the examination of various phases and their distribution. In quantitative metallography, the measurements of the grain sizes and amounts of various phases are established.

Polished cross-sections of a powder compact provide information on the grain and pore

structures. Scanning electron microscopy (SEM) is useful in visualizing three-dimensional nature of pores. The SEM becomes the most important analytical tool in powder metallurgy. Electron microscopy provides an overview of the microstructure. Most compact properties are related to the array of features measurable via metallography. The compact processing and initial powder characteristics will dictate microstructural features. Hence, close examination will reveal the processing history and probable properties of a powder compact. The standard features such as inclusions, oxide particles, second phases, and phase transformation products are observable by metallographic analysis.

Equipment/ Raw Materials:

The raw materials consists of sintered alumina compact

Other accessories/chemicals/equipment:

- X-Ray Diffractometer (Panalytical X’Pert PRO)
- Field emission scanning electron microscope
- Pin Stubs
- Carbon tape

Procedure:

Metallographic sample preparation

1. Remove approximately, 0.2mm of the surface of compacts by grinding on a belt grinder
2. Polish the sample surfaces to mirror finish using abrasive papers
3. Final polishing is done on a flocked rayon cloth using colloidal alumina solution
4. Clean the surface using water or with ethanol
5. Clean the surface using water or with ethanol and dry carefully

Density measurement

The sintered samples were ground, polished, thoroughly cleaned in acetone medium and then dried. The weight of cleaned sample is taken using an electronic digital balance. The density of the sintered pellet is measured using Archimedes principle with water as the immersing medium. Taking the ratio of weight in air to the volume of dispersed water carried out the density measurements. Let the weight of a sample in air is W_a , and that obtained by immersing in a fluid be W_f . So, the apparent weight loss of the sample is $(W_a - W_f)$. We can say that the weight of the dispersed fluid is $(W_a - W_f)$. If the fluid has a density of D_f , then the volume of the displaced fluid is

$$\left(\frac{W_a - W_f}{D_f} \right) \quad (1)$$

which is equivalent to the volume of the sample. Therefore,

$$\text{Density of the sample } (D_c) = W_a / \left(\frac{W_a - W_f}{D_f} \right) \quad (2)$$

For water to be the immersing medium, $D_f = 1$ g/cc, and hence the density of the sample would be $D_c = \left(\frac{W_a}{W_a - W_f} \right)$ (3)

X Ray diffraction (XRD) study

X-ray diffraction studies are carried out for the purpose of phase identification of the sintered samples of Al_2O_3 . This study is made by using Panalytical X'Pert PRO diffractometer operated with $\text{Cu-K}\alpha$ radiation (having wavelength=1.54 \AA) on X-ray diffractometer. The X-ray source is operated at a current of 20 mA and a voltage of 40kV. The diffraction angle is varied in the range of $10\text{-}100^0$ and the range of 2θ is selected such that all the major peaks of the expected phases to be present in the powder are covered. Subsequent peak fitting is performed in Xpert HighScore software, wherein the peaks were analyzed for the presence of different phases.

Scanning electron microscopy study

Microstructural investigation of the polished samples is performed using secondary (SE) electron imaging mode on a Field Emission Scanning Electron microscope (FESEM) to examine the distribution of the constituent phases.

Lab Deliverables:

1. Determine density of the sintered compact.
2. Index XRD pattern of sintered alumina compact
3. Describe microstructural observation of sintered alumina compact and list the microstructural features that are observed in the current laboratory experiment.

EXPERIMENT NO 10

Hardness measurement of sintered products

Objective: Vickers hardness testing of sintered alumina compact

Basic Theory:

Hardness has a variety of meanings. In the metals industry, it may be thought of as resistance to permanent deformation. For the metallurgist, it means resistance to penetration. To the lubrication engineer, it means resistance to wear. For the design engineer, it is a measure of flow stress. To the mineralogist, it means resistance to scratching, and to the machinist, it means resistance to machining. Hardness may also be referred to as mean contact pressure. All of these characteristics are related to the plastic flow stress of materials.

Hardness is not an intrinsic property of any material (like density or melting point). It is rather a characteristic deriving from the composition, the thermal and mechanical history of the material and essentially from the structure (or more properly the microstructure) of the specimen involved. During sintering, the density and microstructure of the compact varies continuously depending on the sintering temperature. Using hardness test because it is simple, easy and relatively nondestructive can easily monitor this variation. Also, the hardness has a relationship with the other properties of material, for e.g., there is an approximate correspondence between hardness data and a range of tensile strength results.

Vickers hardness test involves a diamond indenter, in the form of a square pyramid with an apex angle of 136° , pressed for 10-15 s into the surface of the material under test. The result is a square shaped impression (Figure 10.1).

Vickers hardness value (H_V) is calculated by dividing the applied load by the surface area of the indentation.

$$\text{Area of the indentation} = \frac{d^2}{2 \sin \frac{\theta}{2}} = \frac{d^2}{1.854}$$

Where, d is the mean diagonal length and the included angle (2θ) of the indentation is equal to 136° . Therefore,

$$H_V = 1.854 \frac{P}{d^2}$$

The biggest advantage with the Vickers test is that the hardness value is independent of the magnitude of the applied load.

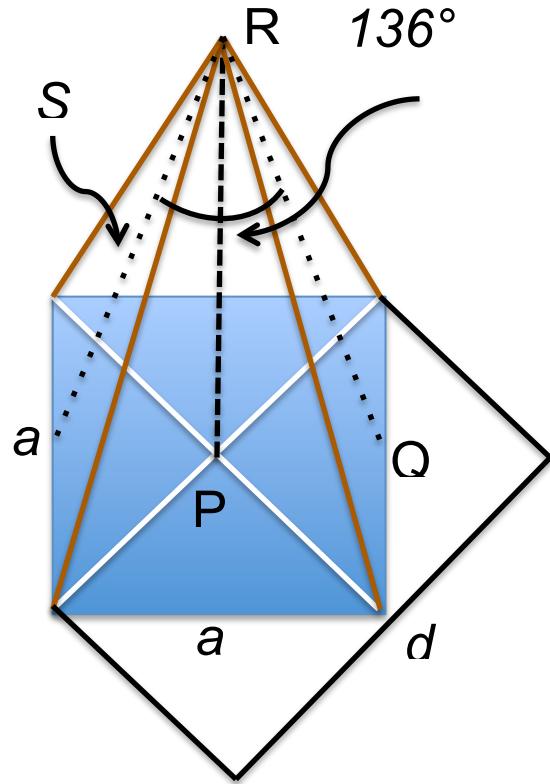


Figure 10.1: Illustrating Vickers indentation

Equipment/ Raw Materials:

Samples: Sintered alumina compact (obtained from experiment 7)

Other accessories/chemicals/equipments:

- Vickers Hardness Tester
- Belt grinder
- Polishing papers of different grades
- Ethanol solution

Procedure:

1. Remove approximately, 0.2mm of the surface by grinding on a belt grinder
2. Polish the sample surfaces to mirror finish using abrasive papers
3. Clean the surface using water or with ethanol
4. Mount the clean surface of the sample facing the indenter on the tester stage
5. Focus the polished surface of the alumina compact
6. Apply a load of 2kgf for 15 s.
7. Unload the sample gently
8. Record and measure the mean diagonal length of square impression
9. Calculate the Vickers hardness value.
10. Repeat the experiment for three times in different locations on the sample in order to obtain the error in the hardness values.

Lab Deliverables:

1. List three types of hardness testing methods along with their principles.
2. Mention some precautions that need to be taken when conduct a Vickers hardness test?
3. Report Vickers hardness value of sintered alumina compact.