EXPERIMENT NO 1

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

Objective:

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

Inevitability 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)

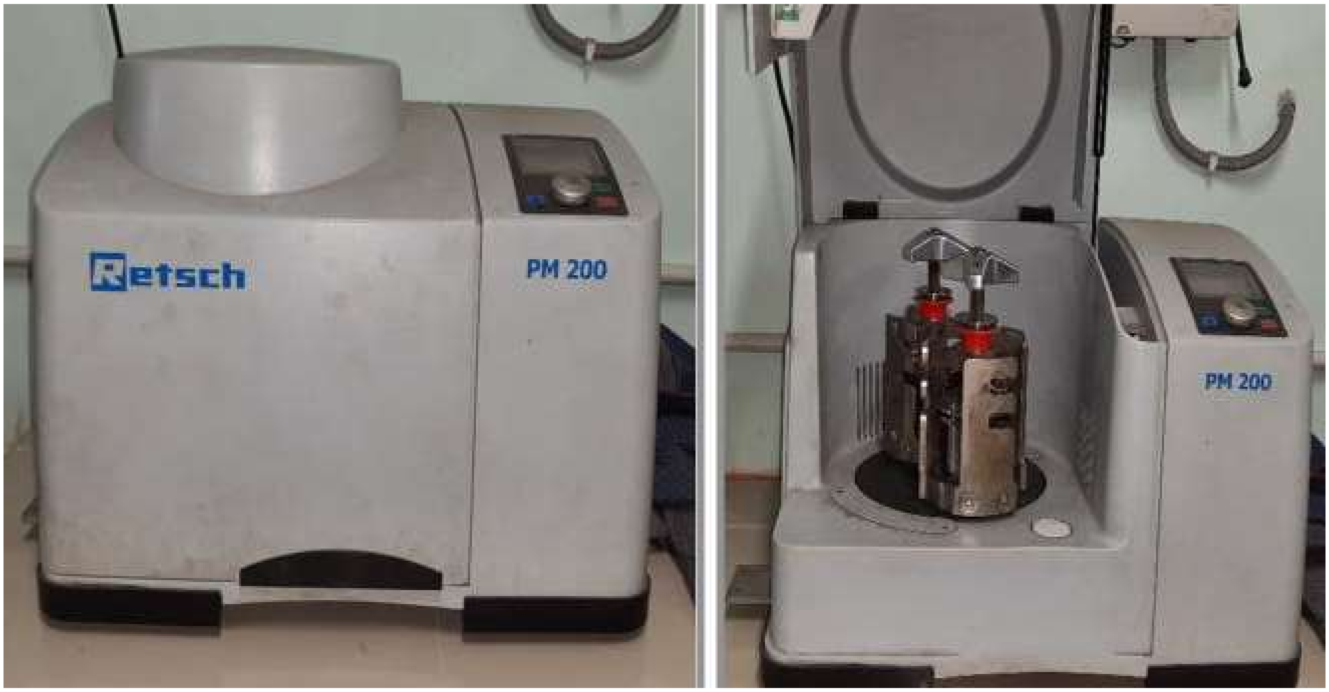


Figure1: Planetary Ball Mill: The PM 200 is a convenient bench top model with two grinding stations.

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

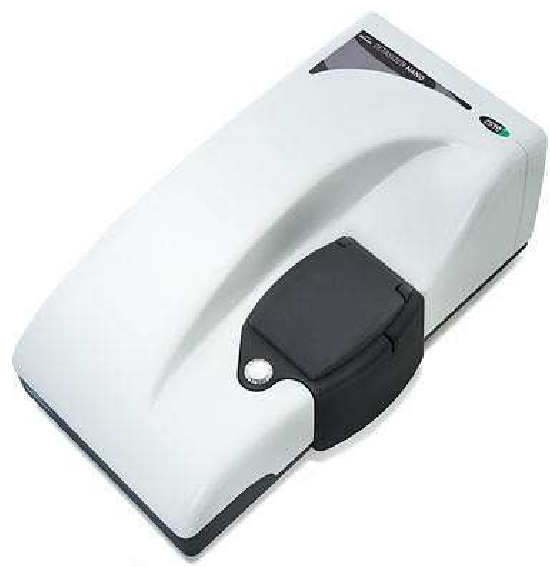
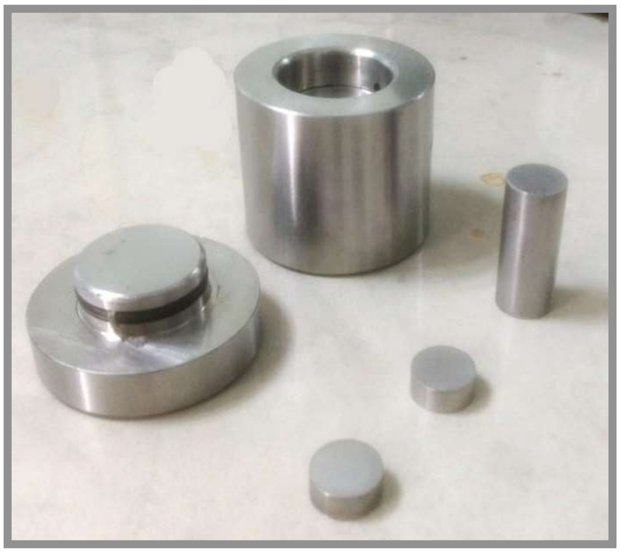


Figure 2: Zetasizer Nano ZS90

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.



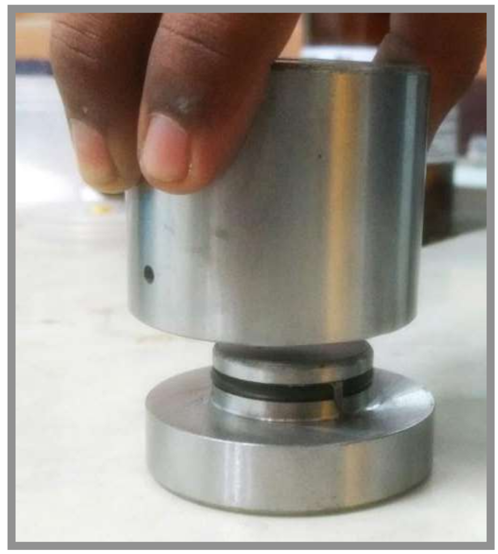


Figure 3: (a) Die, punch and support and (b) die-punch assembly

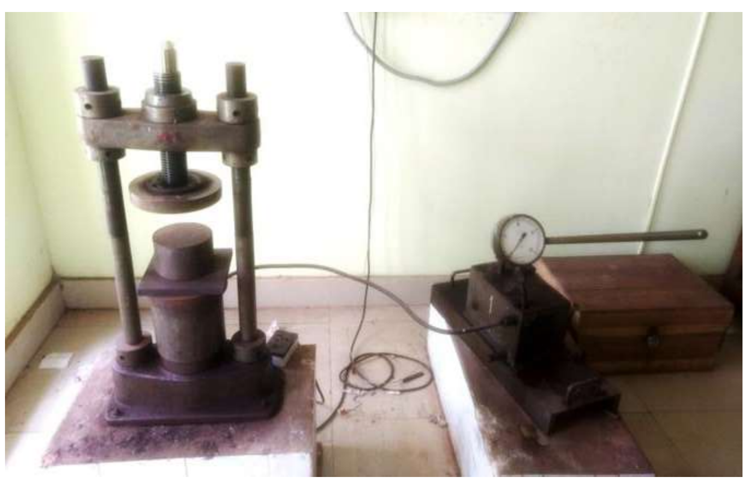


Figure 4: Manual hydraulic compaction machine

High temperature Tube Furnace

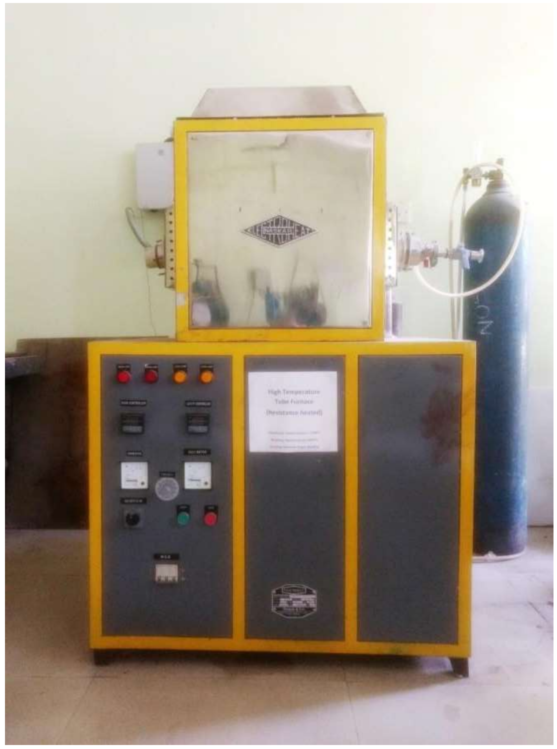


Figure 5: High temperature tube furnace

Samples are sintered in High Temperature Tube Furnace ‘(Resistance Heated, Maximum Teamperature: 1700^0 C, Working Temperature: 1600 ^{0}C, 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.

Sol:

- Mechanical Pulverization: In this method, larger solid materials are mechanically crushed or ground into smaller particles using equipment like crushers, grinders, or mills. This is a common method for producing metal powders, such as iron or aluminum.
- Chemical Precipitation: Chemical precipitation involves the chemical reaction of soluble precursors to form solid particles. For example, in the production of certain inorganic powders, chemicals are added to a solution to create solid particles through precipitation.
- Atomization: Atomization is a technique used to produce fine metal powders. Molten metal is forced through a nozzle, and it is broken into droplets that solidify into spherical particles as they cool. This method is commonly used for producing powders for applications like 3D printing and metal injection molding.
- Electrolysis: Electrolysis involves the use of an electric current to break down a chemical compound into its constituent elements. It can be used to produce powders from compounds that contain elements like metal or non-metal powders.
- Spray Drying: In spray drying, a liquid feed is atomized into tiny droplets, which are then dried rapidly by exposure to hot air. This method is commonly used to produce dry powders from liquid solutions or suspensions.
- Sol-Gel Process: The sol-gel process involves the transformation of a sol (a colloidal suspension of solid particles in a liquid) into a gel and then into a solid powder. It is often used to create ceramic and composite powders.
- Gas Phase Synthesis: Gas phase synthesis methods involve chemical reactions in the gas phase to create powders. One example is chemical vapor deposition (CVD), where solid materials are deposited on a substrate from gaseous precursors.
- Hydrothermal Synthesis: Hydrothermal synthesis utilizes high-pressure and high-temperature conditions to produce powders by promoting chemical reactions within a closed vessel. It is commonly used for the production of advanced ceramic materials.
- High-Energy Ball Milling: This method involves the use of high-energy mechanical milling to produce ultrafine powders by repeated collisions and mechanical alloying of particles.

- Sintering: Sintering is not a powder production method per se, but it involves heating compacted powders to high temperatures to form dense, solid materials. It is often used to improve the properties of powders by creating solid shapes.

2) List and briefly write about different types of mills.

Sol:

- Ball Mill: A ball mill is a type of grinder that uses balls of varying sizes to grind and blend materials for use in mineral processing, ceramics, and other industries. The balls impact and grind the material within the cylindrical drum to achieve the desired particle size.
- Attrition Mill: Attrition mills work by grinding materials in a liquid medium using agitation. They are commonly used for the production of pigments, ceramics, and coatings.
- Planetary Ball Mill: Planetary ball mills are high-energy ball mills that use grinding media and a rotating jar to create high-energy impacts for fine grinding. They are often used in research and laboratory settings.

3) Briefly describe the basic steps involved in the powder metallurgy route to get the final component from powders.

Sol:

a) Powder Production: The first step is to produce the powder feedstock. This can be done using various methods, such as mechanical pulverization, atomization, or chemical precipitation, as mentioned in your first question. The choice of powder production method depends on the material, required particle size, and other specifications.

b) Powder Blending: In some cases, different powders may need to be mixed to achieve the desired material composition. This blending step ensures uniform distribution of the various powders and any necessary additives.

c) Compaction: The blended powders are placed in a mold or die and subjected to high pressure in a compacting press. This compaction step compresses the powders into a green compact, which is a loosely held-together shape of the final component.

d) Sintering: The green compact is then heated in a controlled atmosphere in a process called sintering. During sintering, the compact is exposed to elevated temperatures below the melting point of the material. The particles within the compact bond together through diffusion, and the component becomes denser and stronger. Sintering also helps eliminate any remaining porosity.

e) Cooling and Final Processing: After sintering, the component is slowly cooled to room temperature to prevent cracking or distortion. Depending on the desired properties, additional post-sintering processes like heat treatment, machining, or surface finishing may be performed to achieve the final component's required specifications.

f) Quality Control: Throughout the process, quality control measures, such as dimensional checks, density measurements, and material testing, are conducted to ensure the final component meets the desired standards.

g) Optional Secondary Operations: In some cases, additional operations like machining, heat treatment, coating, or assembly may be required to meet specific design and functional requirements.

4) What are the characteristics of powders through which we classify them?

Sol:

- Particle Size: Particle size is one of the most important characteristics. Powders can be classified as fine, coarse, or have a specific particle size distribution. Particle size affects properties like flowability, surface area, and reactivity.
- Particle Shape: Particle shape can vary from spherical to irregular. Shapes influence packing density, flowability, and compaction behavior. Common shape descriptors include spherical, angular, flake-like, or needle-like.
- Particle Size Distribution: The distribution of particle sizes within a powder is critical. Powders can have a narrow (monodisperse) or broad (polydisperse) size distribution. The distribution impacts the uniformity of properties in the final product.
- Particle Surface Area: Surface area refers to the total area of the particle's external surfaces. High surface area powders are often more reactive and are used in applications like catalysis.
- Particle Density: Particle density is the mass of a particle per unit volume. It influences properties like bulk density and flow behavior. Different materials may have different particle densities.
- Porosity: Porosity measures the fraction of void space within a powder. Porosity can affect properties like strength, permeability, and density.

- Chemical Composition: The chemical composition of a powder is crucial for defining its material properties, reactivity, and compatibility with specific processes or applications.
- Surface Chemistry: Surface chemistry, including surface treatments and coatings, can modify a powder's reactivity, stability, and adhesion properties.

5) State and explain the working principle of

(i) die compaction and

(ii) density measurement by Archimedes’ principle.

Sol:

(i) Die Compaction:

i. Die compaction is a common step in powder metallurgy and involves compressing loose powders into a compact shape using a die or mold. The working principle of die compaction is as follows:
ii. Powder Filling: Initially, the loose powder is poured or placed into the cavity of the die, which is a tool or mold with a specific shape and volume that matches the desired final component.
iii. Compaction: A punch or plunger is then applied with significant force to compress the powder within the die. The powder particles are brought closer together, and interparticle friction and mechanical interlocking occur.
iv. Green Compact Formation: The result is the formation of a green compact, which is a shape held together by mechanical forces but not yet fully dense or solid. The compact retains the shape of the die.
v. Ejection: After compaction, the punch is withdrawn, and the green compact is ejected from the die. The green compact may undergo further processing, such as sintering, to achieve full density and strength.
vi. The working principle of die compaction relies on the application of pressure to reduce the void spaces between powder particles and create a coherent structure. The specific pressure, rate of application, and die geometry are critical factors in determining the density and properties of the green compact.

(ii) Density Measurement by Archimedes' Principle:

i. Density measurement by Archimedes' principle is a method to determine the density of a solid object, including irregularly shaped objects, by measuring the buoyant force experienced when the object is submerged in a fluid. The working principle of this method is as follows:

ii. Selection of Fluid: A fluid, typically water, with a known density is chosen for the measurement. The fluid should be compatible with the object under test.

iii. Weighing the Object in Air: The object's weight is measured while it is in the air. This provides the object's mass and gravitational force acting on it (weight).

iv. Submerging the Object: The object is submerged in the selected fluid, and its weight is measured while it is fully immersed. This provides the weight of the object in the fluid ($weight_{submerged}$).

v. Calculation of Buoyant Force: The buoyant force (F_b) experienced by the object is equal to the weight loss it undergoes when immersed in the fluid. It can be calculated as $F_b = weight - weight_{submerged}$.

vi. Determining Density: Archimedes' principle states that the buoyant force experienced by the object is equal to the weight of the fluid displaced by the object. Therefore, the buoyant force is directly proportional to the volume of the object.

vii. Density Calculation: The density of the object (ρ_{object}) can be calculated using the formula $\rho_{object} = \rho_{fluid} * (weight - weight_{submerged})$ / weight of an equal volume of the fluid, where ρ_{fluid} is the known density of the fluid.