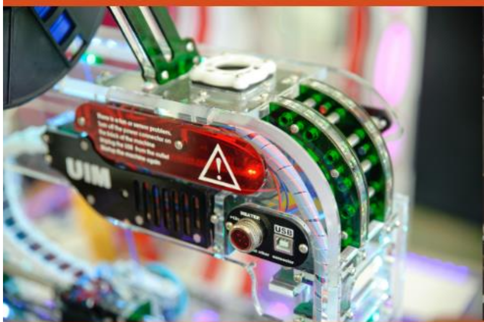
MANUFACTURING PROCESSES

A book by Career Avenues

As per GATE Metallurgy Syllabus



CAREER AVENUES

Manufacturing Processes

Chapter-4
Powder Metallurgy

4.1 Introduction

- Powder metallurgy is the process of blending fine powdered materials, pressing them into a desired shape (compacting) and then heating the compresses material in a controlled atmosphere to bond the material (sintering)
- It is typically used when large amount of small, intricate parts with high precisions are required.

4.2 Steps involved in powder metallurgy

Production of Powders

Powders are manufactured by various methods. The size of the powder largely effects the properties of the part produced.

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Powder Blending

Powders are blended or mixed with lubricants so that the powders are thoroughly intermingled.

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Compacting

It is done for shaping of the powder into the desired shape with sufficient strength to withstand further handling operations.

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Sintering

The green compact is subjected to heating without any pressure for certain period of time under controlled conditions to bond atoms metallurgically.

4.2.1 Production of Powders

4.2.1.1 Properties and characteristics of powders

I. **Purity:** Metal powders should be free from impurities as the impurities reduce the life of the dies and effect sintering process. The oxides and the gaseous impurities can be removed from the part during sintering by use of reducing atmosphere.

II. **Particle size:** The particle size influences the control of porosity, compressibility and amount of shrinkage. It is determined by passing the powder through standard sieves or by microscopic measurement.

III. **Particle size distribution:** It is specified in terms of sieve analysis, the amount of powder passing through 1,00,200 etc., mesh sieves. Particle size distribution influences packing of powders and its behaviour during moulding and sintering.

IV. **Particle shape:** The particle shape depends largely on the manufacturing method of powders. The particle shape can be nodular, irregular, angular and dendritic. The particle shape largely influences the flow characteristics of powders. Some special shaped particles have excellent sintering properties. However, irregular shaped particles have good green strength (compaction) since on compacting; they interlock and attain a strong bond.

V. **Flow rate:** It is the ability of powder to flow readily and confirm to the mould cavity. It determines the rate of production and economy.

VI. **Compressibility:** It is defined as volume of initial powder (powder loosely filled in cavity) to the volume of compact part. It depends on particle size, distribution and particle shape.

VII. **Density**

(a) **True density:** It is the density of the true volume of the material, i.e. the density of the material if the powders were melted into a solid mass.

(b) **Bulk density:** It is the density in the loose state after pouring. Because of the presence of pores between the powder particles, the bulk density is less than the true density.

(c) **Tapped density:** Tapped density of powder is the ratio of the mass of the powder to the volume occupied by the powder after it has been tapped for a definite period of time. The tapped density of a powder represents its random dense packing. Tapped density values are higher for more regularly shaped particles (i.e., spheres) as compare to irregular shaped particles.

(d) **Green density:** The density of the compacted part before sintering is termed as Green density. A smooth surfaced powder results in higher compressibility and hence higher green density whereas an irregularly shaped particle can fold on itself creating porosity that cannot be removed by further compaction, resulting in lower green density.

4.2.1.2 Powder production methods

I. Atomization

- The Atomization process involves the use of high-pressure fluid jets to break the stream of molten metal into very fine particles which when solidify into fine particles.

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(a) Gas atomization:

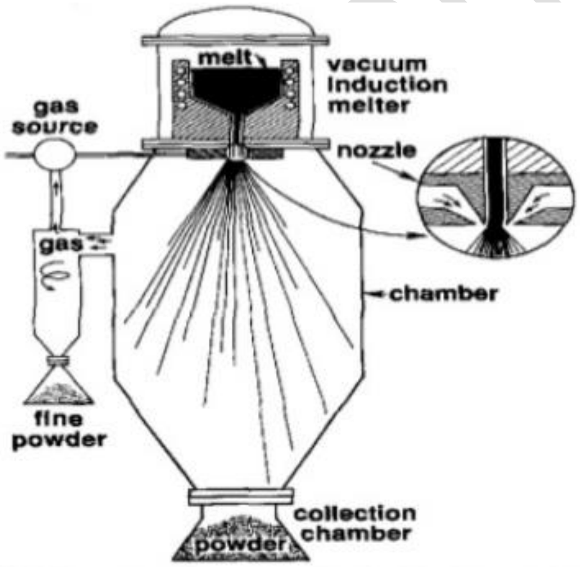


Figure: Gas atomization process

- This process produces a liquid metal stream by injecting molten metal through orifice.
- Stream is broken by jets of inert gas, air or water.
- The size of the particle formed depends on the temperature of the metal, metal flow rate through orifice, nozzle size and jet characteristics.

(b) Water Atomization

- Water Atomization Process refers to a process that sprays and collides water at a high pressure of about 50-150 MPa against a molten metal melted at a high temperature to produce fine metal powder (atomized powder) at the micron level.
- Water jets are mainly used because of their higher viscosity and quenching ability.

(c) Vacuum Atomization

- In this method, when a molten metal supersaturated with a gas under pressure is suddenly exposed into vacuum, the gas coming from metal solution expands, causing atomization of the metal stream.
- This process gives very high purity powder. Usually hydrogen is used as gas. Hydrogen and argon mixture can also be used.

(d) Centrifugal Atomization

- In this method, one end of the metal bar is heated and melted by bringing it into contact with a non-consumable tungsten electrode, while rotating it longitudinally at very high speeds.
- The centrifugal force created causes the metal drops to be thrown off outwards. This will then be solidified as spherical shaped particles inside an evacuated chamber.
- Titanium powder can be made using this technique.

(e) Rotating disc Atomization

- It involves impinging of a stream of molten metal on to the surface of rapidly spinning disk.
- This causes mechanical atomization of metal stream and causes the droplets to be thrown off the edges of the disk.
- The particles are spherical in shape and their size decreases with increasing disk speed.

II. Solid State reduction/ Chemical reduction

- In this method, oxides are first ground to control the particle size and then it is reduced by a gas usually carbon monoxide, hydrogen or cracked natural gas at a temperature which is below the melting point of the metal.
- Particle size and shape can be controlled within rather wide limits by varying the particle size and shape of the oxides, the reducing temperature, pressure and flow of the gas.
- The resulting powder is milled, classified and blended to the desired specifications.
- The purity of the product depends on the purity of the oxide since there is no refining during the reduction process. Generally, the powders produced by this method tend to be spongy porous and have high apparent densities and green strength.
- The powder particles obtained by this method have uniformly sized spherical or angular shapes.
- This process is used for iron, copper, tungsten, molybdenum, nickel and cobalt

III. Carbonyl process

- In this process, powder is obtained from metal compounds which can be transferred to the vapour phase.

  The decomposition reaction involve can be given as:

$$\boldsymbol{M(CO)_x \leftrightarrow M + xCO}$$

  With an increasing temperature, in metal carbonyls dissociation to metal and carbonyl occurs.

- When Iron and Nickel ores react under high pressure (70-300atm) with CO, Iron pentacarbonyl [$Fe(CO)_5$] and Nickel tetracarbonyl [$Ni(CO)_4$] is formed.
- Reactions of carbonyl formation and decomposition can be described as:

$$Fe + 5CO \leftrightarrow Fe(CO)_5$$
$$Ni + 4CO \leftrightarrow Ni(CO)_4$$

  The equilibrium of these reactions depends highly on temperature and pressure.
- The carbonyl decomposition is an endogenous reaction running in a heated cylinder at temperature of 200°C for $Ni(CO)_4$ or at 250°C for $Fe(CO)_5$.
- Small, dense and uniform spherical/angular powders of high purity are formed.

IV. Electrolytic deposition

- In this method, metal powders of high purity deposited at the cathode from aqueous solution of an electrolytic cell.
- This method is mainly used for producing Copper, iron, zinc, tin, nickel, cadmium, lead, antimony, silver, beryllium powders.
- The basic characteristics of powders made using electrolysis is their high purity and a dendritic or sponge structure, good compressibility and high strength of non-sintered compacts and high activity at sintering.

V. Comminution

- This method involves mechanical forces such as compressive forces, shear forces or impact forces.
- These forces on application facilitate particle size reduction of bulk materials.
- Example: Milling, grinding, shooting, graining etc.
- Powder produced:
  Brittle metal- angular shaped
  Ductile metal- flaky (not particularly suitable for powder metallurgy operations)

Mechanical alloying

- *Powders of two or more metals are mixed in a ball mill.*
- *Under the impact of hard balls, powder fracture and join together by diffusion*

VI. Other methods of Powder Manufacturing:

- Precipitation from solution

- Condensation of metal vapours
- Powders of the elements titanium, vanadium, thorium, niobium, tantalum, calcium, and uranium have been produced by high-temperature reduction of the corresponding nitrides and carbides.

> *Almost any metal, metal alloy or nonmetal like ceramic, polymer or wax or graphite lubricant can be converted into powder form by any of the methods. Some methods can produce only elemental powder, often of high purity, while others can produce pre-alloyed particles. Operations such as drying or heat treatment may be required prior to further processing.*

- Iron, nickel, uranium, and beryllium sub-micrometer powders are obtained by reducing metallic oxalates and formates.

4.2.1.3 P

- **Flow rate** is a measure of the ease by which powder can be fed and distributed into a die. Poor flow characteristics can result in non-uniform die filling and in non-uniform density and properties in a product.
- Associated with the flow characteristics is the **apparent density**, a measure of the powder's ability to fill available space without the application of external pressure.
- **Compressibility tests** evaluate the effectiveness of applied pressure in raising the density of the powder, and green strength is used to describe the strength and fracture of resistance.

> ***The size and shape of powder particle can varied and primarily depends on the following factors:***
>
> - *Velocity and media of the atomizing jets or the speed of electrode rotation*
> - *Starting temperature of the liquid (which affects the time that surface tension can act on the individual droplets prior to solidification)*
> - *Environment provided for cooling*
> - *When cooling is slow (such as in gas atomization) and surface tension is high, spherical shapes can form before solidification.*
> - *Irregular shapes are produced due to more rapid cooling, such as water atomization.*

The table mentioned below is very important For GATE MT (Match the following type question)

Sr. No.	Powder production method	Shape of the powder	Examples
1.	**Atomization**	**Spherical shape, Irregular shape**	**Titanium, super alloys such as Ni based, stainless steel, tool steel, brass, Aluminium**
2.	**Chemical reduction**	**Spongy and porous: Spherical or angular shape**	**Fe, Ni, Cu, Co, W, Mo**
3.	**Electrolytic deposition**	**dendritic**	**Fe, Cr, Ag, Cu, Mg**
4.	**Carbonyl process**	**Spherical: Fe Angular: Ni**	**Fe, Ni**
5.	**Comminution**	**Flakes: Ductile material Angular: Brittle material**	
6.	**Rotating Electrode process**	**Uniform and spherical in shape.**	**Ta**

4.2.2 Blending and mixing of powders

- Homogenization of metal powders is necessary to attain effective results in compaction and sintering.
- **Blending:** It refers to the intermingling of powders having same chemistry but varying particle sizes.
- **Mixing:** It refers to the intermingling of powders having different chemistries.

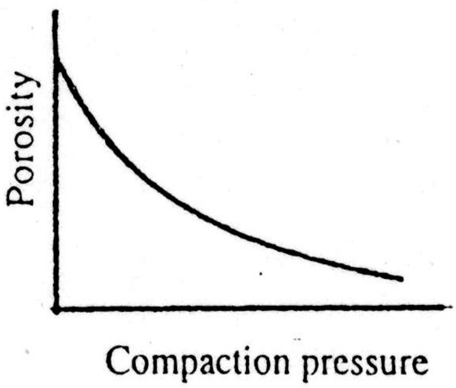
NOTE: Powder metallurgy technology allows mixing various metals into alloys that would be difficult or impossible to produce by any other means.

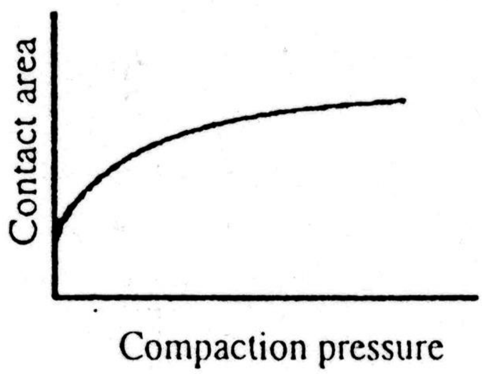
- Mixing powders of different metals/materials
- Add lubricants (<5%), such as graphite and stearic acid, to improve the flow characteristics and compressibility of mixtures

- **<u>Combining is generally carried out in</u>**
  - air or inert gases to avoid oxidation
  - Liquids for better mixing, elimination of dusts and reduced explosion hazards
- **<u>Hazards</u>**
  - Metal powders, because of high surface area to volume ratio are explosive, particularly Al, Mg, Ti, Zr, Th

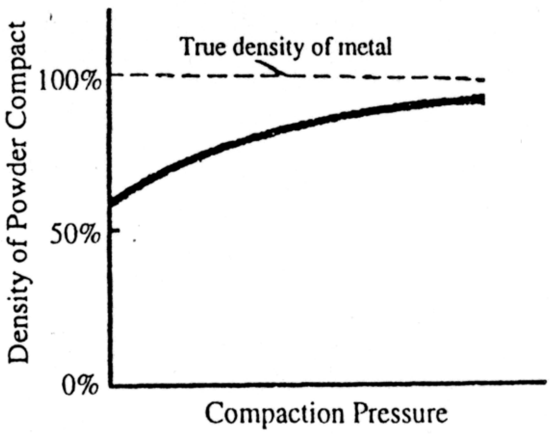
4.2.3 <u>Compaction of metal powders</u>

- Powder compaction is the process of compacting metal powder in a die through the application of high pressures. Typically, the tools are held in the vertical orientation with the punch tool forming the bottom of the cavity.
- The powder is then compacted into a shape and then ejected from the die cavity. In a number of these applications the parts may require very little additional work for their intended use; making for very cost-efficient manufacturing.



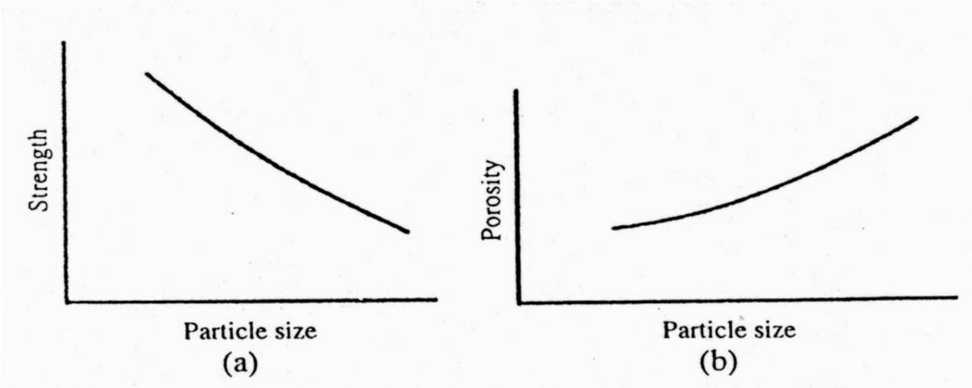


- The density of the compacted powder is directly proportional to the amount of pressure applied. Pressure of 10 tons/in² to 50 tons/in² are commonly used for metal powder compaction.



- Increased compaction pressure provides better packing of particles and leads to ↓ porosity
- At higher pressures, the green density approaches density of the bulk metal
- Pressed density greater than 90% of the bulk density is difficult to obtain

- Compaction pressure used depends on desired density



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NOTE: *In powder metallurgy or ceramics it is possible to fabricate components which otherwise would decompose or disintegrate. All considerations of solid-liquid phase changes can be ignored, so powder processes are more flexible than casting, extrusion, or forging techniques.*

Isostatic Pressing

- In some pressing operations (such as Hot Isostatic pressing) compact formation and sintering occur simultaneously.
- This procedure, together with explosion-driven compressive techniques, is used extensively in the production of high-temperature and high-strength parts such as **turbine blades** for **jet engines**. In most applications of powder metallurgy the compact is hot-pressed, heated to a temperature above which the materials cannot remain work-hardened.
- Hot pressing lowers the pressures required to reduce porosity and speeds welding and

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- **COLD ISOSTATIC PRESSING** (CIP) is a materials processing technique in which high pressure is applied to **metal powder** in a sealed elastomer container shaped for the application
- Press powder into the desired shape and size in dies using a hydraulic or mechanical press
- Pressed powder is known as “green compact”

NOTE: Unfortunately, the handling of aluminum/iron powders poses major problems. Other substances that are especially reactive with atmospheric oxygen, such as tin, are compressed in special atmospheres or with temporary coatings.

Green compact:

- Loose powder is compressed and densified into shape, usually at room temperature.
- Most compacting is done with mechanical presses and rigid tools, but hydraulic and hybrid presses can also be used.
- Compacting pressures generally range between 3 and 120 tons/in^2 (40 to 1650 MPa) depending on material and application with 10 to 30 tons/in^2 (140 to 415 MPa) being the most common.
- Most P/M presses have total capacities of less than 100 tons (9×10^5N).
- Metal-forming processes, such as rolling, forging, extrusion, and swaging, have also been adapted to compact powders.

Green Strength

The mechanical strength which a compacted powder must have in order to withstand mechanical operations to which it is subjected after pressing and before sintering, without damaging its fine details and sharp edges.

Compaction with a Single Punch

When pressure is applied at one punch, maximum density occurs below the punch and decreases as one moves down the column.

Double-Action Press

- More uniform density can be obtained and thicker products can be compacted.
- Since sidewall friction is a key factor in compaction, the resulting density is strong function of both the thickness and width of the part being compressed. For good, uniform compaction, the ratio of thickness/width should be kept below 2.0 whenever possible.

Effect of Compacting Pressure on Green Density

The average density of the compact depends on the amount of the pressure that is applied.

Multi-thickness Part with only One Punch:

- Shows that a single displacement will produce different degrees compaction in different thickness of powder.
- Therefore, it is impossible for a single punch to produce uniform density in multi-thickness part.

Method of Compacting Two Thickness Parts to Near Uniform Density

- By providing different amounts of motion to the various punches and synchronizing these movements to provide simultaneous compaction, a uniformly compacted product can be produced.

Compaction Tooling (Punches and Dies)

- Compaction tools are usually made off harden tool steel.

- Die surfaces should be highly polished and the dies should be heavy enough to withstand the high pressing pressures. Lubricants are also used to reduce die wear.

P/M Injection Moulding

Small, complex-shaped components have been fabricated from plastic for many years by means of injection moulding. While the powdered material does not flow like a fluid: complex shapes can be produced by mixing ultrafine (usually less than 10 um) metal, ceramic, or carbide powder with a thermoplastic/wax material (up to 50% by volume). A water-soluble methylcellulose binder is one attractive alternative to the thermoplastics.

NOTE: (Important Formulae for GATE)

I. **Formula to calculate initial fill height**

As per mass constancy,

$$\rho_a h_a = \rho_g h_g$$

Where ρ_a = apparent density

h_a = height of the powder before compaction (initial fill height)

ρ_g = green density

h_g = height of the green compact

II. **Densification Parameter**

$$Densification\ parameter = \frac{\rho_{green} - \rho_{apparent}}{\rho_{theoretical} - \rho_{apparent}} \times 100$$

Where ρ_{green} = green density

$\rho_{apparent}$ = apparent density

$\rho_{theoretical}$ = theoretical density

4.2.4 Sintering

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- ion. the process of densification leads to the reduction of porosity and increase in the grain size through mass transport.

- The ability to achieve dimensional tolerances in powder metallurgy part arises directly from the control of the sintering process and in particular shrinkage. Thus a detailed understanding of the shrinkage process and the associated shrinkage is of considerable importance.

4.2.4.1 <u>Stages occur during sintering</u>

<u>Stages of Sintering</u>

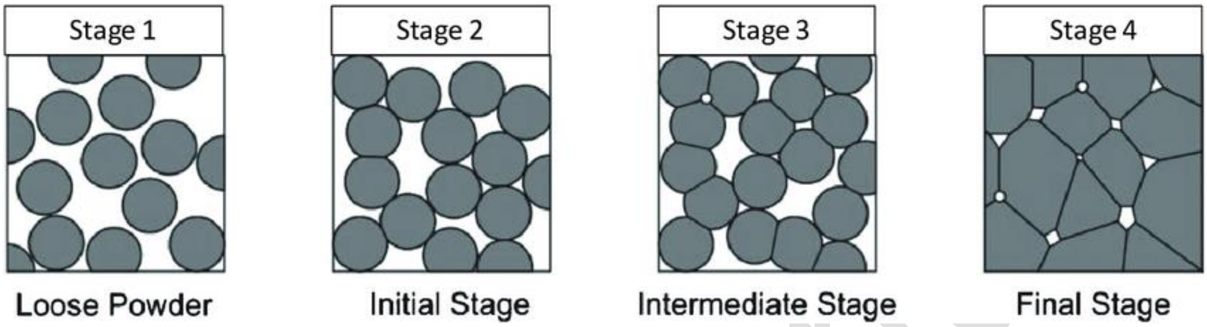

Figure: Stages of Sintering Process

(i) <u>First Stage: Initial growth stage</u>

- Sintering initially causes the particles that are in contact to form grain boundaries at the point
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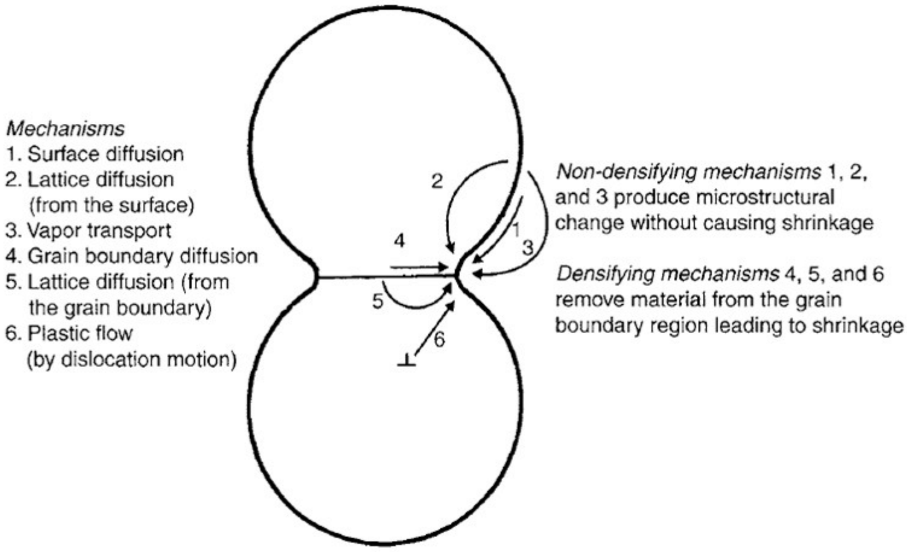
4.2.4.2 <u>Driving force for Sintering</u>

- Sintering is accompanied by an increase in the free energy of the system. The sources that give rise to the amount of free energy are commonly referred to as the driving force for sintering.
- The main possible driving forces are:
  (i) The curvature of the particle surfaces (reduction in total particle surface area: Surface energies are larger in magnitude than grain boundary energies)
  (ii) An externally applied pressure
  (iii) A chemical reaction

4.2.4.3 <u>Sintering mechanism</u>

The process of Sintering occurs due to various mass transport mechanisms.

Sintering mass transport mechanisms



Surface transport mechanism (Non-densifying mechanisms)	Bulk transport mechanism (Densifying mechanism)

NOTE:

Different mechanisms dominate at different points in the sintering process and different materials exhibit different mechanisms.

For example: Viscous flow is the diffusion in the liquid phase and evaporation condensation (vapour diffusion) is in the gaseous phase.

4.2.4.4 **Linear Shrinkage calculation**

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4.2.4.5 **Types of Sintering Process:**

I. **Solid state sintering**

- Solid state sintering is the process of taking metal in the form of a powder and placing it into a mould or die. Once compacted into the mould the material is placed under high heat for a prolonged period of time.
- Under the application of heat, bonding takes place between the porous aggregate particles and once cooled, the powder bonded to form a solid piece.

II. **Liquid state sintering:**

- The densification is improved by employing a small amount of liquid phase (1-10% vol.). The liquid phase existing within the powders at the sintering temperature has some solubility for the solid. Sufficient amount of liquid is formed between the solid particles of the compact sample. During sintering, the liquid phase crystallizes at the grain boundaries binding the grains. During this stage, there is a rapid rearrangement of solid particles leading to density increase. In later stage, solid phase sintering occurs resulting in grain coarsening and densification rate slows down.
- This type of sintering is used for systems like tungsten-copper and copper-tin. Also covalent compounds like silicon nitride, silicon carbide can be made that are difficult to sinter.

III. **Activated Sintering:**

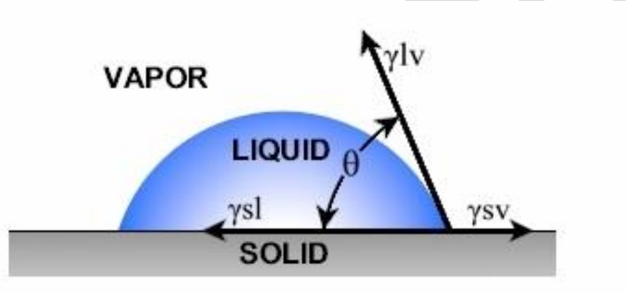
- In this, an alloying element called 'doping' is added in small amount and it improved the densification by as much as 100 times as compare to the un-doped compact samples. Example is the doping of nickel in tungsten compacts.

IV. <u>Reaction Sintering:</u>

- In this process, high temperature materials resulting from chemical reaction between the individual constituents, giving strong bonding.
- Reaction sintering occurs when two or more components react chemically during sintering to create final part.
- Example: the reaction between alumina and Titania to form aluminium titanate at 1553K which then sinters to form a densified product.

4.2.4.6 <u>Liquid phase sintering</u>

- When the liquid forms in Liquid phase sintering, the microstructure consists of solid, liquid and vapour. Liquid spreading on the solid replaces solid-vapour interfaces with liquid-solid and liquid-vapour interfaces.



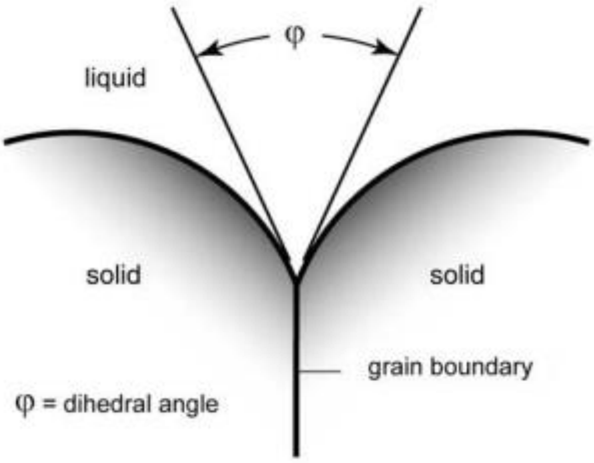
- In the horizontal plane, the contact angle θ is associated with three interfacial energies,

Where

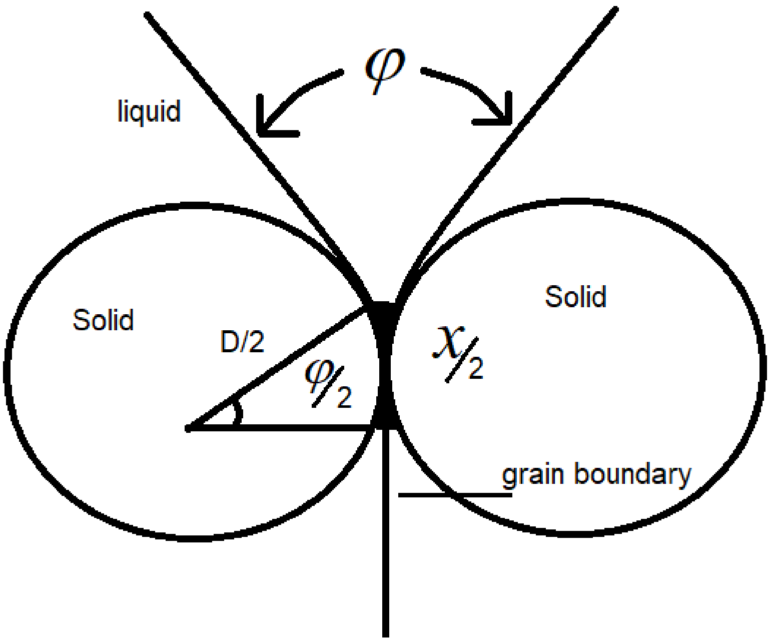
Whereas a high contact angle indicates poor wetting, so the liquid retreats from the solid. This results in compact swelling and liquid discharging from the pores.

Therefore, depending on the contact angle, liquid formation causes either densification or swelling. The magnitude of the capillary effect depends on the amount of liquid, particle size and contact angle.

- A solid-vapour dihedral angle is observed where a grain boundary intersects the vapour phase, but in Liquid Phase Sintering, more concern is given to the intersection of the grain-grain contacts with the liquid phase.



Neck size determination



From the figure given above, the neck size can be calculated as

$$Sin\frac{\varphi}{2} = \frac{x/2}{D/2}$$

Where x = neck size

D = grain diameter

4.2.7 Secondary Operations

- Powder Metallurgy parts are ready to use after they have emerged from the sintering furnace by many products utilize one or more secondary operations to provide enhanced precision, improved properties, or special characteristics.

Secondary operations are be performed to improve:

- Density
- Strength
- Shape
- Corrosion Resistance
- Tolerances

Powder metallurgy processes create porosity. In order to reduced the porosity, Metal Injection Moulding is used

- MIM (Metal Injection Moulding) minimizes total porosity and typically limits interconnected porosity (that porosity connected to a free surface) to less than 0.2%, regardless of the product's percent of full density. This means standard colouring and plating techniques can be used without resin impregnation.
  (Oil impregnation and copper infiltration are not used with MIM).
- When heat treated, parts can be case hardened to closely control case depths equivalent to wrought material.
- Other metalworking techniques such as drilling, tapping, turning, grinding, and broaching work well with MIM.
- All parts are barrel finished unless otherwise specified.

A wide range of additional operations or treatments can be carried out on the parts after they have been sintered.

- **<u>Heat Treatment:</u>** Sintered parts may be heat treated to increase strength and also hardness for improved wear resistance.
- **<u>Oil Impregnation:</u>** The controlled porosity of P/M parts permits their impregnation with oil and resin. This operation is used to give the part self-lubricating properties.
- **<u>Resin Impregnation:</u>** Used to improve machinability, seal parts gas or liquid tight, or prepare the surface for plating.
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- Certain metals that are difficult to fabricate by other methods can be shaped by powder metallurgy. Example: Tungsten filaments for incandescent lamp bulbs are made by PM
- Certain alloy combinations and cermets made by PM cannot be produced in other ways
- PM compares favourably to most casting processes in dimensional control

- PM production methods can be automated for economical production
- Elimination or reduction of machining
- High Production Rates
- Complex Shapes to be Produced
- Wide Variations in compositions are Possible
- Wide Variation in properties are available.
- Scrap is eliminated or reduced.
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Disadvantages

- Cost for making products is very high
- Only small and uniform cross section components can be made
- Inferior Strength Properties
- Relatively High Die Cost
- High Material Cost
- Design Limitations
- Density Variations Produce Property Variations
- Health and Safety Hazards

4.2.7 Applications of Powder metallurgy

Products that are commonly produced by powder metallurgy can generally be classified into five groups.

I. Porous or permeable products:

- Oil-impregnated bearings made from either iron or copper alloys, constitute a large volume of Powder Metallurgy products. They are widely used in home appliance and automotive applications since they require no lubrication or maintenance during their service life. Unlike many alternative filters, they can withstand conditions of elevated temperature, high applied stress, and corrosive environments.

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Past GATE Questions

1. If the solid-solid interfacial energy (γ_{SS}) is 0.87 $J.m^{-2}$and solid-liquid interfacial energy(γ_{SL}) is 0.5 Jm^{-2}, the dihedral angle (ϕ, in degree, rounded off to one decimal place) during sintering is __________ **[GATE 2019]**

2. Consider the following engineering components:
P. Gas turbine blades
Q. Tungsten-based heavy alloy penetrators
R. Self-lubricating bearings
S. Engine block of an automobile
Which of the following two components are produced by powder metallurgy? **[GATE 2018]**
(a) P and Q (b) Q and R (c) Q and S (d) P and S

SAMPLE

8. Match the powder production technique given in Group I with the correspondent shape listed in Group II. **[GATE 2013]**

Group I	Group II
P. Reduction	1. Flaky
Q. Gas atomization	2. Spongy
R. Milling	3. Dendritic
S. Electrolysis	4. Spherical

(a) P-2, Q-4, R-1, S-3 (b) P-1, Q-3. R-2, S-4
(c) P-2,. Q-3, R-4, S-1 (d) P-3, Q-2, R-1, S-4

9. Tungsten filament used in electric bulb is processed by **[GATE 2012]**

(a) extrusion
(b) wire drawing
(c) casting
(d) powder metallurgy

Answers

1. 59.1

Given

γ_{SS} = 0.87Jm^{-2}

γ_{SL} = 0.5 Jm^{-2}

$2\gamma_{SL}Cos\left(\frac{\phi}{2}\right) = \gamma_{GB} \quad (\gamma_{GB} = \gamma_{SS})$

$$2(0.5)\cos\left(\frac{\phi}{2}\right) = 0.87$$

$$\cos\frac{\phi}{2} = \frac{0.87}{1}$$

$$\cos\frac{\phi}{2} = 0.87$$

$$\frac{\phi}{2} = \cos^{-1}(0.87)$$

$$\frac{\phi}{2} = 29.54^o$$

$$\phi = 29.54 \times 2$$

$$= 59.08^o$$

$$= 59.1^o$$

2. (b) Q and R