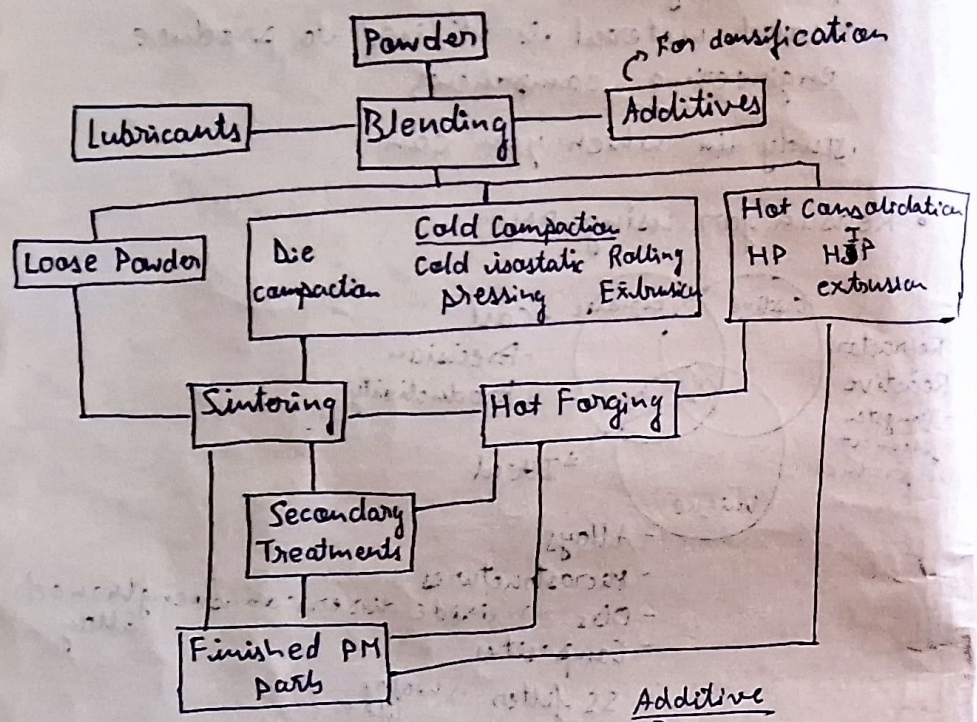
I've tried my best to make sure there's no wrong information, mistakes, or errors. However, I don't take any responsibility if any of these issues come up.

- Conventional Sintering
  - Collect powder
  - Add lubricant
  - Compress
  - Sinter



- Powder production & Characterization
- Powder treatment
- Powder compaction
- Sintering

Additive

ZrB_2 → contains ZrO_2

B_4C is added to remove ZrO_2 and increase in density

Lubricant

Reduce friction b/w powder and die cavity

Surface modification

Cold isostatic

- Rubber mold used (Rubber bag) — Bag filled with powder
- Kept in fluid vessel

HIP

- Thin sheet metallic mould

HP

- Die → graphite, hardened steel (eg: Ti alloy) (eg: Al alloy)

Sintering temp: 0.7 - 0.9 T_m (absolute scale)

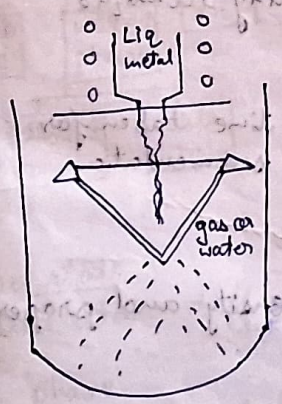
18/07/23

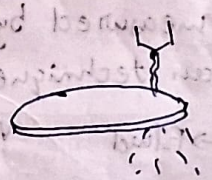
- Powder fabrication and characterization
  - Chemical fabrication (reduce metal oxide)
  - Electrolytic fabrication (used when high purity required) Eg - Cu
  - Atomization → gas, water & centrifugal
  - Mechanical fabrication

  - Particle size & distribution
  - Particle shape
  - Surface area
  - Interparticle friction
  - Phase

- Powder treatment
  - Mixing and blending
- Compaction
- Sintering

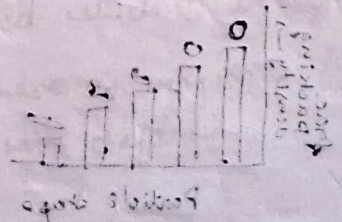
Atomization





Centrifugal atomization - for low melting materials

Med

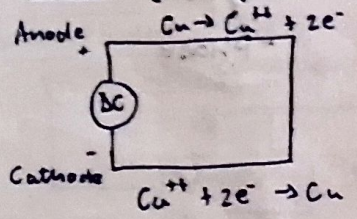


Mechanical fabrication

- Brittle materials suitable, hard and easily fractured materials.

Machining is a type of mech. fab. but it produce large particles.

Electrolytic fabrication



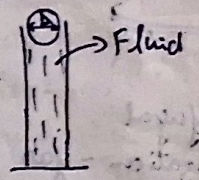
~~Particle~~

Sintering - small particles agglomerate

Easy for smaller particles as temp reqd, surface area ↑, surface energy ↑.

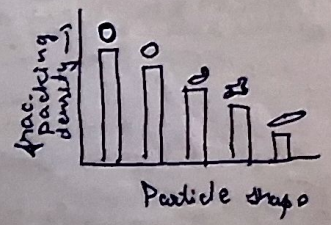
Particle size - measured by screening technique

Sedimentation technique



$V = H/t$ → time taken for sedimentation

Particle shape - affects the density and properties of the material.



Interparticle friction

Flow and packing density is affected

Phase

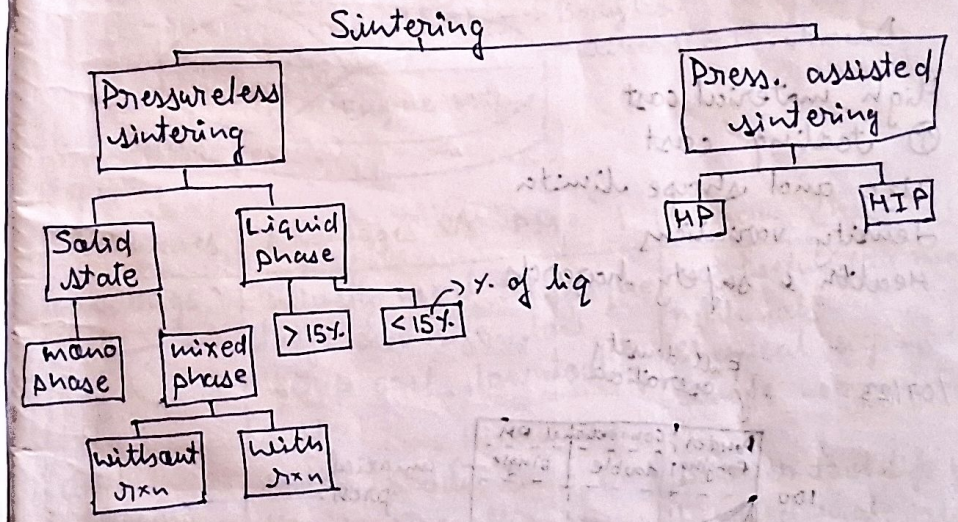
Compaction: carried out to strengthen the object

Sintering: increase the strength

0.7 - 0.9 T_m (absolute scale)

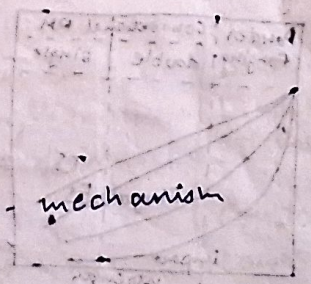
Decreases the surface area.

Chemical fabrication ↑ the surface area.



Solid state

solid state diffusion - mechanism



Liq phase

- Low melting material added which ↑ the density.
- Depends on % of liq - if liq phase is very ↑ material will deform

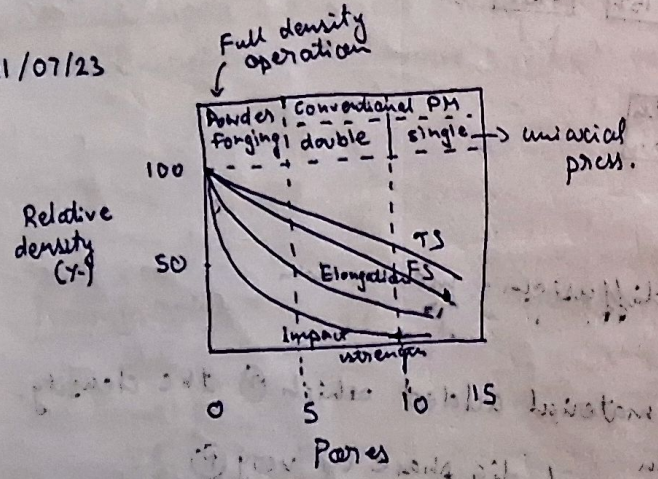
Adv & Disadv of P.M.

1. Elimination/red^n of machining
2. Scrap loss is eliminated/reduced.
3. Maintain close dimensional tolerance.
4. ~~Whit~~ Wide variation in composition
5. Few or no secondary operations
6. Produce good surface finish
7. Non eq^m compos^n are possible.
8. Produce complex/unique shapes which is difficult or impossible to produce with other
9. ~~Coats~~ Provides controlled porosity.

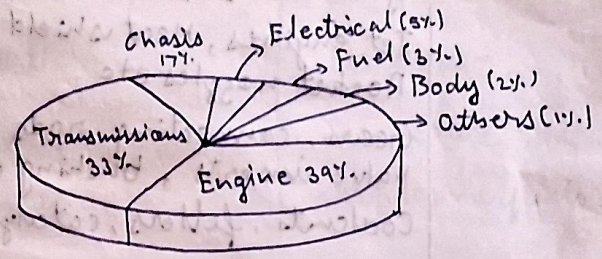
Disadv.

1. High material cost
2. ↑ tooling cost
3. size and shape limits
4. Density variations
5. Health & safety hazards

21/07/23



Application	%
- Automotive	73
- Recreation hobbies hand tools	10.5
- Household appliances	4.3
- Hardware	3.1
- Industrial motors hydraulics	1.9
- Machines	1.2
- Others	6.0



Techniques	Advantages V/S PM	Disadv. V/S PM
Castings	Widely used technique large part size, low setup cost, low tooling cost	Not useful for many materials Chemical segregation Single use molds
Cold forging	Faster production Higher strength Good surface finish	Shorter tool life Limited materials (ductile)
Extrusion	Long parts Smooth surfaces Rapid production	Constant cross section Lower precision, high energy consump^n Simple shape, short tool life
Hot Forging	High mechanical properties large complex shape, fast production, tailored properties	Flash or material waste, poor dimensional control rapid tool wear

Technique	Advantage	Disadvantage
Machining	Widely used in industry Small production vol	Material waste, High cost

Applications of PM

Application	Example / Uses
1. Abrasive	Metal polishing wheels, Grinding media etc
2. Agriculture	Sheet coating, lawn and garden equipment
3. Aerospace	Jet engines, Heat shield Rocket nozzles etc
4. Automotive	Gears, connecting rods valve inserts, bushing etc
5. Chemicals	Coolants, filters, catalysis etc
6. Coatings	Paint, H , Thermal spray barrier
7. Electrical	Contacts, Whack lan, connectors
8. Electronic	Heat sink, ink, microelectronic packages, hardware, locks roaches, cutting tool
9. Heat treating	Furnaces, thermocouples ~~Contact~~ tray conveyor
10. Industrial	Sound absorption, cutting tools, diamond bar etc
11. Joining	Solders, electrodes, weld filler etc
12. Lubrication	~~Greece~~ Grease, lubricant etc
13. Magnetic	Relay, magnets, cores
14. Manufacturing	Dies, tools, bearings, hearth facing etc
15. Medical / Dental	Hip implants, amalgams, forceps
16. Metallurgical	Metal recovery, alloying etc
17. Nuclear	Shielding, filters, reflectors
18. Office equipments	Copies, copier, photocopy process carries
19. Ordanance	Penetrators, Fuses, ammunition
20. Personal	Vitamins, cosmetics, soaps ball point pens
21. Petrochem	Catalysis, ~~reaching~~ drilling bits
22. Plastic	tools, dies,
23. Printing	ink coating, laminates
24. Pyrotechnic	explosives, fuels, coolants

28/07/23

Powder Fabrication Methods

- Chemical Fabrication
- Electrolytic deposition
- Atomization
- Mechanical fabrication

factors of choosing materials –

* process efficiency
* feed stock
* final required powder
* energy input
* contamination

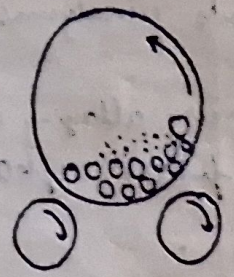
Mechanical Fabrication

- brittle, easy to fracture
- Reactive metals
- Flake powder

4 types

* impactions
* Attritioning
* Shearing
* Compression

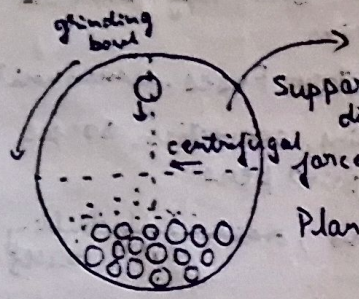
Input energy to create more surface → principle



$\sigma_f = \frac{K_{IC}}{Y\sqrt{c}}$ → Frac. toughness, → flaw size

$\sigma_f \propto \frac{1}{\sqrt{D}}$ → dia of particle

$D \downarrow \sigma_f \uparrow$ centrifugal Ball Mill



Planetary ball mill

- grinding medium
- ball size
- chemical composition
- specific wt.
- hardness
  hardness of grinding medium shd be higher than charge material

* diameter of grinding balls ~ 30 times of charge
* vol. of grinding balls = $\frac{1}{2}$ (Jar vol)
* vol of charge material = 25% of Jar vol.

- proper rotational speed is imp

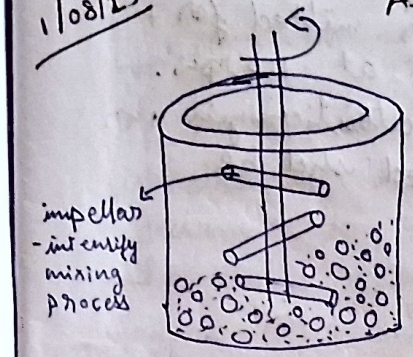
There are 4 fundamental mechanical fabrication processes are available which are impaction, attritioning, shearing & compression. Impaction involves the rapid instantaneous delivery blow to a material, causing cracks and resulting in size redn. Attritioning applies to the redn in particle size by a rubbing motion. Shearing forms a ~~deava~~ cleavage type of fracture by cutting. Powders formed by shearing are coarse in nature and not often found in powder metallurgy, unless the material is extreamly ~~hard~~ hard. Compression forces can break a material into a coarse ~~powders~~ powder if it is brittle. ~~Fo~~ enough

All 4 forces are often combined to form metal powder. For powders in mm size range ~~to~~ hammers mills are efficient but for powders of 1-100 μm ball mills and other intense attritioning devices are more appropriate. Powdery produced by mechanical techniques are typically irregular in shape. Milling is not useful for many ductile materials since they do not easily fracture, insted the ductile particle hold together giving a low process efficiency often in the 1% - 3% range. ~~to~~ Since brittle materials are more responsive it is appropriate to perform milling on oxides prior to redn or certain metals form hydrides that are brittle. These metals can be exposed to H_2, ~~annealed~~ ~~annealed~~ milled and then dehydrated by heating in vacuum. The Nb powder was milled using a hydride technique, giving an angular particle characteristics of a brittle material. The rotation of the jar is adjusted for max impact velocity, fast enough to carry the ball to the top of the jar before falling back on the bed of material being down, too slow a rate will allow the balls to roll back down the jar side while too fast will create a large centrifugal force will keep the balls ~~from~~ falling.

The optimum rotation speed of grinding mill varies with the inverse sqrt of its mill dia. For optimum milling the dia of grinding balls shd be approximately 30 times the dia of charge mate, the volume of balls shd be above $\frac{1}{2}$ of jar vol. and the feed of the balls shd be above 25% of the charge materials That gives a good balance b/w the ball mass and the ball contact. Fluids and protective atmosphere are used to reduce oxidation an ↑ efficiency. Analysis of energy in milling shows tremendous loss in the form of noise & heat. Metal powders form by milling are work hardened irregular, and exhibit coarse flow and packing charac. Other problem are contamination from jars and balls. Milling is widely used to deagglomerate atomize and reduce electrolytic powders after annealing and commonly applied to brittle compounds of borides, carbides and intermetallics.

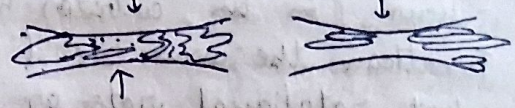
1/08/23

Attritor



Eg:- ODS (imp. for structural application). Matrix of Ni based alloy and Y_2O_3, Al_2O_3 particles dispersed homogeneously.

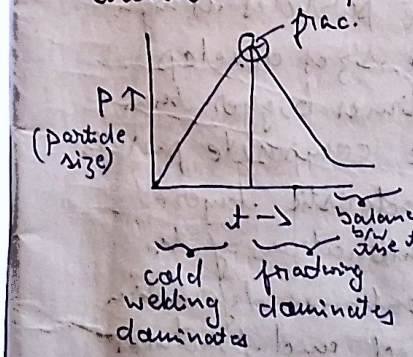
Solid state alloying



- particles are repeatedly flattened, cold welded, fractured or rewelded
- work hardening
- new surface created
- steady state equilibrium is attained.

- reduce only particle size, no transfer of material → milling
- to form new material, true material transfer → mechanical alloying
  → requirements
  material shd be ductile and ceramic

due to strain hardening material becomes brittle and particle size reduces.



milling time

$$t = \frac{c d^2}{N^{1/2}}$$

$d \downarrow \quad t \downarrow$

smaller ball size good for proper mixing.

Milling	Mech. alloying
- Only for ↓ of particle size	- Gen. of new material - New alloy system produced - True transfer of material

Q A Tungsten-Copper powder is milled for 4 hours in an attritor mill at 120 rpm. To attain the same level of attritioning in 1 hr what rotational rate or speed shd be st selected?

Ans During high energy milling the powder particles are repeatedly flattened, cold welded fractured and re-welded. Whenever 2 balls collide some amt. of powder is trapped in b/w them: The force of the impact plastically deforms the powder particles, leading to work hardening and frac. The new surfaces created enable the particles to weld together and this leads to a increase in particle size. Since in the early stages of milling, the particles are soft, their tendency to weld together and form large particles is high. A broad range of particle sizes develop with some as large as 3 times bigger than the starting particle. The composite particle at this stage has a characteristic layered str. consisting of various combinations of the starting constituents, with continued deformation the particles get work hardened and fractures by a fatigue failure mech and by the fragments of fragile flakes. Fragments generated by this mech may continue to reduce in size in the absence of strong agglomerating forces. At this stg the tendency to frac. predominates over ~~welding~~ fracturing. Due to the continued impact of grinding balls the str of the particles is steadily refined but the particle size continue to be same. Consequently the inter layer spacing ↓ and the no. of layers ↑. After milling for a certain length of time, steady state eqm b/w rate of fracturing and welding is achieved which tends to ↑ the avg particle size and the rate of fracturing which tends to ↓ the avg particle size.

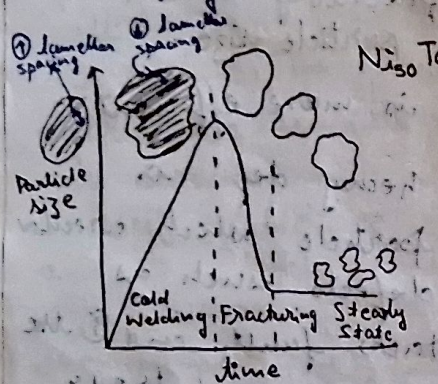
High energy ball milling is more efficient

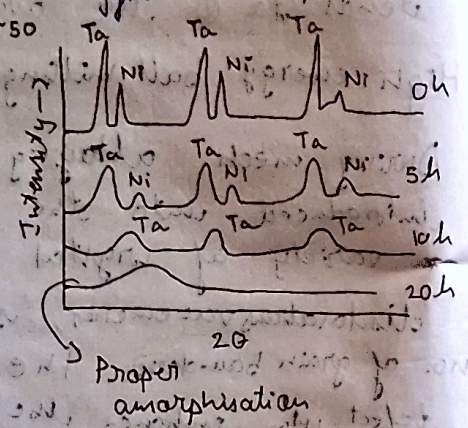
During mech alloying heavy def is introduced into the particle which creates a variety of crystal defects such as dislocations, vacancies, stacks faults and ↑ the no. of grain boundaries. The presence of these defect str enhance the diffusivity of solute into the matrix further refined microstr feature ↓ the diffusion distance. Additionally a slight ↑ in temp during milling further increases the diffusion behavior and consequently the alloying takes place among the constituent element.

4/08/23

Mechanical Alloying

- type of mill
- milling container : dictate the amt. of contamination
- Milling speed
- milling time
- type, size and size distribution of milling medium
- ball to powder wt. ratio : Tungsten carbide balls used
- milling atmosphere
- PCA (Process control agent) : Toluene, Acetone used to reduce cold welding otherwise we will get agglomerated powder
- temp of milling





Mech alloying describes the process when mixtures of powders are milled together. Material transfer is inferred in this process to obtain a homogen alloy. On the other hand milling of uniform comp. powders such as pure metals, intermetallics or pre alloyed powders where material transfer is not req^d for homogenisation has been referred to as mech milling. The raw powders fall into the broad categories of pure metals master alloys, pre-alloying powders and ref. compounds. & Dispersion strengthened materials usually contain addition of carbides, nitrides and oxides. Oxides are the most common and these alloys are known as ODS materials. Ductile-brittle and Brittle-Dri are mixed to produce novel alloys. Occassionally metal powders are milled with a liq media and this is ref wet grinding. If no liq is involved then it is referred to as dry grinding. It has been found that wet grinding is a more suitable method than dry grinding to obtain fines grand pdt. because the solvent molecules are absorbed on the newly formed surfaces of the particles and lowers their surface energy. The less agglomerate condn of the particles in wet condn is also a useful factor. It has been reported that the rate of amorphisation is faster in wet grinding than dry grinding. A disadv. of wet grinding is the ① contamination of the powder.

$Ni_{50}Ta_{50}$

The process was inter after same equal length of time to pull the vial which has been heated up due to the milling. A suitable quantity of the mech alloyed powder was taken out to observe the progress of alloying by XRD and SEM techniques. A plot of particle size vs time show that the size of the powder ① at the begining of milling. The mech alloying process can be classified into 3 distinct

stages
Cold welding, fracturing and the steady state stage respectively. At the cold welding stage there is a sharp ↑ in particle size due to the predominance of cold welding of Ni and Ta elemental powder. In the fracturing stage the particles become harder and the frac of powder becomes dominant resulting in a continuous ↓ of particle size. During the steady stage the balance b/w cold weld and frac is achieved which leads to a relatively const. particle size. A typical lamellar morphology was formed after ball milling of 1 hr. The individual layer thick vary from 0.5 - 4 mm. The lamellas were more highly refine when further milled for 5 hr. After milling for 20 hr, lamellar spacing vanished which indicate that the steady state of M.A process dominated the ball milling proc for the period of 5 - 20 hrs. After M.A. of 5 hr most of brag peaks from pure Ni have already disappeared and the intensity of the brag peaks from Ta have also ↓. The peaks of BCC Ta also broadened asymmetrically towards the large angle side, because of the dissolution of smaller Ni atoms in the BCC lattice. After ball milling for 20 hr only broad diffrac. peaks exist which means that fully amorphous powders were formed.

—X—

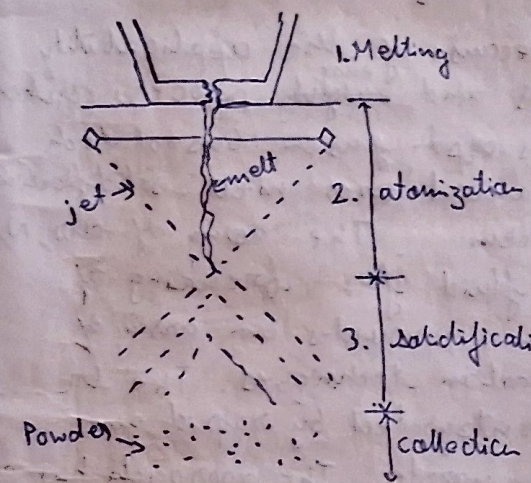
7/08/23

Atomization

Parameters for producing spherical & small powders

- Low melt viscosity
- Small nozzle dia → to avoid premature solidification
- atomizing pressure
- superheat

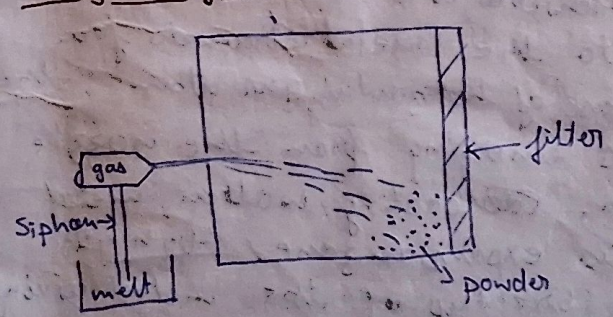
* Gas atomization
* Water atomization
* Centrifugal atomization



1. Atmosphere
   Superheat
   nozzle diameter
   Density
   Surface Tension
   Viscosity
2. Jet pressure / force
   Jet velocity
   Jet geometry
3. Melting range
   Droplet size
   Heat transfer
   Quenching medium
   Flight path

→ spherical powder is produced
- enhance density
- ↓ sintering time

Horizontal gas atomization

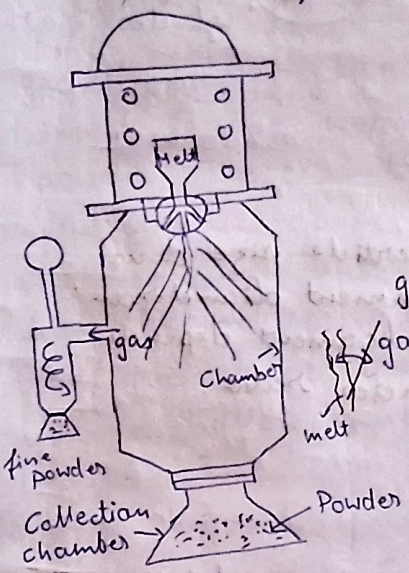


Commercial atomization units operate at production rates as high as 400 kg/min. Prior to the development of atomization, powder chemistry & shape charateristics could not be fully controlled. Now atomization further provides the majority of metal powders. Atomization involves the formation of powder from molten metal using a spray droplet ~~both~~ Both elemental and ~~free~~ pre-alloy powders are formed by such processes.

It is attractive because of the applicability to several alloys and ~~easily~~ easy process control. Atomization depends upon fusion based tech which provides control over melt solidification and alloying chem. The using of air, N_2, He or Argon as a fluid for breaking of molten metal stream, provides an array of versatile powder fabrication techniques. The liquid metal stream is disintegrated by rapid gas expansion out of a nozzle. The approach has proven ideal for Ni based superalloy and many other highly alloyed materials. The design varies wrt the metal feed mechanism and the sofistication of the melting & collection chambers. However the main idea is to deliver energy to the metal stream to form droplets. In case of horizontal gas atom the high velo. gas emerging from the nozzle creates a siphon, ~~cool~~ cooling molten metal into the gas expansion zone. High gas velocity helps breakup of the metal giving a fine spray of molten droplets. During flight through the collection chamber the droplets fast heat & solidify. For some metal a closed inert gas chamber is used to prevent oxidation.

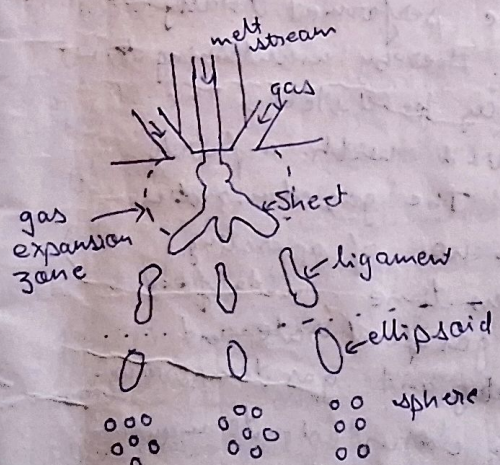
14/08/23

Vacuum induction melter → to avoid oxidation
Al, Ni, Cu, Mg, Co, Fe, Au, Sn, Zn
alloy melting temp. - 1400°c

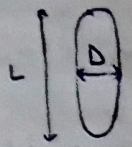


melt temp. - 1550°c
atomization gas - Ar (to avoid oxidation)
gas pressure - 2 MPa
gas flow rate - 8 m³/min
gas velocity, at nozzle exit - 100 m/s
gas-melt impingement angle - 40°
metal flow rate - 20 kg/min
mean particle size - 120 μm

- highly superheated melt
- viscosity free energy
- gas press ↑
- sufficient heigh of chamber



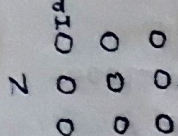
If material is not sufficient superheated then we will get only ligament.



- Surface area remains same
- Ligament is cylinder

$V = \pi \frac{D^2}{4} L$

$A = 2\pi \frac{D}{2} L + 2\pi \frac{D^2}{4}$ → ignore, as ligament is not perfect cylinder.



$V = N \times \frac{4}{3}\pi r^3 = N\pi \frac{D^3}{6} \frac{d^3}{6}$

~~D = 1.5d~~

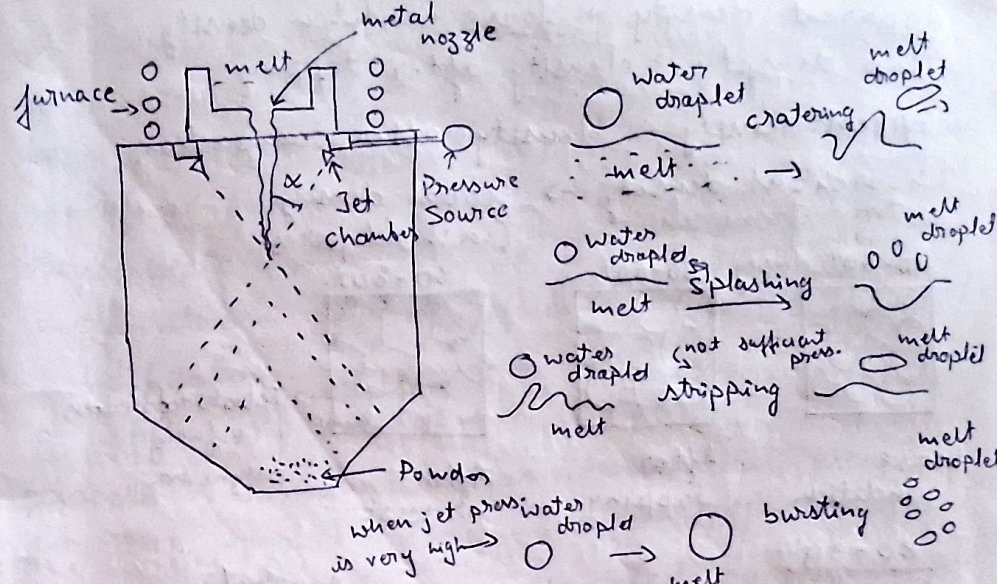
Prove that the final particle size d is 1.5 times the original ligament diameter and the no. of particles per ligament depends on the length over diameter ratio.

d = 1.5 D

Gas atomization can be performed totally under inert conditions. Thereby maintaining the integrity of high alloy feedstock. The particle shape is spherical with a fairly wide size distribution. The gas atomization process has a large no. of operating variables including gas time, metal feed rate, gas pressure, gas feed rate and velocity, nozzle geometry and gas temperature. Those parameters are adjusted to maintain process control and tailor the powder characteristics for various uses. The main adv of gas atomization is in the pdt. homogeneity and the good packing property available with the resulting spherical powders. Gas atomization is capable of production rates as high as 100 kg/min. The press are typically below 5MPa but on a few occasions range upto 18MPa. The technique is applied to alloy of Al, Ni, Mg, Co, Pd, Cu, Fe, Au, Sn, Zn and Be. In gas atomization, the greater the energy input to the metal, the finer the resulting powder. The expanding gas around the molten metal stream causes a dramatic depressurisation and disruption of the melt stream emerging from the nozzle. The depressurization of the gas causes the melt stream to spread into a hollow cone after exit from the nozzle. The thin cone is unstable because of a high surface area to volume ratio assuming sufficient superheat in the liq so that premature solidification is prevent. The liq continues to respond to the surface shear and acc^n deration forces from the gas. The size red^n is limited by the melt viscosity and temp and by the respance to the acc forces superheating the melt above the liquid decrease its viscosity and prolongs the post atomization solidification time. A longer solidification time helps particle spherodization. The droplet shape sequence with dist from the nozzle occurs as sheet, ligament, ellipsoid and sphere. Depending on the amt of superheat & other variable, solidi...

may produce any one of the shape. The gas velocity on exit from the atomizer at the melt superheat are dominant factors in determining the resulting particle size.

- Absolute core size - The max opening in a porous material such as a filter through which no larger particle will pass.
- Acicular powder - Needle shaped powders
- Activated sintering An enhanced sintering process involving a treatment which reduces the activation energy for atomic motion. The ① in sintering rate allows faster sintering, lower sintering temp or improved properties
- Add ~~mixt~~ mixed powder. A ~~sm~~ small discrete powder mixed with another powder called lubrication bonding agent.
- Agglomeration The tendency for the particles to stick together and appear as larger particles
- Alloy powder. A powder in which each particle is composed of the same alloy of two or more metals



Process parameters

Melting temp. → 1365°C

Melt temp. → 1510°C

Metal nozzle dia → 6 mm

Metal flowrate → 22 kg/min

No. of water jets → 8

Water jet apex angle/impinge angle → 38°

Water pressure → 9 MPa

Water flow rate → 230 l/min

Water velocity → 110 m/s

Mean particle size → 60 μm

	G.A.	W.A.
Particle size →	100 μm	150 μm
Particle shape →	spherical	irregular
Agglomeration →	Some	Little
Apparent density →	55%	35%
Cooling rate →	10^4 °C/s	10^5 °C/s
Oxide content →	120 ppm ~~3MPa~~	3000 ppm ~~14 MPa~~
Fluid pressure →	3MPa ~~100 m/s~~	14 MPa 100 m/s
Fluid velo. →	100 m/s	100 m/s
Efficiency →	10 W	moderate

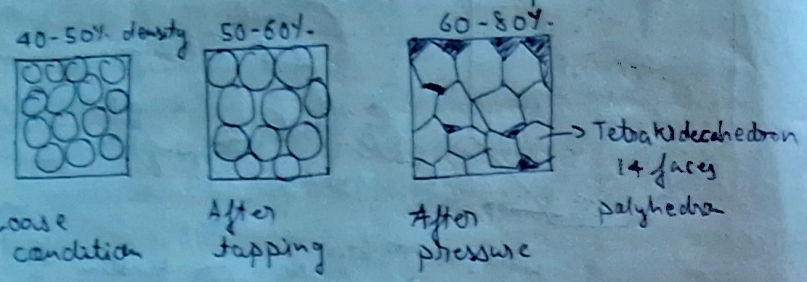
- Jet press. decreases burst → splashing → cratering → stripping

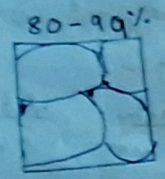
Apparent density -> loose condition density

Tap density -> density after tapping

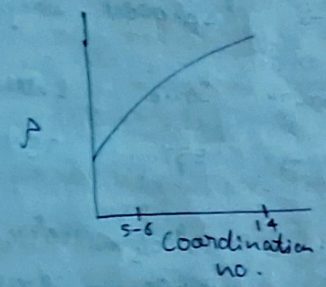
Green density -> density after compaction

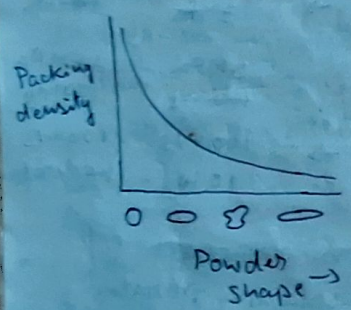
Theoretical density -> Ideal density (without pores)

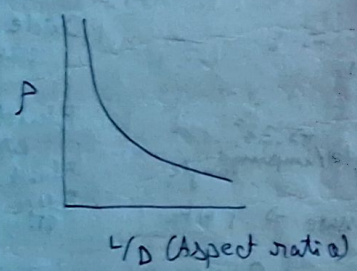




$$\text{Relative density} = \frac{\text{Apparent } \rho}{\text{Theo. } \rho} \times 100$$







packing density depends on L/D ratio

22/08/23

Water Atomisation

Water atomisation is the most common technique for producing elemental and alloy powders from metals which melt below approximately 1600°c. High press water jets are directed against the melt stream forcing disintegration and rapid solidification. The water can be directed by a single jet, multiple jet or annular ring. The process is similar to gas atomisation except for the rapid quenching and differing fluid properties. Particles are generated by cratering splashing, stripping and bursting mechanism. The latter process results in the smallest powder. Typical mass flow rates are abt 5 kg of water for every kg of metal powder. Because of rapid cooling, the powder shape is irregular and rough with some oxidation. Shape control req superheats far above the liquidus. Chemical segregation within an alloy particle tends to be quite limited, since the particles solidify rapidly. Synthetic oils or other non reactive liq can be used instead of water to gain better control of particle shape and oxidation. ~~Syn~~ Pressure is the main process control variable in water atomization, higher water pressure results in higher water velocity and smaller particle size. In one set of experiment steel was atomized at water press of 1.5 MPa giving ~~a~~ ~~Dom~~ 117 um ~~of~~ mean particle size but nearly

Brass 8 fold press increase resulted in a 3 fold reduction in mean particle size. Pressure upto 150MPa have been used in water atomisation resulting in particle in the 5 μm size range.

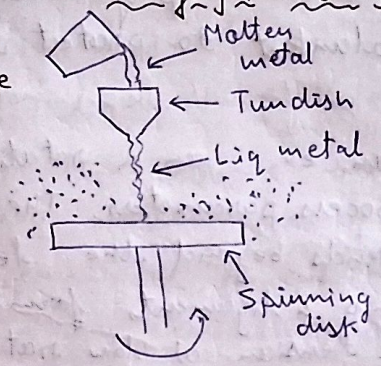
$$D = \frac{\beta \ln P}{V \sin \alpha}$$

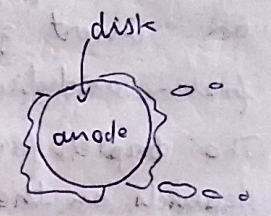
$\alpha \rightarrow$ impinge angle
$P \rightarrow$ pressure
$\beta \rightarrow$ constant

The nozzle to melt distance is less of an influence in water atomization than in gas ato. Water has a much lower compressibility and higher density than gas. Hence distance b/w water exit from the nozzle and impact with the melt stream is a secondary factor. The two outstanding differences are observed in water atomisation are powder shape and surface contamination. In many instances if an oxides coating is detrimental then it can be removed by heating in hydrogen after atomisation. Since the reduction causes particle bonding, subsequent milling may be required to maintain discreate particles. The operating variables for water atomisation have a significant impact on the resulting powder characteristics. In practice atomizing rate as high as 400 kg/min are encountered using velocities upto 230 m/sec. The melt can be the desired final alloy or a precursor composition. In one variant a high C steel with 3.5% C is water atomized, oxidized, milled, decarburized and annealed to form a sponge which is ground to form an iron powder. Other materials produced by water atomization include brass, steel, bronze, Co and Ni alloys and low melting temp metals such as Pb, Sn and Zn alloys.

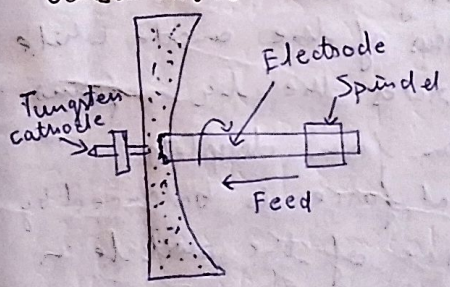
Centrifugal Atomization

- Rotating disk type





- Consumable electrode type



$$D = \left(\frac{A}{\omega}\right)\sqrt{\frac{\gamma}{\rho_m R}}$$

- Tip of electrode is melted

$\gamma \rightarrow$ surface energy
$A \rightarrow$ const
$\omega \rightarrow$ angular velocity
$R \rightarrow$ electrode radius
$\rho_m \rightarrow$ density of melt

The desire to control particle size and the difficulties in aggregating powder from reactive metals have led to the dev of centrifugal atomisation. The centrifugal force throws off the molten metal as a fine spray which solidifies into a powder. The apparatus consists of consumable electrode made from the desired material. The

electrode is melted at its end by a plasma arc or stationary Tungsten electrode. The consumable electrode is the anode and rotates at velocity upto 50K rpm. As melting occurs the electrode is fed into the chamber via an external mechanism. Like gas atomization is performed under an inert gas blanket to protect the powder from oxidation.

The droplet formation ev... on a rotating substrate depends on the process parameters. The liq forms a sheet which extends beyond the lips of the rotating solid. Lump & ligaments form due to shear and surface tension at low rotational velocities and slow melting rate. The particles form directly at the lips of the anode while at very high melting rates the liq forms ligaments which in turn form droplets.

Assuming the centrifugal force is balanced by the surface tension force of the particle on the rim of the rotating device gives a relation b/w expected particle size (D) and the operating parameters

$$D = \frac{A}{\omega}\left(\sqrt{\frac{\gamma}{\rho}}\right)$$

Where
A → process dependent const
ω → angular velo
γ → surface energy of melt
ρ_m → density of melt
R → radius of the electrode

The particle size shifts with approximately the inverse of the rotational velo acc to

Smaller particles are generated with faster rotational rates, larger rotation dia and lower surface energy of the liq. During flight the ligaments pinge into droplets and eventually form spheres. However, if there is insufficient superheat... solidifies with for ligament particle before spheroidal

Generally the particle shape is not and avg particle size of 250 μm is common.

Another empirical formula for mean particle size D is by centrifugal atomization is

$$D = \frac{M_m^{0.12}}{\omega\, d^{0.64}}\left(\frac{\gamma}{\rho_m}\right)^{0.43}$$

M → melt grade
d → anode dia
ω → angular velo
γ → surface energy of the melt
ρ_m → melt density

In terms of process control, the list of variables power, volt, anode dia, ω, and the material involved. Typically the melt rate is of the order of 10^{-7} m³/sec, the rotation velo b/w 1K and 50K rpm and the anode dia b/w 2 and 5 cm. The benefits of a rotating electrode process are the powder cleanness, spherica

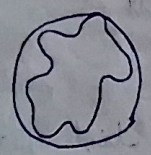
uniform particle size and lack of material contamination. Heavy low production rate, ↑ equipment & processing cost, coarse particle size.

Additionally if a Tungsten cathode is used, there is usually Tu contamination. For this reason Plasma melting is the preferred technique for many application.

Powder Characterization

- Particle size, shape
- Surface area
- Interparticle friction
- Packing
- Compressibility

• Particle size



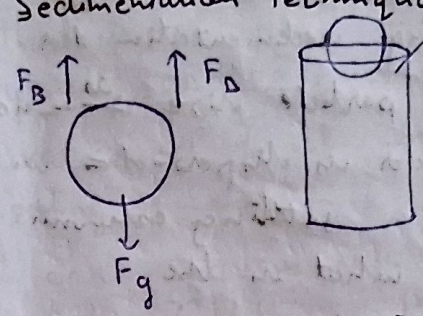
$A = \frac{\pi D^2}{4}$

$D_A = \left(\frac{4A}{\pi}\right)^{1/2}$ → Equivalent area diameter

When powder particle is very irregular

$V = \frac{\pi D^3}{6}$	$S = 4\pi r^2 = \pi D^2$
$D_V = \left(\frac{6V}{\pi}\right)^{1/3}$	$D_S = \left(\frac{S}{\pi}\right)^{1/2}$
Equivalent vol. diameter	Equivalent surface area diameter

Sedimentation Technique



→ Any fluid

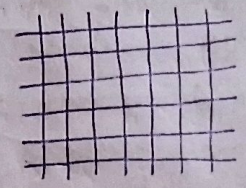
During free falling the particles experience some forces.

At terminal velo. the forces balance each other.

....

Screening Technique

mesh size - no. of wires squares per unit length



Particle size passes through the screen → -ve sign

Limitations

- Cannot determine particle size when particles are very fine

.... Sedimentation techn continue

$$F_g = g\rho_m \frac{4}{3}\pi r^3 = g\rho_m \frac{\pi D^3}{6}$$

$$F_B = g\rho_f \frac{\pi D^3}{6}$$

$$F_D = 3\pi D v \eta$$

At terminal velocity

$$g\rho_f \frac{\pi D^3}{6} + 3\pi D v \eta = g\rho_m \frac{\pi D^3}{6}$$

$$\Rightarrow 3\pi D v \eta = g\frac{\pi D^3}{6}(\rho_m - \rho_f)$$

$$v = \frac{gD^2(\rho_m - \rho_f)}{18\eta}$$

Q A spherical Ni powder is to be analyzed for particle size using sedimentation tech. It is suspected that particle size is near 8 μm. If the powder is dispersed in water at the top of a settling column 100 mm high then what is the expected settling time?

$\rho_{powder} = 8.9 \times 10^3$ kg/m³

$\rho_f = 10^3$ kg/m³

$\eta_f = 10^{-3}$ kg/m sec

$$v = \frac{g D^2 (\rho_m - \rho_f)}{18 \times \eta}$$

$$= \frac{9.8 \times 8 \times 10^{-6} \times 8 \times 10^{-6} (8.9 \times 10^3 - 10^3)}{18 \times 10^{-3}}$$

$$= 34.84 \times 10^{-9} \times 7.9 \times 10^3$$

$$= 275.271 \times 10^{-6}$$

$$= 2.752 \times 10^{-4} \text{ m/sec}$$

$$\text{time} = \frac{100 \times 10^{-3}}{2.752 \times 10^{-4}} = \frac{1000}{2.752} = 363.3 \text{ sec}$$

A

1. What is splat cooling and what are typical cooling rates observed in splat cooling? What kind of str. result from splat cooling of metals

2. In case there is some additional uniform agitation facility in the melt bath. Will it have the same effect on the morphology of the atomized metal powders?

3. Inert gas atomization is an ideal process to produce spherical powder particles However the process is costly. In case it is req^d to produce similar shape powder by much cheaper water atomization what special care will have to be taken.

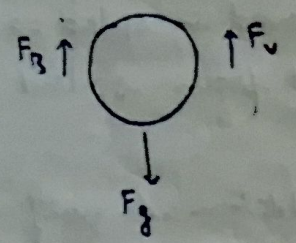
4. Nitrogen added ice pit steel have better wear resistance both abrasive and adhesive. Which method shd. be applied to produce powder of such steels?

1. Splat cooling - is a metallurgical, metal morphing technique used for forming metals with a particular crystal str. by means of quenching

Cooling rate = 101 - 104 °C/sec

28/08/

Sedimentation technique



$$v = \frac{g D^2 (\rho_m - \rho_f)}{18\eta}$$

$$v = \frac{R_N \eta}{D \rho_f}$$

$\geq$ 27 μm sedimentation

$<$ 27 μm DLS

$$\Rightarrow \frac{g D^2 (\rho_m - \rho_f)}{18\eta} = \frac{R_N \eta}{D \rho_f}$$

$$\Rightarrow D^3 = \frac{18 R_N \eta^2}{g \rho_f (\rho_m - \rho_f)}$$

$$D = \left(\frac{18 R_N \eta^2}{g \rho_f (\rho_m - \rho_f)}\right)^{1/3}$$

Q Tungsten spheres settling in air. What is the max particle size for which sedimentation is valid?

$R_N = 0.2$

$\eta_{air} = 1.8 \times 10^{-5}$ kg m^{-1} s^{-1}

$g = 9.8$ m s^{-2}

$\rho_f = 1.2$ kg m^{-3}

$\rho_{Tung} = 19.3 \times 10^3$ kg m^{-3}

10^{-10}

$$D = \left(\frac{18 R_N \eta^2}{g \rho_f (\rho_m - \rho_f)}\right)^{1/3}$$

$$= \left(\frac{0.991 \times 10^{-10}}{(19300 - 1.2)}\right)^{1/3}$$

$$= \left(\frac{0.991 \times 10^{-10}}{19298.8}\right)^{1/3} = (5.1 \times 10^{-15})^{1/3}$$

$$= 1.8 \times 10^{-5}$$

If water is selected instead of air, what is the max. particle size

$\eta_{water} = 10^{-3}$ kg m^{-1} s^{-1}

$\rho_{water} = 10^3$ kg m^{-3}

$$D = \left(\frac{18 \times 0.2 \times (1.8 \times 10^{-5})^2}{9.8 \times 10^3 (18.3 \times 10^3)}\right)^{1/3}$$

$$= \left(\frac{1.19 \times 10^{-5}}{18.3 \times 10^6}\right)^{1/3}$$

$$= (0.065 \times 10^{-11})^{1/3}$$

$$= (6.5 \times 10^{-9})^{1/3}$$

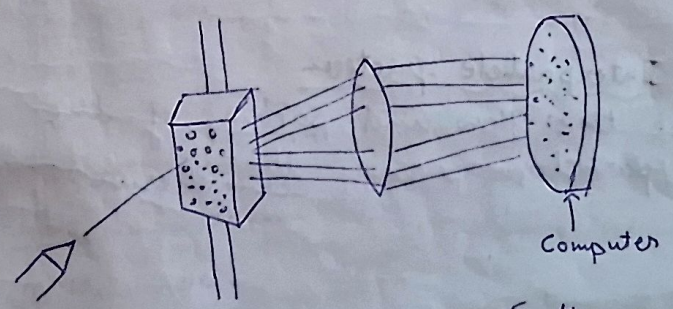
$$= 1.8 \times 10^{-3} \text{ m}$$

$$= \frac{18 \times 0.2 \times 10^{-6}}{9.8 \times 10^3 \times 18.3 \times 10^3}$$

$$= (0.02 \times 10^{-12})^{1/3}$$

$$= 0.27 \times 10^{-4}$$

$$= 27 \text{ μm}$$



Scattering angle $\propto \frac{1}{\text{Particle size}}$

Scattering intensity $\propto (\text{Particle size})^2$

Typical application of light scattering are the characterization of particles, emulsions or molecules which have been dispersed / dissolved in a liq.

The brownian motion of particles or molecules in suspension causes light to be scattered at diff intensities. Analysis of these intensity fluctuations yields the velocity of the brownian motion and hence the particle size using the Stoke-Einstein relationship. Dynamic light scattering technique adv -

Fast, accurate, reliable & repeatable particle size analysis in 1 or 2 mins. Mean size only requires knowledge of the viscosity of the liquid. Simple or no sample preparation, high concentration can be measured directly. Simple setups & fully automated measurement, low vol requirement, size measurement of sizes <1nm is possible.

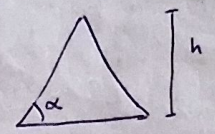
Interparticle friction

Influence the flow and packing of the powder particles.
semicohesive

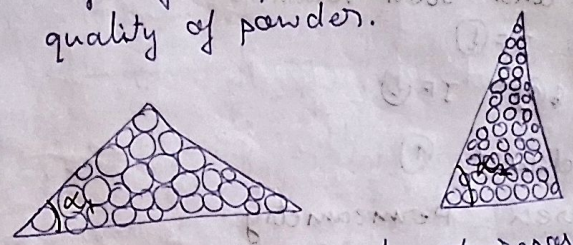
- How to determine interparticle friction
- Apparent density
  S.A. ↑ interparticle fric ↑

- Tap density
  interparticle friction ↓ Tap density ↑

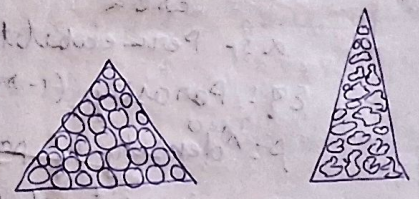
- Angle of repose
  $\mu = \tan\alpha$



Angle of repose is influenced by the quality of powder.



S.A. ↑ ⇒ steep angle of repose



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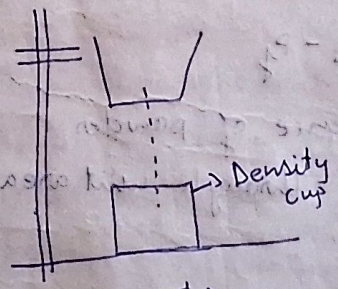
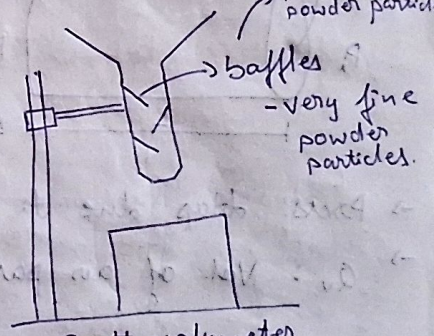


Hall flow meter

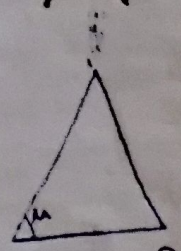


Scott volumeter

- Interparticle friction influence the flow and packing.
- Easy flow ⇒ ↓ interparticle friction
- Interparticle fric. ↓ ⇒ Coordination no. ↑
- S.A. ↑, I.F ↑, App. ρ ↓

How to determine interparticle prop.?

1. Angle of repose



μ(↓) IF(↑)

2. Hall flowmeter and Scott volumeter

flow time (↓) IF(↓)

App density (↑) IF(↓)

IF (↓) Tap density (↑)

Surface area Permeametry

$$S_w = \frac{1}{\rho}\sqrt{\frac{1}{5\alpha}\frac{\varepsilon^2}{(1-\varepsilon)^2}}$$

$$Q_v = \alpha \frac{\Delta P}{L}\frac{1}{\eta}$$

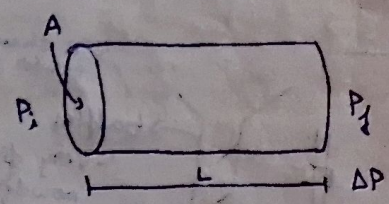
S_w :- Specific surface area

α :- Permeability coeff

ε : Porosity (1 - relative ρ)

ρ : Theo density of powder

η : viscosity of fluid



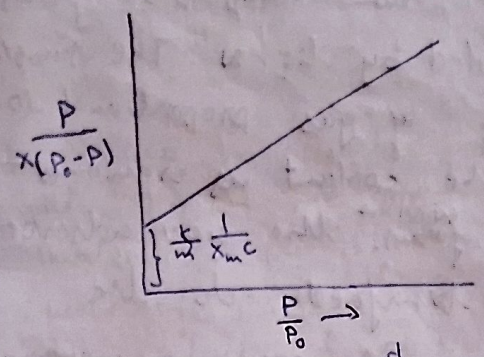
$\Delta P = P_i - P_f$

→ Press. drop due to presence of powder

→ Q_v : Vol. of air passing through unit area

Brunauer Emmet Teller (BET)

$$\frac{P}{X(P_0 - P)} = \frac{1}{X_m C}\left[1 + \frac{P}{P_0}(C-1)\right]$$



X = amt of gas adsorbed

X_m = amt of gas necessary to form a saturated surface coating 1 atm layer thick

P_0 = Saturation press. of adsorbent

P = Partial press " "

C = Const. related to adsorption enthalpy

$$S = \frac{X_m A_0 N_0}{W V}$$

S → surface area

N_0 = Avogadro's no.

A_0 = Avg. area of an adsorbent molecule

V = Molar volume

W = Sample wt.

In order to determine specific S.A. by the BET method, the amt of gas adsorbed in a mono molecular layer on the surface of the powder and the area occupied by the adsorbent gas molecul and the adsorbent gas known

Before measurements are made, gas adsorbed on metal powder must be desorbed by heating the sample in the vacuum.

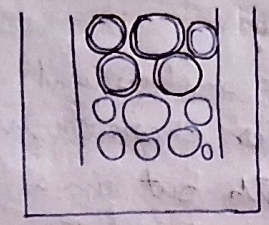
A gas stream of known composition flows over a sample cooled by liq N. The sample adsorbs a quantity of gas proportional to its S.A. When the coolant is removed and the sample warms, the gas adsorbed is dissolved and transferred to the flowing gas stream. Its conc. is measured. Repeating this produces the data need for a std BET plot to determine the S.A. of the sample. Basic concept: Total S.A. can be determined by the amt of gas req^d for the formation of an adsorbed gas monolayer, x_m. x: amt of gas adsorbed by the powder under press P.

P_0: sat. press of gas at sat temp T

C: const

Plotting the expt data $\frac{P}{x(P_0-P)}$ vs P_0 shd be a str line with slope $\frac{C-1}{x_m C}$ and intercept is $\frac{1}{x_m C}$

x_m & C can be determined from the plot.



Q N gas is adsorbed on an alumina powder at 77.4 K. The area occupied by an adsorbed molecule is 0.163 nm^2 at STP. The vol of a mole of gas 2.24×10^4 cm^3 and vol of gas req^d for monolayer adsorption of 1g of powder is 2.52 cm^3. Calculate the specific surface area of the powder.

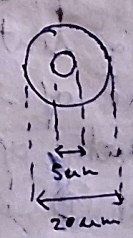
$S = \frac{A_0 X_m N_0}{W V}$ $SSA = \frac{TSA}{\text{mass of sample}}$

Q A spherical 20 μm particle of Ti has a 5 μm spherical void in its centre. What will be the terminal centric velo of this particle in water. How does this compare to terminal velo. of a pore free particle of the same dia

$\rho_{water} = 1 g/cc$

$\eta = 10^{-2} g/cm/sec$

Theo ρ of Ti = 4.5 g/cc



$$v = \frac{D^2 g (\rho_m - \rho_f)}{18 \eta}$$

• With void

$$D_{eff} = \left(\frac{6V}{\pi}\right)^{1/3} = \left(\frac{6(V_{sphere} - V_{void})}{\pi}\right)^{1/3}$$

4/10/12

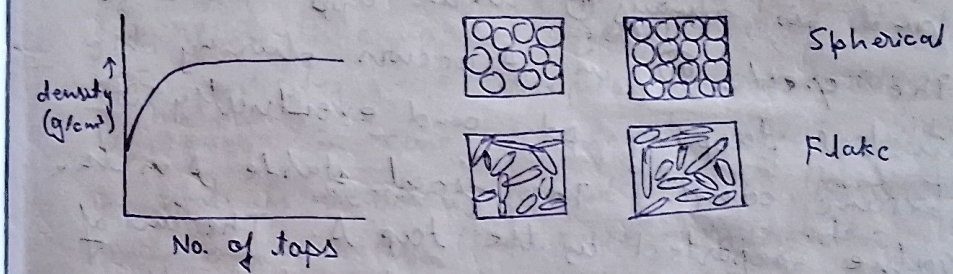
Particle surface Topography

The nature of the surface of the individual particles is also an imp powder characteristic. Spherical particle may appear smooth but on a closer examination at high magnification the surface may actually be rough. Reduced metal powder has a highly roughened surface. Atomized metal powder on the other hand have finer degree of surface roughness which are of granular kind rather than sharp & rough.

S.E. microscope is a powerful tool for examining surface topography. Surface contamination of the particles and agglomeration of fine particles can also be studied by this technique.

The exact nature of S.T. is influenced by friction forces b/w particles. These are imp in the case of bulk movement of the particles when powder is flowing, during settling or compaction. The extent of actual particle to particle contact during sintering will also be affected by the nature of the surface roughness. Chemical reactivity of the powder also tend to ↑ with ↑ in surface roughness especially the irregular type.

Material	Apparent density (g/cm³)	Tap density (g/cm³)	(%) increase
Copper			
Spherical	4.5	5.3	18
Irregular	2.3	3.14	35
Flake	0.4	0.7	75
Iron			
Electrolytic	3.31	3.75	13
Sponge	2.29	2.73	19



Apparent & Tap

The app density of a powder refers to the mass per unit vol. of loose powder usually expressed in g/cm³. It is one of the most critical characteristics of a powder, because of some reasons:

1. It determines the size of the compaction tooling and magnitude of the press motion necessary to compact & densify the loose powders.
2. It determines the selection of equipment used to transport & treat the initial powder
3. It influences the behaviour of the powder during sintering.

Other characteristics which have direct

an app. density are of the solid mate, particle size & shape, surface area, topogr & its distribution. Tap density is determined by the Hall flowmeter where a container of known vol. is completely filled by flowing metal powder through a hall funnel. very often a mass of loose powder is mechanically vibrated or tapped. The density of the loose powder ↑ due to this treatm and is always ↑ than app density. The greater ↑ in ρ occurs during the initial tapping pd. and eventually it become const. The final stable ρ is the value reported as the tap ρ. The amt of ↑ in ρ due to tapping depends on the extent of the original fric forces b/w the particles. The greater the fricti cond of the origin powder due to ↑ size, irregul shap & rough surface, the greater the ↑ in the ρ due to tapping.

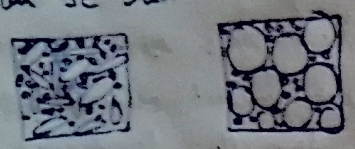
Flow Rate

Rapid rates of P/M part produc^n req a relatively rapid flow of powder from storage containers to dice. The std method for the determination is by the Hall flowmeter where the time necessary for 50 g of powder to flow through a prescribed small orifice are therefore proportional to the reciprocal of the flow rates. Very fine powders do not flow through a small orifice, this is a result of the drastic ↑ in the specific S.A. as the size becomes very small. For a given metal powder the ↑ the app. ρ, ↓ the flow time. When a fine size powder is mixed in a coarse powder because of the ↑ in the app. ρ the flow time is ↓ irrespective of whether the particles were irregular or spherical. However, in case of irregular powder addition an amt. is reached for which no flow behaviour is observed. This corresponds to the presence of an excessive amt. of friction and S.A.

Powder	ρa (g/cm³)	Compaction Pressure (MPa)	ρg (g/cm³)	Green Strength (N/mm²)
Sponge	2.4	415	6.2	2100
		550	6.6	3200
		690	6.8	4100
Atomized	2.5	414	6.55	1900
		550	6.80	2700
		690	7.0	-

Densification Parameter $= \frac{\rho_g - \rho_a}{\rho_T - \rho_a}$

To improve ρ_a, two types of powder particles can be taken



Bimodal particle distribution b/w coarse & fine particles

- A ρ_a is imp for design purpose.

—x—

5/09/23

Compressibility

Densification parameter $= \frac{\rho_g - \rho_a}{\rho_T - \rho_a}$

imp. for tool design

$C_R = \frac{V_L}{V_c} = \frac{\rho_g}{\rho_a}$ ↑ ρ_a ↓ interparticle friction

Compressibility is a measure of ability of a material to which a powder will compress or densify upon application of external press. It is reported as the density in g/cm³. ~~Type~~ Typically a cylindrical or rectangular test piece is made by pressing powder in a die with press. applied simultaneously from top & bottom. It is influenced by factors like invariant hardness of the concerned metal or alloy, particle shape, internal porosity, particle size distribution, presence of non metallics, addition of ~~non~~ alloying elements or solid lubricants

It is defined in terms of densification parameter

$$D.P. = \frac{\rho_g - \rho_a}{\rho_T - \rho_a}$$

I. Compressibility in general ↑ with ↑ in in of ρ_a. Another term which is imp. in for tool design is the compression ratio, It is the ratio of the volume of loose powder to the vol. of a compact. A low compression ratio is desireable because

i) Size of the die cavity and tooling can be reduced

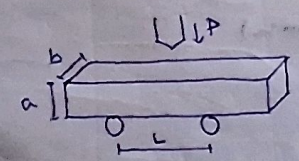
ii) Breaking and wear of tool is reduced.

iii) Press motion can be reduced

iv) A faster die fill and higher production rate can be achieved

Green Strength

Transverse / 3 point bend test



$\sigma_g = \frac{3PL}{2bd^2}$

L → supporting length

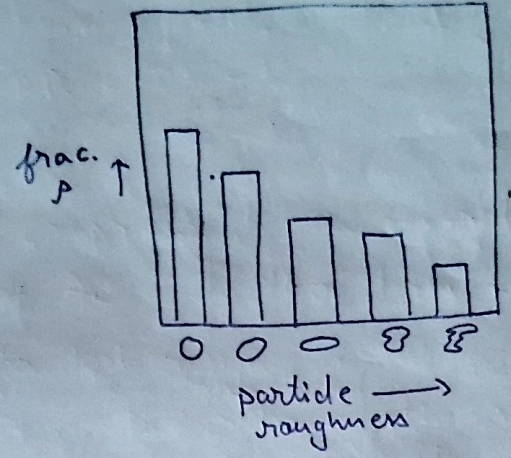
Green strength is the mechanical strength of green (unsintered) powder compact. This characteristic is very imp as it determines the ability of a green compact to maintain

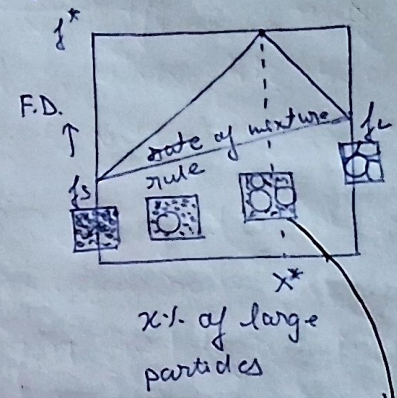
its size and shape during handling prior to sintering. It is promoted by (1) particle surface roughness since more sides are available for mechanical interlocking. It is promoted by (2) in powder surface area. It is promoted by (3) the amt of certain interfering additives for eg. the addition of small amt. of alloying elements such as soft graphite to iron and lubricants prevents metal interlocking. The std. green strength test is a transverse grain test of a rectangular specimen and it is the stress calculated from the ~~Trac~~ Flexure formula req. to break the specimen acc to. $\sigma_{fg} = \frac{3PL}{2bd^2}$ is the

where P: breaking load (N)
L: supporting distance
d: specimen thickness (mm)
b: " width (mm)

Pyrophorasity and Toxicity

Pyrophorasity is a potential danger for many metal for including the most common types when they are in a finely divided form with large surface area to vol. ratio. The toxicity of powders is normally related to inhalation and ingestion and result in toxic affect





$x^* = \frac{f_L}{f^*}$

$f^* = f_L + f_s(1-f$

Bimodal distribution is beneficial for achieving higher F.D.

19/09/23

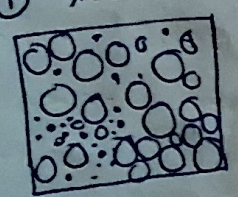
Mixing & Blending

- Unique particle size ~~distance~~ distribution
- Combine powder to generate new alloy
- Add lubricants and binders → provide green strength → added during mixing
- Prepare powder binder mixture for shaping
- *Remove segregation
  - Particle size ~~shape~~
  - density → ↑ density material present at bottom.
  - shape

• Coeff of segregation

$$C_S = \frac{X_T - X_B}{X_T + X_B}$$

ρ↑ hardness ↑



X_T : amt. of powder present at the top of the container

X_B : at the bottom of the container.

- If particles are not distributed uniformally, only some portion of it might produce the desireable alloy.

→ $ZrB_2 - SiC$
↓
ZrO_2 (removed by B_4C)

$A + B$

$W_A \quad W_B$

$\rho_A \quad \rho_B$

Theoretical density of mixture

$$\rho = \frac{W}{V} = \frac{W_A + W_B}{\frac{W_A}{\rho_A} + \frac{W_B}{\rho_B}}$$

Mixing & blending are necessary to prepare during particle size distribution combine powde to generate new alloys during sintering add lubricants for compaction and to prepare a powder - binder mixture for shaping. A major reason for blending powders is to remove segregation typically induced by vibration in transport. There are 3 causes of seg which are - diff in particle sizes, ρ and shape. Size segregation is dominating. For eg- a powder will be segregated by size if the small particles can pass through the voids b/w the large particles. One constituent of size segregation is that overall appearance ρ will ↓ and there will be a point to point density variation. The degree of seg can be expressed thru C_S

$$C_S = \frac{X_T - X_B}{X_T + X_B}$$

where X_T is the frac. of large particles in the top half of the container and X_B is the frac of larg particles in bott

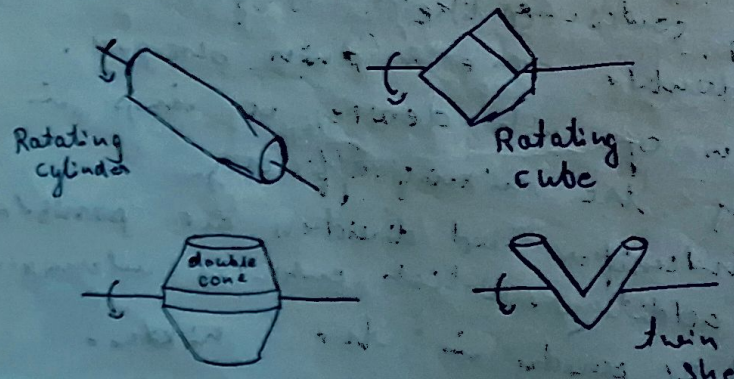
An irregular particle ~~size~~ shape distortion will inhibit size distribution. ① seg occurs with particle sizes ~100 μm because of the greater interparticle friction. Size seg can be ① by using wetting agent through agglomerates created by can contribute to segregation. Mixing process are mostly understood & are a common source of problems in fabrication. The variables involved in blending or mixing powder into the material particle size mixture time, size, relative powder vol. in the mixture, speed of mixing, shear time of mixing etc. Additionally environmental factors such as humidity influence mixing.

Some simple rules to avoid segregation are
- reblend a dry powder after transport
- do not vibrate a dry powder
- do not feed dry powders through a free fall where sizes can segregate
- minimize unnecessary shear for powder-binder mixture.

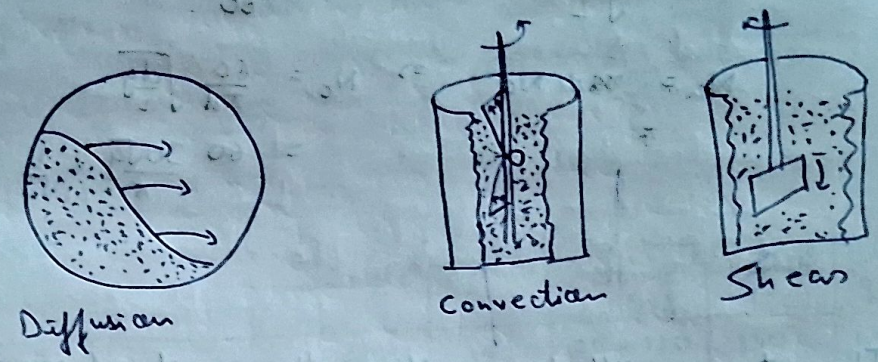
Dry Powder Mix

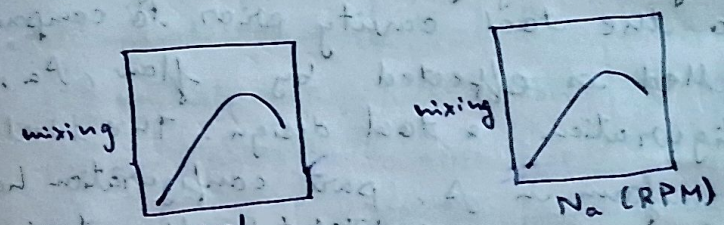
The mechanism for powder mixing are diffusion, convection and shear. Diffusion mixing occurs in a rotating drum, convective mixing occurs in a screw mixture and shear mixing occurs in a blade mixture. The diffusional mix occurs by the motion of individual particles into the powder bed and implying plane of the powder bed breaks down at the outer edge allowing flow over the surface. Continuous rotation of the drum provides the inclined with fresh powder giving diffusional intermixing of the particles. Convective mixing refers to a transfer of adjacent powder to from one location to another. The screw cuts off a small grp of particles from the lower surface & transport them elsewhere in the batch. Shear mixing occurs by continuous and flow of the powder over slip planes. Most mixer powder mixing & blending is performed in rotating containers like rotating cylinders, rotating tubes, double cone & twin shaper. The interior of such devices is imp in determining the mixing efficiency. Baffles intensifier and dividers are possible internal additions which enhances intermixing. The vol of powder in the mixture

mixing efficiency. As the mixture becomes filled with particles, the relative motion inhibited. The powders had to be 20 and 40% of the mixture capacity is usually optimum. The rotating speed also has a large effect on efficiency. A slow rotation with prolonging time necessary for adequate mixing. An excessive free fall of the powder in the mixture will cause preferential size & impact but rapid a centrifugal force to the powder which interferes with flow. Best mixing occurs when centri forces are ⊕ but not so small that turbulence does not occur. A desireable rotational speed is one which balances grav and centrifugal forces. The optimum rotat speed for a cylindri mixture $N_a = \frac{32}{\sqrt{d}}$. Optimum mixing of powder occurs when mixture is run approximately 75% of critical rotation speed.



9. To prove that optimum rotational speed for cylindrical mixture is $N_a = \frac{32}{\sqrt{d}}$.





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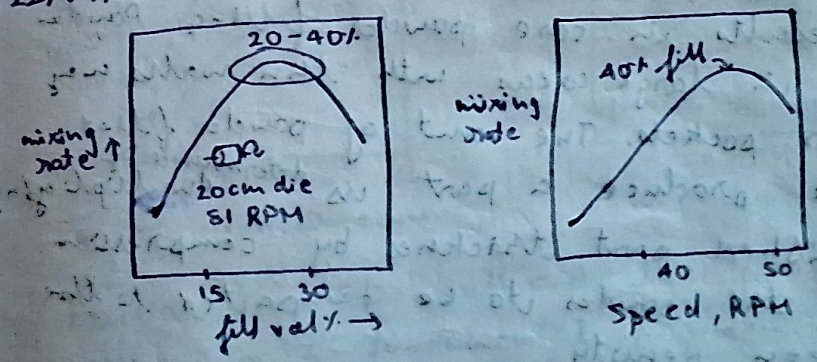


$75\% \cdot N_c = N_a = \frac{32}{\sqrt{d}}$

N_c → critical velo (when centrifugal and gravitational forces balance each other)

N_a → optimum velo

$$\frac{2mv^2}{d} = mg \qquad (F_c = F_g)$$

$$\Rightarrow \quad v = \sqrt{\frac{dg}{2}} \quad \& \quad N_c = \frac{\pi d N_c}{60}$$

$$N_a = 75\% \, N_c \qquad \Rightarrow \quad N_c = \frac{60}{\pi d}\sqrt{\frac{dg}{2}}$$

$$= 60 \, \frac{30\sqrt{g}}{\pi} \times \frac{1}{\sqrt{d}}$$

The terms filled signifies the amt. of powder taken into the tool cavity prior to compaction. Powder filled is effected by flow, ρ_a, part configuration & tool design. The ideal fill shd be uniform in ρ. part configuration has direct influence on the powder filled into the tool cavity.

Thin walled parts have narrow cavity section which results in core powder filling. Powder filling in large areas with thin walls may trap air pockets. The amt of powder filled req to produce a part is determined by multiplying by finished part thickness by compression ratio of the powder to be compacted to the req green density.

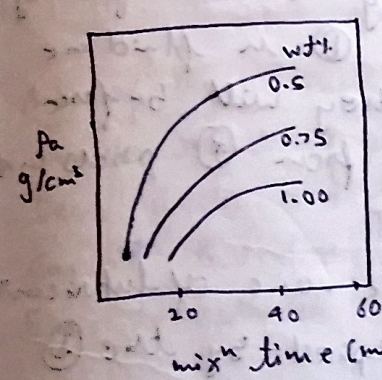
$$\frac{\text{Height of filling capacity}}{\text{Compact H}} = \frac{\rho_g}{\rho_a} = \text{fill vol.}$$

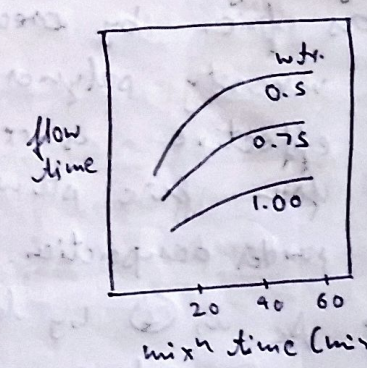
Q The ρ_g for S.S. powder 6.5 g/cc. The ρ_a is 2.7 g/cc. What is the compression ratio & req powder ~~free~~ fill ht. for a final ht of 4 cm.

$$\frac{H}{4} = \frac{6.5}{2.7} \qquad CR. = \frac{6.5}{2.7} = 2.40$$

$$H = 9.63 \text{ cm}$$

	Oxide	% oxide	Softening temp (°C)	melting temp (°C)	ρ g/cm³
Zinc stearate	ZnO	14	100 - 120	130	1.09
Calcium stearate	CaO	9	115 - 120	160	1.05
Lithium stearate	Li_2O	5	195-200	220	1.01





Friction b/w die wall and the powder during pressing is a fundamental problem. As the compaction press is ↑, ejection of the powder mass from the die become difficult. Consequently lubricants used to ↓ die wear and ~~ease~~ easy ejection. There are 2 means of lubrication

- Die wall and powder lubrication.
- Die wall lubrication is preferred in the easy

but not easy to incorporate into automatic compaction equip. thus lubricants needs to be material powder as a final step before pressing. Typical conc are b/w 0.5 & 1.5 wt%. For metal powders stearate powders based on Zn, Al, Li, Mg or Ca are in common use. The molecular chains contain 12-22 C atoms. The chains are surface active & melted relatively ① temp. The stearate is added to metal powder as a small spherical form. A mean size of 10-30 μm is common. Besides the stearates, other lub cellulose additives, wax may be used. During def. the lub. form a fluid that lowers fric. by creating a thick film of high viscosity polymer. ① in fluid are not effective since they will be force away from fric points from ② press used in powder compaction. T

The μ_s is ① by large conc of lubricants because ① vol is occupied by the ① μ of lubricant. The flow time is ① when the amt of lubricant is ①. In the presence of wate lubrication can lower the friction.

25/09/

Negative aspects to the mixing & blending

1. Metallic particles will work harden with prolonged mixing and will become more difficult to press.
2. An improperly designed mixing cycle can cause particle segregation with becomes more of a problem as the size, shape and ρ b/w the two powders differ.
3. During mixing attritioning of the powder is possible hence smaller particle sizes and rounded shape can result from prolong mixing. Coating can develop in mixing of hard & soft phases with the soft phases causing agglomeration or spreading on the hard particle surface.
4.

Adv of dry mixing:-

1. Avoids reach of powder with liq medium
2. Less media and lining wears than wet mixing.
3. Can be started or stopped any time.
4. Easier to optimize.

Adv of wet mixing

1. Low power require
2. No dust problem
3. Higher rotational speed
4. Good homogenisation
5. ① particle size than dry mixing
6. Narrower particle size distribution

W_L = wt of lubricant
f = frac. ρ of compact
ρ_L = ρ of lubricant
ρ_P = ρ of powder

$$W_L = \frac{(1-f)\rho_L}{\rho_L(1-f) + \rho_P f}$$

Prolonged mixing (Disadv)

1. Work hardening
2. Material gets brittle and particle size ↓
   For creep, smaller particles are not desireable

- Some powders generates heat when dry mixed
- Lubricants ↓ ρa. Hence we cannot add more of it.

Q Zn stearate with a theo ρ of 1.09 g/cc is used to lubricate Cu powder having ρ 8.9 g/cc. If the final frac ρ of Cu compact is 85% of theoretical then what is the max. lubricant content?

$$W_L = \frac{(1-0.85)\,1.09}{1.09(1-0.85) + 8.9 \times 0.85}$$

$$= \frac{0.1635}{0.1635 + 7.565}$$

$$= \frac{0.1635}{7.7285} = 0.02\,g$$

Mixing is the first step in the preparation of a powder binder feed stock for shaping. The main purpose of binder is to provide strength to the moulded green body. Various polymers and waxes are used as binders. The binder is a temporary vehicle for packing a powder into the desired shape and holding the particles in that shape until the begining of sintering. Although the binder shd not the finer composition of the moulded material/powder. It has an influence on the process. The binder is mixed with the powder to form feed stock. Unfortunately no binder is perfect so selection of an appropriate binder varies b/w situation. Most binders are multi component mixtures that contain a major ingredient that dictates the basic properties along with additives used to tailor the properties. After shaping the binder holds the particles in place until the particles are heated in sintering. Finally the binder is eliminated without contaminating the powder.

Desired binder characteristics

1. ~~Under~~ flow characteristics
   i) ⓘ inherent viscosity at the shaping temp
   ii) Strong and rigid after shaping
   iii) Low molecular wt.

2. Powder interaction
   i) Wets and adhere to powder
   ii) Chemically passive
   iii) Stable during mixing & shaping
   iv) B

3. Binder burn out
   i) Non corrosive,
      Non toxic decomposition pdt.
   ii) No residue on burnout
   iii) Decomposition temp above mixing temp
   iv) Decomp below sintering temp.

4. Manufacturing
   i) Inexpensive, available, safe & low cost
   ii) ⓣ strength & stiffness
   iii) ⓓ water absorption, ⓓ volatile components.

• Examples of binders

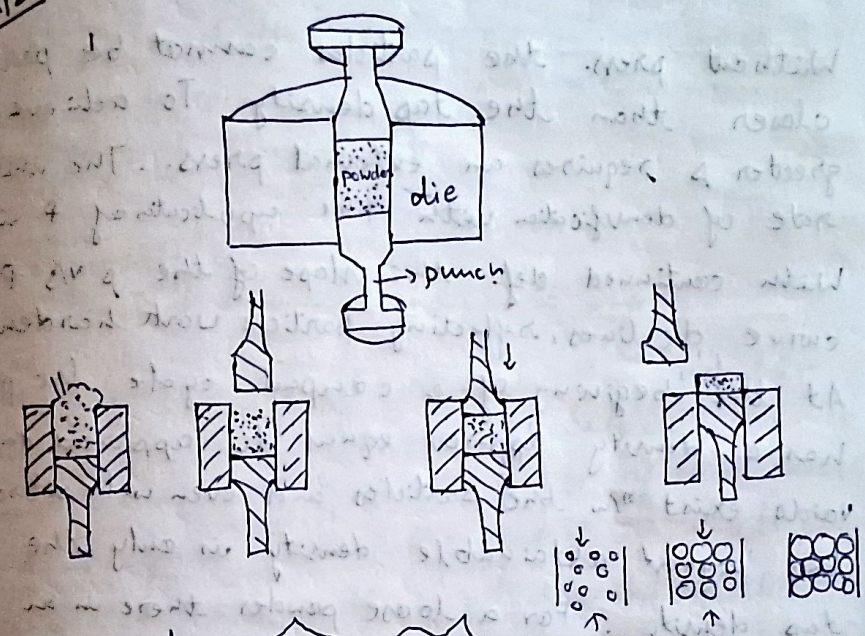
* Extrusion
  → 56% H_2O, 25% Methyl cellulose, 13% glycile
  6% boric acid.

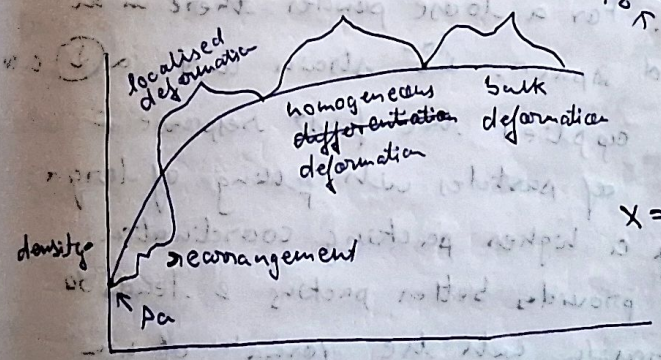
* Injection moulding
  → 75% , 25% polyethylene,

* Slip casting
  97% H_2O, 3% polyvinyl alcohol — Diff b/w lubricant & binder

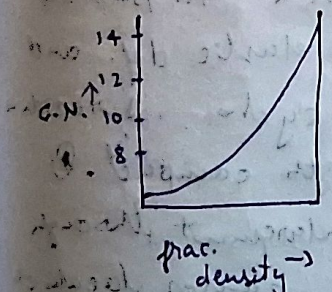
* casting
  80% toluence, 13% PEG, 7% polyvinyl ...

26/09/23



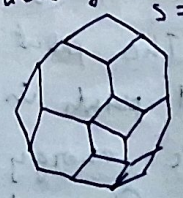


$$X = D\left[1 - \left(\frac{\rho_0}{\rho}\right)^{\frac{2}{3}}\right]^{1/2}$$

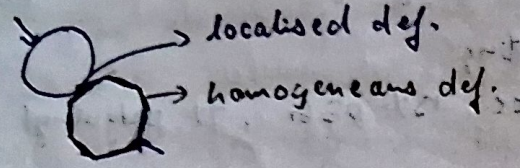


$N_c = 14 - 10.4\,\varepsilon^{*}$

Tetrakaidecahedron
100% density
e = 36
H = 8
S = 6



homogeneous deformation
total powder particle is deformed



Without press. the particles cannot be packed closer than the tap density. To achieve greater ρ requires an external press. The initial rate of densification with the application of P is ↑. With continued def. the slope of the ρ v/s P curve declines, reflecting particle work hardening. At the begining of a compact cycle the powder has a density approx equal to apparent density, voids exist b/w the particles and even with vibration the highest attainable density is only the tap density. For a loose powder there is an excess of void space, low strain and a ↓ C.N.

As press. is applied the first response is rearrangement of particles with filling of larger pores, giving a higher packing coordination. ↑ in press. provides better packing & leads to decreasing porosity with the formation of new particle contacts. The porosity ↓ as the no. of contacts & the contact area b/w particles ↑. The point contacts undergo elastic def. and at all points in the compact cycle. A reversible elastic energy is stored in the compact. ↑ press ↑ density by contact enlargement through elastic def. Thus the press causes localised def. and the contacts giving work hardening & allowing new contacts to form as a gap b/w particles collapse. The interparticle contact flattens take on a ~~vacant~~ flattened appearance with a circular profile

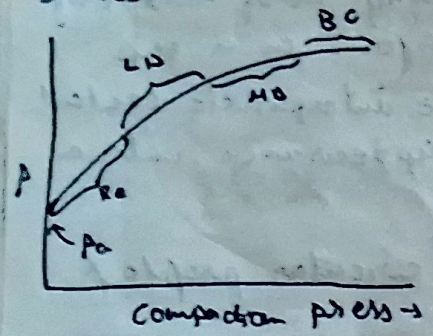
The ρg and dia of the circular profile X are related

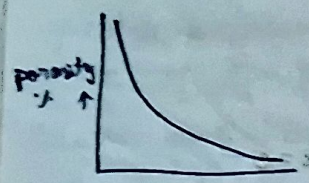
$$X = D\left[1-\left(\frac{\rho_0}{\rho}\right)^{2/3}\right]^{1/2}$$

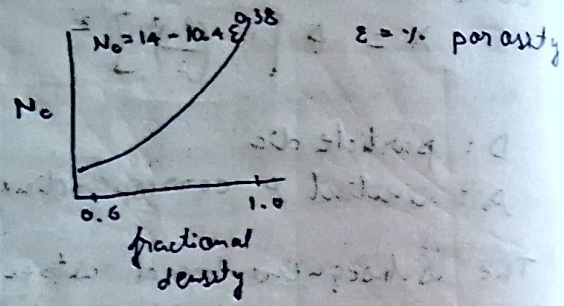
D: particle dia
ρ_0: initial ρ corresponding to x=0

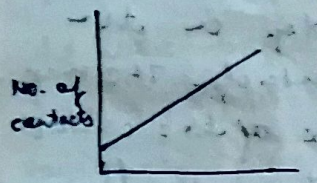
The subsequent bond strength dep on the amt of shear at the particle contacts. The max shear stress occurs at the centre of the contact and is highest when the contact is small. During def cold welding at the interparticle contacts contributes to the dev of interparticle strength. The strength after press. but before sintering is green strength.

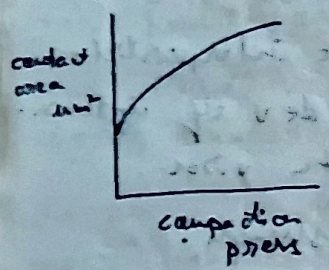
3/10/23









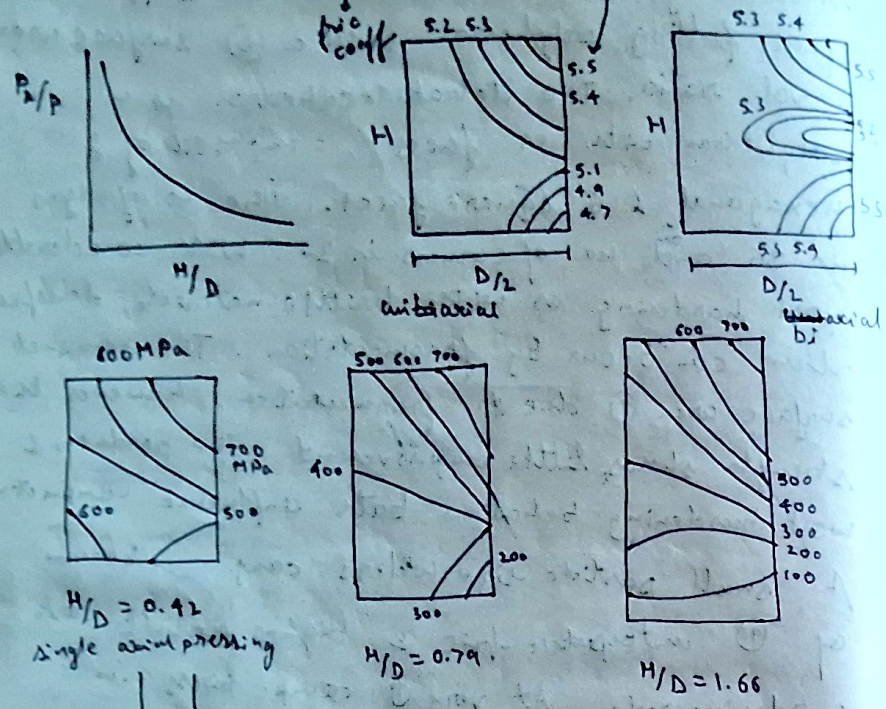


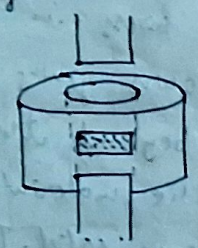
As the compaction pressure ↑ the relative vol of each particle undergoing plastic deformation ↑. At ↓ press. plastic flow is localised to particle contacts. As the press ↑ homogeneous plastic flow spreads from the contacts and the entire particle becomes work hardened. The large pores are eliminated fast & the particle coordination no. ↑ to further distribute the load. The Nc typically starts near 6 → in the loose state. During the N increases to with frac

porosity. At ↑ p the Nc approaches 14 corresponding to a tetrakidecahedron an efficient space filling polyhedron with a ↓ surface area to vol. ratio. The tetrakidecahedron is a polyhedron with 14 faces, composed of 8 hexagonal & 6 square faces. The no. of edges is 36 and no. of corners is 24. With considerable work hardening as with brittle materials, densification can occur by fragmentation. The compact surface area ↑ due to fragmentation, however the strength shows little improvement. The hardness & work hardening behaviors both influence compaction. A small particle size widens comp. because of ↑ interparticle fric & higher particle work hardening rate. At very ↑ comp. press in excess of 1 GPa massive def. occurs giving. Continuous pressurisation beyond that level is of little benefit. The material response is similar to that of dense solid. In all cases relief of the press results in reduction of the material stored elastic energy denoted by dimensional spring back. The elastic illustrator is shown by the compact failure after ejection; the level of spring back ↑ with approximately square of the compaction press.

Prove that

$$P_x = P \exp\left(-4\mu z x/D\right)$$





⇒ Large H/D pdt. cannot be produced by conventional die compaction

How to solve this problem?
Isostatic compaction

As the ratio ↑ ρ gradient in a compact ↑ and the overall compact density ↓. An ↑ in the H/D ratio results in greater ρ gradients and a lower ρ_g. In sintering the amt. of compact shrinkage varies inversely with ρ_g. A ↓ ρ_g results in a larger shrinkage. In many powder metallurgy applications, shrinkage undesirable ∵ creates prob dimensional control.

Furthermore the presence of ρ_g variation in a compact is a prob. ∵ the dimension will change non-systematically. Hence min ρ_g variation ensures uniformity in properties & dimensions after sintering. (cont)

—x—

6/10/23

For a single ended pressing, the avg compaction stress estimated as

$$\sigma = P\left[1 - 2\mu z\left(\frac{H}{D}\right)\right]$$

and double ended pressing

$$\sigma = P\left[1 - \mu z\left(\frac{H}{D}\right)\right]$$

The avg stress is dependent on both, the geometric $\left(\frac{H}{D}\right)$, the axial to radial press distribution z and the die-wall friction. High avg stress is attained in short compacts with large diameters and lubricated die walls. Die wall friction lowers the efficiency of compaction. The compact has a non homogeneous ρ, since ρ_g is highly dependent on the applied press. The specific size & shape of compact can influence the ρ distrib. The most imp para is the $\left(\frac{H}{D}\right)$ of the compact. For complex shapes or large length to dia cold isostatic pressing is a suitable technique. A flexible mould is filled with a powder & pressurised isostatically using a fluid. Compaction press upto 1400 MPa can be achieved however cold isostatic press usually performed @ press below 350 MPa. The field and shield mould is emerged into a fluid chamber which is

pressurised by an external hydraulic system. Isostatic pressures more efficiently reaching ⊕ densities at given press compared to die compaction & density grads are smaller. With isostatic the fluid provides uniform press. and densities

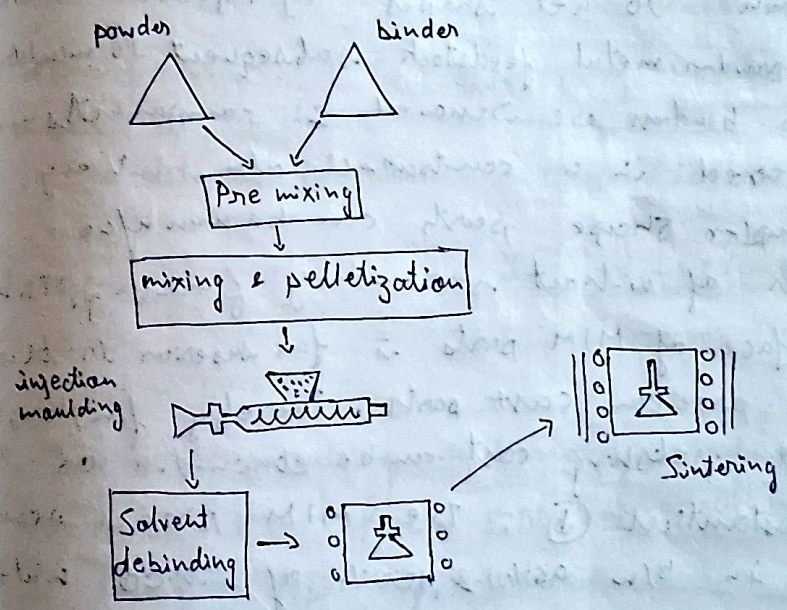
Adv of cold isostatic

1. Uniform ρ of compact bodies
2. ρg (5-15%) higher than die compaction at the same press.
3. High green strength & good handling props
4. ⊖ internal stress
5. Powder can be compacted without binder as lubricant.
6. Bodies with complex shapes or with a large length to cross sec ratios can be compacted with ⊕ uniform ρ
7. Composition can be
8. ⊖ tool cost through the use of rubber plas
9. ⊖ material and machining cost

Disadv

1. Dimensional control of green compact is less precise than
2. Surface is less
3. In general the rate of production is lower
4. The flexible moulds used have shorter life than rigid steel or carbide dies.

Powder Injection Moulding



The PIM process is said to be combination of conventional P.M. and plastic injection moulding tech because it brings together the diversity of materials of conventional P.M. & the geometric freedom of part design associated with thermoplastic injection moulding. The combination allows for complex shapes to be produced to near net fin ρ for high

performance applicat". The concept of I.M. originated from the metal die casting industry wherein the technology of injecting molten metal into close dies to obtain desired shapes evolved. Later this moulding concept was successfully exploited in industry. Powder m is an extension of . Metal injection moulding is a process by which metal are moulded to net shapes by injection moulding. A powder metal feedstock subsequent to moulding the binders are removed & components sintered in a controlled atm. Very complex shape parts can be manufac with of without secondary finishing. The surface of MIM parts is far superior to that of precision cast parts. Thereby finishing and polishing cost can be eliminated or substantial (↓). The MIM process being ∆ in b/w 95% & 100% of theo material ρ. The MIM process allows an accuracy of better than ± 0.3% of the reqd dimension. Opportunity to combine multiple components into 1 machining part & assembly cost. Ability to fill ~~thick~~ thin walls and remove unnecessary areas. The basic stages involved in forming a components by powder for the process. Mixing powder with a suitable binder for producing either a homo slurry or granular pellets of mixed feed stock. IM of feed stock to obtain a reqd shape. Removing binder from moulding parts by debinding. Densifying compact by sintering. Forced m or further densification. The capacity of injection moulding machine are specified by

1. quality of feedstock delivered to the mould.
2. Clamping force of die and clamp open
3. Injection press.
4. Speed of moulding in terms of shots of vol/area or vol/time makes space of moulding in terms of depth of moulding.
5. Mould opening size as crowel
6. Parting line vs ⊥ injection

9/10/

Sintering

- Pressureless
  - Solid (diffusion) state
    - Mixed phase
      - Composites
      - Activated
      - Homogenization
    - Single phase
  - Liquid phase
    - Transient
      - Reactive
      - Solid solution
    - Persistent
      - Mixed phase
      - Supersolidus
- Pressure assisted
  - Low stress
    - Creep
    - Viscous flow
  - High stress
    - Plastic flow

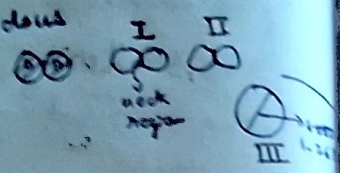
$$D_f = \frac{A_1 - A_2}{A_1 - A_3}$$

under ρ

→ inc. ρ

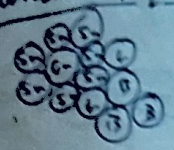
$$\frac{N}{N_0} = \exp\left(\frac{-Q}{RT}\right)$$

$$\rho_0 = \rho_f\left(1 - \frac{\Delta L}{L_0}\right)^3$$



Technique	Material	Application
Solid state		
Single phase	→ Ni	- Battery electrode - Orthopedic bracket
Mixed-homogenization	→ Fe-Cr-Ni	- Magnetic brake sensor
- activated	→ Fe-P → enhance sintering	- Wear or cutting tool
- composites	→ Al_2O_3-ZrO_2	- High temp. filter
Liquid phase		
Transient (Liq vanish at the end of sintering)		
- reactive	→ Ni_3Al-B	- High temp. filter
- solid solⁿ	→ Cu-Sn	- Self lubricating bearing
Persistent-mixed phase	→ W-Ni-Fe	- Armour piercing projectile
- Supersolidus (liq. stays upto end of sintering)	→ Tool steel	- Cutting tool
- Viscous	→ Ag-glass	- Electrical conductive lead
Pressure assisted		
Low stress - Creep	→ Si_3N_4-Al_2O_3-Y_2O_3	- Engine turbine charger
- Viscous	→ Al_2O_3-glass	- Grinding wheel
High stress - Plastic flow	→ Ti alloy	- Artificial hip replacement

Transient liq phase

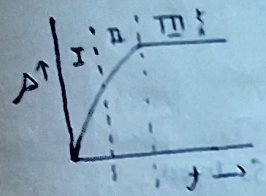


Cu react with liq Sn & form bronze.

Liq Sn vanished at the end of sintering

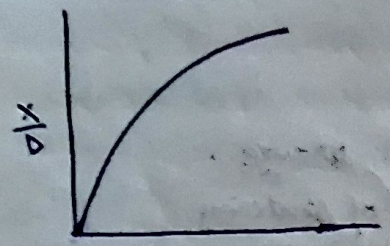
Same happens in ~~Ti~~ Ti-Al alloy.

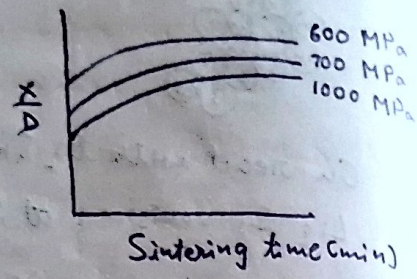
Transient reactive liq phase → reacⁿ takes place at interface

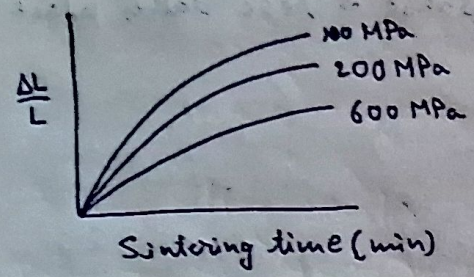


Q If two ~~particles~~ powder particles of dia D coalese with each other, their final dia is 1.26 D.

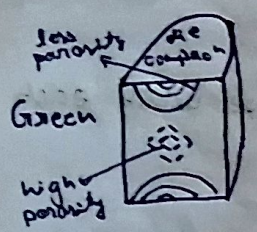
∴ Vol. const

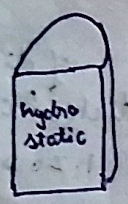


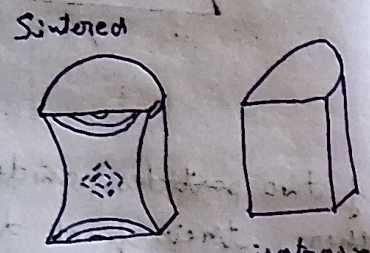




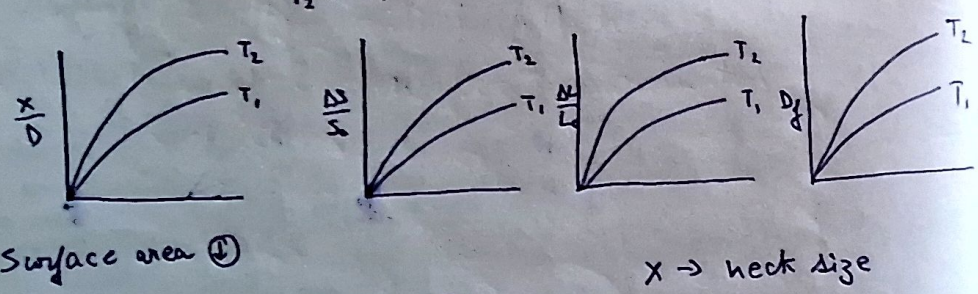
→ ↑ compac pres
↓ porosity
hence shrinkage is less





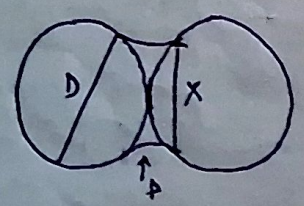


$T_2 > T_1$



Surface area ↓

x → neck size



In several instances a comp press is applied to a powder before sintering. Pressing a powder before sintering ↓ the porosity while ↑ the dislocation density in the powder. The ↑ dislocat^n contribut to the initially faster sintering rate. Compac^n therefore gives ↑ strength, density, shape and dimensional control. Higher compaction press. contribute to one ↑ in P and contact size while reducing the rate of net growth. From the fig higher comp press. leads to large net neck size, however the compac contribution dominates at the high pressure. The shrinkage ↓ as the compaction press ↑. Sintered neck size determine propes like strength and ductl thus ↑ comp press are desireable. ↑ the comp press gives better dimensional control, less sintering shrinkage and higher final properties. Dimensional control during sintering is a significant concern durin component fabrica^n of a part size and compaction die size shd be the same. At other times uniform shrinkage desired to simplify prediction of dimensional changes and final part shape. Shrinkas during sintering varies inversely with P_g. For that reason P gradients can be troublesome due to non-uniform shrinkage. The P_g grad due to die-wall friction becomes dimensional gradients after sintering.

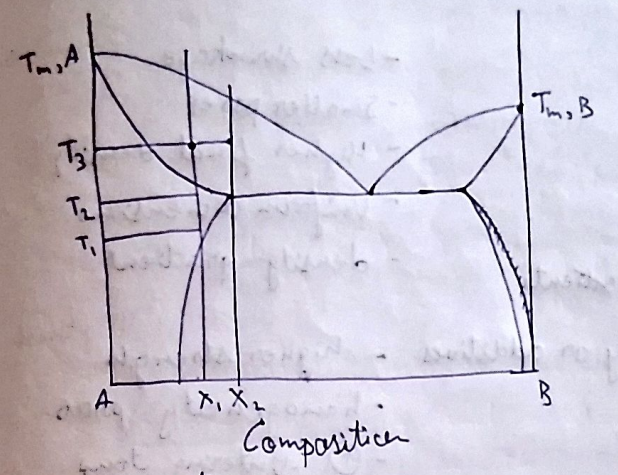
In die compaction or die compacted sts minimized dimensional change is achieved with larger powders high comp. press, low sintering temp., short sintering time, ① comp height and uniform geometry. Unfortunately such attributes are detrimental to compact strength and maybe impractical with respect to the intended application. Whereas hydrostat. exhibits uniform sintering shrinkage.

g A Cu alloy powder has an ρ_a of 3000 kg/m³ and ρ_{tap} of 4500 kg/m³. The powder is compacted in a cylindrical die at 300 MPa to a ρ_g of 6000 kg/m³. Subsequently the compact is sintered to a ρ of 7500 kg/m³. The theo ρ of the alloy is 9000 kg/m³.

i) If the powder is compacted to a 10 mm high what is the initial fill height

ii) What is the densifica^n parameter of the sintered compact.

03/10/23



X_1 composition

T_3 - Liq phase sintering

T_2 - Transient liq phase sintering

X_2 composition

T_3 - Viscous flow sintering

Sintering processing effects

Change to aid sintering	Effects
1. ⓓ in particle size	- Faster sintering - Greater expense - Higher impurity level - ⓐ hazards
2. ⓐ in time	- Greater expense - Grain growth & coarsening - ⓓ productivity
3. ⓐ in temp	- Greater shrinkage - Grain growth - Greater expense - ⓓ precision - Higher properties

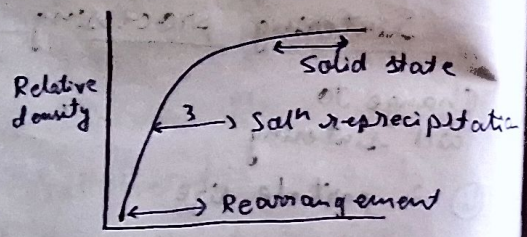
4. ↑ in ρ_g
- Less shrinkage
- Smaller pores
- Higher final density
- Uniform dimensions
- density gradient

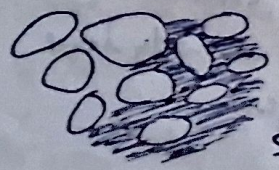
5. ~~Density gradients~~

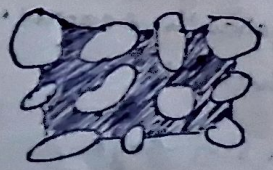
5. ↑ in alloy or additives
- higher strength
- homogeneity prob
- ↑ sintering temp

6. Use of sintering aids
- Faster sintering
- Lower sintering temp
- Embrittlement
- Distortion
- Grain growth control

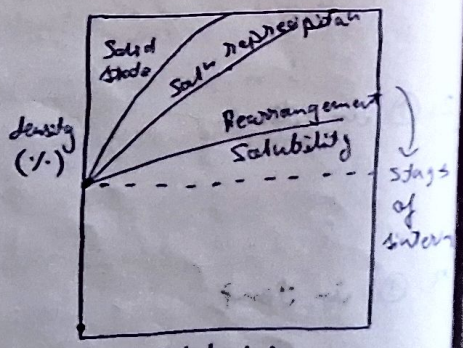
Green

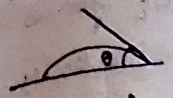



Liquid Spreading

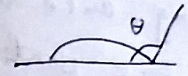

Solⁿ reprecipitation

Solid skeleton

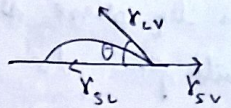




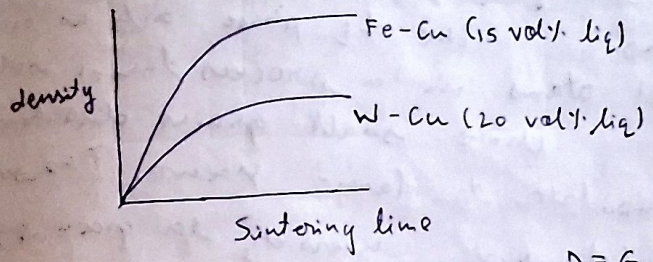
$\theta < 90°$
good

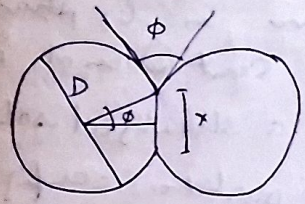


$\theta > 90°$
poor



$\gamma_{SV} = \gamma_{SL} + \gamma_{LV} \cos\theta$





D = G (same)

$x = G \sin(\phi/2)$

$\gamma_{SS} = 2\gamma_{SL} \cos(\phi/2)$

ϕ = dihedral angle

Two phases
- high melting
- low melting

Requirement
→ Production of liq phase
→ Liq shd wet the solid surface
→ Solubility
→ Vol. of liq

Stages of sintering depends upon
→ Temp, composition

⇒ If vol. of liq is high → no chance of solid state stage in sintering.

In liq phase sintering, once the liq forms it will flow to wet the particles. Initially particle rearrangement contributes to densification. The solid phase dissolves into the liq and the amt of liq grows until it is sat. with liq comp. The liq phase acts as carrier for solid atoms in a process termed soln reprecipitation where small grains dissolve & reprecipitate to large grains. The solubility of solid varies inversely to grain size hence ① grains dissolve in liq phase among time. The grain ~~size~~ count ① and size ② after exposure to liq a stabilized solid liq sys emerges by the 2 particle config. The eqm condn occurs when the solid grains form stable neck with surrounding liq. That eqm condn is unlike the one in solid state sintern where neck growth continues throughout sint. The stable neck size is given by G and ϕ $x = G\sin(\phi/2)$ and the contact angle ϕ is determined by surface energy balance $\gamma_{SS} = 2\gamma_{SL}\cos(\phi/2)$

γ_{SS} = S-S interfacial energy

γ_{SL} = Surface energy b/w solid-liq interface

31/10/23

Self ~~cooperatin~~ propagating ① temp synthesis

~~SPHT~~ SHS is a current scientific area at the intersection of inorg. chemistry, physical chem, chem. physics, combustion and explosion physics. The results of SHS have found wide application in such areas physical mat science and metallurgy. The use of SHS methods appears to be highly effective for the production of new materials. Specificall SHS has been used as the basis for developing ~~single~~ simple & efficient methods for the synthesis of metal & non metal powders used in manf of items exhibiting superior surface characteristics such as hardness, durability, resistance to wear, thermal & chemical stab. Existing method for producn of ref ceramic materials are quite costly & labour intensive. Traditional ref ceramic item manf with P.M. tech involving sintering, hot pressing of preliminary pressed blank parts which req ① energy. In contrast to trad method of PM the use of SHS the manf of desired parts possible in 1 stage. Eliminating the st

Sig adv.

1. Simplicity of equipment & min electricity consumption..

Inerty SHS is an exothermic reac^n b/w 2 or more reagent c in the mode of directional combustion in the absence of daily external heat source. Generally in order to perform in SHS it is ensured that process is at ↑ temp and ↑ rate, ensuring self propagation. The reaction is accompanied by heat release. Examine the SHS process, we can consider the syn of TiC as an example. One end of a tablet pressed from a mixture of Ti and C powders is affected by a heat impulse from a electrically heated coil. This sets off an exothermic rea^n in the heated top layer of the reagent mixture. The heat release warms the next layer of the sample, setting off chem^al rxn. A glowing wave of synthesis propagates along sample in the mix of Metal and non metal powders transfor into TiC within seconds. The temp in the SHS combustion can be as ↑ as 1500 to 4000°c. The velo of the linear wave propagation falls within the range 0.5 – 15 cm s^-1. The syn zone thickness 0.01 - 0.5 cm. The heating rate of reagent in the combustion wave is $10^3 - 10^6$ °deg/sec. The impulse energy req^n for initiation of reac^n is 42 - 420 J/cm^2 and the duration of impulse is 0.05 - 5 sec. SHS has used for syn of a no. of req materials characterised by diff chem c phase composition like carbide, borides, nitrides, silicides, hydrides etc. and reduced metals. SHS inorg materials are obtained in the form of powder, porous, cast & nano sized system pdts. SHS can be used for direct pdn of parts of desired shape size and application of coating. SHS occurs in the absence of external source of heat (SHS based production methods do not imply the use of furnace since the process occurs as a result of heat release during the chem. rxn). SHS occurs at ↑ temp in the ~~converted~~ condensed phase which set up unusual synthesis cond^n. The process can be performed however with the use of rather simple equipment. Periodic comb^n reactor of 2 types for a pilot prod^n of req. material by SHS methods. Crucibless syn. reactor for syn of carbides, borides etc. And universal combustion reactor for syn of nitride, hyd carbonitrides etc. With a vol range from 2-30 L

these cylindrical reactors have the capacity to produce very ① amt of combustion pdt per hr. SHS processes can occur under high and super high pressures. These cyl. if possible to combine combus with effects of super ② press & shock wave compaction. Thus performing isostatic processing of SHS pdts. For this type of procedure modern pipe press equipment can be employed. In stand furnace assisted process like combination of ① temp & ② press is problematic. In complex multi comp system consisting of transit metal comp & red ca Mg Al etc oxidising ag N, C, B Si & their compounds. SHS can be accompanied by a stage of reduction. The formation of ref pdt can follow red of metal from oxide. This process is quite big in ... gives rise to new prospects for SHS as a promising prod. methods. In particular it introduces the possibility to use ① expensive raw materials. This method can be used for prod^n of ca combination of ic ref material

is industry.

Chem rxn for the process

$Me_nO_m + Me' + X = Z + Me'O_m$

where

~~The chem~~

Me_n : Metals of Grp 4-6 & B

Me' : reducing metal like Ca, Mg, Al

X: N, C, B, V_2O_5 & others

Z: desired pdt.

B, BC, B_2N_3

Preliminary thermo analysis of such rxn reveals their exothermic nature and hence their possibility to occur in SHS method. Heat released during rxn not sufficient for element in comb^n mod to ensure obtained synthesis. For ~~example~~ eg rxn of

$4B + C = B_4C + 38.9\ kJ/mol$

~~Heat release~~

Comat Lc realised in SHS mode due to using thermal effect. However B_4C can be successfully obtained as a result of a process

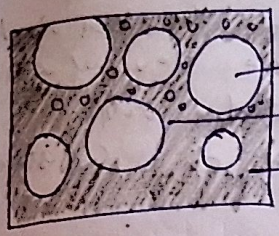
$2B_2O_3 + 6Mg + C = B_4C + 6MgO + 1125.5\ kJ/mol$

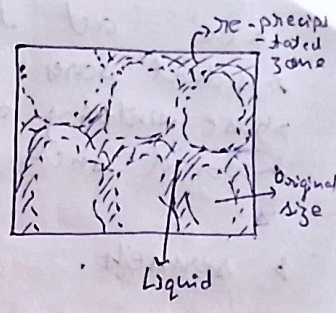
$2B_2O_3 + Mg = 4B + 6MgO + 1087.8\ kJ/mol$

$4B + C = B_4C + 38.9\ kJ/mol$

which includes a stage of red^n.

10/11/23





Solution re-precipitation

One stage

If there is insufficient liq. to fill all pores then continued densification relies on solid diffusion through the liq. If the solid is not soluble in liq then densif. occurs by relat solu solid state chemical densific. However solution re precip dominates when the solid is soluble in liq

3 stages

i) Solid soln into the liq preferentially from ① energy region including point in the microstr area. Areas under compression & small grain

ii) Diff of diss solid into liq

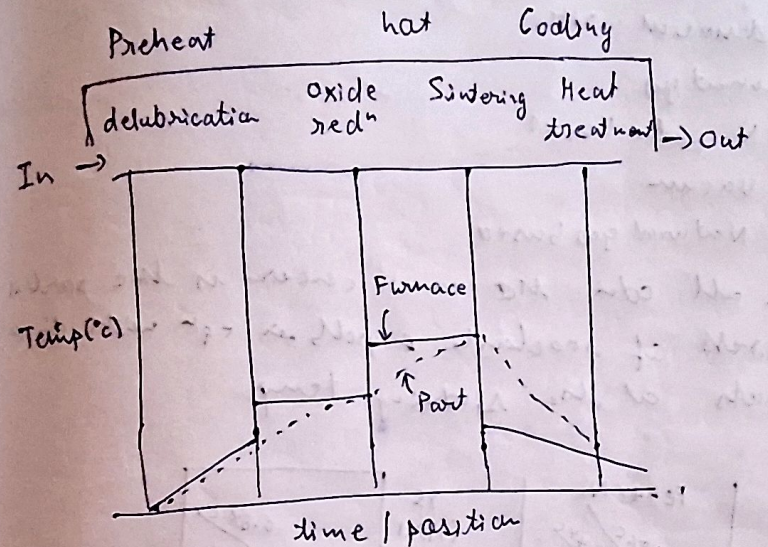
iii) Pptn of diss solid onto concave region on larger grain in areas not under compress. via soln reprecip improves grain packing, releasing liq to fill pores, grain shape. Accomodation is favourable ∵ the overall interfacial energy is ①. A conceptual outline of changes occurring with soln reprecip densification where both g.g. & grain shape

ace at a reduce [illegible]
residual pores: Imp technical advances in liq phase sintering came with the dev of several material such as cemented carbide tungsten Ni Cu, Cu steels & cermets.

Microwave sintering

Modern studies confirm that many ceramic & even metallic systems can be sintered by microwave energy. uwave are electromag energy with a wavelength range of 10-1 mm freq of 300 GHz. Ceramics with a moderate to (N) dielectric loss camp with the uwave and convert the uwave energy to heat energ. Since the uwave penetrate deeply into ceramic thick sections can be heated uniformly & rapidly to the sintering temp. Incase the parts are (N) in size temp uniform is few min provided the part is insulated at surface to avoid heat loss from surface by IR radiation

Heating element	Max. temp. (°c)	Atmosphere
Ni-Cr	1150°c	1,2,3,4,5,6
Kanthal	1300	1,3,4,6
Super "	1600	1,3
SiC	1250/1600	1,2,3,5,6/1,3
Pt	1500	1,3,4
Mo	1800/2200	2,3,4,5,6/4
Ta	1900/2400	3,4/4
W	1900/2800	2,3,4/4
C	2200/3000	3,4,5/3,4
ZrO_2	2200	1,3,4
$MoSi_2$	1700	1,3.



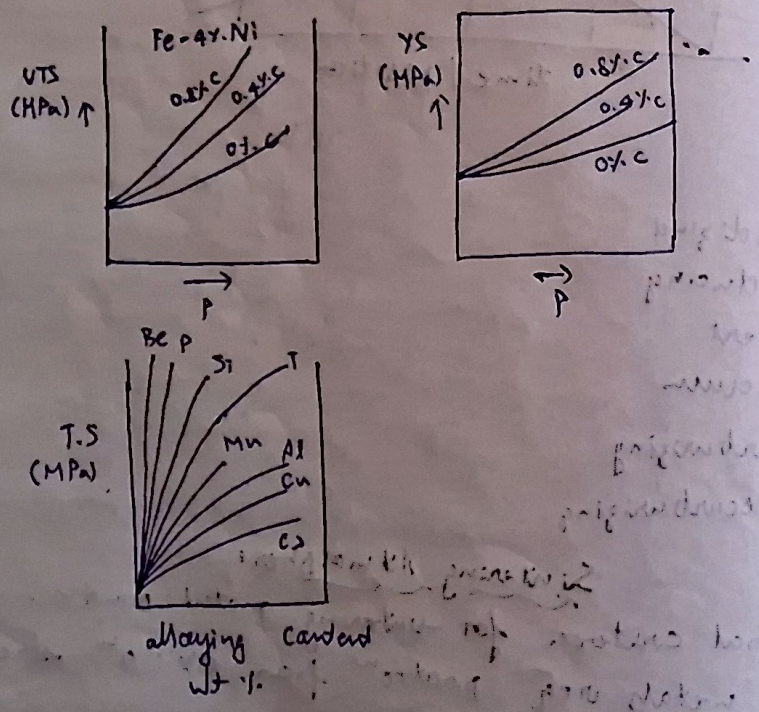
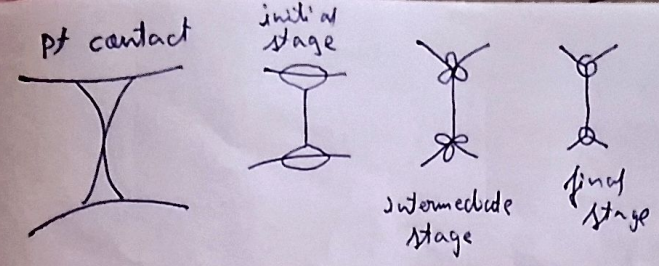
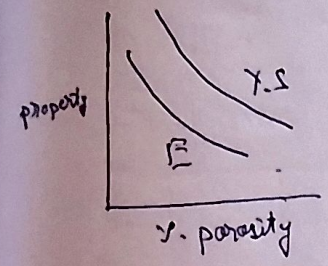
1 - oxidizing
2 reducing
3 inert
4 vacuum
5 carburizing
6 decarburizing

Sintering Atmosphere

Several criteria for sintering metal powder. Many metals req protecn from oxidan since oxide & other contaminates dev of properties. The atm serve lubricants & binders used. Since oxides are present in reducn atm not only provide

protection from oxid but can reduce any existing oxides. The atm can also control the interstitial content of sintered nature.

Types of atmosphere..

1. Hydrogen
2. dissociated NH3
3. inert gas
4. N based
5. Vacuum
6. Natural gas based

In all atm the main concern is the partial pressures of reactants & products in eqm with the oxides at the sintering temp.