NOTES

DATE 25 09 2023

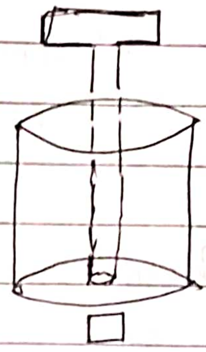
Flow rate:-

Flow rate is the measured amount of powder made to pass through a certain orifice and collected in a die/ container of certain diameter ~~for this~~ and the amount of time required for this process is k/as flow rate.

Significance:-

→ Productivity is affected by flow rate.

→



→ Bulk material

→ Bulk Consolidated material.

→

★ **Apparent density:-** Ratio of mass and volume of free flowing powder without any external force / pressure to densify the powder in As recieved condition.

Significance:- Die fill up or producing a compact.

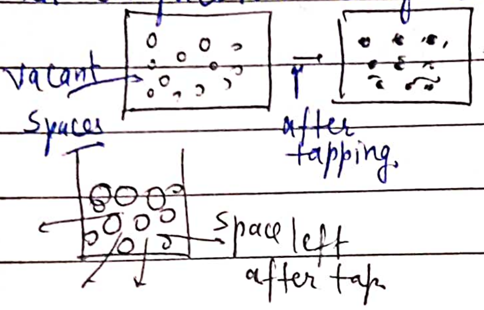
→ Reflects the approx. amount of powder to be used to make a compact of certain height with fixed diameter.

★ **Tap density:-** The density of the powder, after it has been mechanically shaken down or tapped until the level of the powder no longer falls. Tap density is always higher than the free flow apparent density.

We cannot directly compress the powder after fill up because powder has no adjustment. so a lot of space is there in b/w powder particles which should be accomodated w.r.t size, shape and surface morphology of powder particles so we need to give some external f tap to accomodate the space.

Significance:- Powder compaction can be utilized significantly by applying a specified number of taps to accomodate space.

Certain spaces can't be accomodated by taps. Tap density will help in ~~main~~ systematic accomodation of vacant space so that it help in compaction by ~~comp~~ external compression.



→ Uniform distribution of pressure

→ Helps to avoid gas entrapment.

Fill up die under free fall. → Tapping (Tap density) → Compression.

★ Green density :— When there is no interparticle bonding then it is k/as Green condition.

Density of powder before sintering or without interparticle bonding is k/as Green density.

Density of the compact after the application of external pressure at room temperature is called Green compact.

Ex. Fe – 0.2%C , Ti – 6Al – 4V , Al – 4%Cu.

~~→ Cold pressed~~

→ (i) As mixed (Homogenized composition).] Green density will be higher

→ (ii) Mechanical alloyed. → highest hardness | Green density

→ (iii). Prealloyed powders. ↓ decreases.

→ Hardness of powder particles should be low.

★→ The better the green density, better is the sinterability provided composition remains same.

Ex. 75% G.D. & 85% G.D. of same sample → Sinterability. 85% G.D. powder > 75% G.D.

NOTES

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Green Compact :-

No-interparticle bonding.

* relative density is the actual density divided by the theoretical density.

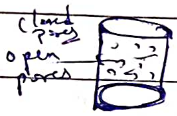
2.69 g/cc → Theoretical density.

2g/cc → after compaction

$$\text{relative density} = \frac{2}{2.69} \times 100 = 74.34\%$$

① Dimensional method :-

Whenever a compact is in green condition, the density of compact is calculated using dimensional method



$\dfrac{\text{mass}}{\text{volume}}$

Limitation :- Porosity may be filled with gases so mass will inc and ~~dens~~ volume may ~~dec~~ inc.

② Archemedes Principle :-

We can't use archemedes principle in green condition because water can ~~pass the flush~~ wash out the compact as there is no interparticle bonding in green condition.

★ Significance of Green density :-

(1) High Sinterability.

(2) Better surface area of contact i.e. Particle contact area.

Better the diffusion of bonds.

(3) Better the Green density better the green strength.

★ Factors affecting the Green strength and Green density :-

(1) Type of powders production technique.

for Same size & shape →

(2) Surface morphology. (Roughness)

PAGE

 morphology - size, shape, ~~regularity~~ roughness, ...

DATE

The higher the roughness, higher the surface area, better the ~~interparticle~~ ~~bonding~~ green density.

(3) Powder particle shape.

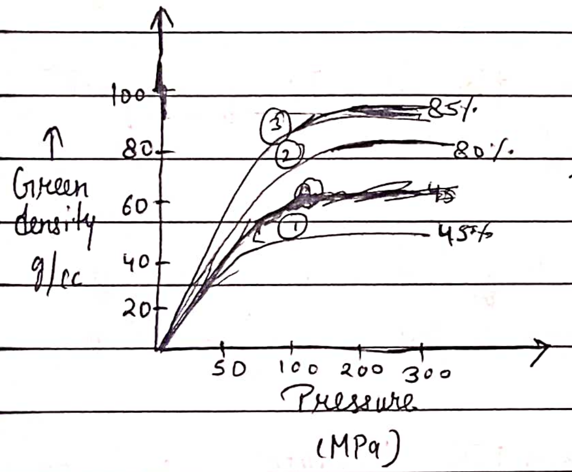
~~↑es Irregular shape~~

↑es particle shape irregularity better the G.D.

(4) Particle size.

↑ fineness better the G.D.

(5) Presence of Oxides, Nitrides, Carbides — brittle — decreases G.D. because pressure required to compact is high due to brittle nature of oxides, nitrides and carbides.



Type of materials are different for ①, ③ & ③

G.D. of ③ inc. with inc. Pressure to larger extent than ① & ②.

Ti - HCP — G.D. is high.

Harder powders have lower G.D.

High pressure may damage die at very high external pressure but it may increase some amt. of ~~gr.~~ G.D.

★ Cold Compaction — at room temperature.

Green ~~density~~ compact is made by cold compaction.

Parameters:—

→(1) Pressure.

(2) Time.

(3) Strain rate.

★ Die compaction   Die parts

(1) Punches — Top, bottom

(2) Die

Mostly cylinderical dies are used to avoid die wear and tear

→ Powder particles without ...

NOTES morphology - size, shape, ~~regularity~~ roughness, ...

DATE

The higher the roughness, higher the surface area, better the ~~interparticle bonding~~ green density.

(3) Powder particle shape.

~~Ies Irregular shape~~

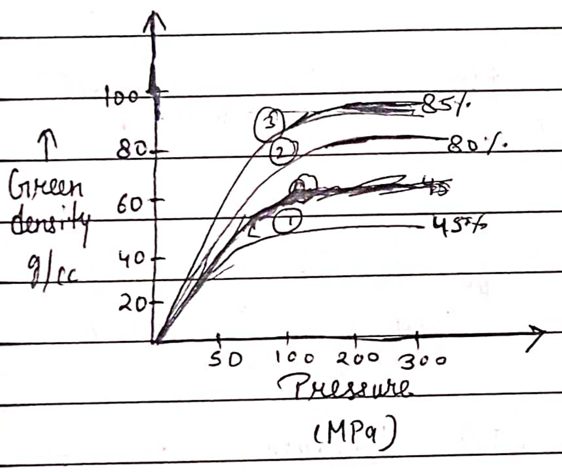
Ies particle shape irregularity better the G.D.

(4) Particle size.

↑ fineness better the G.D.

(5) Presence of Oxides, Nitrides, Carbides – decreases G.D. because pressure required to compact is high due to brittle nature of oxides, nitrides and carbides.

brittle



→ Type of materials are different for 1, 2 & 3

G.D. of 3 inc. with inc. Pressure to larger extent than 1 & 2.

Ti - HCP — G.D. is high.

Harder powders have lower G.D.

High pressure may damage die at very high external pressure but it may increase some amt. of ~~gr~~ G.D.

★ Cold Compaction :- at room temperature.

Green ~~density~~ compact is made by cold compaction.

Parameters :-

→ (1) Pressure.

(2) Time.

(3) Strain rate.

★ Die Compaction — Die parts

(1) Punches — Top, bottom

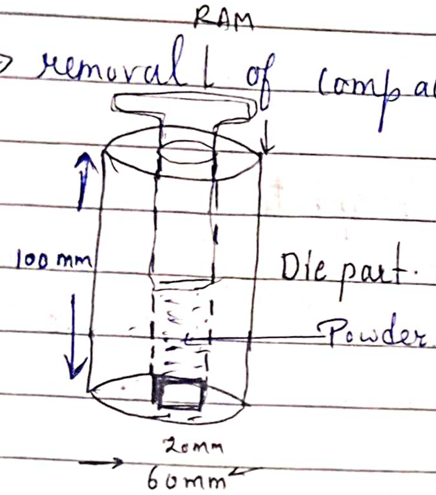
(2) Die

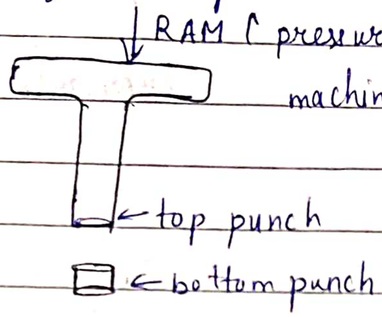
Mostly cylinderical dies are used → to avoid die wear and tear

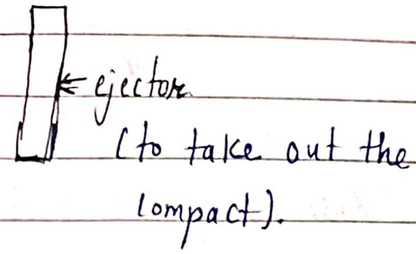
→ Powder particles with...

PAGE

→ removal of compact after compaction will be easier.





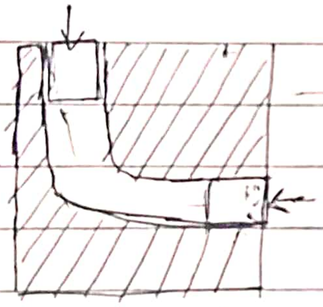


→ uniaxial ~~compound~~ compaction → Pressure is applied only on top side.
~~Biaxial~~ ~~Pro~~ → One directional

~~Split die~~ Uniaxial → One directional / two directional } multiple directions.

Date 06/10/23

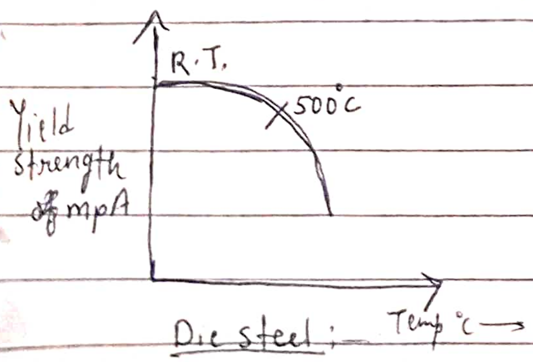
Biaxial compaction:— Equal channel angular pressing (ECAP)



⇒ This can be used for cold compaction, warm compaction (200-300°c) or hot compaction.
→ Can be used to ~~pro~~ make green compact.

L-shaped die (ECAP)

Warm compaction is used for:— → Better density.
→ Some of the powders are difficult to compress because of their ~~As received powders~~ shape, size, surface oxides and roughness.
→ Die material changes in comparison to cold compaction.
→ same design of die can be used for cold, warm and hot compaction.

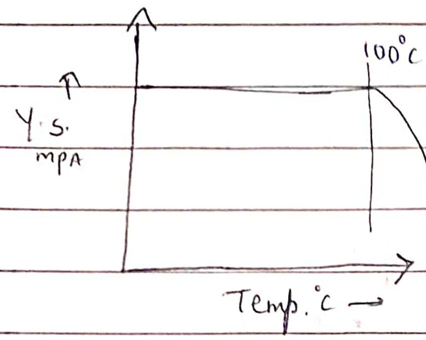


Die material.
→ H13 Diesteel — min
→ H21 Diesteel

Die steel:—

Cu – Tendency to stick
Powder

NOTES DATE



High speed steel – 650-700°C

Lubricants → (1) Graphite — Powder / Colloidal form
(slurry of graphite is made in warm water and then applied.)

Graphite can be used till 1200°C.

~~Disad~~ of Graphite :–
There may be contamination due to graphite in powder.

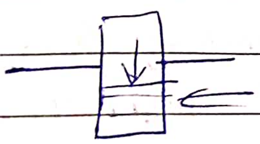
→ (2) Zinc stearate – soft white coloured
→ used at room temp. below 700-800°C

(3) Glass Lubricants :– Type of glass.
→ Heat glass to a high temp. such that it becomes liquid glass (sticky & viscous liquid).
~~Glass~~ → This is only used at elevated temperature.

(4) MoS_2 :– Colloidal powder.
(Molybdenum disulphide).

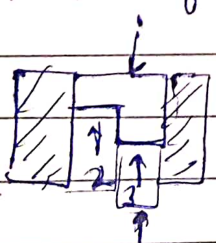
Powders with surface oxides are harder.

Uniform compact density is preferred (i.e. uniform pores distribution).

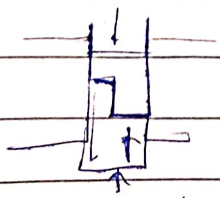


← Top part have better density than bottom part.

In case of Biaxial – top & bottom parts have better density than middle part.



Double lower Punch



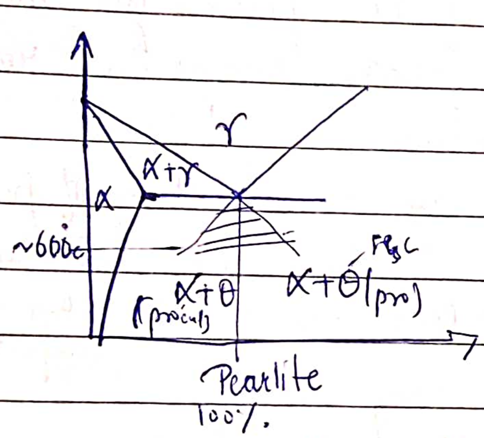
Single bottom punch.

Isostatic Compaction: → Uniform density.
Because of uniform pressure there are lower chances of leakage of powder.

→ Powder protection is required in hot compressing
→ Chances of wear and tear.

Date 12/10/23

Assignment → Apparent density, Tap density, Green density, Lubricants ~~their~~ this is used doing compaction.



γ → α+θ

We may have 100% pearlite beyond eutectoid temperature also but undercooling is required for that.
But it can be found only upto ~600°c. because diffusion of large size Fe atoms will become difficult beyond this temp.

Purpose of Cold Compaction - To get pre-final or final shape.
- It is difficult to handle powder

Die Compaction. - produce green compact.

Cold Compaction — R.T.

Warm Compaction. (~100-200°C) much below the recrystallization temperature.

for steel ~ 300°C
└ mpt > 1500°C

Die material types
- H-13
- H-21
- HSS / Semi HSS
→ INCONEL-718

↓ Temp. inc.

fcc
bcc
hcp

Q.1. Why we go for warm compaction?
ans = → Some of the powders are not able to produce compact at room temperature.
→ Basic crystal structure of powder
→ Powder, shape, size, contamination, oxygen content at high temp. affects. Compaction.

Disad.:-
→ Because of wear and tear of die ~~at~~ in warm compaction.
→ Depending on the size of powder particles they will absorb oxygen content in warm compaction.

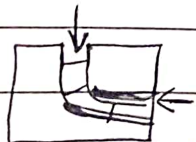
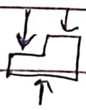
Green compaction max - 85% density

★ Cold-Isostatic Pressing :-
↳ Pressure from all the sides.

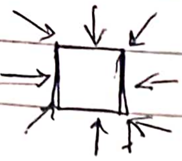
with respect to die compaction:-

(i) Uniaxial single dirⁿ 

(ii) Uniaxial bidirectional 

(iii) ECAP :-   (iv) Multiple punches

→ W.r.t. powder particles
→ To improve the microstructures. (refine)



Uniform pressure is applied along all the directions

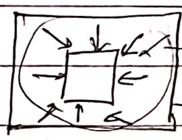
Triaxial state of stress in compression → closes the crack
" " " " in tension → formation/ Propagation of crack.

→ ~~So~~, Compression state — so, cold isostatic pressing closes the pores.

→ In this Cold Pressing is done prior to CIP to
↓
Cold Isostatic Pressing → to ~~increase~~ improve the density & to shape.

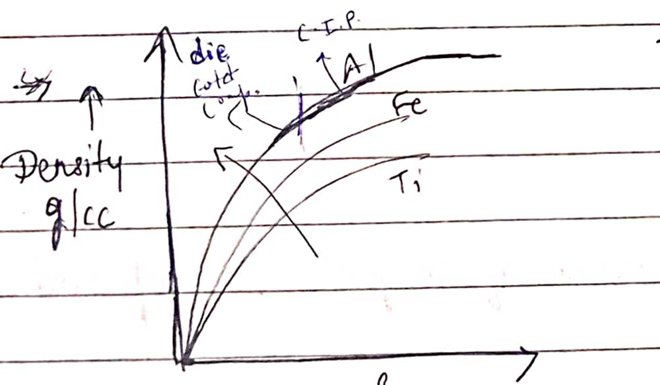
→ This technique (CIP) uses a flexible mold, because pressurising medium is used as fluid → Oil,
→ water

~~We can't directly~~

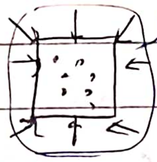


Cold Isostatic pressing needs → flexible mold → to impart powder pressure
→ Rubber mold
→ fluidizing medium.

Purpose of flexible mold :—
→ (i) Impart pressure of fluidizing medium on compact.
(ii) To avoid the contamination of ~~compact~~ fluid to the sample.



★ disadv :-
In cold isostatic pressing gas pores may



not allow the compaction and lead to breakage of mold.

→ 0-200 megapascals (MPa) ← Max Pressure used in Cold Isostatic Pressing

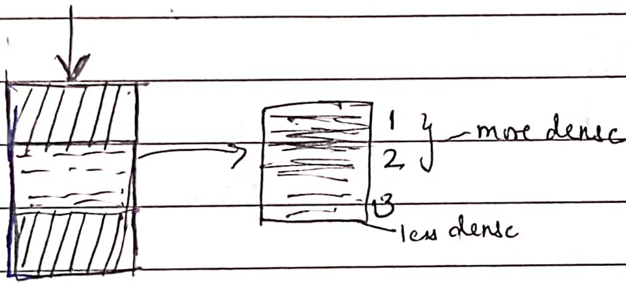
→ In die compaction pressure - 0-1000 MPa

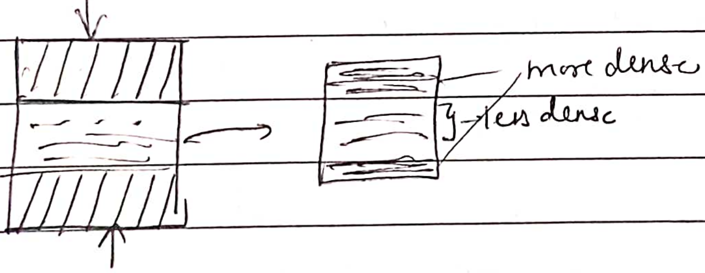
Die	Cold Isostatic
(1) 0 – 1000 MPa	(1) 0 – 200 MPa
(2) Compaction time. 15 minutes – max, Avg – 5-10 minutes.	(2) Compaction time. 5 minutes – max Avg → 1-2 minutes
(3) Die concern.	(3) Tidious w.r.t. encapsulation.
(4) Economical.	(4) Cost high.

Densification process

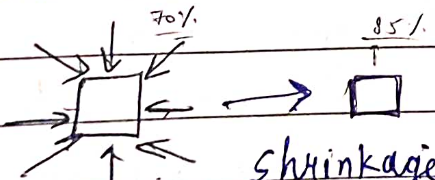
Both densification and compaction ~~occ~~ occurs in hot compaction.

Date 16/10/2023





ECAP ~~has~~ gives more ~~more~~ dense compact



Shrinkage in volume.

If encapsulation is ~~tore~~ perfect then density of compact is improved significantly.

Spherical particles have point contacts so sintering is difficult in case of spherical particles. → Pressurised sintering → inc. contact area.

☆ Sintering :-

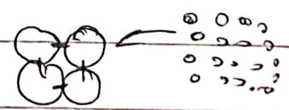
Purpose :- Imparts:
→ Interparticle bonding.
→ Densification.
→ Converting to Bulk-consolidated material.

Types of Sintering :-
(i) Liquid-phase sintering.
(ii) Solid-phase sintering.

(i) Liquid-phase Sintering :-
Three main steps in Liquid phase sintering as follows:-
(1) Liquid phase present. Movement of liquid / dispersion of liquid throughout. ~30-35% volume fraction of liquid in bulk material.
(2) Dissolution & reprecipitation.
Small particles dissolve in liquid and at the same time particles precipitate.
(3) Solid-state sintering.

(i) Liquid phase sintering will happen in binary and ternary alloy composition at sintering temperature.
(ii) Activated → external liquid phase ex Pd, Ni etc. (activator) are added which have low m.pt into an alloy to improve sintering.

(ii) Solid-phase sintering :-



(1) Developing (Proper) particle contacts.

(2) Formation of necks.
(3) Pore channel formation.
(4) Pore channel closure.

Pores will become rounded & spherical

Mechansim of mass transport :— for liq. as well as solid
(2) Evaporation and condensation.  mass transport from concave to convex.
(3) Plastic flow. w.r.t (i) Lattice diffusion (ii) Grain boundaries diffusion.
(4) Viscous flow. — depends on the slf tension of liquid
↳ depends on the viscosity of the liquid.

~~General~~

* Process parameters for sintering:—
(1) Temperature.
(2) Time.
(3) Sintering atmosphere.
~~(4)~~

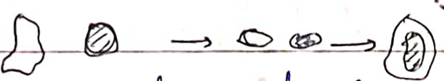
* Metallurgical Process parameters:—
(1) Particle size → finer
(2) Particle shape ← irregular or mix of Particles
(3) Surface roughness. → high
(4) Oxide, nitride, Carbide → adversely affect → less
(5) Entrapped gases → less
(6) Impurity.
(7) Alloying additions → depending on chemical affinity it may or may not affect Sintering positively or negatively.
↳ depend on type of stoichiometric sample. will increase in volume at high temp. due to internal pressure of gases.

→ Formation of intermetallic compounds makes brittle material.

Syllabus Remaining! ~~Forms~~
Reactive Sintering.
Sintering atmosphere
Hot Pressing.
Hot Isostatic Pressing
Spark Plasma Sintering (SPS)
Powder Injection Moulding.
Laser Technology or Inkjet Technology.
Porous material.
Applications.

Reactive Sintering:-

→ Only happens in certain systems.
→ "Special case".
It only happens in a binary or ternary components systems.
Al-Zn-Mg.
→ Needs binary.
→ Two elements will have a affinity towards each other beyond ~~sintering~~ Some
temperature.
Ex Ti-Al system.
Ti-45-48 Al (at %)
→ The temperature acts as activation as the system requires an energy.
→ Affinity towards each other beyond certain temperature (activation energy)
→ Stiochiometric → Composites powders should be there in alloy composition. ~~As re~~ As mixed.



This case can be used for reactive sintering. to make alloy.
If it is already alloyed then there is no requirement for reactive sintering:
Alloyed element → densification
Single element → Phase change may occur at high temp.

→ >750°C → Prealloyed – solid state sintering
→ As mixed or composite powders – Liquid state sintering
→ autogeneous sintering takes place.
Reaction → self propagating
In Ti-Al ~~if~~ we go for alloying so that no liquid state remains
so, sintering will be done in solid state.

→ Self propagation, exothermic reaction takes place beyond some temperature in reactive sintering.

550°C – Al sintering.

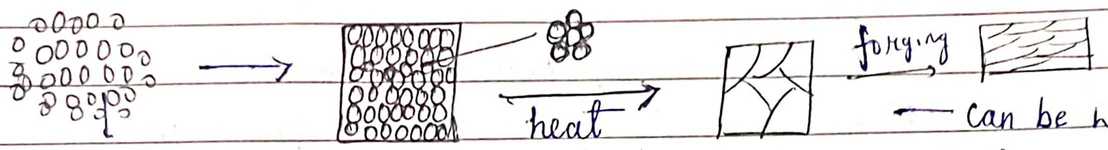
→ Particle size → initiation temp. 725–750°C 5 μm   650°C → nanocrystalline.
700°C — submicrocrystalline.   600°C

→ Type of powder → Composite powder ← Used mainly.
Prealloyed
As received → Thermo-mechanical ~~sinter~~ consolidation.

→ Pressure assisted → Hot pressing.
→ Pressureless → Thermal

Date
20/10/2023

Consolidation :— Thermomechanical



← can be worked on

000
000

Compaction at room temperature.   (hot ~~compact~~ consolidation)   Bulk polycrystalline material (bulk consolidated material) or actual material.

→ ~~For~~ After consolidation we can do rolling, forging, shaping on the bulk consolidated material.
For shaping we need to convert powder into bulk consolidated material by sintering, hot consolidation ~~or an~~, hot pressing, etc.

→ Thermal Consolidation. (T.C)
→ Thermomechanical Consolidation. (TMC)
→ ~~Therop~~ Thermoprocessing Consolidation. ~~(MPC)~~ (TPC)

★ Hot Pressing :—
→ Die compaction

~~H13 H21~~

low temp. { H13, H21 } 400–450°C   HSS, Semi HSS] upto 650°C   INCONEL-718, Superalloy] upto 1100°C

PAGE

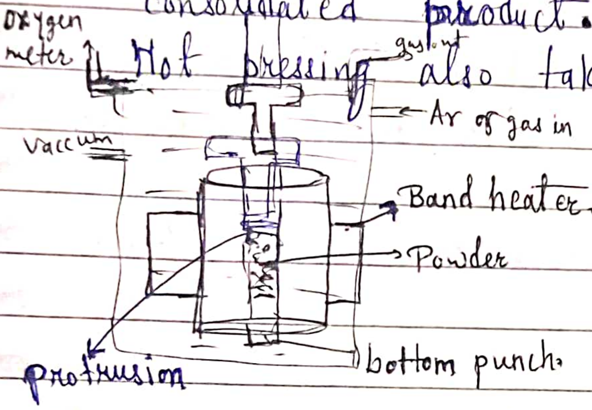
Sintering temp. → 0.8 to 0.9 Tm.

Sintering is a diffusion process which is more efficient at high temperature.

At ~~850~~ 0.85 Tm → diffusion is higher than at 0.8 Tm. But it should be below melting pointing otherwise ~~m~~ it will melt.

→ Hot pressing is a phenomenon ~~to con~~ directly convert powder to hot consolidated product.

Hot pressing also takes place at sintering temperature.



→ All setup is kept in air chamber

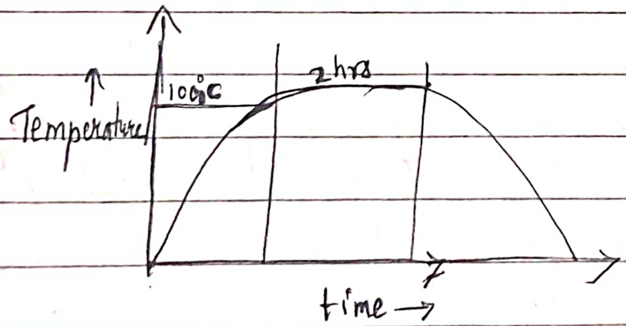
→ Uniaxial single direction ← usually

thermocouples are inserted inside the powder

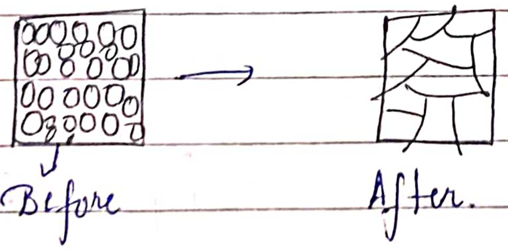
If oxygen content is much less then we go do heating and pressurizing.

Hydraulic or mechanical press.

→ Heating and application of pressure at the same time in hot pressing.



Parameters
→ Oxygen content
→ 1000°C 2hrs holding time.
→ Pressure - 100 MPa.



Hot Pressing {
Alloying
Sintering
Compaction.

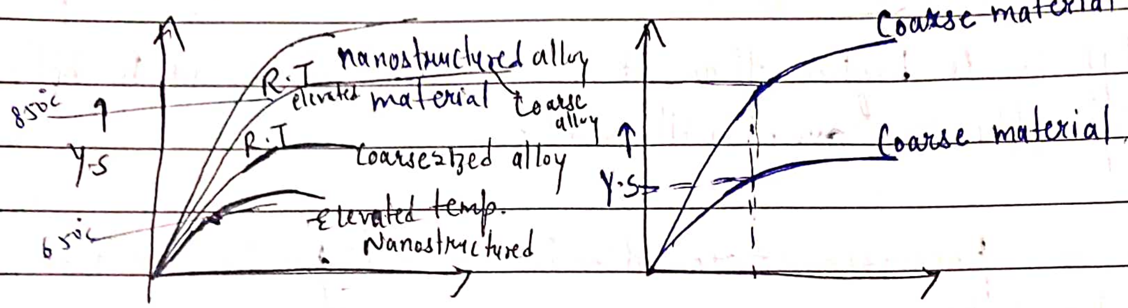
Hot pressing can be done on. → Controlled atmosphere.
→ Vaccum ~~tool~~ hot pressing (costly).

NOTES DATE

→ Nanostructured materials can be made in a easier manner than other processing techniques in Powder metallurgy.

grains are nanosize.

at lower temp.



→ Brittle to ductile transition reduces at lower temp. due to ~~equi~~ ECT.

→ Creep → coarse grained → better because it have high Y.S. at elevated temp.

→ Holding time (2 hrs) and pressure should be achieved at the same time. (3 hrs)

Date 30/10/23

★ Advantages of Hot Pressing :—

1. → No room temp dry compaction or warm compaction are cold isostatic compaction are not needed.
2. Direct ~~p~~ conversion from powder to bulk
3. High quality compact can be produced than sintering provided the same powder ~~shou~~ is taken.
4. Bulk material produced can be used for secondary processing e.g. forging, rolling, ECAE

Disadvantages:—

1. We need a controlled chamber/atmosphere because powders are reactive at high temp
   ~~...~~ Costly process.
2. Density variation will be there. — more dense / less dense
   → invert the sample and do hot pressing to improve density variation
   or
   → improve by forging (secondary process)

ECAE — Equichannel Angular extrusion.

→ Rolling
→ Forging

5. As hot pressed compact or samples can be used as a suitable precursor (starting material for forging).

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★ Hot Isostatic Pressing :—

HIP is not only for P/M products but it is universally used for any of the processed material.

→ Fluid used – Inert gas (Ar). is used for HIP.

Procedure :—

→ Evacuate sample in ~~flexible mold~~ metallic encapsulation. to evacuate all the gas entrapped inside the powder sample.

Metallic encapsulation.
- → Uniform pressure application and it should not react at high temp.
- → To avoid contamination

★(i) Encapsulation is used (needed) : when we use powder, Green compact — HIP.

Types of encapsulation material :—

★ Properties needed for encapsulation products :—

1. should not react with particular product.
2. Material should be ductile at HIP temperature.
   ~~2.~~ It should not go beyond its fracture limit.
   ~~3.~~ Material used ~~—~~ Titanium (Ti)
   Commercially pure Titanium (Ti) → 1670°C m.pt
   Certain type of stainless steel – 304, 316

No need for encapsulation
(ii) Bulk Consolidated material can be processed – HIP
(iii) Ingot metallurgy products (I/M) – HIP
] Provided No entrapped gases

★ HIP used to improve the quality by improving the density of products irrespective of any methods of production. e.g. I/M, P/M, etc.

* Forged (improve density) sample can be densified by HIP.

→ Near-dense products can be obtained by HIP. (99.99%)

NOTES Tensile → triaxial → breakage of sample.
compa stress

Degassing [evacuation → creating vaccum
heating → to remove volatile matter

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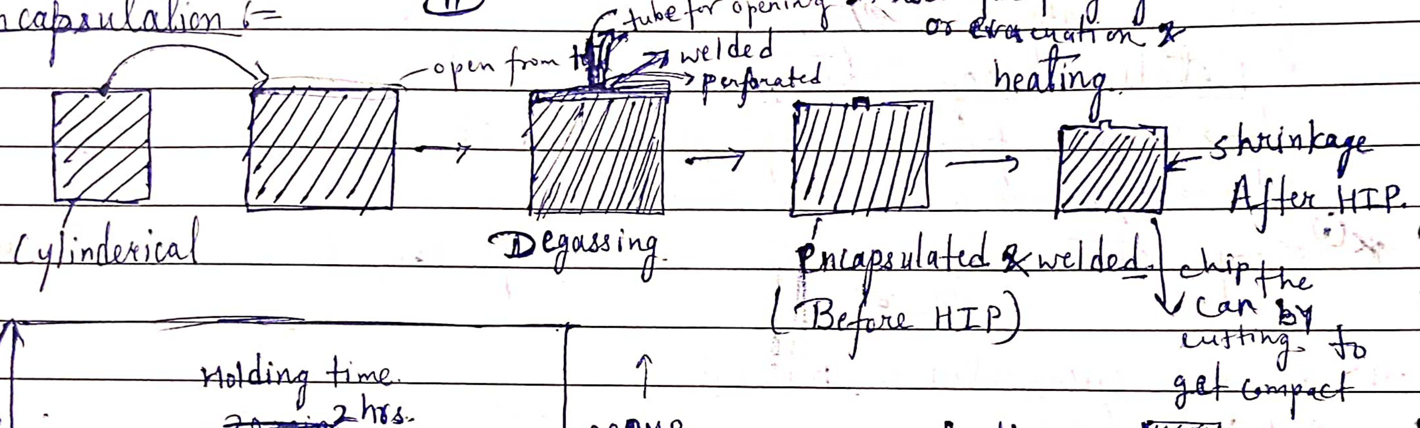
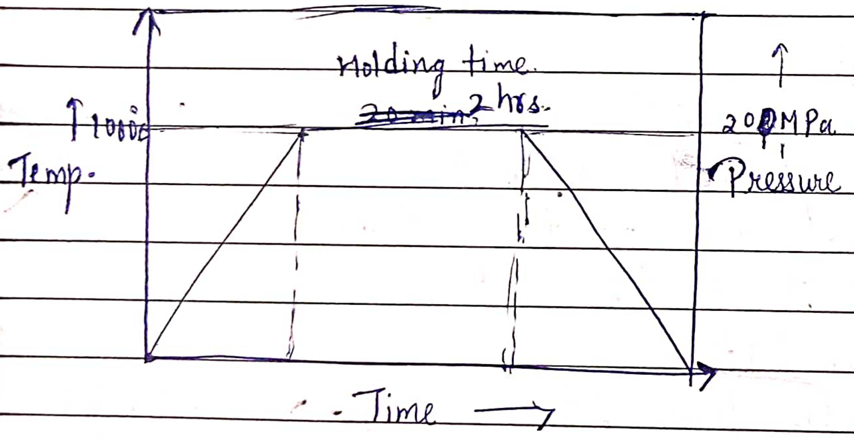
Precursor
→ Starting material → Powders, Green Compact.
→ Encapsulation.

→ Isostatic pressing → there is no ~~e~~ much effect of triaxial state of stress
→ Triaxial state of stress in compression does not have any problem of breakage
Triaxial state of stress in tensile state may break the sample.

Encapsulation :-





(fluid press. is used) mech. pressure is used (mechanical process).
→ Shape does not change in HIP while in forging shape of product changes.

★ Can material → flowable
→ ductile
→ should not react with powder material at high Temp. (i.e. HIP temp.).

★ Powder forging :-
→ Loose powders. → Open die forging
→ Green compact. → No need for Porous membrane.
} densification & interparticle bonding.
Starting material.
Thermo-mechanical consolidation
→ Bulk Consolidated via P/M route. → Only densification.
Thermo-mechanical Processing (TMP), → density will be higher

Hot pressing - die compaction at high temp.
Hot isostatic pressing -

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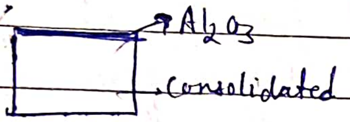
☆ Powder Compact Forging :-

→

Medium to high strain rate forging is done

→ Process is economical provided
→ ~~Po~~ In Bulk consolidated material there is no need for degassing



→ Controlled chamber forging.
→ It is very difficult to maintain controlled chamber.
→ Costly.

Advantages :-

→

Hot pressing - Pressure assisted sintering (TMC)
Powder extrusion - if Powder or G.C. is used → can be called as

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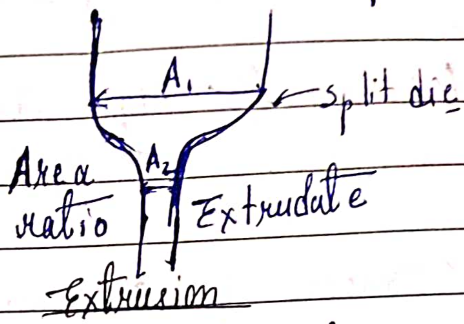
★ Powder Extrusion:- Loose powder or Green compact extrusion

★ Powder compact Extrusion:- P/M product extrusion (Bulk compacted product).



→ Direct extrusion - mostly
→ Indirect extrusion.

→ Powder / Green compact (High temp.) is used then very high strain rates cannot be used because of low fracture toughness.

Forces:- → Dominated by Compression force.
→ Tensile force.
→ Mechanical forces
→ Hydrostatic Pressure
→ Triaxial compression.

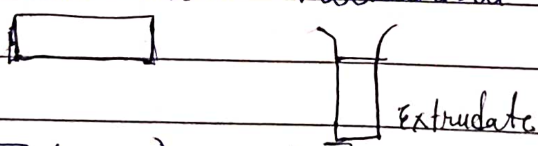
→ Since it is elevated temperature extrusion so, high strain rates are difficult when Powder or, Green compact is used.

→ Pores closure, sintering and formation of product of different size and shape.

→ Medium strain rates are used.

→ Thermo mechanical processing → provided the starting materials are powder or Green compact it can be called as pressure assisted sintering.

→ Refinement of microstructure in extrudate is much better than other processed counter Part (i.e. forging or sintered). So, they have better mechanical properties.



(60% strain)
→ Severe plastic deformation (SPD) is done ~~to refine it~~ for the refinement of microstructure.

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★ Procedure :— (Below 450°c)

→ Hydrostatic (HE) extrusion cannot be done when powder and green compact are used as starting materials.

→ HE can be done below 450°c only when powder is encapsulated.

→ Depends on :—

→ Type of die material used. →

→ ~~Lu~~ Lubrication used w.r.t temp.

→ Type of material.

(flow) movement of material varies with temp.

↳ Temperature differente should not be there for better extrudate.

★ Advantages :—

→ Better mechanical properties are obtained.

→ Wrought shape can be ~~f~~ produced.

→ Forging can't be done after extrusion.

→ Density of extrudate can be improved by hot isostatic pressing (HIP). but generally it is not required as extrusion itself helps in improving density.

★★ Starting material should be near dense (i.e. No porosity). for extrusion or any other shaping process.
Otherwise initial force will be used to remove pores in the starting material.