MANUFACTURING PROCESS IN METALLURGY

POWDER METALLURGY

1. POWDER COMPACTION/PRESSING
2. CHARACTERISTICS OF GREEN COMPACTS

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COMPACTING

Press powder into the desired shape and size in dies using a hydraulic or mechanical press.

- Pressed powder is known as “green compact.” The green strength of the part when pressed is adequate for handling.
Compaction

High pressure is applied to squeeze the powder into the desired shape.
COMPACTION OF METAL POWDERS:

Compaction is an important step in powder processing as it enables the forming of loose metal powders into required shapes with sufficient strength to withstand till sintering is completed. In general, compaction is done without the application of heat. Loose powders are converted into required shape with sufficient strength to withstand ejection from the tools and subsequent sintering process. In cases like cemented carbide, hot compaction is done followed by sintering. This process can not be termed as compaction strictly, as sintering is also involved in this.
Powder compaction methods:

Powder compaction techniques can be classified as:

1. Methods without application of pressure:
   i) loose powder sintering in mould, ii) slip casting,
   iii) slurry casting, iv) Tape casting, v) vibratory compaction,

2. Methods with applied pressure:
   i) cold die compaction (single action pressing, double action pressing, floating die pressing), ii) isostatic pressing, iii) powder rolling, iv) powder extrusion, v) explosive compaction.

3. Hot compaction: Compaction + Sintering simultaneously.
PRESSURELESS COMPACTION TECHNIQUES:

No compaction is done i.e. no pressure is applied in the die. It is used for the production of simple and low density parts such as filters, bearings, other parts that are porous in nature. These techniques involve no external force and depend upon gravity for powder packing:

I) **Loose powder sintering:** - Also known as loose powder shaping, gravity sintering, pressureless sintering. In this method, the metal powder is vibrated mechanically into the mould, which is the negative impression of the product and heated to sintering temperature.
This is the simplest method and involve low cost equipment. The main reasons for not using this method for part production are, difficulty of part removal from the mould after sintering, & considerable shrinkage during sintering.

**Applications:** Amount of porosity ranges from 40 volume% to as high as 90 volume%; Highly porous filter materials made of bronze, stainless steel, and monel, porous nickel membrane for use as electrodes in alkaline storage batteries and fuel cells are typical examples.
II) Slip casting:

It is used for compacting metal and ceramic powders to make large & complex shapes for limited production runs.

A slip is a suspension of metal or ceramic powder (finer than 5 µm) in water or other soluble liquid, which is pored into a mould, dried and further sintered.

• Slip is usually made of, 1) a dispersion agent to stabilize the powder against colloidal forces, 2) a solvent to control the slip viscosity and facilitate casting, 3) a binder for giving green strength to the cast shape, 4) plasticizer to modify the properties of the binder.
For successful slip casting, formation of appropriate and a consistent slip is important. This is achieved by proper control of particle size, size distribution, order of component addition, their mixing time, addition of proper deflocculant - to prevent the settling and aggregation of powders and maintains the desirable viscosity of the slip.

Mostly water is used as suspending medium, but absolute alcohol or other organic liquids may also be employed. Additives like alginates – ammonium and sodium salts of alginic acids, serve three fold functions of deflocculant, suspension agent & binding agent to improve green strength of the compact.
• The slip to be cast is obtained in a form of suspension of powder in a suspending medium. The slip should have low viscosity & low rate of setting so that it can be readily poured. The slip cast should be readily removable from the mould. Low shrinkage and high strength after drying is expected.

• To obtain these properties, 5 µm powder particles should be used. In the case of fine molybdenum powders, a slip can be prepared by suspending the powder in 5% aqueous polyvinyl alcohol with a minimum viscosity, at a pH value of 7.
• For coarser, spherical stainless steel powder, a mixer of 80.7% metal powder, 19% water, 0.3% of sodium alginate as deflocculant having a pH value of 10 can be used.

• Steps in slip casting: i) Preparing assembled plaster mould, ii) filling the mould, iii) absorption of water from the slip into the porous mould, iv) removal of part from the mould, v) trimming of finished parts from the mould.

• Sometimes mould release agents like oil, graphite can be used.

• Hollow and multiple parts can be produced.
Figures shows the steps of slip casting:

i. Assembled mould
ii. Filling mould
iii. Absorption of water from slip
iv. Part removal
Advantages of slip casting:

Products that can not be produced by pressing operation can be made, no expensive equipment is required, works best with finest powder particles.

Disadvantage:

slow process, limited commercial applications.

Applications:

tubes, boats, crucibles, cones, turbine blades, rocket guidance fins; Also products with excellent surface finish like basins, water closets.
III) Slurry casting:

This process is similar to slip casting except that a slurry of metal powders with suitable liquids, various additives, and binders is poured into a mould and dried. The solvent is removed either by absorption into the POP or by evaporation. Very high porous sheet for use as electrodes in fuel cells and nickel-cadmium rechargeable batteries are produced by this method.
IV) Tape casting (doctor blade casting):

This is a variation of slurry casting process and is used to produce thin flat sheets.

- This process involves preparing a dispersion of metal or ceramic powder in a suitable solvent with the addition of dispersion agent (to improve the dispersion of the particles). Then a binder is added and fed to a reservoir. Whole mixture is fed on to a moving carrier film from the bottom of the reservoir.
Tape casting process.

Stages in tape Casting

Slip  Green Tape  Sintered Tape
• This slurry layer is deposited on the film by the shearing action of a blade. The slurry should be free of air bubbles, otherwise result in porosity. During sintering, the binder is burnt off first and densification of material occurs.

• In present days, endless stainless steel belt is used instead of carrier tape. This process can be used for making very thin tapes between 50 to 1000 µm thickness. This method is used for making electronic substrates, dielectrics for capacitors and piezoelectric actuators.
V) Vibratory compaction:

Vibratory compaction uses vibration energy to compact the powder mass. During this process, smaller voids can be filled with particles of still smaller size and this sequence is carried out till a high packing density of powder is achieved even before consolidation. Mechanical vibration facilitates the formation of nearly closed packed powder by settling particles in the voids present in the powder agglomerate. During vibration, small pneumatic pressure is usually superimposed on the powder mass.
• Brittle powders can be compacted by this method as they develop crack if done by pressure compaction.
• This method is generally used when, 1) powders have irregular shape, 2) use of plasticizers for forming is not desirable, 3) sintered density is required to be very close theoretical density.

**Important variables in vibratory compaction:**
1. inertia of system: larger the system, more the energy required for packing
2. friction force between particles: more friction results in need of more KE for compaction
3. particle size distribution: more frequency required if more large particles are present. Vibration cycle is important and not period of vibration.
PRESSURE COMPACTION TECHNIQUES

These techniques involve application of external pressure to compact the loose powder particles; Pressure applied can be unidirectional, bidirectional or hydrostatic in nature.

Die compaction:
In this process, loose powder is shaped in a die using a mechanical or hydraulic press giving rise to densification. The mechanisms of densification depend on the material and structural characteristics of powder particles.
Unidirectional and bidirectional compaction involves same number of steps. These are:
i) charging the powder mix.
ii) applying load using a punch or double punch to compact powders.
iii) removal of load by retracting the punch.
iv) ejection of green compact.
Compaction

High pressure is applied to squeeze the powder into the desired shape.
In single die/unidirectional compaction, powders close to the punch and die walls have higher force than in center. This results in variation in green density across the sample length. Longer the sample, more the density difference. This non-uniformity can result in non-uniformity in properties of sintered part. This density variation and hence, final property variation can be drastically be reduced by having double ended die/bidirectional compaction. In this case, powder experiences more uniform pressure from both top and bottom, resulting in minimization of density variation.
But this variation will still be considerable if the components have high aspect ratio (length to diameter ratio). This means that long rods and tubes cannot be produced by die compaction. In this case, isostatic pressing is more suitable.

i) Single ended compaction  
ii) Double ended compaction
Variables of compaction:

For a good compaction, following is required:

1) Irregular shaped particles give better interlocking resulting high green strength.

2) Apparent density of powders decides the die fill during compaction. Hence, powder size, shape & density affect the apparent density.

3) Flow rate of powders affects the die fill time, which depends on powder size, shape & density.

4) Pressure applied.

5) Rate of pressure applied.
Powder behavior during compaction: Compaction occurs in two stages:

1) Movement of powder particles past one another interacting with each other and with die-punch.

2) Particles deformation and fracture.

In the case of homogeneous compaction, two stages are observed:

In first stage, on application of pressure, rapid densification occurs due to particle movement and rearrangement resulting in improved packing;

In second stage, increase in pressure leads to elastic and plastic deformation resulting inter locking and cold welding of particles. In this stage, large pressure is needed for a small increase in density.
The green compact produced is considered as a two-phase aggregate consisting of powder particles and porosity each having its own shape and size. Compaction can be done at low and high temperatures. Room temperature compaction employs pressures in the range of 100-700 MPa and produce density in the range of 60- 90% of the theoretical density. At higher temperatures, pressures are kept low within the limits for preventing die damage.
LUBRICANTS:

Presence of frictional forces between powder particles & powder particle and powder particles & die walls limits the degree of densification of compacted products. Lubricants, mixed with powders and applied on contact surfaces of die, reduce the friction and aid to better packing. But, they may affect the densification property depending on their volume and density. Amount of lubricant added can be 0.5 to 2% by weight of charge. Even 1 wt% of lubricant can occupy large volume of approximately 5% and maximum attainable density will be 95% (assuming zero porosity) only.
Lubricants mixed with powders reduce the interparticle friction and aid to better packing. But, they may affect the densification property depending on their volume and density. The mixed lubricants should be removed before sintering to avoid distortion of compact.

Graphite & MoS2 can be applied physically on the die wall, punch surfaces. These can be easily removed, but takes longer production times.

**Lubricants:** Paraffin wax, Butyl stearate, Aluminium stearate, Lithium stearate, Zinc stearate, Magnesium stearate, Sodium stearate, stearic acid, Oleic acid, Polyglycols, Talc, Graphite, Boron nitride, MoS2. These lubricants have low boiling points.
DIE MATERIALS:

Dies for powder compaction can be made from a variety of materials. For soft powders like aluminium, copper, and lead, dies are made from abrasion-resistant steel such as air-hardened steels. Die steels can also be used. Tool steels are used for relatively hard powders for making dies because of their low cost and easy machinability. For more hard & abrasive powders like steel, tungsten carbide dies are used. But, carbide dies are costly & high hardness making it difficult for machining. Coated dies with hard & wear resistant material like titanium nitride or titanium carbide can be also used.
Defects occurring in die pressing of powders:

1) Lamination cracking:
   If air is not escaped from the die during compaction, air is trapped in the compacted products. Cracking in green products occurs in a direction perpendicular to the direction of loading. This trapped air prevents the interlocking of particles. Hence, there shall be small gap between die and punch so that air should escape easily during pressing. Further, pressing speed has to be suitably controlled so that air should escape.

2) Blowout:
   It occurs when all the entrapped air tries to escape at the interface between the die and punch.
The powder compacts can be compacted up to 80 - 90% of their theoretical densities. Water/oil/air/gas can be used as pressuring medium. The products result in good surface finish with uniform density and high green strength, which is not possible in die compaction processes. The process has flexibility of operation, cheaper, low tool cost, near-shape products and use of less amount of binder.

Following is the classification of Isostatic compaction process depending upon the temperature at which process is performed:

1. Cold isostatic compaction
2. Hot isostatic compaction
ISOSTATIC COMPACTION (CIP):

Isostatic compaction is a compaction process in which isostatic fluid pressure is applied to a powder mass to compact it into desired shape. It is most suitable for compaction of very hard, and ceramics materials with complex shape. Conventional die compaction processes have a major disadvantage that the size and shape of the products are limited and also the capacity of press is kept limited due to strength of die materials. Die compaction processes has also limitation in production of complex-shaped parts due to difficult in die fabrication and ejection of compact from die.
In cold isostatic compaction, pressure is applied simultaneously and equally in all directions using an isostatic fluid (Water/oil/gas/air) to the powder mass kept in a rubber or polymeric material or tin cane container/mould at room temperature to produce desired shape. The pressure applied is much lesser than die compaction and ranges between 100 to 800 MPa. The compaction time depends upon characteristics of powder, and of the order of few seconds. The powder parts can be compacted up to 80-90 % of their theoretical densities. High density near-net shape green products can readily be produced. After sintering, the products can reach up to 97 % of theoretical density.
In case of Hot isostatic compaction, powder kept in a container/mould, of high melting point, is compressed by applying high temperature of several hundreds to 2000 degree centigrade and isostatic pressure of several tons to 200 Mpa at the same time. Argon is mostly used as pressing media. The process improves plastic deformation during compaction. This process minimizes the internal micro-porosity to the greater extent, and thus increases the density of compact products and thereby improves mechanical properties. This process is most suitable for ceramic, refractory metal, cermet powders, metal matrix composites & dissimilar materials to produce near net-shape products.
SCHEMATIC DIAGRAMMES FOR COLD AND HOT ISOSTATIC PRESSING

COLD ISOSTATIC PRESSING

HOT ISOSTATIC PRESSING

“Green” Compact
Gas Pressure Inlet
Flexible Mold
Gas Inlet
Top Closure
Cylindrical Pressure Vessel
Heat Insulator
Material
Heater
Support
Bottom Closure
The following steps are involved in the production of compacts in these processes:

1. Filling of the powder in a flexible capsule and evacuation the same.

2. Placing the sealed capsule in the pressure vessel.

3. Maintaining the required temperature in case of hot isostatic compaction.

4. Application of pressure using a fluid.

Steps in cold isostatic compaction
(i) Mold filling (ii) Pressuring the mold (iii) Depressurizing (iv) Green compact
Uniform and good mould filling is important in this process because the initial powder distribution and density affect significantly the bag movement and hence the preform shape. Powder size, shape, density and mechanical properties influence the flowability of powder into the mould and the packing density. Optimum pressing is obtained by using a free-flowing powder along with controlled vibration or mould tapping.

Materials used for flexible moulds must be resilient, exhibit uniform elastic behaviour in all direction are natural, synthetic rubber like neoprene, urethane, nitrile, butly & silicones.
During pressing, high density is achieved at a low pressure, while the green strength of the compact rises linearly with pressure. Initially the applied stress serves to improve the density of the compact by particle sliding and rotation. In the next stage, deformation and fracture of powder particles occur and particle characteristics like shape play vital role in deciding this stage. Irregular shaped powders, interlock with one another and also deform during both the stages, tend to densify much easily than spherical powders.
In the case of spherical powders, in spite of their higher initial packing densities, particles do not mechanically interlock with one another and hence do not easily deform. Hence high pressures are required for their compaction.

After compaction stage is completed, the rate of depressurization of the chamber must be controlled to avoid cracking of the compacted products due to the release of entrapped gas in the compact.

Types of cold isostatic pressing:
1. Wet bag process
2. dry bag process
In wet bag process, the mould is removable, which is directly in contact with the fluid. This reduces the productivity, since the bag has to be removed every time before refilling.

In dry bag process, mould is fixed to the pressure vessel and powders are filled in situ. The mould is separated from the pressure fluid by a sleeve made of an elastomer. This is an automated process in which the powder filling, compaction, depressurization and removal of green parts are done continuously. This process gives higher production rate due to the faster cycle time compare to wet bag process. In this process, simple shapes are produced to help easy removal of compact.
POWDER ROLLING:
It is the process, in which, feeding of powders between two rolls moving in opposite direction is done to produce a coherent and brittle green strip continuously. This green strip is then sintered & re-rolled to obtain a dense, finished product. The process in mostly used for production of bimetallic and trimetallic strips and coinage strips from powders of close dimensional tolerance. In this process, rolls are mostly kept horizontally as gravity feeding of powders gives flexibility in controlling the powder feed and feeding of different powders simultaneously for multilayer strip production. Green density of 75-90% is achieved.
Powder Rolling

Powder is compressed in a rolling mill to form a strip
Steps in powder rolling:

1) Preparation of green strip.
2) Sintering.
3) Densification of sintered strip.
4) Final cold rolling and annealing.

1) Preparation of green strip:
In this step, feeding of powder mix (metal powder with binders to improve green strength) between rolls is done to get a green strip. During rolling, rearrangement of particles due to slip between particles and elastic & plastic deformation of particles takes place.
During rolling, powders pass through three zones, viz. free zone, feed zone and compaction zone. The strip produced is

Factors affecting powder rolling:
1. Roll gap: Increasing the roll gap, decreases the green density. Small roll gap results into edge cracking.
2. Roll diameter: Increasing roll diameter, increases density and strength for a given strip thickness.
3. Roll speed: It is kept low 0.3-0.5 m/s.
4. Powder characteristics: Irregular powders with rough surfaces, results in better interlocking & higher strip density.
2) Sintering:
The purpose of sintering is to increase the strength of strip, to remove the undesirable elements such as oxygen present as oxide on the surface of powder by using hydrogen atmosphere and sulphur as hydrogen sulphide from Nickel powder produced by hydrometallurgical process. Protective atmosphere is used to avoid oxidation of surface and subsurface pores. Continuous belt furnace is used for sintering. After sintering, sintered strip moves to rolling mill for densification.
3) Densification of sintered strip:
   The sintered strips are porous (~10%) and hence, to increase density, these are re-rolled. In densification stage, repeated cold rolling followed by annealing or hot rolling of strip is done.

4) Final cold rolling and annealing:
   After densification of sintered strip by repeated cold rolling and annealing, rolling & annealing are done simultaneously. Whereas after hot rolling of strip, a separate final cold rolling and annealing is done.
POWDER EXTRUSION

There are three ways, by which, extrusion is done:

1. Extrusion of loose powder.
2. Cold pressing and extrusion.
3. Canning followed by extrusion.

In extrusion of loose powder, loose powder is kept in a heated container and extruded directly through the die. Heat of container is enough to raise the temperature of the powder for extrusion.

In cold pressing and extrusion, powder is cold compacted, hot pressed and then extruded.
In canning followed by extrusion, powders are canned (placed in a metallic capsule made of copper or low carbon steel) and sealed after degassing. The canned powder is heated and extruded along with can. Then can material is removed by stripping or chemical etching.

(i) Extrusion of loose powder (ii) Cold pressing and extrusion (iii) Canning followed by extrusion.
EXPLOSIVE COMPACTION

In this process, high velocity shock wave is developed by detonating an explosive materials under water to impart compaction of powder kept in a plastic bag. The shock wave produced by the explosion heats up the powders resulting densification. High-pressure gases can also be used in place of explosives. The process is suitable for hard materials as well as for forming composites. Velocity up to 7 to 8 mph and pressure up to 150 to 300 k bar can be achieved depending upon the type of explosives used. The pressure is exerted only for few microseconds during compaction, resulting very good compaction. The amount of explosive to be used depends upon mass of powder.
CHARACTERISTICS OF GREEN COMPACTS:
The characteristics of green compacts affects the quality of end products. Following are important green compacts characteristics:
1. Green density – should be high.
2. Green strength – should be high.
3. Density distribution – should be uniform.
4. Entrapped air – should be minimum.
5. Defects – should be minimum.
6. Type of porosity: Closed pores are not good as compare to interconnected pores.
7. Lubricants-should be minimum after sintering.