A- Pressure compaction:

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

1. Single ended and double ended compaction

   hot pressing(uniaxial)

Die pressing is a very widely used process for forming ceramics. It is suitable for both fine and coarse grained ceramics.

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 stages and are described in this figure. They are

   i) charging the powder mix

   ii) applying load using a punch (uni-) or double punch (bi-) to compact powders,
iii) removal of load by retracting the punch,

iv) ejection of green compact.

The table gives compaction pressure ranges for metals and ceramics.

<table>
<thead>
<tr>
<th>Metals</th>
<th>70–275</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminium</td>
<td>400–700</td>
</tr>
<tr>
<td>Brass</td>
<td>200–275</td>
</tr>
<tr>
<td>Bronze</td>
<td>350–800</td>
</tr>
<tr>
<td>Iron</td>
<td>600–720</td>
</tr>
<tr>
<td>Iron-copper (2%) premix</td>
<td>70–140</td>
</tr>
<tr>
<td>Tungsten</td>
<td></td>
</tr>
<tr>
<td>Ceramics</td>
<td>100–140</td>
</tr>
<tr>
<td>Alumina</td>
<td>140–169</td>
</tr>
<tr>
<td>Carbon</td>
<td>150–400</td>
</tr>
<tr>
<td>Hard metals</td>
<td></td>
</tr>
<tr>
<td>Magnetic ceramics (ferrites)</td>
<td>110–165</td>
</tr>
</tbody>
</table>

**Effect of powder characteristics** For a good compaction,

1) irregular shaped particles are preferred as they give better interlocking and hence 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 affects the die fill time, and once again powder size, shape & density affect the flow rate.

**Powder behavior during compaction**

Compaction involves:

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

2) deformation of particles. In the case of homogeneous compaction, two stages are observed.
First stage => rapid densification occurs when pressure is applied due to particle movement and rearrangement resulting in improved packing;
Second stage => increase in applied pressure leads to elastic and plastic deformation resulting in locking and cold welding of particles.
In the second stage, large increments in pressures are seen to effect a small increase in density.

- The green compact produced can be considered as a two-phase aggregate consisting of powder particles and porosity each having 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
In single die compaction,

powders close to the punch and die walls experience much better force than in center. This results in green density variation 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 greatly reduced by having double ended die 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 can be used.
Die compaction lubricants
- It is known that presence of frictional forces limits the degree of densification.
- Usage of lubricants either mixed or applied to contact surfaces can be done to minimize friction

- Lubricants => organic compounds such as waxes or metallic stearates or salts and they generally have low boiling points; Amount of lubricant added can be 0.5 to 2 % by weight of charge

- Mixed lubrication => Reduce the interparticle friction and aid 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.
- Even 1 wt% of lubricant can occupy large volume of app. 5% and maximum attainable density will be 95% (assuming zero porosity) only.

- Die wall lubrication => Graphite & MoS2 can be applied physically on the die, punch surfaces; They can be easily removed, but takes longer production times.

- Commonly used lubricants in P/M => Paraffin wax, Aluminium stearate, Lithium stearate, Zinc stearate, Magnesium stearate, stearic acid, Oleic acid, Talc, Graphite, boron nitride, Mos2

**Die materials**
Soft powders like aluminium, copper, lead => abrasion resistant steel such as airhardened steels, die steels are used for making die .Relatively hard powders => dies made of tool steel are used More hard & abrasive powders like steel => tungsten carbide dies are used. But carbide dies are costly & high hardness (difficult to machine) .Coated dies with hard & wear resistant coating material like titanium nitride or titanium carbide can be used.
2. **Isostatic pressing**

**Cold isostatic pressing (CIP) and hot isostatic pressing (HIP)**

**Cold isostatic compaction (CIP)**

- CIP is a compaction process in which isostatic fluid pressure is applied to a powder mass at room temperature to compact it into desired shape. The powder parts can be compacted up to 80-90% of their theoretical densities. Water or oil can be used as pressuring medium.

- Process details: High density near-net shape green parts, long thin walled cylinders, parts with undercuts can be readily fabricated.

  In this process, pressure is applied simultaneously and equally in all directions using a fluid to an elastomeric fluid with powder at room temperature.

  Sintered CIP component can reach up to 97% of theoretical density. Steps in this process is shown in flowchart.

![Flowchart](chart.png)

Good mould filling is required in CIP because the initial powder distribution and density affect the preform shape. Powder size, shape, density and mechanical properties affect 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 are natural, synthetic rubber like neoprene, urethane, nitrile, silicones.

- During pressing, high density is achieved at a low pressure, while the green strength of the compact rises linearly with pressure.

The pressure applied can range from 100- 400 MPa.

Initially the applied stress (exactly shear stress) serves to improve the density of the compact by particle sliding and rotation. In the next stage, deformation of powder particles occur and particle characteristics like shape play vital role in deciding this stage.

- Irregular particles which 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.

Types of cold isostatic pressing: CIP

- Wet bag process: IN this, the mould is directly in contact with the fluid. This reduces the productivity, since the bag has to be removed every time before refilling. Tooling costs are reduced in this.

- Fixed mould process: the mould is fixed in the pressure vessel and powders are filled in situ. The tooling has internal channel into which fluid is pumped. This is an automated process in which the powder filling, compaction, depressurization and removal of green parts are done continuously. This involves higher tooling cost, but has higher production rate.

**Hot isostatic pressing**

- Ideal method for consolidation of powders of nickel and cobalt base super alloys, tool steels, maraging steels, titanium alloys, refractory metal powders, cermets. It has got variety of applications including bonding of dissimilar materials, consolidation of plasma coatings, processing hard and soft magnetic materials etc.

- HIP is the application of pressure at elevated temperatures to obtain net or near net shape parts from metal, ceramic, cermet powders.

- HIP unit consists of a pressure vessel, high temperature furnace, pressurizing system, controls and auxiliary systems (material handling, vacuum pumps, metering pumps).

- The pressure vessel is made of low alloy steel. Its function is to heat the powders while applying uniform gas pressure on all the sides.

Furnaces are of radiation or convection type heating furnaces with graphite or molybdenum heating elements.

Nichrome is also used. The furnace heats the powder part, while pressurizing medium (a gas) is used to apply a high pressure during the process.

Generally, argon, nitrogen, helium or even air is used as pressurizing medium.
- The pressurizing gas, usually argon, is let into the vessel and then a compressor is used to increase the pressure to the desired level. The furnace is then started and both temperature and pressure are increased to a required value.

3. **Continuous Compaction**

A. Powder rolling

This process involves feeding of powders between rolls to produce a coherent and brittle green strip. This green strip is then sintered & re-rolled to obtain a dense, finished product.
Steps: 1) preparation of green strip, 2) sintering, 3) densification of sintered strip, 4) final cold rolling and annealing

Parameters affecting powder rolling are roll gap, roll diameter, roll speed, powder characteristics;

- Roll gap => large roll gap leads to decrease in green density; very small roll gap leads to edge cracking;
- roll diameter => increase in density and strength with increase in roll dia. for a given strip thickness;
- roll speed => Kept low, 0.3-0.5 m/s;
- Powder => irregular powder with rough surfaces provide better strip density

In densification stage, either repeated cold rolling followed by annealing or hot rolling of strip can be followed.

HIP presses are available in diameters up to 2m with pressures ranges from 40 to 300 MPa with temperature range from 500 to 2200 °C.
The processing time can last up to 4 hours depending on the material and size of the part.
- during HIP, the pores are closed by flow of matter by diffusion and creep, but also bonded across the interface to form a continuous material.
- Commonly used heating elements: Kanthal heating element – up to 1200 °C; Molybdenum heating element – 1200 to 1700 °C; Graphite heating element – 2000 to 2600 °C
- Typical range of temperature & pressures used in HIP is given in table

<table>
<thead>
<tr>
<th>Material</th>
<th>HIP temperature range (°C)</th>
<th>Applied pressure (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al alloy</td>
<td>350–500</td>
<td>100</td>
</tr>
<tr>
<td>Cu alloy</td>
<td>590–900</td>
<td>100</td>
</tr>
<tr>
<td>High-speed steel</td>
<td>1,000–1,200</td>
<td>100</td>
</tr>
<tr>
<td>Ni base superalloy</td>
<td>1,170–1,280</td>
<td>100–150</td>
</tr>
<tr>
<td>Ti alloy (Ti-6Al-4V)</td>
<td>880–960</td>
<td>100</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>1,350–1,450</td>
<td>100</td>
</tr>
<tr>
<td>Ba Ti O₃</td>
<td>100–1,200</td>
<td>100</td>
</tr>
<tr>
<td>Partially stabilized zirconia</td>
<td>1,350–1,500</td>
<td>100</td>
</tr>
<tr>
<td>SiAlON</td>
<td>1,700–1,800</td>
<td>100</td>
</tr>
<tr>
<td>WC-Co</td>
<td>1,300–1,350</td>
<td>30–100</td>
</tr>
</tbody>
</table>

4. **Powder Extrusion**

Powder Extrusion Moulding (PEM) is a development of conventional polymer extrusion processes. This technology consists of four stages: Mixing, extrusion, binder removal and sintering. In the first stage a metallic or ceramic powder is mixed with a binder system usually formed by a thermoplastic polymer and lubricants. This mixture, named “feedstock”, is fed into an extrusion machine where is melted and passed through a nozzle that shapes the profile. The cross-sectional area of this profile is constant.
where $D_c$ is the compacted density, $D_T$ is the full density of the material, $\Delta D$ is the difference between full and compacted density, and are constants, and $E$ is the net energy absorbed by the powder.

Next, in the binder removal stage, the organic component that served as a vehicle during extrusion stage is decomposed. Commonly, the elimination takes place by means of thermal degradation. Finally, in the sintering stage, the part is subjected to a high temperature thermal cycle to densifie and get final dimension.

Previous to mixing, an important issue is powder selection (ceramic or metallic) in accordance to final application. Powder characteristics, such as size and particle size distribution are crucial for “feedstock” formulation and determine the results obtained at the different stages of the process. Binder system selection should be based on rheological behaviour, powder-binder interaction, processing temperature and decomposition range, etc. It can be said that binder should be tailored to the powder in accordance to the final application.

During mixing stage a suitable powder-binder ratio should be determined. This ratio is a commitment between obtaining the suitable rheological behaviour for the extrusion step and simultaneously to obtain final parts with the required density. For example, if final pieces with high porosity are required it will not be a problem to reduce powder loading in “feedstock” which in addition will improve the flow properties during the extrusion stage. Nevertheless, in this case special attention should be paid to the dimensional stability of the profile after the following stages of the process. In the extrusion stage parameters such as temperature profile, rotor speed and wire
drawing, etc should be carefully controlled. In the removal stage a thermal cycle should be designed in accordance to the binder system used. Binder removal should be carried out gradually to avoid defects in the final part. Finally, in the sintering stage the thermal cycle is designed to obtain the required properties according to the final application.

Innovative Aspects:

This technology allows to produce metallic or ceramic profiles with high accuracy. Since starting material are powders and an organic vehicle, usually constituted by thermoplastic polymers and some lubricants, the materials can be processed at polymer processing temperature avoiding high processing temperatures commonly used for metallic or ceramic processing. On the other hand final parts have high dimensional accuracy and the porosity of the profile can be controlled by establishing a suitable powder-binder ratio or by carefully designing thermal cycle during sintering stage. The research group has managed to produce pieces with the required porosity according to the final application.
One of the advantages of this technology is the possibility of obtaining metallic and ceramic profiles with high dimensional accuracy. In the case of hollow profiles very thin thicknesses of wall can be obtained (of the order of microns), also, in the case of solid profiles the diameters can be very small. Additionally, when certain permeability is required the porosity of the profile can be controlled. If, on the opposite way, the final application requires a watertight profile, parts with high densities can be produced. Additionally, the incorporation of this technology can represent a cost reduction coming from a smaller energy demand since processing temperature of ceramics and metallics materials is diminished substantially.

5. **Explosive compaction**

It used for pressing hard particles. The powder are placed in water proof bags which are immersed in water. It contained in a cylinder having wall thickness. Due to sudden deformation of change at the end of cylinder the pressure in the cylinder increases. The pressure used to press the metal powders to form green compact.

6. **Vibratory Compacting**

Vibration can be very effective in obtaining higher packed densities in powders.
The relative densities of powders vibrated under carefully controlled conditions are much higher than those obtained by simply pouring the powder into the container. Therefore, much lower compaction pressures are required to reach a given density for a vibrated powder than for a poured powder. This is illustrated in Fig. 8 for a carbonyl iron powder.

The density of 5.53 g/cm³ (71% relative density), reached by compacting under a pressure of 245 MPa (35 ksi), is due to the plastic deformation of the iron powder particles, while the 5.37 g/cm³ (69% relative density) obtained by vibrating at 167 oscillations per second is due mainly to vibratory packing. Plastic deformation during the simultaneous compacting at 2.4 MPa (0.36 ksi) is minimal.

The method of consolidating powders by vibrating and simultaneous compacting is, therefore, primarily applicable to hard powders, such as refractory metal and cemented carbide powders, which can be densified relatively little by pressure application alone.

![Image of a vibration press]

Theoretical model of vibration pressing.

\[ K_3 = \frac{C \sigma d}{2} \]

where \( C \) is a coefficient which takes into account the effect of the static force \( P \) and the dynamic force \( f \), \( \sigma \) is the shear modulus of the material, and \( d \) is the diameter of the part to be compacted.
Effect of powder vibration on densities of FeO$_2$ compacts.

- Obtained in static pressing.
- Vibratory compacting at a frequency of 233 oscillations per second.
- Vibratory compacting at a frequency of 167 oscillations per second.