Full Density Processing

In the ideal, a component is fabricated with final dimensions requiring no post-consolidation machining. That goal is termed 'net shaping,' and full density PM is one of the best approaches.

Eliminating residual pores while obtaining the desired final dimensions is the challenge. With prolonged sintering, the pore structure becomes stabilized and is difficult to remove from a compact via diffusion. However, the properties of many PM materials can be improved. The ability to control microstructure, segregation, grain size, inclusion population, and the material texture has motivated considerable exploration of full density PM processing.

A basic conflict of full density processing is that the actions necessary for densification often add the greatest expense. The conflict between performance and cost may be resolved in many ways. The best approach is to determine the properties needed in a situation and then decide on the density necessary for the level of performance.
Some of the options between performance and porosity are shown in Figure. Although not comprehensive, various processes are located on the performance vs. density plot in terms of compact size.

Although the figure is schematic, it provides a first view of full density processing which stimulates 4 questions: First, are the properties actually needed? Second, what are the limitations in terms of materials, processes, properties, and size? Third, can the cost of the processing options be justified? Finally, would changes in the material, technology, or density be most appropriate?

Generally increased performance implies a higher density. For smaller components and moderate performance levels, traditional press and sinter technology is most useful. As the component shape complexity increases, the P/M becomes a better alternative.

Figure  Three of the variables that influence the selection of a powder metallurgy processing methods - component size, density, and performance (as a percentage of wrought). That behavior corresponds to ferrous based P/M systems formed from coarse powder, but is representative of many powder metallurgy materials. The symbols are P/S = press and sinter; reP = press, sinter and repress; P/S+F = press, sinter and forge; CIP+S = cold isostatically press and sinter; HIP = hot isostatically press; HIP+F = hot isostatically press and forge.
High densities are possible via high sintering temperatures.

Larger shapes require isostatic compaction and can be densified by either hot pressing or forging.

Surface contamination of the powder is a chronic problem with PM processing. If the contamination is an oxide or similar inert film (possibly a nitride or carbide), then particle bonding will be weak. Alternatively, if the surface is enriched with one component of prealloyed powder, then subsequent ppt of compounds is possible. Both problems lead to the formation of prior particle boundary ppt in the consolidated compact. The ppt of second phase provides for easy fracture along the particle contacts.

3 steps will counteract prior particle boundary contamination:

i) the powder must be rapidly quenched to avoid initial segregation; thus atomized powders are usually preferred. ii) the powder must be maintained in a high purity environment to avoid the formation of contamination films before consolidation. iii) extensive plastic deformation during consolidation ensures disruption of any continuous surface films which can degrade properties.
Density vs. Alloying

The effects of various alloying additions on the strength of common metals are well known. In powder processing, the alloying addition and the density of the compact both affect final properties. The strength, ductility, and toughness all improve with alloying and densification. alloying additions in the powder (prealloyed) may further increase the inherent material strength (fig.), but also contribute to greater processing difficulty.

![Graph showing tensile strength vs. alloying content](image)

- Tensile strength, MPa
- Alloying content, wt. %
- Ferrite strengthening

![Graphs showing ultimate and yield strength](image)

- Ultimate strength, MPa
- Yield strength, MPa
- Sintered density, g/cm³
- Fe + 4% Ni
- 0.8%C
- 0.4%C
- 0.0%C

![Graph showing impact energy](image)

- Impact energy, J
- Elongation, %
For example, compressibility of iron decreases rapidly with alloying. The penalty of alloying is increased difficulty in achieving full density. However, the baseline mechanical properties are improved by appropriate alloying additions.

For high performance applications, especially those involving impact and cyclic loading, alloying and densification are considered in conjunction with the optimal microstructure. In such cases, it is necessary to develop techniques applicable to full densification. Typically, this implies the use of small powders or the application of an external force on the powder compact to eliminate pores.
Advantages and Disadvantages of Full Density Processing

In most instances, the performance of PM materials improves with a higher density. Attaining full density is difficult. Therefore, full density provides on the one hand improved properties, on the other hand it represents high level of difficulty and expense over traditional pressing and sintering processes.

More specialized equipment is often required to obtain full densities. Additionally, dimensional control can be lost with some of the techniques. The type of powder most responsive to full density processing is a clean, prealloyed powder, usually a specialized input material.

Full density PM techniques use material more efficiently than other metalworking technologies, with better process control and increased production.

Another advantage of full density powder product is the wide range of shapes, sizes and materials available.
Solid-State Sintering: Although this chapter emphasizes the simultaneous use of stress and temperature to achieve full densification, it should be pointed out that many small powders can be sintered to full density, especially at relatively high sintering temperatures.

The initial compact shape can be generated by any of several methods, including die compaction, cold isostatic compaction, injection molding, slip casting, tape casting. Sintering critically depends on combination of processing factors such as particle size, time, temperature, and green density. Significant diffusion can be induced by heating a compact close to the melting temperature using small particle sizes.

Enhanced Sintering Techniques: A fast diffusion phase greatly aids sintering densification. Liquid phase sintering has high potential for full densification of small particles. The liquid provides a high diffusivity pathway for atomic motion and a strong capillary force that induces particle compression at point contacts. However, the liquid must be wetting not the particles and have solubility for them to allow densification.
Liquid phase sintering is widely employed to obtain full density products without the application of pressure during sintering. Applications include tool steels, stainless steel, W-Ni-Fe heavy alloys, Co-Sm and Fe-based magnets, WC-Co cemented carbides, TiC-Re cermets, and nickel superalloys.

Infiltration: Another technique for obtaining toughness is to infiltrate the remaining pore structure after sintering as shown below. For a wetting liquid melt, the capillary pressure $\Delta P$ varies with the inverse of the pore size $d$, as follows:

$$\Delta P = \frac{2 \gamma \cos(\theta)}{d}$$

where $\gamma$ is the surface energy of the liquid and $\theta$ is the contact angle. A good example is copper infiltration of iron. The capillary pressure causes liquid to flow into the open pore structure.
Although the flow of liquid into the pores is beneficial, there are also some possible problems with infiltration. Typically, the infiltrant is formed on one surface of the sintered material and capillary action draws the liquid into the pores. Because of directional flow of the liquid, it may erode the surface from which the infiltrant is fed. For that reason, infiltration cycle times are short, to keep dimensional changes to less than 2%.

Also, since the capillary pressure scales with the inverse of the pore size, there is little driving force for infiltration of large pores. Since the large pores are most detrimental to properties, for max. benefit, one must ensure they are filled.

The ideal infiltrant will completely fill the pore space, exhibit good flow and wetting of the pore structure, and will not leave a residue.
Consolidation Fundamentals

Infiltration and enhanced sintering treatments are full density processing methods which do not require external stress. Many full density methods do employ various combinations of temperature and stress. The traditional cycle involves sequential compaction or shaping at low temperatures followed by sintering as a second operation. Hot consolidation of powder combines the compaction and sintering steps into one operation.

The simultaneous heating and pressurization events add cost and complexity that are best justified by increased performance.

The four concerns in discussing hot consolidation of powders are: i) temperature, ii) stress, iii) strain, and iv) strain rate.

Full density processing is normally ineffective at temperatures below approx. one half of the absolute melting T. Temperatures between 70 and 85% of Tm are typical.
ii) Because pores act as stress concentrators, the effective stress at the inter-particle contacts is higher than the external stress. Only at full density does the effective stress equal the applied stress.

iii) The flow and deformation at particle-particle contacts is important to the quality of the particle bonds. Large shear strains disrupt surface films on the particles but contribute to tool wear and component cracking.

iv) The strain rate is the 4th variable of concern. A high strain rate allows less recovery and reduces ductility, thus fracture is more likely. Alternatively, a low strain rate gives more plastic deformation of the compact with a higher final density. The strain rate determines the rate of work hardening and the recovery rate.
Three mechanisms are important to pore elimination:

1. Diffusional creep is the dominant process in hot pressing and hot isostatic pressing during the final elimination of porosity. Plastic flow occurs when the effective stress exceeds the yield strength at the compaction temperature. Densification occurs instantly as the effective stress exceeds the material yield strength. At lower stresses, diffusional flow along grain boundaries or through the lattice combines with the stress to give densification in a process similar to creep.
The basic features of hot consolidation can be combined into densification maps.

Figure gives 2 such maps for a 50 \( \mu \text{m} \) particle size tool steel consolidated at either a constant temp or constant pressure. The region of dominance for each mechanism is outlined in terms of density vs. pressure and temperature. The left plot is for 1200°C and shows the density vs. applied pressure for isothermal times of 0.25, 0.5, 1, 2, or 4 hours (dashed line).

Densification at that temperature is controlled by diffusion. The right plot corresponds to a variable temperature and constant 100 MPa pressure. The density is shown for various processing temperatures and times.

![Densification maps for tool steel with 50 \( \mu \text{m} \) particles.](image)
Hot Consolidation Techniques

Uniaxial Hot Pressing: Hot pressing can be performed in a rigid die using uniaxial pressurization as shown in Figure. Note the features are similar to die compaction. The die is usually made from graphite to allow external induction heating. Other common die materials are refractory metals and their alloys, and sometimes ceramics such as silicon carbide can be used at low stresses.

If the compact exhibits an incompatible thermal expansion coefficient, then cooling may occur during cooling. In such cases it is appropriate to eject the compact at high temperature.

Uniaxial hot pressing is slow and inherently has poor control over the heating and cooling stages because of the large thermal mass associated with tooling. Typical max Ts are 2200°C and max pressures are 50 MPa. Vacuum is often selected for the process environment to minimize contamination of the compact.
The dies and apparatus can be expensive, especially if pressing is performed under vacuum. Contamination of the compact from the die is a common problem. However, uniaxial hot pressing is widely used to fabricate unique compositions and composites. One large commercial use for uniaxial pressing is in the consolidation of diamond-metal composite cutting tools.

**Hot Isostatic Compaction**: Flexible dies are used in hot isostatic pressing with isotropic pressurization. The primary control parameters are pressure (stress), temperature and time.

Figure shows a schematic of the hot isostatic pressing (HIP) sequence. For loose powder, a gas-tight container is used to shape the powder. The container may be fabricated from any material that is deformable at the consolidation temp. Prior to HIP consolidation, the filled container is heated and vacuum degassed to remove volatile contaminants. After prolonged evacuation and degassing, the container is sealed.
The consolidation of the powder container occurs in an intensely heated, cold-wall pressure vessel. High pressure gases, such as argon or nitrogen, is used to transfer heat and pressure to the compact, giving densification. Because the stress rise is slow, it is considered a low strain rate process. Temperatures up to 2200°C and pressures up to 200 MPa are possible using HIP. After the HIP cycle the compact is removed and the container stripped from the densified compact. That method is applied to the consolidation of many aerospace alloys (nickel superalloys, titanium, and aluminium), composites, and tool steels. It is most useful for large components where full density and isotropic properties are required.

One variant is to use previously sintered compacts with densities over 92% of theoretical, which corresponds to the point of pore closure. In that case, the component already has the desired shape and the closed pores allow for HIP. This approach is widely used to consolidate cemented carbides, wear materials, and titanium implants.
Typically, the surfaces of the compact are contaminated by the container and may need to be removed by chemical dissolution, machining, or abrasion.

**Powder Forging**: Forging is a high strain rate deformation process, typically conducted at elevated temperatures where the material has a low strength and high ductility. Conventional forging of cast material requires several steps to convert a billet into the final shape.

A sintered powder preform with 10 to 25% porosity can be densified in a single forging strike. Traditional press and sinter powder metallurgy is used to fabricate precursor material for forging. The preform shape and density have a large influence on the success of the forging operation.

The behavior of the porous preform in plastic flow is the main concern in powder forging. A schematic drawing of the deformation of a simple PM compact is shown.

<table>
<thead>
<tr>
<th>Density</th>
<th>Preform</th>
<th>Increasing Forging Strain</th>
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<tr>
<td>75%</td>
<td>![Preform Image]</td>
<td>![Increasing Strain Images]</td>
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Powder forging is a combination of densification and flow under uniaxial forces. The pore collapse in forging is significantly different from that encountered under the hydrostatic conditions used in HIP. Figure below illustrates the higher shear experienced by a pore during forging and contrasts that with the uniform deformation experienced in isostatic compaction.

Lubricants have a large effect on the density uniformity of forged compacts. Without lubrication, the forged powder exhibits low density regions because of drag on the punch and die wall. Friction causes circumferential tensile stresses as flow occurs in the die, which may lead to cracking.
Temperature determines the stress necessary to achieve densification. Typical ferrous forging operations do not exceed 1200°C, and many are forged at high temperatures closer to 800°C. The preform size and density are determined by the lateral constraints of the die, the part shape, and the need to obtain full densities in a single forging strike.

The properties from powder forging are attractive, as shown in table below. The strength is significantly higher using powder forging, although the ductility is decreased. Inclusions seriously degrade properties, in spite of full densification. The inclusions result from oxidation of the preform in the porous state, contaminants entered in the pores, and lubricants from the die.

The preform must be kept clean to prevent entrapment of such inclusions.
Powder Extrusion: The extrusion of a metal powder at an elevated temperature is another method to achieve full density. Precalloyed powders are commonly used with a high extrusion deformation to obtain maximal properties. Long shapes with a constant cross section are the main products of extrusion.

By boxing the powder in a vacuum tight container, it can be degassed and extruded hot. Both densification and shaping are achieved in one operation.

A schematic drawing of powder extrusion is shown below. A small penetrator in the extrusion ram directly stresses the powder and thereby avoids buckling the can. The extrusion constant $C$ provides a measure of the difficulty in achieving deformation and flow of the powder. The extrusion force $F$ and extrusion constant are related as follows:

$$F = CAh(R)$$

where $A$ is the cross-sectional area of the feed material and $R$ is the reduction ratio. The reduction ratio equals the area of billet divided by the area of the product.
Cold Consolidation Techniques

Powder Rolling: Most approaches to full density PM products involve simultaneous action of temperature and stress. However, there are techniques that achieve densification with a cold compact. Conventional rolling technology is used to densify a porous feed material. A loose powder is gravity fed into the gap between two rolls which generate the green sheet. A binder can be added to the powder to enhance green strength.

The higher the initial powder density and the greater the reduction in thickness or rolling, then the higher the final density. Green densities after the first pass through the rolling mill range from 60 to 90% of theoretical. The efficiency of densification initially increases with the application of a higher roll force; however, at higher densities the process loses efficiency.
In a single-pass rolling operation, densities of 99.8% of theoretical have been achieved, but multiple-pass treatments are more common. The two major technical problems in roll densification of powders are slipping and cracking of the feed material. Obviously, the final product is limited in geometry and powder rolling is generally restricted to forming sheet products. Powder rolling is employed in the fabrication of iron, copper, aluminum, nickel, steel, stainless steel, Mo-Cu, Co-Fe, Cu-Pb, and composites.

**Cold Forging**: Loose powder and sintered PM preforms can be cold deformed and fully densified under certain conditions. For example, the high pressure cold compaction of a tool steel powder is plotted, showing that pressures over 2 GPa are required for 95% density, and over 3 GPa are required for 98% density. Such high pressures are rarely used in compaction because of severe die wear and possible damage to the tooling. The applied stress for 100% density is typically 5 to 8 times the yield strength of the material. It is expensive and difficult to obtain tooling that can withstand such stresses.
High pressure cold compaction or cold forging is usually applied to low strength materials or compacts heat treated for minimized strength and maximized ductility. In those cases densification without fracture is possible if lateral constraint is provided during compaction. Careful tool design ensures defect-free components.

**Spray Forming**

Liquid Spraying: Spray forming uses an inert gas atomizer and substrate closely positioned below the atomization nozzle. As shown in figure, the spray is collected on the substrate where it is rapidly solidifies after undergoing deformation. Usually the spraying conditions are adjusted so the droplets arrive in semi-solid condition. This allows shear deformation. The rapid heat extraction delivers microstructural homogeneity. In some instances the product is sufficiently dense for direct use while in other instances it is subjected to subsequent hot forging, extrusion, or casting. Only simple shapes are possible using spray deposition.
Plasma Spraying

In a manner similar to spray forming, the use of a plasma torch (or alternatively a flame torch) deposits dense coatings. Powder is fed into a plasma arc and heated rapidly. The plasma is generated by a voltage gradient between the electrodes where the temperature exceeds 5000 °C, with a theoretical limit of 30,000 °C. At high gas flow between the electrodes, the arc extends the arc and accelerates the powder through the arc. The metal powder melts and is projected onto an external substrate as a liquid droplets. On reaching the substrate, the droplets splat into a layered structure. If the droplets have solidified during flight, then they will bounce-off semi-molten droplets adhere and solidify on the substrate.
The heat transfer to the powder is crucial for obtaining high density deposits. The powder must absorb the heat of fusion plus the necessary heat to satisfy the heat capacity. The difficulty in melting a material increases with the enthalpy of fusion and density.

Considering the short dwell time in the plasma arc, the particle size must be sufficiently small to allow adequate heat flow. However, too small particle size can be detrimental because of powder vaporization and agglomeration. Generally, a particle size in 40 to 80 μm range is optimal, but finer sizes are needed with the higher melting point metals.

Deposit density usually is about 85% of theoretical when deposition takes place with 1 atm pressure around the substrate. If the spraying is performed in vacuum, then deposit densities 95 to 99% of theoretical are attainable.

The formation of dense protective coatings is main application. The deposit serves as corrosion, oxidation, or thermal protective to the substrate.
Finishing Operations

Component production does not end with sintering or densification, since additional steps may be needed to meet the application requirements. Successful execution of the finishing operations can add considerable value to PM products by tailoring the component for the application and service conditions.

Examples of finishing operation activities include drilling holes, tapping threads, joining components into assemblies, repressing to adjust dimensions, flattening or smoothing rough surfaces, removing burrs, surface hardening through carburization, strengthening via heat treating, plating with a rust preventing coating.

To remain competitive and viable as a manufacturing technique, PM must deliver functional materials that involves finishing operations.
Components that are porous after sintering may require further deformation to adjust dimensions and to close pores. Figure schematically suggest the dimensional convergence to a narrower distribution by the use of a repressing operation. Depending on the degree of deformation, the post-sintering mechanical deformation treatments are known as repressing, sizing, and coming. They involve less plastic deformation than the classic densification steps of hot pressing, forging or extrusion.

Repressing is a restrike operation similar to forging, except it is used to adjust final dimensions and density without large shape changes. The sintered compact is placed in a closed die and subjected to small strains at moderately high strain rates. As a result of the strike, the compact is densified, strengthened, and adjusted for dimensions and surface finish.
Sizing is also used to adjust the final compact dimensions. For example, it is used to give a precise hole diameter to a bushing or to ensure conformance with a site specification.

Coining is a technique for adding surface configurations to a sintered blank. The term comes from the historic use of the technique for fabrication of coins.

Restrike operations are useful for closing surface pores, improving surface finish, increasing surface hardness, increasing precision and adding detail to the component.

**Machining:** Machining involves the removal of mass from the PM product to add threads, grooves/channels, undercuts, and special features that cannot be incorporated into the original tooling. Further, machining provides another means of adjusting dimension or surface roughness.

Those adjustments are made by removing mass from the component using high speed, cutting techniques.
Example Machining Operations

boring - single point rotating tool, stationary work, used to cut large holes or large diameter circular contours

broaching - bar with multiple shaped tool tips of increasing size, stationary work, reciprocating push and pull motion of tool over work cuts grooves, holes or other features

burnishing - reciprocating round hard tool, stationary work, surface smoothing by friction that causes shear displacement to harden, smooth, or finish surface

drilling - rotating fluted tool (usually with multiple tips), stationary work, cutting rotation removes mass to form hole

electrical discharge machining - electrical spark machining across a small gap filled with a dielectric fluid, progressive sparks evaporate and expel material to form tool profile in work

grinding - moving abrasive wheel, stationary or rotating work, used to generate a smooth surface (similar operations are honing and lapping)

hobbing - rotating cutting tool with teeth arranged along helical thread, used for cutting gear teeth by synchronized rotation of both the hob tool and work

honing - an abrasive finishing operation that uses fine grit rotating abrasive wheels to produce accurate dimensions and excellent finishes

lapping - fine abrasive grit loaded into a rotating flat disk, work moves over rotating disk to attain flat surface and smooth finish

milling - rotating tool with multiple tips, stationary work or reciprocating work, generates flat surface

planing - single point stationary tool, reciprocating work motion, used to form flat surfaces

reaming - rotating tool with cutting edges on fluted outer diameter, stationary work, used to make small cuts to size, contour, or improve hole accuracy

shaping - similar to planing except the work is stationary and the tool moves in a reciprocating motion to form a flat surface

tapping - rotating conical or cylindrical cutting tool with threads on periphery, used to cut screw threads in a hole

turning - stationary tool, revolving work, used to remove surface to form circular profiles
Generally, for PM materials, the machining operations are minimized when possible and only used to add features or adjust dimensions on oversized parts. The material hardness and residual porosity affect the tool life.

The stress on the cutting tool tip is cyclic since the cutting path is interrupted by pores. Accordingly, the tool will experience fatigue failure in cutting a porous material. That problem can be reduced by infiltrating the pores with a low melting temperature metal before machining; for ex. by copper infiltration of the surface to be machined on a steel component.
Lubricants are used in most machining operations, but care must be exercised to minimize contamination by lubricant entrapment in pores. That requires cleaning after machining, especially if the component is to be heat treated. Additives to improve machinability can be included in the initial powder mixture for components requiring it.

In ferrous PM materials, alloying changes and heat treatments influence the ease of machining. Very soft and very hard materials are the most difficult to machine, therefore there is a sensitivity to carbon level. In some instances it is possible to rough machine the component in the green or presintered conditions to minimize final material removal. However, best dimensional precision is attained by machining the component after heat treatment.
Heat Treatment

Heat treatments are used to tailor the phases, microstructure, and distribution of alloying elements after fabrication of a component. Heat treatments are used for surface hardening to improve wear resistance and hardening all through a component to improve strength. In most cases, heat treatments are applied to alloys that undergo phase changes with T. For ferrous PM alloys, approx. 60% are subjected to a heat treatment after sintering. For aluminium alloys, precipitation hardening is common; in titanium alloys, the polymorphic transformation allows considerable manipulation of the final properties. For tool steels, heat treatments are used to develop a high hardness microstructure for strength and wear resistance.

Heat treatments are best understood in ferrous systems. The transformation from austenite (FCC iron) to ferrite (BCC iron) is impeded by alloying additions, especially carbon. Manipulations adjust the carbide $\text{Fe}_3\text{C}$ formation. Formation or generate a metastable phase known as martensite (body-centered tetragonal Fe).
As the alloying level decreases, especially as carbon is removed from the alloy, the ability to manipulate properties decreases. Usually the high hardness and high strength ferrous alloys contain modest to high carbon levels (0.6 to 0.8%).

Hardening the surface of a PM ferrous component improves wear resistance. It is best performed after all forming, sintering, and machining operations have been completed. Carbon is diffused into the surface in a process termed case hardening or surface carburization. Figure plots hardness vs. depth from the surface after one hour of vacuum carburization. The presence of open pores increases the depth of carbon penetration. Open pores contribute to vapor transport and surface diffusion which are faster than bulk diffusion. Therefore, porosity has a mixed effect on the heat treatment of PM steels; it increases carburization depth, but decreases hardenability.