Module 3C1: Materials processing and design
Module 3P1: Materials into products

Powder Processing

1. Introduction

Powder routes are used to make 3D components from metals, ceramics and some polymers. Mechanical properties can be excellent, and the process can be cost effective: it is a near-net shape process so material wastage is small, and energy requirements can be modest. PM (powder metallurgy) steel parts are very widely used in the automotive industry.

What is powder processing?

Material in fine powder form (particle size generally ranging from 50 to 500 µm, though sometimes as small as 1 µm for some specialist applications) is pressed into the required shape and then heated to bond the particles together by interdiffusion to form components, which generally require very little further processing. Occasionally (in the case of some ductile metals) the compacted powder is used as source material for further processing (e.g. extrusion, forging etc).

Powder processing normally involves *cold compaction* followed by a high temperature *sintering* stage, in which heat is applied with or without pressure.

Powder

| Lubricant | → | Additives |

Mix

Press in die

'Green' part

Sinter

Finished part
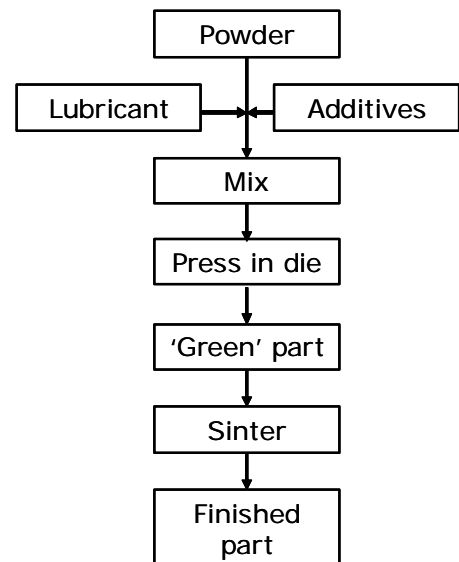
Why use a powder route?

1. High melting-point materials can be formed to final shape: ceramics (e.g. alumina, silicon nitride, zirconia ....); cermets (e.g. tungsten carbide/cobalt); metals (e.g. tool steels, carbon/alloy steels, bronzes, Al alloys, Ti alloys ...); polymers (e.g. PTFE, ultra-high MW polyethylene). Some of these cannot be processed in other ways.

2. It is a *near-net shape forming* process (i.e. involves minimal final machining) with reasonably close tolerances and good surface finish e.g. small ceramic parts, metal gears, bearing bushes, connecting rods etc.

3. Material wastage is low (important for expensive materials)

e.g. Powder route   Cast/forged parts

4. Achieves good dispersion of phases e.g. for reactive materials or of materials which cannot easily be mixed in the molten state. Fine particle distributions can be achieved. e.g. carbide particles in high-speed steel:
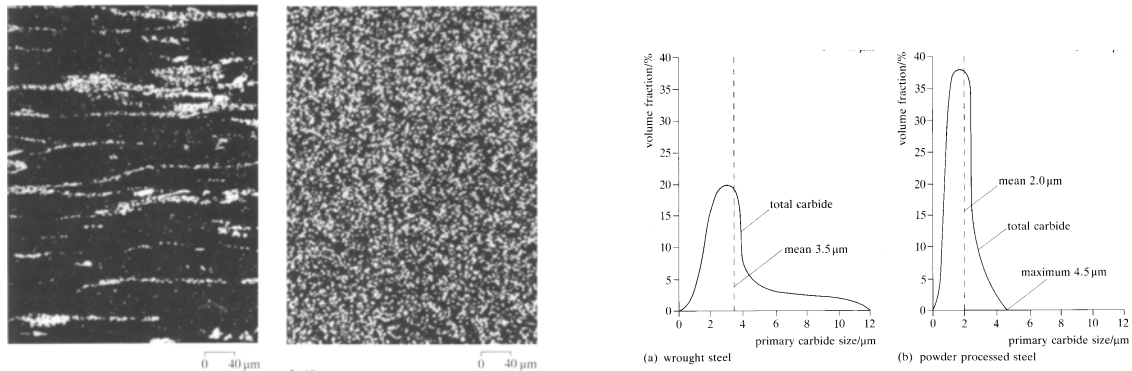
Conventional processing produces 'stringers' of carbide particles whereas powder processing allows higher volume fraction of carbides, which are evenly dispersed: provides better strength, toughness, wear resistance.

5. Avoids segregation effects which occur during casting (e.g. when densities of phases are very different) e.g. solid bearing alloys

6. Porosity can be controlled, either to achieve low porosity(essentially 'fully dense') or high porosity (up to 50%) for porous ('self-lubricating') bearings or filters.

7. Relatively cheap for large production runs (e.g. $>10^4$): materials costs are reduced by lack of wastage but plant and die costs can be high.

2. Powder manufacture

An important process for metals is *atomization*, in which high pressure jets of water (water atomization) or gas (gas atomization) are directed at a stream of molten metal, causing it to break up into droplets and solidify.

Water atomization (fast quenching in a high heat-capacity medium) leads to irregularly shaped particles; gas atomized particles tend to be more spherical. For very reactive materials, inert gas or fluid may be used.
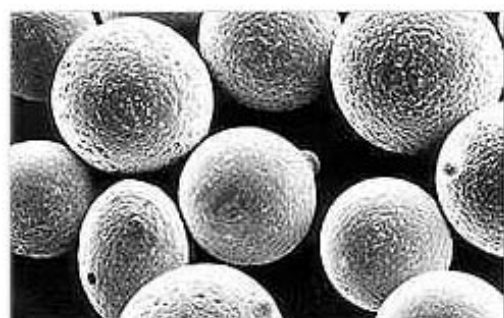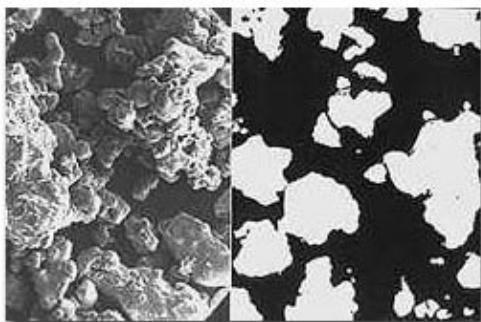
Ceramic powders are made by crushing or grinding (followed by sieving) – the particles are very irregular.

Particle shape is important because:
- irregular particles 'key' together well and so when pressed together tend to adhere, leading to stronger 'green' components than rounded particles. For metals this is because high pressure at particle contact points leads to heavy plastic deformation, interlocking and local welding.
- rounded particles flow more easily than irregular particles and can pack together more uniformly and densely than irregular particles. For highest packing density use mainly rounded particles with a range of particle sizes (small ones to fill up the gaps between big ones). With a single size, can achieve high, controlled porosity.

3. Processing route
Powder blending
The basic metal or ceramic powder is blended with various additives.
- Steel components are made from iron powder mixed with carbon (graphite – up to 1%) and often copper (1 – 4%). The copper strengthens the steel and prevents shrinkage during sintering. Other alloying elements can be used and alloy PM steels are easily made by mixing appropriate powders.
- Non-ferrous alloys (e.g. bronzes, titanium alloys, aluminium alloys) can also be blended as powders.
- Ceramic powders usually incorporate additives as sintering aids and to inhibit grain growth

The mix also includes a lubricant/binder (e.g. stearic acid, zinc stearate) which performs several functions:
- Powder flows more easily, so fills mould better
- Die friction is reduced, so more uniform product density is achieved, part is ejected without cracking and die life is increased.
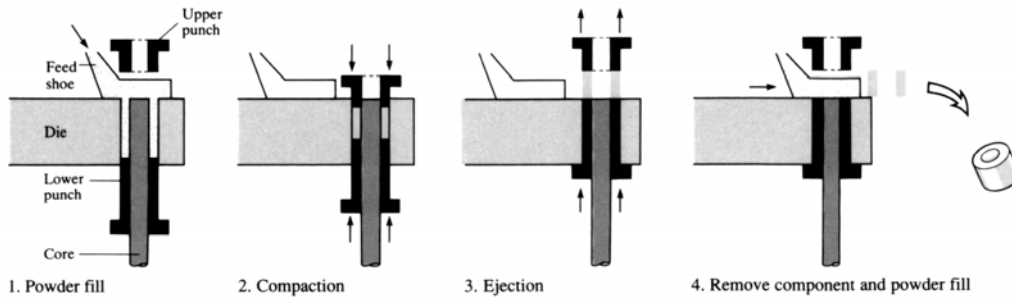
Compaction
The powder is pressed into a shaped mould/die at high pressure: e.g. 350-800 MPa for steels, 70-275 MPa for aluminium, 100-150 MPa for alumina. Presses need capacities (maximum force) up to hundreds of tonnes force (several MN) to achieve these pressures. The press capacity usually limits the size of the part that can be formed.

The component is now called a *'green compact'* with the correct shape, but little strength (typically 10-20 MPa). Since it is so weak it must be handled carefully, but it is very easy to machine if necessary (*'green machining'* to produce features which cannot be produced by pressing – e.g. transverse holes, slots).

For **ceramic** materials the linear dimensions of the green compact will be 15-20% greater than those of the finished part because of the remaining porosity (up to 50%). With **metals** the powder particles themselves deform during compaction and the green compact has a similar density to the final product (up to 95% of bulk metal); there is little or no shrinkage on sintering.

Uniaxial pressing

The mechanical properties of the final product (after sintering) depend critically on the *homogeneity* of the compact. If a compact contains a range of densities, each region will contract to a different extent on sintering. This means that not only will it have different mechanical properties in different regions but even more important the product will contain internal stresses. This causes problems in both metals and ceramics.

Influence of compacting pressure on density for iron and copper
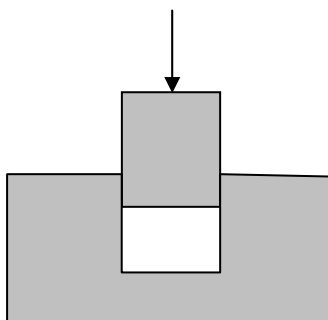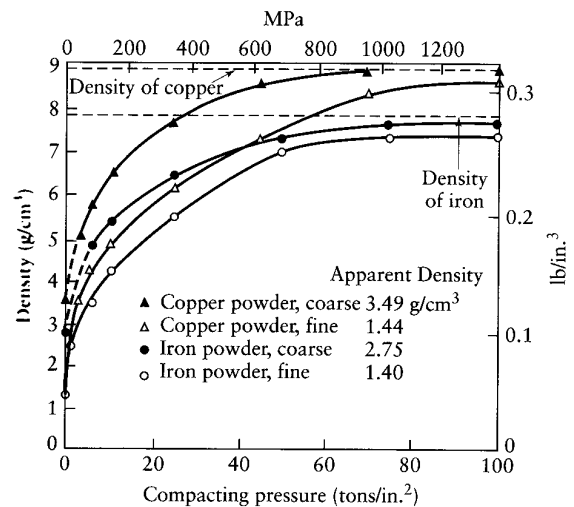
Design of the die/punch is very important. Friction between the powder and the die can lead to a non-uniform density distribution which results in strength variation.
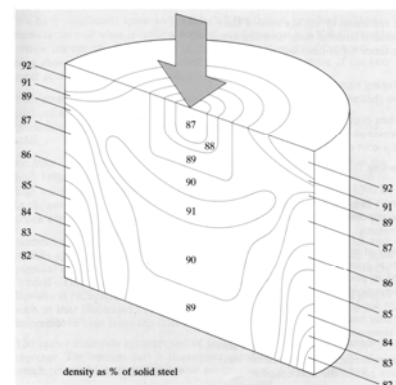
More uniform distribution is achieved by

- better lubrication between powder and die
- multiple punches, e.g. with punches moving at both top and bottom of die. Get highest compaction close to moving punch, so average compaction increased and compaction variation is reduced.

Compacts of non-uniform shape will have a complex and non-uniform density distribution unless multiple punches are used to give constant compression ratios.

The design of presses and dies to achieve optimum filling and uniform compaction can be very complex.
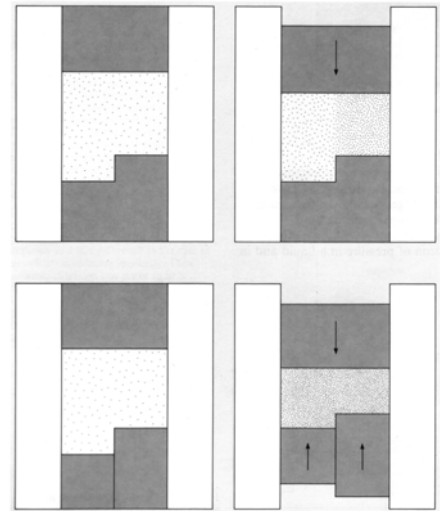
Cold isostatic pressing
Uniaxial (i.e. 1-D) pressing is restricted to simple shapes. For more complex shapes (especially for ceramics) *isostatic pressing* is needed to form a green compact with a uniform density distribution. The powder is contained in a rubber mould (n.b. no heating is used at this point), and pressure is applied by external fluid or gas.
More expensive than uniaxial pressing, and dimensional accuracy low (products are often machined before sintering). However, the need for lubricants/binders is reduced or removed.
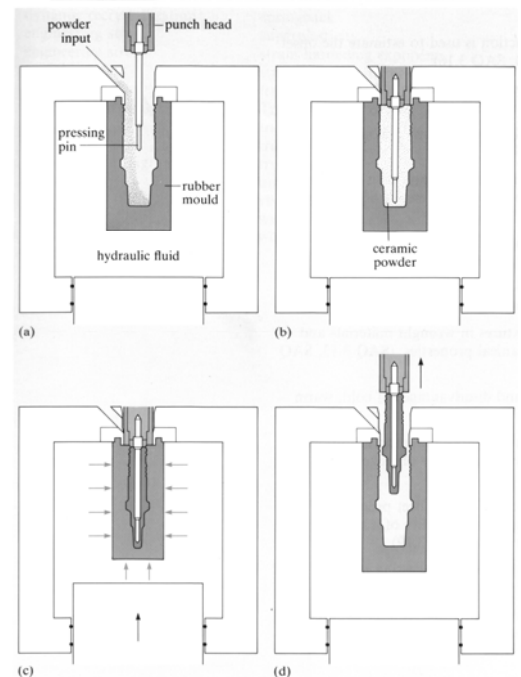
Sintering (Pressureless sintering)
Typically a continuous process: the time at highest temperature can vary from 10-45 minutes (for steels and copper alloys) to several hours (for high T_m metals and ceramics). The furnace atmosphere is closely controlled (e.g. to prevent oxidation or decarburization of steels).
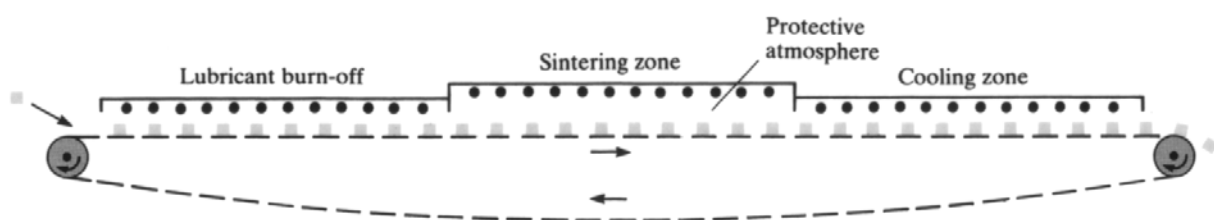
The green compact is heated to a temperature well above 0.5 T_m (typically 0.7 to 0.9 T_m) so that rapid diffusion can occur. Typical sintering temperatures: Iron/steel 1000-1200 °C; Al alloys 600 °C; copper, bronze 750-900 °C; tungsten carbide/cobalt 1500 °C; tungsten 2350 °C; alumina 1600 °C.

Driving force for sintering: diffusion along composition gradients and reduction in particle surface area. (Vapour and/or liquid phase transport can also occur.)

Mechanism: atoms diffuse to fill the pores. Diffusion occurs along different paths: bulk (through the lattice), dislocation core, surface (along surface of unsintered particles), grain boundary (along the boundaries between the particles, once fused). Total rate is the sum of all of these, though one mechanism tends to be dominant at a particular temperature and particle or pore size. (The mechanisms are essentially the same as creep)
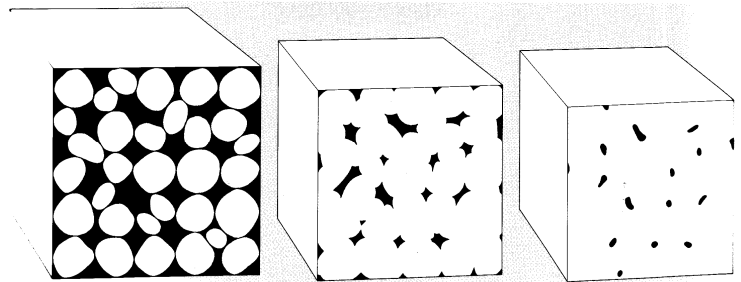
Sintering rate:

$$\frac{\partial \rho}{\partial t} \propto \frac{\gamma}{a^3} D_0 \exp\left(-\frac{Q}{RT}\right)$$

for density ρ, surface energy γ, particle diameter a

Stages in sintering:
1. plastically deformed contacts (in metals)
2. cohesion of particles by formation of bridges. Porosity interconnected.
3. particles grow competitively by diffusion across inter-particle interfaces, leading to grain growth
4. porosity becomes spherical and shrinks. Porosity becomes discrete.

- Rate inversely related to (particle size)^3 : (driving force from surface energy, diffusion distances). Fine compacts sinter faster, and as grains grow, the rate falls.
- Very difficult to get 100% densification by sintering alone.
- Pore size and spacing directly related to original particle size.
- Many advantages in keeping sintering times as short as possible

IM Hutchings, Nov. 2012

Porosity is usually expressed indirectly in terms of density: e.g. for Cu:

For metals, if the sintering process is followed by mechanical working (e.g. hot forging, extrusion etc.) then the porosity can be completely removed and maximum mechanical properties are obtained. The process of forging and sintering can be combined into a single process ('sinter forging').
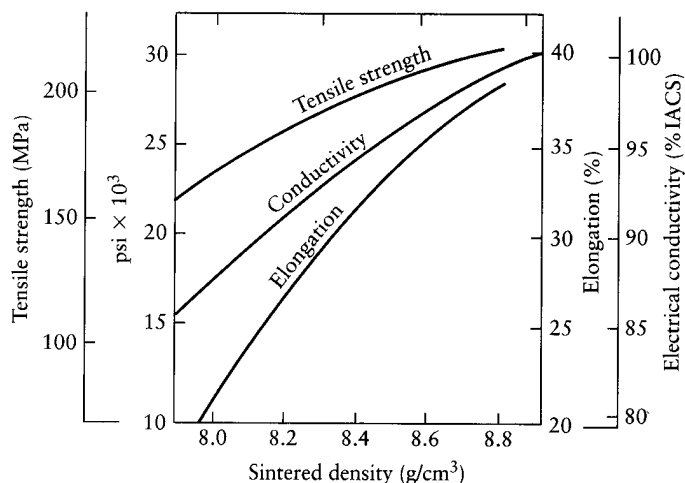

4. Liquid phase sintering
Sintering can be speeded up dramatically if a liquid phase is present which can be drawn (by capillary action) into the spaces between the particles. Example:

- sintering of alumina + 1% MgO which reacts to form a low melting-point glass which bonds the alumina grains together (e.g. for spark-plug insulator). A disadvantage is that the high-temperature strength of the material is reduced.

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5. Hot Isostatic Pressing (HIPing)
Similar to cold isostatic pressing, but powder is canned in a metal container to provide shape and simultaneously subjected to high temperature (e.g. 2000 °C for SiC) as well as high hydrostatic pressure (using gas, generally argon).
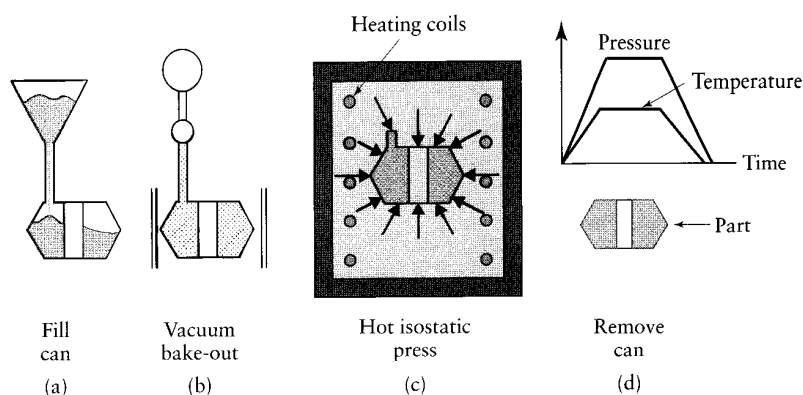

Very low final porosity. The short process time (minutes, rather than the hours needed for conventional sintering) reduces grain growth problems. However, the high gas pressure (typically 10 MPa = 100 bar) mean that the process is expensive (large pressure-vessels, operating at high T). HIPing provides components with good mechanical properties (low porosity), but dimensional accuracy is low. The improvement in ductility and tensile strength can be very significant.

Driving force: contact pressure leading to creep deformation

$$\frac{\partial \rho}{\partial t} \propto \frac{P}{a^2} D_\text{o} \exp\left(-\frac{Q}{RT}\right)$$

6. Metal Injection Moulding (MIM) and Powder Injection Moulding (PIM)

Used for producing small, high-precision, low porosity components from metal or ceramic powder, in large quantities. These processes overcome the restrictions in shape/complexity of uniaxial pressing.
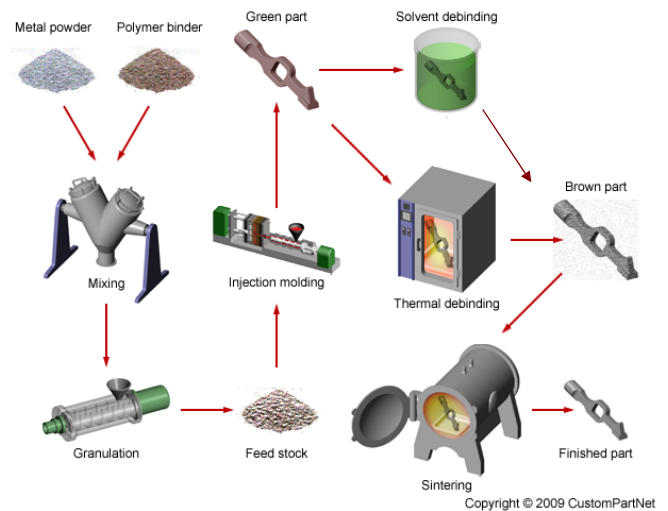
Uses conventional polymer injection moulding technology with a polymer-metal or polymer-ceramic feedstock to produce the initial green compacts. These are therefore highly-filled polymer-metal or polymer-ceramic composites. Typical MIM process uses very fine metal powder (1 – 20 µm) with specially designed polymer binders. The volume fraction of binder is 30-50%.

Binder removal (*debinding*) is a critical stage involving:
- heating the green compact – taking several hours, or
- chemical decomposition by e.g. gaseous nitric acid – quicker, or
- dissolving the binder with a solvent

The debinding process converts the 'green' part (as-moulded, containing the polymer binder) to a 'brown' part (with no binder, but still not sintered, so containing high porosity) which is then sintered.

During sintering, the part typically shrinks 50% by volume (15-20% linearly). Care is therefore needed to retain shapes while dimensions change dramatically.

Advantages of PIM, MIM:
- low die wear rates; complex shapes (variations in wall thickness, moulded-in decoration) with high dimensional tolerances can be made; low and uniformly distributed porosity means products take high surface polish and have excellent mechanical properties. Particularly useful for high-volume production.

MIM and PIM are typically used for small thin-section complex parts produced in high volumes e.g. small gears, disc drive parts, camera parts, surgical instruments, spectacle frames and luxury goods. For some metals it competes with investment casting (lost-wax casting). Maximum section thickness is ~5 mm – limited by the binder removal process.

IM Hutchings, Nov. 2012