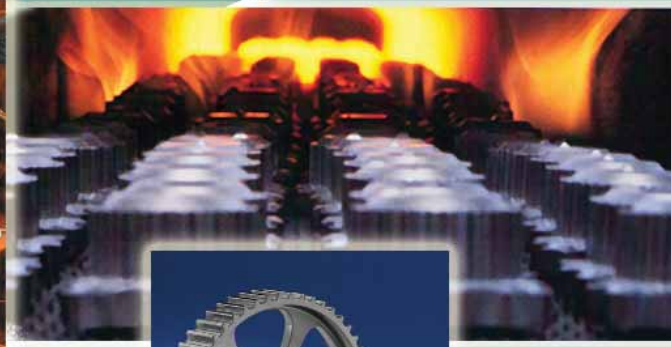
European Powder Metallurgy Association

Introduction to POWDER METALLURGY

THE PROCESS AND ITS PRODUCTS



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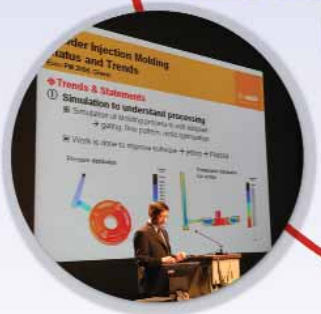

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We were established as a non-profit organisation in 1989, and act as the voice of the European PM Industry at European and International levels. We collaborate with a range of organisations to address issues of strategic importance to the global competitiveness of PM technology. These issues include Environment, Health and Safety, Quality Assurance, Research, Education and Training, Statistics, International Standards, and PM technology promotion.

We have developed a range of educational training materials such as a range of free booklets, and information about these and other publications about the Powder Metallurgy process, PM Materials, and PM Applications is available on our EPMA website at www.epma.com/onlinepublications.

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Powder Metallurgy -
The Process and Its Products
This publication was originally written by Gordon Dowson in 1992 and has been substantially revised by David Whittaker in 2008.

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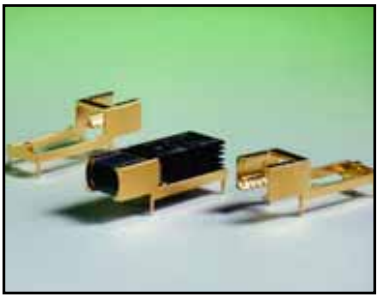
POWDER METALLURGY

THE PROCESS AND ITS PRODUCTS



Powder Metallurgy has been used since the 1920s to produce a wide range of structural PM components, self-lubricating bearings and cutting tools. This publication reviews the PM process and describes recent developments in production technology and PM applications.

Powder Metallurgy (PM) in the context of this publication encompasses the production of metals in powder form and the manufacture from such powders of useful objects by the process known as sintering. In many cases individual engineering components are produced directly by the process such as components being referred to indiscriminately as sintered components, sintered parts, or PM parts. However, wrought products also can be produced from powder and recently a number of scientifically exciting developments of great industrial potential have taken place.

The PM process involves compressing the powder, normally in a container, to produce a compact having sufficient cohesion to enable it to be handled safely, and then heating the compact, usually in a protective atmosphere, to a temperature below the melting point of the main constituent during which process the individual particles weld together and confer sufficient strength on the material for the intended use. It is this heating step that is referred to as sintering.

THE PROCESS

The basic procedure in the manufacture of PM parts is:

(1) Mix the metal powder or powders with a suitable lubricant.

(2) Load the mixture into a die or mould and apply pressure. This gives what is called a compact, which requires only to have sufficient cohesion to enable it to be handled safely and transferred to the next stage. Such compacts are referred to as green, meaning unsintered. Hence the terms green density and green strength.

(3) Heat the compact, usually in a protective atmosphere, at a temperature below the melting point of the main constituent so that the powder particles weld together and confer sufficient strength to the object for the intended use. This process is called sintering, hence the term sintered parts. In certain cases, a minor constituent becomes molten at the sintering temperature in which case the process is referred to as liquid phase sintering. The amount of liquid phase must be limited so that the part retains its shape.

In certain special cases stages 2 and 3 are combined i.e. compaction is done at an elevated temperature such that sintering occurs during the process. This is termed hot pressing, or pressure sintering.

One specialised variant on this process - the production of Soft Magnetic Composite (SMC) components - involves a heating step aimed at curing a resin binder, added to insulate the individual iron powder particles, rather than at sintering of the metal particles.

In many cases the sintered part is subjected to additional processing - repressing, plating etc. and these will be dealt with in later sections.

In certain special cases, e.g. in the manufacture of filter elements from spherical bronze powder, no pressure is used, the powder being placed in a suitably shaped mould in which it is sintered. This process is known as loose powder sintering

WHY MAKE PARTS FROM POWDER?

There are two principal reasons for using a powder metallurgy product:
(1) cost savings compared with alternative processes, or
(2) unique attributes attainable only by the PM route.

The first of these reasons is the driving force for:

Structural (or Mechanical) Parts:
In tonnage terms, this is by far the largest group.

The bulk consists of iron-based parts, but significant tonnages of copper, brass, bronze and aluminium parts are produced, as well as some rarer metals such as beryllium and titanium. In general, such parts often have advantage over forgings in dimensional accuracy, but, in a large number of cases, the main justification for their use is the economic one. Over the past decade or so, however, developments have taken place that now require revision of the foregoing. It is now possible to produce sintered parts with properties equal to or even superior to those of parts made by more traditional routes. Some of these developments have been in the materials utilised, while others have been modifications to the standard production process at the compaction stage (e.g. Warm Compaction, High Pressure Cold Compaction, High Velocity Compaction), during Sintering (e.g. Activated Sintering, Ferrite Phase Sintering) or as post-sintering densification processes (e.g. Powder Forging, Surface Cold Rolling). These developments are considered in more detail in later sections of this publication.

There are a number of examples of the second reason, the attainment of unique properties or characteristics:

Porous Materials: Most forms of metal are porous to some extent, sintered metals more than most, but here we are concerned with the production of parts having a significant, carefully controlled porosity designed to serve a useful purpose. The chief products in the group are filters and oil-retaining bearings often referred to as self-lubricating bearings.

The latter is one of the major PM products and is dealt with in a later section.
The following products cannot readily or satisfactorily be produced by alternative processes.

Refractory metals have very high melting points, such as tungsten, molybdenum, niobium, tantalum and rhenium. They are very difficult to produce by melting and casting and are also frequently very brittle in the cast state. A sintered powder compact can be mechanically deformed at a suitably elevated temperature. It then gradually develops a microstructure with preferred orientation that gives the now dense material useful ductility even at ambient temperatures.

Composite Materials: These compromise of two or more metals which are insoluble even in the liquid state, or mixtures of metals with non-metallic substances such as oxides and other refractory materials. Examples are:

(a) Electrical Contact Material (copper/tungsten, silver/cadmium oxide)

(b) Hard Metals (cemented carbides) ,used for cutting tools and wear parts (e.g. wire-drawing dies and tools for hot forging of metals). Tungsten carbide bonded with cobalt was the first of this class of material and still has the lion's share of the market, but other carbides and, more recently, nitrides, carbonitrides and borides are being used in increasing quantities and substitutes for the relatively scarce and expensive cobalt have been developed. These include Ni, Ni-Co, Ni-Cr, nickel-based superalloys and complex steels.

(c) Friction Materials for brake linings and clutch facings in which abrasive and other non-metallic materials are embedded in a copper or other metallic matrix.

(d) Diamond cutting tools, especially grinding wheels, in which small diamonds are uniformly dispersed in a metal matrix.

(e) In recent years, several wrought products containing finely dispersed non-metallic phases have been developed and put into service. These dispersion- strengthened materials (referred to as ODS materials if the strengthening particles are oxides) have strength, especially at elevated temperatures superior to that of cast and wrought metals of similar basic composition.

(f) Soft magnetic composite (SMC) materials,consisting of iron powder particles insulated from each other by a cured resin binder. It is difficult if not impossible to make these composite products except by PM.

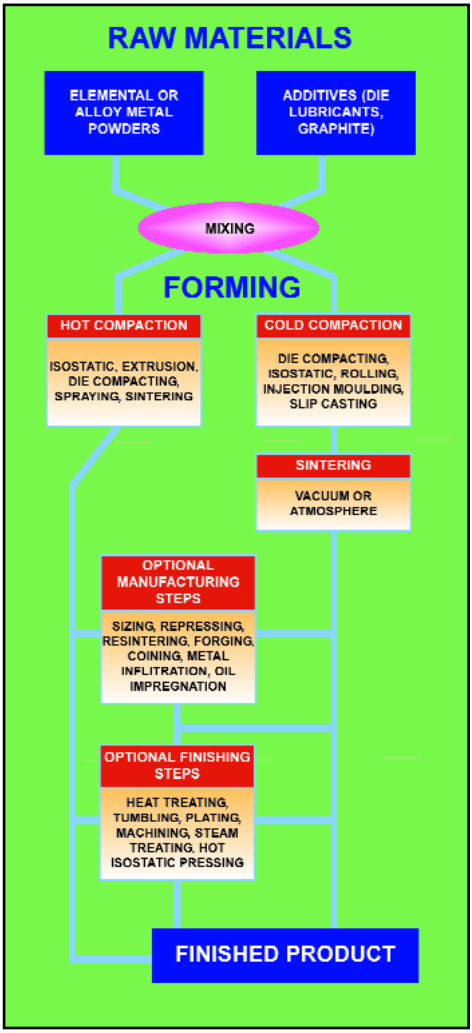


Fig 1: Powder metallurgy processes chart

Magnetic Components: Economic and technical advantages have also been found in the production of components for magnetic applications.

Special high-duty alloys: High speed steels and superalloys based on nickel and/or cobalt can be processed by PM to give a product having superior properties to those achieved by casting and forging. In general, powder is compacted into a blank or billet which is then subjected to forging or extrusion followed by forming in traditional ways. The advantages of the PM route are a higher yield of usable material and a finer, more uniform microstructure that confers improved mechanical properties and, in the case of cutting tools and wear parts, longer life.

The PM process has also allowed the development of new types of materials based on powders having micro-crystalline or even amorphous (glass-like) structures produced by cooling droplets of molten metal at very high rates. The final consolidated product is characterised by very high strength, ductility and thermal stability.

POWDER MANUFACTURE

There are many ways in which metals may be produced in powder form,

- *comminution of solid metal*
- *precipitation from solution of a salt*
- *thermal decomposition of a chemical compound*
- *reduction of a compound, usually the oxide, in the solid state*
- *electrodeposition*
- *and the atomisation of molten metal*.

Of these, the last three account for the bulk of the powders used.

Solid State Reduction: This was for a long time the most widely used method for the production of iron powder. Selected ore is crushed, mixed with carbon, and passed through a continuous furnace where reaction takes place leaving a cake of sponge iron which is then further treated by crushing, separation of non-metallic material, and sieving to produce powder. Since no refining operation is involved, the purity of the powder is dependent on that of the raw materials. The irregular sponge-like particles are soft, and readily compressible, and give compacts of good green strength.

Refractory metals are normally made by hydrogen reduction of oxides, and the same process can be used for copper.

There are several plants producing powder by the reduction of iron oxide (mill scale) by means of hydrogen or carbonaceous material such as coke.

As a variant on a similar theme, high compressibility iron powders are also produced by the decarburisation of granulated and pulverised high carbon iron.

Fig 2: *Metal powders with example of components*

Electrolysis: By choosing suitable conditions - composition and strength of the electrolyte, temperature, current density, etc. - many metals can be deposited in a spongy or powdery state.

Extensive further processing - washing, drying, reducing, annealing and crushing - may be required.

Copper is the main metal to be produced in this way but chromium and manganese powders are also produced, by electrolysis. In these cases, however, a dense and normally brittle deposit is formed and requires to be crushed to powder. Pressures from environmental legislation have now restricted production of electrolytic copper to regions where these constraints are less severe although the powder type continues to be traded more widely by the major copper powder suppliers. Electrolytic iron was at one time produced on a substantial scale, but it has now been largely superseded by powders made by less costly processes. Very high purity and high density are two distinguishing features.

Atomisation: In this process molten metal is broken up into small droplets and rapidly frozen before the drops come into contact with each other or with a solid surface.

The principal method is to disintegrate a thin stream of molten metal by subjecting it to the impact of high energy jets of gas or liquid. Air, nitrogen and argon are commonly used gases, and water is the liquid most widely used.

By varying several parameters - design and configuration of the jets, pressure and volume of the atomising fluid, thickness of the stream of metal etc. - it is possible to control the particle size distribution over a wide range. The particle shape is determined largely by the rate of solidification and varies from spherical, if a low heat capacity gas is employed, to highly irregular if water is used. In principle, the technique is applicable to all metals that can be melted and is used commercially for the production of iron, copper, tool steels, alloy steels, brass, bronze and the low melting point metals, such as aluminium, tin, lead, zinc and cadmium.

The readily oxidisable metals (e.g. chromium-bearing alloys) are being atomised on an increasing scale by means of inert gas, especially argon.

In addition, there are several other processes that are finding increasing application, an important one being centrifugal atomisation in which droplets of molten metal are discharged from a rotating source. There are basically two types of centrifugal atomisation processes:

(1) A cup of molten metal is rotated on a vertical axis at a speed sufficient to throw off droplets of molten metal, or a stream of metal is allowed to fall on a rotating disc or cone; or

(2) A bar of the metal is rotated at high speed and the free end is progressively melted e.g. by an electron beam or plasma arc. This latter process is called the Rotating Electrode Process (REP), and the bar may be rotated either on a horizontal or on a vertical axis.

A special advantage of these processes is that they can be carried out in a sealed vessel in a controlled atmosphere - even vacuum - and thus produce 'clean' powders of highly reactive metals.

With the REP process, the avoidance of contact with refractory is a potent means of reducing the number of non-metallic inclusions in the powder, and in components manufactured from the powder.

Close-coupled (or confined) gas atomisation has been extensively developed in recent years to produce fine powders for a range of applications, including Metal Injection Moulding (see later section). In close-coupled atomisation, the design of pouring nozzle and atomisation head are adjusted so that impingement of the molten metal stream with atomising gas jets occurs immediately below the exit of the nozzle with little or no free fall height.

Atomisation is particularly useful for the production of alloys in powder form, since the constituent metals are fully alloyed in the molten state. Thus each powder particle has the same chemical composition.

Fig 3: *Water Atomisation, courtesy of Hoeganaes Corporation*

Additionally the process is used to produce compositions such as copper-lead, in which the lead, though soluble in the liquid state, comes out of solution on solidification. If a casting of such an alloy is made, serious segregation of the lead results, but if the liquid is atomised, the end product is copper powder containing a very fine and uniform distribution of lead inclusions within each powder particle.

Mechanical Comminution: Brittle materials such as inter-metallic compounds, ferro-alloys - ferro-chromium, ferro-silicon, etc. are pulverised mechanically in ball mills, and a process known as the Coldstream Process produces very fine powders. In this process, granular material, which may be coarsely atomised powder, is fed in a stream of gas under pressure through a venturi and is cooled and thereby embrittled by the adiabatic expansion of the gas before impinging on a target on which the granules shatters.

Chemical Processes: Thermal decomposition of a chemical compound is used in some cases, a notable one being nickel carbonyl.

This Carbonyl Process was originally developed as a means of refining nickel, crude metal being caused selectively to react with carbon monoxide under pressure to form the carbonyl which is gaseous at the reaction temperature and which decomposes on raising the temperature and lowering the pressure.

The same process is used for iron, and carbonyl iron powder finds small scale application where its very high purity is useful.

Recently, demand for very fine powders for the injection moulding process has given a considerable impetus to the carbonyl process. Typically the particle size of carbonyl iron powder is 1 - 5 μm, but, as in the case of nickel, it can be tailored to suit particular requirements.

Another case of thermal decomposition is platinum powder, which is made from sponge produced by heating a salt - platinum ammonium chloride. In the Sherritt-Gordon process, nickel powder is made by hydrogen reduction of a solution of a nickel salt under pressure.

Chemical precipitation of metal from a solution of a soluble salt is used in other cases - e.g. silver, powder of which is produced by adding a reducing agent to a solution of silver nitrate. This is, of course, the same basic process as is used

Fig 4: *Sponge Iron Powder NC 100.24, courtesy Höganäs AB*

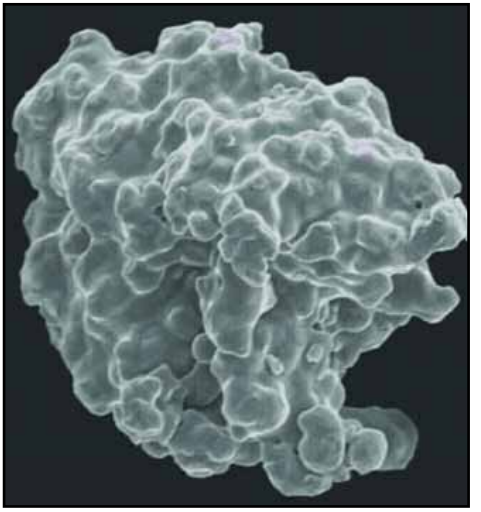


Fig 5: *Atomised Iron Powder ASC 100.29, courtesy Höganäs AB*

to produce black and white photographs. Cobalt powders are also produced by reduction of cobalt carbonate powder, produced by chemical precipitation with CO2, starting from cobalt amino-sulphate solution.

Powder Characteristics: The further processing and the final results achieved in the sintered part are influenced by the characteristics of the powder:

- *particle size,*
- *and size distribution*
- *particle shape,*
- *structure*
- *and surface condition*.

A very important parameter is the apparent density (AD) of the powder, i.e. the mass of a given volume, since this strongly influences the strength of the compact obtained on pressing. The AD is a function of particle shape and the degree of porosity of the particles.

The choice of powder characteristics is normally based on compromise, since many of the factors are in direct opposition to each other:

An increase in the irregularity and porous texture of the powder grain, i.e. decrease in apparent density, increases the reduction in volume that occurs on pressing and thus the degree of cold-welding, which, in turn, gives greater green strength to the compact.

This increase in contacting surfaces also leads to more efficient sintering.

Additionally the greater reduction in volume necessary to give the required green density may require greater pressure and consequently larger presses and stronger dies.

The ease and efficiency of packing the powder in the die depends to a large extent on a wide particle size distribution, so that the voids created between large particles can be progressively filled with those of smaller size.

Fine particle sizes tend to leave smaller pores, which are easily closed during sintering. An excess of fines, however, reduces flow properties.

Fine particle size is, however, an important requirement of the Metal Injection Moulding process (see later section). The emergence of this process since the 1980s has given a considerable impetus initially to the use of carbonyl iron and nickel powders and, subsequently and more significantly, to fine gas atomised powders produced by close-coupled atomisation.

The purity of the powder is critically important. Impurity levels that can be tolerated depend to a large extent on the nature and state of combination of the substances concerned.

For example, the presence of combined carbon in iron tends to harden the matrix so that increased pressures are required during compaction. Free carbon, however, is often an advantage, acting as a lubricant during the pressing operation.

Most metal powder grains are coated by a thin oxide film, but in general these do not interfere with the process, since they are ruptured during the pressing operation to provide clean and active metal surfaces which are easily cold-welded. Their final reduction under the controlled sintering atmosphere is essential for complete metal bonding and maximum strength.

Stable oxide films or included oxide particles, such as SiO2 and Al2O3, are more serious, since these are generally abrasive and lead to increased tool wear. Furthermore they cannot be reduced during subsequent sintering and their presence may adversely affect the mechanical properties especially impact strength of the finished part. This is of major importance when high integrity, high density parts are required - notably powder forgings.

PRODUCTION OF SINTERED PARTS

The general sequence of operations involved in the powder metallurgy process is shown schematically in Fig. 1. The component powders are mixed together with a lubricant until a homogeneous mix is obtained. The mix is then loaded into a die and compacted under pressure, after which the compact is sintered. An exception is the process for making filter elements from spherical bronze powder where no pressure is used; the powder being simply placed in a suitably shaped mould in which it is sintered. This process is known as loose powder sintering.

Mixing: The object of mixing is to provide a homogeneous mixture and to incorporate the lubricant. Popular lubricants are stearic acid, stearin, metallic stearates, especially zinc stearate, and increasingly, other organic compounds of a waxy nature. The main function of the lubricant is to reduce the friction between the powder mass and the surfaces of the tools - die walls, core rods, etc. - along which the powder must slide during compaction, thus assisting the achievement of the desired

uniformity of density from top to bottom of the compact. Of equal importance is the fact that the reduction of friction also makes it easier to eject the compact and so minimises the tendency to form cracks.

It has been suggested that an additional function of the lubricant is to help the particles to slide over each other, but it seems doubtful whether this factor is of much significance: - good compacts can be obtained without any admixed lubricant, e.g. using die wall lubrication or isostatic pressing. Care in the selection of lubricant is necessary, since it may adversely affect both green and sintered strengths especially if any residue is left after the organic part has decomposed.

Over-mixing should be avoided, since this increases the apparent density of the mix. Additionally, over-mixing usually further reduces the green strength of the subsequent compacts probably by completely coating the whole surface of the particles, thereby reducing the area of metal to metal contact on which the green strength depends.

The flow properties also are impaired and good flow is essential for the next step i.e. loading the powder into the die. In the special case of cemented carbides, the mixing process is carried out in a ball mill, one of the objects being to coat the individual particles with the binder metal e.g. cobalt, but as the very fine powders involved do not flow, the mixture is subsequently granulated to form agglomerates.

Pressing (or Compacting): The mixed powders are pressed to shape in a rigid steel or carbide die under pressures of 150-900 MPa. At this stage, the compacts maintain their shape by virtue of cold-welding of the powder grains within the mass. The compacts must be sufficiently strong to withstand ejection from the die and subsequent handling before sintering. This is a critical operation in the process, since the final shape and mechanical properties are essentially determined by the level and uniformity of the as-pressed density. Powders under pressure do not behave as liquids, the pressure is not uniformly transmitted and very little lateral flow takes place within the die. The attainment of satisfactory densities therefore depends to a large degree on press tool design.

The maximum permissible compaction pressure is controlled by the need to avoid tool failures. For complex, multi-level part geometries, thin and fragile punches in the tool assembly would necessitate pressures being limited to around 600 MPa maximum. However, for "chunkier" part geometries not requiring thin punches, it has been found possible to utilise pressures of 1,000 MPa or even more. This High Pressure Cold Compaction has allowed higher density (and therefore higher performance) parts to be introduced in applications such as starter gears, synchroniser rings and cam lobes.

Tool Design: Attention must be given to the following design factors in the light of limited lateral flow and also the necessity of ejecting the green part in the direction of pressing.

(1) Length-to-Width-Ratio. The applied pressure and therefore the density decreases over the length of the compact. Double-ended compaction assists in equalising pressure distribution, but still leaves a lower density region at the middle section of the part. Ratios of length to width in excess of 3:1 are not recommended.

(2) Re-entrant Grooves. Reverse Tapers and Lateral Holes cannot be moulded into the compact because of the impossibility of ejection and must therefore be subsequently machined, although elaborate, sometimes flexible die assemblies have been designed and patented to overcome this limitation.

(3) Bevels require feather-edged tools, which are fragile and easily fractured; so, if design permits, the bevelled edge of the component should end in a small flat.

(4) Abrupt changes in sections should be avoided since they introduce stress raisers, which may lead to crack formation as a result of the stresses induced by the elastic expansion-springback - that takes place as the compact is ejected from the die.

(5) To a first approximation the size of the part that can be made is a direct function of the capacity of the press available, but the complexity of the part and number of punch motions required also influence the equation. These same factors are relevant also to the production rates: the simpler the part the easier it is to press at high speed. With such parts, rates as high as 1 part per second have been achieved using mechanical presses. Hydraulic presses enable greater pressures to be used - up to 5,000 tonnes - but speeds are necessarily much lower, 10 parts per minute being a fairly representative high speed for parts of comparatively simple geometry.

Fig 6: Compacting Press, courtesy of Fette GmbH

It has already been stated that, because of the frictional forces set up between the powder grains and die wall, there is progressive reduction of pressure transmitted and thus of density achieved through the length of the component. It will be obvious that this factor increases in importance as the size of the compact increases, and is one of the factors limiting the size of sintered part that can be produced economically.

These effects are minimised by improving lubrication and by double-ended compaction, and many presses incorporate multiple punch motions operating from above and below the die. The same general results can be obtained with the single-acting press using a floating die assembly. It is possible to continue the downward movement of the die after compaction, thereby withdrawing the die from the component rather than ejecting by an upward movement of the bottom punch.

The dependence of green density upon compacting pressure follows a hyperbolic relationship and there is therefore a practical optimum pressure above which further densification is negligible and tool wear becomes severe.

Another feature that limits the density that can be achieved is the presence of the lubricant. Although this is normally present in no more than 1% by weight, the volume can be 5% or more, so that even if there are no voids in the compact, the density could not exceed 95%. The use of die wall lubrication using unlubricated powder mixes overcomes this problem, but the practical difficulties of applying the lubricant which must be renewed before each die fill have, so far, limited the use of the process to a few special cases where high density is a must.

Tools: The basic parts of a tool set are the die, in which the powder is contained, and punches, which are used to apply the compacting pressure. If, as is frequently the case, the part has holes running through it, these are formed by core rods located in the die before the powder is introduced. Multiple punches acting independently are used if the component being pressed has different levels. The die and core rod(s) form the contour of the compact parallel to the direction of pressing, and must, of course, be free from projections and re-entrants at right angles to the pressing direction; otherwise it would be impossible to eject the compact from the die.

Materials used are hardened tool steels or hardmetals (cemented carbides). The use of the more expensive carbide is increasing because of the greater life it affords, and the increasing cost of tool changes both in lost production and toolsetters' wages. PM high-speed steels are finding increasing application in this field. For short runs, ordinary steel dies may, of course, be more economical.

The importance of precise dimensions and high quality of the surface finish scarcely needs emphasis bearing in mind that one of the major features justifying the use of sintered parts is the ability to produce such parts accurately as regards size with a surface finish that obviates the necessity for subsequent machining operations.

Die life is another important aspect, and here it is impossible to give more than an indication. The life depends not only on what material is being pressed, and to what density, what lubrication is provided and the degree of die wear that can be tolerated, but also on the skill of the toolsetter, and the complexity of the tools. With steel dies, up to about 200,000 compacts can be achieved, with carbide dies 1,000,000 parts or more are possible.

Warm Compaction: A variant on the compaction process that involves the pre-heating of both the powder feedstock and the compaction tooling to around 130°C. This allows green density to be increased by 0.1 to 0.3 g/cm3, compared with conventional cold compaction, resulting in

improvements in mechanical properties by 10-20%.

Several systems for powder and tool heating have been developed and are offered by a number of press suppliers, often to be retro-fitted to existing presses.

Several hundred component applications are now in production. Many of these are various types of helical gear for hand power tools, but automotive applications include sprockets, synchroniser rings and various hubs.

Hot Pressing (pressure sintering): While the bulk of sintered parts is made by pressing the powder mix at ambient temperature followed by sintering, hot-pressing is used in certain cases. At elevated temperatures metals are softer and therefore it is usually possible to press to a much higher density without increasing the pressure required. It may be possible to dispense with a separate sintering operation, but this is not general because hot pressing is only justified by the significantly better properties obtained and a subsequent sintering step almost invariably improves the properties. The use of the process is limited by the much greater cost: special heat-resistant dies are required, a controlled atmosphere may be needed and production rates are, relatively, very slow; but it is used for the production of hardmetal and diamond cutting tools, both of which are, in any case, expensive materials.

OTHER POWDER SHAPING METHODS

Metal Injection Moulding: The production of green bodies by injection moulding of a plasticised powder feedstock was first developed for ceramics in the 1970s and was then first adapted to metal powders (as Metal Injection Moulding) at the end of that decade.

In principle, the process *(see Fig. 7)* is quite straightforward:- metal powder is intimately mixed with a thermoplastic binder and worked (using a kneader or shear roll extruder) to produce a plasticised feedstock (usually in a granulated form), which is then injected at a slightly elevated temperature into a mould in a manner identical with that used for the production of plastic articles. Initially, in fact, the same injection moulding machines, as were in use in the plastics industry, were used. The green body is released from the mould, the binder removed, and the part sintered. Densities of more than 95% theoretical are generally achieved, giving properties close to those of the wrought material, and with remarkable dimensional accuracy.

It can be inferred from the above that the process is significantly more expensive than the traditional PM process. The rationale behind its use is that parts of very complex geometry that are out of the question for normal PM can be produced. By reason of their complexity, such parts would be very expensive to produce by machining.

The process has a number of special requirements. One of the secrets of success - and, in spite of the large amount of literature on the subject, there are still secrets - is the

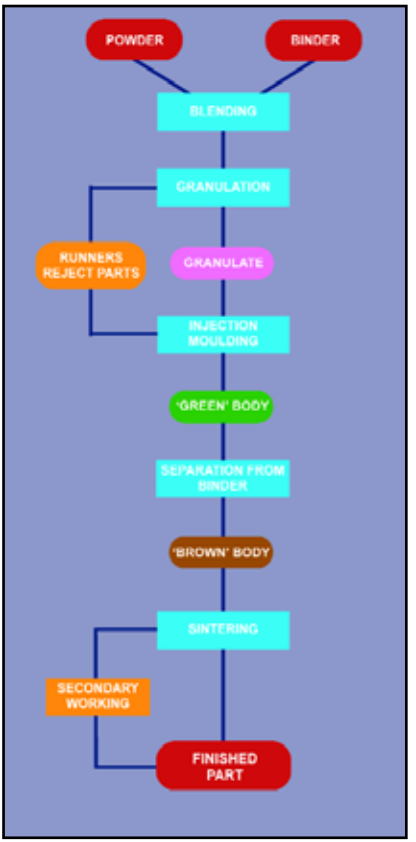


Fig 7: MIM processes chart

formulation of the binder. Among its many necessary attributes, it must be capable of wetting the powder particles and forming a uniform mass, and it must be removable without causing the part to deform or disintegrate. Since the proprietary process was launched, a great deal of research has been conducted and many different formulations are now available.

As regards the metal powder there are special requirements there also. In order to get a readily injectable feedstock and a uniform powder loading, the powder has to be, by PM standards, very fine. The carbonyl iron and nickel powders used in low alloy steel blends are spherical and have particle sizes between 2 and 10 μm. High alloy powders such as stainless steels (a dominant material type in current MIM production) can be either gas or water atomised and have particle sizes less than 40 μm. Gas atomised powders are generally produced by the close-coupled atomisation process, referred to in an earlier section. The amount of binder used is typically of the order of 50 vol.%. The use of fine powder gives an important additional bonus - such powders sinter more readily by reason of their large specific surface area.

The removal of the binder - usually referred to as debinding - is another key operation. The initial method was to heat the parts carefully. and cause the binder to decompose and/or evaporate (thermal debinding). This was a slow process which, depending on the thickness of the thickest section, could take between a few hours and a few days. Other debinding procedures have subsequently been developed which have been able to reduce debinding times to some degree. One is catalytic debinding, a patented process in which a polyacetal binder is removed in a gaseous acid environment (concentrated nitric or oxalic acid) at a temperature of around 120°C, the reaction products being burnt in a natural gas flame. Solvent debinding techniques have also been developed, in which the binder includes a constituent that can be dissolved in a liquid at room temperature so that a network of interconnected porosity is formed (sometimes as an aid to faster thermal removal of other binder constituents). Water soluble binder constituents are preferred to organic solvents for environmental reasons.

The relatively long time required for debinding and its thickness dependence is high on the list of reasons why the process is currently confined to relatively very small parts.

Fig 8: MIM Components

The sintering process for injection moulded parts does not differ significantly from that normally used for die-pressed compacts, but as the original green body had a large volume fraction of binder, up to 50%, the shrinkage on sintering is very large. It is remarkable that very good dimensional tolerances are achieved. A prerequisite is that the original mix be completely uniform so that the shrinkage is identical throughout.

The ability to competitively mass-produce complex, strong shapes in a wide variety of materials makes the process an excellent alternative to pressure die casting and machining.

Metal injection moulding business, after an initial period where it was gaining acceptance with design engineers, has since been following a growth rate of around 10% p.a. for the past decade or more. Continued growth at such rates is anticipated for the foreseeable future.

Isostatic Compaction: A technique which is coming increasingly into use is isostatic pressing.

In Cold Isostatic Pressing (CIP) the powder is contained in a flexible mould commonly of polyurethane. In Wet Bag CIP, the filled mould is then immersed in a liquid, usually water, which is pumped to a high pressure. Thus the powder is compacted with the same pressure in all directions, and, since no lubricant is needed, high and uniform density can be achieved. In Dry Bag CIP, the flexible mould is permanently fixed to the pressure vessel container and is separated from the pressurising liquid by a pressure transmission membrane (hence the term "dry bag").

The process removes many of the constraints that limit the geometry of parts compacted unidirectionally in rigid dies. Long thin-walled cylinders and parts with undercuts present no problem. The process is being increasingly automated with consequent improvements in productivity, and production rates are in some cases comparable with die pressing.

Cold isostatic pressing is commonly used in the forming of a broad range of products; including ceramic spark plug bodies (a leading application of Dry Bag CIP), refractory crucibles, hardmetal parts (e.g. rollers and dies), tungsten heavy alloys, high speed steel semi-products, molybdenum and tungsten arc furnace melting electrodes and long cylindrical filter elements. However, the process has not succeeded in establishing a significant niche in the forming of low alloy ferrous PM materials.

Hot Isostatic Pressing (HIP) also finds extensive use for the compaction of powders. In this case it is not possible to use a liquid pressure medium and argon is normally used. Furthermore, the material used for the container cannot be an organic elastomer, and in general a metal container, referred to as a can, is used. Since, at the temperatures involved, sintering takes place, the question of green strength does not arise, spherical powders, which have the highest AD are favoured. The process is used in the production of billets of superalloys, high-speed steels, ceramics etc. where the integrity of the materials is a prime consideration.

Sinter-HIP: With sintered metals a relative density of about 92% is sufficient to ensure that open porosity - i.e. surface-connected porosity - has been eliminated and, if vacuum sintering has been used so that there is no gas in the pores, such parts may be HIPed to full density without canning. In a recently developed process, sintering followed by HIPping in the same vessel is achieved. The vessel is evacuated, raised to sintering temperature, and then, at a predetermined stage, high pressure argon is introduced.

This process, called Sinter-HIP or Pressure Assisted Sintering (PAS) is rapidly superseding the two stage process of vacuum sintering followed by HIPping in a separate apparatus for hardmetal cutting tools, and it can be expected to find increasing application more generally.

Ceracon Process: In the 1980s, a number of processes were developed, which could be generically described as "pseudo-HIP" processes. Of these processes, Ceracon remains as the one process that succeeded in creating a market niche.

The term "Ceracon" is derived from ceramic granular consolidation. This involves charging heated ceramic grains directly into a die and then inserting a preheated preform or group of preforms into the hot ceramic, which, when compacted by the axial loading of the press, acts as a pseudo-isostatic pressure transfer medium. Densification and deformation of the preform result, 100% of theoretical density being achieved with the selection of the proper variables. Cycle time under pressure is a matter of seconds, the ram being retracted once the desired load has been achieved. Following consolidation, the ceramic granular medium and the now fully dense parts are separated by ejecting the contents of the die and separating the ceramic grains and parts. The system can be fully automated using a robot for handling of ceramic grains and preforms and a variety of shapes such as connecting rods can be produced.

High Velocity Compaction (HVC): HVC is a compaction technology developed by Höganäs AB and Hydropulsor AB around 2000. The technology involves compaction in less than 20 milliseconds by a high-energy impact. Further densification is possible with multiple impacts at intervals as short as 300 ms. Materials based on both pre-alloyed and diffusion-alloyed ferrous grades have been compacted to densities in the range 7.4 to 7.7 g/cm3.

This approach is potentially particularly useful for the production of large components (i.e. weights up to 5kg), but, like high pressure conventional compaction, is likely to be limited to "chunky" part geometries, although developments to take the technology into multi-level parts have been reported. Helical gear forms are difficult to imagine by this technique, but the production of straight spur gear forms at density levels over 7.5 g/cm3 has been demonstrated.

Rapid Manufacturing Processes: Several forming methods, originally aimed at Rapid Prototyping, have now been developed further

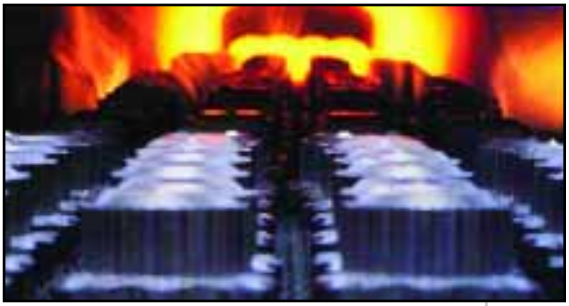


Fig 9: PM Sintering parts entering a mesh belt sintering furnace, courtesy of Ames S.A.

as means of low volume production of components from a wide range of metal powders. These methods are also sometimes referred to as Freeform Fabrication or Direct Writing technologies.

The original process of Selective Laser Sintering involved building up a 3-dimensional shape layer by layer by rastering a laser beam on the surface of a bed of powder to create sintering locally and was restricted in the types of materials that could be processed. Also, the achievable sintered density was restricted to quite a low level by the packing density of the initial powder bed and usable components could generally only be produced by post-sintering infiltration.

Of much more potential is a range of 3-dimensional Powder Melting methods.

These can be separated into two classes; those using a powder bed (i.e. similar to Selective Laser Sintering) and those based on injection of powder into an energy beam. In both cases, there are examples of developed commercial processes that use either a laser or electron beam as the energy source.

Powder bed methods raster the energy beam to build a 3-dimensional shape layer by layer by melting individual powder particles. In essence, therefore, they are micro-casting methods. Commercially available methods include SLM (Selective Laser Melting) and EBM (Electron Beam Melting).

Powder injection methods involve the deposition of a 3-dimensional shape from molten metal created by injection of powder into an energy beam. In essence, these processes are weld deposition on a micro-scale. The commercial processes include Lasform, LDMD (Laser Direct Metal Deposition), DMD (Direct Metal Deposition) and LENS (Laser Engineering Net Shaping).

These methods can become commercially viable at small batch sizes as they obviate the need for the manufacture of the tooling required for conventional processes. They are also very useful for the building of product shapes that could not be produced in any other way - any shape, that can be designed by CAD, can be built by these processes

SINTERING

As the term 'sintered part' implies, sintering is a key part of the operation. It is here that the compact acquires the strength needed to fulfil the intended role as an engineering component. In general, sintering requires heat.

The ISO definition of the term reads: 'The thermal treatment of a powder or compact at a temperature below the melting point of the main constituent, for the purpose of increasing its strength by bonding together of the particles'.

Theories about exactly what happens during sintering have provided the subject matter of innumerable conferences and learned scientific papers, but these need not concern us here.

Suffice to say that atomic diffusion takes place and the welded areas formed during compaction grow until eventually they may be lost completely.

Recrystallisation and grain growth may follow, and the pores tend to become rounded and the total porosity, as a percentage of the whole volume, tends to decrease.

The operation is almost invariably carried out under a protective atmosphere, because of the large surface areas involved, and at temperatures between 60 and 90% of the melting-point of the particular metal or alloys. For powder mixtures, however, the sintering temperature may be above the melting-point of the lower-melting constituent, e.g. copper/tin alloys, iron/copper structural parts, tungsten carbide/cobalt cemented carbides, so that sintering in all these cases takes place in the presence of a liquid phase, hence the term liquid phase sintering.

It is, of course, essential to restrict the amount of liquid phase in order to avoid impairing the shape of the part. Control over heating rate, time, temperature and atmosphere is required for reproducible results.

The type of furnace most generally favoured is an electrically heated one through which the compacts are passed on a woven wire mesh belt. The belt and the heating elements are of a modified 80/20 nickel/chromium alloy and give a useful life at temperatures up to 1150°C.

For higher temperatures, walking beam furnaces are preferred, and these are increasingly being used as the demand for higher strength in sintered parts increases. Silicon carbide heating elements are used and can be operated up to 1350°C.

For special purposes at still higher temperature, molybdenum heating elements can be used, but special problems are involved, notably the readiness with which molybdenum forms a volatile oxide.

Molybdenum furnaces must operate in a pure hydrogen atmosphere.

Controlled Atmospheres: These are essential for almost all sintering processes, to prevent oxidation and to promote the reduction of surface oxides.

In practice dry hydrogen, nitrogen/hydrogen mixtures and partially combusted hydrocarbons are mainly used, although the first named is often precluded because of cost. It is however, used for sintering carbides and magnetic materials of the Alnico type.

Dissociated ammonia containing 75% hydrogen and 25% nitrogen can readily be produced free from oxygen or water vapour, and having a dewpoint of the order of -50°C. It can replace pure hydrogen for many applications at approximately one-third the cost, with the obvious exceptions where reaction with nitrogen cannot be tolerated. It can be used to sinter iron, steel, stainless steel and copper-base components and is particularly useful for high temperature sintering of ferrous grades that contain elements (e.g. Cr), which form oxides that could not be readily reduced in other atmospheres.

Until recently, the most widely used atmospheres, primarily because of their lower cost, were produced by partial combustion of hydro-carbons. By variation of the air-to-gas ratio, a wide range of compositions can be obtained. For practical applications, since the combusted gas contains water vapour it must be dried to a dewpoint of less than 0°C for satisfactory operation with iron components.

Hydrocarbon gas, such as methane, butane or propane, reacted with a limited amount of air may contain up to 45% of hydrogen, some carbon monoxide and dioxide with nitrogen as the remainder. Because of the endo-thermic nature of this reaction, external heat has to be supplied, and for that reason the resulting atmosphere is called endogas.

If the hydrocarbon is burnt with just insufficient air for complete combustion, an atmosphere which may contain 5% or less of hydrogen and a very large percentage of nitrogen is produced, and as this reaction is exo-thermic, the atmosphere is called exogas. It is the cheapest atmosphere available, but its reducing potential is low and thus the removal of oxides from the powder compacts is less efficient and lower sintered strengths may result.

Fig 10: *PM Sintering furnace, courtesy of Elino Furnaces GmbH*

For sintering steels, i.e. ferrous alloys containing carbon as an alloying element, the carbon potential of the atmosphere is very important. It should be in equilibrium with the steel.

Over the past decade or so, the atmosphere type most commonly adopted for the sintering of ferrous parts has become increasingly the so-called 'synthetic' atmospheres, also called

nitrogen-based atmospheres, since they are produced by careful mixing of predominantly nitrogen with hydrogen (usually at the 5 to 10 volume% level). For the sintering of steels, a hydrocarbon gas is also often added in predetermined proportions, in order to reduce any residual oxygen contained within the porosity in the parts. These atmospheres, though possibly more expensive than exo- or endogas, have the advantage of cleanliness, more reliable adherence to the specified composition (e.g. they are designed to be "carbon-neutral", the carbon content of the sintered product being controlled solely by the graphite addition to the initial powder mix) and inherently low water vapour content.

Sinter/Hardening: New types of sintering furnaces allow low alloy steel parts to be sintered with neutral carbon potential (without decarburisation or carburisation) and then to be hardened in a rapid cooling zone.

The heat treatment is achieved by high speed circulation of a water cooled protective gas in the rapid cooling zone of the furnace with cooling rates of up to 50°C/sec achievable between 900°C and 400°C.

In recent years, the major iron powder suppliers have also developed special, high hardenability powder grades that have been specifically tailored to respond to sinter hardening. These materials are often "hybrids", involving full pre-alloying with Mo and diffusion-alloying or bonded mixing of other additions. Other materials have been designed to give satisfactory response to sinter-hardening using low levels of alloying additions and less expensive elements such as Cr and Mn. This process results in a homogeneous martensitic structure in the PM steels.

Close dimensional tolerances are maintained in the sinter-hardened parts thus eliminating the need for sizing. The combination of sintering and hardening in one step has reduced the production costs of low alloy steel parts, which need post sintering heat treatment.

The sinter-hardening furnace also provides other cost benefits through its ability to generate the endothermic sintering atmosphere in the furnace itself from a combustion gas and air and also through the use of the endothermic gas flowing out of the sintering zone to heat the PM parts from ambient temperature to approx. 500-600°C.

Fig 11: *Double-door crucible VIGA atomization unit courtesy of ALD Vacuum technologie sa*

Vacuum Sintering: Vacuum can be regarded as a special case of a controlled atmosphere, and is probably the best from the scientific point of view. Gas within the compact is readily removed and readily oxidisable metals such aluminium, beryllium, niobium (columbium) and tantalum, the oxides of which are not reduced even by the best gaseous reducing atmospheres may be sintered.

Vacuum sintering, however, is very expensive and for that reason is unlikely to find application in the production of standard, run of the mill mechanical parts where the atmospheres referred to earlier give quite satisfactory results. Apart from equipment cost and relatively low rates of production, the process is difficult if not impossible to make continuous and to automate. However, for certain special applications, the number and volume of which is increasing, vacuum sintering is becoming the standard e.g. high speed steels and other highly alloyed steels (especially chromium-containing steels), hardmetals (especially those containing TiC, TaC or NbC) and titanium alloys.

Dimensional Changes During Sintering: As mentioned above, sintering leads to progressively increased strength by causing the particles to weld together by diffusion. Generally, the part tends to increase in density as sintering proceeds and this still further improves the mechanical properties. Increase in density implies, of course, an overall shrinkage which leads to complications. It is possible, however, to get an increase in size, i.e. growth, taking place during sintering.

This growth can result from a number of factors: (a) entrapped gases within the compact (b) water vapour formed within the object by reduction of oxides (c) decomposition products of the lubricant. Rapid heating and high green density intensify all these effects and may lead not only to overall growth, but to blistering and distortion. Clearly such behaviour must be avoided.

Another cause of growth, which is quite distinct from the above, is the result of having mixed powders of different elements. Conspicuous examples are iron containing copper, and copper containing tin (to form bronze). The exact mechanism whereby this growth occurs has not been established to everyone's satisfaction, but there is general agreement that it is a diffusion effect. The growth is most marked above the melting point of the lower melting constituent. In this case, also fast heating rates tend to increase growth. Under controlled plant conditions, reproducible size change can be maintained. It is, of course, necessary to allow for this change in the design and manufacture of tools, but it is possible and increasingly practised so to balance the composition and sintering regime that no dimensional change takes place. It should however be noted that dimensional change is influenced also by compact density: the lower this is the greater the tendency to shrink. This is one of the reasons that uniformity of density of the compact is of such importance. If there is significant variation from one part to another the differential dimensional change in the various sections can lead to warpage.

Liquid Phase Sintering: As already mentioned there are a number of commercial processes during which a liquid phase is present at some stage of the sintering operation. Such a phase markedly assists the sintering operation, and can be regarded as a special case of activated sintering. Tin with copper, copper with iron, the binder metal (often cobalt) in cemented carbides are typical examples.

Ferrite Phase Sintering: A further special case of activated sintering, developed in the 1990s, has involved the use of ferrous material grades, incorporating an addition of an element that stabilises ferrite phase at sintering temperatures (the commercial powder grades contained 3.5%Mo, either fully pre-alloyed or mixed, for this purpose). Sintering activity can be greatly increased, as self-diffusion of iron is around 100 times greater at a given temperature in the ferrite phase rather than in austenite. There is a requirement with this approach for the alloy system and sintering atmosphere to be completely carbon-free, as carbon is a strong austenite stabiliser. The carbon-free product can then be readily sized after sintering and, if necessary, carbon then introduced for strengthening purposes by case-hardening or even through carburising.
The use of this approach, particularly when combined with higher temperature sintering, can produce sintered density levels around 7.5g/cm3.

POST SINTERING OPERATIONS

Re-pressing: Even with the best control that is feasible in practice, there will inevitably be some variation in the dimensions of parts produced from a given material in a given die set.

Typically, it is possible for parts 'as-sintered' to be accurate to a tolerance of 0.002mm per mm, in the direction at right angles to the pressing - direction, and 0.004mm per mm parallel to the pressing direction. Dimensional accuracy can be greatly improved by re-pressing the part after sintering. This operation is called sizing.

Sizing may be done in the die that was used for compacting the powder in those cases where the dimensional change on sintering is controlled at or very near to zero, but commonly separate sizing tools are used.

Re-pressing is used also to imprint or emboss the face(s) of the component in contact with the punch(es), in which case the process is referred to, for obvious reasons, as coining.

During re-pressing the density of the part is generally increased, especially if the as-sintered density is low. In certain cases where strength and other mechanical properties are required to be at maximum, re-pressing is used principally to achieve such densification. Further improvement is achieved by re-sintering

Hot Isostatic Pressing: HIP is used as a post-sintering operation to eliminate flaws and microporosity in cemented carbides. It is

predicted that as HIP vessels increase in size the economics of the process will be sufficiently attractive to allow its use even on low-alloy steel structural PM parts in order to achieve full-density.

Forging: This is a technique in which a pressed and sintered blank is hot re-pressed in a closed die that significantly changes the shape of the part, and at the same time can give almost complete density and hence mechanical properties approaching or even surpassing those of traditional wrought parts. Powder (or Sinter) Forging is dealt with in more detail in a later section.

Surface Cold Rolling: It is possible to increase density level (and therefore performance level) of PM parts by applying an appropriate deformation after sintering. Powder Forging is, of course, a long established example of this approach and, also, a certain degree of densification can be produced by cold calibration or re-pressing. Surface Cold Rolling, on the other hand, is a means of creating densification locally just in the areas where it is most often required, the surface layers of the component.

The technology was originally applied to improve rolling contact fatigue resistance of PM bearing races, but has subsequently been applied to the tooth profiles of both straight spur and helical gears, to improve tooth bend fatigue strength and surface contact fatigue strength (pitting wear resistance).

By rolling a pre-toothed PM perform, with a controlled amount of excess stock on the tooth profile, with a meshing toothed rolling tool, a local densification can be created in the surface layers of the entire tooth profile. With such a densified surface, the PM part can respond to a subsequent case carburising treatment in a similar way to a solid steel gear.

Infiltration: An alternative method of improving the strength of inherently porous sintered parts is to fill the surface connected pores with a liquid metal having a lower melting point. Pressure is not required. Capillary action is sufficient, provided that the infiltrant wets the metal concerned. It is desirable,that the infiltrant has a limited capacity to dissolve the metal being infiltrated otherwise the surface of the part may be eroded.

The process is used quite extensively with ferrous parts using copper as infiltrant but to avoid erosion, an alloy of copper containing iron and manganese, is often used.

Clearly if the molten copper is already saturated with iron its ability to erode the surface is lost. Other proprietary infiltrant compositions are also on the market.

However, as we saw in the section on dimensional change during sintering, the diffusion of copper into iron can lead to growth.

Infiltration is also used to make composite electrical contact material such as tungsten/copper and molybdenum/silver; the lower melting point metal being melted in contact with an already sintered skeleton of W or Mo.

Impregnation: This term is used for a process analogous to infiltration except that the pores are filled with an organic as opposed to a metallic material. An outstanding example is oil-impregnated bearing materials, which are dealt with in detail later; but, increasingly, impregnation with thermo-setting or other plastic materials is being done.

The benefits to be obtained include some increase in mechanical properties, sealing of the pores, which may provide pressure-tightness and will also prevent the entry of potentially corrosive electrolyte during a subsequent plating operation.

Additionally the machining of sintered parts is improved, a feature that is referred to in more detail later.

Fig 12: *Oil Quenching System, courtesy of Hoeganaes Corp*

Heat Treatment: Although many, perhaps the bulk of sintered structural parts are used in the as-sintered or sintered and sized condition, large quantities of iron-based parts, correctly speaking steel parts, are supplied in the hardened and tempered condition. Conventional hardening processes are used, but because of the porosity inherent in sintered parts, they should not be immersed in corrosive liquids - salt baths, water, or brine - since it is difficult to remove such materials from the pores. Heating should be in a gas atmosphere followed by oil-quenching. These restrictions may not apply to very high density parts (>7.2g/cc) nor to parts that have been infiltrated.

Surface-Hardening: Carburising and carbonitriding of PM parts is extensively used, and again gaseous media are indicated. Because of the part's porosity, the 'case' is generally deeper and less sharply defined than with fully dense steels.

Steam Treatment: A process peculiar to PM parts is steam-treatment, which involves exposing the part at a temperature around 500°C to high pressure steam. This leads to the formation of a layer of magnetite (iron oxide) on all accessible surfaces and a number of desirable property changes result.

Firstly the corrosion resistance is increased by the filling of some of the porosity

Secondly, this reduction in porosity of the surface layer leads also to improved compressive strength.

Thirdly, the oxide layer significantly increases the surface hardness and more importantly the wear resistance.

Steam-treatment is often followed by dipping in oil which enhances the blue/black appearance and still further increases the corrosion resistance. The treatment is not generally applicable to hardened and tempered parts because the exposure to the high temperature would undo the hardening.

Blueing: Heating in air at a lower temperature (200-250°C) can also be used to provide a thin magnetite layer that gives some increase in corrosion resistance, but it is much less effective than steam treatment.

Plating: Sintered parts may be plated in much the same way as wrought or cast metals, and copper, nickel, cadmium, zinc, and chromium plating are all used.

However, it is important to note that low density parts should be sealed - e.g. by resin impregnation - before plating, to prevent the electrolyte from entering the pores and causing corrosion subsequently. Parts that have been oil-quenched cannot be plated satisfactorily unless the oil is removed before resin impregnation.

Work has shown that it may be possible successfully to plate unimpregnated porous parts with nickel by electroless plating, which process will plate also the surfaces of any of the pores into which the solution penetrates.

Coatings: A large percentage of hardmetal cutting tool inserts are now coated using chemical vapour deposition (CVD) or physical vapour deposition (PVD).

The lower temperature PVD process also allows steels to be given a wear resistant layer of TiC, TiN, Al2O3 or a combination of these materials and sintered high speed steel tools are also now being coated.

Above- Fig 13: *Watch Parts, courtesy of ETA S.A.*
Below - Fig 14: *Titanium Aluminium Nitride Coated Drill Bits, courtesy of Hilti AG*

Machining: Although a major attraction of PM parts is that they can be produced accurately to the required dimensions, there are limitations to the geometry that can be pressed in rigid dies, and subsequent machining, for example of transverse holes or re-entrants at an angle to the pressing direction is not uncommon.

The existence of porosity alters the machining characteristics and in general tool wear is greater than with the same composition in the fully dense form. Carbide tools are recommended, and lower cutting speeds may be necessary.

The machinability can be improved by incorporating certain additions in the powder mix - e.g. lead, copper, graphite, sulphur or a metal sulphide such as manganese sulphide, and, as already indicated, by infiltration or resin impregnation.

If these points are borne in mind all the traditional machining processes - turning, milling, drilling, tapping, grinding, etc. - can be done quite readily.

De-burring: De-burring is done with sintered parts, and is used to remove any 'rag' on edges, resulting from the compacting operation or a machining step. Tumbling, sometimes in a liquid medium with an abrasive powder, is normally employed. At the same time, brushing and some hardening of the surface layers may occur.

PRODUCTS AND APPLICATIONS

Porous Bearings: The advantage of porous bearings is that the pores can be filled with lubricating oil, so that the bearing requires no further lubrication during the whole life of the machine in which it is used.
Typically these are made of bronze. Starting materials may be mixed elemental powders of copper and tin, fully pre-alloyed bronze powder, or mixtures of the three. If pre-alloyed bronze is used, shrinkage on sintering normally results, while, with elemental powder, a significant growth can be obtained.

Clearly it is possible by using the appropriate proportion of pre-alloyed bronze to achieve a situation in which the dimensions of the finished part are approximately the same as those of the die. In this way the compacting tools may be used for the sizing operation, which is essential with bearings in order to 'true-up' the part.

In addition to the normal lubricant, graphite is often added to the mixes used for porous bronze bearings. This is thought to assist in pore formation and, in addition, it acts as a lubricant in its own right. It is essential that the pores form an interconnected system of controlled size and volume, so that oil is supplied to the entire bearing surface.

The rate of oil supply automatically increases with temperature and, therefore, with increasing speeds of rotation, to achieve optimum working conditions. For most applications, the porosity should lie between 20% and 35% by volume, the upper limit being imposed by strength considerations, which varies inversely with its porosity.

Additionally porous bearings made of iron, iron copper, or iron mixed with bronze may be used in non-critical applications, but 90/10 bronze is the most widely used, having a low coefficient of friction, high wear resistance, and also resistance to seizure with the still shaft.

The processing steps are compacting at between 150 and 230MPa, sintering at approximately 800°C for between 5 and 10 minutes, followed by sizing and oil-impregnation. Very high production rates are possible because of the short sintering times required. Iron-based bearings have the advantage of greater strength and, in addition, their coefficient of thermal expansion is of the same order as that of the steel shaft.

Compacting is carried out between 230 and 380MPa, followed by sintering in combusted gas atmospheres or cracked ammonia at 1100°C to 1150^{O}C for approximately 30 to 45 minutes, sizing and impregnation.

Fig 15: *Self-lubricating PM Bearings, courtesy of Oilite Bearings Ltd*

Fig 16: PM Bearings

Non Porous Bearings: Bearings of copper/lead or copper/tin/lead (leaded bronze) made from powder. For heavy duty applications, steel backed bearings of copper/lead or copper/tin/lead (leaded bronze) made from powder are widely used

Large-scale production is carried out under continuous processing conditions, whereby the powder is loose-sintered onto a steel backing-strip, rolled to a density approaching theoretical and to the required thickness, and finally annealed. In addition to the economic advantage of this production method in comparison with cast alloys, a superior microstructure and better properties are obtained.

Lead, although soluble in molten copper and bronze, is practically insoluble in the solid metal. Thus, when such alloys are made as castings, the lead is rejected and appears as a coarse, non-uniformly distributed second phase.

In the production of powders, which is done by atomisation, the droplets are cooled very rapidly so that a uniform distribution of very fine lead inclusions results. For this application, spheroidal powders are used because they are more free flowing and pack to a higher density than irregular particles.

The demand for materials that can operate under conditions, where the presence of oil or grease is unacceptable, has led to the development of the dry bearing. The PTFE bearing is an outstanding example in this class. Polytetrafluorethylene (Fluon or Teflon) is a polymer of carbon and fluorine, which has an extremely low coefficient of friction and is stable up to 300°C under almost all corrosive and oxidising conditions. It is possible to overcome the inherent disadvantages of massive PTFE, namely low strength, poor heat conductivity, and high thermal expansion, by incorporating a metal powder in the plastic. Bronze is normally used in this application.

Alternatively, a porous metal bearing, generally bronze, may be impregnated with PTFE. This applies especially to steel-backed bearings.

Fig 17: Filters - image supplied by Porvair Ltd.

Filters: Controlled porosity is also employed in the manufacture of metal filters and diaphragms. They have the advantage over their ceramic counterparts of higher strength and resistance to mechanical and thermal shock.

The close control over the pore size and permeability is achieved by the use of powders having a narrow range of particle size. Perhaps the most commonly used filter elements are made of bronze (89/11 Cu/Sn), and spherical powders are used.

The filter profile is formed by a loose packing of the powder in the mould and the inherently poor compressibility of spheres is no disadvantage. Where products are required to have limited or localised porosity, conventional pressing is necessary and irregularly shaped particles are more suitable.

Metal filters are available in a wide range of materials including copper, nickel, bronze, stainless steel and 'Monel', and are widely used for the filtration of fuel oils, chemical solutions and emulsions. They are also efficient in separating liquids of varying surface tension, and have been successfully applied to jet engine fuels, where water is removed at the same time as the fuel is filtered. Similar devices are widely used for sound damping on air compressors and the like.

Friction Materials: Sintered metal friction components are particularly useful for heavy-duty applications, e.g. aircraft brakes, heavy machinery clutch and brake linings etc..

They consist essentially of a continuous metal matrix, into which varying amounts of non-metallic friction generators, such as silica and emery are bonded.

Compositions tend to be complex in view of the characteristics required, and may include copper, tin, iron, lead, graphite, carborundum, silica, alumina, emery and asbestos substitutes. The sintered material has a high thermal conductivity and may be used over a wide range of temperature. Satisfactory performance figures have been reported for copper-based materials, operating at surface temperatures up to 800°C and from iron-based materials up to 1000°C.

Their resistance to wear is superior to resin-bonded materials, and therefore, permits the use of components of thinner section. Because of the large surface area, and this thinness of section, the components are relatively weak. Mechanical strength is imparted by bonding the friction element to a steel backing-plate, either by brazing or welding, or by sintering the two components together under pressure.

Compared with solid phosphor bronze or aluminium bronze friction elements, the sintered material offers many advantages. The most important is probably the much wider range of friction characteristics, which can be obtained from variations in the dispersion of non-metallic particles.

Structural Parts: The bulk of structural parts is based on iron, but significant quantities of copper, brass, nickel silver and bronze parts are also made

Ferrous Structurals Parts: In the applications dealt with in the other sections, the powder metallurgy process is used to produce materials having special characteristics that either cannot be achieved in any other way or that can be achieved only with great difficulty.

In the case of structural parts, the justification for using PM is, in many cases, quite different. No special technical merit is claimed for the product in comparison with similar parts made by alternative processes-casting, forging, stamping.

The justification is economic, i.e. there is a cost saving. At first sight this may seem difficult to understand. The bulk of structural parts is based on iron, and iron powders are significantly more expensive than iron in the solid state.

The cost savings that enable this disadvantage to be overcome are basically twofold:

(a) sintered parts can be produced directly to the specified dimensions, markedly reducing the amount of machining required or eliminating it completely;

(b) in consequence of (a) material usage is very much better, scrap being almost negligible.

The saving in machining costs as a proportion of the total cost is likely to be greater the smaller the part and until recently, the bulk of parts produced from powder were small, less than 450g in weight.

Another factor is that the size and, therefore, the cost of presses increases with the size of the part being made. For this reason the compressibility of the powder mix is of major importance, and has militated against the use of prealloyed steel powders which, inevitably, require greatly increased pressures to make compacts of the required density.

When strengths greater than those obtainable with 'pure' iron powder are required, it is customary to add powder of alloying elements to the mix. The choice is restricted to elements that do not oxidise in commercial protective atmospheres and, in practice, copper is the most widely used.

Copper has the advantage of melting at a temperature below the sintering temperature used for iron (1120°C) and, therefore, alloying is rapid.

The cheapest strengthening element for iron is, of course, carbon, but its use in sintered parts depends on the ability to control the composition, and this has driven the trend towards the use of sintering atmospheres that are "carbon-neutral" i.e. neither carburising nor decarburising (e.g. synthetic nitrogen-hydrogen mixes). Copper and copper plus carbon remain the most widely used additions.

Nickel and molybdenum are also used as hardenability additions, but it is important that such additions be homogenously mixed to ensure a well-controlled response. This requirement led to the development of diffusion-alloyed (or "partially pre-alloyed") grades from the mid-1970s. These grades involved the use of a low temperature diffusion process to "tack" fine particles of alloying additions (Ni, Mo or Cu) to the high compressibility base iron powder

particles. This technique combines high and reproducible hardenability with retention of the high compressibility of the base powder.

Additions such as graphite and lubricant cannot be attached to the base iron powder by diffusion and this has led to the development of "bonded pre-mixes" in which the additions are held in place using an adhesive binder. Bonded pre-mixes were first introduced in the late 1980s and, since 2000, grades have become available where all alloying additions are bonded to the base iron as an alternative to diffusion alloying. These grades can show benefits over their diffusion-alloyed counterparts in terms of higher apparent density and improved flow characteristics.

In recent years, a number of high strength binder-treated grades have been developed that make use of lower cost hardenability agents such as chromium, manganese and silicon. As some of these elements form more stable oxides, the sintering of such grades often requires greater attention to the dew-point of the atmosphere and/or the use of higher sintering temperatures.

During the past decade, it has been recognised that certain alloying additions (particularly Mo) create little or no deterioration in compressibility in full pre-alloying. Consequently, a number of Mo-containing grades have been introduced, Some of these grades, with up to 1.5% Mo, have been specifically developed to make use of their superior hardenability in case hardening. Powder grades, combining prealloying with Mo and diffusion alloying or bonded mixing of the other alloy additions, are now available. Some of these grades have been specifically developed to respond to sinter-hardening.

Infiltration also is used to increase strength, the most common infiltrant being copper with a small percentage each of iron and manganese to avoid erosion. It is not necessary to infiltrate the whole part; quite often local infiltration of highly stressed areas is sufficient.

Achievable strength in ferrous structural parts has also been enhanced significantly over the past two decades by a range of process developments aimed at creating higher density levels. These comprise developments at the compaction stage (e.g. High pressure cold compaction, Warm Compaction, High Velocity Compaction), during sintering (e.g. Activated Sintering, Ferrite-phase Sintering) or in post-sintering densification operations (e.g. Surface Cold Rolling).

As a further means of establishing higher performance structural part applications, the on-line **Global PM Property Database (GPMD)** was developed and launched in 2004 as a tool for designers and engineers

The other major area of advance in ferrous parts over the years has been an attack on limitations as to geometry. For instance these can, in some cases, be overcome by making two parts and joining them, e.g. during the sintering process, by using materials for the different parts of the assembly that give different size changes in sintering to create mechanical interference and eventually diffusion bonding, or after sintering using joining processes such as copper brazing or projection welding.

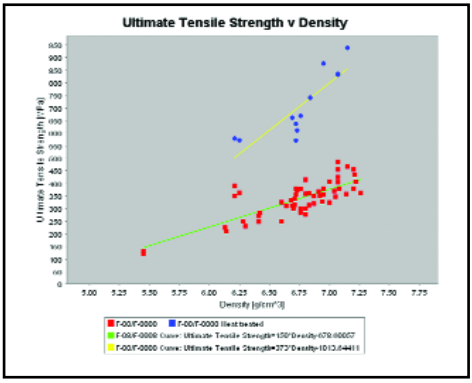


Fig 18: Graph showing Ultimate Tensile Strength v Density, from the GPMD

Fig 19: Planetary Carrier for Automotive Transmission courtesy of Ames SA

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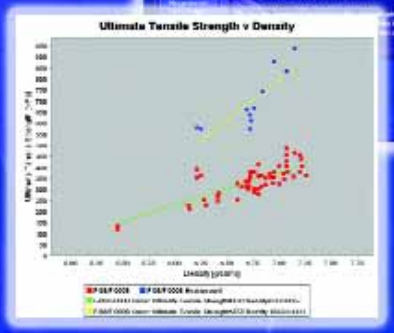

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In this way, undercuts and transverse projections can be incorporated.

Another device for achieving similar results is the use of 'split-die' i.e. a die, which is in two pieces with the junction at right angles to the pressing direction. The compact is removed by separating the two halves and taking the compact out in the middle. This process is known as the Olivetti process.

These advances both in geometrical complexity and performance level have led to a great expansion in the types of PM structural part applications.

The automotive sector has emerged as the dominant customer industry for PM structural parts - worldwide, 75-80% of all PM structural parts go to the automotive sector. Automotive applications are now quite diverse, but engine and transmission components are particularly important, accounting for at least 70% of total automotive usage.

Engine applications include:

- *Engine timing pulleys, sprockets and hubs*
- *Valve train parts - valve seat inserts, valve guides, valve timing control and coupling devices*
- *Balancer gears*
- *Main bearing caps*
- *Engine management sensor rings*
- *Oil and water pump gears*

Transmission applications feature in both manual and automatic transmissions:

- *Synchroniser system parts*
- *Clutch hubs*
- *Gear shift components*
- *Planetary gears and carriers*
- *Turbine hubs*
- *Clutch and pocket plates*

PM parts feature in other automotive systems:

- *Shock absorber components - piston rod guides, piston valves and end valves*
- *Anti-lock Braking System (ABS) sensor rings*
- *Exhaust flanges and oxygen sensor bosses (a major application for PM stainless steel parts)*
- *Gears and bearings in small electric motors*
- *Door lock parts*

PM structural parts also have a number of important non-automotive markets:

- *DIY hand power tools*
- *Domestic appliances*
- *Business machines*
- *Leisure and garden*
- *Industrial motors and controls*
- *Hardware (lock parts, latches etc.)*

A selection of these applications is illustrated in the product photographs in this publication, and on the EPMA website-***www.epma.com.***

Non Ferrous Structural Parts: The production of structural parts in non-ferrous materials is on a much smaller scale but significant quantities of copper, brass, nickel silver, and bronze parts are made, and the production of aluminium from powder is now developing.

Bronze: In the case of bronze there is an important technical advantage. Because of the wide freezing range of copper/tin alloys it is difficult to avoid serious inter-dendritic porosity in bronze castings, and pressure-tight pump bodies and other hydraulic fittings are difficult to cast without a significant percentage of rejects.

By using the powder route, this problem is overcome, since, although there is normally a percentage of porosity in the sintered part, this is in the form of separate small holes rather than the interconnected porosity found in castings. Of course, we are talking of densities much higher than those of self-lubricating bearings.

Fig 20: *Synchronizer hubs, courtesy of PMG Füssen GmbH*

Aluminium: the usage of PM aluminium parts has seen a significant recent growth, driven largely by the automotive sector's desire for weight reduction. Camshaft bearing caps have emerged as a leading application for PM aluminium parts.

Powder Forgings: Powder forging produces fully dense PM steel parts, such as the automotive connecting rod. The production of traditional PM parts has been expanding at a significantly faster rate than the general growth of engineering production and when it was originally developed in the 1970s powder forging or sinter forging was expected to alter fundamentally the scale of the PM industry.

Process: In this process, a powder blank is pressed to a simple shape halfway between that of a forging billet and the required finished part. This compact, referred to as a preform, is sintered and then hot forged to finished size and shape in a closed die. The amount of deformation involved is sufficient to give a final density very closely approaching that of the solid metal and, consequently, the mechanical properties are comparable with those of material forged from wrought bar

Advantages: Indeed the mechanical properties may be superior in some respects because of the freedom of the powder forged part from directionality, the greater homogeneity as regards composition, and a finer microstructure, as well as the absence of internal discontinuities resulting from ingot defects that are possible in forgings made from cast metal.

An additional advantage is the dimensional consistency achievable in consequence of the accurate metering of the quantity of powder used

Limitations: There are limitations to the steel compositions that can be successfully produced on a commercial scale.

Steels containing readily oxidisable elements such as chromium and manganese - which happens to be also the cheaper strengthening elements - cannot easily be used, but special compositions, generally containing as alloying elements, nickel and molybdenum, the oxides of which are reduced in sintering atmospheres, have been developed.

Powder forged steel parts can be heat treated in the same manner as wrought steels.

Production costs in powder forging are generally higher than in conventional casting or forging due mainly to the high price of the starting material and tooling. However, the high precision achieved in powder forging results in considerable savings on machining costs and hence savings on investments in machining operations.

This has particularly proved to be the case for powder forged connecting rods which have gained a high market share in gasoline engine applications due to their improved dimensional accuracy, higher dynamic properties, smoother running in the engine, and significant cost savings.

Companies in North America, Japan and Europe now have large powder forging installations mainly to produce parts for the automotive industry. Such parts can have inside and outside spline forms, cam forms, and other forms that require extensive machining.

Fig 21: *Powder Forged Conrod, courtesy of Metaldyne Sintered Components*

In addition to the well known connecting rod, other applications include bearing races, torque convertor hubs, and gears.

Hardmetals and other Hard Materials: Hardmetal is the term used to signify a group of sintered, hard, wear-resisting materials based on the carbides of one or more of the elements tungsten, tantalum, titanium, molybdenum, niobium and vanadium, bonded with a metal of lower melting point usually cobalt.

Tungsten carbide is the most widely used. These materials are commonly referred to as cemented carbides or simply as carbides as, for example, carbide tools. By varying the carbide particle size, the amount of binder metal, and the sintering conditions, the properties such as wear resistance, impact strength, resistance to cratering, and hot hardness may be optimised for a given application. For example, in the case of a wire drawing die, wear resistance is the major requirement, but, for a cutting tool, especially if subject to intermittent loading, high impact strength is required.

Individual parts are produced by intensive milling together of fine cobalt powder with carbide powders of the appropriate particle size - generally from 0.5 microns to 10 microns average. The milled powders are extremely fine in comparison with those used in ordinary PM production, and consequently do not flow readily if at all. It is common practice therefore, to form small agglomerates of the powders in order to facilitate filling of the dies. This process is called granulation. These granules are mixed with a lubricant, and then pressed in a die. The resulting compacts are pre-sintered in hydrogen or vacuo at about 900°C to remove the lubricant, and then sintered at a higher temperature - usually between 1200° and 1500°C. In the pre-sintered state, the material can be machined, and this technique is used to produce short runs of parts that would not justify the manufacture of a special tool set. More commonly, the parts are pressed and sintered to the required shape and dimensions. Hot pressing in carbon dies is also employed, especially for large pieces, and hot isostatic pressing is now in commercial use. This latter is claimed to give much greater freedom from porosity.

Other materials having metallic borides or nitrides cemented with a binder metal are also known, but there have limited special applications, and although they are hard materials, they are not normally included in the term hardmetal as it is now defined.

Diamond cutting tools are made by similar processes. In this case, it is important to provide a matrix, which gives maximum support to the diamonds in order to keep the tool sharp. Each cutting application therefore requires separate consideration, and matrix materials range from bronzes of different compositions to cemented carbides.

Electric and Magnetic Components:

Electric components: Composite structures attainable only by powder metallurgy methods have been used extensively in the manufacture of electrical contacts and current collector brushes.

Fig 22: *Example of components made from hard materials, courtesy of Sandvik Hard Materials AB*

It is possible to combine the desirable conducting properties and low contact resistance of silver or copper with the strength, heat-resistance, and resistance to arc erosion of tungsten, molybdenum, nickel, etc or with the lubricating qualities of graphite.

Magnetic Components: Economic and technical advantages have also been found in the production of components for magnetic applications. A range of soft magnetic materials can be processed by PM.

Iron, silicon-iron and iron with about 5% of phosphorus are widely used in the manufacture of pole pieces and armatures for DC application.

For AC application, eddy current losses must be minimised. Eddy current losses are found to be proportional to the square of the thickness of the material serving as the flux path. This has been the main stimulus for the interest in Soft Magnetic Composite (SMC) materials. In these materials, the individual powder particles are insulated from one another by a resin addition,

which is cured in a baking process after compaction, so that the thickness of the flux path becomes equivalent to the powder particle size.

A long-standing and important use of this type of approach has been in the production of cores for self-inductance coils in high-frequency communication equipment. Sintering in the accepted sense is not required, although a thermal stabilisation or stress-relieving treatment is usually carried out to obtain optimum magnetic characteristics.

Purity and close control of compositional limits is very necessary, and electrolytic or carbonyl powders and pre-alloyed permalloy-type powder are widely used as raw material.

Recent developments in SMCs have concentrated on finding applicable insulating coatings to permit use at lower frequencies. Progress hinges on the development of coatings that will restrict eddy currents to the individual powder particles while achieving a compacted density that will permit sufficient strength to resist centrifugal forces and provide sufficient magnetic flux at low cost to compete with steel laminations.

Sintered high-permeability laminated components in nickel-iron and permalloy-type materials are also used for transformer applications, incorporating the direct rolling of powders into thin strips.

Another class of magnetic materials, processed by powder metallurgy methods, are the ferrites or magnetic oxides. The combination of ferric oxide with bivalent oxides of magnesium, nickel, manganese, copper, zinc, etc, produces materials having a wide range of both soft and permanent magnetic properties. The 'soft ferrite' has found the widest application in the manufacture of cores for radio and television.

Hard (or Permanent) Magnetic Materials: Today, the vast majority of hard magnets are processed by PM:

As mentioned already, ferrites can exhibit hard magnetic properties and, indeed, this was the first hard magnetic material type to be processed from powders, from around 1930. Because of their low costs, hard ferrites are still the most frequently used hard magnets and, in tonnage terms, represent over 90% of the current market.

Small permanent magnets of the Alnico, Cunife or Cunico types can be pressed and sintered to final shape and size, showing equivalent magnetic properties but finer grain structures and better mechanical strengths than similar cast materials.

Higher strength hard magnets based on rare earths are also processed by PM. The first such category to appear was the Sm-Co alloy materials. A more significant impact in the market has however been created by the later Nd-Fe-B magnets, with their lower costs and higher magnet energy product. Although hard ferrites still dominate in tonnage terms, Nd-Fe-B magnets currently account for over 30% of the hard magnet market in sales value terms and this market share is still growing rapidly.

POWDER ROLLING

This term is applied to the process, now established on an industrial scale, wherein a metal powder is fed continuously into a rolling mill which may be heated, and compacted between the rolls into strip. This strip is then passed through a sintering furnace and rerolled to finished size.

In general, the product does not have any advantage over strip produced by rolling cast billets, although in some cases superior homogeneity can be demonstrated as well as freedom from laminations that can arise from ingot defects.

Fig 23: *Rain sensor ring, courtesy of GKN PLC*

The main advantage is economic, and depends on the following features:

- *The yield of finished strip from castings is low*
- *The cost of fettling the ingot, of the extensive rolling, annealing and pickling, is considerable. Powder rolling is economical therefore especially in cases where the metal is produced cheaply as a powder directly during the extraction process, e.g. nickel.*
- *In the case of a material that work-hardens rapidly and, therefore, requires many intermediate annealing and pickling operations during reduction of a rolling slab, e.g. stainless steel.*

The production of small quantities of special materials by powder rolling is increasing for applications such as cobalt- or nickel-base alloy strip for welding, nickel-iron strip for controlled expansion properties, special Cu-Ni-Sn alloys for electronics, porous nickel strip for alkaline batteries and fuel cell electrodes, composite bearings, etc

OTHER PRODUCTS MADE FROM POWDER

Developments designed to produce, from powder, engineering components that are not merely as good as those currently in production by alternative routes, but to produce better products, have been made possible by improved atomisation techniques that produce clean powders of highly alloyed metals, such as high-speed steels, and complex precipitation hardenable alloys based on nickel and/or cobalt with chromium, now commonly called superalloys.

These powders are isostatically pressed in evacuated metal cylinders and subsequently hot-forged, still in the container, to give a dense billet, which is then processed to the final shape by conventional means.

The process depends on the use of inert gas, usually argon, to atomise the metal, which has been pre-alloyed by vacuum melting. Increasingly centrifugal atomised powders are favoured - e.g. those made by the REP process already referred to.

The advantages of material produced are:

- *much better uniformity of chemical composition (lack of segregation);*
- *freedom from ingot defects; and*
- *fine and uniform distribution of secondary phases (carbides and other hardening precipitates).*

The last results from the fact that each particle of powder is virtually quenched from the liquid state

Similar advantages apply to high speed steels and these are now being produced from powder by two different routes:

(1) irregular, water atomized powders are cold compacted either in rigid dies or isostatically, and then sintered in vacuum to give individual pieces of near finished shape and size; and

(2) in a process analogous to that described for superalloys, gas atomised (spherical) powder is HIPped and extruded to produce wrought material.

A number of processes for the production of near net shapes have been developed. Many of these were pseudo-isostatic hot pressing, the pressure being applied by a hydraulic press to a solid medium in which the powder or powder compact is embedded, the medium being such that, at the operating temperature, it transmits pressure in all directions as if it were a fluid.

One such process is the Ceracon process already described. Another is the so-called fluid die process in which powder is contained in a die made of a metal that is so soft at the compacting temperature that it behaves substantially as a fluid.

Superalloy shapes have been made in this way using copper/nickel alloys as the die material.

Fig 24: *An 'as HIP' High speed steel hollow bar used for producing cutting tools - Courtesy Boehler Uddeholm Powder Technology*

METAL MATRIX COMPOSITES

Metals having a dispersion of a finely divided non-metallic phase have been known for many years, the idea being to provide the strengthening that is produced by precipitation hardening without the drawback that the second phase goes into solution as the temperature rises thus limiting the operating temperature.

An early example was sintered aluminium powder (SAP) which was made by pressing and sintering heavily oxidised aluminium flake powder, the sintered material being subsequently hot worked to break up the Al2O3 films and disperse them in finely divided form.

There was some improvement in the properties but it was insufficient to make large scale use economical.

The picture was, however, changed dramatically as newer procedures for getting very much finer dispersions of the non-metallic phase have been developed, and metal matrix composites (MMC) as such materials are now called, represent a major step forward in the search for improved materials i.e. with better mechanical properties especially at elevated temperatures. Powder metallurgy is the most important route by which such composites are produced.

In the majority of cases so far developed, the strengthening phase is a stable oxide usually of another metal and the term ODS - oxide dispersion strengthening - is in everyday use.

A number of different processes are available for producing the very fine dispersions required.

In one process, an alloy of the matrix metal with the metal that forms the stable oxide is heated in an atmosphere that is reducing to the matrix metal but oxidising to the second metal. The latter is converted to oxide uniformly distributed throughout the matrix.

In the case of precious metals - Ag, Pt etc. - heating in air can be used and a range of electrical contact materials consisting of silver with a dispersion of for instance Cd oxide, Sn oxide, and/or In oxide are now widely used.

The internal oxidation as the process is called occurs as a result of the diffusion of oxygen through the silver lattice and with large sections, this is a lengthy process.

However, if powder is used, a relatively short oxidising cycle is required so that the pressing and sintering of internally oxidised powder is the best procedure. In this case, the object is not to improve the strength but the electrical properties, i.e. the resistance to contact welding.

In other cases, the matrix metal sometimes in the form of a salt is mixed with a solution of a salt of the metal with the more stable oxide and the mixture is heated in an atmosphere that is reducing to the matrix metal but oxidising to the second metal. ODS platinum and tungsten are made in this way.

Other composites use fibres or whiskers as the strengthening agent.

A further process that represented a major step forward in materials for very high temperature applications, gas turbines for jet engines in particular, was mechanical alloying.

This process involves milling, usually in an attritor, a mixture of a metal powder and a refractory material for long periods during which the refractory particles are broken up and incorporated in the metal. The 'alloyed' powder is subsequently compacted, sintered, and normally wrought by extrusion or hot rolling.

Superalloys made in this way are in service, and mechanically alloyed aluminium alloys have also been developed. In the case of aluminium, another mechanical alloy is made by a similar milling process starting with a mixture of aluminium powder and graphite, which, during the milling process, is incorporated in the metal as aluminium carbide. Al4C3.

Another class of wrought sintered material that is beginning to make an impact is based on particulate material - powder or chopped ribbon - that has been solidified and cooled at a very high rate such that metastable non-equilibrium microstructures result. They may be microcrystalline or amorphous.

The process is applicable only to certain alloys, and one important feature is that the matrix metal can retain in solid solution a much higher than the equilibrium percentage of the alloying element.

Providing that the densification and mechanical working is carried out at a temperature low enough to avoid destroying the non-equilibrium structure, remarkably enhanced mechanical properties can be achieved.

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