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History of Powder Metallurgy

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POWDER METALLURGY (PM) has been called a lost art. Unlike clay and other ceramic materials, the art of molding and firing practical or decorative metallic objects was only occasionally applied during the early stages of recorded history. Sintering of metals was entirely forgotten during the succeeding centuries, only to be revived in Europe at the end of the 18th century, when various methods of platinum powder production were recorded (Table 1).

Since the beginnings of recorded history, metal powders, such as gold, copper, and bronze, and many powdered oxides (particularly iron oxide and other oxides used as pigments) were used for decorative purposes in ceramics, as bases for paints and inks, and in cosmetics. Powdered gold was used to illustrate some of the earliest manuscripts. It is not known how these powders were produced, but it is possible that some of the powders were obtained by granulation after the metal was melted. Low melting points and resistance to oxidation (tarnishing) favored such procedures, especially in the case of gold powder. The use of these powders for pigments and ornamental purposes is not true PM, because the essential features of the modern art are the production of powder and its consolidation into a solid form by the application of pressure and heat at a temperature below the melting point of the major constituent.

Early man learned by chance that particles of metal could be joined together by hammering, resulting in a solid metallic structure. In time, man learned how to build furnaces and develop temperatures high enough to melt and cast metals and to form lower-melting alloys, such as copper and tin to make bronze.

Earliest Developments

Long before furnaces were developed that could approach the melting point of metal, PM principles were used. About 3000 B.C., the Egyptians used a "sponge iron" for making tools. In this early process, iron oxide was heated in a charcoal and crushed shell fire, which was intensified by air blasts from bellows to reduce the oxide to a spongy metallic iron. The resulting hot sponge iron was then hammered to weld the particles together. Final shapes were obtained by simple forging procedures. Although the product often contained large amounts of nonmetallic impurities, some remarkably solid and sound structures have been discovered (Ref 1).

Table 1 Major historical developments in powder metallurgy

Date	Development	Origin
3000 B.C.	"Sponge iron" for making tools	Egypt, Africa, India
1200 A.D.	Cementing platinum grains	South America (Incas)
1781	Fusible platinum-arsenic alloy	France, Germany
1790	Production of platinum-arsenic chemical vessels commercially	France
1822	Platinum powder formed into solid ingot	France
1826	High-temperature sintering of platinum powder compacts on a commercial basis	Russia
1829	Wollaston method of producing compact platinum from platinum sponge (basis of modern PM technique)	England
1830	Sintering compacts of various metals	Europe
1859	Platinum fusion process	
1870	Patent for bearing materials made from metal powders (forerunner of self-lubricating bearings)	United States
1878–1900	Incandescent lamp filaments	United States
1915–1930	Cemented carbides	Germany
Early 1900s	Composite metals	United States
	Porous metals and metallic filters	United States
1920s	Self-lubricating bearings (used commercially)	United States
1940s	Iron powder technology	Europe
1950s and 1960s	Powder metallurgy wrought and dispersion-strengthened products, including powder forgings	United States
1970s	Hot isostatic pressing, PM tool steels, and superplastic superalloys	United States
1980s	Rapid solidification, powder injection molding technology, and binder-treated ferrous premixes	United States and Europe
1990s	Intermetallics, metal-matrix composites, spray forming, nanoscale powders, water-atomized prealloyed ferrous powders with molybdenum as the principal alloying element, and warm compaction	United States, United Kingdom
2000s	Warm-die compaction, additive manufacturing (3-D printing) on a commercial basis	United States and Europe

W.D. Jones (Ref 2) wrote of a process modification developed by African tribes. After reduction, the sponge was broken into powder particles, washed, and sorted by hand to remove as much of the slag and gangue as possible. The powder was then either compacted or sintered into a porous material, which was subsequently forged. Another example of ancient reduction of iron oxide was carried out in the fabrication of the Delhi Pillar, which weighs 5.9 tonnes (5.9 metric tons, or 6.5 tons).

These crude forms of PM ultimately led to the development of one of the commercial methods for producing iron powder. By grinding the sponge iron into fine particles, and heating in hydrogen to remove oxides and anneal or soften the particles, this process is today a viable technique for producing high-quality iron powder.

Powder metallurgy practices were used by the Incas and their predecessors in making platinum before Columbus made his voyage to the "New World" in 1492. The technique used was based on the cementing action of a lower-melting binder, a technique similar to the present practice of making sintered carbides.

The technique consisted of cementing platinum grains (separated from the ore by washing and selection) by the addition of an oxidation-resistant gold-silver alloy of a fairly low melting point to wet the grains, drawing them together by surface tension and forming a raw ingot suitable for further handling (Ref 3).

A color change from the yellow of the sintered material to the whitish platinum of the final metal was caused by diffusion during heating prior to working. Heating is thought to have been accomplished by means of charcoal fires fanned by blowpipes. Analyses of these alloys vary considerably. The platinum content ranged from 26 to 72%, and the gold content ranged from 16 to 64%. Silver additions were found to vary from 3 to 15%, and amounts of copper up to 4% were traced.

Powder Metallurgy of Platinum

The metallurgy of platinum, as practiced in the 18th and 19th centuries in Europe, is considered to be one of the most important stages of development for modern PM. For the first time, complete records were available that provided insight into the various methods of powder production and the processing of these powders into solid, useful implements.

Between 1750 and 1825, considerable attention was given to the manufacture of platinum. In 1755, Lewis (Ref 4) discovered that when a lead-platinum alloy was oxidized at high temperatures, a spongy, workable mass remained after lead oxide impurities had been volatilized. Scheffer (Ref 5) found that when platinum was heated with arsenic, the platinum showed signs of melting. This finding was confirmed in 1781 by Achard (Ref 6), who described the production of a fusible platinum-arsenic alloy, probably by forming the eutectic containing 87% Pt and melting at 600 °C (1110 °F). Achard formed solid platinum by hot hammering a sponge, welding the individual particles into a large solid. The sponge was obtained by high-temperature working of the platinum-arsenic alloy, which caused volatilization of the arsenic.

This procedure formed the basis for a method of producing platinum that was first used in about 1790 in commercially manufactured chemical vessels by Jannetty in Paris. Mercury was used later in a similar process by von Mussin-Puschkin (Ref 7). Other metals worked in this way include palladium, by using sulfur instead of arsenic, and iridium (using phosphorus). Ridolfi (Ref 8) made malleable platinum for chemical vessels using lead and sulfur.

In 1786, Rochon (Ref 9) successfully produced solid platinum without using arsenic by welding small pieces of scrap platinum. He produced malleable platinum by uniting purified platinum grains.

Knight (Ref 10) found that if chemically precipitated platinum powder was heated at high temperatures in a clay crucible, it softened and could be compressed and forged. Tilloch (Ref 11) put platinum powder into tubes made of rolled platinum sheet, which were then heated and forged to produce a compact mass. In 1813, Leithner (Ref 12) reported production of thin, malleable platinum sheets by drying out successive layers of powder suspended in turpentine and heating the resulting films at high temperatures without pressure.

In 1882, a French process was reported by Baruel (Ref 13) in which 14 kg (30 lb) of platinum powder was made into a solid ingot by a series of operations. Platinum was precipitated in powdered form, slightly compressed in a crucible, and heated to white heat. The powder was then put in a steel matrix and put under pressure with a screw coining press. The compact platinum was repeatedly reheated and re-pressed until a solid ingot was formed. The final heat treatments were made in a charcoal fire at lower temperatures. Because the platinum powder was placed in the steel die while hot, this process was based on the hot pressing technique.

In Russia in 1826, a high-temperature sintering operation was applied to previously compressed powder compacts on a commercial basis for the first time. This was in contrast to methods based on hot pressing. Sobolewskoy (Ref 14) described sifted platinum powder pressed into a cast iron cylinder that featured a steel punch actuated by a screw press. The resulting compacts were annealed for one day at high temperature in a porcelain firing kiln. The final product was highly workable, especially if the platinum powder had been well washed and was of high purity. Annealing, however, caused a decrease in volume: a cylinder 100 mm (4 in.) in diameter and 19 mm (0.75 in.) in height shrank 19 mm (0.75 in.) and 6 mm (0.25 in.) in these dimensions, respectively.

Another Russian method was reported by Marshall (Ref 15) in 1832. Platinum powder in a ring-shaped iron mold was pressed by a screw press, heated to a red heat, and re-pressed. After working in a rolling mill, the compacted discs were used as coins.

The Wollaston process of producing compact platinum from platinum sponge powder is generally considered the beginning of modern PM. At least 16 years prior to his publication of 1829 (Ref 16), describing the manufacture of a product much superior to that of contemporary manufacturers, Wollaston devised the foundations for modern PM technique. Wollaston was the first to realize all the difficulties connected with the production of solid platinum ingot from metal powder and thus concentrated on the preparation of the powder. He found that pressing the powder while wet into a hard cake (to be subsequently baked at red heat) was best done under considerable pressure. In addition, because available screw presses were not powerful enough, Wollaston developed a horizontal toggle press of the simple construction shown in Fig. 1. Wollaston used the following nine steps in the manufacture of compact platinum metal (Ref 17):

1. Precipitating ammonium-platinum-chloride from diluted solutions
2. Slowly decomposing the finely divided and carefully washed ammonium-platinum-chloride precipitate into loose sponge powder
3. Grinding this sponge powder without applying pressure to the powder particles, thus avoiding any burnishing of the particles and preserving all the surface energy of the particles
4. Sieving the sponge powder
5. Washing the sponge powder with water to remove all remnants of volatile salts
6. Separating fine particles from coarser particles through sedimentation (only the finest sponge particles were used)
7. Pressing the wet mass containing the finest platinum particles into a cylindrical cake
8. Drying the wet cake very slowly and then heating it to approximately 800 to 1000 °C (1475 to 1830 °F)
9. Forging the cake while it was still hot

By applying these steps, Wollaston succeeded in producing compact platinum, which when rolled into thin sheet was practically free of gas blisters. Crucibles made from this sheet were the best-quality platinum implements of their time. Wollaston's process was used for more than a generation and became obsolete only with the advent of the platinum fusion

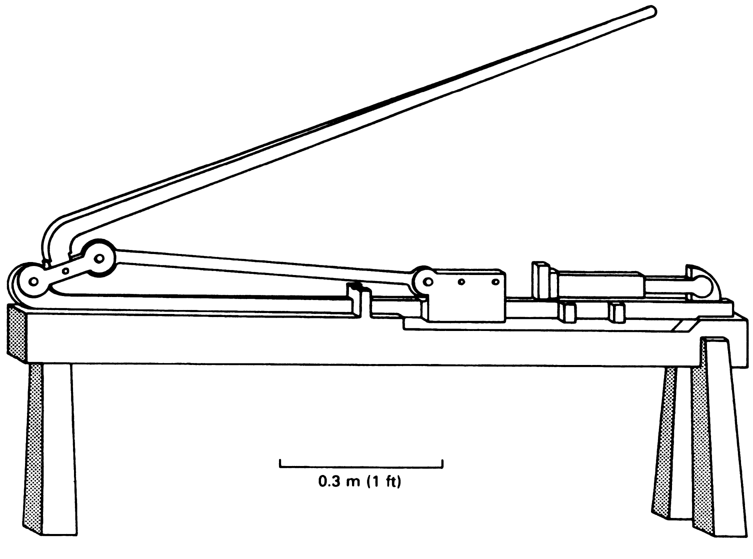

Fig. 1 Simple toggle press used by Wollaston for making platinum powder compacts

procedure developed by Sainte-Claire Deville and Debray in 1859 (Ref 17). They succeeded in producing a powerful flame with illuminating gas and oxygen, the oxygen being manufactured from manganese dioxide. The fused metal that they produced was, however, superior to Wollaston platinum in quality and homogeneity, and the fusion procedure was also less expensive and quicker than the Wollaston method. Every platinum refinery, therefore, soon adopted fusion. It is still considered the superior method for manufacturing standard-quality platinum.

Further Developments

The use of PM technology to form intricately shaped parts by pressing and sintering was introduced in the 19th century. In 1830, while determining the atomic weight of copper, Osann (Ref 18) found that the reduced metal could be sintered into a compact. Osann then developed a process for making impressions of coins from copper powder produced by the reduction of precipitated copper carbonate (Cu_2CO_3).

Osann found that reduction was best done at the lowest possible temperatures that could be used to produce a metal powder of the fineness known in platinum manufacture. High reduction temperatures resulted in granular masses that did not sinter well. Using the powder immediately after reduction or storing it in closed glass bottles eliminated contamination of the powder by the atmosphere. The powder was separated into three grades, determined by particle size, before use. To make an impression of a coin, fine powder was sprayed on the surface, followed by layers of coarser grades. The powder and a die were placed in a ring-shaped mold and compressed by the pressure of hammer blows on a punch or use of a knuckle press. Volume of the copper powder was reduced to one-sixth of the original powder during compression. Sintering was done at temperatures close to the melting point of copper, after the compacts were placed in airtight copper packets sealed with clay. A nondistorted 20% shrinkage occurred, but the sintered copper was harder and stronger than cast copper.

Osann also produced medals of silver, lead, and copper by the same procedure. Although he considered his process especially suitable as an alternative to the electrotype method of reproducing coins and medallions, Osann advocated its use as an initial production method for these articles. He believed PM could be used for producing printing type and for making convex and concave mirrors by pressing on glass. Osann thought that measurement of the shrinkage of copper compacts could be used to calculate temperature, as the shrinkage of clay cylinders was used in the Wedgewood pyrometer.

Among the advancements in the PM industry during the second half of the 19th century were Gwynn's attempts to develop bearing materials from metal powders. Patents issued to Gwynn in 1870 (Ref 19) were the forerunners of a series of developments in the area of self-lubricating bearings. Gwynn employed a mixture of 99 parts of tin powder, prepared by rasping or filing, and 1 part of petroleum-still residue. The two constituents were stirred while being heated. A solid form of desired shape was then produced by subjecting the mixture to extreme pressure while enclosing it in a mold. The patent specifically states that journal boxes made by this method or lined with material thus produced would permit shafts to run at high speeds without using any other lubrication.

Commercial Developments

The first commercial application of PM occurred when carbon, and later osmium, zirconium, vanadium, tantalum, and tungsten, was used for incandescent lamp filaments. Methods were developed from 1878 to 1898 for making carbon filaments by the extrusion and subsequent sintering of carbonaceous materials.

Osmium filaments were used for a short time from 1898 to 1900. Auer von Welsbach (Ref 20) described the production of filaments of osmium by chemical precipitation of the powder and formation of a mixture with sugar syrup, which served both as binder and, if osmium oxide powder was used instead of the metal, as reducing agent as well. The mixture was squirted through fine dies, and the resulting fine threads were subsequently fired in protective atmospheres to carburize and volatilize the binder, reduce the oxide, and sinter the metal particles into a coherent metallic wire for use as an electrical conductor.

The osmium electric lamp was soon succeeded by tantalum filament lights, which were used widely from 1903 to 1911. The general procedure (Ref 21) was similar to that used for osmium, with the exception that tantalum had to be purified by a vacuum treatment to become ductile. Similar techniques were used for the production of filaments from zirconium, vanadium, and tungsten; with tungsten, especially, extruded wires were bent into hairpin shapes before sintering to shape them for use as filaments. Because lack of ductility was the major shortcoming of these filaments, attempts were made to improve this property by the addition of a few percent of a lower-melting, ductile metal. Tungsten powder was mixed with 2 to 3% Ni, pressed into a compact, and sintered in hydrogen at a temperature slightly below the melting point of nickel. The resulting bars could be drawn, and nickel was removed from the final filaments by a vacuum heat treatment at a high temperature (Ref 22). Although this process was not commercially successful, it was an important step toward the industrial development of cemented carbides and composite materials.

Tungsten was soon recognized as the best material for lamp filaments. The problem, however, was to devise an economical procedure for producing these filaments in large quantities. A number of procedures to produce tungsten powder had been worked out earlier. In 1783, the D'Elhujar brothers (Ref 23) first produced tungsten powder by heating a mixture of tungstic acid and powdered charcoal, cooling the mixture, and removing the small cake, which crumbled to a powder of globular particles. The purification of tungsten powder by boiling, scrubbing, and skimming to remove soluble salts, iron oxide, clay, and compounds of calcium and magnesium was reported by Polte (Ref 24).

Coolidge Process. At the beginning of the 20th century, Coolidge (Ref 25) made the important discovery that tungsten could be worked in a certain temperature range and would retain its ductility at room temperature. Few changes have been made over the years on the Coolidge procedure; it is still the standard method of producing incandescent lamp filaments. In this method, very fine tungsten oxide powder, WO_3, is reduced by hydrogen. The powder is pressed into compacts, which are presintered at 1200 °C (2190 °F) to strengthen them so that they can be clamped into contacts. They receive a final sintering treatment near 3000 °C (5430 °F) by passing a low-voltage, high-current-density current through the compacts. During sintering, the compacts shrink and reach a density near 90% that of solid tungsten. The sintered compacts can be worked only at temperatures near 2000 °C (3630 °F). When heated to this temperature, they can be swaged into rounds. With increasing amounts of warm work, tungsten becomes more ductile, the swaging temperature can be progressively lowered, and the swaged bars can be drawn into fine wire at relatively low temperatures.

Other Refractory Metals. The procedures developed for the production of tungsten often were adaptable to the manufacture of molybdenum. Lederer (Ref 26) developed a method of making molybdenum using molybdenum sulfide powder. The sulfide, mixed with amorphous sulfur and kneaded into a paste, was formed into a filament. When exposed to air, the filaments became strong enough to be placed in a furnace. Heating in hydrogen resulted in formation of hydrogen sulfide and sintering of the metal into solid filaments. A similar process was patented by Oberländer (Ref 27), who used molybdenum chloride and other halides as starting materials. When the chloride was treated with a reducing agent such as ether, a paste was obtained.

Tungsten, molybdenum, and tantalum are the three most important refractory metals used today in the lamp, aerospace, electronics, x-ray, and chemical industries. Other refractory metals of minor significance were developed by the PM method in the early 1900s, notably niobium, thorium, and titanium. At the same time, however, another development, originating in refractory metal processing, took form and rapidly grew to such importance that it far overshadows the parent field. Cemented carbides have become one of the greatest industrial developments of the century.

Cemented Carbides. Ordinary drawing dies were unsatisfactory for drawing tungsten wires and filaments. The need for a harder material to withstand greater wear became urgent. Because it was known that tungsten granules combined readily with carbon at high temperatures to give an extremely hard compound, this material was used as the basis for a very hard, durable tool material known as cemented carbide. The tungsten carbide particles, present in the form of finely divided, hard, strong particles, are bonded into a solid body with the aid of a metallic cementing agent. Early experiments with a number of metals established that this cementing agent had to possess the following properties to permit solidification of the hard metal body:

- Close chemical affinity for the carbide particles
- A relatively low melting point
- Limited ability to alloy with the carbide
- Great ductility (not to be impaired by the cementing operation)

Cobalt satisfied these requirements most closely. The early work was carried out mainly in Germany by Lohmann and Voigtländer (Ref 28) in 1914, by Liebmann and Laise (Ref 29) in 1917, and by Schröter (Ref 30) from 1923 to 1925. Krupp (Ref 31) perfected the process in 1927 and marketed the first product of commercial importance, "Widia." In 1928 this material was introduced to the United States, and the General Electric Company, which held the American patent rights, issued a number of licenses. The process entails carefully controlled powder manufacture, compacting a mixture of carbide and metallic binder (usually 3 to 13% Co), and sintering in a protective atmosphere at a temperature high enough to allow fusion of the cobalt and partial alloying with the tungsten carbide. The molten matrix of cobalt and partly dissolved tungsten carbide forms a bond, holding the hard particles together and giving the metallic body sufficient toughness, ductility, and strength to permit its effective use as tool material.

Composite Metals. The next development in PM was the production of composite metals used for heavy-duty contacts, electrodes, counterweights, and radium containers. All of these composite materials contain refractory metal particles, usually tungsten, and a cementing material with a lower melting point, present in various proportions. Copper, copper alloys, and silver are frequently used; cobalt, iron, and nickel are used less frequently. Some combinations also contain graphite. The first attempt to produce such materials was recorded in the patent of Viertel and Egly (Ref 32) issued shortly after 1900. The procedures used either were similar to those developed for the hard metals (Ref 33) or called for introduction of the binder in liquid form by dipping or infiltration. In 1916, Gebauer (Ref 34) developed such a procedure, which was developed further by Baumhauer (Ref 35) and Gillette (Ref 36) in 1924. Pfanstiehl (Ref 37) obtained patent protection in 1919 for a heavy metal, consisting of tungsten and a binder that contained copper and nickel.

Porous Metal Bearings and Filters. In addition to the development of refractory metals and their carbides, another important area of PM that gained attention during the early 1900s was that of porous metal bearings. Special types of these porous bearings are referred to as self-lubricating.

The modern types of bearings, usually made of copper, tin, and graphite powders and impregnated with oil, were first developed in processes patented by Loewendahl (Ref 38) and Gilson (Ref 39 and 40). Gilson's material was a bronze structure in which finely divided graphite inclusions were uniformly distributed. It was produced by mixing copper powder and tin oxides with graphite, compressing the mixture, and heating it to a temperature at which the oxides were reduced by the graphite and the copper and tin could diffuse sufficiently togive a bronzelike structure. Excess graphite (up to 40 vol%) was uniformly distributed through this structure. The porosity was sufficient to allow for the introduction of at least 2% oil. The process was later improved by Boegehold and Williams (Ref 41), Claus (Ref 42), and many others, primarily by utilization of elemental metal powders rather than oxides.

Metallic filters were the next stage in the development of these porous metals, and patents date back as far as 1923 (Ref 43), when Claus patented a process and machine to mold porous bodies from granular powder.

Post-War Developments

Infiltration techniques, porous materials, iron powder cores for radio tuning devices, PM permanent magnets, and W-Cu-Ni heavy metal compositions were developed during the periods between 1900, World War I, and the late 1920s. At the beginning of World War II in Europe, iron powder technology began its advance to commercial viability. The most spectacular development of iron parts made by PM was during World War II in central Europe, where paraffin-impregnated sintered iron driving bands for military projectiles were extensively used. German powder metallurgists found this technique effective as a substitute for scarce gilding metal, a copper-zinc alloy containing 5 to 10% Zn. Production reached a peak of 3175 tonnes (3500 tons) per month for this application.

The advent of mass production in the automotive industry made possible the use of iron and copper powders in large tonnages and spawned many of the technological advances of the modern PM industry. The automobile has been the basis for most industrial applications of PM, even in fields unrelated to the automotive industry. The first commercial application of a PM product, the self-lubricating bearing, was used in an automobile in 1927. It was made from a combination of copper and tin powders to produce a porous bronze bearing capable of retaining oil within its pores by capillary attraction. At about the same time, self-lubricating bearings were introduced to the home appliance market as a refrigerator compressor component.

Through the 1940s and early 1950s, copper powder and the self-lubricating bearing were the principal products of PM. Since then, iron powder and steel PM mechanical components such as gears, cams, and other structural shapes have become dominant. While copper powder remains an important PM material, consumed on the order of 14,500 tonnes (16,000 tons) per year, it is overshadowed by iron and iron-base powders with markets of 345,000 tonnes (380,000 tons) per year.

Since the end of World War II, and especially with the advent of aerospace and nuclear technology, developments have been widespread with regard to the PM of refractory and reactive metals such as tungsten, molybdenum, niobium, titanium, and tantalum and of nuclear metals such as beryllium, uranium, zirconium, and thorium.

All of the refractory metals are recovered from their ores, processed, and formed using PM techniques. With the reactive metals, PM is often used to achieve higher purity or to combine them with other metals or nonmetallics to achieve special properties. Nuclear power plants use fuel elements often made by dispersing uranium oxide in a metal powder (e.g., aluminum) matrix. The control rods and neutron shielding may use boron powder in a matrix of nickel, copper, iron, or aluminum. Tungsten combined with nickel and copper powders is used widely as a shielding component in applications where intricate configuration involving machining is required, such as in cobalt-60 containers. In aerospace, beryllium and titanium are used extensively. Rocket skirts, cones, and heat shields are often formed from niobium. Molybdenum is widely used in missile and rocket engine components. Nozzles for rockets used in orbiting space vehicles often are made from tungsten via the PM process in order to maintain critical dimensional tolerances.

The 1950s and 1960s witnessed the emergence of PM wrought products. These are fully dense metal systems that began as powders. Hot isostatically pressed superalloys, powder forgings, PM tool steels, roll compacted strip, and dispersion-strengthened copper are all examples. Each of these processes and materials is covered in separate articles in this Volume.

The commercialization of powder-based high-performance material emerged as a major breakthrough in metalworking technology in the 1970s by opening up new markets through superior performance coupled with the cost effectiveness of material conservation and longer operational life.

In the late 1970s, the experimental programs involving PM wrought products began spilling over into the commercial industrial sector, principally in the form of PM tool steels and powder forgings. With the advent of powder forgings, no longer were properties compromised by density. Fully dense components capable of combining the alloying flexibility and the net and near-net design features of PM were very marketable. The later 1970s and early 1980s witnessed a significant metallurgical breakthrough in the recognition of PM techniques for eliminating segregation and ensuring a fully homogeneous, fine-grained, pore-free, high-alloy structure. Categorized as PM wrought metals, they led to the perfection of extremely high-purity metal powders and improved consolidation techniques such as hot isostatic pressing (HIP). The 1980s also saw the commercialization of ultra rapid solidification and injection molding technology. Both of these developments are also covered in separate articles in this Volume.

Recent Developments

In the late 1980s and early 1990s, several patents (Ref 44) were issued for additive manufacturing, also known as three-dimensional (3-D) printing. Additive manufacturing is a way of making 3-D objects by building up material, layer upon layer, with the guidance of a digital design. In 2012, as the national accelerator for additive manufacturing and 3-D printing, the National Additive Manufacturing Innovation Institute (NAMII) was founded. It is structured as a public-private partnership with member organizations from industry, academia, government, nongovernment agencies, and workforce and economic development resources and is intended to increase global manufacturing competitiveness.

Commercial PM now spans the density spectrum from highly porous metal filters through self-lubricating bearings and PM parts with controlled density to fully dense PM wrought metal systems. The PM parts and products industry in North America has estimated sales of more than $7 billion (Ref 45). It comprises 150 companies that make conventional PM parts and products from iron- and copper-base powders and approximately 150 companies that make specialty PM products such as superalloys, tool steels, porous products, friction materials, strip for electronic applications, high-strength permanent magnets, magnetic powder cores and ferrites, tungsten carbide cutting tools and wear parts, rapid solidification rate (RSR) products, metal injection molded parts, additive manufactured parts (3-D printed), and tool steels. Powder metallurgy is international in scope with growing industries in all of the major industrialized countries. Annual worldwide metal powder production exceeds 1.36 million tonnes (1.5 million tons).

Trends and new developments include:

- Improved manufacturing processes such as HIP, powder forging, metal injection molding (MIM), and direct powder rolling through increased scientific investigation of PM technology by government, academic, and industrial research and development programs
- Fully dense PM products for improved strength properties and quality in automobiles, diesel and turbine engines, aircraft parts, and industrial cutting and forming tools
- Commercialization of technologies such as MIM, rapid solidification, powder forging, spray forming, high-temperature vacuum sintering, warm compacting, and both cold and hot isostatic pressing
- The use of powder forged connecting rods in automobiles and PM composite camshafts for four- and eight-cylinder automobile engines and the use of PM main bearing caps

A review of major historical developments in PM is presented in Table 1.

Powder Metallurgy Literature

A number of literary works are worthy of mention in connection with the background of PM. One of the earliest works of significance was *Principles of Powder Metallurgy* by W.-D. Jones, published in 1937 in England (Ref 46). It was updated in 1960 and published as *Fundamental Principles of Powder Metallurgy* (Ref 47). The first Russian publication was by Bal'shin (Ref 48) and appeared in 1938; the first comprehensive text in German, *Pulvermetallurgie und Sinterwerkstoffe*, was published by R. Kieffer and W. Hotop in 1943 (Ref 49). In the United States, the first publication was by H.H. Hausner in 1947 (Ref 50), followed closely by P. Schwarzkopf (Ref 51). Two years later, the first of four volumes of a treatise on PM, a major work by C.G. Goetzel (Ref 52), was published. Some current "Selected References" on PM science and technology are listed at the end of this article.

Powder Metallurgy Trade Associations

The advancement of PM from a laboratory curiosity to an industrial technology has been influenced greatly by various professional societies and the PM trade association, whose annual technical conference proceedings chronicle the maturing of the technology. In 1944, an organization called the Metal Powder Association was founded by a group of metal powder producers in the United States. It was reorganized in 1958 as the Metal Powder Industries Federation (MPIF), a trade association whose representation embraced the commercial and technological interests of the total metal powder producing and consuming industries.

International in scope, the Federation consists of the following autonomous associations, which together represent the primary elements of the PM and particulate materials industries:

- *Powder Metallurgy Parts Association:* Members are companies that manufacture PM parts for sale on the open market or captive use.
- *Metal Powder Producers Association:* Members are producers of metal powders in any form for any use.
- *Powder Metallurgy Equipment Association:* Members are manufacturers of PM processing equipment and supplies, including compacting presses, sintering furnaces, belts, tools and dies, and atmospheres.
- *Refractory Metals Association:* Members are manufacturers of powders or products from tungsten, molybdenum, tantalum, niobium, and cobalt.
- *Isostatic Pressing Association (IPA):* Members are companies that use isostatic pressing in the manufacturing processes.
- *Metal Injection Molding Association (MIMA):* Members are international companies that use the metal or ceramic injection molding process to form parts.

The Metal Powder Industries Federation also has both Overseas and Affiliate/Consultant classes of membership.

The Federation generates industry statistics, process and materials standards, industrial public relations and market development, government programs, research, and various educational programs and materials.

The technology's "professional" society is APMI International. As distinguished from the Federation, APMI members are individuals, not companies. Members are kept informed of developments in PM technology through local section activities, conferences, and publications, including the *International Journal of Powder Metallurgy and Powder Technology*.

Many of the major professional societies are also active in PM, usually through committees working on standards, conferences, or publications. This includes ASM International, The Minerals, Metals & Materials Society (TMS), SAE International, ASTM International, and Society of Manufacturing Engineers (SME).

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Powder Metallurgy Methods and Applications

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Powder metallurgy (PM) is the production and utilization of metal powders. Powders are defined as particles that are usually less than 1000 nm (1 mm) in size. Most of the metal particles used in PM are in the range of 5 to 200 μm (0.2 to 7.9 mils). To put this in context, a human hair is typically in the 100 μm (3.9 mils) range.

The history of PM has already been outlined in the article "History of Powder Metallurgy" in this Volume. This article reviews the various segments of the PM process from powder production and powder processing through to the characterization of the materials and their properties. It will cover processing methods for consolidating metal powders including options for processing to full density.

Powders have a high ratio of surface area to volume and this is taken advantage of in the use of metal powders as catalysts or in various chemical and metallurgical reactions. While this article focuses on the use of powders to make functional engineering components, many metal powders are used in their particulate form. This aspect of PM is covered in the article "Specialty Applications of Metal Powders" in this Volume.

Powder technologies are exciting to engineers because processing options permit the selective placement of phases or pores to tailor the component for the application. The capability of press and sinter processing or metal injection molding (MIM) processing to replicate parts in high volumes is very attractive to design engineers. The ability to fabricate complex shapes to final size and shape or to near-net shape is particularly valuable. Powder metallurgy offers the potential to do this in high volumes and also for applications where the volumes are not so large.

The three main reasons for using PM are economic, uniqueness, and captive applications, as shown in Fig. 1 (Ref 1). For some applications that require high volumes of parts with high precision, cost is the overarching factor. A good example of this segment is parts for the automotive industry (where approximately 70% of ferrous PM structural parts are used). Powder metallurgy parts are used in engine, transmission, and chassis applications. Sometimes it is a unique microstructure or property that leads to the use of PM processing: for example, porous filters, self-lubricating bearings, dispersion-strengthened alloys, functionally graded materials (e.g., titanium-hydroxyapatite), and cutting tools from tungsten carbide or diamond composites. Captive applications of PM include materials that are difficult to process by other techniques, such as refractory metals and reactive metals. Other examples in this category are special compounds such as molybdenum disilicide and titanium aluminide, or amorphous metals.

The metal powder industry is a recognized metal forming technology that competes directly with other metalworking practices such as casting, forging, stamping (fine blanking), and screw machining. The industry comprises powder suppliers and parts makers, plus the companies that supply the mixing equipment, powder handling equipment, compacting presses, sintering furnaces, and so forth.

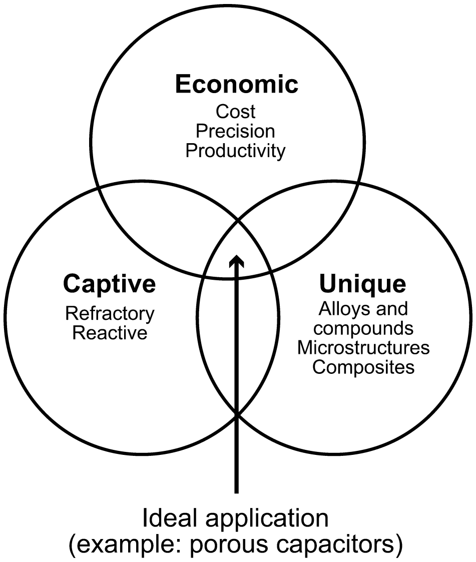


Fig. 1 Three main reasons for choosing powder metallurgy shown in the form of a Venn diagram. The intersection of the three circles represents an ideal area for applying PM techniques. Source: Ref 1

Powder metallurgy processing offers many advantages. The PM process is material and energy efficient compared with other metal forming technologies. Powder metallurgy is cost effective for making complex-shaped parts and minimizes the need for machining. A wide range of engineered materials is available, and through appropriate material and process selection the required microstructure may be developed in the material. Powder metallurgy parts have good surface finish and they may be heat treated to increase strength or wear resistance. The PM process provides part-to-part reproducibility and is suited to moderate-to-high volume production. Where necessary, controlled microporosity can be provided for self-lubrication or filtration. While dimensional precision is good, it typically does not match that of machined parts.

In the case of ferrous PM parts, they have lower ductility and reduced impact resistance compared with wrought steels.

The majority of PM parts are porous and consideration must be given to this when performing finishing operations.

Metal Powders

Metal powders come in many different shapes and sizes (Fig. 2). Their shape, size, and size distribution depend on the manner in which they were produced. Metal powder production is covered in depth in various articles in the Section, "Metal Powder Production" in this Volume.

There are three main methods of powder production:

- Mechanical, including machining, milling, and mechanical alloying
- Chemical, including electrolytic deposition, decomposition of a solid by a gas, thermal

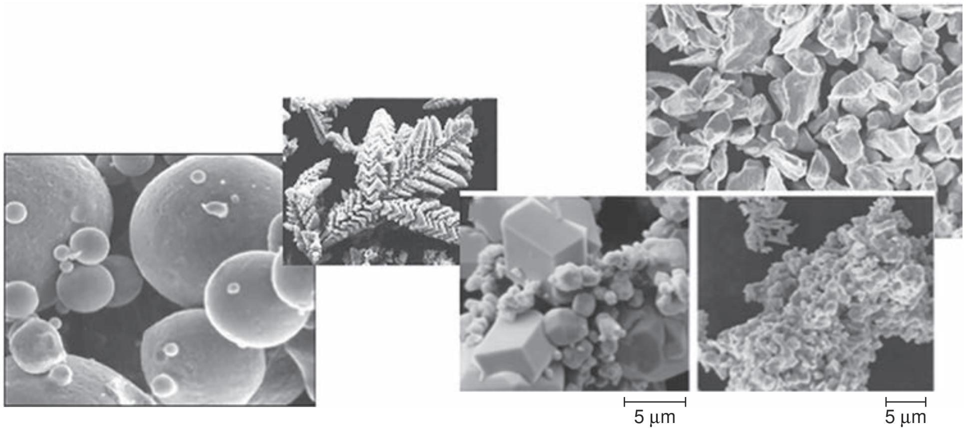


Fig. 2 Example of the different particle shapes possible with metal powders

decomposition, precipitation from a liquid, precipitation from a gas, solid-solid reactive synthesis
- Physical, including atomization techniques

Most metals are available in powder form. Some may be made by many different methods, while for others only a few options are possible. The characteristics of the powder are determined by the method by which it is produced. The shape, size, size distribution, surface area, apparent density, flow, angle of repose, compressibility, and green strength depend on the powder production method. In-depth coverage of the sampling and testing of metal powders is presented in the articles in the Section "Metal Powder Characterization" in this Volume.

Powder Processing

For the production of PM parts in high volumes, compaction is carried out in rigid dies. In most instances, the metallic powders are mixed with a lubricant (e.g., ethylene bisstearamide) to reduce interparticle friction during compaction and to facilitate ejection of the compacted parts by reducing friction at the die-wall and core-rod interfaces.

The metal powders may be elemental powders; mixtures of elemental powders; or mixtures of elemental powders with master alloys or ferroalloys, prealloys, diffusion alloys, or hybrid alloys. See the article "Ferrous Powder Metallurgy Materials" in this Volume for an in-depth review of the alloying methods used in ferrous PM. A consequence of the various alloying methods available is that only the PM materials made from prealloyed powders are chemically homogeneous. The other alloying methods can result in chemically inhomogeneous materials. The hardenability is determined by the local chemical composition, and the resulting microstructures are generally quite complex. Chemical analysis can be a challenge due to the inhomogeneous nature of the materials. Guidelines for sample preparation for the chemical analysis of the metallic elements in PM materials are provided in MPIF Standard 67 (Ref 2).

Complex, multilevel PM parts compacted in rigid dies will not have the same green density throughout. While the objective is generally to achieve a density as uniform as possible throughout the compacted part, taller parts and parts with multiple levels are subject to the presence of density differences between adjacent regions. This is due to frictional effects and compacting tool deflections. Taller parts will have a neutral zone or density line—the region of the compact that has experienced the least relative movement of powder. The position of the neutral zone may be adjusted by varying the pressure exerted by the upper and lower punches.

Compaction in rigid dies is limited to part shapes that can be ejected from the die cavity. Parts with undercuts, reverse tapers, threads, and so forth, are not generally practical. Such features are formed by postsintering machining operations.

There are two main types of compacting press: mechanical and hydraulic. Some hybrid presses offer features of both. A detailed treatment of compaction is provided in the Section "Metal Powder Compaction" in this Volume.

Some PM parts are molded (shaped) rather than compacted. Fine-particle-size metal powders (5 to 20 µm, or 0.2 to 0.8 mils) are mixed with binders and plasticizers and processed to form a feedstock for MIM. Molding is performed using machines similar to those used for plastic injection molding. Shrinkage during the subsequent sintering operation is extensive (15 to 20%) due to the fine-particle-size powders used and the high sintering temperatures. Because the parts are molded and not compacted, they do not contain density gradients that lead to distortion or problems with dimensional control. The process makes complex-shaped, small-to-medium sized PM parts with high relative densities.

Some metal powders are not very compressible. The powder particles are hard and have limited plasticity. Rigid die compaction is not suitable for consolidating such powders, and they must be processed by other means such as hot pressing, extrusion, or hot isostatic pressing (HIP), described subsequently in this article.

Highly reactive metal powders are also not suitable for rigid die compaction. They generally need to be vacuum hot pressed, or encapsulated and extruded, or HIPed.

Rigid die compacted parts and MIM parts are thermally treated to increase their strength in a process known as sintering. The parts are heated, generally in a reducing atmosphere, to a temperature that is below the melting point of the primary constituent of the material, in order to form metallurgical bonds between the compacted metal powder particles. Sintering is a "shrinkage" process. The system tries to reduce its overall surface area via various diffusion processes. Metallurgical bonds (microscopic weldments) form between adjacent metal particles (after oxides have been reduced on the surface of the powder particles), pore surfaces become less irregularly shaped, and larger pores grow at the expense of the smaller pores. Sintering is generally carried out using continuous mesh-belt furnaces. For higher temperatures (>1150 °C, or 2100 °F), pusher, roller hearth, or walking-beam furnaces may be used. Batch furnace processing is used for special applications (e.g., pressure-assisted sintering). More information on sintering may be found in the Section "Sintering Basics" in this Volume.

Powder Metallurgy Material Properties

The majority of PM parts contain pores (see options for processing metal powders to full density later in this article). This is an advantage when metal powders are used to make self-lubricating bearings in which the surface-connected pores of the parts are impregnated with oil. When the bearing surface heats up due to frictional heat, oil is released from the pores. When the bearing cools, the oil is sucked back into the pore channels by capillary action. The porosity in PM parts has an effect on the physical, mechanical, magnetic, thermal, wear, and corrosion properties of the parts.

Thermophysical properties of sintered steels, in particular their coefficient of thermal expansion and their thermal conductivity, are needed when designing parts and when modeling heat treatment processes. Opinions differ in the PM community as to the effect of density on these properties. Danninger has shown, however, that the coefficient of thermal expansion up to 1000 °C (1832 °F), measured through dilatometry, is virtually independent of porosity (density) over a density range from 5.97 to 7.53 g/cm^3 (Ref 3). In addition, thermal conductivity was determined in the same temperature range by using laser flash to measure thermal diffusivity, and specific heat, and then the thermal conductivity was calculated from these parameters and the density in accordance with:

$$a = \lambda/(\rho C_p) \qquad \text{(Eq 1)}$$

where a is thermal diffusivity, λ is thermal conductivity, ρ is density, and C_p is specific heat at constant pressure.

Thermal conductivity was shown to depend on density. The effect of porosity in the technically relevant density range was, however, slightly less pronounced than the effect exerted by the alloying elements; specifically, the variation observed between different standard PM steel grades in the low-to-medium temperature range. Both thermophysical properties are, therefore, significantly less influenced by porosity than by chemical composition. Powder metallurgy steels are more similar to wrought steels than was generally assumed.

The elastic constants are also of interest to the design engineer. Young's modulus, Poisson's ratio, and the shear modulus are related according to:

$$E = 2G(1 + \nu) \qquad \text{(Eq 2)}$$

where E is Young's modulus, G is shear modulus, and ν is Poisson's ratio. E and ν are determined by resonant frequency and G is calculated from Eq 2.

Beiss (Ref 4) has shown that:

$$E = E_0(\rho/\rho_0)^m \qquad \text{(Eq 3)}$$

where E_0 is the Young's modulus of the pore-free material, ρ is the density of the material, ρ_0 is the density of the pore-free material, and the exponent m depends on the pore morphology and varies between 2.5 and 4.5. Nevertheless, over the density range of interest for ferrous PM structural materials, 6.4 to 7.4 g/cm^3, Young's modulus is essentially a linear function of density (Fig. 3) (Ref 5).

Poisson's ratio is a weak function of density, and for ferrous PM structural steels it can be taken as 0.27 ± 0.02.

The mechanical properties of PM materials are a function of density:

$$P/P_0 = (\rho/\rho_0)^m \qquad \text{(Eq 4)}$$

where, P is the property of interest, P_0 the value for the pore-free material, ρ is the density of the material, ρ_0 is the density of the pore-free material, and m is an exponent the value of which depends on a given property (Fig. 4) (Ref 6–7). While tensile strength increases in a linear fashion as density increases, tensile ductility is more dependent on reducing the level of porosity. Fatigue performance is even more influenced by density with an exponent m of between 3.5 and 4.5. Impact energy is the most dependent on density, with an exponent m of approximately 12.

Magnetic properties of ferrous PM materials are affected by density. Induction and permeability increase as the density is increased. Permeability and coercive field strength are structure-sensitive properties that are degraded by the presence of impurities. The sintering conditions are extremely important to keep carbon, nitrogen, and oxygen contents to low levels (C = 0.03 wt% max; N = 0.01 wt% max; and O = 0.10 wt% max). Residual stresses from operations such as sizing, machining, or shot peening degrade the magnetic properties. The properties can be restored through an annealing treatment.

Processing Options to Consolidate Metal Powders

There are three basic approaches to the consolidation of metal powders, as shown in Fig. 5 (Ref 8).

Pressure-based compaction establishes density via the compaction process then sinters to develop strength in the compacts. Rigid die compaction falls into this category and is the most cost-effective method for the high-volume production of PM structural parts. In order for this method to be viable, the metal powders need an irregular shape and good flow characteristics, they must be compressible, and they must have good "green" strength. (*Green* is the term used to describe an as-pressed compact.) Extremely hard particles with a spherical shape are not appropriate for use in rigid die compaction. Compaction takes place at high pressure in confined dies (the dies are generally made from cold work tool steel or cemented carbide). Compacting pressures for ferrous powders are generally in the range from 400 to 700 MPa (60 to 100 ksi), from 100 to 400 MPa (14.5 to 60 ksi) for aluminum and aluminum alloy powders, and approximately 400 MPa (60 ksi) for copper and copper-alloy powders.

The green density increases as the compacting pressure is increased and levels out at higher compacting pressures. Powder particles work harden as the result of plastic deformation and it requires higher pressures to cause further plastic flow. In addition, the lubricant that is typically admixed to aid particle rearrangement and to reduce the fictional forces between the powder and the compacting tools eventually has no place to go because all the voids between particles have been closed—either by metal flow or by the presence of lubricant. More lubricant is beneficial at lower compacting pressures, but there is a transition point at which the additional lubricant impedes further densification (Fig. 6) (Ref 9).

Warm compaction processing was developed to overcome the compressibility constraints of rigid-die compaction (Ref 10). The powder mixture and the compacting tools are heated

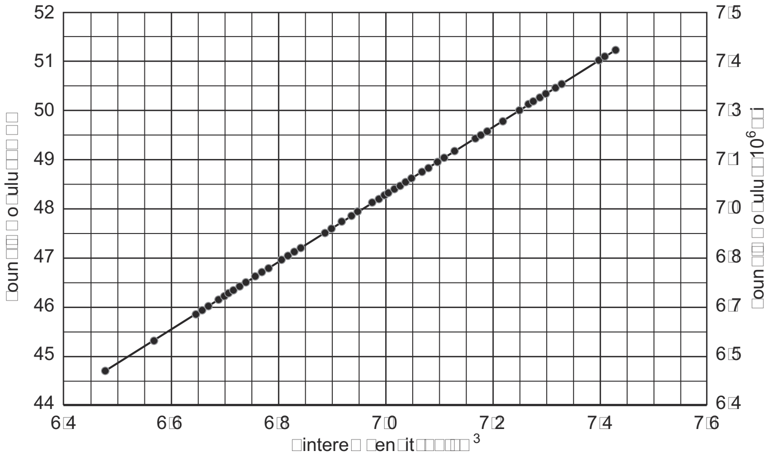
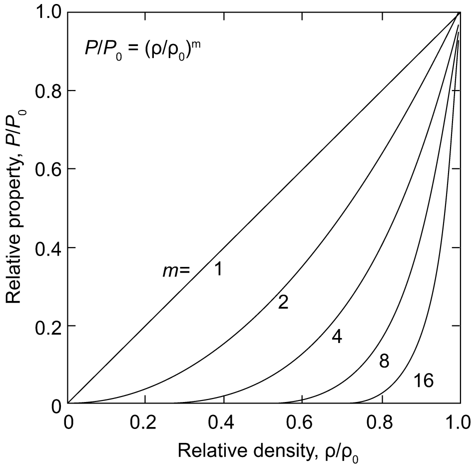


Fig. 3 Young's modulus as a function of sintered density. Data from Ref 5



Thermal conductivity	1.5 ≤ m ≤ 3.5
Elastic moduli	2.5 ≤ m ≤ 4.5
Fatigue strength	3.5 ≤ m ≤ 5.5
Unnotched impact energy	m > 12

Fig. 4 Effect of density on mechanical and physical properties of PM materials. Source: Ref 6

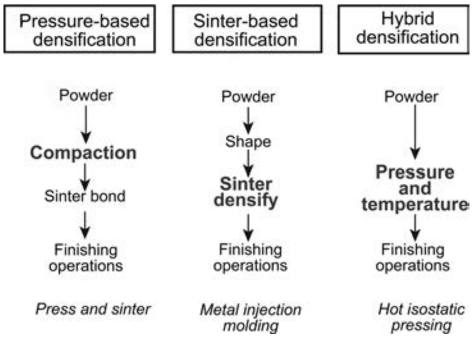


Fig. 5 Three basic approaches to the consolidation of metal powders. Source: Ref 8

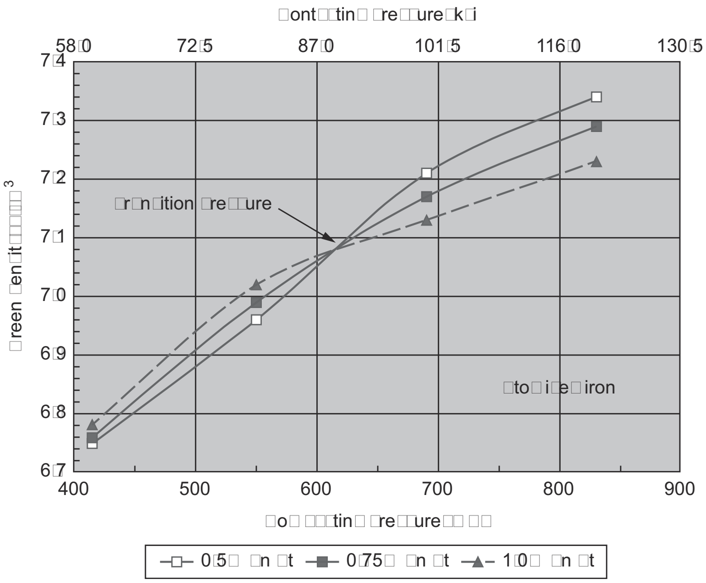


Fig. 6 Effect of lubricant content on the compressibility of metal powders. Source: Ref 9

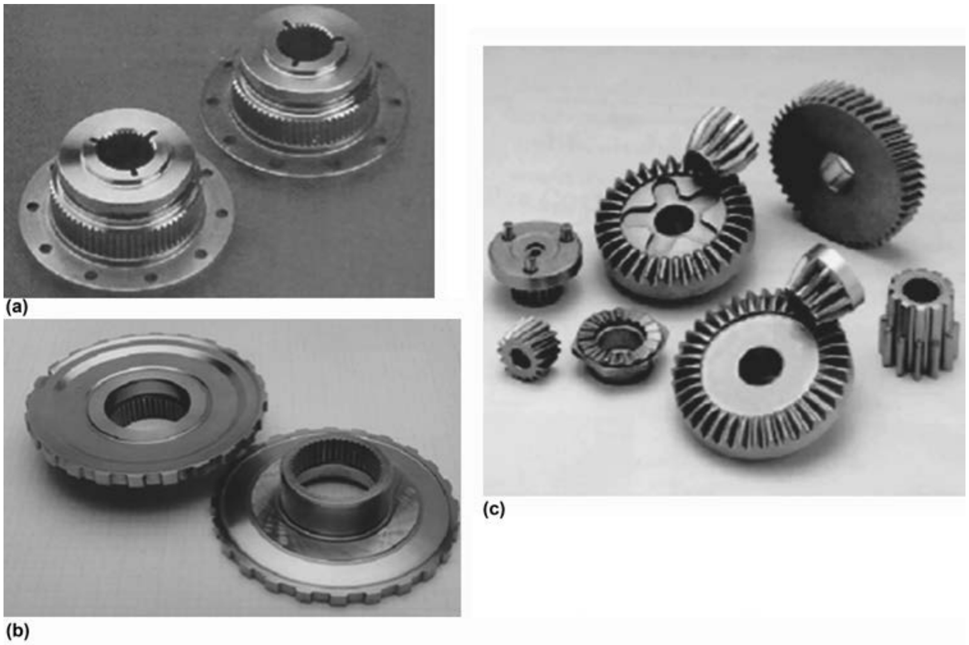


Fig. 7 Examples of warm compacted PM parts. (a) Torque converter hub. Courtesy of Chicago Powder Metal Products. (b) Transmission output shaft hub. Courtesy of GKN Sinter Metals. (c) Hand tool parts. Courtesy of PoriteTaiwan Co. Ltd.

to approximately 120 °C (250 °F) and the powder is compacted in a single press stroke. Green densities of up to 7.3 g/cm^3 are possible with highly compressible ferrous powders. This is approximately 98% of the pore-free density of the powder mixture being compacted (The pore-free density of the powder mixture is the green density that could be reached if all the porosity was removed from the material. It can be calculated for any mixture based on the density and the amount of each constituent in the mixture and the volume they would occupy in the pore-free condition.) Special lubricants and binder-treated premixes were developed for warm compaction. The efficiency of the special lubricant enabled it to be reduced to 0.6 wt% from the 0.8 wt% more typically used for rigid-die compaction: the pore-free density increases by 0.1 g/cm^3 for each 0.2 wt% reduction in the amount of the admixed lubricant. Examples of warm compacted parts are shown in Fig. 7.

More recently, warm-die compaction has been introduced. In this instance, only the compacting tooling is heated (the powder is not heated). The optimal die temperature varies according to the specific lubrication system being used. The die temperature is set so that the surface temperature of the green compacts reaches the desired range for the lubricant system in question. Warm-die compaction is ideal for small-to-medium size parts that weigh less than 700 g (1.5 lb), are up to 32 mm (1.3 in.) high, and have wall thicknesses of up to 19 mm (0.75 in.). For larger parts warm compaction processing is required. Green densities of 7.45 g/cm^3 have been reached using warm-die compaction with lubricant additions of approximately 0.3 wt% (Ref 11).

In sintering-based densification, the shape of the component is formed in a molding operation (e.g., MIM) and sintering is enhanced by the use of high temperatures and fine-particle-size powders. While extensive shrinkage occurs during sintering, it is essentially isotropic in nature so that good tolerance can still be achieved. Metal powder loading in the feedstock used for MIM is approximately 60%. The binders and plasticizers added to make the mixture moldable must be removed prior to final sintering. This "debinding" step is the rate-controlling phase of the MIM process. Other sintering-based densification processes that involve the molding or shaping of powders are slip casting and tape casting.

Hybrid Densification. For some materials, a hybrid densification process is used in which pressure and temperature are applied at the same time. As mentioned previously, some powders are not suitable for rigid-die compaction; they are too hard, are spherical in shape, or are too reactive. In this instance, processes such as powder extrusion (typically after encapsulation) or HIP are used. Hot pressing (often in vacuum) or spark sintering may also be used.

Processing to Full Density

Options for processing metal powders to full density are mapped in Fig. 8 (Ref 12). The vertical axis relates to relative stress (the applied pressure divided by the in situ yield strength of the material) and the horizontal axis relative temperature (based on the melting temperature of the material).

Full-density processing requires the simultaneous application of pressure and temperature. The approach works because most materials soften as temperature is increased. They also become more ductile and deform with less work hardening.

The processing options fall into the following categories:

- Low-stress processes that operate at high temperatures and are dominated by diffusion processes (e.g., liquid-phase sintering)
- Processes that apply intermediate stress levels and operate at intermediate temperatures and rely on diffusional creep processes (e.g., hot pressing or HIP)
- High-stress routes that operate at high strain rates and lower temperatures (powder forging or extrusion)
- Routes that achieve high density via the application of ultrahigh-stress at ambient temperature (explosive compaction)

Liquid-phase sintering results in a composite microstructure that consists of a skeleton of a high-melting-temperature phase in a matrix of a solidified liquid—for example, W-Ni-Fe heavy alloys, WC-Co cemented carbides.

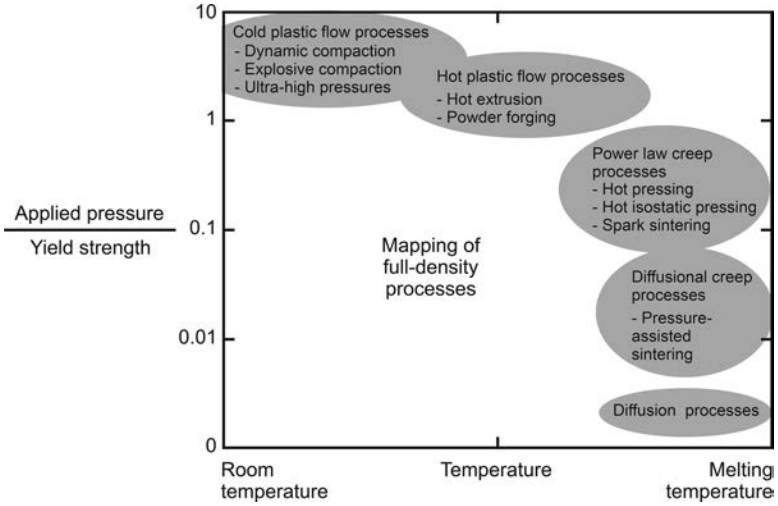


Fig. 8 Options for processing metal powders to full density. Source: Ref 12

Hot pressing is performed in a rigid die using uniaxially applied pressure: it is a low-strain-rate process. Graphite dies may be used, in which case induction heating may be employed. Hot pressing cycle times are slow compared with rigid-die compaction. Vacuum is sometimes used to minimize contamination of the compact. Diamond-metal-composite cutting tools are often hot pressed. Spark sintering is a process related to hot pressing. In spark sintering, direct resistance heating is applied to the punches, die, and powder mass during consolidation.

Hot isostatic pressing applies pressure from all directions simultaneously. In order to establish a pressure differential, powders must be processed to the point where they have no surface-connected, interconnected porosity, or they need to be encapsulated prior to the HIP process. Prior to HIP, a container is filled with powder and heated under vacuum to remove volatile contaminants. After evacuation and degassing, the container is sealed. The container may be fabricated from any material that is soft and deformable at the consolidation temperature, for example, glass, steel or stainless steel (the choice depends on compatibility with the powder that is being compacted). A HIP vessel is illustrated in Fig. 9 and the sequence used to make a HIPed part is shown in Fig. 10 (Ref 13).

Vacuum sintering then backfilling the sintering furnace with pressurized gas to assist final densification is employed in sinter-HIP processing (a pressure-assisted sintering process). A typical cycle is shown schematically in Fig. 11 (Ref 14).

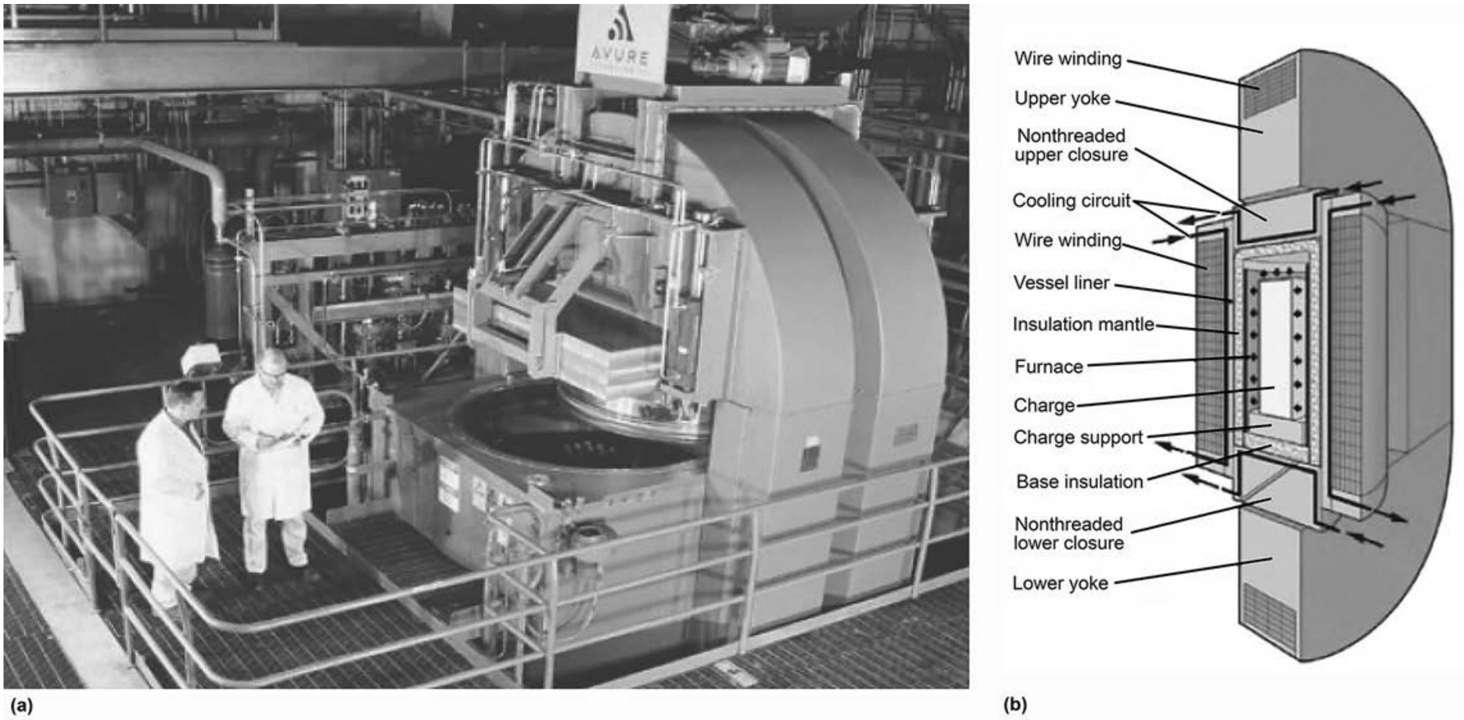


Fig. 9 (a) Typical hot isostatic pressing (HIP) vessel. (b) Schematic of the wire-wound unit. Courtesy of Avure Technologies. Source: Ref 13