POWDER METALLURGY

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Although it may be said that, in one sense, powder metallurgy is a comparatively modern subject, there is plenty of evidence to show that it had been practised for centuries before the art of melting and casting of metals had been mastered by man. Powder metallurgy may be defined as the art of producing objects from metal powders with or without the addition of non-metallic constituents. The powders are usually pressed to a desired form and may be simultaneously or subsequently heated to yield a welded, alloyed, or coalesced mass. The metal parts are described as being " moulded from metal powders by pressing and sintering under various conditions ". Sintering may be defined as the knitting together of small particles to form larger particles, cakes, or masses ; in the case of metal powders it comprises the welding of the particles by heat and pressure.

Furnaces that would melt pure iron were not available until the nineteenth century, yet iron was produced and used at least as early as 3,000 B.C. This was prepared by the reduction of pure iron oxide ore, yielding a sponge composed of small particles of metallic iron, which was then consolidated by forging at a high temperature. This process was practised in India, where amongst other examples the famous Delhi Pillar, made 1,600 years ago and weighing 6½ tons, remains as a monument to those early smiths. The commercial manufacture of metal powders did not begin until the nineteenth century and Sir Henry Bessemer was the founder of the industrial manufacture of a brass powder for use in " Gold " or " Bronze " paints. The manufacture of malleable platinum by powder metallurgy was begun in Britain early in the nineteenth century but, apart from these two exceptions, no fundamental contribution or invention has been made in powder metallugy by British workers until recent years.

It has not been possible to ascertain with certainty when atomized powders were first made commercially in Great Britain. It is probable that low melting point metals such as tin and lead were produced in this way before 1914 and the manufacture of atomized copper and brass was begun about 1925. Evidence concerning the electrolysis-produced powders is very incomplete and all that can be said with certainty is that full commercial production was under way in 1939-40. Tungsten was first made commercially in Great Britain by powder metallurgy about the middle of 1909 by methods evolved by American workers and the two best known powder metallurgy developments, the tungsten carbide tool and the porous bronze bushing, were foreign inventions, the former having been brought from Germany into England about 1930 and the latter from America at about the same time or slightly earlier. The production of powder metallurgy parts in Great Britain is very small in comparison with either the present American production or with the German production during the second World War. There is, however, considerable interest in the subject and a number of firms and companies in Great Britain are manufacturing small components by this process. Evidence of the interest shown in this subject in recent years is shown by the symposia held by the American Society of Testing Materials in 1943 and by the Iron and Steel Institute in 1947.

The chief advantages of this technique are that it is possible to produce articles having unique properties unobtainable by other methods, for example, controlled porosity ; it offers a method of producing parts in metals that cannot be melted commercially or only with considerable difficulty and do not lend themselves to casting. It is suitable for mixtures of metals and non-metals ; it can be used for metals which do not alloy ; it can be applied to metals of widely separated melting points or greatly different densities. It offers mass production methods of producing large quantities of parts identical in size and quality, often of complex shape, without further operations ; and is specially competitive where final machining constitutes a large proportion of the final costs by conventional methods.

At the same time the limitations of the process must be realized. Only in a very few applications can the metal powder be heat treated to form a usable product without compression and the high pressures normally required establish a very real limitation to the process. Special moulds and heavy presses are required which increase in size rapidly as the size of the component to be made increases. By the same token, the depth of the component which can be tackled is dependent on the pressure available and some metal powders have a tendency to stick to the die walls and thus aggravate the depth limitation. The large surface area of the powders presented to the atmosphere sometimes gives rise to difficulties due to oxidation and due to the presence of included moisture, to overcome which atmosphere control may be necessary throughout the whole process.

PRODUCTION OF FERROUS AND NON-FERROUS METAL POWDERS

Metal powders are produced in many ways each giving a powder with its own particular characteristics of density, particle shape, and size distribution.

The shape may be angular, spherical, dendritic or flattened and the particles themselves may be dense or spongy.

The manufacturer's task is to select the method of production which will give a powder having a combination of characteristics precisely suited to the purpose for which it is to be used. Every method of powder production is not suited to all metals, for example, methods involving melting are not so readily applicable to metals of high melting point. The main methods of production of metal powders may be grouped as follows :—

(1) Milling
(2) Machining
(3) Atomization
(4) Granulation or Shotting
(5) Graining
(6) Reduction of metallic oxides
(7) Reduction of metallic salts
(8) Electrolysis
(9) Carbonyl process
(10) Hydride process
(11) Miscellaneous.

Milling

While brittle metals are more readily converted to powder by milling, it is possible with special mills to disintegrate even malleable metals. The mills used are of several types which include ball mills, stamp mills, impact mills, eddy mills and disc crushers. In Germany large quantities of a relatively fine iron powder were produced by the Hametag process, in which steel containing 0.5% of manganese (added to improve the drawing properties) is drawn into wire and cut into small pieces and milled.

Machining

The machining process consists merely of cutting massive metal into small pieces by the use of a lathe or other machine tool. This method, though one of the simplest means of producing metal powders, is not used a great deal as it is expensive. Machining operations may be followed by milling which reduces the coarse cuttings to powder. This process was used by Bessemer to produce imitation gold powders.

Atomization

The production of metal powders by atomization is probably one of the cheapest methods. It is used extensively in the production of powders of the lower melting point metals, such as aluminium, tin, zinc, lead and solder, and it appears to have been used to a great extent in Germany in the manufacture of iron powders.

The simplest form of atomizing equipment comprises a crucible containing the molten metal which passes, by gravity, through an orifice. The stream of metal is disintegrated by high pressure air, steam, or water impinging on it. Another less conventional atomizing process employed in Germany was the Disc process. Molten metal such as steel, cast iron, copper, brass and bronze, is poured in a thin stream vertically downwards through an annular jet from which issues water directed downwards at high pressure. The metal is broken into globules which are then rapidly swept out of the atomizing zone by knife blades set in a horizontal disc rotating at a very high speed. The globules of metal are in a plastic state when struck by the blades and the final metal particles are similar to solder splashes, that is, irregular and flattened.

Granulation or Shotting

Some metals, such as aluminium, zinc and tin, can be broken down into rather coarse particles by pouring the molten metal through a vibrating screen. The globules of metal solidify while falling through the air and are finally collected in water, which prevents the particles sticking together.

Graining

If certain metals such as aluminium and tin are stirred or vibrated while cooling from the molten to the solid state they form a mush of small particles of metal owing to oxidation of the metal surfaces which prevents the particles coalescing. The particles are irregular in shape and contain appreciable amounts of oxide and are not suitable for powder metallurgy purposes.

Reduction of Metallic Oxides

In all the methods described so far the powders are coarse but by the reduction of metallic oxides really fine powders can be obtained, which are well beyond the scope of the finest screen to separate. In fact, the powders are so fine that special methods are necessary for determining the distribution of the particle size. It is possible by this method to obtain iron, cobalt, copper, nickel, tungsten, and molybdenum metal powders. One of the cheapest iron powders is obtained by the reduction of good iron ore with carbon. Cobalt, copper and nickel oxides are readily reduced by hydrogen, and by careful control consistent properties are obtained in the powders.

The reduction of the oxides to obtain metal powders is probably the commonest method used and it can, with proper control, give very excellent and consistent material.

Reduction of Metallic Salts

Some metals do not permit of their reduction from the oxide by hydrogen. Tantalum is probably the most interesting of these metals and it is becoming increasingly important. The metal can be produced by electrolysis of fused potassium tantalum fluoride with metallic sodium and reduced to tantalum metal. Iron powder of a high degree of purity may be obtained by the reduction of ferrous chloride by hydrogen. Zirconium is another metal produced as a powder when its chloride is passed over heated magnesium.

Electrolysis

Many metal powders are produced by electrolysis and the method is often preferred because it can yield very high purity powders practically free from some of the usual impurities present in the products of other methods, such, for example, as reduction of the oxides.

Depending on the conditions of their formation, electrodeposits of metals can vary quite markedly in their appearance and texture. Usually the powders produced by the electrolytic process are dense and have a characteristic dendritic or tree-like appearance. Normally the powders are not used directly as they are rather brittle and coarse, but are milled and annealed at relatively low temperatures in hydrogen.

Carbonyls

Carbon monoxide readily enters into combination with several metals to form metallic carbonyls with varying physical properties. Of particular interest is nickel carbonyl, obtained as a low boiling point liquid by the interaction of carbon monoxide and nickel under atmospheric pressure at temperatures between 30° and 50°C. Nickel carbonyl is readily dissociated by heating above its boiling point and, depending on the condition of decomposition, metallic nickel can be obtained as a very fine powder or as shot, built up from deposited metal. The metal powder so obtained is pure and is widely used in powder metallurgy. Iron powder can also be obtained by a similar process, is of very high purity and very finely divided.

Hydride Process

A number of metals such as titanium, tantalum, niobium, zirconium, and vanadium form hydrides which can be used directly as metal powder additions, or, if necessary, decomposed by heating in a vacuum to form metal powder and hydrogen. The metal hydrides are very fine and similar in appearance to the metal powders, but are much less subject to oxidation during storage.

Miscellaneous

There are a number of other methods of producing metal powders, such as chemical precipitation ; for example, copper is deposited from its sulphate solution by the addition of iron. Powders produced by this process are very fine.

Porous nickel powder can be prepared by producing an alloy of nickel and aluminium by melting and digesting this alloy in caustic soda to dissolve the aluminium, leaving behind spongy particles of the metal. Zinc powder is sometimes produced by the controlled condensation of zinc vapours produced during the reduction of zinc oxide with carbon. The metal powder produced in this manner has a very small particle size which makes it eminently suitable for use in metal spray guns for the deposition of metal coatings on steel.

SIZE AND SHAPE OF METAL POWDERS

The size and shape of particles is extremely important in powder metallurgy. If a finished product is required to be as dense as possible, that is, as near to the specific gravity of the sound cast alloy as possible, then the density of the powder in the mould should be high. This can be obtained in two ways ; the particles can be of uniform size and very finely divided in which case the resistance of such an agglomeration to pressure is high, thus limiting the depth to which moulding pressure is effective ; or the particles can be of different sizes and shapes. The densest compacts are made by mixing coarse, fine, flake and sponge particles which enable the pressure to be effective to the maximum depth and allow the maximum surface area of contact between particles which assist sintering. A somewhat similar analogy can be drawn from the moulding sands of foundry practice. Here all grades are necessary to form a sand with satisfactory " green " strength and permeability, from coarse sand to clay grade (or silt), the more uniform the sand grade the higher the permeability will be. In porous metal powder products, however, the converse of this condition is required and for such compacts particles are chosen which will leave interstices having the maximum total volume. The particles will be as nearly spheroidal as possible.

In making up alloys there is often a choice of powder types of the various metals, which will be most suited to make the best compact. It is for this reason that one metal may be made commercially by many different processes which may produce fine, coarse, flake, sponge, etc., according to the requirements of the compacting and sintering processes.

Property	Type of Powder						
	Electrolytic		Oxide-Reduced		Carbonyl	Milled	Chloride-Reduced
	Range	Average	Range	Average			
Particle density, g./c.c.	7.45–7.89	7.77	7.26–7.86	7.60	7.84	7.60	—
Particle porosity, %	0.78–3.81	2.13	0.19–3.31	1.97	0.03	1.57	—
Apparent density, g./c.c.	2.05–3.37	2.77	0.97–3.03	2.17	3.40	2.49	—
Specific surface, sq. cm./g.	265–1149	534	448–5161	1036	3459	585	—
Density, g./c.c., pressed at 30 tons/sq. in.	4.54–6.32	5.80	5.15–5.84	5.49	5.69	5.79	—
Porosity, %, pressed at 30 tons/sq. in.	19.76–42.36	26.22	25.83–34.48	30.27	27.73	26.44	—
Compression ratio	1.34–2.94	2.18	1.93–5.31	2.71	1.67	2.33	—
Iron content, %	98.54–99–64	99.22	96.70–98–69	98.01	99.53	97.89	99.17
Oxygen, unsintered, %	0.10–1.23	0.55	0.71–2.09	1.16	0.26	1.47	—
Oxygen, sintered, %	0.06–0.19	0.11	0.29–1.18	0.56	0.05	0.48	—
Hydrogen, unsintered, %	0.001–0.025	0.006	0.001–0.007	0.004	0.004	0.005	—
Hydrogen, sintered, %	0.002–0.016	0.009	0.003–0.089	0.047	0.008	0.031	—
Nitrogen, unsintered, %	0.01–0.02	0.014	0.01–0.07	0.03	0.03	0.02	—
Nitrogen, sintered, %	0.01–0.02	0.014	0.01–0.05	0.018	0.01	0.02	—
Ultimate tensile strength, tons/sq. in.	4.09–10.31	7.1	4.19–20.07	7.65	8.08	5.85	11.50
Yield point, tons/sq. in.	4.40–7.30	6.12	2.0 –13.1	6.57	7.68	—	—
Elongation, %	0–8.3	5.04	0.0 –7.15	2.86	3.35	1.1	2.2
Hardness (Vickers diamond)	22.5 –61.2	34.5	26.9 –113.0	42.0	49.8	32.3	39.5
Density, g./c.c., sintered	4.60–6.42	5.81	5.24–7.05	5.80	5.98	5.79	6.38
Porosity, %, sintered	18.43–41.55	26.20	10.42–33.42	26.31	24.02	26.43	18.94
Shrinkage, % : Length	0.00–5.99	1.08	0.55–10.10	1.88	1.87	0.57	0.98
Breadth	0.13–7.06	1.25	0.55–10.30	1.89	2.22	0.72	0.00
Height	—2.13–7.36	1.60	0.20–9.28	2.19	1.54	0.91	0.70
Volume	—1.56–18.37	3.80	1.66–27.00	5.65	5.52	2.22	1.20

TABLE I

The sizes are usually denoted by the mesh of the sieve through which they will not pass in the process of screen classification adopted for powders. They vary considerably from 3 microns (0.003 mm or 0.00012 in) in diameter for iron carbonyl powder up to 6 mesh (0.132 in).

Some details of physical properties of iron powders made by different processes and of compacts made from them are given in Table I on page 158.

METAL POWDER PRODUCTS

A very wide variety of metal and alloy powders are now compacted to form a large number of engineering products.

Some of these products are made in this way because there is no other suitable method of making alloys from the constituents. In this category are the refractory metals such as tungsten, molybdenum and tantalum the high melting points of which preclude the economic use of melting and casting, and the superhard materials such as cemented carbides with which the desired structure is obtained only by powder metallurgy techniques. Bearings and other porous metal parts also can only be made conveniently by this process where oil impregnation and admixture of non-metallic substances are possibilities. Certain magnets and electrical parts have properties which can more conveniently be obtained by manufacturing them from powdered metal compacts than any other way. Owing to the tolerances to which the finished products can be made, many small components such as gears and complicated shapes are more economically produced by compacting in spite of their inferior properties compared with their wrought and machined counterparts. Other items such as bi-metals for thermostats or clad metals can be made by special powder techniques.

During the war many new applications were found, and the Germans did much development work and research to produce shell driving bands in pure iron by powder metallurgy. Initially this work was put in hand to circumvent the shortage of copper which was hitherto universally used for this purpose, but when the iron compact driving bands were perfected, they were found to be superior to copper in many ways. It was found that a driving band made from powdered iron gave a life of 10,000 to 12,000 rounds compared with 8,000-9,000 with copper bands. No undesirable deposit was left in the barrel and the iron band could function at much higher muzzle velocities.

Some of the more common uses to which powder metallurgy techniques are applied are given briefly under the following headings.

Cemented Carbides

A very important field of application of metal powders is the production of cemented carbides for tool tipping. These are made by sintering together usually tungsten carbide and cobalt, but other alloys made by mixing these with titanium and tantalum carbides are also widely used. The essential feature of these materials is that the powdered carbides which are extremely hard are held in a matrix of cobalt and present, after sintering, an excellent wear resistant surface which can be ground to a suitable tool profile.

The resultant alloy after pressing is not sufficiently strong itself to be capable of withstanding the forces which are applied to a tool, so that the compact is moulded into a shape which can be attached to the face of ordinary plain carbon tool steel, usually by brazing. The carbide tool tip has only then to transmit compressive stresses to the shank of the tool. A photomicrograph at × 1,500 magnification of a typical cemented carbide is given in Fig. 1.

FIG 1

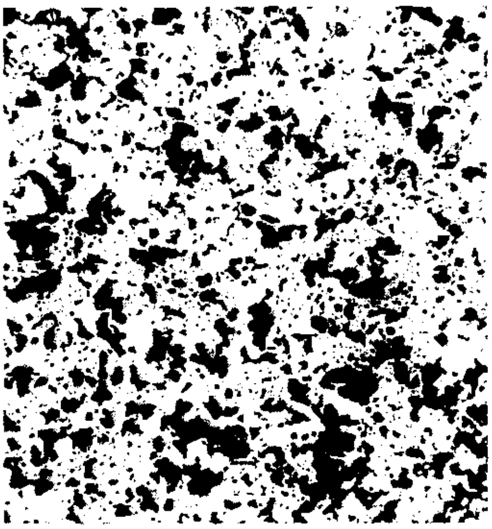

FIG 2

The German hard metal industry evolved rigorous control over the production of powdered carbides and the composition of the mix to be used for tool tips. They also tried substitutes for their dwindling supplies of tungsten during the war; although molybdenum carbide was the best tried, the tips made from this material were much more brittle than those based on tungsten carbide.

Porous Metal Components

It is easy to visualize that by powder metallurgy methods components could be made porous. The process of compacting can be so adjusted that voids remain in the finished article between the grains of the powder.

The type and amount of porosity depends very largely upon the application for which the component is to be used. Foremost of the applications in this field are the bearing materials. Porous bronze self lubricating bearings have been in use for a considerable time and have proved satisfactory in cases where bearing load is not too high ; a maximum static load of approximately 7,500 lb/sq in is usually recommended. They are normally produced by sintering compacts made from a mixture of copper, tin and graphite powders ; in some instances a volatile compound is added to the powder to increase the " green " strength, that is the strength of the pressed powder before sintering. After sintering, the bearings are impregnated with oil by quenching them from the sintering furnace in oil, by soaking in warm oil, or by impregnation under reduced pressure. The composition of these bearings usually corresponds to a graphited 10% tin bronze but more complex alloys containing zinc, lead and iron have been made.

The size and type of the voids in porous bearings is of great importance. It is generally agreed that comparatively large holes interconnected by capilliaries are necessary for maximum lubricant retention but obviously their size is limited by strength requirements. However, once a porous compact has been impregnated it is extremely difficult to get the oil out again, and for analytical purposes it is necessary to extract six or seven times with hot carbon tetrachloride to effect its removal. These lubricant filled voids may be as much as 0.010 in. in diameter but their average size in a loose compact of this sort is approximately 0.003 in. in diameter. Such material has a specific gravity of 6.5 compared with a solid compact of 7.9 and a cast 10% tin bronze of 8.8 specific

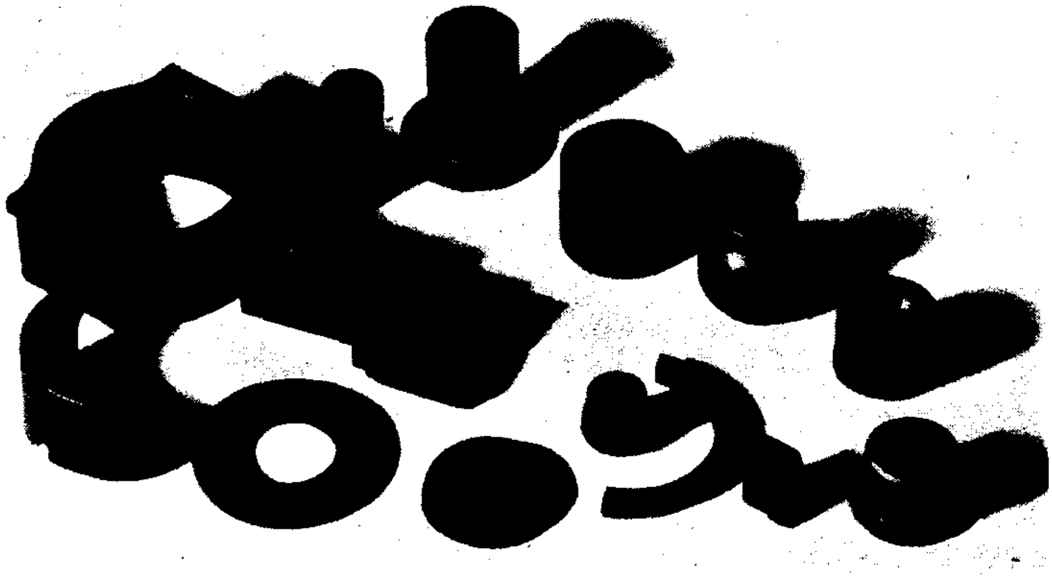

FIG 3

gravity. A photomicrograph of a section of a typical porous material is given in Fig. 2 at × 20 magnification.

It is also possible to manufacture highly porous compacts which can be used as filters. To obtain the best component of this type, two factors concerning the powder itself must be controlled : the grain size must be uniform and the grains must approach the spherical as nearly as possible. These factors will ensure that the maximum percentage of the volume remains as voids and in practice about 37% of the total volume is available for the passage of the fluid to be filtered.

Although the chief application for these metallic honeycombs is for filtering liquids like oil-engine fuel, petrol and water, they can be used for mixing gases and for dampers for pressure gauges. A copper-nickel-tin alloy compact made from powders of the three metals is formed into strips for use as a distributor of the de-icing fluid along the leading edges of aircraft wings. This use of porous metal has contributed greatly to the success of this type of de-icing, because of the uniform distribution afforded thereby.

Magnetic Materials

We now pass from materials having permeability to gases to those of high magnetic permeability. The intrinsic properties of composition, purity and uniform particle size of iron powder, made by the carbonyl process, led to the development of compacts made from this material which have high magnetic permeability, that is, they are capable of forming the cores of powerful electromagnets. Alloys of 80% nickel and 20% iron are amongst those which have the highest known magnetic permeability of any materials and, since both these metals can conveniently be produced in powder form by the carbonyl process, cores of magnets for low frequencies made from these powders give very low core losses ; they are used largely in telephone transmission. The pure iron powder compacts are employed for high frequency uses.

At the other end of the magnetic scale, there are the magnetically hard materials, that is to say, those which will make good permanent magnets. The best of these alloys are the " Alnico " and " Alcomax " types which are alloys of aluminium, nickel, cobalt and iron and have a coercive force of from 2 to 4 times that of the best cobalt steel magnets. They are at the same time extremely hard, brittle and unmachinable as a result of which, if they are made by conventional methods, they can only be cast roughly to shape and ground. Powder metallurgy has helped enormously in the production of the desired shape which requires no machining. Many different methods were tried initially to obtain a satisfactory technique but the presence of aluminium which oxidizes rapidly at 600°C was largely responsible for failure to produce compacts from the individual elements of the alloy. Now a master alloy of aluminium and cobalt is used in powder form together with powders of the other constituents in carefully controlled particle sizes and shapes to form the most homogeneous mixture possible. Sintering operations are carried out in a controlled atmosphere and a finished product of high density is obtained.

Conclusion

It must be realized that a component made by sintered and compressed powdered metal will not be as strong as one made in the same material cast or wrought, provided the latter is sound. The field therefore to which the powder metallurgist turns to work is in those materials which cannot easily be cast or to produce a finished component which becomes an economic proposition to make in a die owing to machining costs. These are apart from the cases where powder methods are the only way in which a desired component can be obtained, for example carbide tips for tools and porous impregnated bearings. Some typical finished products are shown in Fig. 3.

The field of application of this technology will undoubtedly increase. At present, research is in progress to produce gas turbine blades in this way and it is not beyond the bounds of possibility that composite materials using metals and non-metals of a refractory nature should be used in order to produce materials which will withstand the rigorous combinations of temperature and oxidation encountered in these engines.