

HISTORY OF POWDER METALLURGY

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Powder metallurgy principle of shaping metallic objects without melting from powdered materials can be traced back to the early civilizations. These include the ancient Egyptian iron implants which date from at least 3000 B.C. In Greece the manufacture of iron components were widespread in 800-600 B.C. The manufacture of large objects were known to Indians as early as 300 A.D. and the famous Delhi iron pillar weighing more than six tons is a typical masterpiece indeed. These are processed by direct reduction of iron oxide without melting, since the technology to obtain temperature high enough to melt pure iron was not available until about 1800. The significant development in the use of powder metallurgy principle took place during the early part of nineteenth century for processing platinum and the credit to this work is to be given to Wollaston in England and Sobolevskiy in Russia. These developments ultimately led to the modern *renaissance* of powder metallurgy in the beginning of twentieth century with the manufacture of tungsten filaments for the incandescent lamp industry. The invention of electric lamp by Thomas Edison and Swan a century ago has contributed substantially to the rapid progress of this field. Powder metallurgy emerged as a new dimension in materials technology in twentieth century particularly during the world war period and subsequent years. Today the technology is used advantageously to process advanced materials for the nuclear, electronic and aerospace industries. But in modern India the progress made in this field is mainly during the past two decades.

INTRODUCTION

Powder metallurgy is concerned with the production of metal powders and converting them to useful shape. It is a material processing technique in which particulate materials are consolidated to semifinished and finished products. Generally the emphasis is on the metallic materials but the principles of the process apply with little modification to ceramics, polymers and a variety of composite materials composed of metallic and non-metallic phases. Nowadays powder metallurgy techniques are increasingly used to provide exceptional properties that are required in highly sophisticated aerospace electronic and nuclear energy industries. However automobile industry is the major consumer of powder metallurgy products today. There are two important reasons to use powder metallurgy by industries. Products like tungsten filament, tungsten carbide, porous self lubricating bearings etc. are either difficult or impossible to make by other methods. The other reason is that powder metallurgy process of manufacturing structural components competes with other manufacturing methods such as casting, machining and forging. Powder metallurgy process minimises or eliminates the machining, and scrap losses at the same time is suited to high volume pro-

duction of components. The process offers economy, savings in energy and raw materials along with mass production of quality precision components.

THE BASIC PRINCIPLES OF THE PROCESS

The traditional powder metallurgy process consists of blending the metal powders and other constituents followed by compaction to produce the desired size and shape. The green compact is then sintered by heating at elevated temperatures, preferably below the melting point of the major constituent to get a product of desired density, structure and properties. The two stages of compaction and sintering are combined into a single step in hot pressing. Powders can also be rolled continuously and sintered to produce strips and other flat products or can be forged to get high strength finished components. Some of the limitations of die compaction and sintering in traditional powder metallurgy process can overcome by the recently developed isostatic compaction and hot isostatic compaction methods. The latter method is increasingly becoming important for the fabrication of sophisticated and advanced materials.

EARLY DEVELOPMENTS

One of the important characteristics of powder metallurgy process is the shaping of metallic objects from powdered materials without melting. Hence many of the metallic objects of older civilizations can be believed to be the application of powder metallurgy principles^{1,2,3}. Iron objects were known at least from 3000 B.C. much before the iron age of about 1200 B.C. Ancient Egyptians knew the carburizing of iron in 1200 B.C., and the quenching technique in 900 to 700 B.C. Tempering of iron was employed in Roman times. Daggers ornamented with gold powder were found in the tomb of the Egyptian Pharaoh Tutankhamon, who lived sometime in the fourteenth century B.C. These ornamental gold, silver, copper etc. powders were made by grinding the particles or amalgams in a special mortar and pestle. The mercury from the amalgam was driven off by heat and the resulting powders were processed to make the pigments⁴. Powders were also prepared by mechanical disintegration in water and also by the reduction of oxides. The manufacture of iron components were widespread in 800 to 600 B.C. in Greece. In the earlier days metallic iron was used by man as weapons useful in the struggle for existence. In those days the only way to produce useful objects of iron was by hammering together lumps of sponge iron to the desired shapes. The iron lumps or sponge were prepared by reduction of iron ore in Charcoal fires. Majority of the products made by this way were limited in size to pieces weighing a few kilograms. But the smiths of India produced the famous Delhi iron pillar, shown in Fig. 1, weighing about 6½ tons and other objects even larger as early as 300 A.D. These are likely to be processed by hammering of hot pieces of reduced iron, produced by the direct reduction of iron oxide without melting, since the technology to obtain temperature high enough to melt pure iron was not available until about 1800. It is also reported that the natives of Matakam tribe of Central Africa have also formed their iron tools from sponge iron by hammering and heating (see ref. 1). In the above

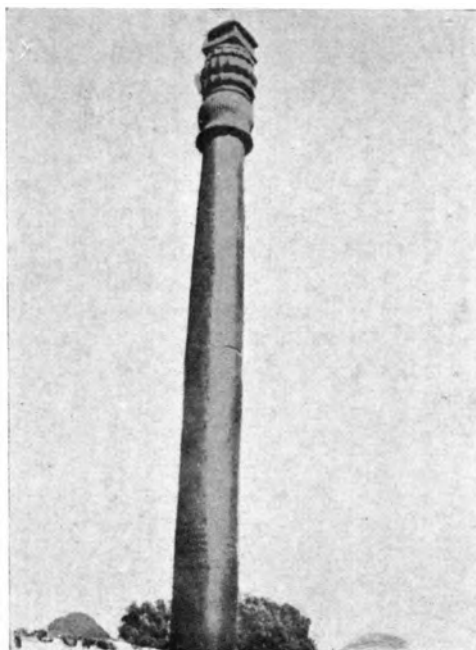


Fig. 1. Delhi Iron Pillar

discussion the hammering process may be compared to the compaction and heating to the sintering of the powder metallurgy process. Powder metallurgy principles were also used by the Arabs and Germans about 1000 A.D. to make their high quality swords from iron powder (see ref. 2). The iron powder was produced by filling the hammered steel lumps. After a certain rusting the powder was again hot forged and the treatment repeated until the carbon content is low enough and the impurities were finely dispersed in the matrix. These principles of powder metallurgy processes for making iron and steel were forgotten and replaced by the metallurgy of molten iron with the development of high temperature furnaces for the reduction and treating of iron and steel in the seventeenth and eighteenth centuries. The same procedure of powder metallurgy principles were adopted in the history of Platinum metal because of its high melting point of the order of 2042°K over a period of 1750 to 1850. Platinum compacts were made out of reduced platinum powder, which were subsequently sintered and hot worked. The credit to this work is to be given to Wollaston in England and Sobolevskiy in Russia who performed the experiments almost simultaneously.^{5,6} Sobolevskiy's powder metallurgy was used to make platinum coins from powders. Nowadays powder metallurgy is also used in Canada to make the 5 cent coins from nickel powder on account of its economic viability. Wollaston succeeded in producing some of the best platinum ware, particularly crucible, of his time. The powder metallurgy of platinum was replaced by melting first, by using the oxy-hydrogen flame. By 1860 the

fusion method became very popular and almost replaced the powder metallurgy process for making platinum products, because the latter method was found to be quicker and cheaper.

MODERN RENAISSANCE OF POWDER METALLURGY

Tungsten is another metal with a higher melting point than platinum, and hence more difficult to melt and process. Therefore tungsten metal has also undergone the same history of development in which principles of powder metallurgy process were used. The invention of electric lamp in October 1879 by Thomas Edison has contributed substantially to the progress of tungsten powder metallurgy for the manufacture of filaments. Although in 1878 Joseph Wilson Swan, an Englishman has already produced some electric lamps with carbon filaments, they were unsuitable for mass production. Edison's electric lamp consisted of an evacuated bulb with carbonized cotton thread as filament glowed for more than forty hours. Metal filaments for electric lamps were introduced, sometime in 1898 with the osmium filament of Austria followed by metallised carbon filament. Tantalum filaments were used in Germany in 1905. But all these filaments were brittle although it was recognised that tungsten would be the best metal for filaments because of its high melting point of the order of 3653°K along with good electrical properties⁷. Many attempts were made and several patents were filed regarding the manufacture of the filaments by powder metallurgy process around 1906. But the development of making tungsten ductile at room temperature is due to W. D. Coolidge, who took his patent in 1909⁸. Coolidge realised that when a sufficiently high temperature was used for sintering and swaging, metal being subjected to mechanical work—its ductility increased, until finally it be-



Fig. 2. Powder Metallurgy Automotive Structural Parts.

came so ductile that it could be drawn into wire at room temperature. Further it is also possible to shape filaments into coils of very small diameter which greatly improved the efficiency of electric lamp filaments. This development may be considered as the modern renaissance of powder metallurgy where the sintering process was used to make filaments for the incandescent electric lamp industry.

The subsequent development include the production of contacts and electrode materials, sintered porous bearings, cemented carbides, a wide range of electrical and magnetic materials and finally the use of powder metallurgy process for producing certain components as a competitive process to the conventional methods of casting, working and machining. The Coolidge process consists of briquetting the tungsten powder into a form which is sintered in a protective atmosphere and subsequently swaged at high temperatures to reduce its cross section and to improve the ductility until a stage is reached where the metal is ductile at room temperature and can be drawn to wire. All the high temperature operations were carried out below the melting point of tungsten. Later other refractory metals such as molybdenum, tantalum, niobium etc. were also processed through the powder route. Powder metallurgy process is also extremely useful to add thorium oxide in a finely divided form to tungsten during the processing so that this prevented the grain growth of tungsten crystal during the use of the finished tungsten filament in an incandescent lamp which greatly increased the resistance of the filament to sagging and embrittlement.

Another advancement in the powder metallurgy took place with the manufacture of metallic objects deliberately made porous so that it could be impregnated with lubricants. Several patents were taken including the one for production of bronze materials with graphite around 1910⁹. Hence considerable developments took place in the field of powder metallurgy during the late 19th and early 20th century. The production of cemented carbides started in 1922 which revolutionised the machining and metal working industry. From this work many other products developed such as heavy alloys, cemented multi carbides, contact materials etc. using the principle of liquid phase sintering or infiltration techniques. From the sintered porous bronze bearings other development of products such as porous filters, electrodes etc. and metal-non-metal aggregates such as friction materials took place. Major consumers of traditional powder metallurgy products are the automobile industries and a variety of these products (sintermetallwerk-Krebsoge) are shown in Fig. 2. Modern applications of powder metallurgy techniques include the processing of ferrites, garnets, piezoelectric materials etc. for electronic industry, beryllium, super alloys and titanium alloys for aircraft and aerospace and a variety of fuel elements for nuclear reactors. Recent developments in powder metallurgy techniques promise to produce a variety of new materials and alloy systems tailored to meet specific technological requirements.

Although our country had an ancient and rich heritage in the iron powder metallurgy (see ref. 2), the production of modern powder metallurgy products such as bronze and iron bushes, filters and structural components started in India only

in 1958¹⁰. Subsequently cemented carbide products such as cutting tools, mining bits, wire drawing dies etc. were manufactured. Other products include metal-graphite brushes, electrical contact materials and tungsten filaments. Powder metallurgy is also used in fabricating nuclear fuel elements and for fabricating components for defence and aerospace applications. Considerable research, development and production activities in powder metallurgy are going on in India in educational institutions, R and D laboratories and private and public sector industries. To co-ordinate our efforts in this field, the Powder Metallurgy Association of India has been formed in the year 1973 as a national organization to promote and develop the growth of powder metallurgy industry and to stimulate interest in the science, technology and application of powder metallurgy.

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