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PROCESS FOR MAKING POROUS METALLIC BODIES

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This invention relates to the production of porous metal articles useful as filter bodies and the like, and particularly to improvements in the manufacture of such bodies from powdered metal.

Porous metallic bodies are commonly made from powdered metal by a variety of processes, including the well known cold compact process with or without a transient pore forming agent and subsequent sintering, and the so-called no pressure-sinter method. It is also known to mix metal powder with a binder and extrude the plastic mixture, the binder being removed in subsequent sintering. Each of these methods finds application in the manufacture of particular porous articles, and selection of the optimum method for any application depends upon the size of the article to be produced, its contour and dimensions, the degree of porosity or permeability required, and other factors.

The extrusion process is highly advantageous for many purposes, among them is that it may produce articles of complicated shape and long length. Porous metal articles produced by extrusion are also characterized by uniformity of pore size and physical strength. For some severe applications, however, even these products are not entirely satisfactory, and further improvement in strength and pore size uniformity is desirable.

A principal object of the present invention is to provide a novel process for making metallic bodies from powdered metal, which bodies exhibit unusual strength and outstanding uniformity of pore size and particle orientation. A related object is to provide a method for manufacturing sintered powdered metal filter bodies having both high strength and uniformly high fluid permeability.

Further objects are to provide a versatile method for making sintered porous metal articles, suitable for utilization of substantially all metals and metal alloys in powdered form, and adapted for the production of articles having a wide range of shapes and sizes. The process is of particular value in the manufacture of porous metallic bodies of large size, and/or of intricate configuration. Further objects will be in part obvious and in part pointed out hereinafter.

According to the present invention, metal powder is mixed with a binder to form a plastic mass which is then extruded through a die. As a salient feature of the invention, substantial pressure is applied to the extruded body, in extruded or subsequently altered form, to reorient or rearrange the metal particles therein, eliminating bridges, arches, air pockets and the like which might result in pore openings of excessive size. The extruded and pressure treated body is subsequently sintered, to volatilize the binder and join the metal particles.

An illustrative procedure according to the present invention will now be described in detail. Metal powder, for example spherical particles of 18-8 stainless steel of about 200 mesh, is mixed with a binder consisting essentially of 25 to 30 parts paraffin, 1 part molybdenum disulphide and 1 part zinc stearate, all parts being by weight.

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The binder may be prepared by first melting the paraffin, and then adding the molybdenum disulphide and zinc stearate to the molten paraffin, with thorough mixing to insure complete homogeneity.

The binder is then mixed with the metal powder in proper proportions, and the mixture, in the case of paraffin, brought to a temperature of approximately 55 to 65° C., at which the binder material is quite liquid. The proportion of binder to metal powder is desirably kept at a minimum, the theoretical ideal being just enough binder to fill the spaces between the metal particles in densely packed arrangement. In the present example, the amount of binder used will ordinarily be from 10% to about 15% by weight of the weight of metal particles, depending on the metal particle size and similar factors.

The metal powder-binder mixture is constantly and smoothly agitated to insure even distribution of the metal particles throughout the mass, and permitted to cool to about 48° C., at which temperature the mixture is plastic and capable of being extruded through a die. At the temperature noted, the consistency of the plastic mass is roughly that of modeling clay.

The homogeneous plastic mass is then extruded in conventional manner through a standard die, the molybdenum disulphide and zinc stearate in the binder serving as die lubricants. The mass is extruded in the form of a shaped body as determined by the die orifice, and the extrusion involves no difficulty or critical conditions. It has been found desirable to chill the extruded body, as by supporting it on a cool surface or by using a water spray located two or three inches from the die orifice, to "set" the binder and facilitate handling of the extruded body.

The metal powder-binder mass may be extruded to final shape, or the extruded shape may be further formed by cutting, bending, blanking, trimming and/or joining operations. The extruded body may be cut or trimmed by means of a conventional knife or shear. Large articles may be formed from smaller articles by joining them through abutting surfaces, this operation being facilitated by local application of heat and pressure.

For example, cylindrical shapes with a height to diameter ratio greatly exceeding any possible by other methods may readily be formed by extruding the powder-binder mixture in sheet form, and then trimming the sheet as necessary and bending it about a mandrel. If necessary, the bending may be facilitated by gently heating the sheet. The abutting edges of the sheet may be joined by passing a hot, flat spatula-like tool along the seam. The hot tool melts the binder in the vicinity of the seam and causes the edges to fuse together so completely that after the final sintering operation no evidence of the original seam can be discerned, even under microscopic examination. The cylindrical body may then be chilled and readily removed from the mandrel.

The extruded or extruded and further formed body is next subjected to the application of considerable pressure, essentially to compact it. The pressure may be applied in the case of sheet material by pressure rolls, in a closed die especially when dimensions are to be retained, hydrostatically in the case of unusual shapes, or otherwise. In applying pressure by rolling, sheet material may be heated to about 40 or 45° C. to make the binder plastic, and the sheet passed one or more times through pressure rolls, in which the pressure applied may be of the order of 5 tons per square inch. In the rolling of sheet material, the body will ordinarily be lengthened and widened, and its thickness reduced. In applying pressure in a die, the article may or may not be heated to plasticity, and pressure of the order noted may be applied in a single stroke. In hydrostatic application of pressure also,

a pressure of the order of several tons per square inch is effective.

After the pressure application step, the articles are sintered. For this purpose, it is usually desirable, particularly in the case of intricate shapes, to fully support the article with ceramic sand, such as a mixture of 60 mesh and 120 mesh Alundum sand. In the case of a cylinder, for example, the core of the cylinder may be packed with sand and the packed cylinder then laid in a supporting bed.

The supported bodies may then be sintered in conventional manner. In the specific example, heating to about 2400° F. in a reducing atmosphere for several hours will volatilize the binder and sinter the metal particles together. In the sintering operation, the sand pack behaves in the manner of a foundry mold, maintaining the particular body in desired configuration after volatilization of the binder, which occurs at about 400° F. After the sintering operation, the metal particles are metallurgically bonded to each other and the resulting porous articles are ready for use.

Extrusion of a plastic mass comprising metal particles and binder makes possible the production of dense, dimensionally accurate shapes. Additionally, the particle orientation in extruded bodies is more uniform than is possible, for example, in the case of loose powder, and the binder present serves to maintain the particle orientation. The relative density of the extruded mass, that is the ratio of the density of the extrusion with the binder eliminated by calculation or suitable test procedure to the density of solid metal of the type comprising the metal powder, may range from about 35% to about 45%, depending on the metal powder mesh size and other factors. During the sintering operation, the relative density usually increases somewhat.

It has been found that the strength of a porous metal body is approximately directly proportional to its relative density, within the range of from 65% to 100%. Below 65% relative density, tensile strength falls off very rapidly. Prior attempts to achieve porous metal articles of high relative density include "coining" after sintering, an operation in which sintered articles have been die coined, swaged, hydrostatically pressed, rolled and similarly treated. In coining sintered articles, however, extreme care must be exercised to prevent excessive surface metal flow, and such surface flow as does occur, if permeability to fluid is of importance in the final product, must be overcome by acid treatment or other expedient to reopen the surface pores. The coining of sintered bodies, moreover, does not achieve an increase in strength proportional to the achieved increase in relative density unless followed by a resinter, nor does it materially affect the range of variation in pore size.

The application of pressure to extruded bodies, prior to sintering, on the other hand, materially increases the relative density of the final product, and very greatly increases its strength. Moreover, pressure treatment of the extruded bodies effects a considerable decrease in maximum pore size, without materially altering the mean pore size of the final product.

As previously indicated, the relative density of extruded powdered metal-binder shapes may range from about 35% to about 45%. Pressure treatment prior to sintering is found to increase the relative density of the extruded bodies from 4% to 10%, apparently due to the destruction of arches and bridges in the particle arrangement, and the elimination of air pockets or bubbles. The pressure treatment of the extruded articles appears to be accompanied by movement of binder to the article surfaces.

In a series of experiments, for example, a plurality of extruded discs comprising stainless steel particles and paraffin binder mixed as previously described were found to have relative densities ranging from 39.3% to 40.7%. After pressure treatment of the "green" articles in a

die, applying pressure of 5.66 tons per square inch, the relative densities were found to be between 44.5% and 45.5%. In both cases, the relative density determinations included calculation to eliminate the effect of the binder present.

In another series of experiments a number of similar discs were prepared and sintered in as-extruded condition. The mean pore size of the sintered discs ranged from 13.0 to 16.9 microns, while the maximum pore size in the same group varied from sample to sample between 62.5 and 98.7 microns. A similar series of discs were prepared and extruded under identical conditions, and subjected to pressure treatment in a die before sintering. After sintering, the resultant articles exhibited mean pore size ranging from 10.6 to 14.8 microns, and a maximum pore size ranging between 45.3 and 51.9 microns. It is evident, accordingly, that pressure treatment of the extruded bodies before sintering effects a substantial decrease in maximum pore size, accompanied by a negligible decrease in mean pore size. The decreased differential between mean and maximum pore size in the sintered bodies, and the increased uniformity in maximum pore size, indicates more uniform particle packing, and the destruction of voids previously existing, due to particles bridging, air bubbles or the like. The maximum pore size and the differential between mean and maximum pore size are of extreme importance in the manufacture of filters, since they affect the fluid permeability and the degree of filtration of which the porous metal bodies are capable.

In a further series of experiments, a number of tensile specimens were prepared as previously described and extruded, and then sintered for 4 hours at 2400° F. in dry hydrogen. The sintered samples exhibited tensile strength ranging from 2430 to 2790 pounds per square inch. Similar specimens were sintered in as-extruded condition for a total of 16 hours at 2400° F., these products exhibiting tensile strength ranging from 2480 to 2790 pounds per square inch. Only minor increase in tensile strength, then, resulted from sintering for an extended period of time.

A similar series of specimens were prepared and extruded under identical conditions, and subjected to pressure treatment in a die at about 5 tons per square inch prior to sintering. The pressure treated specimens were then sintered for 4 hours at 2400° F. in dry hydrogen, and the sintered samples exhibited tensile strength ranging from 4700 to 5160 pounds per square inch. Other specimens treated similarly but at lower pressures exhibited similar improvement in tensile strength, although to lesser degree. At higher pressures, adherence to the die plunger was encountered. It is believed that the improvement in tensile strength due to the pressure treatment prior to sintering is roughly proportional to the pressure applied, up to a practical limit. The improvement in tensile strength and other properties, and the pressure necessary to effect such improvement, will vary of course with different metals, different binders, different metal powder particle size and other variables.

Pressure treatment of the exemplary extruded articles prior to sintering, then, effects a substantially 100% increase in tensile strength of the final product, and the difference between maximum and mean pore size is greatly reduced, and more constant from sample to sample. It is also found that the preliminary pressure treatment effects no noticeable difference in air flow vs. pressure drop characteristics, as compared to articles sintered in as-extruded condition. It is believed that this improvement in properties derives from a more ordered particle orientation in the extruded bodies as a result of the pressure application. In the pressure treatment step, the particles apparently slide or glide past one another to achieve a denser packing, or state of orientation. This results in increased particle to particle contact area per unit of volume in the finished sheet, and accounts for

the very great strength increase, which is a direct function of the total particle to particle contact area. Similarly, destruction of arches and bridges in the porous structure apparently accounts for the decrease in maximum pore size, but inasmuch as the majority of the pores remain unchanged, little or substantially no effect on mean pore size results.

The advantages of the pressure treatment prior to sintering are obtained only with a binding agent present, the binder apparently permitting the particles to slide freely relative to one another within the porous mass, and to reorient themselves into a more closely packed state. This function is enhanced, it is believed, by use of a binder of lubricating character. Application of pressure in the absence of binder does not effect the same results, although it may increase existing particle to particle contact area somewhat by deformation of the particles.

While the invention has been described in connection with stainless steel particles, it is obviously of equal utility with other high temperature alloys, such as Haynes Stellite #31, and with other metals and metal alloys, such as pure iron, brass, bronze and nickel silver. Similarly, in place of paraffin, thermoplastic resins or synthetic polymeric materials or other binders may be employed.

It will thus be seen that there has been provided by this invention a method in which the various objects hereinbefore set forth, together with many practical advantages, are successfully achieved. As various possible embodiments may be made of the novel features of the above invention, all without departing from the scope thereof, it is to be understood that all matter hereinbefore set forth is to be interpreted as illustrative, and not in a limiting sense.

I claim:

1. A process for making porous metallic bodies useful as filters which comprises, in sequence, the steps of mixing metal powder with a binder to form a plastic mass, extruding said mass as a shaped body, applying pressure to said body whereby metal particles therein are reoriented and then heating said body to volatilize the binder and sinter the metal powder, said pressure applied to said body being sufficient to significantly increase the density of the resulting porous metallic body and decrease the maximum pore size thereof without materially altering the mean pore size.

2. A process as claimed in claim 1 wherein said shaped body is cooled to decrease its plasticity and facilitate handling thereof prior to the application of pressure thereto.

3. A process as claimed in claim 2 wherein said cooling is accomplished by means of a water spray.

4. A process for making porous metallic bodies useful as filters which comprises, in sequence, the steps of mixing metal powder with a binder with constant agitation to form a homogeneous plastic mass, extruding said mass as a shaped body, applying pressure to said body whereby metal particles therein are reoriented, and then heating said body to volatilize the binder and sinter the metal powder, said pressure applied to said body being sufficient to significantly increase the density of the resulting porous metallic body and decrease the maximum pore size thereof without materially altering the mean pore size.

5. A process as defined in claim 4 wherein said binder comprises paraffin and a lubricant.

6. A process as defined in claim 4, wherein said binder comprises approximately 25 parts by weight paraffin, 1 part molybdenum disulfide and 1 part zinc stearate.

7. A process as defined in claim 4, wherein said reorienting pressure is applied to said body in a die.

8. A process for making porous metallic bodies useful as filters which comprises, in sequence, the steps of mixing spherical stainless steel particles with a binder to form a plastic mass, extruding said mass as a shaped body, applying pressure to said body whereby metal particles therein are reoriented, and then heating said body to about 2400° F. in a reducing atmosphere to volatilize the binder and sinter the metal powder, said pressure applied to said body being sufficient to significantly increase the density of the resulting porous metallic body and decrease the maximum pore size thereof without materially altering the mean pore size.

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