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METHOD OF CONTROLLING THE DENSITY OF SINTERED COMPACTS

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The present invention relates to an improved process for controlling the dimensions and density of a powdered metal article during sintering.

The process of the present invention represents an improvement over the newly developed metal forming process in which powdered metal is mixed with a binder consisting of a heat-fugitive material, and the mixture is heated to a temperature above the softening points of the binder ingredients but below their vaporization temperatures to produce a mass that will observe the laws of hydraulic flow, with the metal particles in suspension in a continuous matrix of binder. This plastic mass is then formed into a desired shape by means of processes such as injection molding, extrusion, pressing, and the like. The preformed articles are then placed in a sintering furnace and subjected to a relatively high temperature to drive off the binder by depolymerization of the organic polymer contained therein without leaving a carbonaceous residue. The preferred class of resins used as binder materials in this type of process consist of the poly-mono-olefinic resins, such as polybutene, polystyrene, polyethylene, and the like, which decompose at temperatures at or below the sintering temperature of the metal particles to liberate decomposition products including polymers of lower molecular weight than the poly-mono-olefinic resin used as the binder. These depolymerization products are continuously volatilized during the time that the mass is heated to the appropriate sintering temperature until all of the binder is removed by volatilization and the metal particles are sintered together into a coherent structure.

We have observed that it is difficult to control the amount of shrinkage which naturally occurs between the time that the binder-containing plastic mass is introduced into the sintering furnace and the time at which the final sintered article is removed from the furnace. This shrinkage is caused by the drawing together of the powdered metal particles as the plastic binding material is being removed.

We have now found that this shrinkage during volatilization of the binder can be controlled to within closely defined limits if a non-oxidizing protective gas stream is passed over the articles during heating such that the vaporized materials resulting from pyrolitic decomposition of the binder are removed at carefully controlled rates. The specific rate of gas flow for the non-oxidizing protective gas stream will, of course, vary depending upon the ultimate density and dimensions desired in the final articles, the size of the furnace, the number of parts in the furnace charge, and their volume and surface area. However, we have found that for the production of coherent metal articles of controlled uniform porosity, the rate of flow of the protective gas stream should be such that the content of materials liberated from the binder during heating of the plastic mass in the stream does not exceed about 75% by volume.

The change in density upon heating of compacts containing metal particles and a heat fugitive resin is due

to shrinkage or drawing together of the powdered metal particles as the heat fugitive binder is being volatilized. If the binder is rapidly removed, the particles of powdered metal are left with relatively little surface contact and large void areas. If the binder is removed slowly, the particles are brought together leaving relatively few voids and a relatively large amount of surface contact. This effect is noticeable through the solid, liquid, and vapor phases of the heat-fugitive resin. The amount of shrinkage occurring is particularly marked where relatively large amounts, on the order of 35 to 50% by volume, of heat-fugitive binder material is used in preparing the plastic composition. Such relatively large amounts of binder are used so that the plastic mass observes the laws of hydraulic flow, thus facilitating extrusion and making possible a wide variation in the density and porosity of the finished article.

If the volatilization of the heat fugitive binder is prevented, the binder exists as a liquid which tends to coalesce, thereby bringing the metal particles closer together. As a result, when the article is sintered it is very dense, having shrunk considerably from its molded dimensions, and is generally severely distorted from its previous form. On the other hand, where the volatilization of the binder material is forced by rapidly conducting away the vapors as the vapors are volatilized from the plastic composition, the metal particles are left at relatively great distances from each other. Consequently during sintering, there is little tendency for the particles to bond together, and very little shrinkage occurs. However, the finished article is too porous for ordinary use and is structurally weak.

An object of the present invention is to provide an improved method for controlling the shrinkage and density of powdered metal compacts containing heat-fugitive binder materials during heating of such compacts to a sintering temperature.

Another object of the present invention is to provide an improved method for the controlled removal of depolymerization products of a heat-fugitive depolymerizable resin binder during heating to thereby control the density and porosity of a powdered metal compact including such heat-depolymerizable resin material as a binder.

In making the articles according to the present invention, powdered metal and the heat-fugitive binder are mixed together in suitable proportions, preferably in proportions which include about 35 to 50% by volume of binder, and the resulting plastic mass is shaped at a temperature above the softening point of the binder but below the volatilization point. As mentioned previously, the preferred binders are the thermally depolymerizable poly-mono-olefins, particularly polybutene, and other compounds such as polystyrene, polyacrylate, polymethacrylate, polyethylene, polypropylene, and polyvinyl butyl ether. The resulting plastic mass is shaped, as by means of injection molding at a temperature of about 300° F. Upon leaving the injection mold, the articles are introduced into a furnace in which sintering of the metal particles and volatilization of the binder takes place. In the case of iron particles, a temperature of about 2100° F. is sufficient for effective sintering. In order that the volatilization of the binder proceed at a relatively uniform and controlled rate, it is desirable that the plastic mass be heated to the sintering temperature at a rate not in excess of about 30° F. per minute, and preferably at a rate of about 15° F. per minute.

During the heating of the articles in the sintering furnace, a stream of protective, non-oxidizing gas is introduced into the furnace and directed over the articles being treated therein. The nature of the protective gas will depend essentially upon the nature of the particles and for the more common metals, such as iron, gases such as

hydrogen and/or cracked ammonia, or other inert, non-oxidizing gases can be employed.

The control of the rate of introduction of the sweeping gas, such as hydrogen, into the furnace may be varied within wide limits, depending upon the density desired in the ultimate article. Where a substantially porous article is to be produced, the protective gas can be introduced into the furnace at velocities up to those which would cause turbulence within the furnace. It will be understood that the furnace will normally be provided with means for conducting away gaseous binder products picked up by the sweeping gas.

The rate of passage of the sweeping gas rate and the density of the article being sintered can be balanced through analysis of the depolymerization products present in the gas atmosphere. Samples of the protective gas stream after leaving the area in which the articles are treated may be periodically or continuously withdrawn and analyzed for their content of depolymerization products. If the analysis shows that the content of the products in the gas stream is in excess of about 75% by volume, the rate of introduction of the sweeping protective gas is increased until the analysis indicates that the percentage of depolymerization products in the stream is below the value of 75% by volume. It will be appreciated that the proper concentration of gaseous decomposition products in the stream will depend upon the degree of porosity desired in the final article, and the shrinkage desired, so that the actual desired value of the polymerization product concentration in the gaseous stream may be well below the figure of 75%. In fact, the relative concentration may be as low as a few percent if a high gas velocity is employed, provided that the gas velocity is not sufficiently high to cause turbulence in the furnace.

In a specific example of the type of process described above, a plastic composition containing about 100 parts by weight iron powder, 7 parts by weight polystyrene, and 5 parts by weight microcrystalline wax was shaped by means of injection molding at about 300° F. and heated to a sintering temperature of 2100° F. in the presence of a stream of hydrogen. The furnace employed was 3 inches in diameter, about 67 inches long, and contained 5 powdered iron compacts in the furnace. By varying the sweeping gas rate from a value of 1.5 feet per minute to about 10 feet per minute through the furnace within this range of gas velocities, powdered iron compacts were produced having densities ranging from about 90% to about 60% of the theoretical density of iron.

From the foregoing, it will be appreciated that the method of the present invention provides an accurate means of producing powdered metal articles of controlled density and controlled dimensions. As a result, powdered metal articles can be produced to within close tolerances with respect to density and physical dimensions.

This application is a continuation-in-part of our co-pending application Serial No. 166,970, filed June 8, 1950, now Patent No. 2,622,024, granted December 16, 1952, and entitled "Method of Controlling the Density of Sintered Compacts."

It will be understood that modifications and variations may be effected without departing from the scope of the novel concepts of the present invention.

We claim as our invention:

1. In the method of making a porous sintered metal article of predetermined dimensions and density wherein a preformed plastic mass containing metal particles and 35 to 50 volume percent of a heat-fugitive depolymerizable poly-mono-olefinic resin binder are heated to a sintering temperature in the presence of a non-oxidizing protective gas, the step of directing said protective gas as a stream past said article at less than a turbulent rate and a rate sufficient that the content of materials liberated from said binder during said heating in said stream does not exceed about 75% by volume.

2. In the method of making a porous sintered metal article of predetermined dimensions and density wherein a preformed plastic mass containing metal particles and a heat-fugitive depolymerizable resin binder are heated to a sintering temperature in the presence of a non-oxidizing protective gas, the step of directing said protective gas as a stream past said article at a rate sufficient to maintain the content of materials liberated from said binder into said gas during said heating at a value not in excess of about 75% by volume, but at a rate insufficient to cause turbulent flow of said protective gas stream.

3. In the method of making a porous sintered iron article of predetermined dimensions and density wherein a preformed plastic mass containing iron particles and a heat-depolymerizable poly-mono-olefinic resin are heated to a sintering temperature in the presence of a non-oxidizing protective gas, the steps of heating said article to a sintering temperature at a rate not in excess of about 30° F. per minute, and directing said protective gas as a stream past said article at less than a turbulent rate and a rate sufficient so that the content of materials liberated from said binder during said heating in said stream does not exceed about 75% by volume.

4. In the method of making a porous sintered iron article of predetermined dimensions and density wherein a preformed plastic mass containing iron particles and 35 to 50 volume percent of a heat-depolymerizable poly-mono-olefinic resin are heated to a sintering temperature in the presence of a non-oxidizing protective gas, the steps of heating said article to a sintering temperature at a rate not in excess of about 30° F. per minute, and directing a stream of hydrogen past said article at less than a turbulent rate and a rate sufficient that the content of materials liberated from said binder into said hydrogen stream during said heating does not exceed about 75% by volume.

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