

## UNITED STATES PATENT OFFICE

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METHOD OF CONTROLLING THE DENSITY  
OF SINTERED COMPACTSRaymond S. Gurnick, Lakewood, and Robert T.  
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1 Claim. (Cl. 75-222)

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The present invention relates to an improvement in the art of making porous sintered compacts.

In carrying out the method of this invention metal powder is mixed with a binder containing a heat depolymerizable organic binder, 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. 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 thus produced are next 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 temperature of the mass is progressively increased until all of the binder is removed by volatilization and the metal particles are sintered together into a coherent form.

It has been 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 of removal of the finally sintered article from the furnace. This shrinkage is caused by the drawing together of the powdered metal particles as the plastic binding material is being removed. The present invention now controls this shrinkage by regulation of the rate of volatilization of the binder.

It is therefore an object of the present invention to provide a method of controlling the amount of shrinkage and hence the density of porous metal compacts.

Another object of the present invention is to provide a method for regulating the rate of volatilization of the binder from the types of plastic masses previously described so that an article of a predetermined density and predetermined physical dimensions can be produced upon sintering.

Still another object of the present invention is to provide a method for producing sintered compacts in which the degree of shrinkage during volatilization of the binder and sintering of the metal powder can be controlled.

We have found the density and physical dimensions of a sintered metal compact may be controlled by controlling the rate of volatilization of the binder material as the plastic compact containing the metal particles suspended in a

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heat-fugitive resin binder is heated through a temperature range where the binder is volatilized, and up to the temperature used for sintering the particles into a coherent form.

We have found that the change in density upon heating such compacts is due to the shrinkage or drawing together of the powdered metal particles as the heat-fugitive binder is 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 phenomenon takes place through the solid, liquid and vapor phases of the heat-fugitive resin. The amount of shrinkage thus occurring is particularly marked where relatively large amounts, on the order of 35 to 50%, by volume of heat-fugitive binder material are used in preparing the plastic composition. Such relatively large amounts of binder material 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 ultimate article.

We have also found that if the heat-fugitive binder is prevented from volatilizing, 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 same 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 them to bond together, and very little shrinkage occurs. However, the article is too porous for ordinary use and is structurally weak.

The control of the rate of volatilization of the binder can be accomplished by adjusting one or more of three inter-related variables. The first of these is the control of the rate of heating of the preformed mass until the time that the sintering temperature is reached. The second is the rate of introduction of protective gas, such as hydrogen, cracked ammonia, or other inert gas, used to carry off the volatilized binder compounds.

The third is the control of the content of vaporized binder material in the atmosphere surround-

ing the mass being heated. In the case where a non-decomposing binder is used, this involves controlling the vapor pressure of the volatilized binder compound in the surrounding atmosphere.

A preferred binder is a thermally depolymerizable polyolefin, particularly polybutene, and other depolymerizable compounds such as polystyrene, polyacrylate, polymethacrylate, polyethylene, polypropylene, and polyvinylbutyl ether may be employed. These organic polymers decompose into lower molecular weight polymers or monomers during volatilization so that addition of such decomposition products to the atmosphere surrounding the article during volatilization of the binder effectively inhibits the volatilization of the binder at too rapid a rate.

Control of the rate of volatilization of the binder materials through one or more of the above described methods has been found to have a very substantial effect upon the density of the ultimate sintered article. For example, a plastic composition containing 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 and heated to a sintering temperature of 2100° F. in the presence of hydrogen. By varying the amount of polystyrene present in the furnace, sintered iron compacts having densities from 65% to over 90% of the theoretical density of iron were obtained.

The specific ranges of the variables to be used for any given material will, of course, vary with the density desired in the sintered article.

The volatilization of the binder and sintering of the article can be most conveniently accomplished by passing the plastic article after shaping through a furnace having areas of progressively increasing temperature. For example, the temperature at the inlet to the furnace may be slightly above the temperature of the mass after leaving the injection mold, normally on the order of 300° F., and the temperature at the outlet end of the furnace can be maintained constant at the sintering temperature of the metal or higher. In the case of iron, a temperature of 2100° F. is sufficient for effective sintering. The rate of heating of the articles as they pass through the furnace can be conveniently controlled by varying the velocity of the conveyor which carries the articles through the furnace. In the case of producing sintered iron compacts, we have found that a convenient heating rate for the compacts between the temperatures of 300° F. and 2100° F. is in the vicinity of 15° F. per minute.

The control of the rate of the introduction of the sweeping gas, such as hydrogen, into the furnace may also be varied within wide limits depending on the density desired in the ultimate article. In producing iron compacts for most uses where substantial strength is desired, the gas velocity employed for the sweeping gas will be just sufficient to prevent diffusion of gaseous binder products into the sintering zone where they could decompose to form free carbon. On the other hand, where a substantially more

porous article is to be produced, the protective gas can be introduced at velocities up to those which would create 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 prior to reaching the higher temperature zones.

In the manufacture of sintered articles having a very high density, approaching the theoretical density of the metal itself, it is desirable to incorporate in the stream of protective gas passing through the furnace a supply of volatilized binder compound or products of its thermal decomposition. For example, in using a polystyrene binder, the control of the rate of volatilization of the binder can be accomplished by vaporizing a quantity of polystyrene and passing it into the furnace at a point upstream of the volatilization zone.

From the foregoing, it will be appreciated that we have herein provided a convenient method for accurately controlling the density of sintered porous compacts produced from a plastic preshaped mass having a large percentage of organic binder. The control of the mentioned variables can be accomplished to a very accurate degree to produce a sintered article having a density and physical dimensions within closely defined limits.

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:

In the method of making porous sintered metal compacts of predetermined dimensions and density wherein a preformed plastic mass containing metal particles and a heat-depolymerizable polymeric binder is heated in a heating zone to a sintering temperature in the presence of a current of protective gas, the step of adding to said current of gas a quantity of a depolymerization product of said binder, said depolymerization product comprising a material having a lower degree of polymerization than said polymeric binder in an amount effective to diminish the rate of volatilization of compounds resulting from the thermal depolymerization of said binder.

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