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METHOD OF MAKING POROUS METALLIC BODIES

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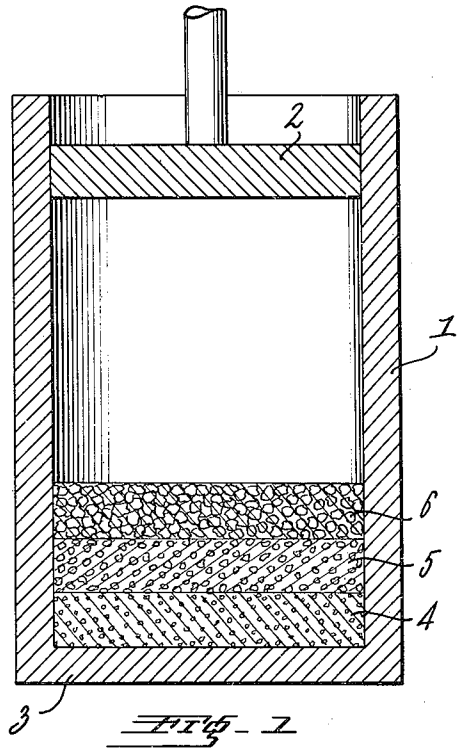



Fig. 1

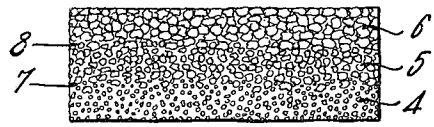



Fig. 2

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## METHOD OF MAKING POROUS METALLIC BODIES

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The present invention relates to porous metallic bodies and methods of making the same. More particularly, it relates to porous metallic bodies suitable for use as filters and while the method may be used with a wide variety of metals, in its preferred form it is applicable to the formation of porous bodies of refractory metals such as tungsten and molybdenum. It has, however, a special adaptation to alloys such as iron molybdenum manganese alloy, and generally to metals that can be cast and suddenly chilled in a manner to produce a brittle formation of large crystals.

The method may generally be described as, first, forming an ingot or body of other shape having large crystals and a consequent brittleness of structure which permits the breaking up of the body into its component crystals. The individual crystals are of considerable strength and hardness although the body as a whole formed from these crystals is quite brittle. The body is first formed into large crystals either by heating the ingot in the case of the more refractory metals to the germinating temperature, or by casting the ingot and immediately chilling it. The ingot is then broken up into its component crystals and these crystals are again broken down by mechanical means such as ball milling, crushing or hammering.

The crystal fragments so produced vary considerably in size and it is, therefore, desirable to screen the fragments into different batches in which the particles are of approximately equal sizes. This may be done by passing the powdered crystalline material through a series of screens varying from say 40 mesh to about 350 mesh. There may thus be screens of the following sizes which are given by way of example merely: 40 mesh, 60 mesh, 100 mesh, 150 mesh, 200 mesh, 250 mesh, 300 mesh, and 350 mesh. By combining the material retained on certain screens with that retained on others, a considerable degree of control of the porosity of the finished body is permitted. Thus, if a given proportion of larger size fragments is mingled with a given proportion of fragments of smaller sizes, a certain degree of porosity of the finished body may be secured, and this porosity may be increased by increasing the proportion of larger size fragments or it may be diminished by increasing the proportion of the smaller size fragments.

When a batch is prepared having a predetermined proportion of large and small size fragments, it is thoroughly wetted with a solution of a soluble salt of a metal such as silver, copper or nickel. The salt used should be one which

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will break down to the oxide under temperature. For instance, an aqueous solution of silver nitrate may be used and the powdered crystalline metal is thoroughly wetted with this solution and dried so as to form a coating of silver nitrate on the individual particles. It is then heated to a temperature sufficient to break down the nitrate to the oxide.

A binding material which is decomposable at temperatures below sintering temperature, of the metal with which the powdered base metal is coated and which will volatilize at that temperature without leaving a residue, should be used. Among such binding materials are paraffin dissolved in carbon tetrachloride, salicyclic acid, naphthalene, benzyldichloride, and ammonium chloride, and many others. The coated powdered metal is thoroughly mixed with this binding material and the mass is shaped in a die under hydraulic pressure.

After pressing and shaping in this manner, the mass is then heated in an atmosphere of hydrogen to a temperature sufficient to volatilize the binder out of the mass without leaving a carbon residue, and when volatilization is complete the temperature is held sufficiently high to reduce the oxide coating to a metallic coating. This temperature should be further increased to a point at which the coating metal on the particles will sinter but it should not be sufficiently high to melt the coating metal to a fluid condition at which it would flow off the particles. There is very little shrinkage of the body under this treatment owing to the fact that at the temperatures used the crystalline fragments show little tendency to coalesce and the voids remaining after the volatilization of the binding material show practically no diminution of size or number. Nowithstanding this lack of shrinkage, the final product has considerable mechanical strength.

The method may be illustrated by describing the formation of such a body from tungsten.

Tungsten powder is first formed under hydraulic pressure in a die to the shape of a bar or ingot. This bar is then sintered and heat treated in a hydrogen atmosphere at the temperature of rapid germination or about 2800° C. and held at that temperature for an adequate time to sinter the ingot and to form the whole body into large crystals. About fifteen minutes is sufficient for this purpose. The ingot so formed is quite brittle and can be broken up into its component crystals and these in turn are further broken down to smaller size in a ball mill or jaw crusher. The material is then screened in the manner herein-

fore described, and the particles thus graded cording to size. Batches selected according to e are then thoroughly mixed together in a pretermined manner to produce the desired degree porosity in the finished body and the mass so med is thoroughly wetted with an aqueous lution of silver nitrate and then dried. Into e same so formed a solution of paraffin dislved in carbon teterachloride is thoroughly xed and dried, and the whole is then formed to e desired shape in a die under hydraulic pressre. The body so formed is then heated to a mperature of about 400° C. until the paraffin ıding material is completely volatilized without ıving any residue. The temperature is regu-ed so that the silver nitrate coating has broken wn to silver oxide and the temperature is then adually raised to about 800° C., the oxide coatg being thus reduced to metallic silver and the etallic silver coated particles are sintered tother. The resulting body is highly porous and considerable mechanical strength.

The invention will be more clearly understood om the drawings in which Fig. 1 represents an paratus for carrying out the method of the inntion and the formation of a porous metallic dy having zones of different degrees of porosity, d Fig. 2 represents a finished porous metallic dy having such zones.

Referring now to Fig. 1, a mold or die 1 having erein a movable plunger 2 and a stationary ınger or stop 3, is provided. An ingot of base etal is heat treated as hereinabove described as to form large crystals and then broken down to individual crystals. The crystals are ıshed, the broken particles screened to form tches graded as to size, and then thoroughly tted with an aqueous solution of silver nitrate d dried. Each batch is then mixed thoroughly th a binding material, such as paraffin dis-ved in carbon tetrachloride. A batch of very ıe screenings consisting, for instance, of parles that pass through a 300 mesh screen and e retained on a 350 mesh screen, mixed with is binder, is then introduced into the die 1 to m a layer as indicated at 4. The plunger 2 then forced down under hydraulic pressure the layer 4 with sufficient pressure to reduce is material to about two-thirds of its original lume. The plunger is then removed and a ond batch of screenings 5 consisting, for ample, of particles that pass through a screen 150 mesh and are retained on a 200 mesh een, is then placed in the die 1 on top of the rtially pressed layer 4. This material is then draulically pressed in about the same degree the bottom layer 4. The plunger 2 is again reved and a third batch 6 consisting of particles ssing a 40 mesh screen and being retained on 60 mesh screen, is then filled into the die 1 top of the partially compressed layer 5 and bjected to very heavy pressure of the order about 30,000 lbs. to the square inch, thus closely mpacting the entire mass.

The block thus formed is then removed from e die 1. It is heat treated, as hereinabove deribed, to volatilize out the binder, to reduce the ver nitrate to oxide and ultimately to a metallic ver coating on the particles and to sinter the ated particles into a mechanically strong, corent but porous mass.

The resultant body is illustrated in Fig. 2 in ıich it will be seen that the original layers 4, 5 d 6 are still roughly preserved but that where e layers contact each other, part of each layer



is forced into the other by reason of the heavy pressure ultimately applied. Marginal layers of this sort are indicated in Fig. 2 at 7 and 8. It will thus be obvious that in this manner the porosity of the finished body may be regulated from a high porosity at 6 through zones of decreasing porosity to 4.

The first step in the process, namely, that of forming the metal into large crystals, may be accomplished in a variety of ways, and it will be noted that the method used will depend upon the nature of the metal dealt with. If the metal concerned is one having a high melting point, such as tungsten, molybdenum and similar refractory metals, the method given in the example of tungsten will be found most convenient under ordinary circumstances. They will be formed into an ingot by pressing and sintering and then heat treated to the germinating temperature of the metal. On the other hand, with lower melting point metals and many alloys, such as the alloy of iron, molybdenum and manganese hereinabove suggested, the metal will be melted and cast and then suddenly chilled by quenching it in cold water. The ingot so formed will be found to have exceedingly large crystals. In fact, any method which is effective to produce large crystals in a given particular metal may be used.

Metallic bodies formed according to the present invention are characterized by a porous continuity permeating the entire structure and permit the passage of fluids therethrough while obstructing the passage of contaminating solid matter that may be contained in said fluids. Such bodies may be constructed with very small pores intercommunicating with each other so as to retain lubricants within the pores that constantly feed to the surface and function very efficiently as self-lubricating bearings. Metals such as an iron molybdenum manganese alloy or tungsten having the particles coated with lead reduced from lead nitrate in the manner hereinabove described, are especially suitable for use as bearings.

Having thus described my invention, what I claim is:

1. Method of making a porous metallic body that comprises making an ingot of metal and treating said metal so as to cause it to form into large crystals; breaking said ingot into its individual crystals, crushing the crystals into crystalline particles; then wetting said particles with a solution of a metallic salt reducible to an oxide under heat, drying said solution on said particles and heat treating said particles until said salt is reduced to an oxide coating on said particles; then mixing said oxide coated particles with a binding medium, pressing said material in a die to a desired shape and heating at temperatures regulated to volatilize said binding material completely, reduce said oxide coating on said particles to metal and sinter said particles into a strong, coherent, metallic body permeated by intercommunicating pores.

2. Method of making a porous body of tungsten that comprises making an ingot of tungsten and heat treating said ingot at a temperature of about 2800° C. until said ingot is formed into large crystals; breaking said ingot into its component crystals and crushing said crystals into crystalline particles of irregular sizes and shapes; grading said crystals into batches according to size by screening and mixing the material from said batches in predetermined proportions according to the degree of porosity desired in the finished

body; then thoroughly wetting said mixed particles with an aqueous solution of silver nitrate and heating until said nitrate breaks down into a silver oxide coating; then thoroughly mixing said silver oxide coated particles with a solution of paraffin in carbon tetrachloride and shaping the mass so formed in a die under hydraulic pressure; and finally heating said formed mass at temperatures regulated, first, to volatilize said paraffin without leaving a residue, then, to reduce said silver oxide coating to metallic silver and then to sinter said silver coated tungsten particles into a strong, coherent body permeated by intercommunicating pores.

3. Method of making a porous metallic body according to claim 1, in which a plurality of batches of crystalline particles from separate screenings through screens of different mesh after being coated and mixed with binding material are each separately partially pressed in a die in layers one above the other and then further pressed and heat treated to volatilize out said binding material, reduce said coating to metal and finally to sinter the same into a strong, coherent, porous body having zones of different degrees of intercommunicating porosity corresponding to said layers.

4. Method of making a porous metallic body according to claim 1 in which an ingot is made of a refractory metal and is formed into large crystals by heating at the temperature of rapid germination until said ingot is formed into large crystals.

5. Method of making a porous metallic body according to claim 1 in which an ingot is formed of a metal that is cast and suddenly chilled, thereby forming said ingot into large crystals.

6. The method of forming a porous metallic body having zones of different degrees of porosity that consists in forming a metallic body and heat treating it to form large crystals therein; then breaking said body down to its component crystals and crushing said crystals; then screening said crushed crystals to separate the crushed particles into batches of different sizes; then wetting said batches with metallic salts and heating to reduce said salts to oxides, thereby forming an oxide coating on the crystalline fragments; then mixing batches of different sized particles with a binding material and filling said batches into a die in layers of different sized particles and pressing said layers under hydraulic pressure; then heating to volatilize said binding material without leaving any residue and to reduce the oxide coating on said particles to metal; and finally further heating so as to sinter said coated metal



powders into a body comprising layers of different degrees of intercommunicating porosity according to the particle sizes of the crystalline fragments composing each layer.

7. Method of making a metallic body permeated by intercommunicating pores that comprises coating a metal powder consisting of broken crystals with a metallic salt, mixing said coated powder with a volatilizable binding material, pressing said mixture to a desired shape and heating said body so formed first to a temperature sufficient to volatilize said binder without leaving any residue, then to a temperature sufficient to reduce said metal salt coating to metal and then to sinter said coated powder to a strong, coherent, porous body.

8. Method of making a tungsten body permeated by intercommunicating pores that comprises coating a tungsten powder consisting of broken tungsten crystals with silver nitrate, mixing said coated tungsten powder with paraffin, pressing said mixture to a desired shape and heating said pressed body at increasing temperatures to volatilize said paraffin without leaving any residue, to reduce said silver nitrate to a metallic silver coating and to sinter said silver coated particles to a strong, coherent, porous body.

JACOB KURTZ.

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