

Feb. 1, 1966

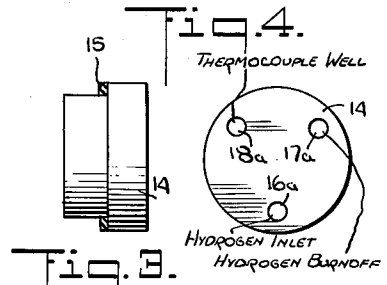
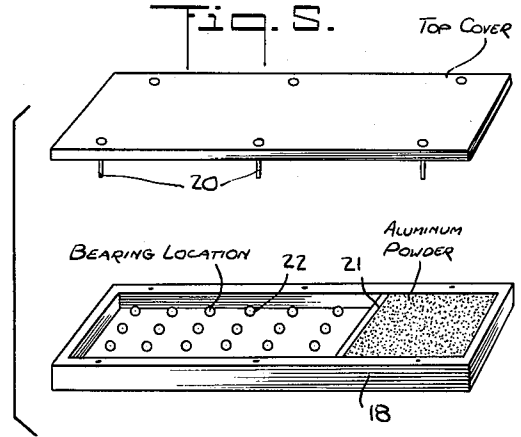
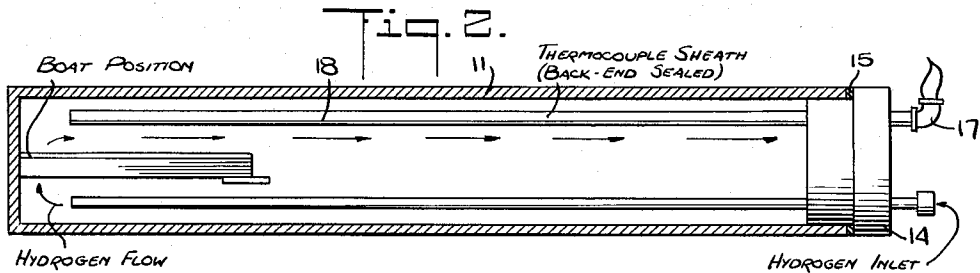
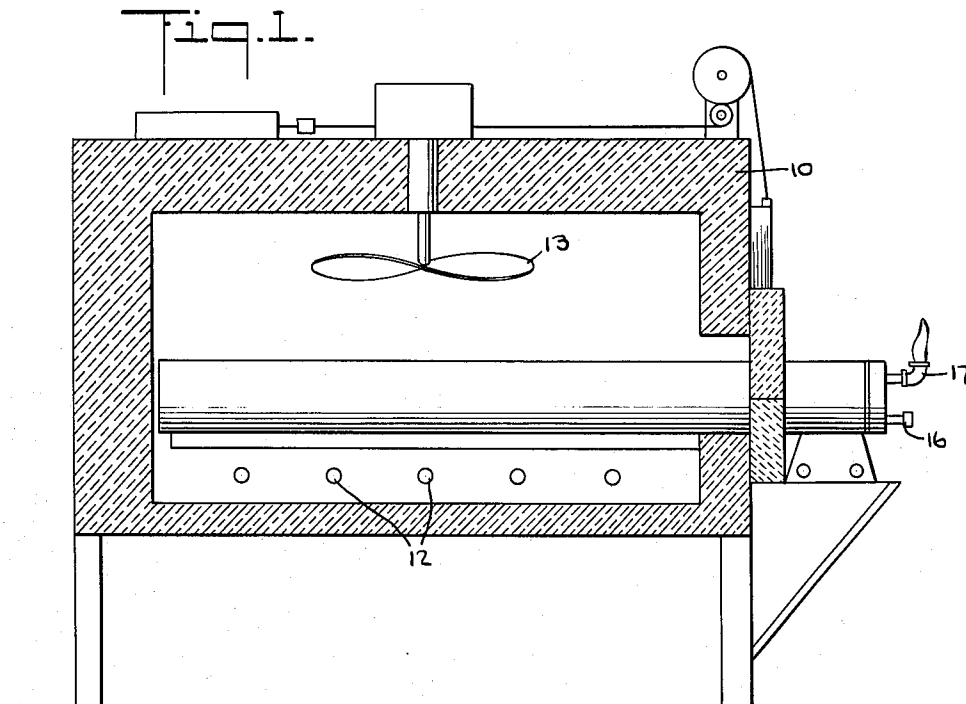
S. STORCHHEIM

3,232,754

POROUS METALLIC BODIES AND FABRICATION METHODS THEREFOR

Filed Nov. 7, 1961

4 Sheets-Sheet 1



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SAMUEL STORCHHEIM

Feb. 1, 1966

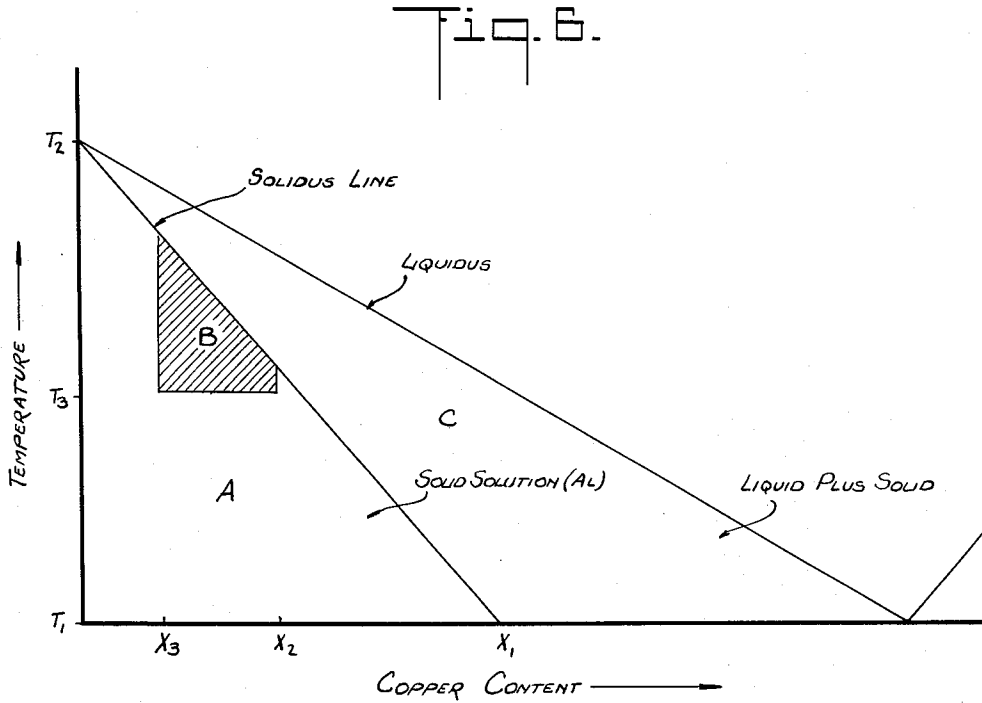
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POROUS METALLIC BODIES AND FABRICATION METHODS THEREFOR

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4 Sheets-Sheet 2



ALUMINUM RICH CORNER OF THE COPPER-ALUMINUM
PHASE DIAGRAM

- A----AREA OF TEMPERATURE-COPPER CONTENT YIELDING INADEQUATE STRENGTHENING BELOW SOLIDUS TEMPERATURE.
- B----AREA OF OPTIMUM COPPER CONTENT-TEMPERATURE FOR CONTROLLED DIMENSIONS AND STRENGTHENING BELOW SOLIDUS TEMPERATURE.
- C----AREA OF EXCELLENT STRENGTHENING, BUT EXCESSIVE SHRINKAGE DISTORTION.
- X₁----5.7% COPPER CONTENT
- T₁----EUTECTIC TEMPERATURE
- T₂----ALUMINUM MELTING TEMPERATURE
- T₃----LOWEST SHORT-TIME SINTERING TEMPERATURE FOR ADEQUATE STRENGTH
- T₂ X₁----SOLIDUS TEMPERATURE LINE

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POROUS METALLIC BODIES AND FABRICATION METHODS THEREFOR

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4 Sheets-Sheet 3

DEPENDENCE OF MAXIMUM EXPANSION ON
COPPER CONTENT AND COPPER PARTICLE
SIZE FOR VARIOUS COMPACT DENSITIES
ALL TYPE H ALUMINUM
SINTERED ONE HOUR IN HYDROGEN

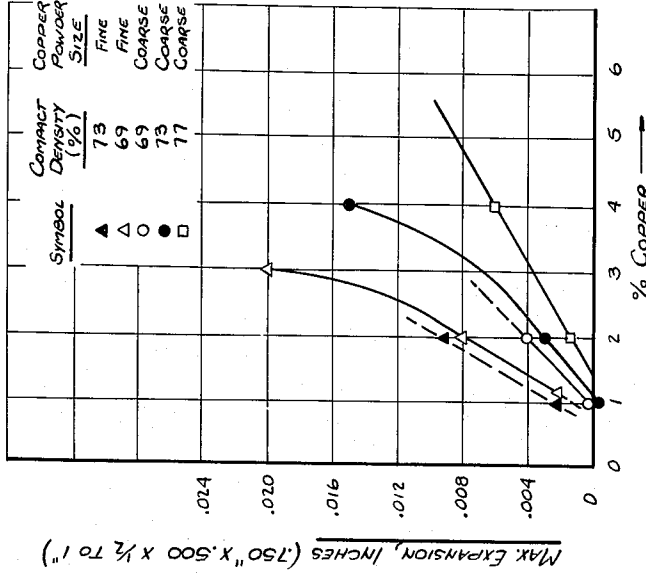


Fig. 6.

EFFECT OF TEMP. VS. DIMENSIONS

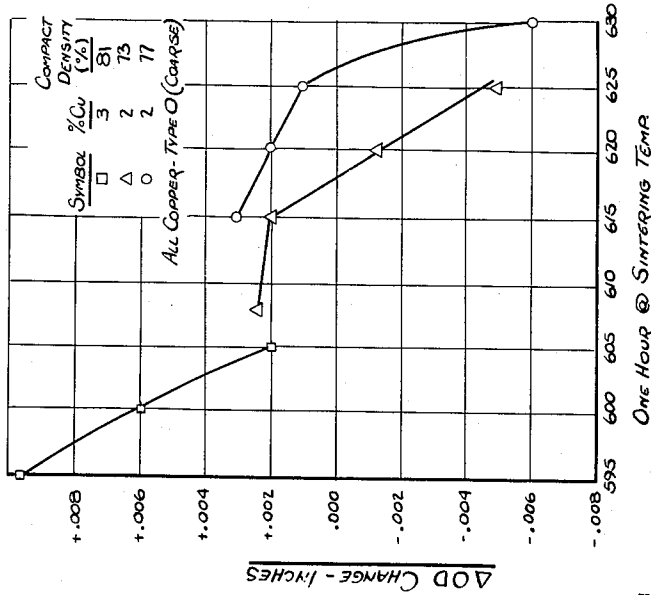


Fig. 7.

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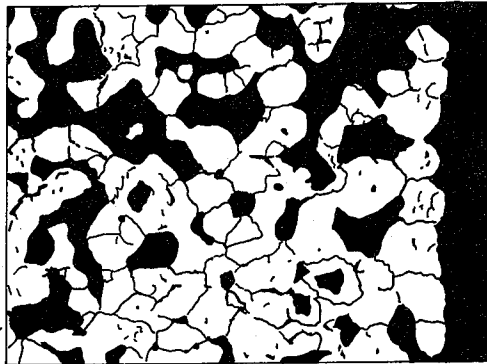
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POROUS METALLIC BODIES AND FABRICATION METHODS THEREFOR

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4 Sheets-Sheet 4

FIG-9

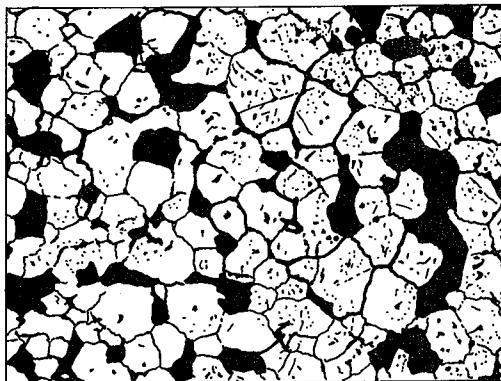


PORE STRUCTURE OBTAINED USING
TYPE O COPPER POWDER

MAGNIFICATION 115X

ETCHED

FIG-10



PORE STRUCTURE OBTAINED USING -325
MESH COPPER POWDER

MAGNIFICATION 115X

ETCHED

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3,232,754

POROUS METALLIC BODIES AND FABRICATION METHODS THEREFOR

Samuel Storchheim, Forest Hills, N.Y., assignor to Alloys Research & Manufacturing Corporation, Woodside, N.Y., a corporation of Delaware
 Filed Nov. 7, 1961, Ser. No. 150,826
 11 Claims. (Cl. 75-214)

This invention relates generally to powder metallurgy and in particular to powder metallurgical methods for fabricating porous and non-porous bodies of various metals such as aluminum and alloys thereof. The present application is a continuation-in-part of my co-pending application Serial No. 53,505, filed September 1, 1960, now abandoned.

The art of powder metallurgy deals with the formation of objects by the heat treatment of compressed metallic powders which may also contain non-metallic substances. This technique is applicable to elemental metal particles or to complex mixtures thereof. In many instances it is possible by powder metallurgical techniques to develop products having properties which are unattainable by the orthodox methods of melting and casting. Thus one may fabricate filters, cermets and self-lubricated bearings as well as many other porous or densified objects. While the present invention will be described in connection with the formation of porous bearings made of an aluminum alloy, it is to be understood that the principles thereof may readily be applied to the formation of other products, and may be used in conjunction with other alloy systems, such as one employing magnesium or titanium as the base metal rather than aluminum.

The conventional practice in making self-lubricating bearings is to press metal powders, such as a copper and tin mixture, into a green compact which is then sintered to form a porous coherent body. Small percentages of graphite and volatile organic compounds are usually included in the powder mixture for the purpose of controlling porosity during the sintering operation. The resultant porosity in the sintered bearing may run as high as 90% by volume. After coining the sintered bearing to its final size it is impregnated with oil. In operation, the oil is exuded from the pores of the bearing to lubricate the shaft journaled therein. As the bearing heats up, the flow-rate of oil increases, hence lower running temperatures favor a longer effective bearing life.

While iron and steel porous bearings as well as bronze bearings are currently in use, because of the problem peculiar to the fabrication of porous aluminum and its alloys, porous aluminum bearings have not heretofore been commercially available. These problems are incidental to certain characteristics of aluminum.

As is well known, aluminum particles exposed to air form an adherent oxide film of a refracting nature, thereby interfering with effective sintering of the particles. In order to overcome the resistance due to the presence of this oxide film, prior attempts to sinter aluminum powder mixtures made use of relatively high compacting pressures; ranging from 20 to 60 t.s.i. But since aluminum is a soft material which flows readily under pressure, the application of high compacting pressures created difficulties in producing continuous interconnecting pores capable of providing proper oil storage and lubricating properties essential to the production of bearings.

Another serious problem encountered with high compacting pressures is that of die wall pick-up and ultimate seizure in the absence of wall lubricants. While such pick-up can be minimized by the use of die wall lubricants, this adds materially to production costs, for the dies then require wiping, and tolerances are harder to control. On

the other hand, where in previous attempts lubricants such as stearates have been mixed with the metal powder, the consequence thereof has been discoloration, oxidation and lower strengths of the sintering compacts.

Another prior approach to the formation of porous aluminum alloy bodies has involved elevated sintering temperatures above the liquid phase. The drawback of this approach is that it gives rise to excessive shrinkage and distortion in the parts. But where solid state sintering has been tried on aluminum alloys at temperatures below the formation of a liquid phase, this has complicated the oxidation problem, for the use of prolonged sintering periods, required by the slower diffusion at low temperatures, causes more oxidation to take place per unit volume of powder.

Another factor which has militated against the successful production of porous aluminum bodies is the inclusion of relatively fine (-325 mesh) powder in the mixture. It has been found that this is one of the major causes of die wall pick-up as well as oxidation during sintering, simply by reason of the vast surface areas presented by powder fines. Also the use of such finely divided powders is hazardous, for they have a tendency to explode, and fires may occur in normal production.

Thus while many attempts have hitherto been made to fabricate porous bodies constituted by aluminum mixtures, expedients applied to overcome particular problems served only to generate new problems and the results have been a commercial failure.

Accordingly, it is the main object of this invention to provide a novel and effective method for fabricating products of aluminum, which method is low cost, simple and efficient and requires neither complicated apparatus nor highly skilled operators.

More specifically it is an object of the invention to provide a method for fabricating porous aluminum bodies having porosities ranging from 10 to 50 volume percent, the bodies being controlled in dimension and being characterized by improved crush strength and good deflection.

A significant feature of the invention is that it facilitates the mass production of commercially acceptable aluminum alloy self-lubricating bearings having advantages lacking in bearings made of other metals. Aluminum alloys have a relatively high thermal conductivity, thereby improving heat dissipation. At the same time they have both a low modulus and softness so that the bearings will not bear overly hard and will wear when subjected to localized pressure. As a consequence, localized pressure is relieved and the deleterious effects of misalignment and shaft deflection are reduced. Furthermore, a soft material such as aluminum will embed grit and thus aid in avoiding hot spots leading to bearing failure. In addition, aluminum bearings have excellent corrosion resistance and high fatigue strength.

Another object of the invention is to provide light weight aluminum bodies which are highly useful as structural elements, particularly in the cryogenic field, for aluminum is not rendered brittle at extremely low temperatures.

A significant feature of the invention is that the porous aluminum body may be formed into a small wafer for use as a nicotine filter in a cigarette or any smoking appliance.

Still another object of the invention is to provide an improved process for fabricating porous aluminum bodies which process makes use of relatively light compacting pressures in conjunction with unlubricated dies, lubricants being added only to the powder mixture in a manner whereby discoloration and oxidation of the sintered parts do not occur.

For a better understanding of the invention, as well as other objects and further features thereof, reference is made to the following detailed description to be read in conjunction with the accompanying drawing, wherein:

FIG. 1 is a sectional view of a furnace for carrying out sintering in accordance with the invention.

FIG. 2 is a separate view, in longitudinal section, of the retort used in the furnace.

FIG. 3 shows the closure for the retort, in side view.

FIG. 4 shows the same closure in end view.

FIG. 5 is a perspective view of the boat or covered tray placed within the retort to house the bearings.

FIG. 6 is a graph explanatory of the invention, showing a copper-aluminum phase diagram.

FIG. 7 is a graph indicating the effect of sintering temperature on the bearings.

FIG. 8 is a graph showing the effect of the copper content and particle size on the bearings.

FIG. 9 is a photograph of the pore structure using one type of copper powder.

FIG. 10 is a photograph of the pore structure using another type of copper powder.

THE POWDER MIXTURE

In general, the process for making porous aluminum alloy products involves the steps of compacting a powder mixture into the desired form, and the green compact under such condition of temperature as to cause sintering of the powders into a coherent but porous mass.

Consideration shall first be given to the nature of the powders employed in accordance with the invention. The mixture is composed of aluminum and copper powders to which an organic lubricant is added. The aluminum constituent of the powder mixture should be one having a very low oxide content. Relatively coarse aluminum powders have lower oxide contents because the surface areas are relatively small per unit weight (less than .3%). Preferably it is prepared by atomizing molten aluminum with an inert gas, such as helium, neon, argon, krypton or nitrogen. I have found, for example, that helium-atomized aluminum powder has a very low oxide content as compared to air atomized powder and consequently will sinter well.

I also prefer to make use of aluminum powder in which fines are virtually eliminated so that the fraction of powder of minus 325 mesh size ranges from 0.0 to almost about 0.4 weight percent. Three suitable examples of helium-atomized aluminum powder are given below in Table I in terms of mesh size percentages.

Table I

Type.....	A	B	C
Screen analysis (percent)—			
Mesh size:			
+100.....	0.501	2.0	1.73
-100+140.....	41.5	35.4	22.98
-140+200.....	49.8	44.0	48.5
-200+270.....	8.03	16.9	18.6
-270+325.....	.250	1.31	1.16
-325.....	0	0.402	0.302

These powders are free of fines, and die-pick-up in compacting is thereby obviated. Another significant point is that the flow rate of air atomized powders has been found to be slower by a factor of about two. This means that less compacts can be produced in a given time in an automatic press. Also the helium-atomized aluminum powder has a somewhat higher density.

To make the helium-atomized aluminum powder, molten aluminum is atomized in a helium atmosphere and cooled in the same atmosphere, oxidation thereby being avoided.

In the case of the copper powder, I prefer to use relatively coarse powder of good purity coupled with a high density and good flow rate. Suitable for this purpose is an electrolytic copper powder (type "O") which in screen

analysis has 60 to 75% of -80+100 mesh size, 20 to 35% of -100+150 mesh size and .25% maximum of -150+200 mesh size.

The lubricant which is supplied to the powder mixture is preferably in the form of Sterotex which is added in small amount solely for the purpose of eliminating die friction problems and the prevention of powder pick-up by the die. I have found that the addition of at least 1% weight percentage of Sterotex powder acted to limit the pick-up problem, whereas optimum compaction was obtained at 2%. Sterotex is a refined vegetable oil produced by Capital City Products Co. of Columbus, Ohio, and has the following properties and characteristics.

Color (Lovibond)—20 Yellow	1.2 red.
Free fatty acid (as oleic)03.
Melting point (capillary)	143-144% F.
Setting point	69% C. plus.
Iodine number	2-10.
Through:	
100 mesh (wet method)	99.4%.
200 mesh (wet method)	92.8%.

The helium aluminum powders are thoroughly mixed with the powders in a ratio in which the copper content is not in excess of 5% by weight and preferably in which the copper content is in the range of 1/4 to 2/4 weight percent copper. The Sterotex lubricant is added to this aluminum-copper mixture in a range of 1 to 3% weight percentage.

COMPACTING

Let us assume by way of example that a bearing is to be produced having of an O.D. of .750" and an I.D. of .4985". The powdered mixture is first placed in a die capable of producing the required outside diameter and a core rod is inserted therein to establish the required inside diameter. To maintain uniform densification throughout the compact, equal punch movement by top and bottom punches is used. Green densities of 69 to 89% of theoretical (2.74 g./cc.) is attained at compacting pressures of 3 to 7 tons per square inch (t.s.i.). No lubrication is necessary other than that provided by the Sterotex in the powder mixture.

Thus obtained from the die is a green compact in the shape of a bearing composed of an aluminum-copper mixture.

Despite the low compacting pressure, the nature of the powder mixture is such that the green compact is sufficiently consolidated for further handling without loss of integrity.

SINTERING

The compact is now ready for sintering. The furnace used for this purpose is illustrated in FIG. 1 and comprises an insulated chamber 10, into which is insertable a retort 11, the chamber being provided with suitable heating element 12 and a circulating fan 13. As shown separately in FIG. 2, the retort 11 is in the form of an elongated rectangular box the rear end of which extends outside of the furnace, the rear end being sealed by a removable closure 14 provided with a gasket 15. The retort is filled with an atmosphere of hydrogen through an inlet tube 16, the hydrogen passing out of the retort and being burned off through an outlet jet 17. The temperature in the retort is measured by means of a sheathed thermocouple 18. This is best seen in FIGS. 3 and 4. The closure is provided with three bores 16a, 17a and 18a for receiving the tubes passing therethrough.

Placed within the retort 11, adjacent the front end thereof, is a boat 18 provided with a removable cover 19, locating pins 20 projecting from the cover which are receivable in correspondingly positioned apertures in the side walls of the boat to hold the cover in place. The boat is divided by a partition wall 21 into a main section for accommodating green compact bearings 22 to be sintered, and an auxiliary section filled with aluminum

powder acting as a getter to pick up oxygen and moisture.

The arrows in FIG. 2 indicate the direction of hydrogen flow in the retort. The covered boat 18 is not sealed from the hydrogen, but free circulation and turbulence of the gas within the boat is inhibited. It has been discovered that use of heavy walled iron boats and covers is essential to the production of clean sintered bearings free from any evidence of contamination. It has also been found that thin walled boats and covers are not as effective as those making use of heavier metal, for the temperature gradients in the furnace cannot be absorbed to prevent dumbbell distortion in the bearings.

Following completion of sintering, the retort is water quenched. While sintering has been disclosed as carried out in a dry hydrogen atmosphere, nitrogen or any inert gas as well as vacuo can be used for sintering. The surface disclosed herein is for purposes of illustration, and other furnace arrangements are feasible, such as pot-types, as long as the principles disclosed above are maintained.

Table III.—Summary of sintered densities and crush strengths for various temperatures and green densities (2% Cu-containing bearings)

Temp., °C.	Green density (percent)	Sintered density (percent)		Crush strength, p.s.i.		Dim. change, in. on O.D.	
		Coarse Cu ²	Fine Cu ²	Coarse Cu ²	Fine Cu ²	Coarse Cu ²	Fine Cu ²
500	69	67.5		1,150		+ .003/+ .005	
595	69	67-69		1,948		+ .001/+ .006	
600	77	³ 73.6-74.8		³ 6,900-8,270		³ + .002/+ .004	
601	73	73.1-73.7	73.4-73.6	2,820-3,590	2,240-3,890	+ .001/+ .003	+ .002/+ .003
601	77	75.4-76.4	74-77.1	3,280-3,740	18,650-20,900	+ .001/+ .002	- .016/- .003
605	69	68-69	66-67	3,380-3,380	2,200-2,520	+ .002/+ .006	+ .006/+ .010
605	73	72.5-73.5	73-73.5	2,350-2,650	1,300-1,680	+ .001/+ .003	+ .003/+ .004
605	77	75.1-75.8	72.1-75.1	2,020-3,950	10,100-19,800	+ .002/+ .004	- .007/+ .007
606	73	73.9-74.2	73.9-74.9	2,440, 3,460, 5,380	2,240-3,660	- .002/+ .001	+ .002/+ .003
606	77	75.5-76.8	75.5-78.6	3,150-4,180	20,900-23,100	.000/+ .001	- .017/- .001
610	73	72.5-73.4	73.0	2,920-2,940	1,840-1,680	+ .001/+ .004	+ .003/+ .006
610	77	75.4-76.0	72.1-74.6	1,920-2,100	10,610-11,180	+ .001/+ .002	- .002/+ .011
612	77	³ 75.0-75.9		³ 10,350-12,680		³ + .001/+ .004	
615	69	71.0		16,120		- .003/- .010	
615	73	72.3-72.7	71.5-72.3	4,380-5,170	3,060-3,570	+ .001/+ .003	+ .005/+ .007
615	77	72.4-75.7	78	10,300-12,420	22,800	+ .001/+ .003	- .019/- .009
620	73	71.8-73.6	70.8-74.0	7,900-9,680	7,160-8,600	- .004/+ .004	+ .001/+ .011
620	77	74.0-76.2	80	5,100-6,180	24,700	+ .001/+ .005	- .019/- .025
625	73	72.7-76.0	72.9-74.5	14,900-15,200	6,720-8,700	- .007/+ .003	- .004/+ .007
625	77	73.5-76.1	81	6,300-6,600	27,000	+ .001/+ .003	- .004/- .011
630	77	76.5-78.1		16,700-19,800		- .004/- .011	- .012/- .037
630	81	80.1-82.1		22,500-22,800		- .001/+ .007	

¹ Sintering time, 1 hr., in all cases.
² Coarse copper—"O" type. Fine copper—"90" type.
³ Sintered in Hevi-Duty furnace.

For example, a continuous belt furnace may be used in conjunction with boats or trays for the bearings.

The results shown in Table II below are for "O" type of copper powder (4%), as defined previously, mixed with helium-atomized aluminum powder in which fines are eliminated, the mixture being compacted in the manner previously described to form a porous bearing. Samples in three green compact densities (69, 73 and 77% theoretical) were sintered in iron boats as described above at various temperatures ranging from 588° C. to 595° C. The resultant crush strength and dimensional characteristics were excellent, as indicated by the optimum sintering temperatures and properties listed below for various green densities.

Table II

Green density, percent	Sintering temperature, °C.	Crush strength, k.s.i.	Dimensional change (x.001")	
			O.D.	I.D.
69	588	12.3-13.7	+1	0
73	590	13.4-15.9	+2	+1
77	592-595	16.2-19.75	+6	+3

It is to be noted that in the results set out in Table II the final dimensions were expanded several thousands of

an inch. This provides good coining characteristics, as will be discussed later.

Table III demonstrates the difference in results between using O type coarse copper and a finer copper hereinafter referred to as "90" type (.1% of -80+100, .5% of -100+150, 4.0% max. of -150+200, 1.5% max. of -200+250, 2-7% -250+325, 90% min. of -325 mesh). In both cases the percentage of copper in the mixture was 2% by weight relative to the aluminum.

It will be seen that excellent dimensional control is experienced with the O type powder independent of crush strength. It will be further noted that comparable results were obtained for sintering 77% green density bearings in two different furnaces. The only apparent differences between furnaces is a 3° C. temperature differential. But under the same sintering conditions the "90" type powder exhibited excessive shrinkage which is a definite disadvantage in coining operations.

In Table III, the results are for 2% copper. To show the sintering effect when using smaller or larger increment of copper, Table IV below summarizes the results obtained with type "O" copper in a range of 0 to 4 weight percent relative to the aluminum powder.

Table IV

Copper, w/o	Crush strength, k.s.i.	Green density (g./cc.)	Sintered density (g./cc.)	Sintering temperature, °C.
0	19.41	77	80	650
1	13.14	73	72.5	640
1 1/4	6.60	77		615
1 1/2	8.80	77		615
1 3/4	10.30	77		615
2	12.42	77	75.7	615
2 1/4	13.80	77		615
3	124.20	77	85.4	620
65	119.75	77		592-595

¹ Samples showed excessive shrinkage.

It will be evident from Table IV that adequate strength coupled with dimensional control is attained wherein the copper content lies in the range of 1 1/4 to 2 1/4 weight percent copper.

THE SINTERING MECHANISM

I have found that by sintering above the eutectic temperature but below the solidus temperature, preferably

utilizing very dry (-80 to -100° F.) dew point hydrogen, one may eliminate or minimize the problems encountered by previous investigators relative to contamination and distortion.

FIGURE 6 summarizes my results on sintering in a schematic representation of the pertinent corner of the Aluminum-Copper phase diagram at temperature ranging from the eutectic up to the melting point of the major alloying element, aluminum. Area B on the diagram defines the optimum range of temperature and composition which is requisite to producing Al-Cu bearings or other porous structures having excellent strength and dimensional control. It is noted that for lower percentages of copper in this range the allowable sintering temperature range increases upward. This is significant, in that, at the higher temperatures, approaching the solidus, for these compositions the sintering time can be reduced considerably without a loss in crush strength. This results in more economical production of parts.

As far as the sintering mechanism is concerned, additions of copper to aluminum powders up to near 5.7 weight percent (the maximum solubility of copper in aluminum at the eutectic temperature) allow a low melting point eutectic to form at 548° C. for a short period of time. This liquid phase (Al-Cu) apparently is capillary drawn throughout the structure of compacted elemental powders and fluxes away any residual aluminum oxide on the surface of the aluminum particles. During these early stages of the sintering cycle the compact apparently expands, the amount of expansion depending on the copper content which controls the quantity of liquid phase present above the eutectic temperature.

As the temperature and time increase and all liquid has solidified on the particle surfaces, rapid diffusion of aluminum and copper continues in the direction of equilibrium concentrations. The maximum temperature that can be used for sintering, once diffusion is relatively complete, is given by the phase diagram as the solidus temperature for the alpha aluminum phase. If this temperature is exceeded, extensive shrinkage and distortion occurs in the compacts.

If excessive copper is added and the final sintering temperature is below the solidus, a grossly expanded compact results after sintering. As the sintering temperature nears and/or exceeds the solidus temperature, for such a copper content, shrinkage occurs rapidly and little or no dimensional control can be attained (see FIGS. 7 and 8). Note in FIG. 7 that the curve for 3% copper has a steep slope which limits the control attainable. FIGS. 7 and 8 also indicate the desirability of using coarse copper powders.

Further, FIG. 7 indicates that when copper content and particle size are properly chosen, a plateau in the curve results which allows for dimensional control over a range of temperatures below the solidus. Note that for 2% copper, the final compact size is slightly expanded, which has been found to be ideal for subsequent coining operations.

The following generalizations for sintering elemental powders can be said to apply in the early stages:

(1) The coefficient of diffusion depends on the concentration gradient.

(2) The coefficient of diffusion is an exponential function of the absolute temperature—i.e., the diffusion rate increases rapidly with temperature.

(3) In general, the diffusion rate of an individual metal into a given lattice is higher, the closer the temperature to the melting point. In a binary system, the lower-melting constituent will, therefore, show the higher diffusion rate at a given temperature.

(4) The rate of homogenization-equilibrium by diffusion-during sintering of mixed powders depends on the particle sizes, which determine the distances between maximum and minimum concentrations. With a given mixture

the most rapid homogenization occurs when the particles of the minor constituent have the smaller size.

(5) In general, the minor component of a binary system will become more quickly alloyed than the main constituent, the diffusion layers forming envelopes about the particles of the minor component.

As the process proceeds and the compact nears the final sintering temperature (above or below the solidus), growth and shrinkage processes start to take effect. Here powder characteristics once again show a significant effect. It has been established that two different mechanisms govern the shrinkage mechanism, namely volumetric particle shrinkage or packing effects which change particle shapes and/or relative position of particles, with particles volume remaining constant. These effects can overlap each other and it has been determined that the finer-grained powders augment these effects. In the case of coarse powders, the pores produced in the green compacts will not permit extensive densification by the packing mechanism.

In summary, it is observed that copper additions to aluminum provide faster sintering, and better strengthening for lower sintering temperatures than is the case for pure aluminum powders. Copper also limits contamination from sulfides or oxides which might be present on powder surfaces and such additions make possible sintering of low pressure compacts.

It has been found also that the use of coarse aluminum powders in the production process is important in that it eliminates explosion hazards normally present when handling finely divided powders having large surface areas. This is particularly true when using hydrogen atmospheres.

THE SINTERED STRUCTURE

FIGURES 9 and 10 are a comparison of the pore structure obtained with the coarse and fine types of copper powders. It is apparent that coarse pores are randomly distributed throughout the structure when coarse copper powders are utilized. It has been explained in the discussion of the sintering mechanism that fine copper powder additions lead to densification and finer pore sizes. Coarse, randomly distributed holes are beneficial to the operation of bearings in that the coarse pores provide wells for storage of oil which can be fed to the fine capillaries formed by the finer particles. In addition, this type of structure has the advantage that the large holes on the surface are more difficult to close by burnishing or wear, which provides more continuous lubrication properties and a greater factor of safety in operation and in changing bearings.

Further, the better the surface finish and the more uniform the dimensions the lower will be the PV temperature. This is accomplished by burnishing which "iron-out" non-uniformity in dimensions and provides a smooth polished bearing I.D.

IMPREGNATION AND COINING

As an example of the effects of coining and oil impregnation, 4% copper compacts having a sintered density of 69.7% of theoretical and somewhat expanded dimensions were impregnated with oil and then coined. Oil impregnation took place under vacuum for $\frac{3}{4}$ hour and coining was at 14 t.s.i. Density increased to 75.7% and crush strengths rose from 13,700 p.s.i. to 25,000 p.s.i. while deflection dropped from 7.9 to 1.6%.

It is noteworthy that impregnation can take place prior to or following the coining operation. Impregnation prior to coining results in very uniform dimensions after coining. The oil acts as a hydrodynamic pressure equalizer which results in more uniform application of coining pressure. It has also been found that dip impregnation is unsatisfactory in that considerable length variations result after coining. Also the end porosity tends to close up in coining dip impregnated samples. As a result vacuum impregnation has become a standard operation. It is also

possible to impregnate the pores with lead to prevent burn out of the bearing should the oil be exhausted.

From the data obtained for sintering, it is also apparent that using proper composition and sintering conditions, adequate crush strengths with good deflection and dimensional control are attainable without coining. Therefore it is feasible to eliminate the coining operation to bring the cost of production down. All that is necessary with such bearings is a button burnishing operation to assure uniform dimensions and a smooth surface.

OTHER ALLOY SYSTEMS AND PRODUCTS

I have found that other metals in elemental powder form can be blended with aluminum to form good sintered compacts providing that a low melting point constituent exists and that the solidus temperature line in the equilibrium phase diagram is established to control maximum sintering temperature. The following elemental additions were tested:

Element or alloy added:	Weight percent added
Tin -----	1.5
Titanium -----	1.5
Molybdenum -----	1.5
Magnesium -----	1
Magnesium (ZK-10) -----	2.4
Iron -----	1.5
Nickel -----	1
Lead -----	1.5
Chromium -----	1.5
Cobalt -----	1.5
Copper plus nickel ----- (each)	4
Brass -----	1

Results show that 1% tin sintered at 625° C./1 hr. has a crush strength of 11,310 p.s.i. and deflection of 9.89%. Aluminum-tin samples have been vacuum and hydrogen sintered in producing satisfactory parts. Magnesium (ZK-10) additions of 4% and sintered at 600° C./1 hr. yielded crush strengths ranging from 14,000 to 15,300 p.s.i. with deflections 7.43 to 9.50 p.s.i. Additions of 4% each of copper and nickel sintered at 600° C./1 hr. had crush strength ranging from 12,010 to 13,880 p.s.i. coupled with deflections of 5.08 to 5.50%.

It has been noted that magnesium apparently volatilizes to some extent in the sintering operation leaving behind appropriate pores depending on the original metallic particle size. This has also been noted for Sterotex, so that it can be concluded that both metals and non-metals can be used to produce requisite porosities.

Porous structural parts can be produced by using an aging treatment following the sintering operation. For example, an aluminum sample containing 4% copper was sintered to a density of 86.3%, having a crush strength of 30,300 p.s.i. and a deflection of 12.1%. Heat treating at 500° C. for 1¾ hours, followed by water quenching and then vacuum heat treating at 150° C. for 2 to 89 hours results in strengths up to 35,700 p.s.i. with deflections of 4.1%.

In addition to the above, it has been discovered that mixed elemental powders can be loaded into boats, levelled off and sintered to high strengths without prior compacting. For example, a mixture of 4% copper, balance aluminum (all coarse powders) was intimately mixed for ½ hour, loaded into boats, levelled and sintered, as previously described, at 620° C. for one hour. The result was a porous, high strength sinter-cake product. Helium or air atomized aluminum can be used. If more strength or density is required, the cake could then be coined or rolled, cold or hot. These sintercakes can also be impregnated with lead. The sintercake can be consolidated into full density structural parts by hot or cold rolling or extrusion. Such parts can also be heat treated and aged to develop requisite structural properties.

A tobacco smoke filter may be constructed by forming a highly porous plug of aluminum alloy fabricated in the

manner described above. The green compact may be very lightly compacted to produce a high degree of porosity, the sintered product having interconnected parts permitting the flow of smoke therethrough. The plug may be inserted in a cigarette holder or in the stem of a pipe. The filter may also be in the form of a small wafer inserted in the tip of a cigarette, the tip being of the type conventionally now in use with fibrous filters. The advantages of aluminum smoke filters are that by reason of their high conductivity, they act to cool the smoke, to condense harmful nicotine-containing vapours and to filter out smoke and tobacco particles. On the other hand the filter will not affect the taste or odor of the tobacco smoke. A filter of this form is of negligible weight and may readily be inserted in a cigarette tip.

SUMMARY

This process disclosed herein makes possible the production of porous metal parts having porosities ranging from 10 to 50 volume percent coupled with improved crush strength, good deflection and excellent dimensional control. The new process offers a number of technical and economic advantages over methods established by others. Solutions to the problems which existed heretofore and the advantages of the new process are in the following points:

Point 1.—The problem of sintering atmosphere causing contamination of the part. This is accomplished by the use of a protective sintering tray for containing parts in the furnace and by employing an alloying element, such as copper, which forms a low melting constituent (in the early stages of sintering) and acts as a fluxing agent on the surface of the powder. It is also to be noted that the corrosion resistance of the Al-Cu, or other, solid solution formed on the surface of the powders is superior to pure aluminum. Such sintering has been accomplished successfully in various atmospheres, namely, dry hydrogen, nitrogen and vacuum.

Point 2.—The problem of refractory oxide or other contaminant films on the surface of elemental powders which cannot be reduced at the sintering temperatures employed. This result has been achieved by providing a low melting fluxing agent as noted in Point (1) above. Also the degree of fluxing required can be controlled by the alloying content. For example, if a very pure powder is utilized (e.g., helium atomized aluminum) loss of the constituent for fluxing (e.g., copper) need be added. The purity of the powder, in turn, is controlled by eliminating particle fines (—325 mesh). This can be done for both the major and minor alloying constituent.

Point 3.—The problem of die wall pickup and seizure in pressing green compacts. This problem is solved by utilizing low compacting pressures, 3 to 7 t.s.i., and by powder particle size control to provide optimum compactibility. Also the successful application of a powder lubricant is very helpful. The use of die lubricants, which would be necessary if high compacting pressures are used and/or the powder could not be lubricated, leads to more cost in production in that dies require wiping, wear would be greater, tolerances would be harder to control, etc. The use of low compacting pressures creates the possibility that dies might be manufactured from plastic materials. In any event, the use of tool steels is eliminated and cheaper alloys can be used, such as cold rolled steel.

Point 4.—The problem of using lubricants mixed in with the powders. Approximately 1 to 3% Sterotex mixed with the elemental powders combined with the use of the special sintering tray arrangement in the sintering operation has eliminated discoloration and oxidation of sintered parts.

Point 5.—The problem of long, costly sintering times encountered in completely solid state sintering below any liquid temperature. Sintering times are shortened to those that are economically feasible by sintering above

the eutectic temperature and just below the solidus temperature.

Point 6.—The problem of excessive shrinkage distortion resulting from liquid phase sintering. This problem was eliminated by maintaining the sintering temperature below the solidus temperature. Where absolutely necessary to use liquid phase sintering (e.g., for 4% Cu), it is possible to obtain results by careful control of the time cycle, particle size and the quench cycle.

Point 7.—The problem of controlling dimensions in sintering to obtain proper sizing control. By producing a slightly expanded sintered part, the coin-out of parts has been facilitated in this investigation. This is done by proper selection of copper content, particle size, and sintering conditions such as temperature and time.

Point 8.—The problem of producing adequate crush strength simultaneously with reliable dimensional control. This is accomplished by using coarse copper powders and maintaining sintering temperature below the solidus for proper length of time.

Point 9.—The problem of control of pore size and production of randomly distributed coarse pores having a gradation of finer pores. This problem has been overcome by utilization of coarse aluminum powder and adding the proper percentage of coarse copper powder which maintains the pore size during the sintering operation.

Point 10.—The problem of producing sintered parts having no contamination after low pressure compacting (3–7 t.s.i.). It is now possible to eliminate contamination despite low compacting pressures by utilizing the sintering tray and fluxing constituent as detailed above.

Point 11.—The problem of storage of mixed powders. This is facilitated by the use of coarse aluminum and copper powders, thereby limiting the exposed surface area of particulates.

Point 12.—The problem of explosion hazards encountered with aluminum powders and various atmospheres. By elimination of fines (–325 mesh) in the powder mixtures particulate surfaces are stabilized so that ignition and/or explosive tendencies are eliminated.

Point 13.—The problem of producing non-contaminated sintercakes directly from powders with no pre-compactation. This can be done by coating the major alloying element particles with the minor alloying elemental powder (e.g., copper coated on aluminum) and sintering at an appropriate temperature and time. Particle size is controlled to develop proper strength and porosity, if required.

Point 14.—The use of aluminum and its alloys offers a number of advantages:

(a) Aluminum and its alloys are softer and less elastic than bronze, for example, and therefore offer less dimensional springback when loading into housings, etc. In the case of Al-Cu bearings, the close-in is only 40% while bronze bearings show a close-in of 77% or approximately twice as much.

(b) Bearings made from aluminum operate at lower temperatures in PV tests than equivalent-size bronze bearings. The temperature differential depends on the PV value of the test.

(c) Because the density of aluminum is approximately $\frac{1}{3}$ that of bronze, the power required to sinter aluminum is much less.

(d) The good thermal conductivity properties of aluminum allows more uniform heat-up in the sintering cycle, which is beneficial for short-time cycles.

(e) Coupled with the above points aluminum has the following properties beneficial in bearing applications: excellent corrosion resistance, high fatigue strength, high compression yield strength, good embedability, good conformability, wear resistance, good antiseizure characteristics, high thermal conductivity and low cost.

Point 15.—The following summarize the advantages of porous bearings in accordance with the invention, exclusive of those cited in (14) above.

(a) Coarse pores in the bearing are beneficial for the storage of oil which can be fed to the fine capillaries. Also this type of structure can be sized readily by various processes (e.g., burnishing) without danger of sealing off the oil supply.

(b) It is possible to produce bearings for use either coined or uncoined. All that is required with an uncoined bearing is a burnishing or similar operation prior to use. The elimination of the sizing operation aids the economics of the process.

Point 16.—Other miscellaneous processing advantages afforded by the process are:

(a) The compaction method eliminates high pressure ejection and oxidation problems encountered by others.

(b) By using the protective means detailed for sintering it is unnecessary to make high initial furnace investments to produce such bearings in quantity.

(c) Metallic (e.g., magnesium) or non-metallic (e.g., Stercotex) can be added in controlled amounts to volatilize or melt during the sintering operation and contribute to production of a proper gradation of pores.

While there has been shown what are considered to be preferred methods in accordance with the invention, it will be appreciated that changes may be made therein without departing from the essential features of the invention as set forth in the appended claims.

What is claimed is:

1. The method of forming a porous sintered aluminum structure substantially free of contamination and distortion comprising forming a powdered metal mixture whose metallic components consist essentially of particulate aluminum, from 1% to 5% by weight of particulate copper, shaping the mixture and heating the shaped mixture to a sintering temperature above the eutectic temperature but below the solidus temperature for the particular composition, the rate of heating being such that a copper-aluminum liquid phase is formed which thereafter solidifies as equilibrium conditions are achieved, thereby bringing all increments of the mixture above the eutectic composition and below the solidus level, and maintaining the sintering temperature for the period of time necessary to achieve sintering, the heating being carried out in an atmosphere, selected from the group consisting of inert gases and hydrogen, whose dew point is at least as dry as -80° F., any compaction of the mixture being carried out prior to heating.

2. The method as set forth in claim 1 wherein the powder metal mixture is shaped and compressed prior to heating by the application of pressure within the range of from about 3 to 7 t.s.i. and the copper is present in an amount of from about $1\frac{1}{4}\%$ to $2\frac{1}{4}\%$ by weight, whereby a porous product is obtained.

3. The method as set forth in claim 1 wherein said particulates are relatively coarse and are free of particles having less than a 300 mesh size.

4. The method as set forth in claim 1 wherein said primary metal particulates have a particle size of about 100 to 300 mesh and an oxide content of less than .3% by weight.

5. The method, as set forth in claim 1, wherein said copper content is in the range of $1\frac{1}{4}\%$ to $2\frac{1}{4}\%$ by weight.

6. The method, as set forth in claim 1, wherein the temperature of sintering is in the range of 550° to 625° C.

7. The method, as set forth in claim 1, wherein said sintering is carried out in hydrogen.

8. The method as set forth in claim 1, wherein said mixture includes an organic lubricant in an amount facilitating compaction thereof.

9. The method as set forth in claim 1, wherein the metals in said mixture are in pre-alloyed particulate form.

10. The method of forming a porous sintered aluminum bearing substantially free of contamination and distortion comprising forming a powdered metal mixture whose metallic components consist essentially of particu-

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late aluminum with from 1¼% to 2¼% by weight of particulate copper, said mixture also including an organic lubricant in an amount up to 3% by weight, shaping said mixture with an applied pressure not exceeding 7 t.s.i. to form a green compact having the general shape of the required bearing, heating said green compact to a sintering temperature above the eutectic temperature but below the solidus temperature for the particular composition, the rate of heating being such that a copper-aluminum liquid phase is formed which thereafter solidifies as equilibrium conditions are achieved, thereby bringing all increments of the mixture below the eutectic composition and below the solidus level at the particular temperature, and maintaining the sintering temperature for the period of time necessary to achieve sintering, the heating being carried out in an atmosphere, selected from the group consisting of inert gases and hydrogen, whose dew point is at least as dry as -80° F., any compaction of the mixture being carried out prior to heating.

11. The method of claim 10 wherein said copper particulates are relatively coarse to produce coarse pores in said bearing.

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References Cited by the Examiner

UNITED STATES PATENTS

2,155,651	4/1939	Goetzel	75—200
2,241,095	5/1941	Marvin	75—200
2,287,251	6/1942	Jones	75—200
2,746,741	5/1956	Naeser	266—5
2,746,742	5/1956	Comley	266—5
2,994,606	8/1961	Goodzeit	75—214
3,007,794	11/1961	Gordon et al.	75—214

OTHER REFERENCES

Goetzel, Treatise on Powder Metallurgy, vol. 2, Interscience Publishers, Inc., N.Y., 1950, pp. 523-527 and 722-723 and 838.

Groom et al.: Developments in Powder Metallurgy, The Metal Industry, Jan. 28, 1938, pp. 131-133.

Schwarzkopf, Powder Metallurgy, The MacMillan Co., N.Y., 1947, pp. 30 and 31.

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