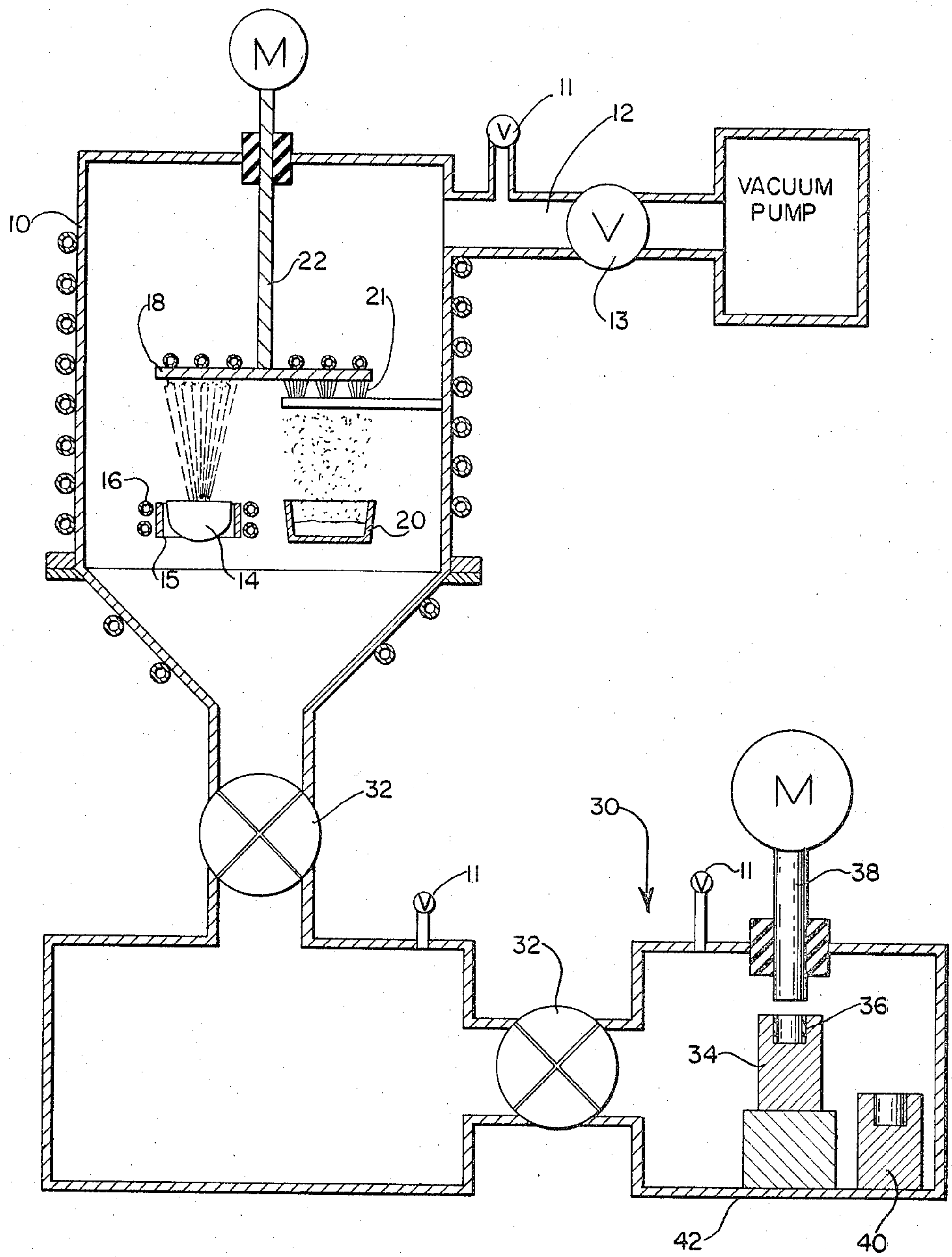
Feb. 6, 1968 L. R. ALLEN 3,367,775

POWDER METALLURGY

Filed Oct. 22, 1965



United States Patent Office

3,367,775

Patented Feb. 6, 1968

3,367,775
POWDER METALLURGY

Lloyd R. Allen, Belmont, Mass., assignor to National Research Corporation, Cambridge, Mass., a corporation of Massachusetts

Filed Oct. 22, 1965, Ser. No. 500,618
4 Claims. (Cl. 75—213)

This invention relates to the production of powder metallurgy billets, suitable for forging extrusion or other machining operations to produce impact resistant products. More particularly, the invention relates to the production of such billets from beryllium powders.

In the prior art, consolidation and densification of beryllium powders is accomplished by the hot pressing process described in U.S. Patent 2,818,339 and by other heating techniques, such as casting, warm isostatic pressing and cold pressing compaction and sintering. The workers in the art try to maintain high purity, yet retard grain growth, during the treatment in order to attain a high degree of triaxial ductility in the finished product. Nevertheless, despite intensive development effort in this field for 20 years, large billets made from beryllium and associated metals, zinc, magnesium, cadmium, titanium, zirconium, have been unsatisfactory. This has forced the aerospace and atomic energy industries to forego wider usage of beryllium despite its very attractive strength-weight ratio and low neutron capture cross-section.

I have discovered a process for producing beryllium billets by providing submicron powders which have a high initial purity and clean reactive surface, forming the powder into green state compacts and then densifying the compacts by cold pressing. While cold pressing, per se, is of course an old process, it is indeed surprising that it could be applied to a metal such as ultrafine beryllium because of known problems of flow of the metal between the die and ram. Whenever the workers in the art tried to overcome the flow problem by closely fitting press assemblies, the result was excessive galling of the ram or die and/or contamination of the metal being pressed. Since the metal must be handled in a non-contaminating environment, such as vacuum or a pure inert gas atmosphere, the galling problem is accentuated.

Therefore, my invention can be characterized as having the principal object of providing a new method of consolidating ultrafine light metal powders into triaxially ductile shapes.

It is a further object of my invention to provide an improved cold pressing process which is useful for consolidating ultrafine light metal powders, without substantial loss of purity or adverse grain re-orientation.

It is a further object of my invention to form triaxially ductile beryllium shapes characterized by fine grain size.

Other objects, features and advantages of my invention will in part be obvious and will in part appear hereinafter.

For a more detailed description of my invention, reference should be had to the following specification which discloses my best known embodiment of the invention and the accompanying drawing which is a schematic diagram of structure used in the practice of my invention.

In the drawing there is shown a water cooled hermetically sealed tank **10** which is evacuated through a conduit **12** connected to a vacuum pump via a valve **13**. There is also provided a valve for introducing inert gas (argon) through conduit **12**. Within the tank is a metal vapor source, which in this embodiment comprises a beryllia crucible **14** surrounded by a water-cooled induction coil **16** and tantalum sleeve **15** which is inductively coupled to the coil. The sleeve **15** is surrounded by insulation (not shown). The coil **16** is coated with a flame sprayed deposit

of enamel to prevent short circuits due to condensing powder.

In operation, a charge of about 2 pounds of beryllium is placed in the crucible. The coil is operated at a level of 20–30 kilowatts to thus heat beryllium to about 1350° C. The pressure in the chamber is adjusted to about 0.5 micron by backfilling with argon. Under these conditions the beryllium evaporates at a rate of 0.1 lb./hr. and the evaporated material is condensed on a water-cooled plate **18**.

The condenser plate **18** is supported from a rotary shaft **22** driven by a ratio motor which rotates the plate at about 100 revolutions per minute. The plate which may be flat or drum shaped is made of stainless steel and has an adherent beryllium coating on its lower face which is prepared in a preliminary beryllium evaporation run at 0.2 micron pressure with the plate heated at 300° C. Collected powders on plate **18** are removed as the plate passes over beryllium-alloy (5% copper) brushes **21**. Powder is brushed from the plate **18** and falls into a collection cup **20**. A plurality of such cups can be provided on a conveyor and fed under the brushes in series.

The operator reaches in through glove ports (not shown) to move the collection cups to dry box **30** though one or more valves **32** and transfer the collected powders to a die **34** mounted within the dry box. The die **34** is made of cold rolled low carbon steel, and is fitted with a polyethylene terephthalate sleeve **36** about .002 inch thick. A motor drives a mild steel ram into the dry box through a conventional seal to press the powders at about 2000 p.s.i. to a green density of about 20%. A series of five compacts produced in die **34** each about 1¼ inches diameter by ¼ inch high are canned (mild steel can 2 inches I.D., .25 inch wall) and placed in a hardened steel die **40** which the operator mounts on block **42**, replacing die **34**. The operator then operates the ram **38** at 94 tons to press the compacts to about 50–55% of theoretical density.

After the above initial and final cold press steps, the beryllium compact is sintered for 10 hours at 1800° F. in a dry nitrogen atmosphere, whereupon the can is cut away.

In the above described second pressing step, the green pellets are crumbled in the loose can (or die container, if no can is used) and repressed to high density. This has the salutory effect of eliminating laminations and density gradations in the final product. The axial travel of the ram in contact with the stack of pellets is very short since the first step of compacting powders takes care of the requirement for long travel length by eliminating trapped gasses from the loose powders.

What is claimed is:

1. An improved powder metallurgy process for producing triaxially ductile bodies of beryllium metal characterized by fine grain size and comprising the steps of:
   (a) providing beryllium powder in the form of high purity, reactive, submicron size particles in uncompacted form,
   (b) cold pressing the powder to self-supporting green pellets with a density in the range of 5–50% theoretical density using a soft die wall made of inert material, with the wall being free of directly applied pressure from the pressing means, the pressing being conducted in a non-contaminating environment,
   (c) crumbling and repressing the pellets in a press with a hard ram and hard die to produce a final pellet of at least 50% or greater theoretical density, the pressing being conducted in a non-contaminating environment, to produce a beryllium structural product characterized by finer grain size than prior art production methods.

2. The process of claim 1 wherein the densified body is sintered after the above step (c) and prior to further fabrication shaping steps.

3. The process of claim 1 wherein the green compact from step (b) is canned prior to step (c).

4. The process of claim 1 wherein the die wall used in step (b) is a polyethylene terephthalate sleeve of a thickness of .002–.020 inch.

References Cited

UNITED STATES PATENTS

3,049,421	8/1958	Allen	75—213 X
3,071,463	1/1963	Hausner	75—213 X
3,150,975	9/1964	Beaver.	
3,175,903	3/1965	Herron	75—213

OTHER REFERENCES

"Fundamental Principles of Powder Metallurgy," Jones, Edward Arnold (publishers), L7D., 1960, pp. 350–351 and 337–342.

CARL D. QUARFORTH, *Primary Examiner.*

BENJAMIN R. PADGETT, *Examiner.*

A. J. STEINER, *Assistant Examiner.*