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CONSOLIDATION AND FORMING BY HIGH-ENERGY-RATE EXTRUSION OF POWDER MATERIAL

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ABSTRACT OF THE DISCLOSURE

This invention is concerned with the production of dense structural shapes from low-density powder compacts by high energy rate extrusion at elevated temperatures. Applicants have found that by rapidly heating low-density billets and forming with the application of very high energy, high quality dense products may be produced and the conventional steps of sintering and forming may be combined.

BACKGROUND OF INVENTION

The production of dense shapes from powdered material is taught by the prior art in several processes.

In one process, loose powder is first cold compacted, frequently under isostatic conditions, where pressures are limited to approximately 100,000 p.s.i. These compacts are then sintered at high temperatures for a long period of time. The sinter-compacted billet is formed into final shape by forging, swaging or extrusion. An example of this method may be found in the processing of tungsten. In such a process loose tungsten powder is compacted in a cold press to about 50 percent theoretical density. It is then sintered at about 70 percent of its melting temperature, that is a sintering temperature of about 2,400° C. for some 15 hours in an atmosphere of hydrogen, thus increasing its density to 85 to 95 percent of theoretical. The sinter-compacted billet is formed by forging, extrusion or rolling.

Compacts forged in this manner require high temperatures for long periods of time to assure adequate density for subsequent forming operations. These high sintering temperatures require special furnaces and treatment inevitably results in coarse-grained structures. In addition, another thermal cycle is usually required during the forming operation. Thus to achieve a product having an adequate density, long periods of time, high temperatures, and considerable expense are necessary.

A second technique of the prior art is hot-pressing followed by forging, extrusion or rolling. In hot-pressing, the operations are severely limited by die materials that must be operated at the pressing temperatures. Again, considerable time and temperature is involved in producing adequate density. Furthermore, the limited pressure available in hot-pressing presents problems in equipment and construction materials.

Still another technique deployed in the prior art with respect to metal products is the deposition of metal vapor on a colder surface. This process has some severe drawbacks among which are loss of metal, difficulty of process control and expense.

OBJECTS OF THE INVENTION

Accordingly, the objects of the present invention are:

To provide an improved process for the consolidation and forming of powder materials;

To provide an improved process for the formation of powder materials into billet stock, sheet, bar, and structural shapes;

To provide an improved process for the consolidation and formation of powdered metals; and

To provide an improved process for producing dense structural shapes from low-density powders by high-energy-rate extrusion at elevated temperatures.

Still other objects and features of the invention will become apparent from the following description.

DESCRIPTION OF INVENTION

In brief, we have found that the sintering and forming operations can be combined by a unique process that can be considered dynamic hot-pressing whereby low-density billets can be heated rapidly and formed by applying energy at a high rate.

Our process consists of preparing the extrusion billet, heating, and extruding at a very high reduction ratio. In preparing the billet, the powder material is vibration-compacted to approximately 20 percent theoretical density in a rubber container of the proper shape and dimensions. The material is then isostatically pressed at approximately 30,000 p.s.i.g. to achieve a density of approximately 50–70 percent theoretical. Following this, the compact may be sintered at a low temperature to remove impurities and/or provide additional strength for sizing operations. Density may increase approximately an additional 10 percent during this operation, and in this condition the compact is machined to size if required. For example, tungsten sintered at 1,100° C. for 1 hour in hydrogen reached a density of 60 percent. For most materials a sintering temperature in the range of 0.3 to 0.4 absolute melting point (homologous temperature) will be adequate. Heating of the compact to extrusion temperature can be done in any suitable manner that prevents contamination of the compact. Metallic materials can be heated rapidly by induction heating techniques with an inert atmosphere provided if required.

After reaching the proper temperature, the compact is held at the same temperature for a length of time of the order of 5 minutes to insure uniform heating. Selection of the temperature is important and may require several trials. The following temperature ranges give good results for the materials listed:

	°C.
Beryllium	750–950
303 stainless steel	750–950
Titanium	550–750
Columbium	850–1050
Molybdenum	1050–1200
Tungsten	1600–1900
Tungsten-ZrN [1]	1600–2000
Tungsten-25 rhenium [2]	1600–2000

[1] .5–5% ZrN (volume basis).
[2] 25% rhenium (weight basis).

For tungsten, a temperature of 1800° C. was reached in 7 minutes with a holding time of 3 minutes for a billet 1.5-inches in diameter by 2.5-inches long. The billet is transferred rapidly from the furnace to the extrusion chamber of a high-energy-rate forming machine and extruded at the proper reduction ratio to produce the final shape having a density of 99 percent theoretical or greater. Extrusion temperature and reduction ratio must be determined experimentally for each material with the combination chosen to produce a minimum of grain growth. During the extrusion operation, the material is subjected to extremely high energy input rates and high temperatures to produce a strong, high-density product. Examples

of energy input rates for various materials are listed below:

	Inch-pounds per cubic inch of final volume per second (in millions)
Beryllium	20–40
303 stainless steel	35–45
Titanium	20–30
Columbium	25–35
Molybdenum	42–58
Tungsten	50–65
Tungsten-ZrN[1]	75–90
Tungsten-25 rhenium[2]	65–85

[1] .5–5% ZrN (volume basis).
[2] 25% rhenium (weight basis).

Commercial tungsten powder with a 5-micron average particle size was extruded into 0.5-inch diameter rods at 1800° C. and at an energy input rate of approximately 55,000,000 inch-pounds per cubic inch of final volume per second.

These rods exhibit the following properties:

Denstiy, percent theoretical	99
Oxygen content	p.p.m. 40
Nitrogen content	p.p.m. 23
Hydrogen content	p.p.m. 0.5
Grain size	ASTM 7–8
Hardness	Rockwell A–70

	At 300° C.	At 500° C.
Tensile strength, p.s.i.	93,000	83,000
Yield strength, p.s.i.	87,500	83,300
Total elongation, percent	10	5
Uniform elongation, percent	10	8
Reduction in area, percent	9	8

These were sound extrusions with good dimensional tolerance and surface finish.

Variations in the preparation of the sintered charge can be made within the conditions outlined above. For example, the powder can be vibration compacted in a suitable die and heated directly to extrusion temperature in a hydrogen atmosphere, thus eliminating the separate sintering steps. Sintering treatments, if required, also could be done with different techniques, and the process could be arranged to eliminate machining of the compact. The energy source may be varied, but the extrusion or forming must be done at high-energy rates, proper deformation and compaction ratios, and proper temperatures. Other forming techniques such as closed die forging could be used.

Our process is applicable to other metals and there is reason to believe also to non-metals such as ceramics and cermets which are combinations of ceramics and metals. Furthermore, it is accomplished with commercially available equipment.

Our process as applied to tungsten starts with cold-pressing the tungsten powder to a density 50–70 percent or less of theoretical. The pressed powder is sintered in hydrogen, at about 1100° C. for about 1 hour. This is primarily to decrease oxygen content, although the density increases incidentally another 10 percent of theoretical during the oxygen cleanup. This step, as a separate one, can be omitted where the equipment includes a hydrogen-atmosphere furnace located close to the forming machine.

The low-density billet sintered for oxygen removal is heated to a temperature close to 1800° C. This temperature can be critical. Temperatures higher than 1900° C. induce grain growth. Temperatures lower than 1600° C. produce a wrought structure. The temperature can be selected in accordance with the structure desired.

The heated billet is transferred quickly to a high-energy-forming machine where the speed of forming results in application of energy at a rate of the order of 55,000,000 inch-pounds per cubic inch of product per second.

In comparison the highest rate of energy transfer which we have been able to find in the prior art is of the order of 500,000 inch-pounds per cubic inch per second. It is apparent that while our invention has superficial similarities to processes of the prior art, the ratio of energy application involved which is permissible under the temperature conditions specified is of a different order of magnitude from those achieved in the prior art.

A second example to indicate the nature of our invention is that of beryllium, a metal much different from tungsten. Beryllium is cold-pressed to about 64-percent of theoretical density. We have then hot-pressed it at 960° C. at 800 pounds per square inch for 3½ hours to 91 percent of theoretical density. It is extruded at 900° C. with an energy input rate of the order of 20,000,000 inch-pounds per cubic inch per second. The final density is very close to 100 percent of theoretical density. This process can be simplified with appropriate apparatus to omit the hot pressing, simply subjecting the cold-pressed ingot to heating and appropriate atmospheric treatment and high-energy impact pressing the ingot when still only in the range of 60 to 70 percent of theoretical density.

In these illustrative examples of tungsten and beryllium the extrusion reduction ratio was 9 to 1, however ratios of from 6 to 1 to 12 to 1 may also be used. That is the cross sectional area of the finished product after pressing was one-ninth of that of the ingot after it had become pressed to final density. The process, thus, can be conceived as taking place in two successive stages, first compression to near final density and immediately after extrusion with the specified reduction ratio.

The process, involving as it does a transfer of energy at the high rate of 20 to 90 million-inch pounds per second of cubic inch of ingot, requires a die for combined compression and extrusion capable of sustaining the combination of high pressures and temperatures. We have found that ceramic dies can sustain these temperatures and pressures, and that one ceramic in particular, zirconium oxide, is well adapted to this service.

In the dies the base and shoulders of the orifice sustain the ingot during the compression when subjected to the rapid application of energy. The continuing pressure extrudes the metal through the orifice after it has been heated and compressed. The orifice is necessarily small enough to resist flow of the ingot until compressed to the desired density and accordingly, our process is adapted especially to extrusions with a large reduction ratio. A larger orifice relative to the die area, with consequently lesser reduction ratio, can be used if the metal is hot pressed to a density approximating the theoretical as in the example of the beryllium hot pressed to 91 percent of theoretical prior to final pressing and extrusion. However, tungsten has been satisfactorily extruded as low as 1580° C. and with a 6.25 to 1 extrusion ratio.

Thus, while there is illustrated and described herein certain preferred procedures which are presently regarded as the best mode of carrying out the invention, it should be understood that various changes may be made without departing from the spirit and scope of the invention concepts which are particularly pointed out and claimed herebelow.

What is claimed is:

1. A method of forming high density products from powdered metals selected from the group consisting of beryllium, stainless steel, titanium, columbium, molybdenum, tungsten, tungsten-zirconium nitride alloys containing from .5 to 5 percent zirconium nitride on a volume basis and tungsten-rhenium alloys containing about 25 percent rhenium on a weight basis which comprises:

(a) cold-pressing a metal powder selected from said group to densities in the range of from about 50 to 70 percent of theoretical density;

(b) sintering the compacted powder with hydrogen at a temperature in the range of from 0.3 to 0.4 of its absolute melting point whereby the material is deoxidized and decontaminated; and

(c) extruding the compact at a temperature of from about 900–1800° C. at a reduction ratio of from 6 to 1 to 12 to 1 and with energy applied at the rate of from 20 to 90 million inch-pounds per second per cubic inch of metal being processed.

2. The method of claim 1 wherein the metal is hot-pressed to 90% theoretical density during step (b).

3. The method of claim 2 wherein the metal is beryllium and the extrusion is carried out at a temperature in the range of from about 750°–950° C. and at an energy input rate of from 20 to 40 million inch-pounds per cubic inch of metal being processed.

4. The method of claim 1 wherein the metal is tungsten and the extrusion is carried out at a temperature of from about 1600°–1900° C. and at an energy input rate of from 50–65 million inch-pounds per cubic inch of metal being processed.

5. The method of claim 1 wherein the metal is molybdenum and the extrusion is carried out at a temperature of from about 1050°–1200° C. and at an energy input rate of from 42–58 million inch-pounds per cubic inch of metal being processed.

6. The method of claim 1 wherein the metal is titanium and the extrusion is carried out at a temperature in the range of from about 550°–750° C. and at an energy input rate of from 20–30 million inch-pounds per cubic inch of metal being processed.

7. The method of claim 1 wherein the metal is columbium and the extrusion is carried out at a temperature in the range of from about 850–1050° C. and at an energy rate of from 25–35 million inch-pounds per cubic inch of metal being processed.

8. The method of claim 1 wherein the metal is a tungsten-zirconium nitride alloy having from 0.5%–5% zirconium nitride on a volume basis and the extrusion is carried out at a temperature in the range of from about 1600–2000° C. and at an energy input rate of from 75–90 million inch-pounds per cubic inch of metal being processed.

9. The method of claim 1 wherein the metal is a tungsten-rhenium alloy having a rhenium content of about 25% by weight and the extrusion is carried out at a temperature in the range of from about 1600–2000° C. and at an energy input rate of from 65–86 million inch-pounds per cubic inch of metal being processed.

10. The method of claim 1 wherein the metal is stainless steel and the extrusion is carried out at a temperature of from about 750°–950° C. and at an energy input rate of from 35–45 million inch-pounds per cubic inch of metal being processed.

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