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PROCESS TO PRODUCE VOID FREE REFRACTORY BORIDE PRODUCT

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2 Claims. (Cl. 75—202)

This invention relates to a method of producing powder metallurgy compacts substantially porefree in nature. More particularly it relates to a method of incorporating an infiltrant into the voids in a powder metallurgy compact which contains as a principal constituent at least one material which is not readily wet by the infiltrant.

Prior efforts to produce materials for seals and bearings have focused attention on the carbides, silicides, borides and nitrides of the group IVA, VA and VIA metals and the prior art discloses the infiltration of compacts of such materials, as described for example, in Schwarzkopf Patent 2,422,439. We have found that the infiltration technique described in the patent does not yield entirely satisfactory results when applied to systems in which the infiltrant does not wet one or more of the materials comprising the powder compact to be infiltrated and that to effectively produce an infiltrated article substantially devoid of pores, it is necessary to incorporate at least a fraction of the infiltrant into the preform prior to infiltration.

For purposes of illustration the present invention will be described in systems in which silver is the infiltrant and preforms of nickel and various borides comprise the matrix material which is to be infiltrated; it will be readily understood by others skilled in powder metallurgy that other refractory constituents may comprise the material infiltrated and that iron, cobalt and a host of ferrous and non-ferrous alloys could be present individually or in combination, in place of the nickel in the following examples and further that in place of metallic silver, silver-base alloys or other non-ferrous metallic materials could be employed, without departing from the scope of the present invention.

The following examples will serve to further illustrate the present invention and are to be taken as illustrative rather than limitative thereof.

Example 1

A powder metallurgy compact consisting of 27 parts by weight of tungsten boride and 3 parts of nickel was formed by intimately mixing plus 200 mesh, minus 325 mesh (Tyler standard) nickel and minus 325 mesh tungsten boride, and pressing the mixture in a die at about 25 tons per square inch. The resulting slug had an apparent density of approximately 80% of the theoretical density of such a mixture.

A solid silver slug weighing 10 grams was placed in physical contact with the porous slug and the silver and compact were heated in a hydrogen atmosphere to about 1150° C. and maintained at that temperature for 120 minutes; during this heating, the silver melted and was drawn into the pores of the compact. When the infiltrated article was examined under a metallurgical microscope, it was observed that the silver had only partially penetrated the pores. Increasing the duration or temperature at which infiltration was effected did not materially decrease the extent of the unimpregnated area.

The resulting material when tested in wear was found to be inferior to standard materials such as graphite.

Example 2

The procedure of Example 1 was repeated except that between one third and one half of the silver was incorporated into the mixture of refractory boride and nickel prior to compacting and a lump of silver was placed in contact with the silver-containing slug. After heating, to melt the silver, as in Example 1, and to infiltrate the compact, it was found that the resulting material was completely free from any voids and that it possessed an excellent combination of properties as a rotating seal material, at elevated temperatures, e.g. 1000° to 1300° F., even in the absence of a lubricant.

Example 3

The procedures of Examples 1 and 2 were repeated with mixtures of the borides of the metals W, Mo, Cr, V, and Ti with and without the addition of some of the silver to the composition, prior to compacting, and in each instance it was found that the infiltrations performed as in Example 1 produced porous products, incompletely impregnated, whereas those processed in accordance with the procedure of Example 2 were free of gas pockets.

The following is a tabulation of some of the compositions processed as in the examples above. All infiltrations were for 120 minutes at 1150° C. in hydrogen atmosphere.

Table I

[No silver in mixture compacted]

Refractory	Powder Mixture		Solid Infiltrant, Ag Parts by wt.	Product, Density (percent of Theoretical)
	Refractory, Parts by wt.	Nickel, Parts by wt.		
Chromium Boride	27	3	10	45
Molybdenum Boride	27	3	10	40
Tungsten Boride	27	3	10	54
Vanadium Boride	21	9	10	66

Table II

[Silver in compacted mixture]

Refractory	Powder Mixture, Parts by weight			Solid Infiltrant, Solid Ag parts by wt.	Product, Density (percent of Theoretical)
	Refractory	Ni	Ag		
Chromium Boride	18	6	6	10	97
Molybdenum Boride	21	3	6	10	96
Tungsten Boride	23.7	1.8	4.5	10	99
Tungsten Boride	18	6	6	10	99.5
Vanadium Boride	13.5	6	10.5	10	94

While it is evident that substantial amounts of silver are infiltrated into the compositions in any event, it appears that to produce a void free product, it is necessary that at least 15–20% of the silver be included in the mixture prior to infiltration.

We claim:

1. A process for producing a pore-free product which comprises: forming an intimate mixture consisting essentially of nickel, silver and a refractory material which is not wet by silver and which is selected from the group consisting of borides of the metals W, Mo, Cr, V and Ti; compacting the mixture of refractory material and metals to form a compact having a density somewhat less than the theoretical density of said mixture; maintaining the compact in physical contact with additional silver and

simultaneously therewith heating the additional silver and compacted material to a temperature and for an interval at least sufficient for the additional silver to melt and to impregnate the compact to substantially theoretical density; the relative amounts of silver being such that at least 15% of the total silver is present in the original mixture and the remainder of the silver being at least sufficient to completely fill the voids in said compact.

2. The method of claim 1 wherein the compact is sintered and infiltrated at about 1115° C. in a hydrogen atmosphere.

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CERTIFICATION OF CORRECTION

Patent No. 2,974,040 March 7, 1961

Elwyn D. Fisher et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 1, for "1115° C." read -- 1150° C. --.

Signed and sealed this 1st day of August 1961.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents

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