

[54]	REMOVAL OF LUBRICANTS AND BINDERS FROM SINTERABLE POWDER COMPONENTS	3,663,317	5/1972	Westendorp et al.....	148/103
		3,728,110	4/1973	Klar et al.	29/192 R
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[57] ABSTRACT

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Powdered metal parts, particulary cobalt-rare earth magnets, having a smooth surface and strong structure are prepared by mixing the powder with an organic lubricant, press-forming the mixture to a desired configuration, removing the lubricant by solvent extraction, evaporating off residual solvent, and sintering.

[56] References Cited

UNITED STATES PATENTS
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8 Claims, No Drawings

REMOVAL OF LUBRICANTS AND BINDERS FROM SINTERABLE POWDER COMPONENTS

BACKGROUND OF THE INVENTION

Powder metallurgy is directed to the press-forming of powder particles into a desired shape and then sintering the shaped particles to produce a strong and cohesive product. Conventionally, organic lubricants and binders are mixed with the powder particles prior to compacting or press-forming the particles. The use of lubricants and binders in powder compaction makes it possible to compact good parts automatically at fast rates with very good tooling life. The lubricants and binders are normally removed from the formed material prior to sintering by a burn-out step.

Some powder systems are very reactive chemically and will retain some or all of the organic lubricants and binders during the burn-out step. During the subsequent sintering step such powders will form carbides, hydrides and oxides which can be very deleterious to the properties of the final product. The present invention provides a way to remove organic lubricants and binders immediately after press-forming. While the invention can be used with chemically inert powders, it is of particular advantage in powder systems that are chemically reactive. An example of such a system is the intermetallic compound powder used in producing cobalt-rare earth magnets.

SUMMARY OF THE INVENTION

The present invention provides for the removal of organic lubricants and binders from metal powders immediately after the compacting step. This removal is achieved by solvent extraction performed under relatively low-temperature conditions such as 50°-85° C. At these temperatures there is no reaction between the lubricants or binders and powder particles even in the case of powders which are chemically highly reactive. After the solvent has removed the lubricant and binder it is sometimes necessary to remove residual solvent by evaporation. After the lubricant, binder, and residual solvent have been removed, the compacted powder is sintered in the conventional manner.

DESCRIPTION OF PREFERRED EMBODIMENTS

The present invention applies with particular advantage to chemically reactive metal powders such as those used in cobalt-rare earth magnets, titanium alloys, and zirconium alloys.

Lubricants used in powder metallurgy are typically organic materials having a wax-like consistency. They can be fatty acid esters, which may be modified, such as are sold under the designation "Acrax-C" by Glyco Chemical Company, or N; N'-ethylene-bis-stearamide sold by the Chemetron Corporation under the designation "Chemetron Wax - 100." Zinc stearate is another material frequently used as a lubricant in powder metallurgy. Since the purpose of the lubricant is to provide effective lubrication between powder particles and between powder and compacting die, the minimum lubricant which will provide such lubricity is used. Normally the powder mixture contains between one half percent and one and one half percent by weight of lubricant. The lubricant and metal powder are normally mixed together by a blending operation.

The powder and lubricant are usually press-formed to provide a green, i.e. unsintered, compact. The lubri-

cant is then extracted from the green compact by a conventional extraction step with an extractor. It is required that the solvent used in the extraction have properties which will dissolve the lubricant in the compacts. Hydrocarbon solvents are useful with many binders. Chlorinated hydrocarbon solvents, such as trichloroethylene, tetrachloroethylene, and trichloroethane are particularly useful with conventional organic lubricant materials.

In order to remove all the lubricant, solvent extraction is allowed to proceed for long periods, for example, 40 hours or more. At the conclusion of the extraction process there may be residual solvent present in the green compact. This can be removed by an evaporation process such as exposure of the compact to a temperature above the boiling point of the solvent for several hours.

After the removal of all lubricant and solvent, the compact is subjected to a conventional sintering treatment as described in more detail hereafter.

The following examples involve processing of cobalt-samarium magnets made from Co₅Sm powder of 1-10 micron particle size. The magnets were made in the form of rings of two different sizes before sintering. Ring A was 0.28 inch inside diameter, 0.610 inch outside diameter, and 0.17 inch thick. Ring B was 0.28 inch inside diameter, 0.790 inch outside diameter, and 0.15 or 0.20 inch thick. Solvent treatment time was 48 to 72 hours. Temperature of the solvent was 50° to 85° C. during treatment in a Soxhlet extractor. After the extraction operation, the solvent was allowed to cool. The lubricant precipitated out in the cold solvent. The mixture was filtered to separate the lubricant and the lubricant was weighed. These weightings demonstrated that the lubricant was completely removed.

The sintering time on all samples was one hour at 1130° C. The sintering treatment was applied to (1) green compact from which the lubricant had been extracted, (2) green compact which included the lubricant, and (3) green compact which had never had lubricant present. The parts that never had lubricant present required die wall lubrication by separate means during pressing in order to eliminate seizing in the die and physical defects in the compact.

EXAMPLE 1

A batch of 150 grams of Co₅Sm powder was mixed with 1.5 grams of N, N'-ethylene-bis-stearamide in a blender for 1 hour. The powder was pressed into Ring A in an axial magnetic field of 3,500 gauss and 120,000 psi pressure. The pressed ring was then extracted with trichloroethylene in a Soxhlet extractor for 47 hours at a temperature of between 50° and 60° C. The ring was then placed in a vacuum dryer for 5 hours at 30° C. to remove residual solvent. The ring was then sintered for 1 hour at 1130° C. in a dry argon atmosphere. It was then cooled to 915° C. at the rate of 6° C. per minute and then quenched to room temperature in hot silicone oil.

EXAMPLE 2

A ring was made in the manner described in Example 1 except that the extraction and vacuum drying steps were omitted.

EXAMPLE 3

A ring was made as described in Example 2 except

that the Co₂Sm powder did not contain any lubricant material.

The following Table 1 gives the results of tests performed on the magnets of Examples 1-3. The packing factor (P.F.) is the percent of density of the theoretical density. With rare earth magnets a packing factor of 87 minimum is needed to produce a good stable magnet.

Table 1

Example Number	Lubricant Content Wt. %	Solvent Treatment	% Shrinkage	Density	P.F.	Open Circuit Flux-Gauss	Surface Appearance
1	1%	Yes	10.5	8.0	93	7030	Smooth
2	1%	No	5.2	6.6	76	1330	Smooth
3	0	No	12.0	8.2	95	7280	Rough

The data of Table 1 show that the magnets that have the lubricant removed (Example 1) were substantially equivalent to the magnets with no lubricant (Example 3) as regards density and magnetic properties. The magnets with no lubricant had rough galled surfaces while magnets with the lubricant removed had very smooth surfaces. The magnets that were sintered without removal of lubricant (Example 2) had low shrinkage, poor density, and very poor magnetic properties.

EXAMPLE 4

Example 4 repeated the procedure of Example 1 except that the solvent was tetrachloroethylene and treatment time was 48 hours at a temperature of 75° to 85° C.

EXAMPLE 5

In Example 5 the procedure of Example 4 was repeated except that the lubricant was a modified fatty acid ester identified as "Acrawax."

EXAMPLE 6

In Example 6 the procedure of Example 5 was repeated except that the lubricant was zinc stearate and the extraction time was 72 hours.

The results of tests made on magnets of Examples 4-6 are shown below in Table 2.

Table 2

Example Number	Lubricant	Magnet Size	% Shrinkage	P.F.	Open Circuit Flux Gauss
4	N,N'-Ethylene-bis-stearamide	A	11.7	94.7	6940
5	Acrawax	A	10.9	93.6	6830
6	Zinc Stearate	B	8.4	94.3	7050

The data of Table 2 show that different lubricants give equivalent results. In all of Examples 4-6, the magnets had a smooth texture. Thus, it appears that in Examples 4-6 the solvent treatments effected substantially complete removal of the lubricant with resultant benefit to the magnetic properties.

EXAMPLE 7

In Example 7 the procedure of Example 6 was followed except that the lubricant was N, N'-ethylene-bis-stearamide. The pressure in the forming press was 118,000 pounds per square inch.

EXAMPLE 8

In this Example the procedure of Example 7 was fol-

lowed except that the press pressure was 60,000 pounds per square inch.

EXAMPLE 9

In this Example the procedure of Example 7 was followed except that the press pressure was 30,000 pounds per square inch.

EXAMPLE 10

In this Example the procedure of Example 7 was followed except that the press pressure was 20,000 pounds per square inch.

Test data covering the magnets of Examples 7-10 are shown below in Table 3.

Table 3

Example Number	Press Pressure psi	% Shrinkage	P.F.	Open Circuit Flux-Gauss
7	118,000	8.2	91.8	6220
8	60,000	9.9	89.5	5730
9	30,000	12.4	91.8	6020
10	20,000	11.2	92.7	5940

The data of Table 3 show that the pressures used in forming ring compacts do not greatly influence the properties of the final product. A moderate pressure of 20,000 pounds per square inch gives about the same results as the extremely high pressure of 118,000 pounds per square inch. Conversely, the practice of the invention enabled all lubricant to be removed even where compacting took place under very high pressures. In all cases the magnets tested to produce the data of Table 3 had a smooth texture.

In some of Examples 1-10, the data shown in Tables 1-3 are averages of two or more runs. These data show that the detrimental effect of lubricants and chemically reactive powder metallurgy systems can be avoided while the beneficial effect of such lubricants during the forming of green compact can be retained.

While the invention has been described with reference to certain embodiments thereof, it is obvious that there can be variations which properly fall within the scope of the invention. Therefore, the invention should be limited in scope only as may be necessitated by the scope of the appended claims.

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What I claim as new and desire to secure by letters patent of the United States is:

1. The method of preparing sintered powdered metal parts which comprises:

mixing powdered metal with an organic lubricant;
forming the mixture to a desired shape;
removing the lubricant from said formed shape by solvent extraction;
and sintering the shaped material.

2. The method of claim 1 in which the extraction step is followed by a solvent evaporation treatment.

3. The method of claim 1 in which the solvent is trichloroethylene.

4. The method of claim 1 in which the solvent is tetrachlorethylene.

5. The method of claim 1 in which the solvent is trichloroethane.

6

6. The method of producing a cobalt-rare earth magnet which comprises:

mixing particles of cobalt-rare earth intermetallic compound with an organic lubricant;

press-forming the mixture into a magnet configuration in aligning magnetic field;

removing the lubricant from said configuration by solvent extraction;

and sintering said configuration to fix the particles thereof firmly in position.

7. The method of claim 6 in which the sintering step is followed by cooling at the rate of about 5° C. per minute to about 915° C., after which the magnet configuration is quench-cooled to room temperature.

8. The method of claim 7 in which the extraction step is followed by a solvent evaporation treatment.

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