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PRODUCTION OF METAL POWDER COMPACTS
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by the Secretary of the Army
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The invention described herein may be manufactured and used by or for the Government for governmental purposes without the payment to us of any royalty thereon. This application is a continuation-in-part of application Serial No. 810,973 filed on May 4, 1959, in the names of Robert A. Powell and Frank I. Zaleski for "Production of Metal Powder Compacts" and assigned to the assignee hereof.

This invention relates to the production of metal powder compacts, and more especially to the provision of an improved method whereby metal powder compacts are produced with properties superior to those produced by previous methods and at less expense. A problem heretofore encountered in the powder metallurgy industry is low ductility and low strength. A typical example is that of brass. A 70-30 prealloyed brass powder compacted at 30 t.s.i. and sintered at 1615° F. for about 30 minutes has the following average properties as compared to annealed wrought 70-30 brass strip.

TABLE I

	Powder Metal Compact	Annealed Wrought
Tensile Strength (p.s.i.).....	26,800	45-48,000
Elongation (percent).....	8 (in 1")	48-64 (in 2")
Density (gm./cc.).....	7.8-7.6	8.57

As a result of their inferior tensile strength, ductility and density, brass parts made by powder metallurgy have not been useful where high strength is required. The application of such parts is further limited by the care required and high cost of sintering. The heretofore sintering procedure for producing 70-30 brass powder compacts involves (1) mixing 0.75% zinc stearate with the powder for lubrication purposes, (2) compacting the powder in a mold at approximately 30 t.s.i., (3) covering the compacts with graphite, (4) preheating the compacts in an air heating furnace at 255 to 345° F. for 5 minutes, (5) preheating the compacts at 1100° F. for 30 minutes, (6) sintering for 30 minutes at a temperature of about 1615° F. Steps 5 and 6 are to be done in a reducing atmosphere such as hydrogen or dissociated ammonia. Using this method compacting pressures up to 40 t.s.i. can be utilized without expansion (density decrease) occurring during sintering.

In accordance with the present invention, the necessity of preheating the compact in two steps is eliminated, the sintering is preferably done in a nitrogen atmosphere, and lithium stearate, rather than zinc stearate is used as the lubricant. The resulting product has good strength, ductility and density.

The mechanisms of sintering powder metallurgical compacts, in general, have been discussed by many investigators and several hypotheses have been formulated. None of these theories, however, explain all aspects of the sintering phenomena. One fact which most investigators agree upon is that the reduction of surface oxides is required before sintering occurs. From this, it would appear that lithium within the compact reduces surface oxides to a greater extent than a pure hydrogen sintering atmosphere.

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The selection of lithium stearate as the best lubricant was based on the test of a considerable group of lubricants. It was found that, at a compacting pressure of 30 t.s.i. and with a 0.75% addition of lithium stearate, the highest green densities could be obtained. Pressures as high as 70 t.s.i. have been used during compacting without expansion occurring during sintering.

Compacts thus made from 70-30 brass powder, i.e., compacted at 30 t.s.i. and 0.75% lithium stearate, were placed in a graphite covered boat and sintered at 1615° F. in an inert atmosphere for about 50 minutes. About 11 minutes were required to bring the furnace up to temperature. No preheat cycle was used before sintering. The resulting properties are indicated by the following table:

TABLE II

Tensile strength (p.s.i.).....	40,500
Elongation in 1" (percent).....	30
Density (gms./cc.).....	8.17

From this table and Table I, it can be seen that the properties of these compacts are decidedly superior to those made in accordance with the currently used method. Thus there is obtained a 51% increase in tensile strength, a 275% increase in elongation (ductility) and a 6.6% increase in density. Another advantage is that the zinc losses are smaller on these compacts than on those produced by the previous method. Experiments were conducted using various percentages of lithium stearate. It was found that optimum properties of the compacts were obtained by the use of 0.5% to 1.0% of this lubricant.

A study of the microstructure of a 0.75% zinc stearate compact molded at 30 t.s.i. and sintered in an inert atmosphere, and the micro-structure of a 0.75% lithium stearate compact similarly molded and sintered, showed that the porosity of both was in a spheroidized form. The degree of spheroidization in the compacts made with lithium stearate, however, was much greater. The disposition of the porosity was also quite different. The porosity of the compacts with the lithium stearate was more uniformly distributed than that of the compacts with the zinc stearate. The grain size of compacts made with lithium stearate was much larger than that of the compacts made with zinc stearate. In wrought materials, excessive grain growth would cause a loss of strength. Apparently, the increase in the grain size of the lithium stearate compacts was not sufficient to cause any decrease in strength. For grain growth to occur during sintering a rather clean particle surface (oxide free) is required. This indicates that the lithium reduced the surface oxides to a great extent.

The fact that lithium stearate did provide a reducing medium and prevented oxide formation was determined by sintering compacts at 1615° F. for about 50 minutes in graphite covered and uncovered boats with no atmosphere. The results obtained are as follows:

TABLE III

	0.75% ZnSt		0.75% LiSt	
	Covered	Uncovered	Covered	Uncovered
Tensile Strength (p.s.i.).....	3,000	2,500	35,000	33,000
Elongation in 1" (percent).....	0	0	23	18

It was observed that the zinc stearate compacts were highly oxidized while the lithium stearate showed no apparent oxidation. The important fact is that the properties of the lithium stearate compacts sintered with no protective atmosphere are considerably higher than the

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optimum properties attained with the use of zinc stearate in a highly reducing atmosphere.

When 70-30 brass compacts using lithium stearate were sintered in nitrogen at higher temperatures and upwardly to about two hours, even better results were obtained:

TABLE IV
Sintered 70-30 Brass Compacts

	108 minutes at 1,615° F. in nitrogen	48 minutes at 1,675° F. in nitrogen	Annealed Wrought Brass
Tensile Strength (p.s.i.)-----	42,500	38,500	45-48,000
Elongation (percent)-----	34 (in 1 inch)	42 (in 1 inch)	45-64 (in 2 inches)
Density (gm./cc.)-----	7.99	8.01	8.53

In each of Tables I through IV, the nitrogen used was commercially pure, oil-pumped, in cylinders, and dried prior to entry in the furnace. The cold specimens were introduced into the already hot furnace which was subjected to a constant flow of nitrogen of 20 c.f.h. at all times, including the cooling cycle. All specimens were sintered in groups of three in a 2 inch diameter tube furnace of the electrical resistance type having a uniform 6 inch long hot zone. After being maintained at the desired sintering temperatures, the specimens were pushed into a cooling zone maintained at about room temperature. Neither the time required to reach temperature nor the time to cool are given in Table IV, only the actual sintering time for the given temperatures. The green densities on all test bars were approximately 7.33 gm./cc.

As previously indicated, the use of lithium stearate in the compacting and sintering of 70-30 prealloyed brass powder with a nitrogen sintering has the advantage that compacts of superior properties are produced more economically and with less loss of zinc. Importantly contributing to these results are the superior lubricating and oxide reducing and oxide inhibiting properties of lithium

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stearate. Thus due to the fact that lithium stearate is a better lubricant, higher green densities can be obtained at equivalent pressures and also extremely high compacting pressures can be used without expansion occurring during sintering. The oxide reducing and oxide inhibiting effects of lithium stearate reduce the cost of sintering in that the problems incident to the use of highly reducing atmospheres are eliminated and very cheap atmospheres such as scrubbed exothermic gas or possibly even no atmosphere can be utilized. While the invention has been largely described as applied to 70-30 prealloyed brass powder compacts, it has been found to be equally applicable to other types of brass powder compacts. For example, it has been found to be useful with 90-10 brass, leaded 80-20 brass, bronzes, copper and iron.

We claim:

1. In a process for producing a compact of metal powder consisting essentially of about 70% copper and 30% zinc comprising mixing said powder with a lubricant, compressing said mixed powder and sintering said compressed powder in a nitrogen atmosphere, the improvement which consists in the step of employing lithium stearate as said lubricant.

2. A single-sintered, prealloyed 70-30 brass powder compact produced according to the process of claim 1, said compact being characterized by a tensile strength ranging between 38,500 and 42,000 p.s.i., a tensile elongation in 1 inch ranging between 30 and 42% and a density ranging between 7.99 and 8.17 grams per cubic centimeter.

References Cited in the file of this patent

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OTHER REFERENCES

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