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H. L. ANDREWS ET AL
PRODUCTION OF ELONGATED METALLURGICAL MILL PRODUCT
FROM LOOSE METAL POWDER

3,810,757

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2 Sheets-Sheet 1

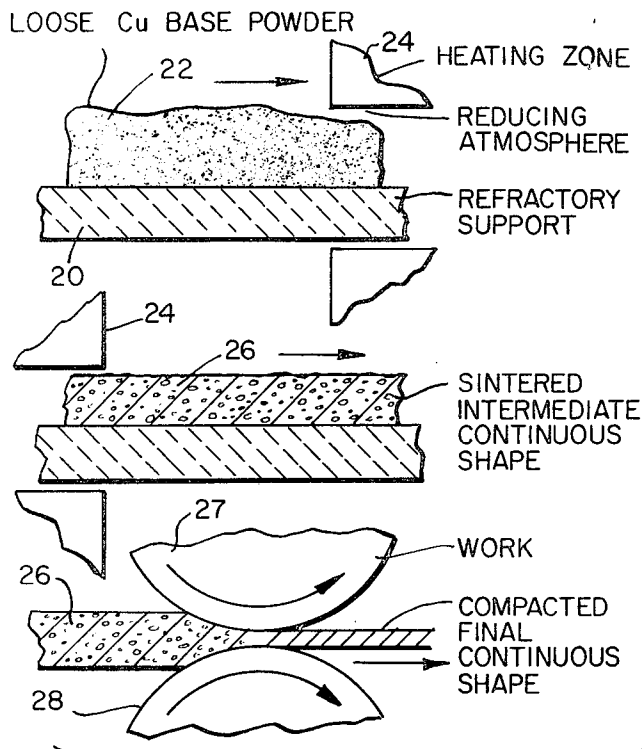


FIG. 1

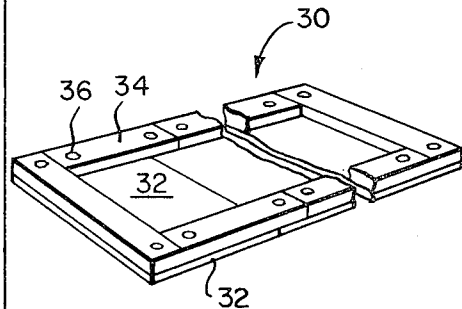


FIG. 2

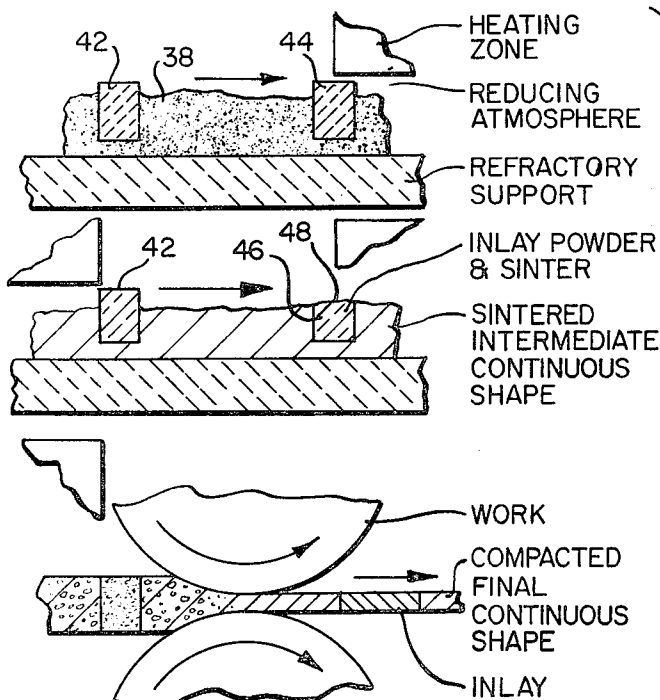


FIG. 3

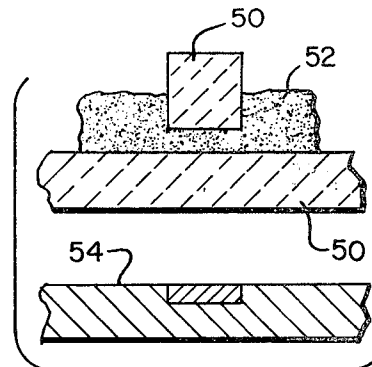


FIG. 4A

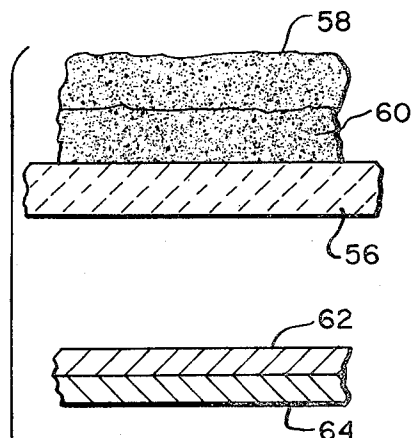


FIG. 4B

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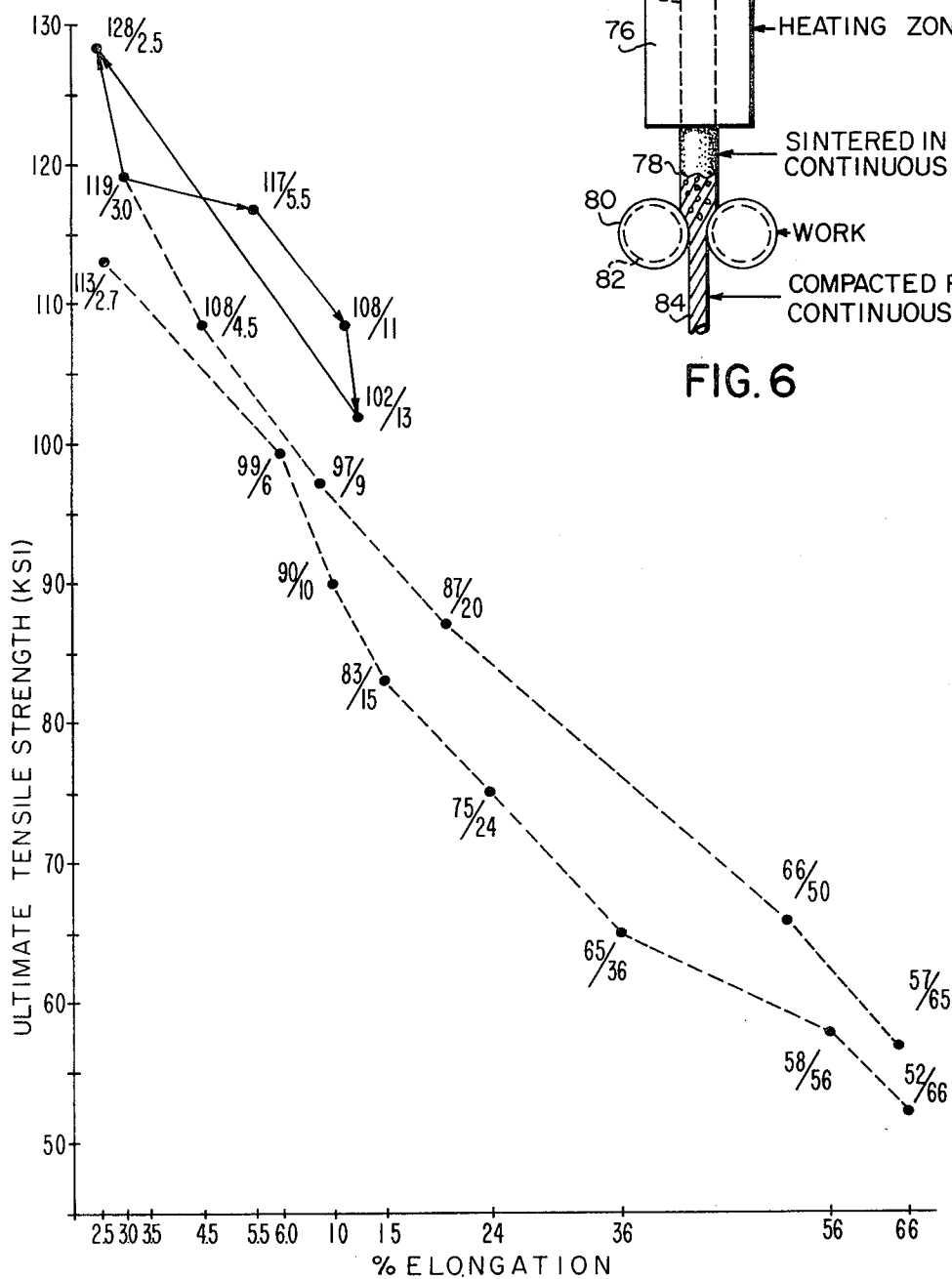


FIG. 5

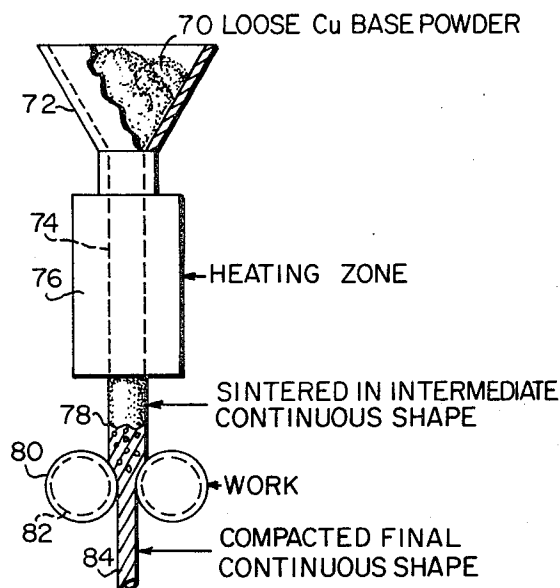


FIG. 6

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PRODUCTION OF ELONGATED METALLURGICAL MILL PRODUCT FROM LOOSE METAL POWDER

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4 Claims

ABSTRACT OF THE DISCLOSURE

A copper base continuous mill shape is produced by the steps of advancing a copper base powder having an initial density on a support through a heating zone in a reducing atmosphere in order to sinter successive increments of the powder for a sufficiently long period to produce a self-supporting intermediate shape having a higher density, and advancing the intermediate shape in a single pass through a working zone that reduces one or two dimensions to produce an elongated shape having at least 90% theoretical density.

BACKGROUND AND SUMMARY

The present invention relates to powder metallurgy and, more particularly, to the production of continuous metallurgical mill shapes from copper base powders.

Prior powder metallurgical processes have involved initial densification of the powder under pressure or adherence of the powder particles together in a binder and thereafter sintering a densified mass of the powder to produce a final shape. When applied to the production of continuous mill shapes, such processes have required particles of specific shape and/or size, large roll diameters, forced feed, slow speeds, special purpose hoppers, and electro-mechanical controls, any or all of which have tended to limit versatility and to increase expense. In particular, powders characterized by particles less than 10 microns in diameter or particles that are spherical in shape have not been useful in continuous powder metallurgical processes because such particles do not frictionally engage each other in such a way as to create a continuous intermeshing mass when fed between working surfaces.

The primary object of the present invention is to provide a process for producing continuous mill shapes, which process comprises the steps of advancing a layer of copper base, loose powder on a refractory support through a heating zone in a reducing atmosphere in order to sinter successive increments of the powder layer for a sufficiently long time to produce a rigid intermediate shape, and advancing this intermediate shape in a single pass through a working zone that reduces one or two dimensions of the intermediate shape to produce a compacted shape. The arrangement is such that spherical powder particles and less than 10 micron powder particles can be delivered and worked continuously. In particular, the successful use of less than 10 micron particle size powders enables unusually fine grain strip, sheet and wire. Also the arrangement is such as to permit a ratio of roll diameter to first yield strip thickness ranging from 100:1 down to 40:1 and lower.

Other objects of the present invention will in part be obvious and will in part appear hereinafter.

The invention accordingly comprises the process and product, including the steps, the components and their interrelationships, which are exemplified in the accompanying disclosure, the scope of which will be indicated in the appended claims.

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BRIEF DESCRIPTION OF THE DRAWINGS

For a fuller understanding of the nature and objects of the present invention, reference is made to the following detailed description, taken in connection with the accompanying drawings, wherein:

FIG. 1 is a flow diagram illustrating a process of the present invention and the resulting product;

FIG. 2 is a component useful in the process of FIG. 1;

FIG. 3 is a flow diagram illustrating another process of the present invention and the resulting product;

FIGS. 4A and 4B illustrate alternative components useful in the process of FIG. 1 and products thereof;

FIG. 5 is a graph illustrating principles of the present invention; and

FIG. 6 is a partly broken away structural view of components of another system embodying the present invention.

DETAILED DESCRIPTION

Generally the process of FIG. 1 comprises the steps of distributing a loose copper base powder on a refractory support 20 to provide a powder layer 22 of initial depth and initial percentage of theoretical density, advancing powder layer on the refractory support through a heating zone 24 to subject each increment of the powder layer to a sintering temperature for a sufficiently long time to produce a rigid intermediate continuous shape 26, and advancing this intermediate shape in a single pass through a working zone, shown as the nip between a pair of rollers 27, 28, to permit working to a compacted continuous shape. Ordinarily the loose powder layer, which preferably ranges from 0.1 to 1½" in thickness, initially is less than 50% of theoretical maximum density. Preferably the rigid intermediate continuous shape is at least 5% greater in density than that of the initial powder and the compacted continuous shape is greater than 90% of theoretical maximum density. The phrase "continuous shape" is defined as a geometrically extended product, for example a sheet, rod, strip or wire, which is produced incrementally and which has a like cross section throughout its length. Loose or free flowing powder includes powder which has been vibrated. The phrase "maximum theoretical density" is defined as the density of a non-porous mass of a composition like the composition of the powder. The phrase "apparent density" is defined as the weight of a sample of copper powder that will fill a 25 cubic centimeter cylindrical cup multiplied by .04. The phrase "tape density" is defined as the maximum density of the aforementioned sample that results from vibration.

Preferably the copper base powder is finer than 300 mesh with any surface and/or shape including particles from a hydrometallurgical process. Such particles from a hydrometallurgical process normally are 10 microns or less in average diameter. Preferably the composition of the copper base powders are such as to contain copper as at least one of their essential ingredients, i.e. more than 50% copper component, the remainder being any alloying metal. Specific examples of copper materials useful herein are as follows. Commercial copper powders that are oxygen free and copper alloy powders including the following. Aluminum bronze, which contains by total weight in the remainder of copper: approximately 9% aluminum and optionally up to 0.21% silver. 90-10 cupronickel copper, which contains by total weight a remainder of copper: approximately 10% nickel and optionally from 1 to 1.6% iron and up to 0.21% silver. 70-30 cupronickel copper, which contains by total weight in a remainder of copper: approximately 30% nickel and optionally up to 0.19% silver. Monel, which contains by total weight in a remainder of copper: approximately 67% nickel and optionally from 1.4 to 1.8 iron and up to 0.06% silver.

65-12 nickel silver, which contains by total weight in a remainder of copper: from 23.3 to 24.8% zinc, approximately 12% nickel and optionally up to 0.20% silver. 65% nickel silver copper, which contains from 16.7 to 19.5% zinc, from 18.1 to 19.2 nickel, and up to 0.225% silver. Low phos copper, which contains by total weight in a remainder of copper: from 0.004 to 0.006% phosphorous and optionally up to 0.22% silver. High phos copper, which contains by total weight in a remainder of copper: from 0.018 to 0.025% phosphorous and optionally up to 0.21% silver. Phosphor bronze, which contains by total weight a remainder of copper: approximately 2% tin, approximately 0.21% phosphorous, and optionally up to 0.193% silver. 90-10 brass, which contains by total weight in a remainder of copper: approximately 9.8% zinc and optionally up to 0.21% silver. 85-15 brass, which contains by total weight in a remainder of copper: approximately 15% zinc and optionally up to 0.21% silver. 70-30 brass, which contains by total weight in a remainder of copper: approximately 30% zinc and optionally up to 0.21% silver.

In one form, the support for the initial metal powder is a refractory bed composed, for example, of graphite. Such a bed is shown in FIG. 2 as comprising a segmented flat base 32 and a segmented shoulder 34, which extend around the periphery of the base and are connected thereto by a series of pins 36. In another form, the support, which is composed of a material such as stainless steel, is in the form of an endless belt. This endless belt may have moving shoulders along its longitudinal edges. In the cases both of the graphite bed and the stainless steel belt, the height of the shoulders is selected to determine the thickness of the initial powder layer.

Preferably, sintering occurs in heat zone 24 at a temperature ranging from just below the melting point of the metal powder to approximately 75% of the melting point, the preferred temperature ranging between 1600 and 1800° F. Preferably, each advancing increment of the powder layer is subjected to the sintering temperature in the heating zone for time long enough for the resulting self supporting sintered stratum to be strong enough to take at least a 50% reduction to 90% of theoretical maximum density without breaking, this 50% reduction referring to at least one dimension in a cross section perpendicular to the direction of travel. Ordinarily this time ranges, for example, between 10 and 30 minutes. Working involves rolling, swaging or forging. In the case of rolling, for example, the ratio of roll diameter to first yield strip thickness ranges below 100:1 down to 40:1 and lower. Preferably the heating zone has a reducing atmosphere for example, carbon monoxide or cracked ammonia.

EXAMPLE 1

In an example of the process of the present invention with reference to FIG. 2, graphite bed 30 has walls that are 350 mils high so as to be capable of retaining a height of copper powder equal to 350 mils. As shown, the bed is approximately 2 feet wide and approximately 20 feet long so that it can be advanced through the sintering furnace at a steady rate. In the present example the copper base powder is composed of commercial copper characterized by particles of less than 10 microns. The heating zone ranges in temperature between 1900 and 2000° F. and is 30 inches long. The bed is advanced through the heating zone at a rate of three inches per minute so that any increment of the copper particle distribution is exposed to the hot zone for a period of approximately ten minutes. The heating zone has a cracked ammonia atmosphere. The resulting 20 foot long self supporting sintered sheet then is compacted between 13 inch diameter rolls to 95% theoretical density at a temperature of 600 to 800° F. to produce a copper sheet that is about 100 mils thick.

EXAMPLE 2

The process of Example 1 is repeated except that the copper powder is composed of 6.4% tin Phosphor bronze strip and that the resulting strip is approximately 0.2 inch thick.

An alternative technique embodying the present invention is shown in FIG. 3 as enabling inlays and the like. This process comprises the steps of producing overlays, inlays, edgelays and throughlays with the aid of refractory shaping components that determine the gross outlines of the copper powder layer before sintering. In the form shown in FIG. 3, copper base powder 38 is spread on a refractory base 40, on which are arranged a series of refractory separators 42, 44. Sintering causes definition of the gross shape of the sintered sheet as having reticulations 46. Following sintering and prior to working, reticulations 46 are filled with an alternative powder 48. Thereafter, the composite intermediate shape is worked in order to produce a composite final sheet, in which metal 48 appears as an inlay. In one form, powder 38 is sintered prior to working and, in another form, powder 38 simply is rolled in position with the remainder of the composite sheet without sintering. An alternative bed 50 for intermediate shape 52 and final inlay configuration 54 is shown in FIG. 4A. An alternative bed 56 for intermediate shape 58, 60 and final ply configuration 62, 64 is shown in FIG. 4B.

The following examples illustrate the properties of 6.4% tin Phosphor bronze strip that has been produced from 10 microns diameter powder in accordance with Example 2 above.

EXAMPLE 3

Steps	Ultimate tensile strength	Percent elongation (in 2 inches)	0.2% offset grain	
			Yield strength	Size (mm.)
1. Sinter roll to .159"				
2. Anneal: 1 hr., 1,600° F				
3. Cold roll to .142" (11% reduction)				
4. Anneal: 3 hrs., 1,100° F				
5. Cold roll to .044" (69% reduction)	117	3.1	113	.006
6. Anneal (3 hrs., 1,110° F.)	55	65		.025
7a. Cold roll to .028" (37% reduction)	96	9.6	91	.023
7b. Cold roll to .022" (50% reduction)	107	4.1	99.5	.018

EXAMPLE 4

Steps	Ultimate tensile strength	Percent elongation (in 2 inches)	0.2% offset grain	
			Yield strength	Size (mm.)
1. Sinter roll to .182"				
2. Anneal: 1 hr., 1,600° F				
3. Cold roll to .142" (22% reduction)				
4. Anneal: 3 hrs., 1,100° F				
5. Cold roll to 0.044" (69% reduction)	120	2.9	115	.006
6a. Anneal 1 hr., 350° F	117	5.5		.006
6a1. Cold roll to 0.022" (50% reduction)	127	2.5		.010
6b. Anneal 1 hr. 450° F	108	11.1	101	.006
6c. Anneal 1 hr. 500° F	102	12.4	94	.006
6d. Anneal 1 hr., 550° F	102	13.6		.008
6d1. Cold roll to 0.022" (50% reduction)	128	2.4		.013
6e. Anneal 1 hr., 650° F	66	50		.003
6f. Anneal 3 hrs., 900° F	57	65	28	.013
6f1. Cold roll to 0.031" (29.4% reduction)	90	10.8		.013
6f2. Cold roll to 0.028" (37.1% reduction)	98	9.1		.013
6f3. Cold roll to 0.022" (50% reduction)	107	3.4		.010
6f4. Cold roll to 0.0175" (60.5% reduction)	119	2.7		.007

As shown in FIG. 5, the samples resulting from the foregoing examples were comparable or better than corresponding standard alloy, of like composition and treatment, in ultimate tensile strength and elongation.

Another modification of a process the present invention is shown in FIG. 6 as involving vertically feeding a loose

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copper base powder 70 through a hopper 72 into the central duct 74 of a cylindrical heater 76 in order to produce a shelf supporting intermediate shape 78, and rolling this shape between the circularly cross-sectional nip 82 of a pair of rolls 80 to produce a compacted final continuous shape 84. This process is useful in the production of rods and tubes.

The present invention thus provides a process by which copper base powder may be continually worked into mill shapes without references to the size and shape of its particles. Since certain changes may be made in the foregoing disclosure without departing from the scope of the present invention, it is to be understood that all matter contained in the foregoing specification and shown in the accompanying drawings is to be interpreted in an illustrative and not in a limiting sense.

What is claimed is:

1. A powder metallurgical process comprising the steps of:

- (a) spreading a free flowing copper base powder on a refractory support to provide a powder layer of an initial thickness ranging from 0.1 to 1½ inches, said powder being characterized by particles of less than 10 microns maximum diameter, said powder being less than 50% in theoretical maximum density;
- (b) advancing said powder layer in contact with said refractory support through a heating zone in a reducing atmosphere in order to sinter successive increments of the powder layer for a sufficiently long time to produce a rigid intermediate continuous shape, the temperature of said heating zone ranging from

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1600 to 1800° F., said rigid intermediate shape being at least 40% of theoretical maximum density;

- (c) advancing said intermediate continuous shape in a single pass between a pair of approximately equal diameter rolls having a ratio of roll diameter to nip distance ranging below 100 to 1 in order to produce a compacted continuous shape having a theoretical density in excess of 90%.

2. In the powder metallurgical process of claim 1, the steps of shaping said powder layer by inserting therein refractory elements, maintaining said refractory elements in position during the advancement of said powder layer through said heating zone, and removing said refractory elements from the rigid intermediate continuous shape prior to the advancement between said pair of rolls.

3. In the powder metallurgical process of claim 1, said powder layer containing a first powder stratum of one composition and a second powder stratum of another composition.

4. In the powder metallurgical process of claim 1, the particles of said powder being spherical.

References Cited

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R. E. SCHAFER, Assistant Examiner

U.S. Cl. X.R.

29-420.5; 75-200