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PROCESS FOR PRODUCING ELONGATED CONTINUOUS BARS AND RODS FROM METAL POWDERS

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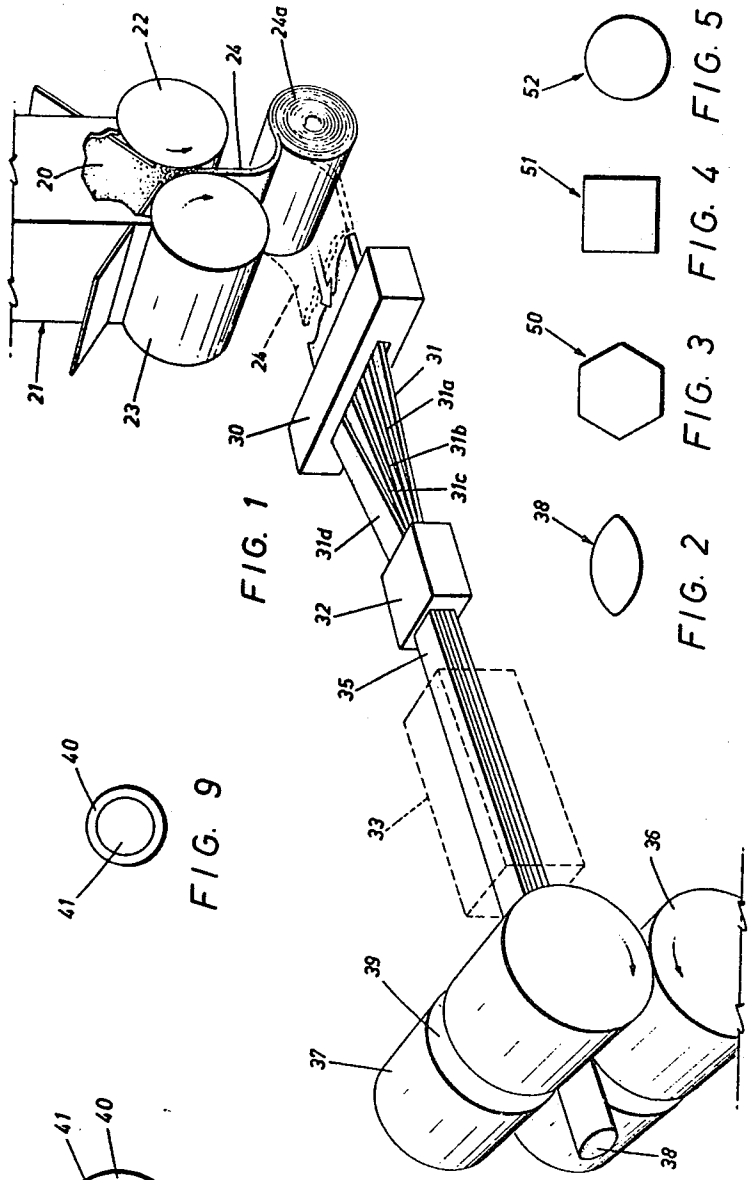


FIG. 1

FIG. 5 FIG. 4 FIG. 3 FIG. 2

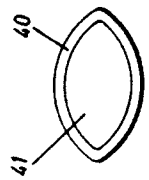


FIG. 7

FIG. 9

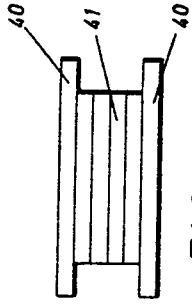


FIG. 6

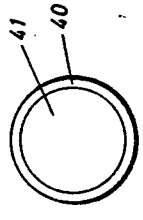


FIG. 8

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PROCESS FOR PRODUCING ELONGATED CONTINUOUS BARS AND RODS FROM METAL POWDERS

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Continuation-in-part of application Ser. No. 84,726, Jan. 24, 1961. This application Mar. 5, 1965, Ser. No. 437,412

4 Claims. (Cl. 75—208)

This application is a continuation-in-part of our prior application Ser. No. 84,726 filed Jan. 24, 1961 and now abandoned.

This invention relates to a process for producing useful wrought shapes from compactible, finely divided metals, metal alloys, composite metal-non metal particles and mixtures thereof. It is particularly directed to providing a process for producing continuous elongated bars and rods and the like wrought shapes from finely divided compactible metallic particles.

Methods are known in the powder metallurgy art by means of which metal particles can be roll compacted to form continuous strips or sheets. Prior to the development of the method of the present invention, however, the usefulness of roll compacting as a means of fabricating metal powder into wrought shapes was severely limited in that the only continuous wrought forms which could be produced with any degree of success were relatively thin strips or sheets. This is because the maximum thickness of self-sustaining "green" strips or compacts that can be produced from powder by roll compacting is limited by the diameter of the compacting rolls utilized to form same. Generally, strip thickness is limited to about 1/100 to about 1/50 of the diameter of the compacting rolls. Thus, very large diameter rolls and massive support frames are required to roll compact relatively thick sectioned products such as are presently obtainable by conventional melting-casting-working procedures. For example, to produce a 0.5 inch diameter rod, rolls 250–500 inches in diameter would be required. The cost of such equipment alone has been sufficient to discourage the adoption of direct powder rolling as a means of forming metal powders into bars and rods. However, even aside from the cost factor of the equipment involved, efforts to directly roll powders into bars and rods using large diameter profiled rolls have never been commercially successful for various other reasons including the difficulty in obtaining a strong, self-supporting, continuous compact with sufficiently uniform cross-sectional properties to enable it to be hot rolled after sintering.

Thus, in order to make bars and rods and the like elongated wrought products from metal powders, it has heretofore been necessary either to melt and cast the particles into a conventional billet or ingot or the like and then to follow conventional hot and/or cold working procedures to reduce the billet or ingot to a desired shape or to follow conventional powder metallurgy techniques of static compaction or direct extrusion followed by sintering and hot and/or cold working by conventional techniques. These procedures add materially to the cost of forming the desired products; they require specialized and often very costly equipment; they limit the types of products which can be produced from particulate matter; they increase the danger of contamination; they destroy desired physical characteristics of the particles which may be valuable; and they conventionally involve a multiplicity of rolling, swaging and other stages which must be applied to permit adequate size reduction.

Accordingly, it is a principal object of the present invention to provide a simple and economic process wherein powder metal roll compacting techniques are utilized to produce continuous elongated bars and rods and the like products. Another object of this invention is to provide a process wherein metal powders are roll compacted into relatively thin green strips and said strips are subsequently fabricated into continuous bars and rods and the like elongated products which have cross-sectional dimensions unrestricted by the thickness limitation imposed on the green strips by the roll diameters of the compacting mill.

Broadly, the process of this invention comprises the following sequence of steps: roll-compacting powdered metallic starting material into a self-supporting green strip of 45–95% theoretical density and having a thickness in the range of 1/50 to 1/100 of the diameter; longitudinally slitting the green strip emerging from the mill into a plurality of strips of predetermined width; forming the plurality of strips so obtained into a vertical stack; and sintering, hot rolling and otherwise hot and/or cold working the sintered stack to produce a continuous rod, bar or the like elongated product, all in a continuous sequence of steps.

The final product is of substantially 100% of the theoretical density, and it is in the form of a unitary structure, i.e. there is substantially complete metallurgical bonding between the particles and/or strips in the final product.

The term "green" strip as used herein is intended to mean strip which is formed of roll compacted metallic particles and which is in a relatively porous state but is sufficiently self-supporting that it can be handled in the initial steps of the process without disintegration.

Compactible particulate matter which can be utilized in the process of this invention includes metal powders, metal alloy powders and composite powders comprised of a metal and at least one other metal or non-metal distinct phase. In this latter category, for example, are composite metal powders, such as nickel-thoria powders, which are suitable for producing dispersion strengthened wrought materials.

The process is independent of the method by which the powders are produced. They can be produced by conventional pyrometallurgical processes involving melting followed by atomizing, sputtering, mechanical attrition and the like. However, we have found that optimum results are obtained when the powders have been derived, at least in part, from hydrometallurgical processes involving chemical precipitation of metals from aqueous media by hydrogen reduction. Such powders are readily obtainable in a wide variety of compositions and physical forms and generally possess characteristics which make them particularly suitable for roll compacting. Regardless of composition, the powder particle size should be below about 300 microns and preferably at least about 40% of the particles should be less than about 40 microns in size.

An understanding of the manner in which finely divided metallic particles can be formed into continuous bars, rods and the like elongated products by the process of this invention can be obtained from the following description, reference being made to the accompanying drawings in which:

FIGURE 1 is a schematic perspective view, with a portion broken away, of an embodiment of the process which illustrates a sequence of operations which take place in producing an elongated rod from particulate starting material in accordance with this invention;

FIGURES 2, 3, 4 and 5 are sectional end views of illustrative cross-sectional shapes which can be produced by the process of the present invention; and

FIGURES 6, 7, 8 and 9 are sectional end views of products formed in a sequence of steps applied to stacked

green strips illustrated in FIGURE 6 arranged to form bimetallic rods.

Like reference characters refer to like parts throughout the description and drawings.

Referring to FIGURE 1 which illustrates, schematically, an embodiment of the process, finely divided, compactible particles **20** are fed from a source of supply **21**, which conveniently may be a hopper shaped bin. Particles flow by gravity or are fed at a relatively uniform rate, such as by a vibrating mechanism, to the nip of rolls **22–23** which define roll gap **25**. Particles **20** are compacted by rolls **22–23** into a green strip.

The exact thickness of strip **24** will depend on the roll gap setting and the type, size, shape and hardness of the particles to be compacted. Maximum green strip thickness obtainable from any given rolling mill will be equal to about 1/50 to about 1/100 the roll diameters. Generally strips from 0.02 to 0.20 inch thick with a density of from 45% to 95% of the theoretical density of the particulate material are contemplated by the present invention.

A self-supporting green strip or sheet can be produced from dry, compactible metal particles. Alternatively, the compacting characteristics of the particles may be improved by mixing them, prior to compacting, with from 0.1% to 2.0% by weight of a liquid, such as water or other transient liquid or a lubricant which does not contaminate the resulting shape and which, preferably, is volatile or self-disposable at the temperature at which the subsequent sintering step is conducted. The specific amount of liquid or other lubricant to be used with a particular material can be readily determined having regard to the fact that if too much is used, the particles will not flow at a uniform rate and a green sheet or strip of uniform density will not be obtained when the process is conducted on a continuous basis. Water is suggested as a dampening agent but other liquids, such as carbon tetrachloride, alcohol and the like, which are evaporated during the compacting or sintering steps without leaving undesired impurities, can be employed, if desired.

The green strip or sheet **24** leaving the rolls **22–23** normally has a density of from about 45% to about 95% of the theoretical density of the particulate matter of which it is formed. It must be self-supporting and coherent to the extent that it can be passed directly to the subsequent steps of the overall process and can be slit, as shown in FIGURE 1, into strips of desired width to form the laminar structure described in detail hereinafter.

The green strip leaving the rolls **22–23** is continuously slit in a slitter **30** to form a plurality of strips **31**, **31***a*, **31***b*, **31***c*, and **31***d*, of predetermined width which are superimposed into a stack **35** in a stacker device **32** to form a laminated structure of desired thickness. The slit strips **31–31***d* can be of the same or different widths as desired.

In an alternative embodiment, not illustrated, the green strip **24** can be sintered prior to the slitting step in order to ensure that it has sufficient strength to withstand the slitting operation without disintegration. Powders which cannot readily be rolled into green strip of sufficient strength to withstand slitting are treated in this manner.

As stated above, the laminar structure **35** formed by superimposing the narrow strips one on the other can be hot worked to form an integral structure. Thereafter, the integral structure can be further cold and/or hot worked to produce the desired shape. Cold working, as known in the art, is plastic deformation below the recrystallization temperature of the particulate matter which forms the structure. It usually involves intermediate annealing steps. Hot working is plastic deformation above the recrystallization temperature of the particulate matter.

In the embodiment of FIGURE 1, the stacked strips **35** are passed to a sintering furnace **33** wherein they are heated to a hot working temperature which is above the recrystallization temperature of the particulate matter but is below the melting temperature of the stack as a whole. Low melting point particles, such as lead and tin, require a relatively low heating temperature, for example, about 30° F. below their melting temperatures. Nickel, copper and iron particles require higher temperatures but still safely below their melting temperature. The structure is sintered at a temperature sufficient to bond the particles together without using excessive temperatures which would be uneconomic and may cause undesired grain growth.

The heating step can be conducted in an inert or reducing protective atmosphere to inhibit the formation of oxide or other films on the metal surfaces which would prevent welding between particles and/or strips in the laminated structure and to remove any undesirable films, if any, which were present prior to the heating step.

The sintered, laminated structure is passed continuously from the heating furnace to the rolls **36–37**. The surfaces of these rolls are profiled, as indicated by the numerial **39** to produce a unitary continuous bar or rod of desired shape, for example, the oval shaped bar **38**, the hexagonal bar **50**, rectangular bar stock **51** and circular bar stock **52**, as illustrated by FIGURES 2, 3, 4 and 5 respectively. The working of the compacted structure can be continued according to conventional procedures to produce a final elongated bar or rod of desired shape and size and of substantialy 100% density.

FIGURES 6, 7, 8 and 9 illustrate an important embodiment of the invention whereby composite structures having a core of one composition and an outer layer of another composition are formed. The structure is formed by positioning relatively wide strips **40–40***a* formed of one material at the top and bottom respectively of a stack **41** of narrow strips formed of a different material from strips **40**, **40***a*. The top and bottom strips **40**, **40***a* extend a distance beyond the interior strips **41** sufficient to enclose the interior strips completely during the working of the structure. In carrying out this embodiment of the process, top and bottom strips **40**, **40***a* and interior strips **41** are fed from separate coils of green strip and are superimposed on stack **35** either immediately ahead of behind the stacker **32**. The structure is then hot and cold worked to produce an elongated rod or bar having an exterior surface formed of one material and a core of a different material.

The following examples illustrate the operation of the process of this invention and the results which can be obtained therefrom.

Example 1

A pure nickel powder of from about 100 to about 10 microns in size was roll-compacted to produce a porous strip compact about 0.042 inch thick with an average density of about 7.2 grams per cubic centimetre, or about 80% of the theoretical density of nickel. The green strip was heated in an atmosphere of cracked ammonia for 30 minutes at 2200° F. and then cooled to about 65° F. in the same atmosphere. The strip was then slit along its length to form eight narrow strips about 0.5 inch in width. The strips were stacked to form a bundle with dimensions of about 0.5 inch wide by 0.34 inch thick.

The bundle was heated to 1900° F. in an atmosphere of cracked ammonia and hot-rolled directly through forming rolls to produce a rod which was oval in cross-section as illustrated in FIGURE 2.

The resulting oval rod had a density of about 8.8 grams per cubic centimetre which is equivalent to 99% of the theoretical density of nickel.

The oval bar was re-heated to 1900° F. in an atmosphere of cracked ammonia and passed through forming rolls, to provide a rod which is diamond-shaped in cross section. This rod was of substantially 100% density.

Subsequent passes through oval and round forming rolls produced a substantially round rod of 0.2 inch diameter. The rod was annealed to 1500° F. and drawn through dies

to yield wire 0.125 inch diameter. After a second annealing at 1500° F., the properties of the wire were:

Ultimate tensile strength	lb./in.^{2}	50,000
Yield strength, 0.2% offset	lb./in.^{2}	8,500
Elongation—in 2 inches	percent	40

Example 2

A copper powder having an average particle size of 20 microns and produced by reacting an aqueous ammoniacal copper sulphate solution with hydrogen at elevated temperature and pressure was compacted with cold rolls having a six inch diameter. The strip had a thickness of 0.034 inch and a density 88% of the theoretical density of copper.

A similar strip was roll-compacted from nickel powder produced by hydrogen reduction from ammoniacal nickel sulphate solution. The average particle size of the nickel powder was 15 microns. This green strip was 85% dense and 0.040 inch thick.

The green copper strip was sintered in hydrogen for one hour at 1700° F. The green nickel strip was sintered for two hours in hydrogen at 2200° F. The sintered strips were slit in the rolling direction to provide 0.7 inch wide strips of copper and 0.4 inch wide strips of nickel.

A stack was then formed from 8 slit strips, the top and bottom strip of the stack being copper and the 6 intermediate strips being nickel. The stack was passed continuously through a sintering furnace and into an oval-shaped pass on the rolls of a hot rolling mill at a speed of 3 feet per minute, which permitted the stack of strips to be heated substantially uniformly in the furnace to a temperature of 1600° F. While being heated and conveyed to the entrance of the hot rolls, the stack was in a long tubular muffle through which a flow of pure hydrogen was passed. A tubular guide at the roll entrance also maintained the alignment of the strips in the stack.

The hot rolling pass was oval-shaped and gave a 40% reduction in the area of the strips. This was sufficient to fully densify the components of the stack as well as to weld the individual strips of the stack completely together. The product of the rolling operation was a fully dense, oval-shaped rod, such as illustrated in FIGURE 7, consisting of a nickel core completely coated with copper.

Example 3

Iron power of 25 microns average particle size was roll compacted to produce 4 inches wide green strip of 0.030 inch thickness nad of 70% of the theoretical density of solid iron.

A similar green strip was rolled from nickel powder produced by reduction from an aqueous ammoniacal nickel sulphate solution with hydrogen at elevated temperature and pressure. The average particle size of the nickel particles was 18 microns. This strip was 4 inches wide, 0.045 inch thick, and 88% of the theoretical density.

Each of the above strips was sintered by heating in cracked ammonia for two hours at 2150° F. The porous sintered strips were then slit along their lengths in the rolling direction to form four strips of iron and four strips of nickel, each 1 inch wide.

The strips were then formed into a stack of alternate nickel and iron strips.

The stack was heated in a flow of hydrogen in a small tubular muffle by induction to a temperature of 200° F. The exit end of the muffle was located at the entry to the roll gap between two profiled hot rolls, 4 inches in diameter, rotating at 3 r.p.m. The rolls were used to pull the stack through the induction heating coil continuously, and simultaneously to consolidate the porous strips of the stack into a dense rod by effecting a reduction in cross sectional area of 35%. The resultant dense, hot rolled rod was an iron-nickel laminated structure with complete metallurgical bonding between the components of the laminate.

Example 4

Substantially pure cobalt particles of random sizes within the range of from 50 to 200 microns were compacted into 83% dense strip 4 inches wide and 0.03 inch thick. The strip was slit into a plurality of strips 0.5 inch wide. A stack of strips was formed. The stack was sintered at 1150° C. in an atmosphere of cracked ammonia. The sintered stack was then hot rolled through each of four sets of profiled rolls at a temperature of 1000° C. for the first pass and 700° C. for the following three passes. The final product was a round rod 0.212 inch in diameter and of substantially 100% density. The rod had an ultimate tensile strength of 99,000 pounds per square inch and an elongation of 7%.

The process of the present invention possesses a number of important advantages. It permits the utilization of powder rolling techniques to form compactible metallic material into elongated continuous products, such as rods and bars, which has cross-sectional dimensions not normally obtainable by powder rolling because of the inherent limitation on compact thickness imposed by the roll diameters.

Also, the development, in recent years of thermal reactors and ancillary apparatus, thermal engines, such as used in jet aircraft, rockets, missiles and the like has increased a demand for metal products which are capable of high resistance to creep for prolonged periods of time at temperatures above the normal re-crystallization temperature of common metals and their alloys. This has created a demand for what are now known as dispersion strengthened materials comprised of finely divided particles of a reinforcing or a strengthening component or components dispersed in a solid solution, or matrix phase, of a metal or metal alloy in which the stiffening or strengthening component is substantially insoluble. It is found to be very difficult, if not impossible, to form, by conventional melting and casting procedures, products in which the reinforcing or stiffening element is uniformly dispersed throughout the entire shape. It is found, however, that finely divided particles of metal or metal alloy and stiffening or strengthening component or components can be prepared as a substantially uniform mixture and compacted by the process of this invention to produce elongated rods and bars of substantially 100% density with substantially uniform chemical and physical properties throughout.

It will be understood that modifications can be made in the preferred embodiment of the invention described and illustrated herein without departing from the scope of the invention defined by the appended claims.

What we claim as new and desire to protect by Letters Patent of the United States is:

1. A process of forming finely divided compactible metallic powders into continuous bars and rods and the like elongated products which comprises the following steps in sequence: feeding a finely divided, compactible metallic powder to a roll compacting unit to produce a continuous self-supporting green strip having a thickness in the range of $\frac{1}{50}$ to $\frac{1}{100}$ of the diameter of the rolls of said compacting unit and a density equivalent to about 45% to 95% of the theoretical density of the material of which said compactible metallic powder is formed; slitting said green strip along its length into a plurality of strips of predetermined width; forming a vertical stack of predetermined thickness of said plurality of strips; passing said stack to a sintering operation at a temperature below the melting point of said metallic powder; and thereafter mechanically working the sintered stack to form a continuous unitary bar, rod or the like elongated wrought product having a density substantially equivalent to 100% of the theoretical density of the material from which said product is formed.

2. A process according to claim 1 including the additional step in which said green strip is subjected to a sintering step at a temperature below the melting point of said metal prior to the slitting and stacking steps.

3. A process according to claim 1 in which said vertical stack comprises at least three strips, the top and bottom strips of said stack being of wider width than the interior strips whereby said interior strips are caused to be enclosed along their lengths by said top and bottom strips when said stack is subjected to said working operation to form it into an elongated wrought product.

4. A process according to claim 1 in which said finely divided metallic powder is formed of a member selected from the group consisting of copper, nickel, cobalt, iron and composite powders containing at least one of said metals as a major constituent.

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