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[54] **EXTRUSION OF CANNED METAL POWDERS USING GRAPHITE FOLLOWER BLOCK**

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[51] **Int. Cl.** **B22f 3/20**

[58] **Field of Search** 75/214, 226; 264/.5; 72/273

[56] **References Cited**

UNITED STATES PATENTS

3,428,717	2/1969	Accary et al.	264/.5
2,736,429	2/1956	Sejournet et al.	72/273
2,659,131	11/1953	Leontis et al.	75/226

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[57] **ABSTRACT**

In hot extruding a batch of dispersion-strengthened metal powder confined in a metal can through an extrusion die in which a uniformly high extrusion strain rate must be obtained, a graphite follower block of substantially the same diameter as the can is inserted between the end face of the extrusion ram and an end face of the heated can, the temperature of the block being at least substantially that of the canned powder, such that during extrusion, friction is markedly reduced by substantially inhibiting metal flashback from the can and the necessary high strain rate is obtained.

9 Claims, No Drawings

EXTRUSION OF CANNED METAL POWDERS USING GRAPHITE FOLLOWER BLOCK

This invention relates to a method for extruding metal powders confined in a metal can or sheath and, in particular, to a method of hot extruding canned metal powder at elevated temperatures while substantially reducing working friction during extrusion.

STATE OF THE ART

It is known that wrought metal shapes may be produced from metal powders by confining and sealing metal powder in a metal can, such as in a can of mild steel or nickel, and then hot extruding the canned assembly at an elevated extrusion temperature through a die to form a dense wrought metal structure. The resulting extruded shape has a thin skin thereon remaining from the metal can which may be removed, if desired, by acid pickling, by grinding or by other suitable means.

Generally, in hot extruding metal shapes from cylindrical billets of heat resistant alloys, it is not uncommon to use extrusion lubricants, such as certain temperature resistant lubricating oils, or inorganic lubricants, such as low melting glass in the form of a fibrous glass pad placed between the head of the extrusion ram and an end face of the billet and/or a fibrous sheath of low melting glass (e.g., several plies of glass fabric) in which the hot billet is wrapped before it is inserted into the extrusion chamber. While such lubricants have been extremely useful in inhibiting friction during extrusion, they have not been sufficient in the hot extrusion of canned dispersion-strengthened powders in which achievement of uniformly high strain rate is important.

During the extrusion of canned powders, considerable hydrostatic pressure is generated, building up hoop stresses in the extrusion container. The stresses may cause radial expansion of the container with concurrent development of an annular gap between it and a steel follower block. Because of this, there is a tendency for the soft can material to be back-extruded, forming a thin flashing. This generally results in high extrusion friction with the attendant increase in power required to continue the extrusion. Flashings up to 10 square inches or more in area have been observed on extrusion stubs.

Because of the foregoing problem, it was generally difficult to assure the desired extrusion conditions for dispersion-strengthened heat resistant alloys using oil and/or glass lubrication and, consequently, the desired mechanical properties and metallographic structure of the finally extruded product were not always obtained.

This was particularly true of dispersion-strengthened mechanically alloyed composite metal powders produced in accordance with the method described in Benjamin U.S. Pat. No. 3,591,362 assigned to the same assignee. In its broad aspects, the method comprises mixing a compressively deformable metallic powder with at least one other powdered material from the group consisting of a nonmetallic material and another metallic material and dry milling the mixture under conditions of repeated mutual impact compression sufficiently energetic to substantially reduce the thickness of at least the compressively deformable metallic constituents of the mixture and for a time sufficient to produce non-pyrophoric wrought composite particles which individually have substantially the composition of the mixture.

In a particular embodiment of the said method, a dry charge of attritive elements (e.g., nickel balls of plus one-fourth minus one-half inch average diameter) and a powder mass of predetermined composition is provided comprising a plurality of constituents, at least one of the constituents being a compressively deformable metal in an amount of at least 15 percent by volume, with the remainder of the powder mass being at least one other constituent from the group consisting of a non-metal and another metal, the metals having a melting point of at least 1,000°K. The volume ratio of the attritive elements to the powder mass is at least about 4:1 and, more advantageously, at least about 10:1. The charge is then subjected to agitation milling under conditions in which a substantial portion of the attritive elements is maintained kinetically in a highly activated state of relative motion, whereby to cause the constituents to unite and form composite metal particles, the milling being continued until cold worked composite metal particles are produced characterized by markedly increased hardness (that is, the particles contain a substantial amount of stored energy) and further characterized by an internal structure in which the constituents are intimately dispersed. Thus, when the particles, which, in a preferred embodiment, are heavily cold worked to reach substantially the saturation hardness of the system involved, are subjected to a diffusion heat treatment, the intimately interdispersed constituents diffuse one into the other rather quickly to produce a homogenized matrix.

The foregoing method is particularly applicable to the production of wrought composite metal particles of a broad range of compositions, such as heat resistant alloy compositions, comprising by weight up to about 65 percent chromium, up to about 8 percent aluminum, up to about 8 percent titanium, up to about 40 percent molybdenum, up to about 40 percent tungsten, up to about 20 percent columbium, up to about 40 percent tantalum, up to about 5 percent vanadium, up to about 15 percent manganese, up to about 2 percent carbon, up to about 3 percent silicon, up to about 1 percent boron, up to about 2 percent zirconium, up to about 6 percent hafnium, up to about 0.5 percent magnesium, up to about 10 percent by volume of a hard refractory compound, preferably 0.5 to 10 percent by volume, and the balance of the composition essentially at least about 25 percent by weight of at least one metal from the group consisting of iron, nickel and cobalt.

In its more particular aspects, the foregoing method is applicable to the production of dispersion-strengthened superalloys having a matrix composition normally very difficult to produce by conventional powder metallurgy techniques, including alloys falling within the range of about 5 to 35 percent or even up to 60 percent chromium, about 0.5 to 6.5 percent aluminum, about 0.5 to 6.5 percent titanium, up to about 15 percent molybdenum, up to about 20 percent tungsten, up to about 10 percent columbium, up to about 10 percent tantalum, up to about 3 percent vanadium, up to about 2 percent manganese, up to about 2 percent silicon, up to about 0.75 percent carbon, up to about 0.1 percent boron, up to about 1 percent zirconium, up to about 0.2 percent magnesium, up to about 4

percent hafnium, up to about 35 percent iron, about 0.5 to 10 percent by volume of a refractory dispersoid, and preferably about 0.5 to 5 percent by volume, and the balance essentially at least one of the metals nickel and cobalt in an amount at least about 40 percent of the total composition. Alternatively, dispersion-strengthening of mechanically alloyed powder may occur during milling due to the inadvertent pickup of oxygen in forming an oxide with titanium, aluminum or other strong oxide formers.

Mechanically alloyed dispersion-strengthened composite metal powders of the foregoing type are hot worked by confining the desired composition in a mild steel can. The can at the extrusion temperatures employed is very soft and, at a temperature of 1,900°F., exhibits a strength of about 4,200 psi at a strain rate of about 1.67×10^{-3} per second. Because of the low absolute strength of the can and the considerable hydrostatic pressure generated during extrusion which causes the extrusion chamber to expand and form an annular gap between it and the metal follower block, the soft can material may be back extruded and thus form a thin flashing which results in high extrusion friction which interferes with achieving uniformly high strain rates essential in the subsequent production of desirable metallographic structures.

When the foregoing occurred, the desired mechanical properties were not always obtained in the final product. This was particularly true in the case where extrusion was employed at correlated extrusion ratios and temperatures preliminary to the production of products having preferred coarse grained microstructures obtained by subsequently heating the extended product to an elevated germinative grain growth temperature to produce coarse elongated grains oriented in the extrusion direction. It was found, according to copending application Ser. No. 52,378 now abandoned, filed on July 6, 1970 in the names of John S. Benjamin, Robert L. Cairns and John H. Weber, and assigned to the same assignee, that when the proper extrusion ratio and temperature were employed for dispersion-strengthened alloys, the desired coarse grains could thereafter be obtained by proper heat treatment at the germinative grain growth temperature. It was also found that where the desired coarse grains were obtained, preferably oriented in the direction of working, such microstructures generally resulted in markedly improved stress-rupture properties at elevated temperatures as high as 1,900°F. and higher. However, it was observed that the results tended to be inconsistent, particularly where strain rates where low due to high friction in the extrusion chamber caused by back extruded flashings. Where such conditions prevailed, the microstructure following the grain growth heat treatment tended to be non-uniform throughout the metal shape and the mechanical properties were adversely affected.

It is thus the object of this invention to provide a method of hot extruding canned metal powders while avoiding back extrusion of the metal can.

Another object is to provide a method of hot extruding canned metal powders while substantially inhibiting friction in the extrusion chamber during extrusion, thereby maintained the required high strain rates.

A further object is to provide an improved method for hot extruding dispersion-strengthened superalloys, whereby to provide enhanced high temperature stress-rupture and creep properties.

These and other objects will more clearly appear when taken in conjunction with the following disclosure.

STATEMENT OF THE INVENTION

Stating it broadly, the invention resides in a method of producing a wrought dispersion-strengthened metal shape by hot extruding a batch of dispersion-strengthened metallic powder confined in a metal can, a substantial portion of the particles thereof having a hardness substantially greater than that of the metal can, the conditions of extrusion being such that metal flash-back from the metal can within the extrusion chamber is substantially inhibited while markedly reducing friction during extrusion. The improvement resides in inserting a hot graphite block between the head of the extrusion ram and the canned assembly in the extrusion chamber, the diametral dimension of the graphite block being substantially that of the assembly. The temperature of the hot graphite block should be at least substantially the temperature of the canned assembly. The hot graphite follower block crushes or otherwise deforms under the applied extrusion force and forms a seal between the canned assembly and the metal follower block, thus preventing back extrusion of metal flashing from the can. The graphite follower block should be of sufficient thickness to prevent formation of metal flash, e.g., a thickness of four inches was found satisfactory.

As stated hereinbefore, the invention is particularly applicable to the hot extrusion of mechanically alloyed powder, particularly the hot extrusion of such powders composed of dispersion-strengthened heat resistant alloys, e.g., age hardenable superalloys. One aspect of the invention resides in providing a batch of mechanically alloyed composite particles formed of constituents which, when alloyed together, provide an age hardenable dispersion-strengthened superalloy, the composite particles having a hardness of at least about 50 percent of the difference between its base hardness in substantially the unworked condition and its saturated hardness is substantially the fully cold worked condition. The expression "substantially saturated hardness" is meant to cover the hardness range mentioned hereinabove. The foregoing hard composite particles are characterized metallographically by an internal structure comprising said constituents substantially intimately united and interdispersed. A confined shape of the mechanically alloyed composite particles, that is, a canned assembly thereof, is hot extruded at a temperature of over about 1,690°F. and ranging up to about 2,150°F. or 2,210°F. correlated to reduction ratios ranging broadly from over about 6.3 to less than about 35, and at a strain rate greater than a minimum value defined hereinafter, such that when the resulting hot extruded alloy is subsequently heated to an elevated germinative grain growth or annealing temperature, coarse grains are formed elongated in the working direction of the extruded alloy shape. For example, where the alloy shape is one obtained by extruding a canned cylindrical assembly of 3.5 inches in

diameter heated to 2,000°F. at a ram speed of at least 1 inch per second to a rod three-quarters of an inch in diameter using a graphite block heated to the same temperature, the coarse grains formed by germinative grain growth heat treatment of the extruded rod are elongated like fibers in the direction of working, that is, in the longitudinal direction of the rod. Similarly, where the final extruded shape has a rectangular cross section, the coarse grains may be platelike in shape, the major axis of each grain being generally disposed in the extrusion direction. By using a hot graphite block disposed between the head of the extrusion ram and the end face of the can, a smooth extrusion stroke and a controllable strain rate in extrusion are assured due to markedly reduced friction, whereby the product has markedly improved properties and grain structure.

The invention is particularly applicable to the hot extrusion of a dispersion-strengthened, age hardenable nickel-base alloy having a nominal composition consisting essentially by weight of about 20 percent chromium, about 2.4 percent titanium, about 1.2 percent aluminum, about 0.07 percent zirconium, about 0.007 percent boron, about 0.05 percent carbon, and the balance essentially nickel. The dispersoid added to the composition, e.g., THO_2, Y_2O_3, and the like, may be nominally about 2.25 volume percent. The foregoing superalloy hot extruded at a controlled strain rate in accordance with the invention exhibits improved high temperature stress-rupture properties after it is subjected to a germinative grain growth heat treatment at a temperature of at least about 2,250°F. Thereafter, the alloy may be further heat treated and age hardened. When the conditions of extrusion and of subsequent germinative grain growth heat treatment are right, stress-rupture properties in excess of 1,000 hours at 15,000 psi at 1,900°F. are obtainable on the foregoing alloy.

As stated hereinbefore, the strain rate should be greater than a minimum value to be defined hereinafter. The actual strain rate during extrusion cannot be determined by direct measurement; however, the ram speed of the extrusion ram can be measured directly. It is considered (see Feltham, "Extrusion of Metals" *Metal Treatment and Drop Forging*, November 1956, pages 440 to 444) that strain rate during extrusion is a direct function of ram speed V and an inverse function of extrusion billet diameter D. Thus, Feltham propounds the following equation for strain rate as applied to extrusions of circular sections:

$$de_d/dt = 6V/D \cdot 1n\ D/d \qquad (1)$$

where d is the diameter of the extruded bar.

It is thus shown that strain rate is directly proportional to the speed of the extrusion ram and is inversely proportional to the diameter of the extrusion billet (or the diameter of the press liner). Clearly, strain rate is affected by the size of the extrusion press liner as well as temperature and extrusion ratio (strain). Examination of a multitude of data obtained from extruded bar produced in a 750 ton Loewy-BLH Hydropress extrusion press having a 3.5 inch diameter extrusion liner and having the exemplary nominal composition set forth hereinbefore produced using varying combinations of extrusion temperature and extrusion ratio demonstrated that a single number describing the minimum required strain rate or extrusion ram speed would not be satisfactory. Consideration of the data developed has led to a semi-empirical relationship which may be expressed as follows:

$$E_{tm} = 1n\ \phi - KD/V \exp(-Q/RT) \qquad (2)$$

where

ϕ = extrusion ratio

D = extrusion press liner diameter

T = extrusion temperature in °K.

Q = 65,000 calories per mole

R = gas constant

V = ram speed in inches per second

K is calculable as 2.175×10^{10} per second and E_{tm}, the thermomechanical energy component, is calculable as 2.028 on the basis of experimental data.

It is considered that the sum of the energy contributed by mechanical alloying of the initial metal powders "E_{MA}" and the thermomechanical energy component "E_{tm}" should equal or exceed a value "$E_{Crit.}$ in order for the consolidated bar to exhibit germinative grain growth in the subsequent high temperature annealing operation.

Equation (2) may be solved for the quantity V/D to provide the required minimum extrusion ram speed as follows:

$$V/D = K \exp(-Q/RT)/1n\ \phi - E_{tm} \qquad (3)$$

In equation (3), the value "E_{tm}" may range from 1.793 to 2.250 with the constant "K" ranging, respectively, from 0.64×10^{10}/sec. to 6.40×10^{10}/sec. to provide the required minimum extrusion ram speed applicable to the reduction ratio-temperature parameters described hereinbefore. The use of the graphite block assures achieving and maintaining at least the minimum extrusion strain rate.

DETAIL ASPECTS OF THE INVENTION

As stated hereinbefore, the invention is particularly applicable to the hot extrusion of canned metal powder in which the powder has a hardness substantially higher than that of the metal can containing it. Examples of materials employed in the construction of metal cans include mild steel, nickel and 18/8 stainless steel. It is particularly advantageous for economic reasons to employ mild steel. These materials, mild steel especially, are relatively soft at the extrusion temperature and tend to back extrude in the extrusion chamber and increase extrusion friction, unless the precautions of the invention are taken.

As illustrative of the invention when applied to the hot extrusion of mechanically alloyed powders, the following example is given:

EXAMPLE I

In preparing composite metal particles for extrusion corresponding to the composition of the preferred alloy set forth hereinbefore, except for the additional presence of about 2.25 percent by volume of yttria as the dispersoid, a nickel-titanium-aluminum master alloy is first prepared by vacuum induction melting. The resulting ingot is crushed and ground to minus 200 mesh powder. The powder (powder A) contains 72.93 percent nickel, 16.72 percent titanium, 7.75 percent aluminum, 1.55 percent iron, 0.62 percent copper, 0.033 percent carbon 0.050% Al_2O_3 and 0.036% TiO_2. About 14.9 percent weight percent of this powder is

blended with 63.7 percent carbonyl nickel powder having a Fisher subsieve size of about 5 to 7 microns; 19.8 percent chromium powder having a particle size passing 100 mesh, 0.25 percent of a Ni-28 percent zirconium master alloy passing 200 mesh, 0.04 percent of a Ni-17 percent boron master alloy passing 200 mesh and about 1.3 percent by weight of yttria of particle size of about 350 A. A 10 kilogram weight of the powder blend is dry milled in an attritor mill of the Szegvari type using 10 gallons (about 390 pounds) of plus ¼ inch carbonyl nickel pellets or balls, at a ball to powder volume ratio of about 18 to 1 in a sealed air atmosphere for about 20 hours with an impeller speed of 182 rpm.

Such an attritor mill is shown at page 8–26 of Perry's *Chemical Engineers Handbook*, Fourth Edition, 1963, and comprises an upstanding cylinder which may be provided with a surrounding cooling jacket having inlet and outlet ports for circulating a coolant, such as water. A coaxially supported rotatable shaft having horizontally extending arms integral therewith is fitted within the cylinder. The mill is filled with attritive elements, e.g., balls, sufficient to bury at least some of the arms so that, when the shaft is rotated, the ball charge, by virtue of the agitating arms passing through it, is maintained in a continual state of unrest or relative motion throughout the bulk thereof. The time of milling is sufficient to produce wrought composite metal particles of substantially saturation hardness. Several batches of the powder are made by the foregoing method, the batches being thereafter sieved to remove abnormally large particles, for example, plus 45 mesh. The microstructure of the particles making up the powder is characterized by nearly complete homogeneity, when viewed optically at 250 diameters, comprising each of the constituents substantially intimately united and dispersed. Increasing the time of milling at 132 rpm from 20 to 40 hours markedly improves homogeneity of the mechanically alloyed powder to the point that fragments of the starting ingredients become practically indistinguishable upon optical examination at 250 diameters. Experience indicates that in the aforementioned mill, the structural homogeneity obtained after 20 hours milling at 182 rpm is about the same as that obtained upon 40 hours milling at 132 rpm.

In producing an extruded shape of the alloy, sufficient weight of the composite powder of minus 45 mesh is confined within a mild steel extrusion can which may, if desired, be evacuated at 350°C., and is sealed by welding. The size of the assembly corresponds to about a diameter of about 3.5 inches. A plurality of billet assemblies was produced in this way and each assembly was then hot extruded at a full throttle setting for the press using varying conditions within the container including a hot graphite follower block behind the billet.

Tests were conducted comparing the results obtained using only the ram of the extrusion press behind the billet, using a pad of glass wool over the head of the ram, using a cold graphite follower block between the ram and the billet and using the hot graphite follower block of the invention. In each case, billet and die lubrication comprised swabbing the container with a high temperature grease, wrapping the hot billet with fiberglass cloth and using a fiberglass pad between the die and the leading end of the billet as a die lubricant. The graphite blocks used were about four inches thick. The nominal composition by weight of the mechanically alloyed composite metal powder was as follows: about 20 percent chromium, about 2.4 percent titanium, about 1.2 percent aluminum, about 0.07 percent zirconium, about 0.007 percent boron, about 0.05 percent carbon, about 1.3% Y_2O_3, and the balance essentially nickel. The results set forth in the following Table I were obtained.

TABLE I

Extrusion designation	Extrusion ratio	Temp., °F.	Follower block	Extrusion* speed in/sec.	Metal flash	Recrystallization behavior
A	31.4	2,000	None	Stalled	Yes	Poor.
B	21.8	1,950	do	1-0.5	Yes	Do.
C	21.8	1,950	do	1-0.5	Yes	Do.
D	21.8	2,075	Fiberglass pad	0.5-1	Yes	Fair.
E	21.8	1,950	Cold graphite	1-0.25	None	Poor.
1	16.0	1,900	Hot graphite	5	do	Good.
2	16.0	1,950	do	6	do	Do.

*=Speed range from beginning to end of extrusion.

The data show clearly that the use of hot graphite follower blocks will eliminate or inhibit metal flash-back and increase the general range of extrusion speeds above the minimum required. The use of no follower block (only the extrusion ram) resulted in low extrusion speeds and gave poor results. The use of a glass wadding or pad ahead of the ram likewise did not provide good results. A cold graphite follower block resulted in an increase in friction and gave poor results. However, when a hot follower block was employed at substantially the temperature of the canned assembly, smoother extrusions were obtained as evidenced by higher extrusion speeds.

In general, the poor results obtained are reflected in the recrystallization behavior and the mechanical properties of the product after extrusion. As stated in copending application Ser. No. 52,378 now abandoned, when the extrusion conditions are satisfactory (that is, the proper extrusion ratio and extrusion strain rate is correlated to the proper extrusion temperature), coarse elongated grains are obtained, after heating to an elevated germinative grain growth temperature, such that markedly improved mechanical properties are obtained.

In working with dispersion hardened, age hardenable nickel-base superalloys, the proper correlated extrusion ratio and extrusion temperature have been determined in conjunction with the aforementioned extrusion press having a 3.5 inch diameter container as being broadly extrusion ratios of over about 6.3 to about 35 correlated with billet heating temperatures (extrusion temperatures) of about 1,690°F. to about 2,210°F., preferably extrusion ratios of about 8.5 to about 25 correlated with billet heating temperatures of about

1,775°F. to about 2,150°F. It is to be appreciated that other extrusion presses may not be so limited in their capacity to deliver a given strain rate at a given combination of extrusion temperature and extrusion ratio. In such cases the area of acceptable extrusion conditions will be determined by the combinations of extrusion temperature and extrusion ratio at which the press delivers a ram speed in excess of that defined by Equation (3).

Following the production of the hot extruded alloy shape using a hot graphite follower block, the extruded product is subjected to a heat treatment comprising at least a first step at an elevated annealing temperature to solution treat, homogenize and germinatively grow the grains and form large coarse grains with one or two major axes disposed in the direction or directions of working. An optional step may be employed in which the alloy is treated to prepare it for aging. A third heat treating step may or may not be employed to age the alloy to the desired hardness and strength. However, an aging step may not be required where the alloy is used at a temperature at which aging occurs in situ. The latter step may comprise a series of aging sub-steps of succeeding lower temperatures where desirable. Thus, for an alloy comprising nominally the preferred composition set forth hereinbefore, a three-step heat treatment found particularly advantageous comprises: (1) heating at a grain growth temperature of about 2,325°F. to 2,400°F. for up to 2 hours in a protective environment, e.g., argon, and air cooling; (2) thereafter heating at a solution temperature of 1,975°F. for 7 hours in air followed by air cooling; and (3) finally aging the alloy at 1,300°F. for 16 hours in air and then air cooling. Optionally, steps (2) and (3) can be replaced by a heating step comprising heating at 1,300°F. for about 24 hours in air and then air cooling. The first step in either case results in a marked increase in grain size having a preferred orientation relative to the extrusion direction. For example, as stated hereinbefore, in the case of the elongated extruded product, the coarse grains are elongated and are disposed or exhibit a preferred orientation in the direction of extrusion, that is, the longitudinal axis of the elongated product. In the case of a hot extruded product in which the cross section is somewhat rectangular, the grains tend to be plate-like and to be disposed or show a preferred orientation in the direction of extrusion.

The coarse grains generally exhibit aspect ratios of greater than about 3:1, in some cases greater than 10:1 or 15:1 or even higher. The aspect ratio is that ratio that defines grain configuration correlated to the direction of interest, e.g., direction of applied stress. The ratio is determined as the average dimension of the grain parallel to the direction of interest divided by its average dimension along a minor axis.

Commensurate with the formation of the coarse grain structure is an incremental improvement of the stress-rupture properties at both intermediate temperatures, e.g., 1,400°F., and at high temperatures, e.g., 1,900°F., determined along the direction of interest.

While the invention has been described in conjunction with a nickel-base alloy, the invention is particularly applicable to the following range of compositions: about 15 to 25 percent chromium, about 0.5 to 2.5 percent aluminum, about 1 to 5 percent titanium, up to about 5 percent molybdenum, up to about 5 percent tungsten, up to about 2 percent columbium, up to about 4 percent tantalum, up to about 1 percent vanadium, up to about 2 percent manganese, up to about 1 percent silicon, up to about 0.2 percent carbon, up to about 0.1 percent boron, up to about 0.5 percent zirconium, up to about 0.2 percent magnesium, up to about 2 percent hafnium, up to about 10 percent iron, about 0.5 volume percent to 5 volume percent of a dispersoid, the balance essentially at least about 40 percent nickel. Generally speaking, the alloys have a melting point of at least about 2,300°F. The composition may comprise cobalt since nickel is generally considered an equivalent of cobalt. The dispersoid may include those selected from the group consisting of ThO_2, Y_2O_3, ceria and the rare earth mixtures didymia and Rare Earth Oxides, and other oxides having free energy of formation exceeding 90 kilocalories per gram atom of oxygen at about 25°C. The size of dispersoid found advantageous in producing dispersion-strengthened superalloys may range from about 50 A to 5,000 A, and, more advantageously, from about 100 A to 1,000 A.

The product provided in accordance with the invention is useful in the production of articles such as gas turbine blades and vanes and other articles subjected in use to the combined effects of elevated temperature and stress.

Although the present invention has been described in conjunction with preferred embodiments, it is to be understood that modifications and variations may be resorted to without departing from the spirit and scope of the invention as those skilled in the art will readily understand. Such modifications and variations are considered to be within the purview and scope of the invention and the appended claims.

We claim:

1. In a method of producing a wrought metal shape by hot extruding a batch of dispersion-strengthened metallic powder confined in a can made of metal which tends to become relatively soft at the extrusion temperature such that during extrusion metal flash-back from the can tends to occur between the extrusion ram and the extrusion cylinder which interferes with the extrusion efficiency by increasing extrusion friction, the improvement which comprises,

inserting a hot graphite block of substantially the same diametral dimension as the canned powder between the end face of the ram and the end face of the canned powder at a temperature at least substantially that of the canned powder, and then hot extruding the canned powder through the extrusion die by force applied by the ram against the hot graphite block, whereby metal flash-back of the can is substantially inhibited and the friction of extrusion markedly reduced.

2. In a powder metallurgy method of producing a wrought metal shape by hot extruding a batch of dispersion-strengthened metallic powder confined in a can made of metal which tends to become relatively soft at the extrusion temperature such that during extrusion metal flash-back from the can tends to occur between the extrusion ram and the extrusion cylinder which interferes with the extrusion efficiency by increasing extrusion friction, the improvement which comprises,

providing a canned batch of mechanically alloyed composite metal powder of substantially saturated hardness formed of constituents which, when alloyed together, provide a dispersion-strengthened heat resistant alloy, inserting a hot graphite block of substantially the same diametral dimension as the canned powder between the end face of the ram and the end face of the canned powder at a temperature at least substantially that of the canned powder, and then hot extruding said canned batch of powder at a temperature of over about 1,690°F. and at a reduction ratio of at least about 6.3 by force applied by the ram against the hot graphite block,

whereby metal flash-back of the can is substantially inhibited and the friction of extrusion markedly reduced, said hot extrusion being carried out at a strain rate such that when the resulting hot extruded alloy is subsequently heated to an elevated germinative grain growth temperature, coarse grains are formed with a major axis disposed in the extrusion direction of the alloy shape.

3. The method of claim 2, wherein the hot extrusion is carried out at an extrusion ram speed at least as great as that determined by the formula:

$$V/D = K \exp(-Q/RT)/1n\,\phi - E_{tm};$$

where

V = ram speed in inches per second
D = billet diameter in inches
ϕ = extrusion ratio
Q = 65,000 calories per mole
R = gas constant
T = temperature in °K.
K = a constant ranging from 0.64 to 10^{10}/sec. to 6.40 $\times 10^{10}$/sec.
E_{tm} = a value ranging from 1.793 to 2.250

such that when the resulting hot worked alloy is subsequently heated to an elevated germinative grain growth temperature, coarse grains are formed with a major axis disposed in a principal working direction of the alloy shape.

4. The method of claim 2, wherein the hot extrusion is carried out at a temperature selected from the range of 1,775° to 2,100°F. and a reduction ratio between 8.5 and 25.

5. The method of claim 2, wherein the dispersion-strengthened heat resistant alloy has a composition ranging by weight from about 5 to 60 percent chromium, about 0.5 to 6.5 percent aluminum, about 0.5 to 6.5 percent titanium, up to about 15 percent molybdenum, up to about 20 percent tungsten, up to about 10 percent columbium, up to about 10 percent tantalum, up to about 3 percent vanadium, up to about 2 percent manganese, up to about 2 percent silicon, up to about 0.75 percent carbon, up to about 0.1 percent boron, up to about 1 percent zirconium, up to about 0.2 percent magnesium, up to about 6 percent hafnium, up to about 35 percent iron, about 0.5 to 10 percent by volume of a refractory dispersoid, and the balance essentially a metal from the group consisting of nickel and cobalt in an amount at least about 40 percent of the total composition.

6. The method of claim 5, wherein the composition ranges from about 15 to 35 percent chromium, about 0.5 to 2.5 percent aluminum, about 1 to 5 percent titanium, up to about 5 percent molybdenum, up to about 5 percent tungsten, up to about 2 percent columbium, up to about 4 percent tantalum, up to about 1 percent vanadium, up to about 2 percent manganese, up to about 1 percent silicon, up to about 0.2 percent carbon, up to about 0.1 percent boron, up to about 0.5 percent zirconium, up to 0.2 percent magnesium, up to 2 percent hafnium, up to about 10 percent iron, about 0.5 volume percent to 5 volume percent of a dispersoid, the balance essentially at least about 40 percent nickel.

7. The method of claim 6, wherein the chromium content of the alloy does not exceed about 25 percent.

8. The method of claim 7, wherein the alloy following hot working is heated to its germinative grain growth temperature to form coarse elongated grains disposed in the working direction of the alloy.

9. The method of claim 1 in which the metal of the can is selected from the group consisting of mild steel, stainless steel and nickel.

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