United States Patent [19]

Nadkarni et al.

[11] **3,884,676**

[45] **May 20, 1975**

[54] **DISPERSION STRENGTHENING OF METALS BY IN-CAN PROCESSING**

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[73] Assignee: **SCM Corporation,** New York, N.Y.

[22] Filed: **June 27, 1973**

[21] Appl. No.: **373,998**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 217,506, Jan. 13, 1972, Pat. No. 3,779,714.

[52] **U.S. Cl.** ..... **75/.5 BC;** 148/11.5 R; 148/11.5 F

[51] **Int. Cl.** **B22f 1/04**

[58] **Field of Search** ..... 75/.5 BC, 206, 211; 148/11.5 R, 11.5 F; 29/182.5, 191.2

[56] **References Cited**

UNITED STATES PATENTS

3,505,059	4/1970	Cerulli	75/.5 BC
3,515,542	6/1970	Larsen	75/135
3,552,954	1/1971	McDonald	75/.5 BC

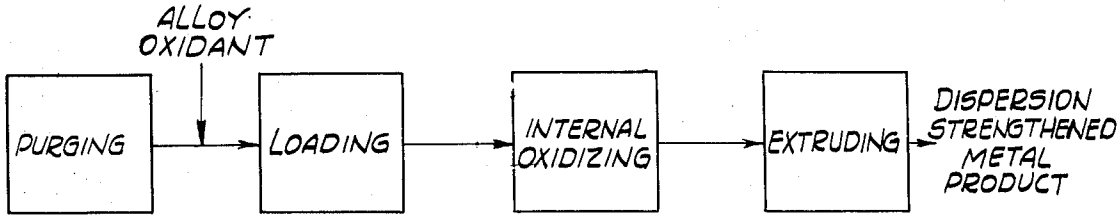
Primary Examiner—W. Stallard

Attorney, Agent, or Firm—Thomas M. Schmitz

[57] **ABSTRACT**

A self-contained powder metal mixture is dispersion strengthened within a sealed container by internal oxidation and thereafter extruded from the same sealed container to produce dispersion-strengthened metal stock or articles. The metal mixture comprises a mixture of powdered alloy and oxidant for complete internal oxidation of the alloy by the oxidant, and is adapted to dispersion strengthen the residue of oxidant upon coalescence and hot-working thereof in the extrusion step to produce dispersion-strengthened metal articles directly from the same container utilized for internal oxidation.

8 Claims, 4 Drawing Figures



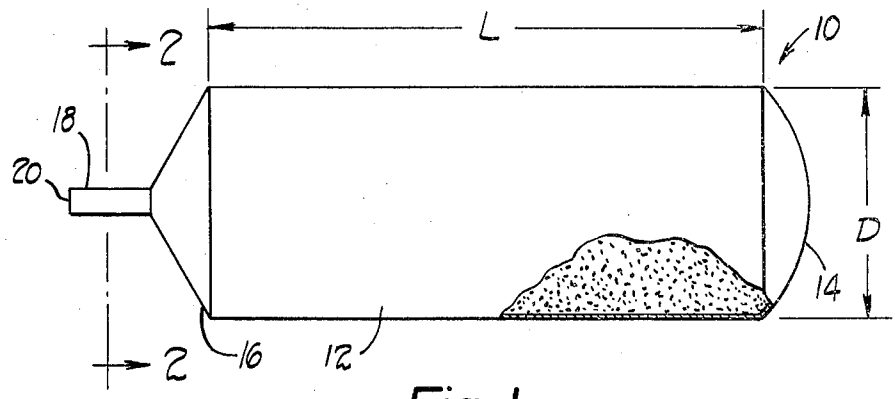


Fig. 1

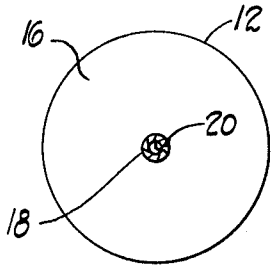


Fig. 2

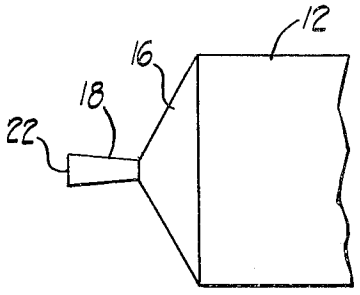


Fig. 3

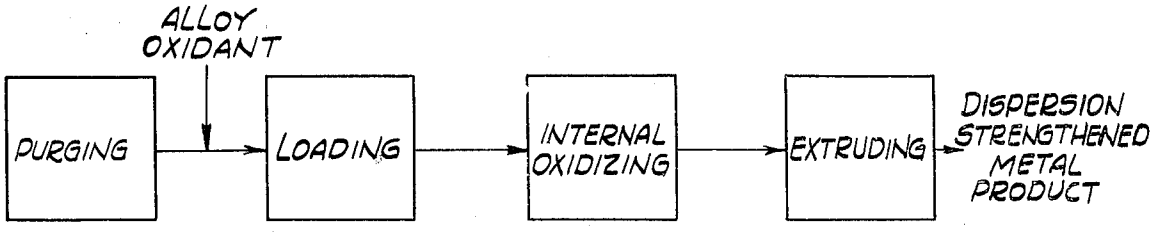


Fig. 4

DISPERSION STRENGTHENING OF METALS BY IN-CAN PROCESSING

BACKGROUND OF THE INVENTION

This is a continuation-in-part of our copending application Ser. No. 217,506 filed on Jan. 13, 1972, now U.S. Pat. No. 3,779,714 and said application is incorporated herein by reference.

Dispersion strengthening has been recognized in the past as a method for increasing strength and hardness of metals. A solid solution alloy comprising a relatively noble matrix metal having relatively low heat or free energy of oxide formation and a solute metal having relatively high negative heat or free energy of oxide formation is heated under oxidizing conditions to preferentially oxidize the solute metal. This technique is known in the art as in situ internal oxidation of the solute metal to the solute metal oxide or more simply "internal oxidation."

Dispersion-strengthened metal products, such as copper dispersion strengthened with aluminum oxide, have many commercial and industrial uses wherein high temperature strength properties and high electrical and/or thermal conductivities are desired or required in the finished product. Such commercial uses include frictional brake parts such as linings, facings, drums, and other machine parts for friction metal applications. Other commercial uses include electrical contact point resistance welding electrodes, electrodes generally, electrical switches and switch gears, transistor assemblies, wires for solderless connections, wires for electrical motors, and many other uses requiring good electrical and thermal conductivities as well as improved strength and hardness at elevated temperatures.

Several prior art processes for internal oxidation have been suggested, such as disclosed in the Schreiner patent, U.S. Pat. No. 3,488,185; the McDonald patent, U.S. Pat. No. 3,552,954; and the Grant patent, U.S. Pat. No. 3,179,515. The prior art processes, however, invariably require delicate control over partial pressure of oxygen during oxidation, or require removal of an oxidant residue which otherwise would form defects in the dispersion-strengthened metal.

Our copending application Ser. No. 217,506 provides a novel solution to these prior art problems by providing for assimilation of the oxidant residue into the dispersion-strengthened mixture wherein the oxidant residue is dispersion strengthened during thermal coalescence by a hard, refractory metal oxide provided in the power mixture to be dispersion strengthened. Hence, the oxidant residue formed during internal oxidation is not required to be removed from the dispersion-strengthened metal mixture but is dispersion strengthened and consolidated into the final metal product and thereby forms an integral part of the dispersion-strengthened metal stock.

It now has been found that a powder metal mixture of powdered alloy and oxidant may be internally oxidized and dispersion strengthened within a sealed container and subsequently extruded from the same container during an extrusion process whereby the dispersion-strengthened metal mixture remains in the sealed container and is hot-worked and coalesced during extrusion to produce a dispersion-strengthened metal article. Accordingly, a major objective of this invention is to provide for internal oxidation of a self-contained

powder metal mixture disposed within a sealed container and adapted for extrusion. The self-contained powder metal mixture eliminates the intermediate steps of removing the internally oxidized material from the container after internal oxidation, pulverizing the sintered cake, and hydrogen reducing the powder mixture if desired, which steps are generally the practice of prior art processes. Elimination of these intermediate steps essentially eliminates the problem of introducing impurities into the dispersion-strengthened metal which often occurs in various handling steps prior to coalescing and hot-working the metal mixture into dispersion-strengthened metal stock. Contamination is particularly detrimental to electrical conductivity.

Futher advantages of this invention include elimination of the prior art step of pulverizing the sintered mass produced by internal oxidation and screening.

A further advantage of this invention is the elimination of a hydrogen reduction step required in prior art processes.

Another advantage of this invention is the elimination of a double heat-up of the material wherein the powder mixture is first heated to high temperatures during internal oxidation and subsequently heated again to high temperatures prior to extruding. Both heating processes are combined in this invention without intermediate cooling which is more economical and substantially improves the quality of the final dispersion-strengthened product.

Still another advantage is increased extrusion output capacity wherein the packed density of the material in the can for extrusion is increased about 20% over typical prior art methods.

These and other advantages will become more apparent from the detailed description of the invention.

SUMMARY OF THE INVENTION

Briefly, this invention provides a self-contained powder metal mixture of alloy and oxidant enclosed within a sealed container wherein the mixture is dispersion strengthened by internal oxidation to form a hard, sintered cake therein, and thereafter hot-worked and coalesced by extrusion from the same sealed container. The powder metal mixture of powdered alloy and oxidant is intimately intermixed wherein the alloy comprises matrix metal and solute metal and the oxidant comprises heat-reducible metal oxide and hard, refractory metal oxide. Sufficient oxidant is combined with the alloy in approximately stoichiometric proportions for complete oxidation of the alloy solute metal. A residue of heat-reducible metal oxide produced during internal oxidation becomes dispersion strengthened during coalescence and extrusion whereby a dispersion-strengthened metal product is produced without removing the dispersion-strengthened metal mixture from the container. In the drawings:

FIG. 1 is a side elevation view of the container partially broken away exposing a powder metal mixture therein;

FIG. 2 is an end elevation view of the rearward portion of the container shown in FIG. 1;

FIG. 3 is a partial side elevation view of the rearward portion of the container showing the nozzle end portion pinched; and

FIG. 4 is a block diagram indicating the processing steps of this invention.

DETAILED DESCRIPTION OF THE INVENTION

Referring now to the drawings wherein like numerals identify like parts, shown is a cylindrical metal container **10** having side walls **12** of uniform thickness, a forward end wall **14**, and a rearwardly disposed end wall **16.** The forward wall **14** and rear wall **16** are secured to the side wall **12** to hermetically seal the container **10.** The rearward wall **16** is conical and protrudes outwardly from the interior of the container **10** and is provided with a centrally disposed nozzle **18** having an opening **20** therein. The nozzle **18** is adapted to receive the powder metal mixture whereby the container **10** may be loaded with the powder metal mixture prior to internal oxidation and extrusion. The nozzle opening **20** is of convenient size relative to the size of the container **10** and, for example, may have a diameter of about ½ to 1 inch. The cylindrical metal container typically has a diameter D of **8** inches and a length L of about 24 inches. The diameter D and/or cylinder length L may be relatively smaller or larger depending on the size of the extruded article, or depending on the size of the extrusion chamber of the press. For efficiency and economy, an elongated container is preferred whereby the cylinder length L is maximized relative to the cylinder diameter D to minimize extrusion costs.

The overall size of the container is usually dictated by the extrusion press available wherein the container **10** desirably has about a ½-inch clearance within the extruder feed cavity. The forward end wall **14** of the container **10** is shown as convex although a flat or concave forward end wall may be utilized. The side wall thickness of the container desirably is greater than about one sixteenth inch and preferably about one eighth inch. Larger wall thickness may be used if desired. The forward wall **14** and the rearward wall **16** have wall thicknesses similar to the side walls **12.** The container **10** is of metal and desirably metallurgically compatible with the alloy to be extruded. For example, a copper container would be desirable for a copper alloy, a nickel container would be desirable for a stainless steel or nickel alloy extrusion, a silver container would be desirable for a silver alloy extrusion, and a steel container is desirable for a steel extrusion. Preferably, the metal of the container is compatible with the alloy contained therein since the container metal itself is completely extruded with the alloy in the extrusion process.

In practice, the container **10** is purged with inert gas such as argon or nitrogen by introducing the same under pressure into the nozzle **18.** The powdered alloy and oxidant mixture is then introduced into the container **10** via nozzle **18** until the container **10** is completely filled. The alloy and oxidant mixture is compacted or settled by mechanical vibration. Thereafter the nozzle **18** is sealed such as by welding or by pinching, as indicated in FIG. **3,** but with the provision for a small leak or pin hole **22** through the rearwardmost portion of nozzle **18.** The small leak **22** provides an escape passage for residual gas remaining within the container **10** whereby pressure build-up is effectively avoided during subsequent heat-up required in internal oxidation.

After the container **10** is loaded and sealed, the loaded container **10** is subjected to high heat of at least about 1400°F for internal oxidation, and desirably heated at about 1750°F for at least about one-half hour, as more particularly set forth in our copending application Ser. No. 217,506. After the internal oxidation step, the sealed container **10** may be placed directly into a conventional extrusion press and extruded at temperatures of about 1600°to 1700°F without prior cooling. The extrusion step may immediately follow the internal oxidation step, or alternatively, the container may be cooled to room temperature and thereafter re-heated prior to extrusion. After internal oxidation, the pin hole **22** is preferably sealed prior to extrusion to prevent air from entering the container **10.**

The foregoing self-contained powder metal mixture adapted to be dispersion strengthened by internal oxidation in the same container utilized for extrusion uniquely eliminates the intermediate step of removing the internally oxidized material from the container after internal oxidation whereby considerable expediency is achieved in processing and the quality (purity) of the product is improved.

Referring now to the self-contained powder metal mixture disposed within a metal container and adapted to be dispersion strengthened and extruded from the same sealed metal container, the powder metal mixture comprises an intimate mixture of powdered alloy of matrix metal and solute metal and an oxidant of heat-reducible metal oxide and hard, refractory metal oxide.

The preferred powder alloy comprises a relatively noble matrix metal having a negative free energy of oxide formation at 25°C of up to 70 kilocalories per gram atom of oxygen, and a solute metal having a negative free energy of oxide formation exceeding that of the relatively noble matrix metal by at least about 60 kilocalories per gram atom of oxygen at 25°C. The preferred oxidant comprises an intimate mixture of heat-reducible metal oxide having a negative free energy of formation at 25°C of up to about 70 kilocalories per gram atom of oxygen, and finely divided hard, refractory metal oxide having a negative free energy of formation exceeding the negative free energy of formation of the heat-reducible metal oxide by at least about 60 kilocalories per gram atom of oxygen at 25°C. The heat-reducible metal oxide is present in the oxidant in an amount sufficient for complete oxidation of the solute metal in the alloy. The hard, refractory oxide in the oxidant is present in substantially the same equivalent elemental proportion as the solute metal in the alloy, and both are of a particle size suitable for dispersion strengthening of the oxidant residue resulting from the internal oxidation, as set forth in our copening application Ser. No. 217,506. After internal oxidation, the oxidant residue comprises particles of in situ residue of heat-reducible metal oxide and particles of hard, refractory metal oxide uniformly distributed therein and the residue of heat-reducible metal oxide is intimately dispersed within the alloy powder. The dispersion-strengthened metal mixture is eventually coalesced and consolidated by hot-working to form a solid metal workpiece whereby the residue of heat-reducible metal is dispersion strengthened by the hard, refractory metal oxide and forms an integral parts of the dispersion-strengthened resulting workpiece.

To achieve the proper proportion of oxidant, about 0.1 to about 10 parts by weight of oxidant are employed per 100 parts of alloy to be internally oxidized. The exact proportions depend on the solute metal to be oxidized, the concentration of solute metal in the alloy, and the oxygen content of the oxidant. The heat-

reducible metal oxide is present in substantially stoichiometric proportions for internally oxidizing all of the solute metal in the alloy. At least about 0.1 weight parts of oxidant are combined per 100 weight parts of powder alloy, desirably between about 0.1 to 20 weight parts of oxidant, and preferably about 0.1 to 10 weight parts of oxidant are combined with about 100 weight parts of powder alloy. Preferably, the proportion of the heat-reducible metal oxide and the hard, refractory metal oxide is predetermined so that the composition of the oxidant residue and internally oxidized alloy are substantially identical in the dispersion-strengthened metal after the internal oxidation step is completed. The amounts of such oxidants to be added may be determined by the stoichiometric amount of oxygen required to oxidize the solute metal completely, but preferably excessive amounts of oxidant are not used so as to avoid leaving more than about 0.1% of reducible oxygen in the internally oxidized metal mixture.

Thus, the internally oxidized metal mixture would not lose greater than about 0.1% by weight after subjecting the same to a hydrogen reduction test. The hydrogen reduction test may be applied to a representative sample of internally oxidized metal mixture by removing the same from the sealed container after the internal oxidation step is completed. The sintered mass is then pulverized, screened, weighed, and then subjected to a hydrogen atmosphere at a temperature of 1600°F for one half hour. The measured weight loss preferably is no greater than about 0.1% based on the total sample tested.

In practicing this invention, the powdered alloy comprising a relatively noble matrix metal and a solute metal is produced by conventional techniques such as melting the metal under inert or reducing conditions and thereafter comminuting the alloy by atomization or other conventional size-reduction techniques such as grinding or ball milling to form a particulate alloy having an average particle size of less than about 300 microns.

The noble matrix metal in the alloy and the in situ, heat-reduced metal in the oxidant residue are defined broadly as those metals having a melting point of at least about 200°C and whose oxides have a negative free energy of formation at 25°C of from 0 to 70 kilocalories per gram atom of oxygen. Suitable alloy matrix metals and corresponding heat-reducible metal oxides for practicing the present invention include the following: iron (FeO, Fe_2O_3); cobalt (CoO); nickel (NiO); copper (Cu_2O, CuO); cadmium (CdO); thallium (Il_2O); germanium (GeO_2); tin (SnO, SnO_2); lead (PbO); antimony (Sb_2O_3); bismuth (Bi_2O_3); molybdenum (MoO_2,MoO_3); tungsten (WO_2, WO_3); rhenium (ReO_3); indium (In_2O_3); palladium (PdO); osmium (OsO_4); platinum (PtO); and rhodium (Rh_2O_3) as more particularly set forth in our copending application Ser. No. 217,506.

In any particular combination of matrix metal and solute metal in the alloy to be dispersion strengthened by internal oxidation, the matrix metal must be relatively noble with respect to the solute metal so that the solute metal will be preferentially oxidized. This is achieved by selecting the solute metal such that its negative free energy of oxide formation at 25°C is at least 60 kilocalories per gram atom of oxygen greater than the negative free energy of formation of the oxide of the matrix metal at 25°C. Such solute metals have a negative free energy of oxide formation per gram atom of oxygen of over 80 kilocalories and generally over 120 kilocalories. Suitable alloy solute metals and corresponding hard, refractory metal oxides include: silicon (SiO_2); titanium (TiO_2); zirconium (ZrO_2); aluminum (Al_2O_3); beryllium (BeO); thorium ThO_2); chromium (Cr_2O_3); magnesium (MgO); manganese (MnO); niobium (Nb_2O_5); tantalum (Ta_2O_5); and vanadium (VO), as more particularly set forth in our said copending application Ser. No. 217,506.

The metal moiety of the heat-reducible metal oxide in the oxidant preferably is the same metal as the matrix metal present in the alloy to be internally oxidized, although the heat-reducible metal oxide moiety can be different. For instance, alloy matrix metal/oxidant heat-reducible metal oxide combinations include:

ALLOY MATRIX METAL	OXIDANT HEAT-REDUCIBLE METAL OXIDE
copper	cobalt oxide, nickel oxide, copper oxide
nickel	cobalt oxide, nickel oxide, copper oxide
cobalt	cobalt oxide, nickel oxide, copper oxide

Similarly, the hard, refractory metal oxide in the oxidant preferably is the same as the solute metal oxide formed in the alloy during internal oxidation of the alloy, although the refractory metal oxide in the oxidant can be different from the solute metal oxide in the internally oxidized alloy. For example, solute metal oxide/oxidant hard, refractory metal oxide combinations include:

ALLOY SOLUTE METAL OXIDE	OXIDANT HARD, REFRACTORY METAL OXIDE
Al_2O_3	Al_2O_3, BeO, ZrO_2, ThO_2
BeO	Al_2O_3, BeO, ZrO_2, ThO_2
ZrO_2	Al_2O_3, BeO, ZrO_2, ThO_2
ThO_2	Al_2O_3, BeO, ZrO_2, ThO_2

In accordance with this invention, the stoichiometrically porportioned alloy and oxidant are contained within a sealed container preparatory to the internal oxidation step. The container must be adaptable for both high temperature internal oxidation and subsequent high temperature extrusion.

The following illustrative examples are included to further explain the invention and are not intended to be limiting. All parts are by weight and all temperatures are in degrees Fahrenheit, unless otherwise stated.

EXAMPLE 1

Part A — Preparation of the Alloy Powder

Electrolytic tough-pitch grade copper rods were melted in an inert refractory crucible in an induction heating furnace under reducing conditions at a temperature of about 2300°F. Metallic aluminum shavings were introduced into the molten copper in the proportion of 0.33% by weight of the resulting molten metallic mass.

The molten solution of aluminum in copper was then super-heated to 2400°F, atomized through an atomizing aperture in a jet of nitrogen to yield an atomized copper-aluminum alloy powder which substantially all

passed a 100-mesh U.S. Sieve indicating that the average particle size was less than about 140 microns.

Part B — Preparation of the Oxidant

One hundred parts of commercially available cuprous oxide (Cu_2O) with an average particle size of about 1 to 2 microns were mixed with 4.1 parts of $Al(NO_3)_3 \cdot 9H_2O$ dissolved in water to form a slurry of cuprous oxide in aluminum nitrate solution. The solution of aluminum nitrate was slurried with cuprous oxide particles, and stirring was continued with mild heating at 200°F until the water had evaporated and the mixture had become almost dry. The mixture was then heated at a temperature of about 500°F for one-half hour to decompose the aluminum nitrate into aluminum oxide. The resulting agglomerate was then ground to form fine oxidant powder which passed a 325-mesh sieve. The resulting oxidant powder comprised 77.43% Cu_2O, and 0.56% Al_2O_3 by weight.

Part C — Preparation of Powder Metal Mixture

The alloy powder of Part A was thoroughly mixed with the oxidant powder to Part B in the proportion of 2.12 parts of oxidant to 100 parts of alloy powder to provide a powder mixture of alloy and oxidant. Mixing was achieved in a ball-mill.

Part D — Internal Oxidation

The alloy powder-oxidant mixture of Part C was then charged into an internal oxidation vessel. The oxidation vessel was a cylindrical copper container having an overall length of 24 inches, a diameter of 8 inches, and a wall thickness of one-eighth inch. Approximately 234 pounds of the foregoing alloy-oxidant powder mixture were charged into the container, purged with argon gas, and thereafter sealed leaving a pin hole opening for pressure release.

The alloy oxidant powder mixture was then brought to a temperature of about 1750°F and maintained at this temperature for about 30 minutes to effect internal oxidation of the alloy powder.

At the end of the 30-minute internal oxidation period, substantially all of the aluminum in the alloy powder was oxidized to Al_2O_3 and substantially all of the cuprous oxide in the oxidant had been reduced to metallic copper. The particles of internally oxidized alloy comprises 99.37% by weight of copper plus negligible amounts of impurities and 0.63% by weight of Al_2O_3 and the oxidant residue comprised 99.37% copper particles and 0.63% Al_2O_3 particles. The overall internally oxidized metal powder composition comprised 98.21% internally oxidized alloy powder and 1.79% oxidant residue.

Part E — Thermal Coalescence and Extrusion

The container of internally oxidized metal mixture was then placed in a ram-type extrusion press and was extruded to form extrudate in the shape of cylindrical bar stock having a diameter of about 1.125 inches. This corresponds to an extrusion ratio of about 50:1 (i.e., the ratio of the cross-sectional area of the can to the cross-sectional area of the extrudate).

The bar stock was about 99.37% copper having dispersed throughout 0.63% (or about 1.5% by volume) of Al_2O_3 particles. The bar stock had a density of about 99.3% of the theoretical density, an electrical conductivity of 88% IACS, a tensile strength of about 72,000 psi, an elongation of 19% using ASTM Test E-8 (for a test specimen 0.16 inch in diameter and 0.65 inch gage length) and a Rockwell hardness of about 75 units on the B scale. All property measurements were made at room temperature.

A sample of the bar stock was reduced by 50% in cross-sectional area by cold swaging whereby the tensile strength became 80,000 psi, the elongation 13% Rockwell B hardness 84 units, and conductivity 86% IACS.

EXAMPLE 2

The procedures of Example 1 were repeated except that the 8-inch diameter copper can was replaced by a 1.25-inch diameter copper can. The extrusion was carried out in an extrusion chamber of 1.38-inch diameter at an extrusion ratio of 30:1 yielding a 0.250-inch diameter rod. Such rod had an electrical conductivity of 86.7% IACS, a tensile strength of about 73,000 psi, and an elongation of 19.8% in a gage length of 0.650 inch.

EXAMPLE 3

The material of Part D of Example 1 was fed into a thin-walled copper can of 1.25-inch diameter and extruded in an extrusion chamber of 1.38-inch diameter at an extrusion ratio of 45:1 to yield a rod of 0.206-inch diameter. This rod had an electrical conductivity of 89% IACS. The rod was swaged and drawn to a 0.010-inch diameter wire and heat treated at 500°C for one-half hour in helium and produced an ultimate tensile strength of 84,000 psi, a yield strength of 71,200 psi, and an elongation of about 5% in 10 inches

EXAMPLE 4

Dispersion-strengthened metal bar stock is formed by the above-described method by oxidizing 100 parts of a powdered alloy of 98.86% nickel and 1.14% aluminum with 4.76 parts of pulverulent oxidant comprising 4.68 parts of nickel oxide and 0.08 parts of aluminum oxide. In this example, a nickel metal container is utilized for internal oxidation and subsequent extrusion. The resulting dispersion-strengthened bar stock has increased tensile strength and hardness at elevated temperatures relative to bar stock of a similar nickel-aluminum alloy which has not been internally oxidized.

EXAMPLE 5

Dispersion-strengthened metal bar stock is formed as indicated in Example 1 by oxidizing 100 parts of a powdered alloy of 98.72% iron and 1.28% aluminum with 3.865 parts of pulverulent oxidant comprising 3.800 parts of iron oxide and 0.065 parts of aluminum oxide, but with the provision that a steel container is utilized for internal oxidation and extrusion. The resulting dispersion-strengthened bar stock has increased tensile strength and hardness at elevated temperatures or after annealing as compared relative to bar stock of a similar iron-aluminum alloy which had not been internally oxidized.

We claim:

1. A self-contained powder metal mixture adapted to be dispersion strengthened by internal oxidation and extruded under heat and pressure, the self-contained powder metal mixture comprising:

an intimate mixture of 100 weight parts of a powdered alloy and at least about 0.1 weight parts of an oxidant, said powdered alloy having an average

particle size of less than 300 microns and consisting of a relatively noble matrix metal having a negative free energy of oxide formation at 25°C of up to 70 kilocalories per gram atom of oxygen, and a solute metal having a negative free energy of oxide formation exceeding the negative free energy of oxide formation of said matrix metal by at least about 60 kilocalories per gram atom of oxygen at 25°C, said oxidant consisting of an intimate mixture of heat-reducible metal oxide having a negative free energy of formation at 25°C of up to 70 kilocalories per gram atom of oxygen, and finely divided hard, refractory metal oxide having a negative free energy of formation exceeding the negative free energy of formation of said heat-reducible metal oxide by at least about 60 kilocalories per gram atom of oxygen at 25°C;

said heat-reducible metal oxide being present in substantially stoichiometric proportion for complete oxidation of said solute metal in said alloy whereby a residue of heat-reducible metal oxide remains in said powder metal mixture after internal oxidation adapted to be dispersion strengthened during coalescence by said hard, refractory metal oxide; and

a metal container comprising side wall portions, a forward wall, and a rearward wall, said walls defining a cavity within said metal container for containing said powder metal mixture of alloy and oxidant free from contamination during internal oxidation and extrusion whereby said powder metal mixture is adapted to be dispersion strengthened within said metal container and directly extruded from said metal container.

2. The self-contained powder metal mixture of claim 1 wherein said oxidant exceeds the stoichiometric proportion for complete oxidation of said solute metal to leave reducible oxides of less than 0.1% by weight of oxygen based on the dispersion-strengthened metal mixture.

3. The self-contained powder metal mixture of claim 1 wherein said walls have a wall thickness greater than one-sixteenth inch.

4. The self-contained powder metal mixture of claim 1 wherein said container is copper and said noble matrix metal of the alloy is copper.

5. The self-contained powder metal mixture of claim 1 wherein said container is nickel and said noble matrix metal of the alloy is nickel.

6. The self-contained powder metal mixture of claim 1 wherein said container is steel and said noble matrix metal of the alloy is iron.

7. A process for dispersion strengthening a self-contained powder metal mixture and extruding metal stock from the same container, comprising:

providing a metal container having a cavity therein for receiving a powder metal mixture adapted to be dispersion strengthened, said metal container for containing the metal mixture during internal oxidation and extrusion;

filling said metal container with powder metal mixture comprising about 100 weight parts of a powdered alloy and at least about 0.1 weight parts of an oxidant, said powdered alloy having an average particle size of less than 300 microns and consisting of a relatively noble matrix metal having a negative free energy of oxide formation at 25°C of up to 70 kilocalories per gram atom of oxygen, and a solute metal having a negative free energy of oxide formation exceeding the negative free energy of oxide formation of said matrix metal by at least about 60 kilocalories per gram atom of oxygen at 25°C, said oxidant consisting of an intimate mixture of heat-reducible metal oxide having a negative free energy of formation at 25°C of up to 70 kilocalories per gram atom of oxygen, and finely divided hard, refractory metal oxide having a negative free energy of formation exceeding the negative free energy of formation of said heat-reducible metal oxide by at least about 60 kilocalories per gram atom of oxygen at 25° C, said heat-reducible metal oxide being present in substantially stoichiometric proportion for complete oxidation of all said solute metal in the said alloy;

heating said container at a temperature of at least about 1400°F to internally oxidize said solute metal of the alloy and form a residue of heat-reducible metal oxide whereby said alloy is dispersion strengthened; and

extruding said dispersion-strengthened alloy from said container under heat and pressure to thermally coalesce said internally oxidize metal mixture and oxidant residue into dispersion-strengthened metal stock whereby said hard, refractory metal oxide dispersion strengthens said residue of heat-reducible metal oxide.

8. The process of claim 7 wherein oxidant exceeds the stoichiometric proportion required to completely oxidize said solute metal in the alloy whereby less than 0.1% by weight of oxygen of reducible oxides remain after the step of heating to internally oxidize said solute metal of the alloy.

* * * * *

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

PATENT NO. : 3,884,676

DATED : May 20, 1975

INVENTOR(S) : ANIL V. NADKARNI and ERHARD KLAR

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 4, line 49, "copening" should read ---copending---

Column 5, line 52, "Il_2O" should read ---Tl_2O---

Column 6, line 45, "porportion" should read ---proportion---

Column 7, line 19, "Cu_2O, and" should read ---Cu_2O,
22.01% CuO, and---

Signed and Sealed this

thirtieth **Day of** *December 1975*

[SEAL]

Attest:

RUTH C. MASON
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents and Trademarks