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3,070,440

PRODUCTION OF DISPERSION HARDENED METALS

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6 Claims. (Cl. 75—206)

This invention relates to a method of processing dispersion hardened metals and, in particular, to a method of optimizing the strength of matrix metals having distributed therethrough a uniform dispersion of a secondary phase characterized by a crystallographic transformation temperature.

In recent years, new metal compositions have been proposed comprising a metal matrix having dispersed therethrough a finely divided secondary phase, for example, a fine dispersion of refractory oxide particles. Of particular interest is an aluminum material referred to as SAP (sintered aluminum powder) which in the wrought state exhibits greatly enhanced strength properties due to the presence of a uniform dispersion throughout the aluminum matrix of Al_2O_3 particles. Methods have been proposed for applying the foregoing phenomenon to other metals by utilizing the technique of powder metallurgy.

The most direct method, employed with varying degrees of success, involved the simple mechanical mixing of desired inert hard phase (e.g. refractory oxide) and metal powders by either dry or wet ball-milling, followed by compacting and extruding as in the case of producing copper alloys dispersion hardened with Al_2O_3. While this method resulted in some measure of success, certain anomalies in behavior of the product occurred accompanied by a falling off in the improved properties. In another method, attempts were made to utilize the phenomenon of internal oxidation to produce the hard phase, this being done by producing an alloy from a matrix metal, such as copper, containing an easily oxidizable solute metal, such as silicon or aluminum, powdering the alloy followed by the selective oxidation of the contained solute metal into a dispersed hard phase and then consolidating the thus treated powder into a wrought metal product. While this technique generally resulted in a further improvement of physical properties, certain anomalies similarly occurred with respect to the behavior of the metal product. Similar anomalies were noticed on dispersion strengthened copper in which the oxide phase was added as a decomposable compound, e.g. aluminum nitrate, which was thereafter decomposed to leave a fine dispersion of Al_2O_3. Another method comprised forming an alloy of say Cu-Al, crushing it into a powder, completely oxidizing the powder, then selectively reducing the copper oxide to give a product in which solute oxide is dispersed throughout the copper. The work indicated that particle size control of the disperse phase in the submicron range was important for obtaining optimum strength properties. However, in the foregoing methods, it was noticed that while the requisite particle size could be obtained, in some instances a coarsening in particle size of the disperse phase would occur during processing whereby the strength properties expected were not always obtained.

We have now discovered that the foregoing disadvantages can be overcome by controlling the processing steps so as to avoid coarsening of the dispersed phase.

It is the object of this invention to provide a method of producing a wrought dispersion hardened metal product in which coarsening of the disperse phase has been greatly inhibited.

Another object is to provide a method for consistently producing a dispersion hardened material having optimum

strength properties while inhibiting coarsening of the disperse phase.

As a further object, the invention provides an improved, wrought, dispersion strengthened metal product characterized by optimum strength properties.

These and other objects will more clearly appear from the disclosure which follows.

We have now discovered that the anomalies observed in the treatment of disperse strengthened metals appear related to whether or not the disperse phase has a crystallographic transformation temperature. In addition, we have discovered that base metal oxides such as NiO, CuO, Cu_2O, FeO, Fe_2O_3, CoO, and the like, as impurities appear to have some effect. We have found, for example, that where alumina is the dispersion hardener, the disperse phase may exits in one or more crystalline states depending on either its initial existence or its existence after the processing of the metal. If in the case of dispersion hardened copper, the alumina is originally present in the gamma form (low temperature form) and during processing it converts substantially to the high temperature alpha form, the physical properties obtained tend not to be as high as those obtained where the crystallographic change has not taken place. We have observed that when the transformation occurs, it is usually accompanied by a coarsening of the phase structure followed by a dropping off in strength properties. We have also observed that the presence of small amounts of base metal oxide tends to activate the coarsening of the phase structure.

In the case of copper dispersion hardened with gamma alumina, and other metal-metal oxide systems, we found that where the fabricating temperatures, e.g. reduction temperature, sintering temperature, extrusion or working temperature, etc., are within or above the transformation temperature range of the disperse phase, there was a tendency for the disperse phase to coarsen by agglomeration due to crystallographic conversion of the phase from gamma to alpha alumina. It was observed that this tendency was particularly noticeable at higher concentrations of alumina and for smaller particle sizes as affected by packing or pressing.

Product processes geared towards making ultra fine particles of alumina (e.g. 50 to 1000 angstroms in average diameter) usually yield the low temperature gamma crystal structure, which over the temperature range of about 800° to 1000° C. transforms to the more stable alpha form. Generally speaking, the occurrence of agglomeration appeared to be connected with phase transformation whereby optimum strength properties were not always achieved. This much was observed: the agglomeration tended to become more noticeable as the concentration of the disperse phase exceeded about 5 v/o and the particle size fell to below .05 micron, for example in the neighborhood of about .03 micron and smaller. The greater the surface area of the disperse phase, the greater was the tendency to agglomerate during transformation, especially where other oxides were present as impurities to aid and abet in the transformation.

In one instance where the dispersion hardened alloy, e.g. Cu-Al_2O_3, was produced by internal oxidation, the strongest compositions were those which contained 80 to 200 angstrom size particles (average diameter) of gamma alumina. However, when the particles were transformed by heating the alloy above the transformation temperature to form alpha alumina particles of coarser size, e.g. up to 1000 angstroms in diameter and higher, the strength properties were not as good.

In overcoming the foregoing difficulty, we provide a method of producing dispersion strengthened material characterized by improved strength and resistance to creep at elevated temperatures by providing a matrix

metal having associated uniformly therewith a finely divided disperse phase of a material of particular crystallographic structure and working said matrix metal to a wrought shape under conditions which preserve the original structure of the disperse phase or which inhibit the transformation of said disperse phase to another structure. In the case of copper or copper powder having uniformly commingled therewith or uniformly dispersed therethrough finely divided gamma alumina, we can inhibit the conversion of gamma alumina to alpha by using fabrication temperatures below that at which gamma transforms to alpha. Actually, an ideal structure would be one containing an ultrafine dispersion of alpha alumina, if available, since this structure would have the inherent high temperature stability necessary. Thus, by starting with alpha alumina, its structure is preserved over a very wide hot working range. However, where ultra fine alpha alumina particles are not available, then we prefer to control the working temperature to inhibit crystallographic transformation and preserve the original character of the particles.

Putting it broadly, therefore, the method for carrying out the invention comprises either starting with the crystallographic structure of the disperse phase which is stable at elevated temperatures or, if the stable phase is not initially available, then controlling the fabrication temperatures to inhibit transformation of the disperse phase into coarser particles and thereby preserve the original character of the particles and assure optimum strength properties in the final wrought product. Where a high concentration of the disperse phase is present at extremely small sub-micron sizes, we prefer that the temperature of fabrication be below the transformation temperature range.

As illustrative of the invention, the following examples are given:

EXAMPLE I

Copper dispersion hardened with gamma alumina was prepared by using the mechanical mixing technique of first uniformly incorporating the disperse phase within a batch of copper powder. An amount of alumina powder, e.g. 0.033 micron powder sufficient to correspond to about 10 volume percent of the total composition, was dry mixed with a batch of copper powder, e.g. one micron powder, preferably by using a Waring Blendor operating at about 15,000 r.p.m. The powders were mixed for about two minutes after which they were poured out on paper and spatulated by hand, the whole procedure being repeated five times to give a total mixing time in the Blendor of about 10 minutes. Two separate batches of 250 grams each were prepared and later combined and intimately mixed to produce 500 grams of material. While other mixing techniques could have been used, such as dry ball milling, wet ball milling, with or without a ball charge, we found the foregoing technique to give us the type of consistent results required for our purposes.

In order to eliminate any copper oxide that was formed during mixing, the powders were reduced in dry hydrogen for a minimum time of 6 hours at a temperature of 400° C., it being observed that after three hours at that temperature no further moisture was given off.

In producing a compact for working into a wrought shape, a given weight of the powder mix was introduced into a rubber tube supported within a two inch diameter perforated steel canister. One end of the tube was rubber stoppered and after the powder was introduced into the rubber tube a second rubber stopper, which contained a hypodermic needle pushed through it, was inserted. Vacuum was applied to the outside end of the needle simultaneously while pushing this stopper into position. After the assembly was evacuated for five minutes, the needle was then removed.

The canister was then subjected to hydrostatic pressure at 30,000 p.s.i. to yield a compact of about 1.4 inches in diameter and 2.5 inches in length. Compacts of copper produced in this manner could be handled easily and could be squared off and turned down in diameter by regular machining to fit the extrusion liner in the extrusion press.

The compact was then sintered in dry hydrogen, first at a temperature of about 500° C. for one hour to enable adsorbed gases and entrapped air to be removed, and then at a higher temperature of 900° C. near the lower end of the transformation temperature range for Al_2O_3 for two hours to promote sintering of the metal particles followed by about 2 to 4% shrinkage in the compact. Generally the temperature is between 800 to 900° C.

The compact was then extruded at 760° C. from a 1.375 inch liner through a 0.3 inch diameter die, giving an extrusion ratio of about 21 to 1. The alloy exhibited a good surface after extrusion with only a thin oxygen-containing layer on the outside of the bar, this layer being easily removed by machining.

For the purposes of illustrating this invention, this alloy and alloys of other compositions produced similarly were subjected to tensile and stress-rupture testing. The test specimens were machined from the extruded rods having a gauge section of about 0.160 inch diameter by 1.0 inch long.

A spectrogoniometer with copper radiation was used to obtain the diffraction patterns of all oxides in the "as received" condition. After the extrusion step, the oxides were extracted from a small sample of each alloy by dissolving the matrix metal in a 25% nitric acid solution and collecting the oxide residues in a dialysis bag. After repeated washings in distilled water, the residues were dried and prepared for X-ray analysis by depositing the oxide powders on a glass slide in a parlodion film and the crystal structure after extrusion being determined.

Alloys which were produced and tested in accordance with the foregoing are given in Table I as follows:

Table I

Alloy No.	Size of Cu, Microns	Size of Al_2O_3, Microns	v/o Al_2O_3	Structure of Al_2O_3 as Received
1	1	0.033	10	gamma.
2	1	0.018	10	Do.
3	1	0.018	2.5	Do.
4	1	0.018	5	Do.
5	1	0.018	7.5	Do.
6	5	0.033	10	Do.

Room temperature tensile and 100-hour rupture properties of the foregoing alloys are given in Table II below:

Table II

Alloy No.	Y.S., p.s.i., 0.2% Offset	T.S., p.s.i.	Stress for 100 Hour Rupture, 450° C.	Al_2O_3 Phase in Product
1	60,500	77,400	24,800	10% alpha, 90% gamma.
2	25,800	33,900	3,600	All alpha.
3	27,400	35,000	8,000	All gamma.
4	48,400	58,800	18,800	Do.
5	69,900	88,500	23,500	Do.
6	59,200	66,300	20,000	80% gamma, 20% alpha.

It will be noted that Alloy No. 2, which contained 10 v/o of 0.018 micron Al_2O_3, was completely converted to alpha Al_2O_3 while Alloy No. 1 which contained 10 v/o of 0.033 micron powder was only partially converted to 10% alpha and exhibited markedly superior strength properties as opposed to Alloy No. 2 at both room and elevated temperatures. That the difference in the initial particle size could not have had a major effect on the falling off in properties is confirmed by the fact that alloy No. 3 which contained only 2.5 v/o of gamma alumina of 0.018 micron size exhibited over double the

stress of 8,000 p.s.i. for 100-hour rupture life at 450° C. as compared to the low value of 3,600 p.s.i. obtained for the all alpha converted Alloy No. 2. This is further confirmed by Nos. 4 and 5 which contained 5 v/o and 7.5 v/o, respectively, Al_2O_3 of 0.018 micron size and yet exhibited much higher room temperature strength (over double the value) than Alloy No. 2 as well as over five to six times the 100 hour rupture stress. Apparently, as the concentration of the Al_2O_3 reaches 10 v/o for finer sizes, there is an increased tendency for gamma alumina to convert to alpha at the sintering temperature of 900° C. because of the clustering tendency of the oxide which enables the transformation to continue to completion once it starts. Examination of Alloy No. 2 under the microscope revealed that the alpha alumina which formed was extremely coarse as compared to Alloy No. 1 in which hardly any agglomeration was noticeable, despite the slight conversion to alpha which occurred. In other words, some alpha may be tolerated without adversely affecting to any great extent the physical properties.

This is shown by referring to Alloy No. 6 which is the same as No. 1 except that the starting copper powder in No. 6 was 5 microns in size as against 1 micron used in No. 1. Here, 20% alpha was formed by conversion at the sintering temperature of 900° C. yet the physical properties are substantially as favorable as those obtained for No. 1 while markedly superior to those obtained for the more inferior Alloy No. 2. One speculative explanation as to why more conversion occurred in No. 6 as compared to No. 1 is that, since the particle size of copper used in the mix for forming Alloy No. 6 was five times greater than that of the copper used in forming No. 1, thereby yielding a surface area of copper in the mix of No. 6 about one-twenty fifth that of No. 1 (ratios of diameter squared), the particles of Al_2O_3 were more closely packed so that conversion could more easily proceed through particle contact once transformation is initiated. Even then the 20% conversion was not sufficient to have a marked adverse effect on the physical properties.

Our observations have indicated that a phase conversion of more than about 25% will have an adverse affect on the physical properties.

It is to be understood that it is not the presence of alpha that is to be avoided but rather the manner in which it forms in the alloy by conversion. So long as it doesn't agglomerate into coarse particles, it can have a beneficial effect on the alloy. For example, a copper-alumina Alloy No. 7 produced from a mixture of one micron copper powder and 10 v/o of 0.3 micron alpha alumina exhibited a 100-hour rupture stress at 450° C. of about 12,000 p.s.i. as compared to the much inferior value of 3,600 p.s.i. for Alloy No. 2.

Another alloy No. 8 in which 10 v/o alpha alumina of very fine particle size was employed exhibited a 100 hour rupture stress value of 15,000 p.s.i., more than four times that of Alloy No. 2. The alpha alumina in Alloy No. 8 was obtained by mixing sufficient aluminum nitrate with specified amount of one micron copper powder to be equivalent to 10 volume percent of Al_2O_3. This was achieved by dissolving the nitrate in a minimum amount of methanol necessary to completely wet the copper powder. After mixing by spatulation, the charge was held under vacuum at 65° C. for 24 hours to reduce the methanol and the bulk of water of crystallization. This enabled the heating of the mixture to the decomposition temperature of the nitrate without melting it. The mixture was canned, consolidated and heated for one hour in vacuum at 450° C. to decompose the nitrate. This was followed by sintering in hydrogen at 800° C. for four hours, machined and then extruded to the desired dimension. The nitrate decomposed to finely dispersed alpha alumina of the order of about 320 angstroms in diameter and it made no difference at what temperature the compact was thereafter hot worked since this form of alumina was stable up to very high temperatures, even up to temperatures higher than the melting points of the various matrix metals.

In the production of dispersion strengthened iron, iron powder of about 3 microns average particle size was dry mixed with 8 v/o of gamma alumina having an average particle size of about 0.027 micron. Lots of 500 grams each were prepared in a Waring Blendor at a speed of about 15,000 revolutions per minute. The mixing was carried out for about 5 minutes followed by alternate mixing by spatulation for a few minutes, the procedure including the Blendor and subsequent spatulation being repeated about 4 times.

The blended powder batches were thereafter subjected to a reducing treatment in dry hydrogen for a minimum of five hours at a temperature of about 800° F. to clean particle surfaces as much as possible for subsequent consolidation of the mixture into wrought shapes. Each batch of the mixed powders was introduced into a rubber tube supported within a perforated steel canister about two inches in diameter, one end of the rubber tube being rubber stoppered at the start. After the powder was introduced, a second rubber stopper having in communication therewith a hypodermic needle was inserted, a vacuum connection being made through the needle to remove the air from within powder mass. After completion of evacuation, the needle was removed and the canister assembly subjected to hydrostatic pressure at about 30,000 p.s.i. to yield compacts about 1.4 inches in diameter and 3 inches long.

The compacts produced as aforementioned were then subjected to sintering in dry hydrogen for a minimum of 10 hours at 830° C. After that they were each canned by insertion in a mild steel can and welded vacuum tight followed by extrusion at an elevated temperature. The extrusion ratio was about 16 to 1.

One of the compacts was extruded at about 840° C. and the other at about 1050° C. Yield strength was obtained for each and 100-hour rupture stress determined at 650° C. as follows:

Table III

Alloy No.	Extrusion Temp., ° C.	Yield Strength, p.s.i., 0.2% Offset	100 Hour Rupture Stress at 650° C., p.s.i.	Percent Elong., 650° C.
9	840	21,300	18,000	2
9A	1,050	10,800	6,500	3

Analysis of Alloy No. 9 showed the alumina to be substantially all gamma while Alloy No. 9A revealed coarse agglomerates corresponding to the spinel structure $FeAl_2O_4$, this structure probably resulting from the presence of small amounts of iron oxide. Alloy No. 9A with the coarse agglomerates resulting from transformation at 1050° C. was noticeably inferior to Alloy No. 9 produced in accordance with the process of the invention, the yield strength of No. 9 being almost twice and the 100-hour rupture stress almost three times that of No. 9A produced outside the invention.

In processing dispersion hardened nickel from nickel powder and gamma alumina, it was not possible to obtain the desired optimum properties. Using the same preparation technique employed in preparing the copper alloys, a compact was produced from a powder mixture containing 5 micron nickel powder and 9 v/o of .018 micron alumina powder. However, because nickel has a much higher melting point than copper, much higher processing temperatures were required, e.g. in excess of 1000° C. whereby all of the gamma alumina converted to a coarse agglomerated alpha during hot working. To avoid formation of the coarse agglomerates, it would be necessary to start with alumina in the stable alpha state.

While the invention has been described with respect to

the production of dispersion strengthened metal products from metal and oxide powder mixtures, we find that our inventive concept is also applicable to the production of such products from internally oxidized powders. In producing internally oxidized copper power containing about 3.5 v/o Al_2O_3, an alloy copper powder of minus 44 microns containing the desired amount of aluminum is prepared and surface oxidized to form a coating of Cu_2O by heating a given amount of powder in a measured amount of oxygen at about 450° C. The oxygen from the surface oxide is then diffused into the sample by heating at the desired temperature, e.g. 650° C. to 850° C. under substantially inert conditions. This method was found adequate for obtaining up to about 3 to 3.5% by volume of solute oxide within the matrix metal.

As an example of one method employed in effecting the internal oxidation of the alloy powder, the surface oxidized powder, for example 450 grams, is sealed in a 1.5 inch diameter tube by flattening of the ends. The tube is placed in a large muffle furnace held at the desired temperature, e.g. 650° C. or 750° C. or 850° C. for a time, determined by pilot test, sufficient to obtain a uniform dispersion of solute metal oxide in each of the matrix metal particles.

The internally oxidized matrix metal powder is thereafter hydrogen reduced at an elevated temperature, e.g. 450° C. for one hour, to clean the surface of each particle and then packed by vibration in a copper container of about 1.4 inch I.D. by 4.5 inch long to achieve a pack density of about 50%. The container is evacuated and sealed and made ready for direct extrusion. The extrusion is carried out at a temperature of about 760° C.

An extruded alloy containing about 3.5 v/o gamma alumina of very fine particle size exhibited improved rupture life properties at 450° C. However, when the alloy after extrusion was heated to about 1050° C., whereby about half of the alumina was converted to an agglomerated alpha phase of increased particle size, a lower rupture stress value resulted.

Besides alumina, other dispersion hardeners are transformable crystallographically to other structures. We have found that silica, when used in the amorphous form, transforms to alpha cristobalite. While silica is not as good in its effect as a hardener compared to alumina, nevertheless it does have a beneficial strengthening effect, which effect can be optimized by preserving as far as possible the original character of the oxide during processing. Silica in an alloy produced from one micron copper powder and 10 v/o of amorphous SiO_2 under the same conditions used in producing alloys Nos. 1 to 6 was all converted to alpha cristobalite. The alloy exhibited a 100-hour rupture stress at 450° C. of about 6,600 p.s.i. Another composition containing 7.5 v/o SiO_2 which converted to alpha tridymite exhibited a higher rupture stress of about 10,200 p.s.i., thus showing the different results which are obtainable depending upon the type of phase change. For optimum dispersion strengthening, we prefer to start with silica initially in the form of alpha cristobalite For example, a copper alloy powder containing 1.59% Si and which was thereafter internally oxidized at 750° C. to produce a dispersion of cristobalite exhibited at 100-hour rupture life stress of 15,000 p.s.i. at 450° C. after extrusion.

Examples of other hardeners which present transformation problems during processing are TiO_2 which as the anatase transforms to rutile. To avoid transformation to agglomerated rutile during processing, the temperatures employed during fabrication should not be allowed to exceed the transformation temperature.

Some refractory oxides, e.g. ThO_2 (face centered cubic), do not present transformation problems as they are stable over a wide range of temperatures. Zirconia, which may appear in the monoclinic form, also crystallizes at relatively high temperatures to the cubic form. However, it is possible to stabilize zirconia in the cubic form prior to its use as a dispersion strengthener by the addition to it of such stabilizers as calcia, baria, strontia.

In sum, this invention is directed to the treatment of dispersion strengthened alloys in which the dispersoid is prone to transform to other phase strurctures having an adverse effect upon the strength properties of the final alloy. Among the metals which may be dispersion strengthened in accordance with the invention are included the copper group (Cu, Ag, Au), iron group (Fe, Ni, Co) and platinum group (Pt, Pd, Ir, Ru, Rh, etc.) metals. Alloys based on these metals are likewise included.

Examples of alloys based on the copper group metals are: 95% copper and 5% zinc; 90% copper and 10% zinc; 60% copper and 40% zinc; 71% copper, 28% zinc and 1% tin; 65% copper, 17% zinc and 18% nickel; 90% silver and 10% copper; up to 15% nickel and the balance silver; 70% gold and the balance palladium; 69% gold, 25% silver and 6% platinum, and the like.

Examples of iron group alloys include: certain steels; 64% iron and 36% nickel; 31% nickel, 4 to 6% cobalt and the balance iron; 54% iron and 46% nickel; 99% nickel and the balance cobalt; 68% nickel and 32% copper, and the like.

Examples of platinum group alloys are as follows: platinum-rhodium alloys containing up to 50% rhodium; platinum-iridium alloys containing up to 30% iridium; platinum-nickel containing up to 6 or 10% nickel; platinum-palladium-ruthenium containing 77% to 10% platinum, 13% to 88% palladium, and 10% to 2% ruthenium; alloys of palladium-ruthenium containing up to 8% ruthenium; 60% palladium and 40% silver, and the like.

For the purposes of our invention, we refer to these metals as matrix metals. We prefer to include those metals that have a negative free energy of formation of the oxide not exceeding about 70,000 calories per gram atom of oxygen at 25° C. Such metals and/or alloys preferably have melting points about 800° C.

Also for the purposes of our invention, we refer to the metal oxide dispersion hardeners as those characterized by a negative free energy of formation at 25° C. of 90,000 or over calories per gram atom of oxygen and further characterized by a melting point above that of the matrix metal, preferably above 1600° C.

The amount of oxide employed may range from about 0.5 v/o to 15 v/o of the alloy and preferably from about 1 to 12 v/o. The particle size of the oxide should preferably not exceed 0.2 micron and preferably should be maintained below 0.05 micron.

Where the alloy is produced by mixing the matrix metal with the oxide prior to consolation, we prefer that the matrix metal powder not exceed 20 microns in size and preferably not exceed 5 microns. We also prefer that the dispersoid mixed therewith be related in particle size so that it ranges from about 30 to 250 times smaller than the size of the starting matrix metal powder.

Where the dispersoid is produced by internal oxidation of a matrix alloy powder, we prefer that the particle size be controlled over the range of up to about 100 angstroms in diameter for up to about 6 v/o of oxide, the particle diameter being preferably controlled over the range of about 60 to 300 angstroms.

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.

What is claimed is:

1. A method of producing a dispersion strengthened metal characterized by improving resistance to creep at elevated temperatures which comprises, providing a matrix metal of melting point at least about 800° C. char-

acterized by a negative free energy of formation of the oxide at about 25° C. of not exceeding about 70,000 calories per gram atom of oxygen having associated therewith a uniform distribution of finely divided disperse refractory oxide particles characterized by a crystallographic phase transformable to another crystallographic phase at an elevated temperature, said refractory oxide being also characterized by a negative free energy of formation at about 25° C. of at least about 90,000 calories per gram atom of oxygen, and hot deforming said metal to a wrought shape at an elevated temperature below the temperature at which said refractory oxide crystallographically transforms to said other phase.

2. A method of producing a dispersion strengthened metal characterized by improved resistance to creep at elevated temperatures which comprises, providing a matrix metal of melting point of at least about 800° C. characterized by a negative free energy of formation of the oxide at about 25° C. of not exceeding about 70,000 calories per gram atom of oxygen having associated therewith a uniform distribution of about 0.5 v/o to 15 v/o of a finely divided disperse refractory oxide phase of average particle size not exceeding about 0.2 micron characterized by a crystallographic phase transformable to another crystallographic phase at an elevated temperature, said oxide being also characterized by a negative free energy of formation at about 25° C. of at least about 90,000 calories per gram atom of oxygen, and hot deforming said metal to a wrought shape at an elevated temperature below the temperature at which said refractory oxide transforms to said other phase.

3. A method of producing a dispersion strengthened metal characterized by improved resistance to creep at elevated temperatures which comprises, providing a matrix metal of melting point of at least about 800° C. characterized by a negative free energy of formation of the oxide at about 25° C. of not exceeding about 70,000 calories per gram atom of oxygen having associated therewith a uniform distribution of about 0.5 v/o to 15 v/o of a finely divided disperse refractory oxide phase of average particle size not exceeding about 0.05 micron characterized by a crystallographic phase which transforms to another crystallographic phase at an elevated temperature, said oxide being also characterized by a negative free energy of formation at about 25° C. of at least about 90,000 calories per gram atom of oxygen, and hot deforming said metal to a wrought shape at an elevated temperature below the temperature at which said refractory oxide transforms to said other phase.

4. The method of claim 13, wherein said matrix metal is comprised substantially of copper and wherein said refractory oxide is gamma alumina.

5. A method of inhibiting agglomeration of a disperse phase in the production of a dispersion strengthened metal which comprises, providing a matrix metal of melting point of at least about 800° C. characterized by a negative free energy of formation of the oxide at about 25° C. of not exceeding about 70,000 calories per gram atom of oxygen having associated therewith a uniform distribution of about 1 v/o to 12 v/o of a finely divided disperse refractory oxide phase of average particle size not exceeding about 0.05 micron characterized by a crystallographic phase transformable to another crystallographic phase at an elevated temperature, said oxide being also characterized by a negative free energy of formation at about 25° C. of at least about 90,000 calories per gram atom of oxygen, and hot deforming said metal to a wrought shape at an elevated temperature below the temperature at which said refractory oxide transforms to said other phase.

6. The method of claim 5, wherein said matrix metal is comprised substantially of copper and wherein the refractory oxide is gamma alumina.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,070,440

December 25, 1962

Nicholas J. Grant et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 8, line 73, for "improving" read -- improved --; column 10, line 6, for the claim reference numeral "13" read -- 3 --.

Signed and sealed this 4th day of June 1963.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

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