

[54] **METAL DISPERSOID POWDER COMPOSITIONS**

3,591,362 7/1971 Benjamin..... 75/5 BA

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[22] **Filed:** Oct. 1, 1970

[21] **Appl. No.:** 77,217

[30] **Foreign Application Priority Data**

Jan. 27, 1970 Canada ..... 073,147

[52] **U.S. Cl.**..... 75/0.5 AC, 75/206

[51] **Int. Cl.**..... B22f 9/00

[58] **Field of Search**..... 75/.5 AC, .5 BC, 75/206

[56] **References Cited**

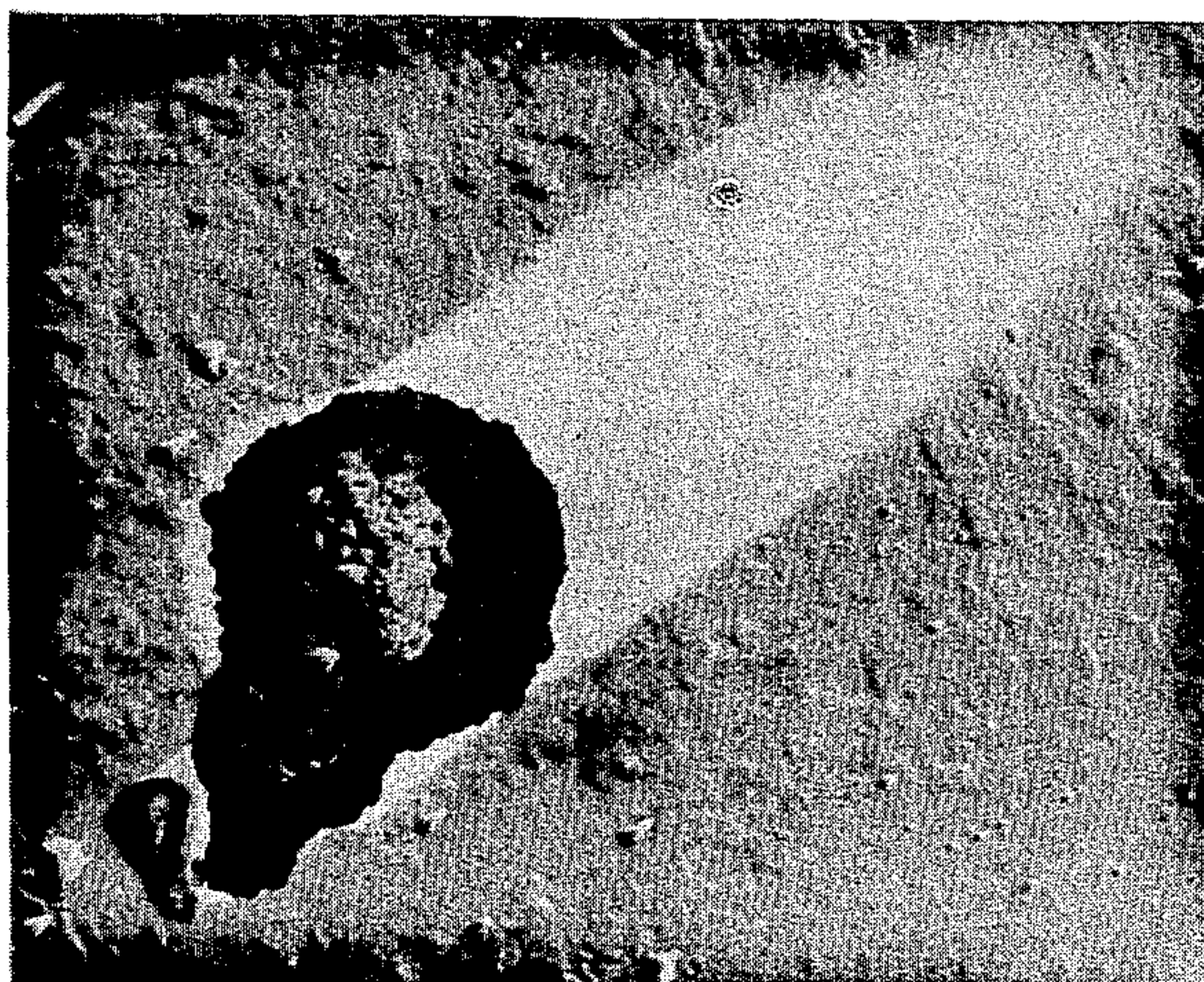
**UNITED STATES PATENTS**

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[57] **ABSTRACT**

A powder composition suitable for powder metallurgical preparation of high temperature resistant wrought metal and metal alloy products. The composition contains highly disseminated and uniformly spaced refractory dispersoid particles fixed to metallic host particles. The host particles are in the form of anisodimensional platelets of nickel, cobalt, iron or an alloy based on at least one of these metals. The platelets have a maximum thickness of about 1,000 millimicrons and a minimum width at least 5 times the thickness. The refractory dispersoid particles have a mean radius in the range of 2–20 millimicrons and are fixed in the planar surfaces of the platelets in a volume fraction of about 0.001 to 0.10.

**9 Claims, 2 Drawing Figures**





Patented June 26, 1973

3,741,748

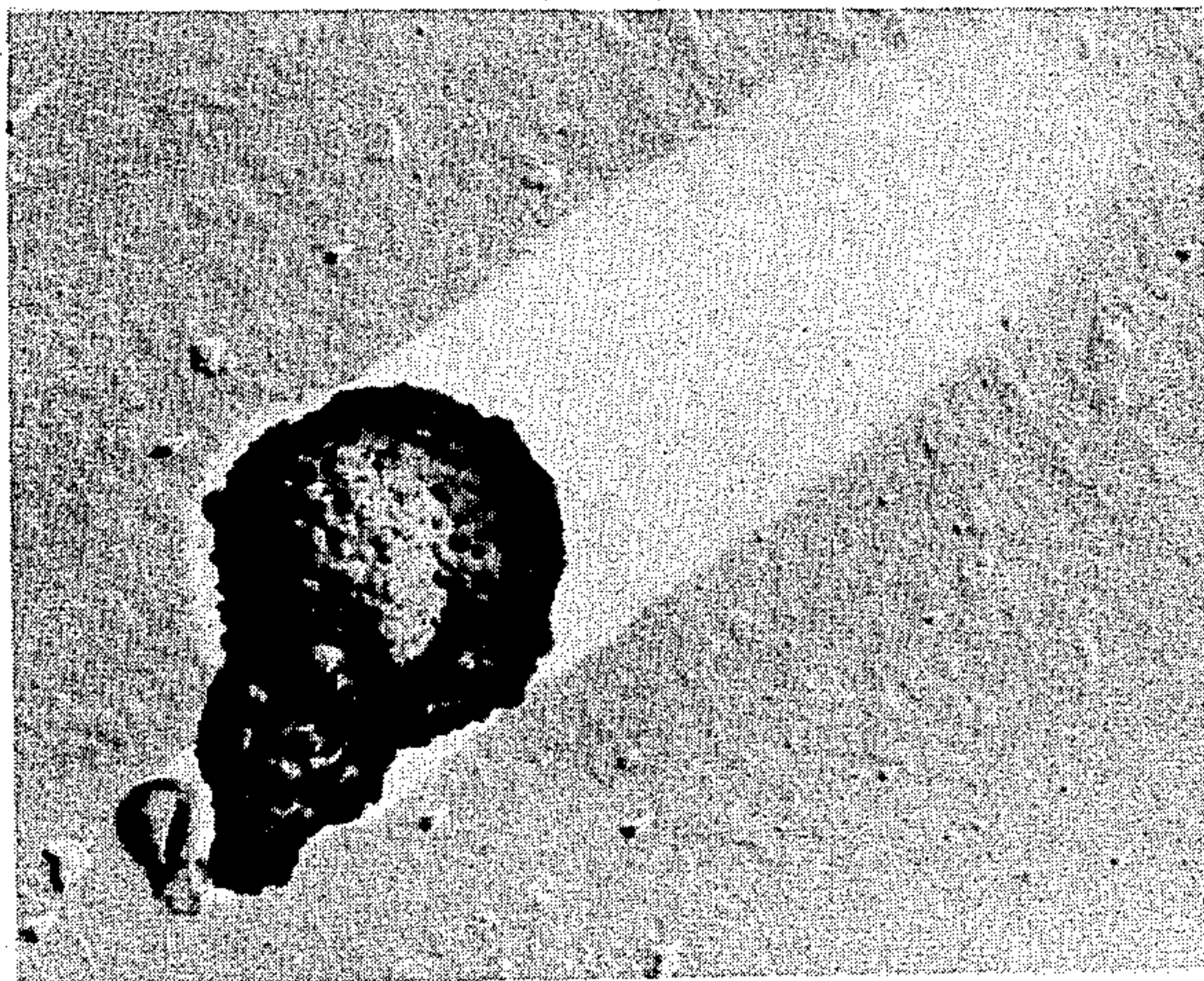


FIG. 1

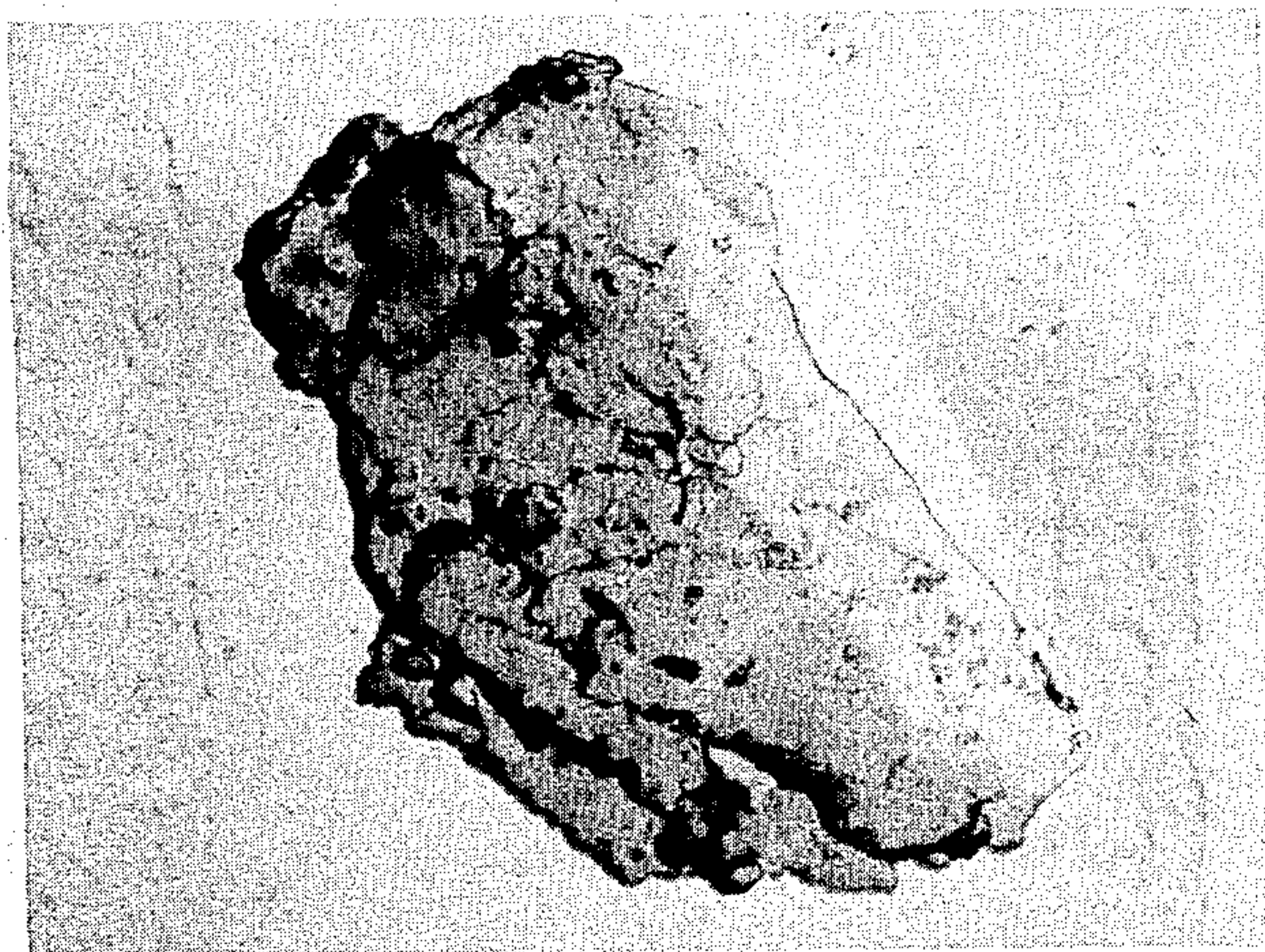


FIG. 2

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## METAL DISPERSOID POWDER COMPOSITIONS

This invention relates to dispersion modified metal and metal alloy powder compositions suitable for powder metallurgical preparation of wrought dispersion strengthened products having improved high temperature service characteristics. More particularly, it relates to a novel form of composite metal or metal alloy-dispersoid particles in which the metallic constituent is nickel, cobalt, iron or alloys of one or more of these metals including, for example, nickel-chromium and nickel-iron-chromium alloys.

Dispersion strengthened metals and alloys are well known and in use in applications where the stability of their strengths at high homologous temperatures is necessary to meet specific materials engineering requirements. Generally, these materials are made by powder metallurgy methods which involve compacting, sintering and hot and/or cold working of metal powder compositions containing the desired metal constituent and uniformly disseminated submicron dispersoid particles, such as refractory oxides, which are inert towards the metal constituents and stable at high temperatures.

It is generally agreed that the strength of such dispersion strengthened materials is inversely dependent on the inter-particle spacing of the dispersoid phase. In order to obtain minimum dispersoid particle spacing, it is customary in the art to utilize metal-dispersoid powder compositions which have been specifically prepared to provide the smallest possible inter-particle spacing between the dispersoid particles in the powder. A variety of such powder compositions and the methods for preparing them are well known in the art. Up to the present time, the existing products can be classified into two categories, namely: (1) powders in which dispersoid particles are disseminated uniformly throughout the matrix of metal particles and (2) powders in which dispersoid particles are disseminated on the surface of host metal particles of a size sufficiently small to give the desired inter-particle spacing between the dispersoid particles. Examples of the first type of powder compositions are described in U.S. Pat. Nos. 2,823,988 and 3,087,234. Powders of the second type are described in Canadian Pat. No. 824,101 and U.S. Pat. No. 3,469,967.

The processes used to produce powders of the first type are technically complex and difficult to control with the result that the powder products are very costly and of irregular quality. Powders of the second type can be produced more economically than the first type and with more consistency of product quality; however, inter-particle dispersoid spacing obtainable in these powders is dependent on the diameter of the host metal particles. Although this can be decreased by obtaining progressively finer host particles, the limiting feature in this approach is the minimum host particle size which can be attained without encountering serious technological and economic difficulties. Also, many metals, if the particle size is reduced down to the submicron range, tend to be pyrophoric and very difficult to handle in the initial stages of processing by standard power metallurgy techniques.

A method has been proposed in U.S. Pat. No. 3,434,830 for obtaining extremely small host metal particle size in metal-dispersoid powders while avoiding the problem of pyrophoricity, but this method and the resulting products have a number of serious draw-

backs which, up to the present time at least, have apparently discouraged their adoption on a commercial scale. Although the problem of pyrophoricity may be avoided by the method of this patent, because of their extremely small size, e.g. 0.2-0.5 microns, in their preferred form, the powder particles still are very difficult to handle because of their inherent low density and tendency to cluster. In addition, there are several limitations on the quantity and size of the dispersoid particles that can be fixed on the host metal particle surfaces without agglomeration of the dispersoid particles. Dispersoid particle agglomeration is undesirable because it adversely affects the strength characteristics of the wrought product fabricated from the powder by increasing the dispersoid particle size.

An object of the present invention, therefore, is to provide an easily handled and relatively inexpensive powder composition comprised of composite metal or metal alloy-dispersoid particles which are characterized by an absence of pyrophoricity and exceedingly small and uniform dispersoid inter-particle spacing over a wide range of dispersoid sizes and contents.

A more specific object is to provide such powder composition in which the host particles are formed of nickel, cobalt, iron or alloys of these metals with each other and/or other metals such as chromium, molybdenum and tungsten.

Another object of the invention is to provide an efficient and easily controlled method for preparation of such powder compositions.

The powder compositions of the invention are comprised of powder particles formed of nickel, cobalt, iron or alloys based on one or more of these metals which are in the form of extremely thin flakes or platelets having submicron sized dispersoid particles fixed in and uniformly distributed over the surfaces thereof. The plate-like particles have a maximum thickness of 1000 millimicrons and a minimum planar width 5 times the thickness. The volume fraction of dispersoid particles fixed on the host metal particles is between 0.001 and 0.10 and the mean particle radius of the dispersoid particles is between about 1 millimicron and about 20 millimicrons. These flake-like particles are non-pyrophoric, are easily handled and compactible and yet have very small dispersoid inter-particle spacing because of the extreme thinness of the flakes and the distribution of the dispersoid particles over the flakes' planar surfaces.

In the preferred form of the powder composition, the thickness,  $t$ , of the host particles is less than 600 millimicrons and relates to dispersoid particle variables  $f$  and  $r$  in accordance with the expression:  $t < (0.5(1-f)r)/(f)$  where  $f$  is the volume fraction of dispersoid in the powder composition and is between 0.015 and 0.04 and  $r$  is the mean radius of the dispersoid particles and is between 2 and 10 millimicrons. Particles whose physical characteristics satisfy this relationship have the desired small dispersoid inter-particle spacing with minimum probability of dispersoid particle agglomeration for dispersoid contents and sizes within the indicated ranges and are therefore particularly well adapted for powder metallurgical fabrication of wrought dispersion strengthened metal or metal alloy products.

Metals dispersoid powder compositions of the invention preferably are prepared by altering to platelet form substantially equi-axed powder particles of a size be-



tween about 0.2 and about 5 microns which are formed of nickel, cobalt, iron or an alloy based on at least one of these metals and which have submicron refractory dispersoid particles fixed on the surfaces thereof. The equi-axed powder particles must be prepared or selected such that they contain from about 0.1 to about 10 volume percent dispersoid particles in the size range of about 2.0 to about 40 millimicrons particle diameter. In the preferred case, these dispersoid particles are controlled such that upon alteration of the shape of the equi-axed particles to thin platelets, the volume fraction of dispersoid and mean particle size of the dispersoid relate to the platelet thickness in accordance with the expression:  $t < (0.5(1-f)r)/(f)$  where  $t$  is the platelet thickness and is less than 600 millimicrons,  $f$  is the volume fraction of dispersoid in the powder composition and is between 0.015 and 0.04 and  $r$  is the mean radius of the dispersoid particles and is between 2.0 and 10 millimicrons. Alteration to platelet or flake form is effected by milling the equi-axed composite metal or metal alloy-dispersoid particles under specifically controlled conditions which effect a flattening of the individual composite particles while distributing the dispersoid particles on the surfaces thereof.

The word "dispersoid" as used herein means refractory metal oxide particles or any of the other high melting point refractory particles such as borides, nitrides and carbides which may usefully be employed in the dispersion strengthening of metals. The term "metal" or "metallic" is used hereinafter in the broad sense to include alloys as well as pure metals.

In the powder compositions of the invention, the metallic host particles may be formed of nickel, iron or cobalt as well as alloys of these metals with each other and with chromium or other metals such as beryllium, magnesium, manganese, molybdenum, niobium, tantalum, titanium, vanadium, zirconium and tungsten. Particularly useful compositions are those in which the host particles are pure nickel or 80-20 nickel-chromium alloy.

Particularly suitable dispersoid particles are the high melting point refractory oxides including  $Y_2O_3$ ,  $ThO_2$ ,  $MgO$ ,  $CaO$ ,  $ZrO_2$ ,  $SiO_2$ ,  $UO_2$ ,  $La_2O_3$ ,  $BeO$ ,  $Al_2O_3$ ,  $HfO_2$ ,  $CeO_2$  and  $TiO_2$ .  $ThO_2$  is generally recognized as a preferred dispersoid because of its high melting point, availability at reasonable cost and because it can be readily prepared in the preferred particle size ranges. The dispersoid particles, which must be fixed to and uniformly distributed on the surfaces of the host particles, have a mean particle diameter within the range of about 4 to about 40 millimicrons. For optimum high temperature strength properties in wrought products fabricated from the powders, dispersoid particles with mean particle diameter between 4 and 20 millimicrons are preferred. The amount of dispersoid is between about 0.1 and 10 volume percent (0.001 and 0.10 volume fraction) depending on the type of dispersoid employed and the properties desired in the end product. Normally, for optimum mechanical properties, the dispersoid volume fraction should be in the range of about 0.015 to about 0.04. Preferably, the volume fraction  $f$  of dispersoid particles, the dispersoid mean particle radius  $r$  and the host particle thickness  $t$  are correlated in accordance with the relationship;  $t < (0.5(1-f)r)/(f)$  with  $t$  being less than 600 millimicrons. Maintenance of this relationship ensures optimum inter-particle spacing of the dispersoid particles within the specified

quantity and particle size ranges while minimizing the probability of dispersoid particle agglomeration on the surfaces of the metallic host particles.

For compositions containing dispersoid particles within the above-noted ranges of quantity and size, the maximum host particle flake or platelet thickness that results in a satisfactory product is 1 micron. Preferably, however, it is desirable that the platelet thickness be as close as possible to the value obtainable from the equation:  $t = (0.5(1-f)r)/(f)$ . For example, for plate-like host particles containing 0.03 volume fraction of dispersoid particles having a mean particle radius of 8 millimicrons, the preferred plate thickness,  $t$ , is 140 millimicrons. This particle thickness value, and the resulting dispersoid, interparticle spacing, is very small and cannot, as a practical matter, be approached by equi-axed particles without pyrophoricity and other production and handling problems. For the plate-like metal-dispersoid particles of the invention, there are no restrictions on the planar dimensions of the host metal particles. That is, the flakes are relatively large and therefore nonpyrophoric and easily handled while they still exhibit dispersoid inter-particle spacing which is equivalent to that obtainable with only extremely small equi-axed particles by reason of the control of flake particle thickness within the limits described.

The metal dispersoid powder compositions of the invention are prepared by converting to flake-form finely divided metal-dispersoid particles which have certain specific physical and chemical qualities. The host metal particles must be formed of nickel, cobalt, iron or an alloy of one or more of these metals such as nickel-chromium or nickel-iron-chromium alloy; they must be generally equi-axed or spherical in shape; they must have a particle size in the range of about 0.2 to about 5 microns and they must have submicron dispersoid particles, such as thoria particles, fixed in the surfaces thereof.

Nickel-dispersoid particles which are ideally suited as a starting material for the method of the invention may be prepared by the process described in the aforementioned Canadian Pat. No. 824,101 and U.S. Pat. No. 3,469,967. Suitable nickel-chromium alloy-dispersoid particles may be prepared by chromizing nickel-dispersoid particles of the aforementioned patents in accordance with the method described in co-pending application, Ser. No. 813,214.

Suitable nickel-dispersoid powders may also be prepared by depositing a hydrated salt, e.g. thorium hydroxide, of the dispersoid on the surfaces of nickel powder particles having the required physical characteristics and then calcining the precipitate to oxide form. The nickel powder described in U.S. Pat. No. 3,399,050 is particularly well adapted for this purpose because of the knobby, equi-axed character of its constituent particles. Cobalt-dispersoid, iron-dispersoid and various nickel, cobalt and/or iron base alloy-dispersoid powder, can be prepared by similar methods.

Regardless of the composition of the metal host particles, it is necessary for the purposes of the invention, that the quantity and particle size of the dispersoid particles associated with the starting powder be selected within the ranges discussed above. For the preferred case, these variables must be controlled such that when the metal host particle is altered from an equi-axed shape to a plate-like shape, the thickness of the result-



ing platelets relate to the dispersoid variables  $f$  and  $r$  in accordance with the expression:  $t < (0.5(1-f)r)/(f)$  where  $t$  is the platelet thickness and is less than 600 millimicrons,  $f$  is the volume fraction of dispersoid particles and is between 0.015 and 0.040 and  $r$  is the mean particle radius of the dispersoid particles and is between 2 and 10 millimicrons. Observation of this condition ensures desired small inter-particle spacing for the dispersoid particles within the indicated volume fraction and particle size ranges.

The alteration of the equi-axed metal-dispersoid starting powder particles to flake or platelet form is achieved in a surprisingly simple manner. It has been found that, quite unexpectedly, the equi-axed starting particles, despite their extremely small particle size (0.2–5.0 microns) can be converted to very thin flakes by milling provided the milling parameters, particularly milling time, are properly controlled.

Milling may be carried out in conventional ball or attrition mills provided with suitable liners and milling mediums to prevent contamination of the powder charge. E.g., for milling nickel-dispersoid powders, it is preferred to employ a nickel-lined mill and nickel or stainless steel milling balls. The attrition type mill is generally preferred because of its characteristic high rate of energy input. Also, it is preferred to utilize wet milling with between about 20 percent and 40 percent water being added to the powder charge.

Control of milling time (or energy input) is extremely important in order to ensure conversion of the particles to flakes. The optimum time required will vary depending on the nature of the mill employed, the composition of the metal-dispersoid powder and the milling medium-powder ratio and can only be precisely determined for a given case by conducting trial runs for various time periods. In general, for wet milling with powder-ball ratios in the preferred range of 0.1 to 0.5 milling time required will be in the range of 10 to 48 hours for attrition milling and 50 to 200 hours for conventional ball milling.

After the powder has been milled to flake form, it is separated from the milling medium such as by screening and then dried.

In the case of powders which are susceptible to oxidation during milling, e.g., nickel and iron powders, the powder preferably is deoxidized by heating in hydrogen for about 30 minutes at a temperature in the range of about 1,000° F. to about 1,700° F.

The powder products which have the novel physical characteristics described in detail hereinabove are particularly well adapted for powder metallurgical fabrication of dispersion strengthened sheets and the like products. The following examples further illustrate the invention and the particular utility of the product.

#### EXAMPLE 1

A nickel-thoria powder sample prepared in accordance with the method described in Example 1 of U.S. Pat. No. 3,469,967 was employed for this example. The powder contained 2.7 volume percent thoria having a mean particle diameter between 5 and 15 millimicrons, balance Ni and had an apparent density of 1.5 gm/cc and a Fisher Number of 1.4. Approximately 3,500 grams of powder were added to a Szegvari attritor mill which contained 2 liters of distilled water and 40 pounds of three-sixteenths inch stainless steel balls. The charge was then milled for 48 hours ( approxi-

mately 550,000 revolutions). The grinding balls were wet screened from the resulting slurry and the slurry was then decanted, dried at 200° F. in a forced air furnace for 15 hours and de-oxidized at 1,400° F. for 2 hours in purified hydrogen.

Electron microscopic examination of the powder using the shadowed carbon extraction replica technique showed that the particles were in flake or platelet form having a particle thickness,  $t$ , in the order of about 400 millimicrons — well within the limits prescribed by the expression  $t < (0.5(1-f)r)/(f) < 1,000$  millimicrons. The average width to thickness ratio of the flake particles was relatively high, in the order of 10:1.

A sample of this powder was fabricated into wrought form by first forming a 60 gm billet by static compaction, sintering the billet for 2 hours at 2,200° F. in purified hydrogen, hot rolling the sintered billet at 2,000° F. and twice cold rolling the strip with 70 percent reductions and intermediate and final anneals at 2,200° F. for 30 minutes in purified hydrogen. The tensile properties of the resulting strip at 2,000° F. were: UTS — 18,900 p.s.i., YS — 14,300 p.s.i.

For comparison purposes, a sample of the unmilled starting powder was fabricated into strip using the same procedure as for the flake powder. The UTS of the strip at 2,000° F. was 11,600 p.s.i. This shows a strength increase of over 60 percent by conversion of the powder to flake form.

#### EXAMPLE 2

A nickel-thoria dispersoid powder was prepared as follows: The starting material was a nickel powder prepared by the method of U.S. Pat. No. 3,399,030. The powder had an apparent density of 1.0 gm/cc and a Fisher Number of 4.2 (particle size between 0.2 and 5.0 microns). 3,600 grams of this powder were added to 2.6 liters of distilled water containing 90 gm/l of dissolved  $\text{Th}(\text{NO}_3)_4 \cdot 4\text{H}_2\text{O}$ . The resulting slurry was stirred for 10 minutes and then dried in a forced air furnace at 200° F. for 15 hours. The dried powder was then de-oxidized by heating in air at 1,400° F. for 3 hours. The calcined powder was cooled in purified hydrogen.

The powder was then milled in a Szegvari attritor in the same way as described in Example 1. Electron microscopic examination of the powder showed that it had the same flake-like appearance as the milled powder of Example 1. Carbon extraction replicas of the starting powder and the milled powders were made and shadowed with chromium at an angle of 70° from the normal. These are shown in FIGS. 1 and 2 of the drawing and illustrated clearly the alteration of the shape of the particles from equi-axed to plate or flake-like form. Samples of this powder were fabricated into strip using the procedure of Example 1. The tensile properties at 2,000° F. were: UTS — 19,200 p.s.i., YS — 14,800. Tensile properties of strip obtained from the unmilled starting powder by the same fabrication procedure were: UTS — 5,700 p.s.i., YS — 5,400 p.s.i., indicating, in this case, a strength increase of more than 200 percent by conversion of the nickel-dispersoid starting powder to flake form.

#### EXAMPLE 3

Thirty-two pounds of nickel-thoria powder prepared in the same manner as described in Example 2, together with 140 pounds three-sixteenths inch diameter stainless steel grinding balls and 10 liters of distilled wa-



ter, were charged into a 16 × 16 inch ball mill constructed from 316 type stainless steel. The mill was rotated at 42 r.p.m.

Milling was continued for 132 hours with samples being taken periodically and examined and fabricated into wrought strip. The fabrication procedure consisted of two 50 percent hot rolling reductions and an anneal at 2,450° F. for one-half hour, a 70 percent cold rolling reduction and a final anneal at 2,450° F. for one-half hour. The results are shown in Table I along with the characteristic strength of the strip fabricated from the powder samples.

TABLE I

Ball Milling Time (Hours)	Number of Revolutions	2,000°F. Tensile *Properties of Strip UTS p.s.i.	YS p.s.i.
0	0	7,500	6,300
20	48,000	11,600	11,300
32	81,000	12,600	11,900
50	127,000	13,000	12,500
64	163,000	13,300	11,400
80	203,000	14,200	12,400
98	246,000	17,300	13,100
115	289,000	18,900	16,900
132	332,000	21,500	18,300

\* Average of two tensile tests.

It can be observed that an initial increase in strength was obtained after the first 20 hours of milling and that for the subsequent 60 hours, very little additional improvement was obtained. In the subsequent 40 hours, however, a marked improvement was obtained with the 132 hour strength being almost double the 20 hour strength. Observation of the powder samples taken at the various time intervals showed that very few of the powder particles were altered to flakes of less than 1000 millimicrons thickness in the first 50–60 hours of milling. The strength increase in this period was attributed primarily to redistribution of the dispersoid on the nickel powder particles rather than to any significant change in the nickel particle shape. The 80 hour powder sample contained some flake-like particles but the major portion of the powder still consisted of generally equi-axed particles most of which were larger than 1 micron in their smallest dimension. The proportion of flake-like particles less than 1 micron thick increased gradually in the 98 and 115 hour samples and the 132 hour sample was virtually all flake-like particles having thickness in the 200–600 millimicron range.

## EXAMPLE 4

400 grams of the de-oxidized flake-like nickel/thoria powder produced in accordance with Example 1 were blended with 100 grams of air classified electrolytic grade chromium powder having a Fisher Number of 8 by mixing for two minutes in a high speed mechanical blender. The resulting powder blend was placed into an open boat and inserted in the cooling zone of a tube furnace. The powder blend was then contacted with dry hydrogen in the cooling zone for 15 minutes, then inserted into the heating zone where it was heated at 1,900° F. in a flowing H<sub>2</sub> atmosphere having a dew point at point of entry of –130° F. for 50 hours. The boat was then removed from the furnace and cooled. The slightly caked powder product was broken into a free flowing non-magnetic powder. X-ray diffraction analysis indicated that the chromium was in uniform solid solution with the nickel. Electron microscopic ex-

amination showed that the major portion of the powder particles had the same flake-like shape as the starting nickel/thoria powder particles.

## EXAMPLE 5

Carbonyl iron powder having a Fisher Number of 4.8 and an apparent density of 2.6 gm/cm<sup>3</sup> was used to prepare thoriated iron powder. 3,000 grams of the carbonyl iron powder were added to 2 liters of a 100 gram/liter solution of thorium nitrate. The reaction was much more rapid than for the nickel-thorium nitrate system of Example 2 and occurred almost instantaneously as the iron powder was added to the thorium nitrate solution. The powder was dried and calcined at 1,500° F. in purified hydrogen for 1 hour.

The de-oxidized thoriated iron powder which contained 3.13 wt. percent thoria was ball milled in an 8 inch laboratory ball mill using 20 pounds of three-sixteenths inch stainless steel balls along with 3,000 grams of the thoriated powder and 1.0 liter of methanol. Methanol was used rather than water to prevent oxidation of the iron. The charge was milled at 60 r.p.m. for 139 hours. Following milling, the powder was dried and de-oxidized at 1,500° F. for 1 hour in purified hydrogen. Micrographic examination of the final powder product showed that the particles had the same flake-like shape as the milled nickel powders of Examples 1–3.

Samples of the iron-thoria powders taken before and after the milling step were compacted at 33 t.s.i. into a 1¼ in × 2.4 inch billets weighing 60 grams and sintered for 2 hours at 2,200° F. in purified hydrogen. The sintered billets were then placed in a furnace at 2,000° F. and held at this temperature for 15 minutes prior to a 50 percent hot roll reduction. This procedure was repeated and the billets were then cold rolled 70 percent following the second hot roll reduction. The cold rolled iron-thoria was then annealed at 2,550° F. in purified hydrogen for 30 minutes. The product properties and other data are shown in Table II.

TABLE II

Treatment	ThO <sub>2</sub> Content (%)	Particle Shape Factor	UTS at 2,000°F. (psi)
Pure Fe	0	Generally equi-axed	2500
As calcined	2.47	Generally equi-axed	4500
Attritor milled 139 hrs.	2.42	Anisodim- ensional plates	6500

## EXAMPLE 6

Fine cobalt powder was obtained by the reduction of Co(NH<sub>4</sub>)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub>·6H<sub>2</sub>O at 1,000° F. in purified hydrogen. The powder had a Fisher Number of 1.2 and an apparent density of 0.7 gm/cm<sup>3</sup>. 3,000 grams of this cobalt powder were added to 2 liters of a 100 gram/liter solution of thorium nitrate. After the reaction was completed, the powder was dried and calcined at 1,500° F. for 2 hours in purified hydrogen. 2,600 grams of this calcined powder were milled for 48 hours in a laboratory ball mill using 40 lbs. of three-sixteenths inch stainless steel balls and 2 liters of water. After milling, the powder was dried and then de-oxidized at 1,500° F. for 1 hour in H<sub>2</sub>. The powder particles had the same flake-like shape as the previously described products.



Samples of milled and unmilled cobalt-thoria powders were compacted at 33 t.s.i. into a 1¼ inch × 2.4 inch billet weighing 60 grams. The compacts were then sintered for 2 hours at 2,200° F. in purified hydrogen. The sintered compacts were then heated in a furnace at a temperature of 2,200° F. and held at that temperature for 15 minutes. The billets were then given a 30 percent hot roll reduction following which they were immediately replaced in the furnace and held at 2,200° F. for 10 minutes prior to a second 30 percent hot reduction. This procedure was repeated until four 30 percent hot roll reductions had been accumulated, the overall reduction being 75 percent. The hot rolled billets were then given a final anneal in purified hydrogen for 30 minutes at 2,550° F. The product properties and other data are shown in Table III.

TABLE III

Treatment	ThO <sub>2</sub> Content (%)	Particle Shape Factor	UTS at 2000°F. (psi)
Pure Co	0	Generally equi-axed	4700
As calcined	3.03	Generally equi-axed	9300
Milled 48 hrs.	2.97	Anisodimensional plates	12,800

EXAMPLE 7

3,200 grams of fine copper powder having a Fisher Number of 1.4 and an apparent density of 0.6 gm/cm<sup>3</sup> were added to 2.1 liter of a 85 gm/liter solution of thorium nitrate. After the reaction was complete, the powder was dried and calcined at 1,200° F. for 1 hour in purified H<sub>2</sub>.

3,000 grams of the calcined product were milled for 48 hours in a laboratory attritor mill with 2 liters of water and 40 lbs. of 3/16 inch stainless steel balls. The powder product was separated from the balls, dried and de-oxidized by heating in dry H<sub>2</sub> at 1,200° F. for 1 hour. The product properties and other data are shown in Table IV.

TABLE IV

Treatment	ThO <sub>2</sub> Content (%)	Particle Shape Factor	UTS at 1650°F. (psi)
Pure Cu	0	Generally equi-axed	1,100
As calcined	2.7	Generally equi-axed	7,700
Attritor Milled 48 hrs.	2.7	Anisodimensional plates	10,200

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

1. A finely divided, compactible and non-pyrophoric powder composition suitable for powder metallurgical preparation of high temperature resistant wrought metal and metal alloy products containing a highly dis-

seminated and uniformly spaced dispersoid particles which comprises metallic host particles formed of nickel, cobalt, iron or an alloy based on at least one of said metals, said host particles being in the form of anisodimensional platelets having a maximum thickness of about 1,000 millimicrons and a minimum width at least 5 times the thickness, said host particles having fixed in the planar surfaces thereof from about 0.001 to about 0.10 volume fraction of refractory dispersoid particles having a mean particle radius between about 2 and about 20 millimicrons.

2. The powder composition according to claim 1 wherein the thickness, *t*, of the host particles relates to dispersoid particle variables *f* and *r* in accordance with the expression  $t < (0.5(1-f)r)/(f)$  wherein *f* is the volume fraction of dispersoid in the powder composition and *r* is the mean radius of the dispersoid particles.

3. The composition according to claim 2 wherein *t* is less than 600 millimicrons, *r* is between 2 and 10 millimicrons and *f* is between 0.015 and 0.040.

4. The composition according to claim 3 wherein the host particle is formed of nickel and the dispersoid is formed of thoria.

5. The composition according to claim 3 wherein the host particle is formed of nickel-chromium alloy and the dispersoid is formed of thoria.

6. A process for producing metal and metal alloy refractory dispersoid powder suitable for use in powder metallurgy fabrication of wrought dispersion strengthened metal and metal alloy products which comprises the steps of: providing a powder composition comprises of substantially equi-axed metallic particles formed of nickel, cobalt, iron or an alloy based on at least one of these metals, said metallic particles being in the size range of about 0.2 to about 5 microns and having fixed in the surfaces thereof from about 0.001 to about 0.10 volume fraction of refractory dispersoid particles having a mean particle radius of about 1 to about 20 millimicrons, milling said powder composition for a time sufficient to alter said substantially equi-axed particles to plate-like particles having a maximum plate thickness of about 1,000 millimicrons.

7. The process according to claim 6 wherein wet attrition milling is employed.

8. The process according to claim 7 wherein milling is continued until the plate-like particles have a thickness of less than 600 millimicrons.

9. The process according to claim 7 wherein the substantially equi-axed metallic particles are selected such that upon alteration of the shape of said metallic particles to platelets, the dispersoid volume fraction and mean particle size relate to the platelet thickness in accordance with the expression  $t < ((0.5(1-f)r)/(f))$  where *t* is the platelet thickness, *f* is the dispersoid volume fraction and *r* is the mean dispersoid particle radius.

\* \* \* \* \*