United States Patent [19]

Evans et al.

[11] **3,716,357**

[45] **Feb. 13, 1973**

[54] **PREPARATION OF THORIATED NICKEL-CHROMIUM ALLOY POWDER**

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[22] Filed: **April 3, 1969**

[21] Appl. No.: **813,214**

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

[51] **Int. Cl.**.....**C22c 1/10**

[58] **Field of Search**.....75/206, 211, 224, 221, 0.5

[56] **References Cited**

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[57] **ABSTRACT**

Nickel-chromium alloy powder containing dispersed refractory oxide particles is produced by heating nickel powder particles containing physically inseparable sub-micron refractory oxide particles, a major portion of which are fixed in the surfaces of the nickel particles, in contact with a chromium source material, such as chromium powder or chromium oxide, in a flowing atmosphere of pure dry hydrogen at an elevated temperature and for a time sufficient to effect diffusion of chromium into the nickel particles and to reduce excess oxygen to an acceptable level.

8 Claims, 2 Drawing Figures

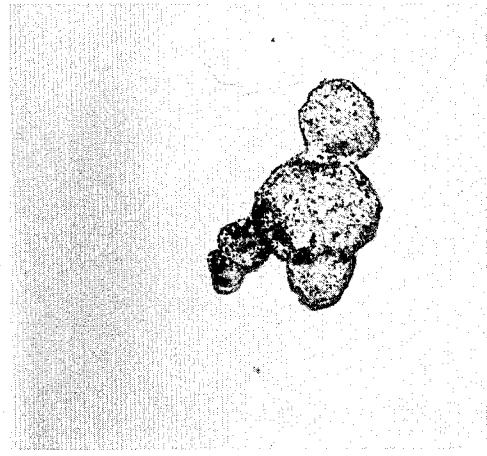


FIG. 1

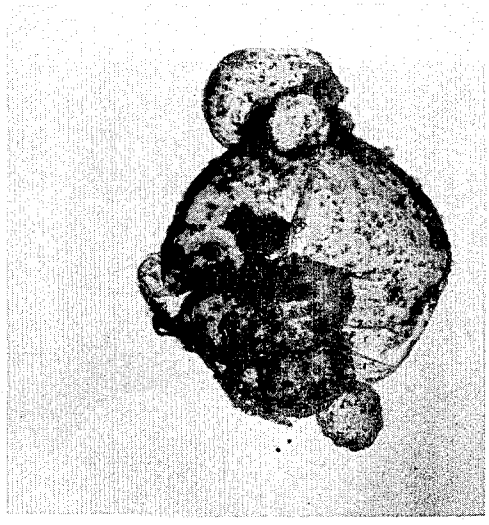


FIG. 2

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PREPARATION OF THORIATED NICKEL-CHROMIUM ALLOY POWDER

This invention relates to nickel-chromium alloy powders containing dispersed refractory oxide particles and to a process for producing such powders. More particularly, it is concerned with such powders and their production in a form which is particularly adapted for direct fabrication into wrought dispersion strengthened nickel-chromium and nickel-chromium base alloys by powder metallurgy techniques.

Advances in the design of turbo-jet engines and the advent of aerospace vehicles have resulted in the need for materials which can withstand high temperatures for extended periods. Although improvements in super alloys and in particular nickel base super alloys have greatly extended the high temperature capabilities of these materials, the gap between the requirements of the design engineer and the materials commercially available has not narrowed appreciably. One new group of materials which appears to have considerable potential for application in the temperature range of 1,800°F. to 2,400°F. are the so-called dispersion strengthened or dispersion hardened nickel and nickel base alloys.

In these materials, the high temperature strength properties are considerably improved by the presence in the metal or metal alloy matrix of the material of uniformly dispersed, ultrafine refractory particles which are stable and substantially insoluble in the matrix at elevated temperatures. These materials are generally fabricated by powder metallurgical methods involving the compacting and sintering of metal powder mixtures containing the desired metal and refractory oxide ingredients, followed by mechanical working of the sinter body to produce a fully dense, wrought shape. Optimum refractory oxide dispersion and maximum strength properties are obtained by the use of powder compositions in which the refractory oxide constituent is in the form of discrete particles incorporated in one or more of the metal powder constituents of the mixture such that they are mechanically inseparable therefrom.

Serious difficulties are encountered in applying the conventional powder metallurgy techniques to the fabrication of dispersion strengthened nickel-chromium alloys. The problems result largely from the tendency of chromium to form stable oxides. When the chromium powder particles are exposed to air, they acquire surface oxide layers that are extremely difficult to reduce during sintering. The oxide contaminates have a deleterious effect on the properties of dispersion strengthed wrought products fabricated from powder mixtures containing nickel-chromium powders. They hinder the inter-diffusion of chromium and nickel during the sintering operation with the result that only incomplete homogenization of the nickel-chromium matrix is obtained and the benefit which can normally be derived from alloying chromium with nickel is not fully realized. Also, chromium oxide contaminates adversely affect the oxidation resistance of the wrought product and are apparently a factor in causing undesirable agglomeration of the refractory oxide particles during processing, which further adversely affects the high temperature service characteristics of the alloy product. Further, the presence of chromium oxide contaminants in the wrought products cause problems in the joining of the wrought material in that the oxides tend to react with the brazing alloy causing gas holes which weaken the brazed joint.

These problems can be obviated to some extent by providing a small amount of a getter material, such as magnesium, in the nickel-chromium-refractory oxide powder mixture, by taking precautions to employ chromium powders with an extremely low oxide content or by sintering the billets in a very dry hydrogen atmosphere for extended periods. However, these methods are generally unsatisfactory for a commercial scale operation. Also, even in the absence of chromium oxide contaminants, with the conventional powder mixing approach there is a tendency for undesired refractory oxide stringers to form at the interfaces between the nickel and chromium powder particles during fabrication.

An obvious and expedient solution to all these problems is to employ a chromium oxide-free, pre-alloyed nickel-chromium powder containing uniformly dispersed, physically inseparable refractory oxide particles. However, heretofor, no economic, effcient method for producing such powders was known. One known method, described in U.S. Pat. No. 2,972,529, involves the coprecipitation of preformed, sub-micron refractory oxide particles together with the hydrous oxides of nickel and chromium, followed by filtering, washing, drying, pulverizing and reducing the coprecipitate at elevated temperature with dry hydrogen. Although this process may be capable of producing a powder product which is suitable for direct powder metallurgical fabrication of wrought, dispersion strengthened nickel chromium, it is technologically complex and it requires substantial expenditure of time in the steps of washing and drying the coprecipitate and drying the hydrogen as well as high capital investment in equipment for carrying out these operations. The result is that the final product is very costly and its use is necessarily restricted to applications where material costs are not an important consideration.

A principal object of the present invention, therefore, is to provide a simple, efficient and economic process for the production of nickel-chromium alloy-refractory oxide powder compositions suitable for powder metallurgical fabrication of dispersion strengthened nickel-chromium and nickel-chromium base alloy products. A further object of this invention is the provision of a novel form of pre-alloyed nickel-chromium alloy powder containing mechanically inseparable sub-micron refractory oxide particles in integral association therewith.

These and other objects of this invention are accomplished by means of a surprisingly simple process involving the steps of providing nickel powder particles containing physically inseparable, sub-micron refractory oxide particles a major portion of which are fixed in the surfaces of said nickel particles; mixing the nickel-refractory oxide particles with a finely divided chromium source material consisting of chromium metal, chromium oxide, chromium hydroxide or mixtures thereof in an amount sufficient to provide from about 10 to about 35 wt. percent chromium in the powder mixture; forming a stationary bed of the resulting

powder mixture and heating said bed at a temperature above about 1,200°F. but below the sintering temperature of the powder mixture in a flowing atmosphere of pure dry hydrogen; continuing the heating for a time sufficient to effect diffusion of substantially all available chromium into the nickel particles and to reduce the amount of oxygen in the powder mixture to less than 0.6 percent oxygen in excess of the oxygen contained in the refractory oxide constituent; and recovering the resulting refractory oxide containing nickel-chromium alloy powder product.

Substantially complete alloying of nickel and chromium and removal of excess oxygen generally can be obtained by heating within the aforesaid temperature range in flowing hydrogen having a dew point below about −4°F. for from about 1 to about 100 hours with the actual time depending on the sintering temperature and the hydrogen dew point employed. This result is surprising in that the literature indicates that only about 80 percent reduction of pure Cr_2O_3 should be obtainable under these conditions. The increase in the reduction reaction rate and efficiency apparently results from the fact the nickel in the powder mixture acts as a sink for free chromium during the Cr_2O_3 reduction reaction and thus changes the equilibrium of the reaction.

Chromizing of the nickel-refractory oxide starting material in accordance with the invention takes place more rapidly as the reaction temperature is increased. However, for any given nickel-thoria powder, there is an upper limit on the chromizing temperature above which excessive sintering occurs with the result that the product is not a free-flowing powder. We have found that for nickel-refractory oxide powders having a major portion of the refractory oxide particles fixed in the surfaces thereof, the maximum permissible chromizing temperature for production of a free-flowing powder product is a function of the volume loading of the refractory oxide in the nickel powder. For nickel-refractory oxide powders containing up to 25 volume percent refractory oxide, the relation may be expressed by the equation:

$$Tcm = -19.4/(f + 0.028) + Tm$$

where Tcm is the maximum chromizing temperature in °K, Tm is the melting point in °K of nickel-chromium alloy of the selected composition, and f is the volume fraction of refractory oxide in the nickel-dispersoid powder.

The particles of the nickel-chromium alloy-refractory oxide powder product of the invention retain essentially the same physical characteristics as the nickel-refractory oxide starting particles. That is, the particle size and shape, the refractory oxide size and the refractory oxide distribution of the nickel-refractory oxide starting material determines what these characteristics are in the nickel-chromium alloy-refractory oxide powder product. Thus, the quality and character of the alloy powder product is controlled primarily by controlling the quality and character of the starting nickel-refractory oxide powder.

A preferred novel product of the process is nickel-chromium alloy-refractory oxide powder containing about 10 wt. percent to about 35 wt. percent chromium, about 0.5 to about 20 percent by volume sub-micron refractory oxide and the balance nickel and consisting essentially of grape-like clusters, up to about 50 microns in size, of generally spheroidal nickel-chromium alloy particles about .5 micron or smaller in size and having a major portion of the refractory oxide content fixed in the surfaces of the nickel-chromium alloy particles. An important characteristic of the alloy particles is that they have a uniform nickel-chromium alloy composition throughout, i.e. they do not have a chromium rich outer layer and a chromium deficient inner core.

In the practice of the invention, the precise source or manner of preparation of the nickel-refractory oxide powder starting material is not important. However, since the characteristics of the nickel-refractory oxide powder are directly reflected in the product, it is desirable that the refractory oxide constituent be in discrete, sub-micron sized form uniformly distributed throughout the nickel powder and that there be essentially no large volume of nickel devoid of refractory oxide particles. Since it is essential that the major portion of the refractory oxide be fixed on the surfaces of the nickel particles this latter requirement can best be met by using nickel-refractory oxide powder comprised of clusters of very fine particles of nickel, 0.5 micron in size or smaller, having refractory oxide particles fixed in their surfaces.

Co-pending U.S. applications Ser. Nos. 543,495 and 604,129 now U.S. Pat. Nos. 3,469,967 and 3,526,498 respectively describe composite nickel-thoria powders which are particularly suitable for use in the process of this invention. These powders, which are produced by methods involving direct hydrogen reduction of basic nickel compounds in aqueous suspensions, consist of irregular-shaped particles of nickel comprised of clusters of sub-particles of nickel between about 0.2 and about 0.5 micron in size. The nickel sub-particles have ultrafine thoria fixed in their surfaces and may occur singly or be agglomerated in grape-like clusters up to 50 microns or more in size. The thoria particles preferably are between 2 and 50 millimicrons in size and are uniformly distributed on the surfaces of the nickel-sub-particles. Typical powders have an apparent density between 0.5 and 2.5 grams per cubic centimeters, a Fisher number of less than 2.0 and preferably contain from about 2.0 to about 4.0 percent by volume of one or more refractory oxides. The refractory oxide particles must have a melting point higher than the matrix metal, a good thermal stability, low solubility in the matrix metal and should be non-reactive with the matrix metal at elevated temperatures in the order of about 2,400°F. There are a large number of refractory oxides which satisfy the conditions necessary for use as a dispersed phase. For example, yttria, ceria and thoria have all been shown to be particularly suitable. Because of its ready commercial availability and high free energy of formation value, thoria is a preferred dispersoid.

Suitable starting powders for the process may also be prepared by mechanical mixing, e.g. by ball-milling high purity fine nickel powder to which refractory oxide particles have been added. Nickel powders prepared by the carbonyl process or by direct hydrogen reduction of basic nickel carbonate slurries as described in Canadian Pat. No. 774,036 may ad-

vantageously be employed for this purpose. The refractory oxide constituent, e.g. thoria, can be added to the nickel powder in the form of discrete particles, e.g. by the addition of thoria powder or sol or thorium nitrate can be mixed with the nickel powder and then calcined to form thoria in situ. Nickel refractory oxide powders can be produced by the latter procedure which have characteristics similar to the powders produced by the direct hydrogen reduction of refractory oxide impregnated basic nickel carbonate in that the particles may be very fine, in the order of 2 microns or less, the refractory oxide is mechanically inseparable from the nickel and a substantial portion of it is fixed on the surfaces of the nickel particles.

The chromium source material employed in the process of the invention may be commercial, high purity chromium powder, oxides of chromium, chromium hydroxide or mixtures of these. Chromium powder is preferred because it blends easily with the nickel-refractory oxide particles and requires less time for reduction of oxides. Chromium powder having a particle size smaller than the particle size of the nickel-refractory oxide powder particles is preferred, although satisfactory results can be obtained with relatively coarse chromium powder having an average particle size up to 50 microns.

According to the invention, the nickel-refractory oxide powder and chromium source powder are thoroughly mixed in any conventional mixing or blending equipment to provide a substantially homogeneous powder mixture. The relative proportions of each ingredient will, of course, depend on the type of chromium source material employed and the chromium content desired in the nickel-chromium alloy product. Generally, the mixture should contain the equivalent of at least 10 percent by weight chromium, which is the minimum amount required to give some improvement in the oxidation resistance of nickel. Preferably, about 16–21 percent by weight chromium or the equivalent amount of chromium compound should be provided to ensure optimum high temperature oxidation resistance in wrought dispersion strengthened nickel-chromium alloys produced from the power product. The maximum amount of chromium is about 35 percent, which is close to the solubility limit of chromium in nickel.

The powder mixture containing the appropriate quantity of chromium or chromium compound is formed into a bed in a suitable heat resistant container which will enable the mixture to be heated in contact with a flowing dry hydrogen atmosphere. For example, shallow, elongated open boats or trays are suitable for this purpose.

The container with the powder bed is inserted into a furnace such as an indirectly heated tube furnace and is heated in a flowing atmosphere of dry hydrogen at a temperature above about 1,200°F. but below the powder mixture sintering temperature. This may be done on a batch basis or on a continuous basis, e.g. by placing the trays on a conveyor, such as a belt, which moves continuously through the furnace. Heating in flowing dry hydrogen is continued for a time sufficient to cause the chromium content of the powder mixture to diffuse into the nickel-refractory oxide powder and to reduce the amount of oxygen in excess of that in the refractory oxide to below about 0.6 wt. percent. The chromizing rate is not affected by the depth of the powder mixture bed or the rate of hydrogen flow, as long as sufficient hydrogen is available to reduce the chromium oxides in the powder mixture. Also, the hydrogen contacting the powder mixture must be pure and very dry, having a dew point below about −4°F. and preferably between about −40° and −150°F.

The actual time required for complete chromizing in any given case will depend on the chromizing temperature, the hydrogen dew point and the nature and size of the chromium source material. The chromizing rate increases with increasing temperature so, preferably, chromizing temperatures only slightly below the powder mixture sintering temperature are employed. The maximum temperature that can be employed for any nickel-refractory oxide-chromium source material powder mixture without causing sintering of the powder mixture is a function of the volume of refractory oxide in the nickel-refractory oxide constituent. For nickel-refractory oxide powders containing up to 25 volume percent refractory oxide, the relation may be expressed by the equation:

$$Tcm = -19.4/(f + 0.028) + Tm$$

where Tcm is the maximum chromizing temperature in °K, Tm is the melting point of the final nickel-chromium alloy composition and f is the volume fraction of refractory oxide in the nickel-refractory oxide powder. In some cases, where chromizing temperatures near the upper limit as determined from the foregoing equation are used, the alloy powder product may be slightly caked but it is readily comminuted to a free-flowing powder by light grinding. Preferably the chromizing operation is carried out at temperatures 50° to 300° F. below the maximum as determined from the foregoing equation. For example, in the case of nickel-refractory oxide powder containing about 3 volume percent thoria, the preferred chromizing temperature range is about 1,700° to 1,950°F.

For any given temperature, the oxide reduction reaction is much slower than the chromium diffusion reaction for chromium oxides, chromium hydroxide and very fine chromium powder, i.e. powder having a particle size below about 2 microns. This is illustrated in the following Table I.

TABLE 1

Time Required to Prepare Ni/Cr/ThO_2 Powder at 1,900° F.

Reaction	Time for Reaction at 1,900° F. (hrs.) Chromium Source			
	Fine Cr Powder (F.N.=0.9)	Medium Cr Powder (F.N.=8)	Coarse Cr Powder (F.N.=44)	Cr_2O_3
Oxides Reduction Reaction (to 0.30 wt. % O_2)	50	35	25	64
Chromizing				

Reaction (to 19 wt. % Cr)	6	50	80	20

The results show that in the case of the coarse and medium chromium powders, the overall reaction time for Ni-Cr-refractory oxide powder preparation depends primarily on the chromizing reaction rate and, in the case of fine chromium powder or Cr_2O_3, the overall reaction time depends primarily on the reduction reaction rate.

When the chromizing and oxide reduction reactions are completed, the powder is removed from the furnace, cooled in air and, if necessary, lightly comminuted to break up caked particles into free-flowing powder. Typical Ni-Cr-refractory oxide powders produced by this process are characterized by a Fisher number in the range of 1.0 to 3.5 and apparent density in the range of 1.0 – 2.0.

FIGS. 1 and 2 of the drawing, which are electron micrographs of a nickel-thoria particle and a nickel-chromium-thoria particle produced therefrom in accordance with the present invention clearly indicate the similarity in the physical characteristics of the two powders. Physically, each of the powders is comprised of clusters up to about 5.0 microns or more in size of generally spheroidal particles up to about 0.5 micron in size which have sub-micron refractory oxide particles fixed in their surfaces. In the nickel-chromium alloy powder of the invention, the spheroidal particles contain from about 10 to about 35 wt. percent, preferably about 20 wt. percent chromium, uniformly alloyed with nickel. In the preferred powders, the uniformly alloyed, spheroidal nickel chromium particles have a diameter between about 0.2 and about 0.5 micron and contain up to about 25 volume percent, preferably 0.5 to about 6.0 volume percent, of discrete refractory oxide particles between about 2 and about 50 millimicrons, preferably between 2 and 30 millimicrons in size, the major portion of which is fixed in and uniformly distributed over their surfaces. The oxygen content of the powder, exclusive of oxygen combined in the refractory oxide is below about 0.6 wt. percent and preferably below 0.01 wt. percent.

The Ni-Cr refractory oxide powders of this invention are particularly useful for fabrication of dispersion strengthened wrought nickel-chromium or nickel chromium base alloy products by powder metallurgy techniques. The powder used alone or combined with one or more alloying metals such as cobalt, molybdenum, tungsten, copper, aluminum, titanium, etc., enables the production of wrought products having greatly improved high temperature service characteristics.

The process and product of the invention is further illustrated by the following examples.

EXAMPLE 1

Nickel-thoria powder prepared in accordance with the process of Canadian Pat. No. 786.268 was de-oxidized by heating at 1,500°F. for 15 minutes in dry hydrogen. The de-oxidized nickel-thoria powder had the following characteristics:

Apparent density	1.51 gm/cc
Fisher Number	1.38
Buckbee Mears Screen Analysis (wet)	
−44 +30	10%
−30 +20	0
−20 +10	0
−10 +5	30%
−5	60%
Thoria content	2.7 volume percent
Oxygen in excess of oxygen contained in thoria	0.51%
Carbon	0.012%
Nitrogen	0.017%
Sulphur	0.0055%

The chromium source material was finely divided chromium powder having the following characteristics:

Fisher Number	8.0
O_2 wt. %	0.67
N_2 wt. %	0.021
C wt. %	0.04
S wt. %	0.03

The de-oxidized nickel thoria powder was mixed with the chromium powder in a high speed blender for 2 minutes to produce a blend containing 78 wt. percent nickel, 19.7 wt. percent chromium and 2.1 wt. percent thoria. A 400 gram sample of the powder blend was placed into a 20 inch open boat and inserted in the cooling zone of a tube furnace. The powder blend was contacted with dry hydrogen in the cooling zone for 15 minutes and then inserted into the heating zone where it was heated at 1,900°F. in a flowing H_2 atmosphere having a dew point at point of entry of −130°F. for 52 hours. The boat was removed from the furnace and cooled. The powder product was slightly caked but was easily broken up into a free-flowing powder having a Fisher number of 2.9 and an apparent density of 1.5 gm/cc. The product contained 19.7 percent chromium, 3,000 parts per million O_2 (including 2,800 ppm oxygen contained in the thoria), 80 parts per million nitrogen, 125 parts per million carbon and 35 parts per million sulphur. X-ray diffraction analyses indicated that the chromium was in a uniform solid solution with the nickel. The powder was non-magnetic confirming the X-ray diffraction results. Microscopic examination showed the powder had essentially the same appearance as the nickel-thoria starting material.

A sample of the powder was statically compacted into 60 gram billet measuring 1.25 inches × 0.2 inches. The billet was sintered in dry hydrogen, hot rolled, annealed and hot rolled a second time to produce a wrought strip. The strip was prepared into a tensile specimen which has a 2 inch gauge length and ½ inch gauge width. The specimen was heated to 2,000°F. in air and tested in tension using a strain rate of 0.025 in/per min. The results were as follows: UTS − 12,400 p.s.i., YS − 10,600 p.s.i., elongation percent − 5.

EXAMPLE 2

A sample of the nickel-thoria powder of Example 1 was blended into an aqueous slurry of chromium hydroxide. The resulting mixture was filtered and dried. 300 grams of the mixture were placed into a 2 inch diameter tube which was positioned in an indirectly heated furnace. The charge was purged with purified hydrogen until the dew point was −50°F. The tube was then placed inside the furnace and heated at a temperature of 2,100°F. The dew point was measured on the exit side of the tube. The hydrogen flow through the charge was maintained at 0.2 to 0.6 standard cubic feet per minute. After about 20 hours, the dew point of

the exit gas dropped to below −50°F. and the charged tube was removed from the furnace and cooled. The reduced product was slightly sintered but was easily comminuted to a free-flowing powder. No magnetic material was present indicating that the chromium had alloyed with the nickel during the reduction. A sample of the powder was fabricated into a wrought strip and tested as described in Example 1. The UTS at 2,000°F. was 9,100 p.s.i., YS −7,900 p.s.i. and elongation percent 3.

EXAMPLE 3

A nickel-thoria powder was prepared by adding 6,000 grams of type B carbonyl nickel powder to 4.5 liters of distilled water containing 339 grams of hydrated thorium nitrate, vigorously stirring, drying and then calcining at 1,400°F. for 3 hours in dry hydrogen. The resulting powder which contained 2.5 volume % ThO_2 was blended with chromium powder as in Example 1. The mixture was heated at 1,900°F. for 15 hours in purified dry hydrogen. The product was a free-flowing powder having 16.8 percent chromium in solid solution with the nickel. A sample fabricated and tested as in Example 1 exhibited a UTS of 7,800 p.s.i. at 2,000°F.

It will be understood, of course, that modifications can be made in the preferred embodiment of the present invention as described hereinabove without departing from the scope and purview of the appended claims.

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

1. The process for producing refractory oxide containing nickel-chromium alloy powders which comprises:
   a. providing nickel powder particles containing uniformly dispersed sub-micron refractory oxide particles at least a portion of which are fixed on the surfaces of said nickel particles;
   b. forming a mixture of said nickel particles and a finely divided chromium source material selected from the group consisting essentially of chromium metal, chromium oxide, chromium hydroxide and mixtures thereof;
   c. forming a loose bed of the resulting powder mixture and heating said bed at a temperature above about 1,200°F. but below the sintering temperature of said mixture in a flowing atmosphere of hydrogen having a dew point below about −4°F.
   d. continuing said heating for a time sufficient to effect diffusion of substantially all available chromium into the nickel particles and to reduce the amount of oxygen in the powder mixture to less than 0.60 percent oxygen in excess of the oxygen contained in the refractory oxide; and
   e. recovering the resulting refractory oxide containing nickel-chromium alloy powder product.

2. The process according to claim 1 wherein said bed is heated at a temperature, Tcm not greater than

$$-19.4/(f+0.028)+Tm$$

degrees Kelvin, where f is the volume fraction of refractory oxide contained in the nickel powder particles and Tm is the melting temperature of the final nickel-chromium alloy product the volume fraction, f, being between 0.005 and 0.25.

3. The process according to claim 2 wherein the heating temperature is from about 50°F. to about 300°F. below Tcm.

4. The process according to claim 2 wherein the powder mixture contains from about 16 to about 21 percent by weight chromium.

5. The process according to claim 1 wherein nickel powder particles are provided which contain thoria particles within the size range of about 2 to about 50 millimicrons, a major portion of which are fixed in and uniformly distributed over the nickel particle surfaces.

6. The process according to claim 5 wherein said nickel powder particles are obtained by direct hydrogen reduction of an aqueous suspension of thoria impregnated basic nickel compounds.

7. The process according to claim 2 wherein the chromium source material is high purity chromium powder having a particle size up to about 50 microns.

8. The process according to claim 2 wherein the heating is conducted in a flowing atmosphere of pure dry hydrogen.

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