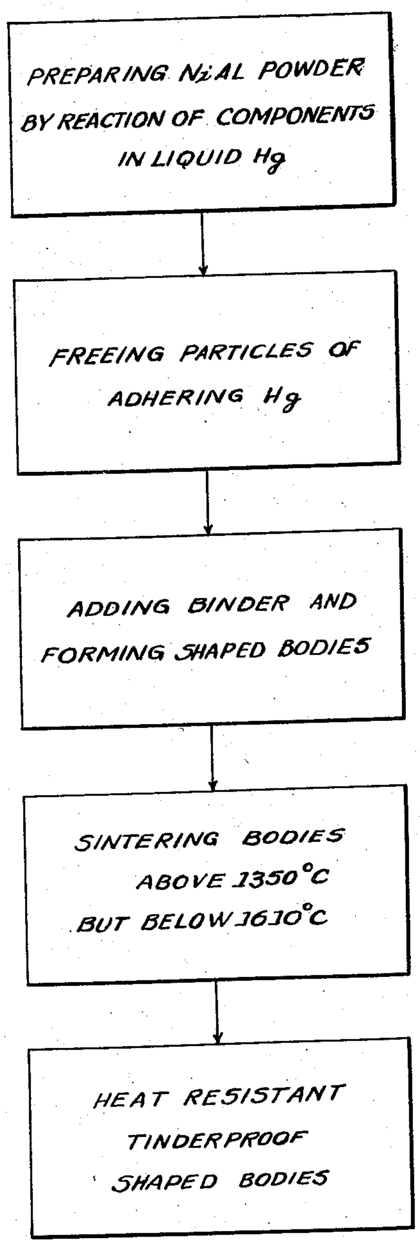
March 10, 1959 E. FITZER 2,877,113

METHOD OF PRODUCING SINTERED NICKEL-ALUMINUM ARTICLES

Filed July 2, 1956



Inventor:
Erich Fitzer.
By [signature] Atty.

United States Patent Office

2,877,113

Patented Mar. 10, 1959

2,877,113

METHOD OF PRODUCING SINTERED NICKEL-ALUMINUM ARTICLES

Erich Fitzer, Vienna, Austria, assignor to Siemens-Planiawerke Aktiengesellschaft fuer Kohlefabrikate, Meitingen, near Augsburg, Germany, a corporation of Germany

Application July 2, 1956, Serial No. 595,483

Claims priority, application Austria July 23, 1955

4 Claims. (Cl. 75—211)

This invention is concerned with a method of producing from powdered materials highly heat resisting tinderproof shaped articles based upon nickel-aluminum alloys containing aluminum from about 17–35% by weight.

Binary nickel-aluminum alloys as basis for highly heat resisting and tinderproof metallic work pieces have been proposed before. The corresponding cast and heat treated alloys exhibit at room temperature, for example, a tensile strength from 50–60 kg./mm.^{2} and at 815° C., and at elevated temperatures, tensil strength from 23–25 kg./mm.^{2}. However, these alloys have not been found to be proof against creeping. They are at higher temperatures and dependent on time relatively strongly plastically deformable.

The forming of high melting phases, for example, hard materials or oxides, into such alloys has produced greater brittleness at low temperatures, a disadvantage which is characteristic for cermets. It is of course known that such cold-brittle behavior of compound materials is essentially affected by the structural properties of the phases going into the making thereof. However, no methods whatever have become known for the carrying out of operations involved in the production of nickel-aluminum allows from powdered materials. The commonly experienced difficulties in the production of well sinterable aluminum and aluminum powder and especially in the sintering of such powder or powder mixtures point on the contrary to the advisability to avoid sintering as much as possible in the production of aluminum or aluminide work pieces.

The object of the invention is to produce nickel-aluminum alloying powders with extraordinarily superior sinter properties, adapted for easy pressure molding to form shaped bodies. The forming may be effected by the addition of metallic or non-metallic binders or auxiliary substances.

This object is according to the invention realized by producing the alloying powder by reaction of the components in liquid mercury, freeing the powder at least of most of the mercury adhering thereto, forming from the powder shaped bodies, if desired by the addition of metallic and/or non-metallic binders or auxiliary substances, and sintering the resulting bodies in a non-oxidizing atmosphere at temperatures in excess of about 1350° C. but below 1610° C.

The first step in the method according to the invention, namely, the production of the nickel-aluminide powder, is based upon the startling discovery that nickel amalgam in a liquid mercury phase with aluminum can be converted to finest mercury-free nickel-aluminum alloying powder floating in the mercury to the top. This behavior could not be expected either based upon the properties of nickel and aluminum amalgam or from the knowledge of other alloy formations in mercury. No other alloy formations in mercury have become known in which amalgam-forming elements react to mercury-free compounds as directly as in the case of nickel aluminides.

The nickel-aluminum alloying powder produced by conversion in liquid mercury, after mechanical removal of the mercury adhering thereto, can be formed directly, without any further mechanical treatment thereof, to produce shaped bodies, if desired by adding thereto metallic or non-metallic auxiliary substances or binders, and the shaped bodies can be sintered in a non-oxidizing atmosphere at temperatures exceeding about 1350° C. and below 1610° C.

It has been found that nickel-aluminum alloying powders are obtained, in the conversion in liquid mercury, in the particle size which is most favorable for high temperature work pieces, by reacting nickel amalgam with aluminum or aluminum amalgam at temperatures exceeding about 370° C., preferably about 450° C. to about 750° C. After cooling, the floated nickel aluminide powder, is freed of mercury by filtration or decanting.

It has further been found that nickel aluminide powder with NiAl-phase is directly produced, in the described treatment, only when the corresponding initial materials are processed in a liquid mercury phase by heating them to about 500–700° C., followed by cooling, and then reheating a second time to about 500–700° C. In a single heating, there will be produced primarily an $NiAl_3$-phase and nickel powder. It is striking that the $NiHg_4$-phase started as a nickel amalgam does not revert in any case upon the cooling. This discovery is of great technical importance because the mechanical separation of $NiAl_3$ and of the nickel powder, from the adhering mercury, can be carried out without distilling of the latter. The separation may be quantitative, by decanting or filtering, if desired by slushing in water, alcohol or other liquids. It has been found satisfactory in many cases to form the desired bodies of the nickel-aluminide powder containing from about 5–15% adhering mercury residue and to remove such mercury residue by vaporization during the sintering. Important for the method according to the invention is the good sinterability of the nickel-aluminide powder obtained in the described first step thereof. As mentioned before, the sintering requires temperatures exceeding about 1350° C. in a non-oxidizing atmosphere. Sinter temperatures must in no case exceed 1610° C.

The forming of the desired bodies from the nickel-aluminide powder produced according to the invention is particularly facilitated by enriching such powder with aluminum and using thickly viscous nickel amalgam as a binder. The mercury is during the sintering vaporized from the added amalgam, the remaining nickel serving as added metal for the forming of an excellently sintering final alloy which is richer in nickel than the nickel-aluminide powder employed.

A similar particularly good and quick sintering is obtained by using nickel-aluminide powder consisting of $NiAl_3$ and Ni-phase. However, in such case, the first sintering must not be carried out at temperatures exceeding 1400° C.; a higher sinter temperature can be applied only after about ½ hour annealing at such lower temperature.

It has proved of advantage to use oxydic binders in the working of the nickel-aluminide powders prepared according to the invention. The formation can be carried out in accordance with the known dross procedure. Hydrolyseable silicic acid compounds, preferably silicic acid ester may be used as binder substance in the forming of the desired bodies. After the sintering there will in such cases remain silicic acid skins between the individual aluminide particles. It was surprising to find that the sintering of the aluminide particles is not completely stopped. Only sinter bridges are obviously formed between the individual nickel-aluminide particles. The remaining fine silicic acid skins constitute a high melting embedded phase and increase considerably the resistance against creepage of the shaped bodies made according to the invention at high temperatures exceeding about 800° C. The metallic character and therewith many properties such, for example, as conductivity is, however, considerably changed by such embeddings. The SiO_2 content may go as high as 10%.

A method has for this reason been devised for partially or totally removing, during the sintering, the silicic acid added as a binder in the form of hydrolyseable compounds. This is accomplished by addition of silicon in the form of elementary powder or in the form of high melting disilicides, these materials forming with the silicic acid during sintering above 1400° C. volatile silicon suboxyde. The amount of added silicon should correspond of the amount of the silicic acid to be volatilized. Other oxydic binder materials may be used, for example, alumina in colloidal suspension.

All sinter bodies produced according to the invention have the NiAl-phase. As is known, such phase is by itself very brittle. The incomparably lower brittleness of the sinter bodies according to the invention, as compared with cast alloys of like composition, may be traced back to the formation of a skeleton of finest NiAl particles. The improved heat resistance and resistance to creeping may likewise be traced to this NiAl skeleton. It is possible that these properties are (even in the absence of oxydic material) due to the presence of submicroscopic oxyde skins, just as in the case of known heat resisting sinter aluminum. This skin may in the course of the method according to the invention be reinforced by slight anoxidation of the nickel-aluminide powder. Of course, such pretreated powder cannot be worked to form shaped bodies without one of the known or above described sinter expedients.

A sinter alloy containing in addition to the NiAl skeleton a Ni_3Al binder phase has been found particularly useful.

The low specific weight of the sinter bodies according to the invention may be particularly noted in addition to good heat resistance, tinder proofness and resistance to creeping thereof. These features impart to fast rotating structural parts made from the material qualities which are superior to those of structural parts made of cast high temperature alloys.

The principal steps of the method according to the invention are indicated in the accompanying drawing.

Changes may be made within the scope and spirit of the appended claims.

I claim:

1. A method of making shaped heat resisting and tinderproof articles of nickel-aluminum compounds containing from about 17% to about 35% aluminum by weight, comprising reacting the components, nickel and aluminum, in liquid mercury at temperatures between 370° C. and 750° C. to produce an alloyed nickel-aluminum compound powder floating in the mercury, freeing said alloyed powder of the major part of mercury adhering thereto, working said powder to form said articles therefrom, and sintering said articles in a non-oxidizing atmosphere at temperatures above 1350° C. but below 1610° C.

2. A method according to claim 1, comprising reacting nickel amalgam with aluminum in a liquid mercury phase at temperatures between 450° C. to 750° C. to produce said powder, said powder floating on said mercury after cooling, and separating said floating powder from the mercury.

3. A method according to claim 1, for directly producing alloyed powder with NiAl phase, comprising converting the initial components, nickel and aluminum, in a mercury phase at a temperature between 500° C. and 700° C. followed by cooling and reheating to like temperature, said powder floating on said mercury, and separating said floating nickel-aluminum powder from the mercury.

4. A method according to claim 1, comprising working said powder with nickel amalgam as a binder to form shaped articles therefrom, residual mercury vaporizing during said sintering and leaving additional nickel, the resulting shaped sintered articles having a nickel content which exceeds that of the alloyed powder.

References Cited in the file of this patent

UNITED STATES PATENTS

1,599,618	Koehler	Sept. 14, 1926
1,607,245	Dhume	Nov. 16, 1926
2,156,002	Cooper	May 2, 1939
2,491,866	Kurtz	Dec. 20, 1949
2,620,555	Lenz	Dec. 9, 1952
2,751,668	Turner et al.	June 26, 1956