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STABILIZED NICKEL POWDER

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This invention relates to finely divided nickel.

Finely divided nickel may be produced by a method disclosed in U. S. Patent No. 1,893,879, granted January 10, 1933, on an application filed by the present applicant and J. C. W. Frazer. In accordance with the invention of that patent a nickel amalgam is subjected to vacuum distillation, in the absence of gas reactive with nickel, to completely evaporate the mercury and leave the nickel as a residue. Nickel thus produced possesses unusual properties. For example, the particle size is so small that the resultant nickel powder will smear on glass much in the manner that talc does. An outstanding property of such nickel is that it is exceptionally reactive relative to finely divided nickel produced in other ways.

Nickel produced in this way from a nickel amalgam is particularly useful for the production of exothermic compositions, and particularly fuse powders. For instance, high speed fuse compositions composed of this form of nickel and sulfur, with or without an inert diluent to regulate the burning speed, are disclosed in Patent No. 2,434,067, granted January 6, 1948, on an application filed by J. C. W. Frazer and the present applicant.

This form of nickel is especially useful in the production of delay fuse compositions. Particularly desirable compositions of this type are disclosed in a co-pending application, Serial No. 469,978, filed December 24, 1942, now Patent No. 2,440,579, granted April 27, 1948, by J. C. W. Frazer and the present applicant, and assigned to the assignee of the present application. In accordance with that invention delay compositions are composed of nickel of the aforesaid origin and an oxidizing agent such as a per-salt of an oxygen-containing acid, most suitably potassium perchlorate, with or without an inert diluent, such as diatomaceous earth. Nickel in this form is useful also with other types of delay fuse compositions, for example those disclosed in co-pending application Serial No. 600,415, filed June 19, 1945, by the present applicant and J. Dubin, now U. S. Patent No. 2,457,860 and likewise owned by the assignee of the present application. The compositions of that co-pending application comprise this form of nickel together with finely divided zirconium, an oxidizing agent adapted to oxidize those metals at a high rate of speed, for example an alkali metal perchlorate, and an oxidizing agent adapted to oxidize them at a slow rate of speed, for example barium chromate.

A particular feature of such compositions is

that their burning times may be varied not only by variation of the oxidizing agent used but also by variation in the proportions of oxidizing agent to metal and of inert diluent, when used. Those compositions also are characterized by the fact that they burn without production of gaseous products of combustion to form solid residues so that the fuses need not be vented to the atmosphere, which is recognized as highly desirable for ordnance purposes.

Nickel produced in accordance with Patent No. 1,893,879 is so reactive that it is pyrophoric in nature, i. e., it ignites upon exposure to air and other oxidizing gases. However, if before contact with oxygen the nickel powder is contacted with carbon dioxide it may be handled freely thereafter in air without danger of ignition so that it can then be used for various purposes, as for the compounding of the foregoing fuse compositions.

Extended experience has shown that if fuse compositions of the kinds alluded to are made from such carbon dioxide-treated nickel promptly or within a short time after its production, the burning time of the compositions changes progressively upon storage, which is obviously undesirable because of the requirement that a delay fuse burn within a rigidly defined period of time. On the other hand, experience has shown likewise that if after being treated with carbon dioxide the finely divided nickel be stored for a substantial length of time before use, fuse compositions made from it retain their predetermined burning time, at least within the permissible variation limitation. This necessity for aging the nickel powder before using it to compound fuse compositions, in order to supply compositions of reliably sustained predetermined burning time, is objectionable in that it requires the carrying of greater stocks of nickel than would otherwise be necessary, with attendant cost, coupled with the inability to supply fuse compositions promptly after production of the nickel powder. It is undesirable also because it is by no means certain that the aging of every batch of amalgam-produced and carbon dioxide-treated nickel for a given length of time will result in complete aging.

The primary object of the present invention is to provide a method of treating nickel produced by evaporation from nickel amalgam to render it, and compositions containing it, insensitive to alteration upon storage, which is simple, inexpensive, easily practiced, and productive of nickel powder that is stabilized against oxidation and is useful for making fuse and other compositions

that reliably maintain over long intervals of time predetermined burning rates.

Other objects will appear from the following specification.

I have discovered, and it is upon this that the present invention is predicated, that the necessity for aging nickel produced by the vacuum distillation of mercury from a nickel amalgam as described in Patent No. 1,893,879, and treated with carbon dioxide prior to contact with oxygen to render it non-pyrophoric, can be eliminated by the simple expedient of heating the carbon dioxide-treated nickel in air, or a similar oxidizing gas, for a short period of time.

In the practice of the invention the nickel after treatment with carbon dioxide, and as promptly thereafter as may be desired, is placed in a container to which air has free access, and the powder is then heated. The exact temperature of heating is not material to the efficacy of the treatment provided the powder does not reach a temperature at which it will ignite while being so heated. The heating is continued for a time, dependent upon the applied temperature, to render the nickel stable against further oxidation upon storage. The higher the temperature, with the limitation just noted, the more rapid will be the stabilization of the nickel against further change. Thus, the stabilizing effect will be produced more rapidly at 200° than at 100° C. I find that the stabilization is effected rapidly and without damage of ignition of the nickel by heating for about 30 minutes at 300° C., which constitutes the preferred practice of the invention.

Heating for a longer time than necessary to stabilize the nickel against further change is merely uneconomical.

Extended experience has shown that fuse compositions made from nickel treated in the manner just described, for example compositions in accordance with the inventions of the aforesaid copending applications and Patent No. 2,434,067 do not undergo alteration of burning time, at least within permissible ordnance limits, upon prolonged storage, and thus the necessity for aging such amalgam produced nickel prior to this invention is eliminated and predetermined and sustained burning times are more reliably attained. Moreover, the compositions remain unaffected by extremes of temperature and humidity experienced in transportation and storage in temperate and tropical climates.

While I do not commit myself to this or any other theory, I now believe that the alteration in burning time alluded to above as occurring with fuse compositions made from this finely divided nickel promptly after its production is due to a slow, progressive oxidation of the nickel, and further that the heating with free access of oxygen in accordance with the present invention promptly causes the nickel to be oxidized to the same extent so that it does not undergo any fur-

ther oxidation, or at least to any detrimental extent, upon long continued storage. Queerly enough, the exceptional reactivity that makes this form of nickel so desirable for fuse compositions is not depreciated materially.

According to the provisions of the patent statutes, I have explained the principle of my invention and have described what I now consider to represent its best embodiment. However, I desire to have it understood that, within the scope of the appended claims, the invention may be practiced otherwise than as specifically described.

I claim:

1. That method of stabilizing nickel produced by evaporation of mercury from a nickel amalgam which comprises contacting the said nickel with carbon dioxide prior to contact with oxygen, and then heating it in a gas containing free oxygen at a temperature below that at which the nickel ignites while being so heated.

2. That method of stabilizing nickel produced by evaporation of mercury from a nickel amalgam which comprises contacting the said nickel with carbon dioxide prior to contact with oxygen, and then heating it in air at a temperature below that at which the nickel ignites while being so heated for a time to stabilize it against further substantial oxidation upon long continued storage.

3. A method according to claim 1, said nickel being heated between about 200° and about 300° C.

4. A method according to claim 2, said nickel being heated at about 300° C.

5. That method comprising the steps of subjecting a nickel amalgam to vacuum distillation in the absence of gas reactive with nickel to evaporate mercury therefrom and leave a residue of nickel in finely divided form that is highly reactive with oxygen, then, prior to contact with oxygen, contacting the said nickel residue with carbon dioxide, and then heating the nickel in air at a temperature below that at which the nickel ignites when so heated, and thereby stabilizing the nickel against further substantial oxidation by atmosphere oxygen upon storage.

OWEN G. BENNETT.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
1,001,279	Kayser	Aug. 22, 1911
1,127,911	Morey	Feb. 9, 1915
1,987,660	Bennett et al.	Jan. 15, 1935
2,205,552	Arnold	June 25, 1940

FOREIGN PATENTS

Number	Country	Date
4,702	Great Britain, 1912	May 26, 1913