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(54) **Process for preparing nickel film.**

(57) An amorphous, non-magnetic film of nickel is prepared by reducing an aqueous solution of a nickel compound simultaneously with a mixture of a phosphorus containing reducing agent and a boron containing reducing agent. The film is exceptionally stable and retains its amorphous, non-magnetic state when heated to a temperature of 300°C.

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PROCESS FOR PREPARING NICKEL FILM

This invention relates to a process for preparing nickel film. In particular, it is concerned with a process for preparing a nickel film which has increased stability and is non magnetic.

In the past many metals have been prepared by reducing an aqueous solution of a salt. Nickel has been prepared by reducing an aqueous solution of a nickel compound with a hypophosphite reducing agent. Nickel has also been prepared by reducing an aqueous solution of a nickel compound with an amine borane reducing agent. Nickel, produced by either of these processes, has a metastable non-crystalline form, ie it has a tendency to lose its amorphous state and crystallise when heated.

Crystallisation is associated with three changes in properties which may be harmful. Firstly, the volume generally contracts. This promotes cracking and loss of adhesion. Secondly, precipitation of second phases is harmful in that it increases the susceptibility of the metal to galvanic corrosion. Thirdly, the remaining crystalline metal matrix becomes ferromagnetic.

The invention seeks to provide a nickel film which retains its non-magnetic, amorphous state when heated to high temperatures.

US-A-3,567,525 discloses use of a mixture of two reducing agents, one a phosphorus containing agent and one a boron containing agent, for the reduction of nickel. The conditions are such, however, that the process produces a material, which is strongly ferrogmagnetic and in powder form.

A process for preparing a continuous film of nickel, is characterised, according to the invention, by reducing an aqueous solution of a nickel compound in the substantial absence of any magnetic material with a mixture of a phosphorus containing reducing agent and a boron containing reducing agent in amounts so that the film retains its amorphous non-magnetic state when heated to a temperature of 300°C.

When a mixture of reducing agents is used according to the present invention, the resulting nickel film has a greater stability than that obtained when only a single reducing agent is used. The rate of deposition of the nickel film is also increased. The film retains its amorphous, non-magnetic state when heated to a high temperature.

The increased ability of the nickel to withstand high temperature and still be amorphous and non-magnetic greatly increases its usefulness in a wide variety of end uses. For example, the non-magnetic property is very important and desirable when the nickel is used as an undercoat on a substrate for a thin film magnetic record disk.

In carrying out the process of the present invention, it is advantageous to use the hypophosphite reducing agent and the amine borane reducing agent in a ratio of about 10 equivalents to 1 equivalent. The preferred hypophosphite reducing agent is sodium hypophosphite, and the preferred amine borane reducing agent is dimethylamine borane.

The relative amounts of the phosphorus containing reducing agent and the boron containing reducing agent should be such that the final product is a non-magnetic nickel containing from 10% to 15% by weight phosphorus and from 0.1% to 1% by weight boron. It is only within these critical ranges that the nickel films maintain their low level of stress and their non-magnetic character when heated.

It should be emphasised that the present invention yields nickel deposits which are continuous films, and not layers of powder as is obtained in much of the prior art. These continuous films are also adherent to the substrate.

Since the object is to form a non-magnetic film, the reduction should be carried out in the substantial absence of any magnetic materials, ie the process and composition limits would be greatly affected by the presence of more than trace amounts of magnetic materials such as iron, cobalt and manganese.

Example

An aqueous solution of nickel sulfate containing 7.0 grams of nickel per litre was prepared. The pH was 4.5. Sodium hypophosphite in an amount of 45 grams per litre and dimethylamine borane in an amount of 2.15 grams per litre were added at a temperature of 91°C. A continuous adherent film of nickel which contained 12% phosphorus and 0.5% boron was precipitated out. This nickel film remains amorphous and non-magnetic when heated to much higher temperatures than those sufficient to crystallise nickel made by a similar process in which only one of the reducing agents is used. Samples prepared according to the present invention can be heated to 300°C and still not become magnetic.

CLAIMS

1. A process for preparing a continuous film of nickel, the process being characterised by reducing an aqueous solution of a nickel compound in the substantial absence of any magnetic material with a mixture of a phosphorus containing reducing agent and a boron containing reducing agent in amounts so that the film retains its amorphous non-magnetic state when heated to a temperature of 300°C.

2. A process as claimed in claim 1, in which the reducing agents are an amine borane compound and a hypophosphite.

3. A process as claimed in claim 2, in which the ratio of hypophosphite reducing agent to amine borane reducing agent is about 10 equivalents to 1 equivalent.

4. A process as claimed in claim 2 or claim 3, in which the hypophosphite reducing agent is sodium hypophosphite and the amine borane reducing agent is dimethylamine borane.

5. An amorphous, non-magnetic nickel film containing from 10% to 15% by weight phosphorus and from 0.1% to 1% by weight boron when prepared by a process as claimed in any preceding claim.

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EUROPEAN SEARCH REPORT

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DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
X	PATENT ABSTRACTS OF JAPAN, unexamined applications, field C, vol. 6, no. 214, october 27, 1982 THE PATENT OFFICE JAPANESE GOVERNMENT page 164 C 131 * Kokai-no. 57-120 664 (MATSUSHITA DENKI SANGYO K.K.) *	1,5	C 23 C 18/36 C 23 C 18/52
X	SOVIET INVENTIONS ILLUSTRATED, section Ch, week C 17, June 4, 1980 DERWENT PUBLICATIONS LTD., London M 13 * SU-681 111 (AS LITH CHEM TECHN) *	1	
X	US - A - 4 152 164 (M. GULLA et al.) * Column 1, lines 45-65 *	1,2,4	TECHNICAL FIELDS SEARCHED (Int. Cl.4) C 23 C
X	DE - A - 2 028 950 (SHIPLEY COMPANY INC.) * Claims 1,2,11-13,22-25, 32-34 *	1,2,4	

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
VIENNA	29-10-1984	SLAMA

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
Y : particularly relevant if combined with another document of the same category
A : technological background
O : non-written disclosure
P : intermediate document

T : theory or principle underlying the invention
E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons

& : member of the same patent family, corresponding document

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