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Electroplating Condition for Ni Nanocrystalline on Metal (Ni and Cu) Plates

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1. Introduction

Ni-63, a beta radiation source, is prepared by the electrical deposition of radioactive Ni-63 ions on a thin non radioactive nickel foil or Cu plate. Given a half life of 100 years, a nuclear battery will still produce half of its initial starting power after 100 years. A speck of a radioisotope like nickel-63, for example, contains enough energy to power a nano-nuclear battery for decades, and to do so safely [1-2].

Ni-63 plating is similar to other electroplating processes that employ soluble metal anodes. It requires the passage of a direct current between two electrodes that are immersed in a conductive, aqueous solution of nickel salts. The charged Ni ions are formed by sulfate, sulfamate, chloride, and a Watts bath. However, the charged Ni-63 ions are formed by dissolving metal Ni-63. To established the coating condition of Ni-63, non radioactive metal Ni particles are dissolved an acid solution and electroplated on a Ni sheet. The nickel plating process is used extensively for decorative, engineering and electroforming purposes because the appearance and other properties of electrodeposited nickel can be varied over a wide range by controlling the composition and operating parameters of the plating solution. A continuous increase in the grain size versus current density has also been recognized in the direct current electrodeposition of nickel coating. On the other hand, Aruna et al. [3] reported that the current density has no significant effect on the grain size of nickel electrodeposits. A review of the literature shows that saccharin has often been added to a nickel plating bath since the 1980s to improve the ductility and brightness, and in later periods as a grain refiner agent.

In the present paper, not only the preparation of a Ni-plating solution prepared by dissolving metal particles, but also an optimization of the deposition conditions, such as the current density, saccharin concentration in the bath, and different metal substrates were investigated. The proposed model can also be applied for radioactive Ni-63 electroplating.

2. Methods and Results

2.1 Electroplating of Ni

Nickel (Ni) coatings were deposited by direct current (DC) electroplating at a current density at 1 to 40 mA/cm^2. The basic composition of the bath was 0.2 M Ni, 0.4 M boric acid (H_3BO_3). Ni metal powders were dissolved in mixed HCl and distilled water. Boric acid is used in the nickel plating solutions for buffering purposes. The pH of the bath was adjusted to 4.0 ± 0.2 by the addition of drops of KOH (1 N).

A nickel sheet (99.99%, Aldrich) with dimensions of 10 mm × 20 mm × 0.27 mm, and a Cu plate (99 %, Nialco) with dimensions of 10 mm × 20 mm × 0.125 mm was used as cathode (substrate), and Pt coated Ti mesh with dimensions of 25 mm×135 mm×1 mm as anode materials. The deposition time was adjusted to achieve an average thickness of 30 μm based on Faraday's law [4]. The experiments were carried out in the baths containing amounts (2 g/l) of sodium saccharin as a grain refiner. The microstructure of the coatings was studied by scanning electron microscope (SEM), and X-ray diffraction (XRD). XRD investigations were carried out using a Philips X'Pert-Pro instrument operated at 40 kV and 30 mA with a CuKα radiation (λ=1.5418 Å).

2.2 Effect of substrate

Nickel deposition was produced at a current density of 1, 5, 10, 15, 20, and 30 mA/cm^2, a bath temperature of 27 °C, and pH=4. Saccharin was added up to 2 g/l as a grain refiner. XRD patterns showed that the crystal structure of the coating is pure fcc nickel, and no characteristic peaks of other phases have been recorded.

The crystal orientation of the films was estimated by a degree of high (200) Ni orientation in the XRD patterns. Ni films prefer a plane orientation. From the peak broadening of XRD patterns, by means of a Scherrer relationship, the average crystalline size calculated from the XRD line broadening of the (200) peak, using the classical Scherrer relationship[3], $D(h\ k\ l) = k\lambda/B \cos\theta$, where D(h k l) is the particle diameter, k is the constant (shape factor) with a value of 0.9, B is half of the maximum line width, and λ is the wavelength (λ = 1.5418 Å), were determined. The size of the deposited particles was at or below 70 nm. The smallest size of the particles was 25 nm, which was formed at 10 mA/cm^2 of current density.

Fig. 1 shows the results of scanning electron microscopy (SEM) for the Ni coated Ni sheet, and deposted Cu sheet at a current density of 10 and 20 mA/cm^2. The results showed that the average grain sizes were 25 nm and 40 nm for Ni coating on Ni and on Cu plates, respectively. Figs. 1(a), and 1(b) show SEM images for electrodeposited Ni on a Ni sheet at a urrent density of 10 and 20 mA/cm^2, respectively. The particles on the Ni sheet were formed as in a spherical shape. In Figs. 1(c) and 1(d), electroplating Ni on a Cu plate was observed at current densities of 10 and 20 mA/cm^2, respectively. The particles on the Ni sheet were formed as needle-like shapes. The substrate affects the formation of their shape of particles.

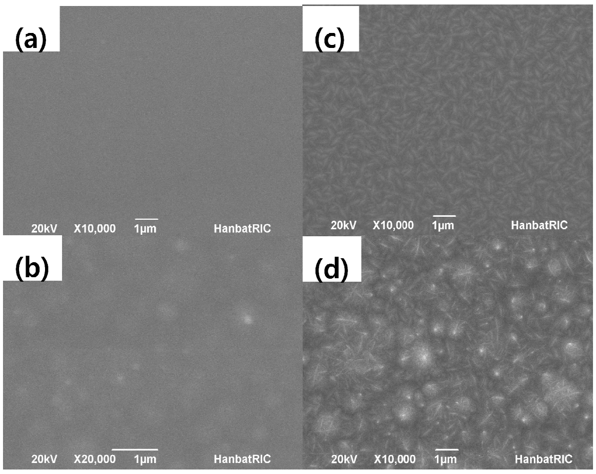

Fig. 1. SEM images for the surface of Ni coated on the Ni plate at a current density of (a) 10 and (b)20 mA/cm^2, and Ni deposited on Cu plate at a current density of (c)10 and (d) 20 mA/cm^2.

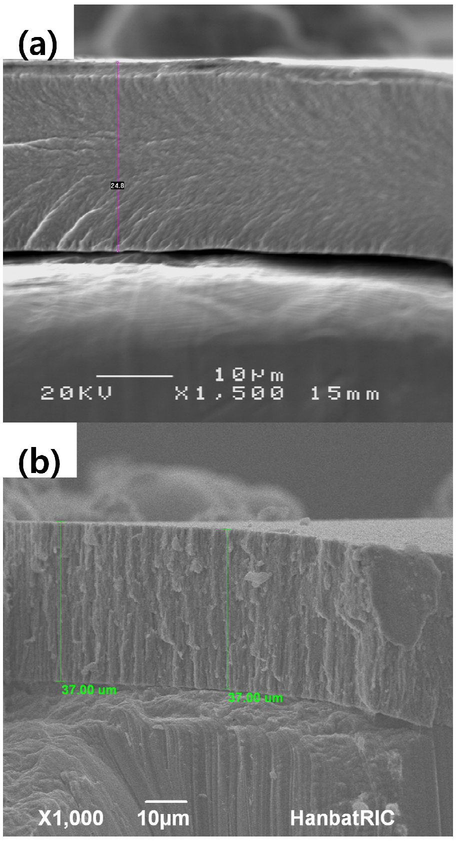

Fig. 2 SEM images for the thickness of coating layer of Ni on (a) Ni sheet and (b) Cu plate at the current density for 10 mA/cm^2 in a bath adding saccharine.

Fig. 2 shows SEM images for the thickness of the Ni coating layer at same current densities and on a different substrate. The deposition time was adjusted to achieve an average thickness of 30 µm based on Faraday's law [4] as below;

$$\mathbf{T\ (cm)} = \frac{\mathbf{t \times I \times MW}}{\boldsymbol{\rho} \times \mathbf{valance} \times \mathbf{faraday\ constant} \times \mathbf{A}} \qquad (1)$$

where T is the thickness to be deposited, t is the time of the deposition, I is the current, MW and ρ are the molecular weight and density of Ni, and A is the area of the film. Estimated time to reach 30 µm in thickness was determined to be 8,786 s at a current density of 10 mA/cm^2. The thickness of the Ni layer on a Cu substrate is well matched with the theoretical thickness. However, the coating layer on a Ni substrate is insufficient to be 30 µm, because of different cathode size (substrate), from 92 to 97%. The thickness of the Ni layer was 24.8 and 37 µm for depositing on Ni and Cu plates, respectively.

3. Conclusions

A nanocrystalline Ni coating was synthesized by direct current electroplating at a current density of 10 mA/cm^2 and pH=4. The basic composition of the bath was 0.2 M Ni ions, which was prepared by dissolving Ni metal particles in HCl. The effect of the substrates was investigated by an SEM technique. The substrate affected not only formation of their shape of particles, but also the thickness to be deposited..

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