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The Effect of Current Density and Saccharin Addition on The Grain Size of Nickel Coatings

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1. Introduction

Recently, the main advantage of a radioisotope "fuel" is concentrated, because it is "burned" at the rate of the isotopes half life [1]. In other words, given a half life of 100 years, a nuclear battery would 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]. Ni-63, a beta radiation source, is prepared by electrical deposition of radioactive Ni-63 ions on thin non radioactive nickel foil. 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 establish the coating condition of Ni-63, non radioactive metal Ni particles were dissolved in an acid solution and electroplated on the Ni sheet.

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. [2] 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 [3]. In the present paper, not only the preparation of the Ni-plating solution prepared by dissolving metal particles but also an optimization of the deposition conditions, such as the influence of current density and saccharin concentration on the grain size, was 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 of 1, 5, 10, 15, 20, 30, and 40 mA/cm^2. The basic composition of the bath was 0.2 M Ni, 25 g/l boric acid (H_3BO_3). Ni metal powders were dissolved in mixed HCl and distilled water. Boric acid is used in a nickel plating solution 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 (Aldrich) of 99.99% purity with dimensions of 10 mm × 20 mm × 0.125 mm was used as a cathode (substrate), and Pt coated Ti mesh with dimensions of 25 mm×135 mm×1 mm as an anode. The deposition time was adjusted to achieve an average thickness of 3 μm based on Faraday's law [14]. The experiments were carried out in the baths containing 0-5 g/l of sodium saccharin as a grain refiner. The microstructure of the coatings was studied by a 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 CuKα radiation (λ=1.5418 Å). The average grain size of the nanocrystalline nickel coatings was calculated from XRD patterns using a modified Scherrer relationship expressed in Ref [4].

2.2 Grain size of Ni coated on Ni sheet

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. XRD patterns showed that the crystal structure of the coating is pure fcc nickel and with no characteristic peaks of other phases. The crystal orientation of the films was estimated based on the degree of high (200)Ni orientation in the XRD patterns. Ni films preferred a plane orientation. From the peak broadening of the 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; $D(h\ k\ l) = k\lambda/B \cos\theta$, 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 37 nm, which was formed at a current density of 10 mA/cm^2.

Fig. 1 shows the results of scanning electron microscopy (SEM) for the Ni coated Ni sheet at a current density of 1, 5, 10, 15, 20, and 30 mA/cm^2. The average particle size was decreased with an increase in current density to 10 mA/cm^2. The shape of the particles was changed from needle-like to a spherical type, as the current density increased. Figs. 1(a), 1(b), and 1(c) describe SEM images observed at different magnification for electrodeposited Ni on a Ni sheet at a current density of 1, 5, and 10 mA/cm^2, respectively. The minimum particle size was observed at a current density of 10 mA/cm^2. The particle size became lager as current density was increased at or above 15 mA/cm^2.

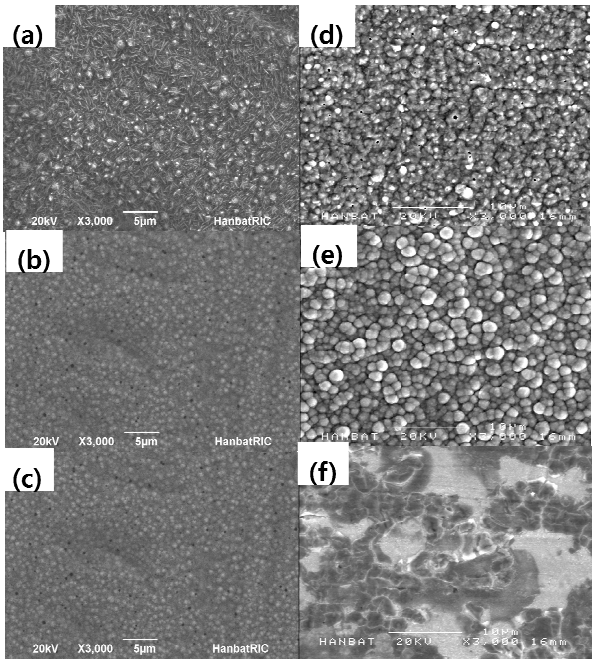

Fig. 1. SEM images for the Ni coated Ni sheet at a current density of (a) 1, (b) 5, (c)10, (d)15, (e)20, and (f)30 mA/cm^2.

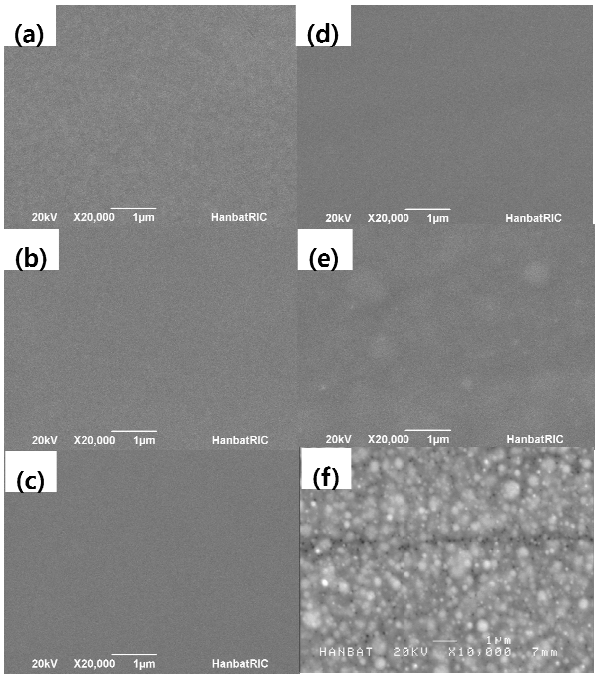

Fig. 2 SEM images for the Ni coated Ni sheet at a current density of (a) 1, (b) 5, (c) 10, (d) 15, (e) 20, and (f) 30 mA/cm^2 in a bath with saccharine (2 g/l).

Figs. 1(c), 1(d), and 1(e) show SEM images observed at different magnification for electrodeposited Ni on a Ni sheet at a current density of 15, 20, and 30 mA/cm^2, respectively. Particles on the Ni plate were formed as in a spherical shape, as shown in Fig. 1(c) and 1(d). In Fig. 1(e), electroplating of Ni was impossible at and above a 30 mA/cm^2 current density due to salts in the solution, also and was coated on the Ni substrate.

Fig. 2 shows SEM images for the Ni coated Ni sheet at the same current densities and different bath conditions when adding saccharine (2g/l) into the solution. The average particle size was decreased with an increase in current density to 15 mA/cm^2. Figs. 2(a), 2(b), 2(c), 2(d), 2(e), and 2(f) describe SEM images observed at different magnification for electrodeposited Ni on a Ni sheet at a current density of 1, 5, 10, 15, 20 and 30, mA/cm^2, respectively. The particles on the Ni sheet were formed in a spherical shape. The average size of the particles was observed as independent of the current density. In Fig. 2(f), electroplating Ni was impossible at and above a 30 mA/cm^2 current density due to co-deposited salts in the solution on the Ni substrate. Further increase in saccharin concentration had no significant effect.

3. Conclusions

Nanocrystalline nickel(Ni) coating was synthesized by direct current electrodeposition at a current density at 1 to 30 mA/cm^2, and pH=4. The basic composition of the bath was 0.2 M Ni ions, which was prepared by dissolved Ni metal particles in HCl. The effect of current density saccharin addition (0–5 g/l) on the average grain size of the deposits was investigated by XRD and SEM techniques. The results showed that the surface roughness decreased as the saccharin concentration increased to 2 g/l, while further increase in saccharin concentration had no significant effect. The experimental results showed that the increases in the current density had a considerable effect on the average grain size of the deposits.

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