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Magnetic Properties and Residual Stress of electroplated Ni

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1. Introduction

A speck of a radioisotope such as 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 a thin non-radioactive nickel foil. Ni-63 plating is similar to other electroplating processes that employ soluble metal anodes. The charged Ni-63 ions are formed by dissolving metal Ni-63. To establish the coating conditions for Ni-63, non-radioactive metal Ni particles are dissolved in an acid solution and electroplated onto a Ni sheet. A continuous increase in the particle size versus the current density has also been recognized in the DC electrodeposition of nickel coating [2-3]. The Ni metal is magnetic materials. The saturation of magnetizations for the perpendicular and the parallel direction are influenced by crystalline easy direction [4]. In this research, a plating film with a face centered cubic (fcc) structure was obtained [3-5]. At the same time, their thickness dependent crystalline easy direction and magnetic properties were investigated by main peak intensity of the X-ray diffraction (XRD) and saturation magnetization. Also, the current density dependent of the residual stress was estimated by deposit analyzing system during electroplating Ni. The proposed model can also be applied for radioactive Ni-63 electroplating.

2. Experimental Technique

Nickel (Ni) coatings were deposited by DC electroplating at current densities of 10, 15, 20, and 25 mA/cm^2. The basic composition of the bath was 0.2 M Ni and 25 g/l of boric acid (H_3BO_3) A nickel sheet of 99.99 % purity with dimensions of 10×20×0.125 mm^3 was used as a cathode (substrate) and a Pt-coated Ti mesh with dimensions of 25×135×1 mm^3 as an anode. A Ni sheet with a high purity of 99.99 % (Aldrich) was used as the substrate. The deposition time was adjusted to achieve an average thickness of 3 μm based on Faraday's law [3]. The microstructure of the coatings was studied by scanning electron microscopy (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 CuKa radiation (k = 1.5418 Å). The saturation magnetizations for the perpendicular and in-plane were measured by vibrating samples magnetometer (VSM). The residual stress between substrate (Cu-plate) and coating layer was measured by deposit stress analyzer (Model 683EC). Figure 1 represents deposit stress analyzer and Cu-strip substrate.

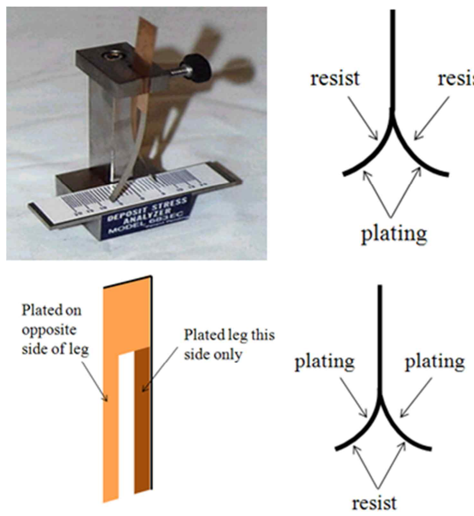

Fig. 1. The equipment of bent strip.

3. Results and discussion

Figure 2(a) shows the XRD patterns of nickel coatings with different thickness produced at current density of 10 mA/cm^2, a bath temperature of 27 °C, and pH 4. It can be observed that the crystal structure of the coating is pure fcc nickel and no characteristic peaks of other phases were recorded. Crystal orientations of the films were estimated by the high degree (200) in the XRD patterns, as shown in Figure 2(a). The main peak of the bulk Ni is presented generally at (111). However, the plane orientation on the substrate was formed, because of the impression of the seal for the production of the foil. The (200) peak strength was decreased as the coating layer was increased. Figure 2(b) shows SEM images for the thickness of the Ni coating layer at same current density. The deposition time was adjusted to achieve an average thickness based on Faraday's law [3] as below;

$$\mathbf{T\ (cm)} = \frac{\mathbf{t \times I \times M\,W}}{\boldsymbol{\rho} \times \mathbf{valence} \times \mathbf{faraday\ \ constant} \times \mathbf{A}} \qquad (1)$$

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 6 μm in thickness was determined to be 1,757 s at a current density of 10 mA/cm^2. The thickness of the Ni layer is matched with the theoretical thickness.

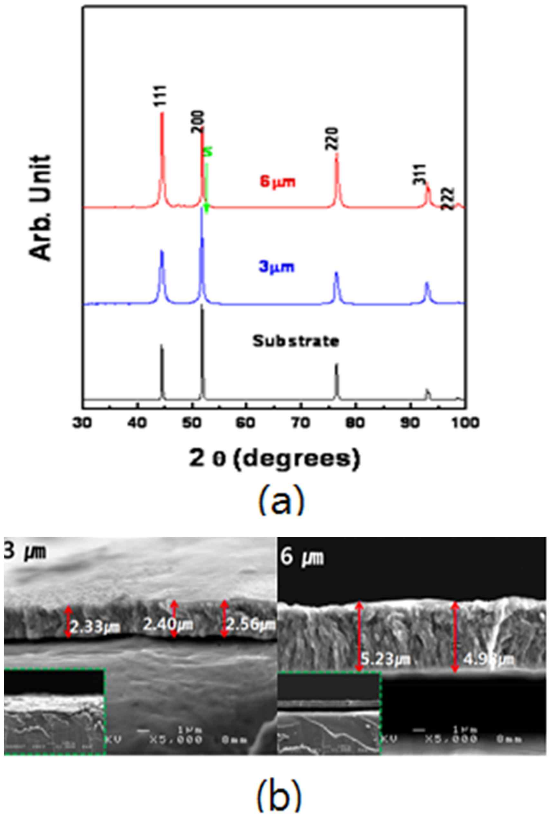

Fig. 2. (a) XRD patterns, and (b) SEM images of the cross section of the coating layer for the Ni on Ni sheet.

The saturation magnetizations of both in-plane and perpendicular hysteresis loops for the Ni substrate were measured as to be 56.5 emu/g and 39.0 emu/g, respectively. The coercivity of both in-plane and perpendicular were measured as to be 67.45 Oe and 65.078 Oe, respectively. Large coercivity for the on-plane hysteresis loop results plane orientation of the substrate. The coercivity of the perpendicular was decreased as the coating layer was increased. This result shows same tendency of the XRD. The saturation magnetizations of both in-plane and perpendicular hysteresis loops for the electroplated Ni on Ni sheet were measured as to be 61.4 emu/g and 41.3 emu/g, respectively. The coercivity of both in-plane and perpendicular were measured as to be 66.95 Oe and 59.96 Oe, respectively. The increase of the coercivity for the in-plane direction was larger than those of perpendicular direction. The residual stresses were measured by deposit stress analyzer during electroplating Ni at the current densities to be 10, 15 20, and 25 mA/cm^2. Table 1 represents the current density dependent of residual stress. The residual stress was shown as large values, as the current density was increased. The low current density played role of effective deposition of Ni ions on the substrate. The current efficiency was also decent, as the residual stress and the current density were decreased.

Table 1. Residual stresses measured by deposit stress analyzer (Model 683EC) at current densities to be 10, 15 20, and 25 mA/cm^2

Current density	Current efficiency	Thickness	Residual Stress
10 (mA/cm^2)	91.31 %	2.74 μm	75 MPa
15 (mA/cm^2)	89.86 %	2.70 μm	227.6 MPa
20 (mA/cm^2)	84.64 %	2.54 μm	322.6 MPa
25 (mA/cm^2)	62.74 %	1.89 μm	434.3 MPa

4. Conclusions

Nanocrystalline nickel (Ni) coating were synthesized by direct current electrodeposition at current density from 10 to 25 mA/cm^2 and pH=4. The basic composition of the bath, which was prepared by dissolving Ni metal particles in HCl, was 0.2 M Ni ions. The results showed that the surface roughness decreased as the saccharin addition of 2g/l. The experimental results showed that the increase in the current density had a considerable effect on the large residual stress of the Ni deposits. Crystal orientations of the films were estimated by the degree of high (200)_Ni orientation in the XRD patterns and M-H curves.

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