Ni electroplating using self-developed automatic electroplating device

Gujin Kang, Jin Kim, Sangmu Choi, Jongbum Kim*
Radioisotope Research Division, Korea Atomic Energy Research Institute, Daejeon, Korea
**Corresponding author: jong@kaeri.re.kr*

1. Introduction

Radioactive nickel sources are used in random number generators, beta cells, electron capture detector, and X-ray fluorescence. Among them, Ni-63, which emits 100% β-rays (E_{max} = 66.7 keV), can be manufactured in thin film form to be used for random number generators and beta batteries[1]. Electroplating method is usually used to manufacture the thin films due to its easy to control, thin thickness and low cost[2].

Several extra precautions should be considered when RI is electroplated. First, insoluble anode should be used to eliminate the possibility of natural nickel plated and to deposit Ni-63 dominantly. Second, since radioactive isotopes are expensive, in order to relatively increase the concentration of radioisotopes, a small plating bath should be used[3]. Third, the amount of radioactive waste after plating should be kept as low as possible to be managed easily and contained safely.

A small plating bath is difficult to be stirred, which results in some bubbles remained in the bath. Pitting phenomena due to the bubbles can include lower quality problem in small bath compared to large plating bath. In addition, the concentration of nickel decreases rapidly during plating in small baths. Therefore the temperature and current density should be changed to maintain the best plating conditions. It is important to optimize the conditions for each concentration of the plating solution to maintain exact mass thickness and homogeneity per unit area.

In this study, an automatic electroplating device was developed to obtain uniform electroplating results. Also, optimization conditions for electroplating were set resulting in accurate mass thickness.

2. Methods and material

2.1 Automatic plating machine

Figure 1 present the automatic electroplating machine developed for this work. Main electroplating part is located at the left side. Control box is located right side. Media bottles are stored the plating solution and washing solution in the upper right corner, and a solution circulating device is in the lower right corner. Waste collection bottles are located the upper left and there are containing KOH used to entrap chloride gas generated during plating. Two waste solution bottles are located lower left. The control box has ability to adjust the temperature of the plating bath, control circulator speed, and an ultrasonic.

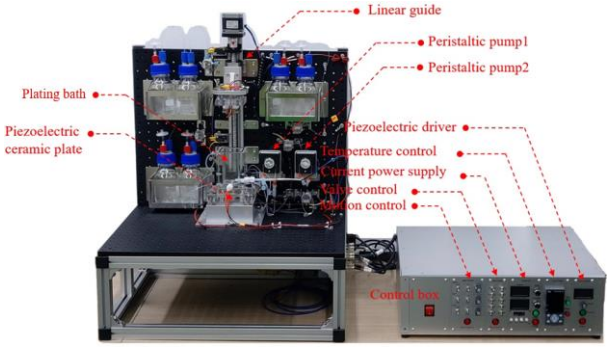

Fig. 1. Automatic electroplating machine for radioisotopes

2.2 Solution

Nickel Chloride hexahydrate (97%, SAMCHUN) was dissolved in 20 mL of distilled water in a beaker. In another beaker, 1.5996 g of boric acid (99.5%, SAMCHUN) and 45 mL of distilled water were added then, dissolved under stirring on a hot plate set at 80°C. After boric acid solution was cooled to room temperature and then moved to the first beaker under agitation. Finally, 65 mL of Ni plating solution could be prepared by adjusting the pH 4 using HCl (37%, MERK) and KOH (85%, SAMCHUN).

2.3 Electroplating

Pt coated Ti bar was used as the insoluble anode and a Cu plate was connected to the cathode. Nickel was electroplated on 2 cm x 2 cm Cu plate. Plating was then performed at room temperature, 40℃, and 50℃ with 0.06, 0.04 and 0.02 M plating solutions. Current densities for plating were 5, 10, 15 and 20 mA/cm^2.

2.4 Sample preparation for analysis Scanning Electron Microscope & Energy Dispersive Spectrometer

Special treatment is required to analyze the electroplating thickness, leveling and composition of the sample. Mounting press (Metpress-A, R&B) was used to make the samples. The sample surface was buffered with SiC sand paper and polishing cloths to be use on Auto polisher (RB 210 FUZZYPOL, R&B) of KAERI. The thickness, leveling and composition of the sample was measured by FE-SEM (SU-5000, HITACHI) & EDS (X-MAX, Oxford) of Hanbat University.

3. Results

3.1 Electroplating

We fabricated the Ni thin film samples with various conditions of current density and temperature. The best result of solution temperature was 40℃. At room temperature, nickel ions did small circulation, so electroplating result were uneven. Over the 50℃, solution was made vapor in bath. The Current density is kept low as possible. At high current density, the samples were burned. At 0.06 M concentration, the burning occurred at 20 mA/cm^2, and as the concentration was lower, the burning occurred at lower current density (Fig. 2.). Based on the above results, the best plating condition was concluded 0.06 M of concentration and 4o℃.

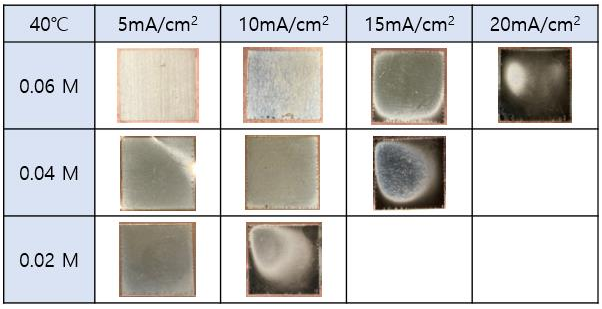

Fig. 2. Result of electroplating at different concentrations and current density at 40℃

3.2 SEM & EDS

The sample fabricated at the best condition was inspected, by SEM and EDS. In figure 3, total thickness of the sample was 550 ㎛ and nickel plating thickness was measured as 1.6 ㎛ on average. This sample was made at 40℃, 0.06 M and 5 mA/cm^2 on Cu plate while 19 min 31 sec. However, it did not reach the target thickness of 2 ㎛, and it was confirmed that the surface was rough. From the EDS image, we could find that Ni was electroplated stably on the Cu plate. The red points were the electroplated nickel, green points were copper plate and no other impurities were found.

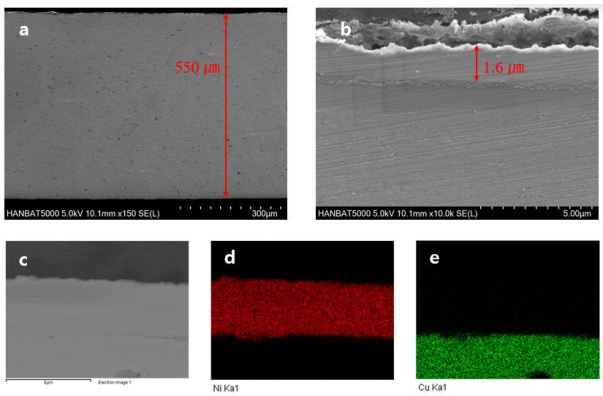

Fig. 3. SEM & EDS image (a) full sample, (b) plating part, (c) EDS mapping, (d) EDS image of Ni, (d) EDS image of Cu.

4. Conclusions

In order to obtain an accurate mass thickness, experiments were carried out using the self-developed automatic electroplating device. Plating machine produced a good quality nickel source at 40℃ with 5 mA/cm^2 current density. It was confirmed through SEM and EDS that the Ni plating thickness was 1.6 ㎛. However, the exact mass thickness could not achieved because the surface was rough and the target thickness was not reached. To solve this problem, we focus to eliminate air bubbles and optimize the plating the conditions.

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