Transactions of the Korean Nuclear Society Autumn Meeting
Gyeongju, Korea, October 29-30, 2015

Synthesis of Co diffused Cu matrix for applying Mössbauer source

Sang Mu Choi, Young Rang Uhm, Jong Bum Kim, and Kwang Jae Son*

[1]Radioisotope Research Division Korea Atomic Energy Research Institute, 989-111 Daeduckdaero, Yuseong-Gu, Daejeon 305-353, Republic of Korea
**Corresponding author: uyrang@kaeri.re.kr*

1. Introduction

For the preparation of ^{57}Co Mössbauer sources, the radioisotope was deposited onto the surface of a selected metal matrix, and then thermal diffusion process of the deposition into the metal lattice was carried out [1-2]. The thermal annealing of different metal matrices (Cu, Au, Fe, Co and Pt) with electrodeposited ^{57}Co described by Stephen [3] was performed in a continuously pumped vacuum furnace (10^{-4}~10^{-5} hPa) at temperatures of 950–1100 ℃ depending on the matrix metal. The annealing time was selected to be between 12 and 50 h. An optimized electro deposition process of carrier-free ^{57}Co onto the rhodium foil was described earlier. ^{57}Co plating is similar to other electroplating processes that employ soluble metal anodes. Specifically, it requires the passage of direct current (DC) between two electrodes that are immersed in a conductive, aqueous solution of nickel salts. The flow of a DC causes one of the electrodes (the anode) to dissolve and the other electrode (the cathode) to become covered with nickel. The cobalt in the solution is present in the form of divalent positively charged ions (Co^{2+}). When the current flows, the positive ions react with two electrons (2e-) and are converted into metallic cobalt (Co^0) at the cathode surface. The reverse occurs at the anode where metallic nickel is dissolved to form divalent positively charged ions that enter the solution.

Charged Co ions are formed by sulfate, sulfamate, chloride, and a Watts bath [4]. However, charged ^{57}Co ions are formed by dissolving metal ^{57}Co. To establish the coating conditions for ^{57}Co, non-radioactive Co ions are dissolved in an acid solution and electroplated on to a copper plate. Then, the thermal diffusion of electroplated Co into a copper matrix was studied to apply a ^{57}Co Mössbauer source [1].

In the present study, the influence of different annealing conditions was investigated. The diffusion depth of Co depends on the annealing temperature and time.

2. Experimental Technique

Cobalt (Co) coatings were deposited by DC electroplating at current densities of 20 mA/cm^2. The metal composition in the sulfamate bath was 0.16 M of Co prepared by applied surface concepts (SIFCO). The pH of the bath was adjusted from 1.89 ± 0.2 to 5 ± 0.2 by the drop wise addition of KOH (1N). A copper (Cu) plate 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. The deposition time was adjusted to achieve an average thickness of 6 μm based on Faraday's law [5]. 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 average particle size of the nanocrystalline Co coatings was calculated from XRD patterns using the modified Scherrer relationship expressed in [6].

3. Results and discussion

Co deposition was produced at a current density of 20 mA/cm^2, a bath temperature of 27 ℃, and various pH levels from 1.89 to 5. XRD patterns showed that the crystal structure of the coating is pure hcp cobalt, and no characteristic peaks of other phases have been recorded. The crystal orientation of the films was estimated by a degree of high (hcp) Co orientation in the XRD patterns. From the peak broadening of XRD patterns, the average crystalline size calculated from the XRD line broadening of the (101) peak was determined using the classical Scherrer relationship [6], $D(h\ k\ l) = k\lambda/B \cos\theta$, 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). The size of the deposited particles was at or below 90 nm. The smallest size of the particles was 23 nm, which was formed at a current density of 20 mA/cm^2. In the XRD results, the second phase was observed in electroplated samples at and above pH 4, and 5. Although the second phase was performed during electroplating, the thermal diffusion process should be carried out by annealing under a vacuum and Ar atmosphere. Thus, the second phase will be changed to a metal phase. This should be dealt with in a further study. The deposition was carried out at a current density of 20 mA/cm^2. The creation of the hole and pitch was observed on the coating layer prepared at and above pH 4. The particle size was increased, as the pH was increased up to 5. Figure 1 represents average particle sizes for Co on Cu plate deposited at different pH conditions and current densities (20 and 30 mA/cm^2) analyzed by Scherrer relationship of XRD patterns.

Figure 2 show results of magnified images of SEM for the electroplated Co at a pH (a)1.89, (b)3, (c)4, and (d) 5. Also, the cross section images were represented at Fig 2 (e) and 2(f). The deposition was carried out at a current density of 20 mA/cm^2. The creation of the hole and pitch was observed on the coating layer prepared at

and above pH 4. The deposition time was calculated to achieve an average thickness of 3~5 μm based on Faraday's law [5].

The diffusion degree was evaluated by mapping using scanning electron microscopy (SEM). The influence of different annealing conditions was investigated. The deposited Co was diffused almost completely into a copper matrix without substantial loss at an annealing temperature of 900 ℃ for 2 hours.

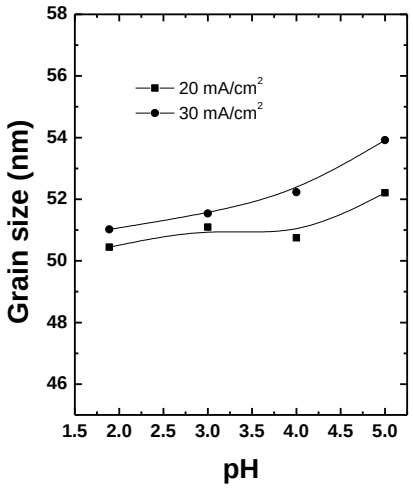

Fig. 1 Average particle sizes for Co on Cu plate deposited at different pH conditions and current densities (20 and 30 mA/cm^2) analyzed by Scherrer relationship of XRD patterns.

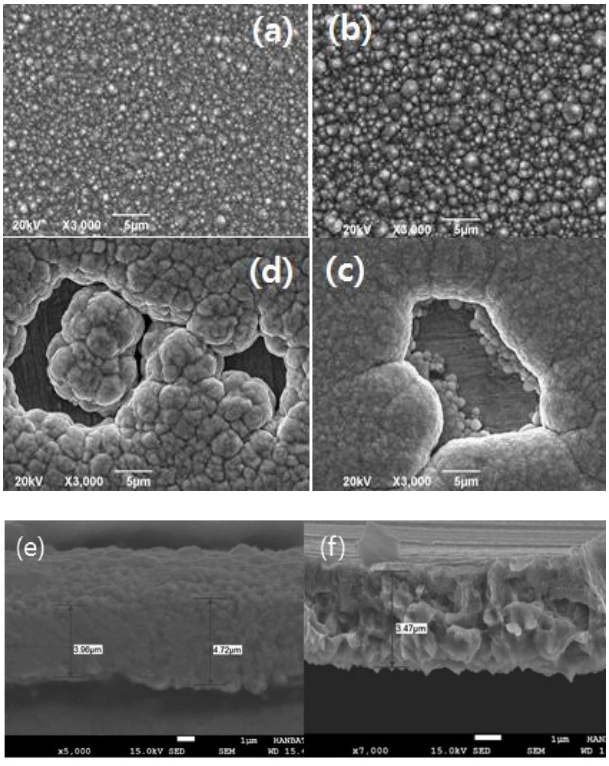

Fig. 2 SEM images for the Co coated Cu sheet at a current density of 20 mA/cm2 and pH at (a) 1.89, (b) 3, (c) 4, and (d) 5. The cross section of the Co deposited at (e) pH 1.89 and (d) pH 4.

4. Conclusions

To establish the coating conditions for ^{57}Co, non-radioactive Co ions are dissolved in an acid solution and electroplated on to a copper plate. Then, the thermal diffusion of electroplated Co into a copper matrix was studied to apply a ^{57}Co Mössbauer source. Nanocrystalline Co particles were coated on a Cu substrate using DC electro-deposition at a pH of 1.89 to 5 and 20~30 mA/cm^2. The average grain size was up to 54 nm as the pH increased to 5. The second phase of Co-oxide was formatted as the pH was increased above 4. The influence of different annealing conditions was investigated. The diffusion depth of Co depends on the annealing temperature and time. The results obtained confirm that the deposited Co diffused almost completely into a copper matrix without substantial loss at an annealing temperature of 900 ℃ for 2 hours.

Acknowledgement

This work was performed under the financial support from Creative Research Program of Korea Atomic Energy Research Institute in Republic of Korea.

REFERENCES

[1] I. Cieszykowska, M. Zoltowska, P. Zachariasz, A. Piasecki, T. Janiak, and M. Mielcarski, Thermal diffusion of ^{57}Co into rhodium matrix as a second step in preparing Mössbauer Sources, Applied Radiation and Isotope, Vol. 69, pp. 1193-1197, 2011.

[2] I. Cieszykowska, M. Zoltowska, and M. Mielcarski, Electrodeposition of carrier-free ^{57}Co on rhdium as an approach to the preparation of Mössbauer Sources, Applied Radiation and Isotope, Vol. 69, pp. 142-145, 2011.

[3] J. Stephen, An electrolytic method of preparing Mössbauer Sources and absobers, Nuclear Instruments and methods, Vol. 26, pp. 269-273, 1964.

[4] A.M. Rashidi, A. Amadeh, The effect of saccharin addition and bath temperature on the grain size of nanocrystalline nickel coating, Surface Coating and Technology , Vol. 204, No.3, pp.353–358, 2009.

[5] R. Orinakova, A. Turonova, M.G. Kladekova, R.M. Smith, Recent developments in the electrodeposition of Nickel and some nickel-based alloys, Journal of Applied Electrochemistry, Vol. 36, No.9, pp.957–972 2006

[6] Y.R. Uhm, J.H. Park, W.W. Kim, C.-H. Cho, C.K. Rhee, Magnetic properties of nano-size Ni synthesized by the pulsed wire evaporation(PWE) method, Matererial Science Engineering B, Vol. 106, No.3, pp. 224–227, 2004.