Ni electroplating using an automatic plating machine

자동 도금 장치를 이용한 Ni 전기 도금

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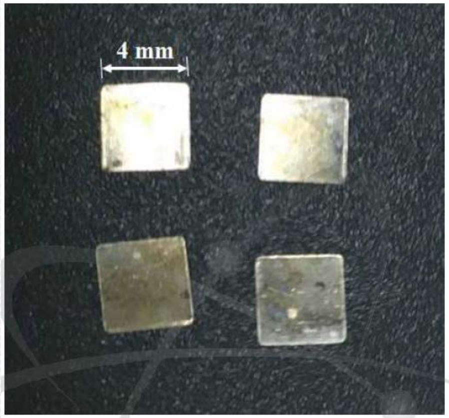
01 Purpose

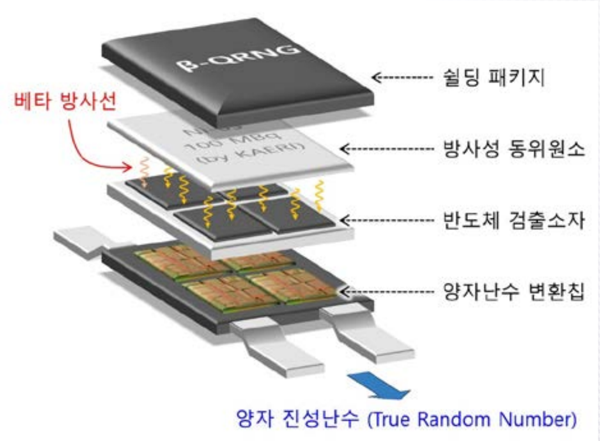
» Purpose

Optimizing plating conditions to achieve uniform quality using automatic machine developed for the establishment of a Ni-63 source mass production system.



Ni-63 source

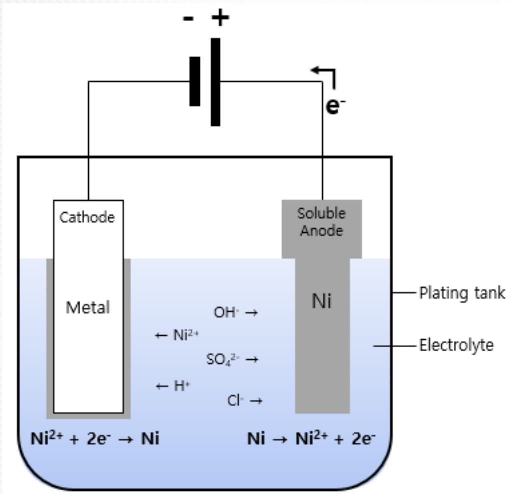
Beta battery

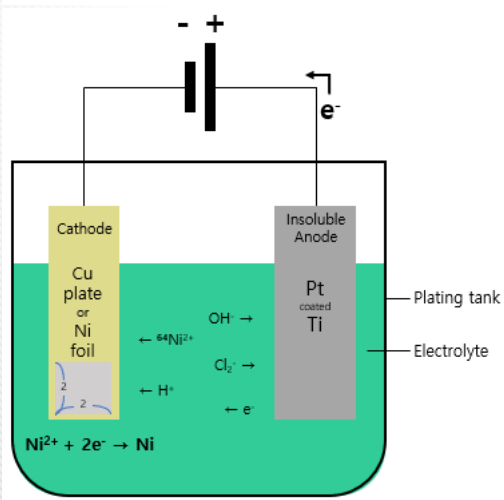


Random number generator

02 Electroplating Theory

Electroplating





- **Electroplating**

  - Metal (Ni) is connected to the anode
  - Material (backing material) is connected to the cathode

- **Soluble anode**

  - Deliver current to the electrolyte and supply metal ions
  - Cathode is shorter than anode
  - Bar, bags, strip, Ti baskets, pellets, etc

- **Insoluble anode**

  - Pt, Pt plated Ti, graphite, Pb
  - pH of electrolyte decrease due to oxidation of water on the surface

- **Radioactive electroplating**

  - Only pure Ni-63 is used to increase the radioactivity

02 Electroplating Theory

» Radioisotope electroplating

- Normal electroplating

  - Principle purpose is to enhance the decorative, corrosion resistance, and etc

  - No limits of plating solution, concentrate and anodes type

- **Radioisotope electroplating**

  - Manufactured to use radiation

  - Only radioisotopes must be electroplated

  - Radioisotope is very rare and expensive, so metal amount is limit

  - Concentration is decrease rapidly during the electroplating

  - Radioactive waste is generated

02 Electroplating Theory

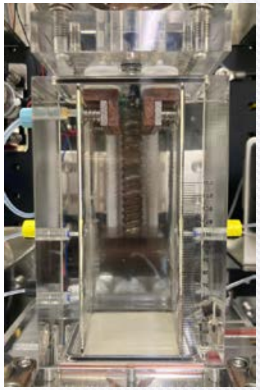
» Problems and solutions

- Low radio activity from radioisotopes source

  - Use insoluble anode and only soluble radioisotopes solution

- Low concentrate of electroplating solution and radioactive waste

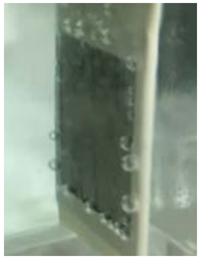
  - **Electroplating on small bath**



- Small electroplating bath

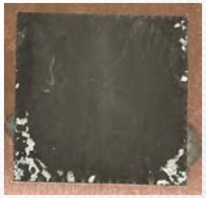
  1. Difficult of remove bubbles

     - Install the piezo driver



  2. Burning phenomenon occurs

     - Control the temperature and decrease current density



02 Electroplating Theory

Bath component

Component	Watts bath	Characteristic
Nickel Sulphate ($NiSO_4 \cdot 6H_2O$)	240 ~ 300 g/L (0.91 ~ 1.14 M)	Supply Ni^{2+}
Nickel Chloride ($NiCl_2 \cdot 6H_2O$)	30 ~ 90 g/L (0.12 ~ 0.37 M)	Supply Ni^{2+}, the conductivity of the electrolyte, internal stress
Boric Acid (H_3BO_3)	30 ~ 45 g/L	pH buffer, uniform color, poor adhesion
Temperature	40 ~ 60 ℃	Physical properties such as gloss and stress
pH	3.5 ~ 4.5	Physical properties such as gloss and stress
Cathode Current Density	2 ~ 7 A/dm^2	Physical properties such as gloss and stress
Deposition rate	25 ~85 µm/h	Control the thickness

- Ni-63 exists in the form of $NiCl_2 \cdot 6H_2O$, so the experiment was carried out under all chloride bath conditions.
- Due to the use of a small amount of radioactive materials, the capacity is smaller than that of general electroplating solutions.

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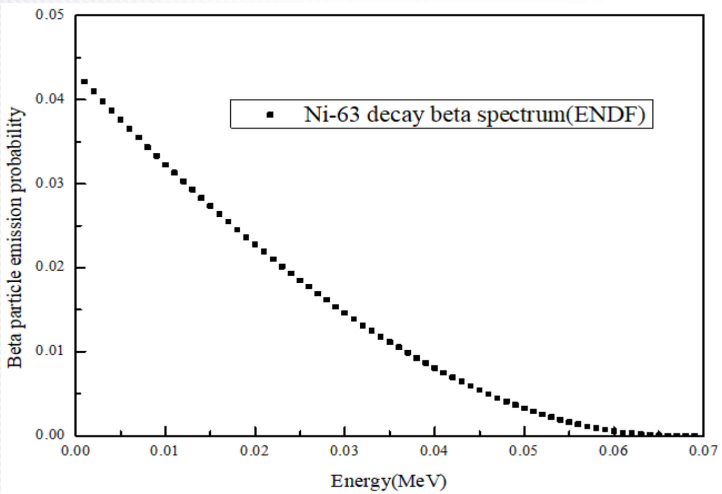
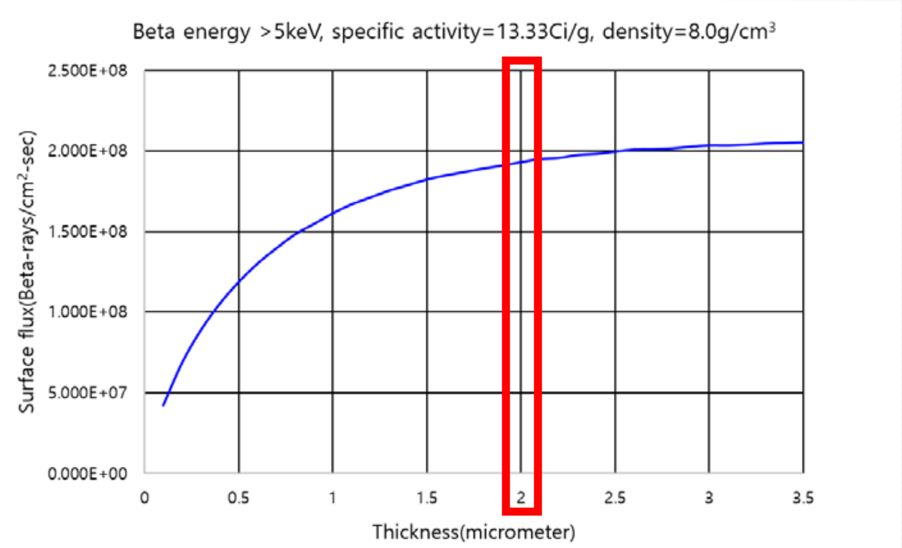
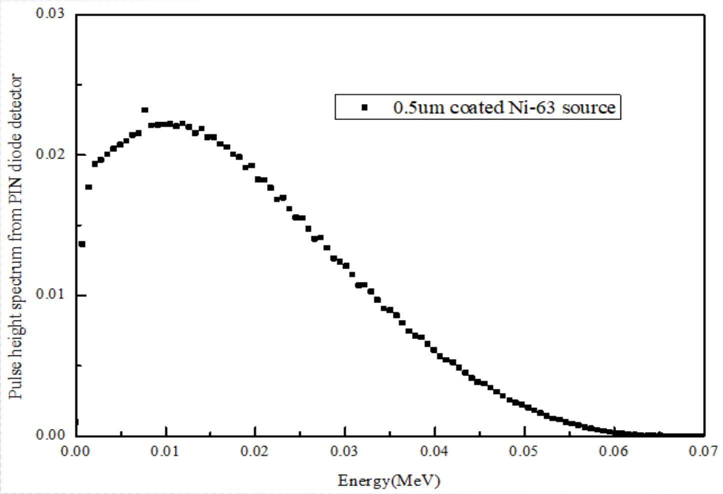
02 Electroplating Theory

» Thickness







- Ni-63: average 17 keV, maximum 66.94 keV, 100% β^- particle emission
- self shielding characteristics, which decrease with high energy
- 85% of the total energy is emitted at a thickness of 2 μm

Faraday constant (F)

1. The amount of dissolution at the anode or plating at the cathode is proportional to the amount of electricity passed (current x hours).
2. The amount of the substance dissolved or plated on the cathode by a certain amount of electricity is proportional to its chemical equivalent.

'F' is the amount of electricity required to electroplate or melt an equivalent of metal

1F = Ne = 96,500 C

$$t(s) = \frac{T \times valance \times F \times A \times d}{I \times M.W}$$

$$292.85 = \frac{0.0002 \times 2 \times 96{,}500 \times 4 \times 8.907}{0.08 \times 58.70}$$

t = Plating time (s)

T = Average thickness (cm)

F = Faraday constant

A = Plating area (cm^2)

I = Current (A)

d = density = Ni density = 8.907 g/cm^3

t = Plating time

T = 0.0002 cm

F = 96,500 C

A = 4 cm^2

I = 0.08 A

d = 8.907 g/cm^3

Current density (mA/cm^2)	Plating time (s)
5	1171.41
10	585.71
15	390.47
20	292.85
30	195.24

» Plating solution

Ni solution

1. Add 0.927 g (0.06 M), 0.618 g (0.04 M), 0.309 g (0.02 M), 0.155 g (0.01 M) Nickel(Ⅱ) chloride hexahydrate and 10 mL distilled water to a 50 mL beaker and stir

0.4 M Boric acid solution

1. In a 100 mL beaker, add 1.5995 g boric acid and 35 mL distilled water
2. Stir while heating the solution to 80 ℃ with a hot plate
3. After all is melted, cool to room temperature

Add solutions

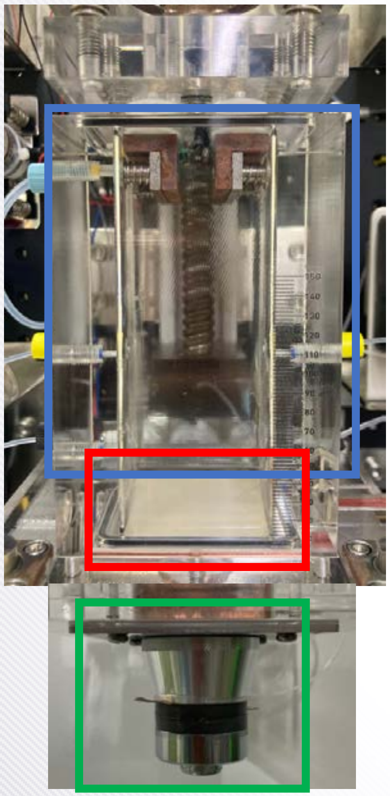
1. After moving the Ni solution to the beaker containing boric acid, wash the 50 mL beaker with 10 mL of distilled water, and then move again
2. After adjusting the pH 4 with HCl and KOH, add distilled water to complete 65 mL of the plating solution

pH 4 ± 0.2

Electrolyte 65 mL

03 Experiment method

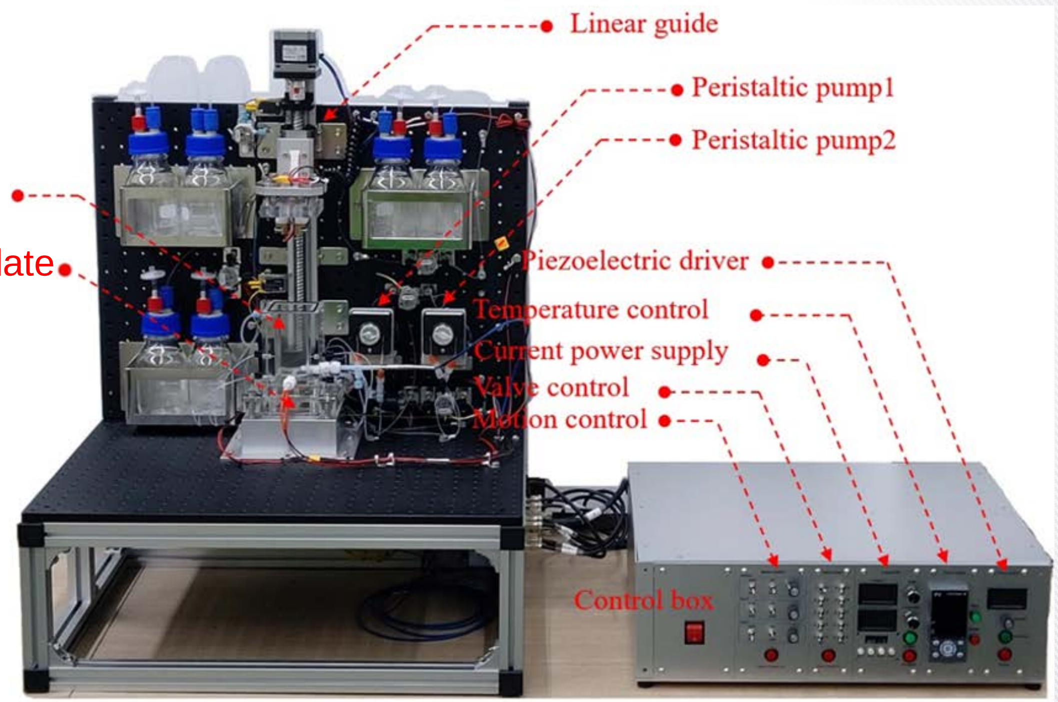
»



Plating bath

Ceramic heater plate

Piezo driver



Main part

Control box

04 Results

» Results of electroplating under different conditions

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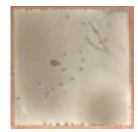
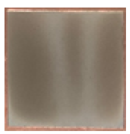
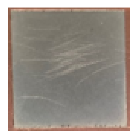
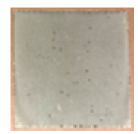
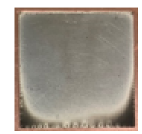
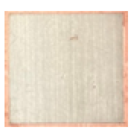
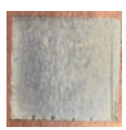
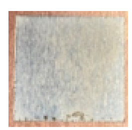
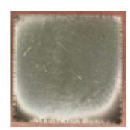
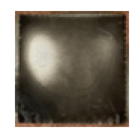
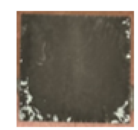
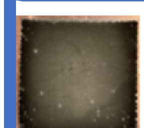
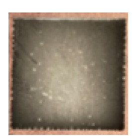
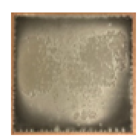
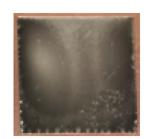
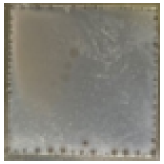
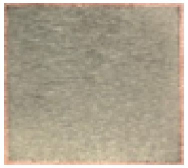
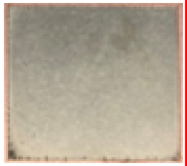
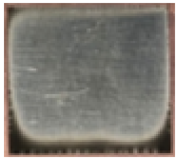
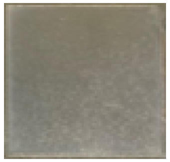
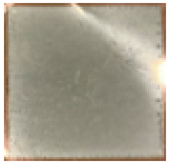
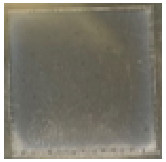
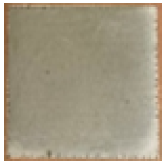
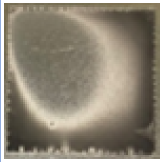
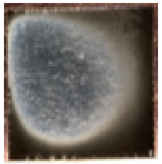
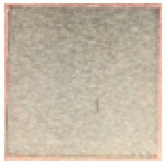
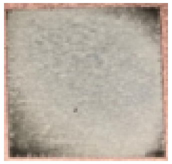
60°C					
50°C					
45°C					
40°C		 	 		
R.T		 			

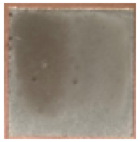
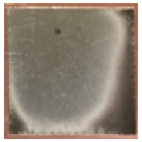
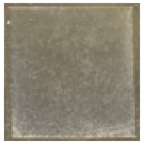
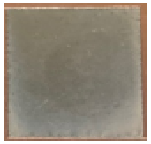
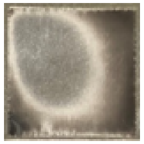
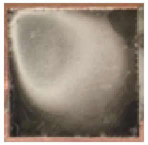
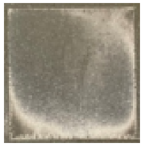
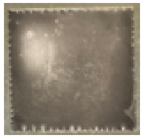
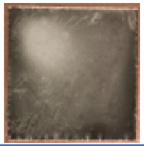
R.T = Room temperature

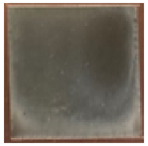
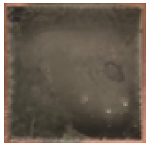
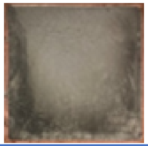
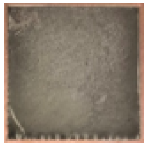
04 Results

Results of electroplating under different conditions

0.04M	5mA/cm²	10mA/cm²	15mA/cm²	20mA/cm²
50℃	 	 		
40℃	 	 	 	
R.T				

» Results of electroplating under different conditions

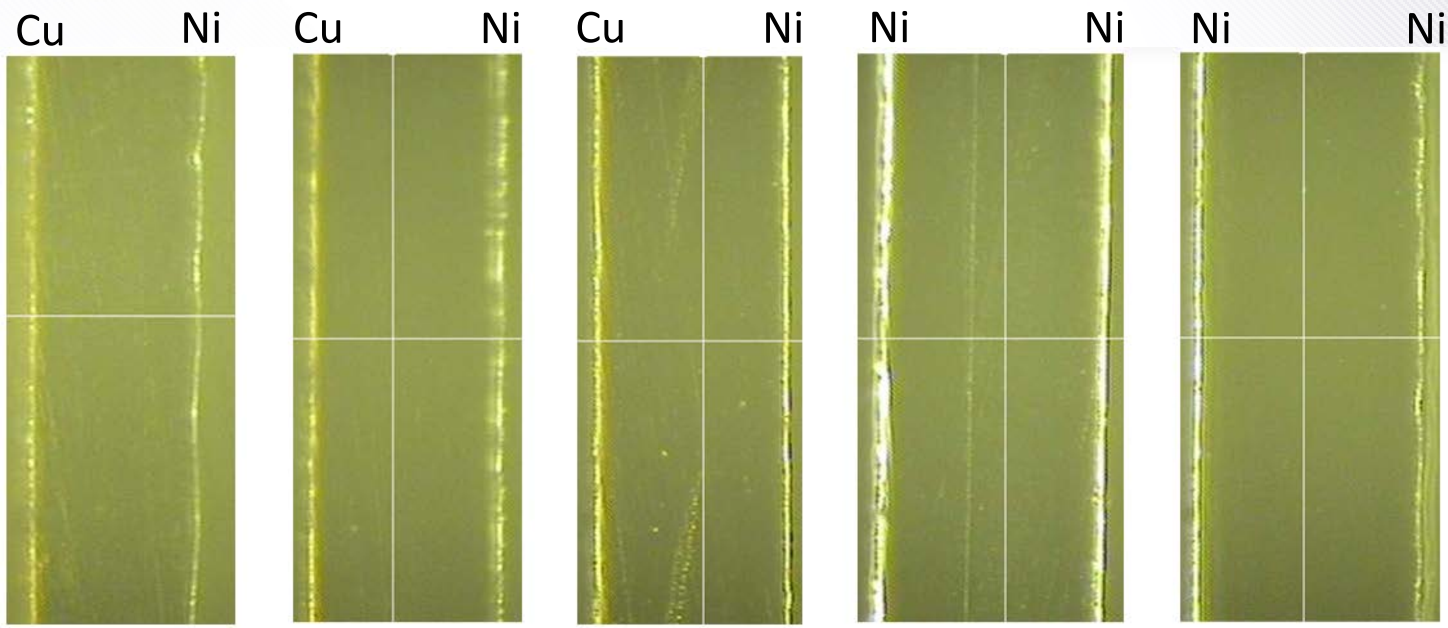
0.02M	5mA/cm²	10mA/cm²
50℃		
40℃	 	 
R.T	 	 

0.01M	5mA/cm²	10mA/cm²
50℃		
40℃		
R.T		

- Burning phenomenon occurs
  (0.06 M, 20 mA/cm²), (0.04 M, 15 mA/cm²), (0.02 M, 10 mA/cm²), (0.01 M, 5 mA/cm²)
- Best plating results at 40 ℃ and 5 mA/cm² all concentration

04 Results

» Optical microscope

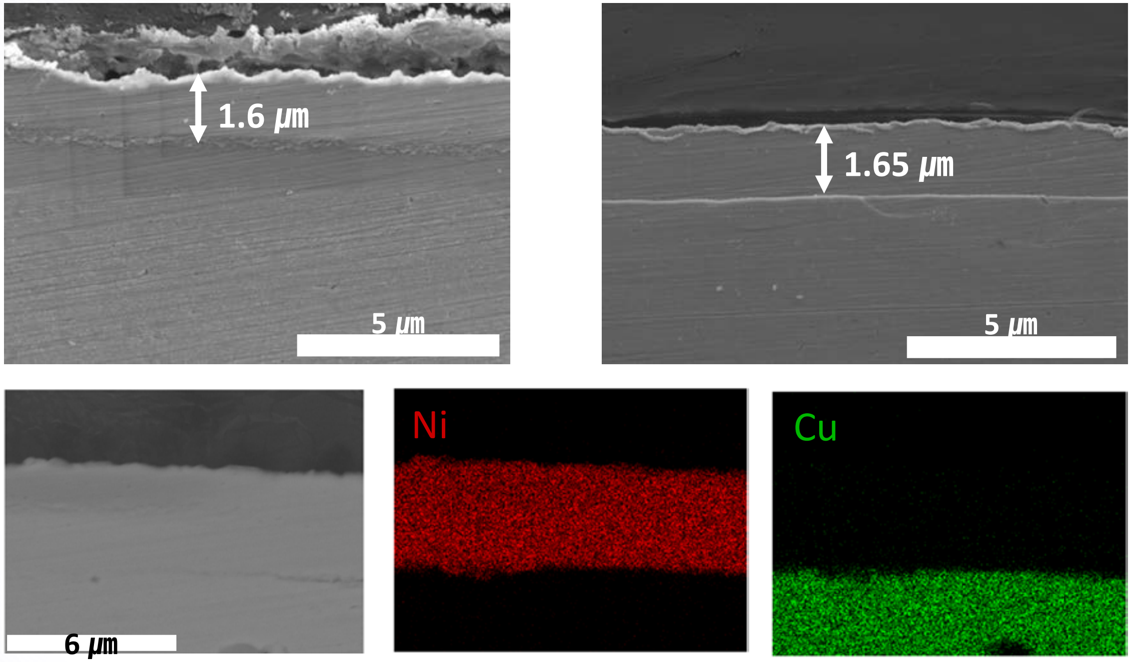


1	2	3	4	5
Cu plate	Cu plate	Cu plate	Ni foil	Ni foil
0.5 mm	0.2 mm	0.2 mm	0.125 mm	0.125 mm

- On the left is the substrate, and on the right is the electroplating

04 Results

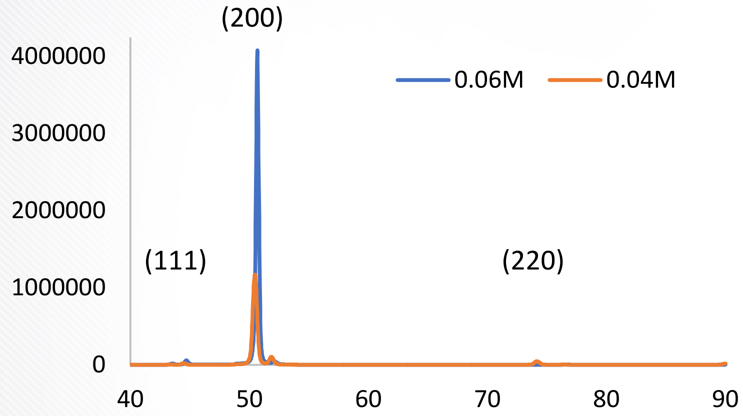
» SEM & EDS



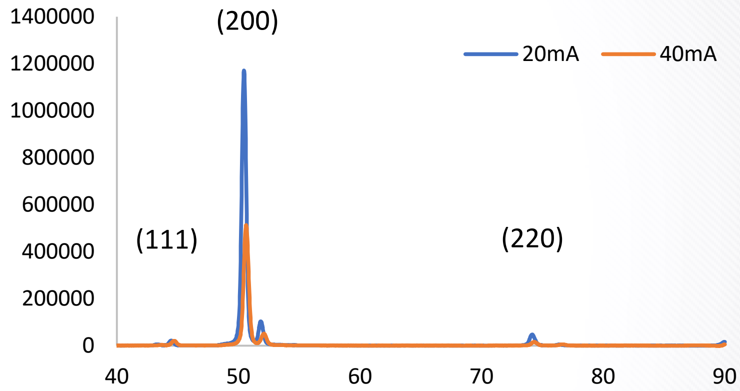
- Average thickness was 1.625 μm at SEM
- Only Ni was plated on Cu

04 Results

X-ray diffraction (XRD)



Compared result 0.06 M and 0.04 M at 20 mA, 40 ℃

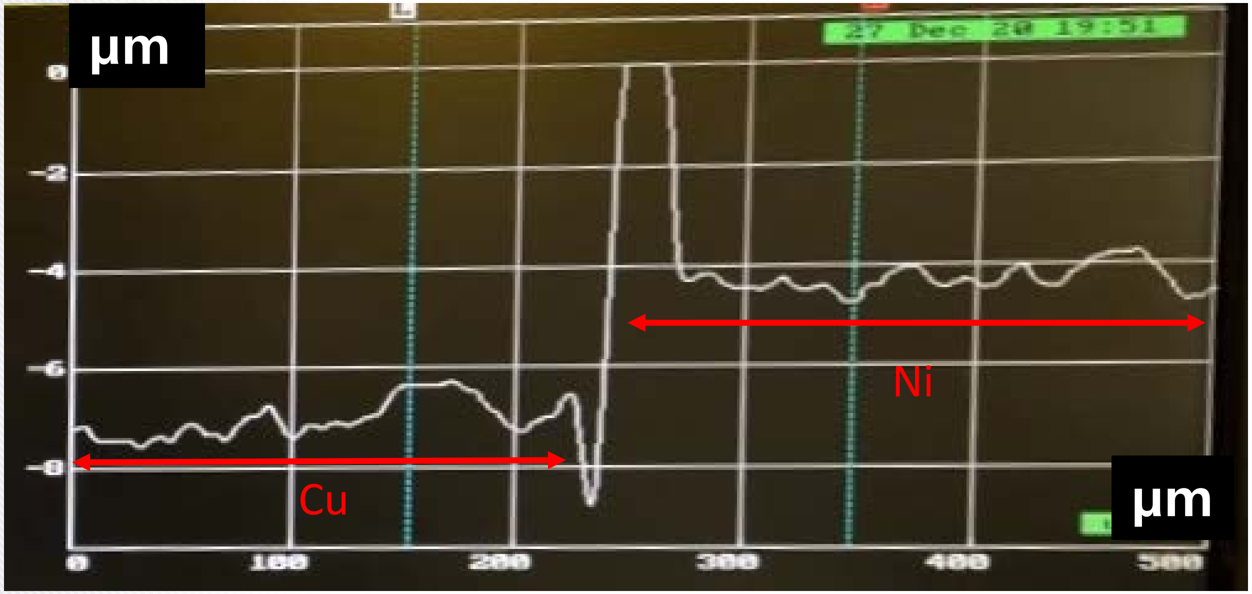


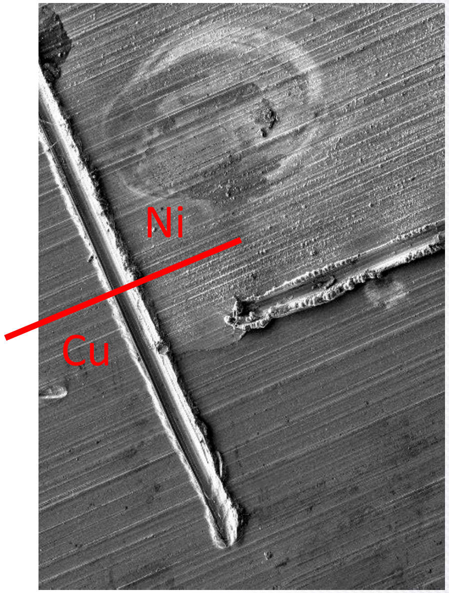
Compared result 20 mA and 40 mA at 0.04 M, 40 ℃

- As a result, they have crystallinity in the (111), (200), and (220) planes
- The 2θ value formed by each crystal differs depending on the conditions
- The smaller the concentration and the higher the current, the smaller the crystal size

04 Results

» Alpha-step





- There was a step difference due to Ni plating on the Cu surface.
- Step range was almost 1.5 ~ 2 μm

Conclusion

The cross-section, composition and thickness of the coating are measured by SEM, EDS, XRD and alpha step instrument. So far, it has been proven that the best electroplating result can be obtained when electroplating at 40 °C and 5 mA/cm^2.

THANK YOU

Korea Atomic Energy Research Institute