

Nickel Electroplating: Effect of Current and Voltage on the Amount of Deposited Metal

Metallurgical and Materials Engineering, Laboratory Sheet (2nd year)

1. Objective

To electroplate nickel onto copper and stainless steel substrates from a nickel sulfate solution using a DC power supply; to measure how the applied current and voltage affect the amount of deposited metal; and to compare the measured mass gain with the theoretical value from Faraday's law, in order to determine the cathodic current efficiency and the coating thickness.

2. Theory

At the cathode the reaction $\text{Ni}^{2+} + 2\text{e}^- \rightarrow \text{Ni}$ takes place. The nickel anode dissolves ($\text{Ni} \rightarrow \text{Ni}^{2+} + 2\text{e}^-$) and keeps the bath concentration roughly constant. Hydrogen evolution ($2\text{H}^+ + 2\text{e}^- \rightarrow \text{H}_2$) can occur at the cathode at the same time, so part of the current does not go into plating.

$$m_{\text{theor}} = (M \cdot I \cdot t) / (n \cdot F)$$

$M = 58.69 \text{ g/mol (Ni)}$, $n = 2$, $F = 96485 \text{ C/mol}$, I : current (A), t : time (s).

$$\eta (\%) = (m_{\text{exp}} / m_{\text{theor}}) \cdot 100 \quad j = I / A \quad \delta (\mu\text{m}) = 10^4 \cdot m / (\rho \cdot A)$$

$\rho_{\text{Ni}} = 8.90 \text{ g/cm}^3$, A : total plated surface area (cm^2), j : current density (A/dm^2).

Example: for $I = 0.5 \text{ A}$ and $t = 15 \text{ min (900 s)}$, $m_{\text{theor}} = 58.69 \cdot 0.5 \cdot 900 / (2 \cdot 96485) \approx 0.137 \text{ g}$.

Note: the passive chromium oxide film on stainless steel makes adhesion of the coating difficult. For this reason the steel specimens are activated in acid (step 5.2).

3. Equipment and chemicals

- DC power supply (adjustable current and voltage), ammeter and voltmeter (or the instrument display)
- 600 mL beaker or plating cell, magnetic stirrer (with heater if available)
- Cathodes: copper sheet and AISI 304 stainless steel sheet (e.g. $2 \times 3 \text{ cm}$); anode: pure nickel plate
- Electrolyte: nickel sulfate solution ($\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, concentration prepared in the lab: _____ g/L, pH: _____, temperature: _____ °C)
- Analytical balance (0.1 mg), caliper, abrasive paper (800–1200 grit), acetone or ethanol, distilled water, dilute H_2SO_4 and HCl , stopwatch, crocodile clip leads, drying oven or hot-air blower

4. Safety warnings

Caution:

- Nickel salts can cause skin sensitization and allergies, and may be carcinogenic when inhaled. Wear gloves, goggles and a lab coat; weigh powdered salts under ventilation.
- Hydrogen is released at the cathode during plating. Keep open flames away and keep ventilation on.
- Wear gloves and goggles when working with acids. Always add acid to water when diluting.
- Do not pour waste solution or rinse water down the drain; collect it in the "heavy metal waste" container.
- Connect and change leads only while the power supply is switched off.

5. Procedure

5.1 Specimen preparation

1. Measure the specimen dimensions with a caliper, calculate the total surface area (A) and record it.
2. Grind the surfaces, degrease with acetone or ethanol, rinse with distilled water and dry.
3. Weigh each specimen on the analytical balance (mo).

5.2 Surface activation

1. Copper: dip in dilute H₂SO₄ (about 5%) for 10–20 s, then rinse with distilled water.
2. Stainless steel: dip in dilute HCl (about 10–15%) for 30–60 s, rinse, and transfer to the bath immediately.

5.3 Plating

Two experiments are run for each substrate in each series (8 experiments in total). Specimens are 2 × 3 cm and immersed on both sides, so the total area is $A = 12 \text{ cm}^2 = 0.12 \text{ dm}^2$. The plating time is 15 min in every experiment.

Substrate	Control mode	Setting
Cu	Constant current	0.12 A
Cu	Constant current	0.36 A
Cu	Constant voltage	3 V
Cu	Constant voltage	5 V
Steel	Constant current	0.12 A
Steel	Constant current	0.36 A
Steel	Constant voltage	3 V
Steel	Constant voltage	5 V

1. Prepare the bath and record its temperature and pH. Place the anode and cathode with a fixed spacing (e.g. 3–4 cm) and keep the same spacing in all experiments.
2. **Series A (constant current):** set the power supply to current-controlled mode. Keep the time constant and apply two different currents ($j = 1$ and 3 A/dm^2 , i.e. 0.12 A and 0.36 A). Record the voltage you read.
3. **Series B (constant voltage):** set the power supply to voltage-controlled mode. Apply two different voltages (3 V and 5 V) for the same time. Record the current at the start and at the end and take the average.
4. When the time is up, switch off the power supply. Remove the cathode, rinse with distilled water, rinse with ethanol, dry and weigh again (m_1).
5. Use a fresh specimen for each experiment, with identical conditions for copper and steel. Note the surface appearance (brightness, roughness, burning, peeling, blistering).

6. Measurement and calculation tables

Series A: Constant current (time $t = \underline{\hspace{1cm}}$ min)

Substrate	I (A)	V (V)	A (cm^2)	j (A/dm^2)	m_0 (g)	m_1 (g)	Δm (g)	m_{theor} (g)	η (%)	δ (μm)
Cu	0.12									
Cu	0.36									
Steel	0.12									
Steel	0.36									

Series B: Constant voltage (time $t = \underline{\hspace{1cm}}$ min)

Substrate	V (V)	I start (A)	I end (A)	I avg. (A)	A (cm^2)	m_0 (g)	m_1 (g)	Δm (g)	m_{theor} (g)	η (%)
Cu	3									
Cu	5									
Steel	3									
Steel	5									

Surface observations

Experiment	Cu: appearance and adhesion	Steel: appearance and adhesion