

OPTIMISATION OF AN ELECTROPLATING PROCESS TO PREPARE A SOLID TARGET FOR (p,n) BASED PRODUCTION OF COPPER-64

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Introduction

Research into the production of copper-64 from a nickel-64 solid target utilising a semi-automated solid target assembly coupled to an IBA 18/9 MeV proton cyclotron is ongoing. The target is prepared using an electroplating method adapted from McCarthy et al (1997), which uses a solution of nickel ammonium sulfate (adjusted to pH 9 with ammonium hydroxide) to plate nickel onto a gold substrate. While this method of production is sometimes very successful, it has also proved unreliable, producing poorly plated disks in approximately 50% of experiments. The irregularities observed in the nickel surface include - flaking, crazing, formation of spheres or pits, loose/powdery Ni, poorly adhered Ni, a lack of 'lustre' and a black deposit forming on the anode. An article from Kim et al (2009) described the black anode deposit, and suggested that ammonium hydroxide and/or ammonium sulfate added to counter residual acidity in the nickel ammonium sulphate solution was the cause. Kim et al suggested an electroplating method to resolve this issue. Further work was carried out to optimise our electroplating procedure, based on their method.

Aim

To develop a method that reliably and reproducibly generates a solid target for copper-64 production by electroplating nickel-64 onto gold; and to optimise the electroplating conditions to enable maximum nickel deposition for minimal time and use of nickel-64.

Method

Preparation of purified NiSO₄ [adapted from Kim et al (2009)]

Nickel metal is dissolved in nitric acid and evaporated to dryness. The solid is treated with sulfuric acid and dried to a yellow solid. The residue is dissolved in milliQ water and recrystallised by adding acetone. The solid is collected by vacuum filtration, and dried over vacuum for two hours, followed by drying in an oven at 120°C for a minimum of two hours. The resulting yellow-green solid is NiSO₄.

Preparation of electroplating solution

Purified NiSO₄ (0.13770g to 0.30079g) was dissolved in milliQ water (5mL, 10mL, or 15mL). Ammonium sulfate (~0.06g) was also dissolved into the solution.

Electroplating experimental conditions

Anode: initially carbon rod (rotating), then platinum rod (non-rotating)

Cathode: initially 2mm x 20mm gold disk, then 125µm x 15mm gold foil

Solution: initially nickel ammonium sulfate, pH 9, with ammonium sulfate buffer, Ni concentration ~3mg/mL (McCarthy et al, 1997); then nickel sulfate, pH 4.5, with ammonium sulfate buffer, Ni concentration ~5mg/mL (Kim et al, 2009)

Plating area: 10mm diameter, 78mm²

Current: Constant 6mA

Time: 12 hours (10 experiments, varying masses of NiSO₄), plus 6 experiments with time varied from 12-96 hours (constant mass of NiSO₄)

Results

16 experiments were conducted with nickel sulfate - 14 considered were successful.

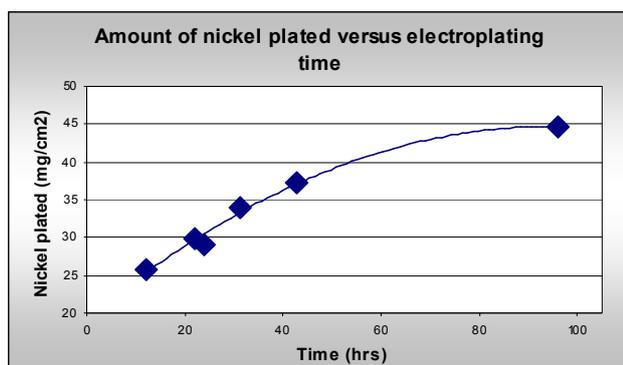


Figure 1: Mass of nickel plated versus electroplating time (constant concentration of nickel in solution, 150mg NiSO₄ in 10mL)

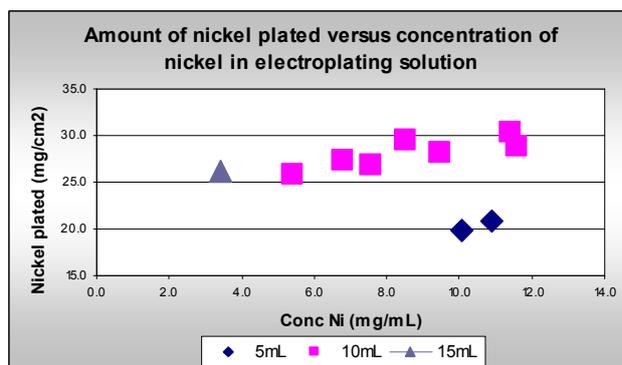


Figure 2: Mass of nickel plated versus concentration of nickel in electroplating solution (for constant electroplating time, 12 hours)

Discussion and Conclusion

Fourteen of the 16 NiSO₄ experiments resulted in a lustrous, well-adhered layer of nickel, with no black residue on the platinum anode. The two failures were the result of variation in the constant current applied to the cell, and a change in the volume of water (increased to 15mL). Some divots have been observed in the nickel surface, indicating that bubbles have adhered to the surface during plating, but they are small and not considered a defect. The electroplating solution is stable over time (ie. no precipitate formed), and it is easy to prepare. The average yield of nickel plated using NiSO₄ is much lower than that achieved with Ni(NH₄)₂.2SO₄ (37-63%, versus ~70-95%), which is a disadvantage.

Effect of time (constant NiSO₄ concentration): Figure 1 shows the amount of nickel plated plateaus rapidly. Doubling the time (12 to 24 hours) results in a 1.1x increase in Ni plated, while quadrupling the time (12 to 96 hours) only results in 1.7x more nickel plated. Run times less than 24 hours are therefore most efficient.

Effect of varying NiSO₄ concentration (constant time): Figure 2 shows a low yield was achieved using a volume of 5mL. One experiment using 15mL of water resulted in a poor nickel surface despite a reasonable amount of nickel plated. The best yield with minimal amount of nickel in solution was achieved with a 10mL solution of 8.5mg/mL of nickel.

Overall, we are satisfied with the reliability and reproducibility of our method.

References

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