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# Effect of plating time on surface morphology and coating thickness of nickel plating on copper surface

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**Public Interest Statement**

While many of the advances in materials science have been driven by breakthroughs in material design and fabrication, understanding the changes that occur in a material during its utilization or operation in devices is of immense importance for its successful integration. Considering these aspects and the continued growth in materials research, there is a clear need for new topical journals which can serve researchers to understand the phase transitions and exploit the phenomena to current, state-of-the-art research in the field of materials science, moreover with full accessibility.

# Effect of plating time on surface morphology and coating thickness of nickel plating on the copper surface

**Abstract**-
In the present work, Ni thin films were deposited by electrodeposition method on copper substrate. The coating time was varied systematically in the range of 10 – 25 minutes during the deposition process resulting in variation in coating microstructure and properties. Structural properties and morphological studies were investigated by using Energy Dispersive Spectroscopy (EDS), X-ray diffraction (XRD), and Scanning Electron Microscopy (SEM) techniques. Coatings were analyzed for their variation in current efficiency and coating thickness with plating time. XRD peaks were observed to be broad and short indicating nanosized coating. The morphology of coating was observed to be made of smaller primary nodules and large secondary nodules. The secondary nodules were observed to be of coffee bean structure. With the increase in plating time, the secondary nodules growth rate was observed to increase forming a truncated pyramid kind of structures, leading to increased roughness. Plating of Ni by electrodeposition has an advantage due to its porous nature of coating leading to self-lubrication effect.

**Keywords**—nickel, copper, electrodeposition, scanning electron microscope, energy dispersive spectroscopy

## Introduction

Metal Coating is a process of applying a thin layer of metal on the surface of another metal commonly known as substrate. Most widely used coating techniques include physical vapour deposition (PVD), chemical vapour deposition (CVD), electrodeposition, lithography, sol–gel etc [1]. Electrodeposition is a surface modification technique that involves the deposition of metallic coatings on a conductive cathode by electrolysis of aqueous electrolyte solutions. This can be performed at ambient temperature or elevated temperatures, under normal pressure. Coatings of nanosized grains are usually preferred due to their good mechanical properties and this can be easily achieved by controlling the deposition parameters [2]. Even though there are different surface modification techniques, electrodeposition is significantly used because of its low cost and high deposition rate benefits. Moreover, this technology enables coating of complex geometry substrates and large surfaces with high thickness uniformity. Therefore, electrodeposition gained significant importance as an attractive surface-finishing technique for wear and corrosion protection [3]. The electrodeposition process is usually aimed at decorative, engineering, and electroforming applications. The process involves metallic dissolution from one electrode (Anode) and deposition on the other electrode (cathode), and it depends on various parameters out of which the three main factors include the chemical type and concentration in electrochemical bath, the current density applied, and hydrodynamic conditions [4].

Nickel (Ni) is a precious and noble metal with ideal hardness properties, wear resistance, and brightness. It is also highly catalytic in nature. However, due to the high cost of nickel metal, industries go for coating components made of Zinc, Steel, Brass, Copper, Aluminum, and Magnesium alloys with Ni. Because of their good corrosion resistance and mechanical properties, Ni-plated articles find application in transport and service apparatus [5, 6]. From literature it is seen that nanocrystalline Ni coating exhibits many unusual mechanical and electrochemical properties compared to other polycrystalline or amorphous materials. So introducing a coating of Ni on copper would be promising because of good wear and corrosion resistance [6]. Ni plated copper substrates can also resist thermal shocks and high-temperature exposure due to which copper wires are plated with Ni and used in aerospace, aviation, and industrial application [7].

Electroplating is governed by Faraday's law of electrolysis;

$$Q=It \qquad \text{Eqn (1)}$$

Where;

Q is the charge passed, I is the current passed, and t is the time current is passed [8].

In the present work Ni is coated onto copper strips by electrodeposition technique by varying the plating time. This Ni plating is expected to exhibit better properties compared to pure copper base metal with respect to hardness, wear, and tribological properties. From the literature, the coatings are characterized for their surface morphology, composition, and atomic arrangements [9-11]. In the present work Scanning Electron Microscope (SEM), X-ray Diffraction (XRD), and Energy Dispersive Spectroscopy (EDS) techniques are used to characterize the coatings for their surface morphology, composition, and atomic arrangements.

## EXPERIMENTAL PROCEDURE

The electroplating technique was followed to deposit nano-crystalline Ni onto the copper substrate. Thin strips of polished copper, approximately 15mm × 15mm, were prepared as a substrate. Pure Ni strip was used as an anode and copper as the cathode in the electroplating cell. Watts electroplating bath comprised of Nickel Sulfate (NiSO4.6H20): 300 g/l, Nickel Chloride (NiCl2.6H20): 150 g/l, Boric Acid (H3BO3): 52 g/l is used [12]. Plating was performed at room temperature by applying air agitation. The plating time was varied in the range of 10 minutes to 25 minutes in steps of 5 minutes. The samples were then analyzed for their structure, atomic arrangements, and composition using SEM, EDS, and XRD. The variation in the rate of coating and current efficiency with time is plotted. Theoretical coating thickness was calculated using Faraday's Law.

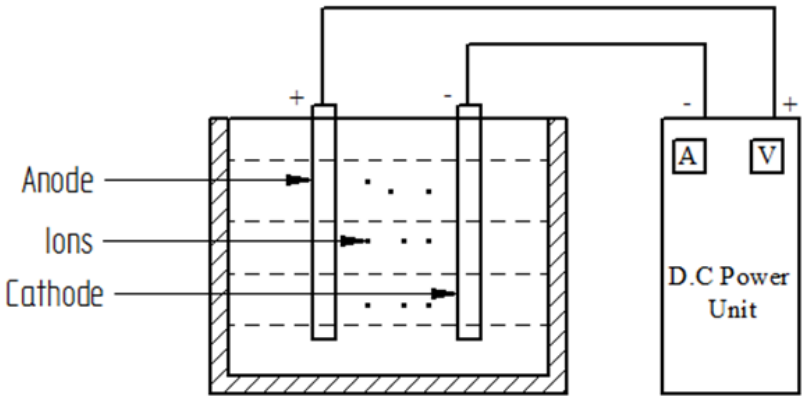
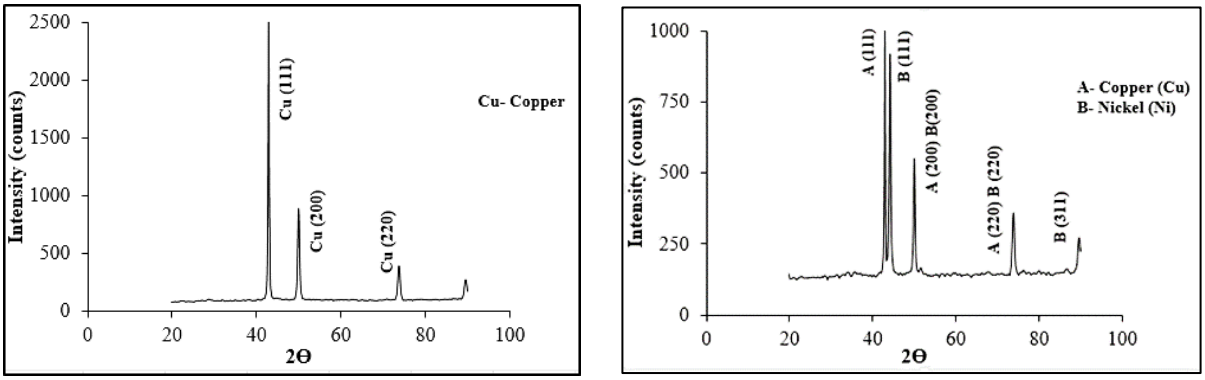



Fig 1: Schematic representation of experimental procedure

| | |
|---|---|
| Nickel Sulfate (NiSO4.6H20) | 300 g/l |
| Nickel Chloride (NiCl2.6H20) | 150 g/l |
| Boric Acid (H3BO3) | 52 g/l |
| **Operating Conditions** | |
| Bath Temperature | $30^0$C |
| Type of Agitation | Air |
| Anode | Nickel |
| Cathode | Copper Strip |
| pH | 3 |
| Current Density | 10 A/dm$^2$ |
| Plating Time | 10 – 25 minutes |

Table 1: Plating parameters and electrolyte composition



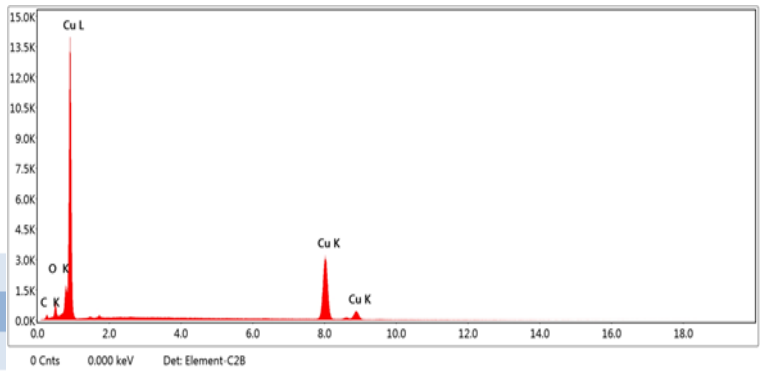

(a) (b)

Fig 2: XRD graphs for a) Pure Cu sample and b) Ni plated Cu sample

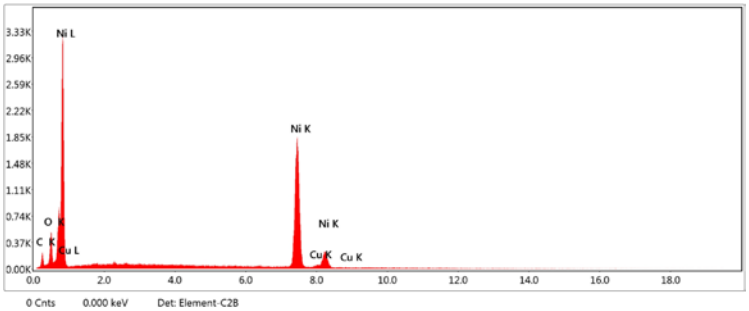
## III RESULTS AND DISCUSSION

XRD peaks are analyzed for the Cu sample, and Ni plated sample. The preferred orientation of the electrodeposited Ni is represented at a current density of 10A/dm2 for a plating time of 15 minutes. The same peaks are observed for Ni plated samples, but there is a considerable reduction in intensity. The orientation of the substrate greatly influences the growth process. The [111] and [200] orientation observed in the plating is expected to provide free growth property [13,14]. It is observed that the peaks for pure copper sample are narrow and long, whereas for Ni plated samples, the peaks are broad and short. Broad and short peaks are characteristic of a nanosized structure with porous nature. Porous nature will lead to self-lubricating effect of the coating. Wider XRD peak represents an increase in stacking faults and structural disorder, thereby providing better hardness and wear properties [15].

| Element | Weight % | Atomic % |
|---|---|---|
| C K | 3.82 | 15.77 |
| O K | 3.92 | 12.17 |
| CuK | 92.26 | 72.06 |




(a)




| Element | Weight % | Atomic % |
|---|---|---|
| C K | 7.69 | 26.02 |
| O K | 5.54 | 14.07 |
| NiK | 84.45 | 58.43 |
| CuK | 2.31 | 1.48 |

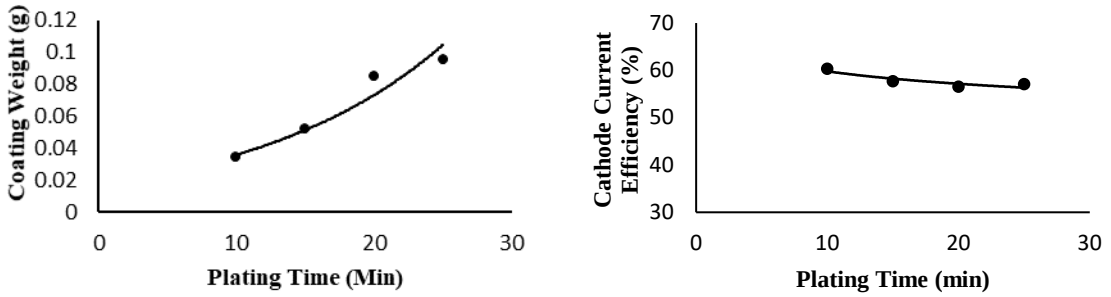
(b)

Fig 3: EDS characterization results for a) Pure Cu sample and b) Ni plated Cu sample.

The coatings are characterized for their composition by EDS technique. EDS study is helpful in determining the elements present in the base metal and coatings of Ni. The abscissa of the EDS spectrum indicates the

ionization energy, and ordinate indicates the counts. The higher the counts of a particular element, higher will be its presence at that point or area of interest. The results provided for heavier elements are very accurate, and it shows clearly a higher weight percentage for Ni, indicating Ni deposition. A small weight percentage of Cu is observed in the result indicating the detection of base metal atoms. C and O are light elements and its presence in the result is unavoidable. The results provided for light elements may not be a highly accurate one [15].

Coating weight and Cathode Current Efficiency are two very important parameters in electrodeposition [16]. The graphs show that the amount of Ni plated increases almost linearly with plating time indicating a constant deposition rate throughout the deposition. This can be attributed to the fact that cathode current efficiency remains almost constant (slight dip) for increasing plating time. Almost constant current efficiency is observed, indicating constant electrochemical reactions occurring at anode and cathode. This shows that plating time will have a direct affect on coating thickness.




a) b)

Fig 4: Coating Weight and Cathode Current Efficiency variation with plating time for a) Cu sample and b) Ni plated Cu sample.

The coating thickness increases linearly with an increase in time. Theoretical coating thickness is calculated using the formula;

$$T=(I \times t \times A \times 10000)/(n \times F \times rho \times S) \qquad \text{eqn (2)}$$

Where, T = Coating Thickness in µm, I = current in coulombs per second, t = time in seconds, A = atomic weight of the metal in g/ mol, n = valence of the dissolved metal in the solution, F= Faraday's constant, rho = density in g/cc and S = surface area of the plated part.

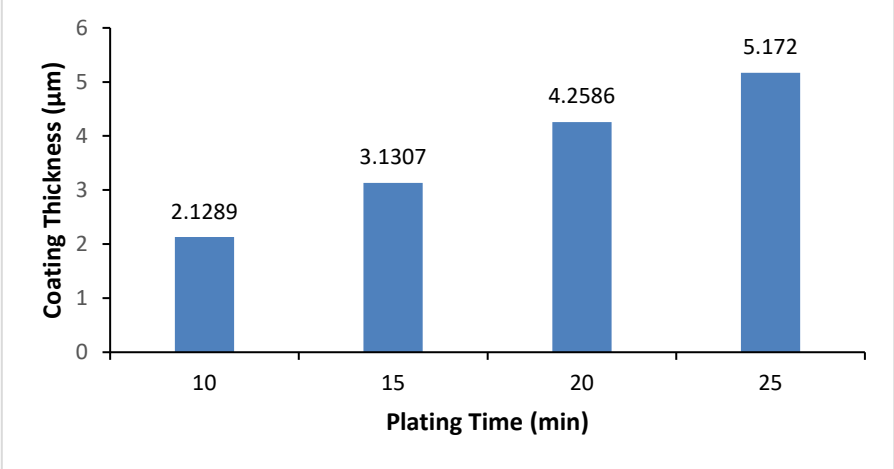
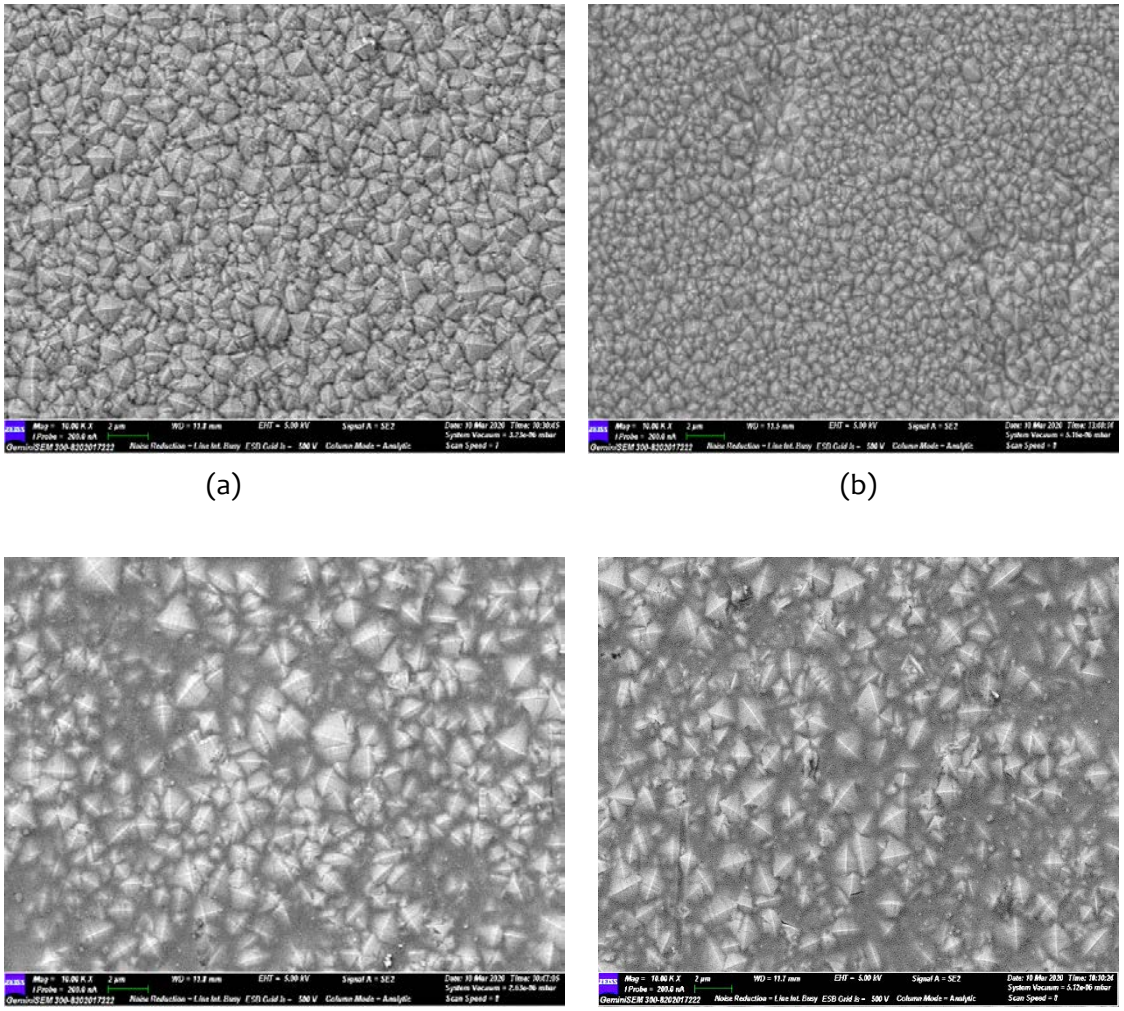



Fig 5: Coating thickness variation with plating time

It is observed from figure 4 that coating thickness increases almost linearly with plating time, attributing to the fact that amount of nickel material deposited increases almost linearly with plating time. There is an almost 60% increase in the value of coating thickness for a plating time of 25 minutes compared to the coating thickness for a plating time of 10 minutes.



(a) (b)

c) d)

Fig 6: Surface Morphology of Ni plating at 1000X magnification for a) 10 min, b) 15 min, c) 20 min, and 25 min

The morphology due to the electroplating method is observed to be of porous nature suggesting a self-lubricating property. Advocating for the widespread use of electrodeposition as a coating method. Plating process is because of germination happening due to the roughness of the substrate. Ni + ions are deposited on the substrate layer by layer forming congresses that are similar to coffee bean structure.

Time has a direct effect on coating thickness. The thickness of the coating increases linearly with time. The coating is desired to have a high degree of quality and uniformity. Low coating thickness variation across the surface is desired. Increase in plating time increases the thickness of coating because the base plate resistance to deposition decreases as the coating is progressed.

The metallurgical area of the coating can be divided into primary and secondary nodule regions. The secondary nodules grow like coffee bean structure, ups, and downs of level increases by increasing the plating time. This leads to increased surface roughness. But this results in an increase in coating hardness and corrosion resistance. With the increase in plating time, suitable places will be more to grow the structure of secondary nodules. Increasing the growth time leads to stagnation of the germination locations leading to more coarse structures.

The nickel coating is observed to be stable for 10 min and 15 min plating time from the figure. The

coating consists of small, roughly spherical primary and secondary nodules. Microstructures of 20 min and 25 min coated samples reveal, large secondary nodules forming truncated pyramid structures with rigid terrain oriented perpendicular to the growth direction. The variation in surface topography of coating is roughly on the order of the size of the clusters. This property enhances the optical behaviour of the coating by minimizing first surface reflection at the film/ air boundary.

**CONCLUSION**

The following conclusions are drawn from the experimentation and results;

Coatings of Ni developed were observed to be of nanocrystalline and porous in nature, indicating self-lubricating property.

The coating rate and coating thickness increases almost linearly with plating time.

Cathode current efficiency almost remains constant throughout, indicating constant electrochemical reactions at anode and cathode.

The metallurgical area of the coating can be divided into primary and secondary nodule regions.

Nickel coating is observed to be stable for 10 min and 15 min plating time, consisting of small, roughly spherical primary and secondary nodules. Microstructures of 20 min and 25 min coated samples reveal large secondary nodule forming truncated pyramid structures with rigid terrain oriented perpendicular to the growth direction.

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