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RESEARCH ARTICLE

EFFECT OF CU PARTICLES CONTENT ON THE SURFACE MORPHOLOGY AND MECHANICAL PROPERTIES OF NI-CU COMPOSITE COATING

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Abstract. Nickel (Ni) coating is commonly used in many industrial applications. Still, the challenge has recently been encountered when Ni coatings experience aggressive stress and load, while their durability may not be sufficient to maintain performance in such conditions. Thus, Ni composite coating offers significant improvement in terms of mechanical properties and wear resistance. Therefore, in this study, metallic particles, such as copper (Cu) particles, can be added to Ni composite coating to refine the coating structure while increasing its durability. However, the conductivity of Cu particles in the electrolyte may influence the surface morphology while contributing to increasing the hardness of the composite coating. Thus, this study aims to determine how optimal Cu particle concentration influences coating surface morphology and hardness. The Cu particles in a range of 0.1–5 g/L were deposited in the Ni matrix, and the growth of composite coating was observed based on deposition times of 5, 10, 20 and 30 minutes. The coated surfaces were examined by surface morphological analysis, and mechanical properties were determined through microhardness testing. The result showed that the formation of a broccoli-like structure increased as the concentration of Cu particles in the Ni composite coating increased. The result also revealed that the higher hardness of Ni-Cu composite coating was achieved when 1 g/L of Cu was loaded into the electrolyte bath. Meanwhile, based on a growth study, Ni ions prefer to be deposited on the carbon steel substrate and at the Cu particles at the early stage of 10 mins. Subsequently, as time increased, Ni-coated Cu formed an accumulated, broccoli-like structure and was incorporated into the growing Ni deposits.

Keywords: Electrodeposition, composite coatings.

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1. INTRODUCTION

Nickel (Ni) coating is a metallic coating that commonly deposits a thin layer of Ni onto a metal substrate. Over the years, electrodeposited Ni coating has become the most common method due to its cost-effectiveness, simplicity of operation, and ease of controlling the microstructure. Notably, electrodeposited Ni coatings are typically deposited on steel and alloys for both surface protection and decorative purposes. Therefore, Ni coating has a wide range of applications in various industrial sectors, including automotive, manufacturing, machining, electronics, and many others. However, the challenge lies in determining when Ni coatings experience aggressive stress and load, while their durability may need to be improved to maintain performance in such conditions. To date, Ni composite coating has provided an alternative to overcome the limitations of Ni coating. Ni composite coating includes two or more materials; Ni itself serves as the matrix, and a co-depositing particle is used as reinforcement. The co-depositing particles are loaded on an electrolyte bath and integrated into the growing metallic Ni during the electrodeposition process. Matrix performance in some applications is improved by co-deposition.

The physical, mechanical, and tribological properties of Ni composite coating depend on the co-depositing particles during the electrodeposition process. Co-depositing particles are generally categorized into two types based on their properties: hard particles and soft particles. Hard particles (SiC, Al_2O_3, W, WC) refer to the particles that enhance the coating's hardness, durability and corrosion resistance. In contrast, soft particles (such as MoS_2, PTFE, and graphite) enhance the wear performance of the coating by providing self-lubricating effects within the coating system. However, among them, the particles can also be classified as conductive, including carbon nanotubes (CNTs), graphene, graphite, tungsten disulfide (WS_2), and many others. In addition, the co-deposition of conductive particles altered the surface morphology of the coating, thereby enhancing its durability, as reported by Long et al. [1] conductive particles enhance the electrodeposition process, promoting more effective current distribution due to the high electric current density induced by these particles. They also mentioned that the conductive particles facilitate better coating growth and increase the coating hardness. However, due to this characteristic, the co-deposition of conductive particles using electrodeposition presents its challenges. Unlike inert particles, conductive particles tend to increase the electric current density, accelerating Ni ions to be deposited around them. Consequently, the coating's surface morphology demonstrates protrusion microstructures [2].

Furthermore, the coating morphology is substantially affected by the increase in the concentration of conductive particles in the electrolyte bath. Numerous investigations have demonstrated that the microstructure formation on the coating surface becomes inconsistent, irregular, and non-dispersive as the concentration of conductive particles increases [3-5]. Xue et al. [6] fabricated CNT/Ni composite coating under severe conditions. They discovered that the nodular-like structure was vertically growing. However, further studies are needed regarding the obtained structure and coating hardness. He et al.[7] demonstrated that Ni-P-WS_2 exhibits a hierarchical, broccoli-like structure, which contributes to achieving a hardness of 720 HV. This finding indicates that the hardness of the nickel composite coating is enhanced by the dispersive distribution of WS_2 and the refinement of the grain structure. Varadaraj et al. [5] fabricated and investigated the effect of graphene content on the surface morphology of Ni-graphene coatings, finding that the coatings exhibited protrusion bulge structures that dispersed unevenly on the surface. They also reported an increase in coating hardness due to the uniform distribution of graphene in the Ni composite coating and grain refinement at the grain boundary, which resisted grain dislocation. Other studies reported [8,9] by adding conductive particles to the Ni composite coating, they also observed a similar surface morphology. In contrast, the structure was vertically grown, forming a unique protrusion structure on the coating surface. Conductive particles have been emphasized in all the literature as a method for improving the durability of nickel composite coatings. However, no extensive research has been conducted in the literature on the relationship between conductive particle concentration and mechanical properties.

Copper (Cu) possesses composite coating properties that enhance hardness while also being lightweight, durable, and an excellent conductor of electricity. Previously, numerous efforts have focused on incorporating Cu particles to enhance the coating's durability. Rahmati et al. [10] fabricated an Al-Mg-Cu coating and investigated the effect of Cu particles on the composite coating. Incorporating Cu particles into the coating led to an increase in its tensile strength and hardness. Anijdan et al. [11] synthesized Ni-P-Cu composite coating by controlling the coating parameters. They suggested that the coating has a higher concentration of Cu particles, which improves the coating's tribology and enhances its durability, but they did not emphasize the coating's hardness. Thus, the addition of Cu particles improves coating durability. Nevertheless, the study regarding the addition of Cu particles in electrodeposited nickel composite coatings is still unfolding.

Hence, Cu particles were incorporated into a Ni composite coating in this investigation through an electrodeposition process. Additionally, this study aims to clarify the effect of conductivity by modifying the Cu particle concentrations in hardening the Ni matrix of the composite coating. Additionally, the optimal concentration of Cu particles was used to investigate the growth of the nickel composite coating. As for future research directions, this study has discovered the potential of Cu particles to act as a solid lubricant in Ni composite coatings, thereby enhancing tribological performance.

2. MATERIALS AND METHODS

2.1 Materials

A carbon steel alloy (AISI 1045) was used as the substrate, purchased from Misumi Malaysia Sdn. Bhd. Figure 1(a) illustrates that the substrate is a circular disc with an outer diameter of 25 mm, a 4 mm thickness, and a centrally drilled hole of 4 mm. All the substrates were mechanically grounded with silicon carbide (SiC) abrasive paper 100, 240, 600, 1000, 2000, 3000 and 5000, followed by diamond slurry polishing to obtain a surface roughness of 0.08 µm. To enhance the adhesion layer of the coating on the substrate surface and eliminate the oxide layer, the substrate surface was activated with diluted HNO_3 (5%) prior to the deposition procedure. Cu particles used as reinforcement in the coating were obtained from Changsha Advanced Engineering Materials Limited. The average size of the Cu particles was ~ 1 µm, and they had a purity of 99.60%. Figure 1(b) shows the surface morphology of the as-received Cu particle.

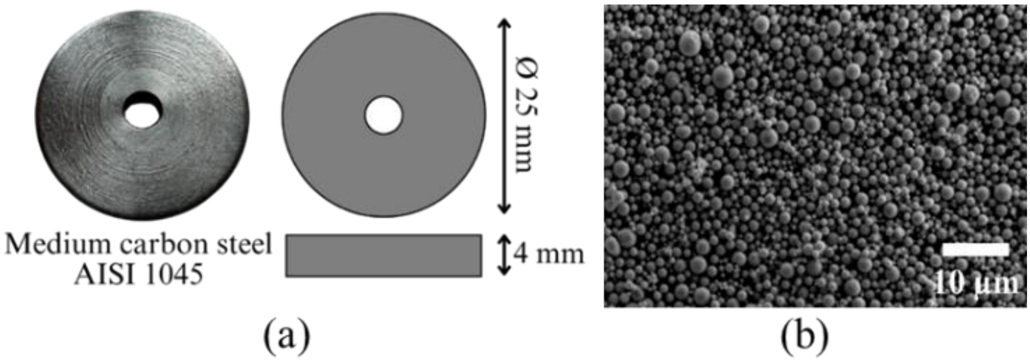


Figure 1: (a) Geometry of the carbon steel and (b) SEM image of Cu particles (average size of 1 µm)

2.2 Electrodeposition Process

A Watts bath composition is used to deposit Ni composite coatings. This study employed a bath chemical composition consisting of nickel (II) sulfate hexahydrate ($NiSO_4.6H_2O$) (488 g/L), nickel (II) chloride hexahydrate ($NiCl_2.6H_2O$) (30 g/L), boric acid (H_3BO_3) (25 g/L) and sodium dodecyl sulfate (SDS) (0.1 g/L). The electrolyte was agitated using a magnetic stirrer for 30 minutes until it was fully dissolved. The bath operation system was operated at temperatures ranging from 25 °C to 30 °C, a pH of 4, and a current density of 3 A/dm². A Ni pure sheet was used as an anode, and carbon steel was used as a cathode.

In this study, the Ni composite coating was synthesized in two phases. In the first phase, substrates were electrodeposited with varying Cu particle contents (0.1, 0.5, 1, and 5 g/L) in the electrolyte bath for 30 minutes to clarify the influence of Cu content on the coating morphology and mechanical properties. Meanwhile, at this stage, an ideal copper content was also determined by evaluating the characteristics of the coating structure and its improved mechanical properties. Afterwards, the study continued with different deposition times: 5, 10, 20 and 30 minutes at the ideal Cu content in an electrolyte bath. The purpose of the latter stage is to elucidate the growth behaviours of the composite coating.

2.3 Coating Characterization

The Ni composite coatings' surface morphology and elemental analysis were analyzed using a scanning electron microscope (SEM) (JEOL, JSM-6010PLUS/LA) that was integrated with an energy-dispersed x-ray spectrometer (EDS) (Oxford Instruments NanoAnalysis Plc). The surface roughness of the coatings was examined using a 3D optical profiler, ZeGageTM (Zygo Corporation and Ultra Precision Technology Division of Ametek Inc).

The structural changes of Ni-Cu composite coating were quantified using X-ray diffraction analysis (XRD) (PW 3040/60 MPD X'PERT High Pro Panalytical). The XRD patterns were identified in a range of $2\theta = 20°$ to $80°$ using Cu-Kα radiation at 1.54 Å. The reinforcement particles are predicted to change the grain size of the nickel composite coating [7]. Therefore, the crystallite size was calculated based on Scherrer's Equation as follow: [12].

$$D = \frac{k\lambda}{\beta \cos\theta} \tag{1}$$

In this equation, k is the Scherrer constant (0.9); λ is the wavelength of Cu-Kα x-ray (0.154 nm); β is the full width at half maximum (FWHM); θ is the diffraction angle at the position peak.

Finally, the HMV-Micro Vickers hardness (Shimadzu Corporation) was employed to determine the hardness of composite coatings. The penetration time was 10 seconds, and the test load was 490.3 mN using the regular quadrangular pyramid diamond indenter. The Vickers hardness values (HV) were converted into gigapascals (GPa).

3. RESULTS AND DISCUSSION

3.1 Surface Morphology and Chemical Composition

The SEM micrographs of fabricated pure Ni coating and Ni-Cu composite coatings, which contain varying amounts of Cu particles in an electrolyte bath, are shown in Figure 2. The microstructure of the pure Ni coating is illustrated in Figure 2(a). As can be seen, the coating's surface morphology is characterized by a uniform and dense nodular structure. The formation of this structure can be attributed to the selection of a low current density range during the deposition process [13].

At 0.1 g/L of Cu particles, the coating exhibits small and irregular growth randomly distributed on the coating surface, as shown in Figure 2(b,c). Further increase of 0.5 g/L of Cu particles to the electrolyte bath, the irregular growth expands and develops a rough morphology, whereas it starts to show broccoli-like structure formation, as presented in Figure 2(e,f). These broccoli-like structures are composed of various blocks of irregularly grown Ni that accumulate together. Interestingly, the hierarchical broccoli-like structure in the Ni-Cu coating was found to be larger and more prominent when 1.0 g/L of Cu particles were added to the electrolyte bath, as shown in Figure 2(h,i).

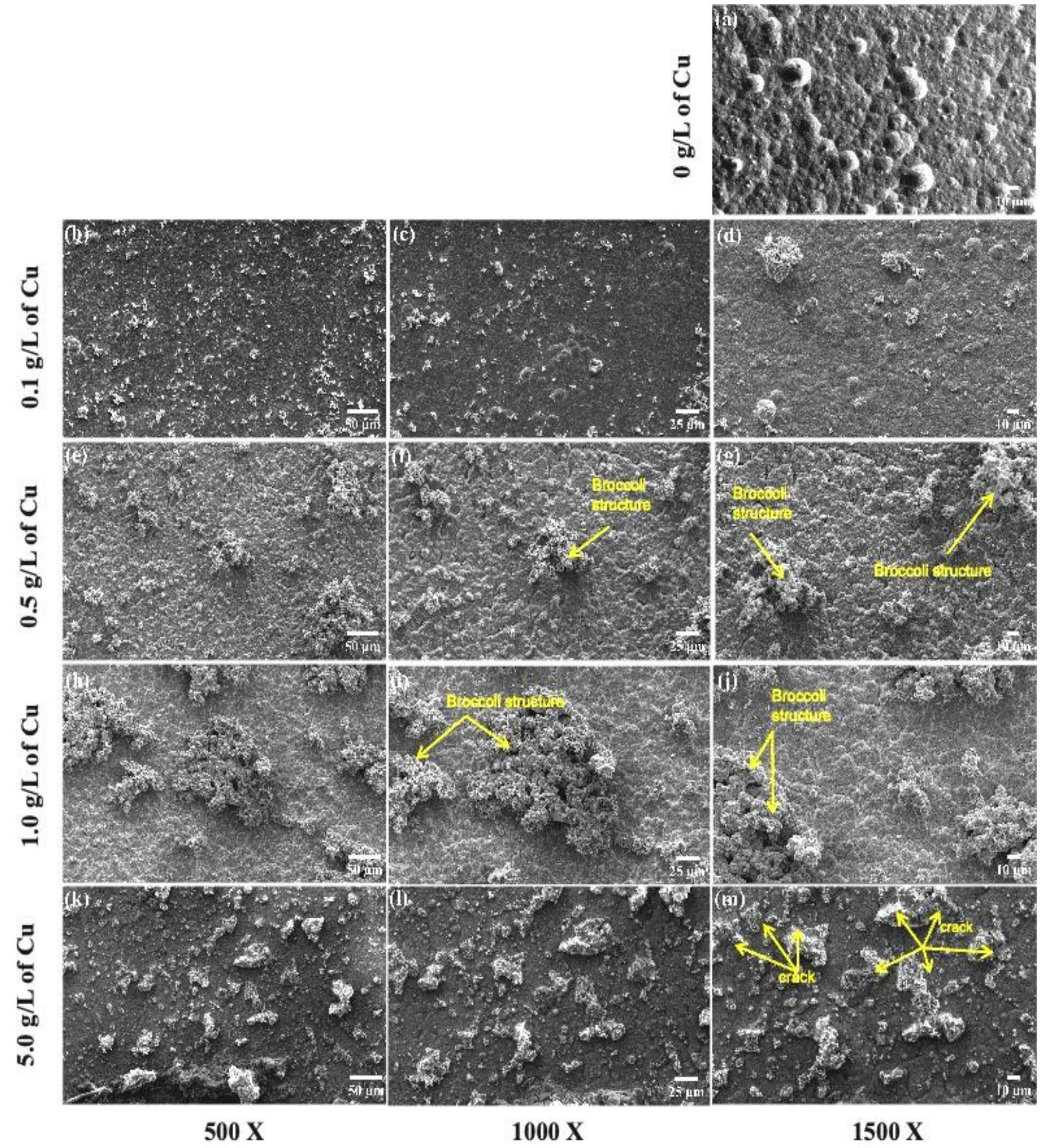
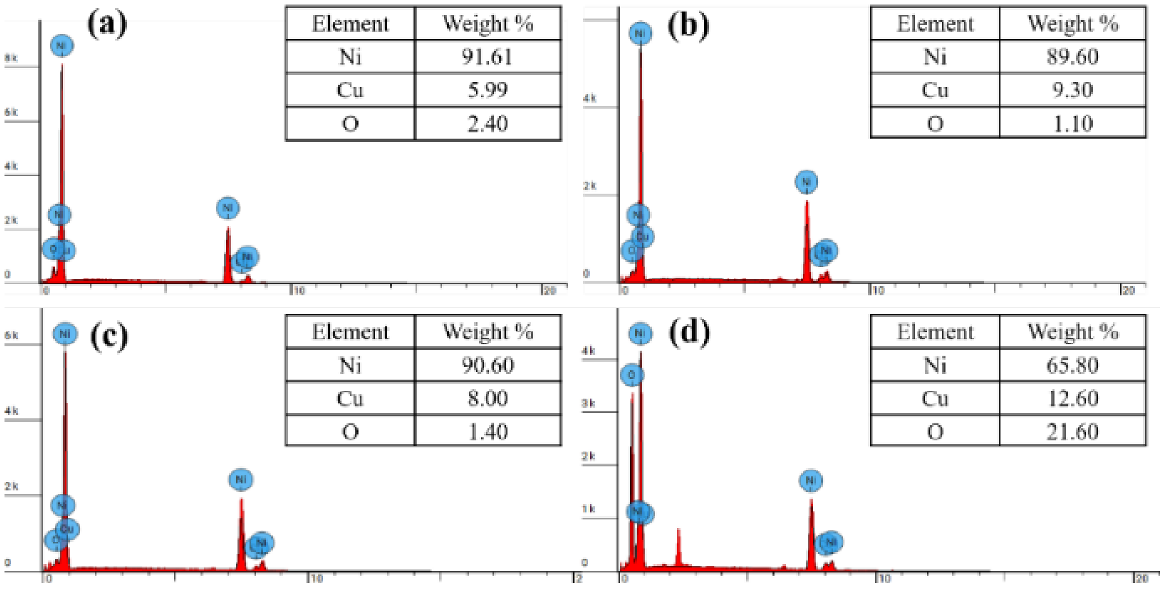


Figure 2: Surface morphologies of Ni-Cu composite coatings at different Cu particle contents in the electrolyte bath observed to varying magnifications of 500X, 1000X and 1500X : (a) 0 g/L, (b, c, d) 0.1 g/L, (e, f, g) 0.5 g/L, (h, i, j) 1 g/L and (k, l, m) 5 g/L

The growth mechanism of Ni composite coating with conductive particles was explained by He et al. [7] and was adapted in this study. Initially, the Ni ions were reduced at the cathode surface, forming planar Ni growth. However, when Cu particles are loaded into the electrolyte bath, they are mechanically transported and absorbed onto the cathode surface. Due to the high conductivity of Cu particles, the current distribution increased, promoting more active sites for reducing Ni^{2+} ions. Therefore, Cu particles create more nucleation sites which retarding Ni crystal growth, then smaller crystal grains are formed. Thus, these smaller crystal grains play a role in strengthening the composite coating.

Regarding the surface morphology, He et al. [7] suggested that this arises from a localized redistribution of current, which causes the Ni ions to preferentially reduce on raised regions and form a hierarchical structure resembling a broccoli-like shape. Essentially, the incorporation of conductive particles in Ni composite coatings can lead to two types of structure formation, known as protrusion and hierarchical structure, which resemble a broccoli-like or cauliflower-like shape. The structure formation depends on coating parameters such as deposition time and current density [14]. For instance, the surface morphology of the hierarchical structure can be found by adding WS_2 [7], and protrusion structures can be identified by adding MoS_2 [3], CNT [4], and graphene [14]. However, based on this study, it is suggested that the high conductivity of Cu particles (5.977×10^5 S/cm [15]) contributes to the formation of a hierarchical structure, even though the deposition time is shorter compared to other studies [3,4,7]. Conversely, when the Cu particle content reaches 5 g/L, the Cu particles are considered excessive, as they cannot disperse perfectly in the electrolyte, resulting in particle agglomeration. This condition prevents Ni from growing on Cu particles, resulting in a flatter region that dominates when Ni is grown on the cathode surface. Meanwhile, the hydrogen evolution reaction also occurred, as evidenced by gaps and cracks on the coating surface, as shown in Figure 2(m). Thus, this is considered coating degradation.

The results reveal that the pure-Ni and Ni-Cu composite coatings were successfully fabricated, and that Cu is present in the coating composition. The EDS analysis provides information on the weight percentage (wt.%) of Cu in the composite coating, as well as the proportions of other elements. Therefore, as shown in Figure 3, the wt.% of Cu increased accordingly due to the increase in Cu content in the electrolyte bath. Another finding from the EDS analysis indicated the presence of O , suggesting that the Cu particles may have undergone oxidation during electrodeposition. Additionally, changes in the wt.% of Ni and O are correlated with an increase in Cu content. Furthermore, following the EDS analysis, the presence of Ni and O in the broccoli-like structure indicates that Ni had completely covered the Cu particles, confirming that the Ni composite coating was successfully fabricated.



(a)

Element	Weight %
Ni	91.61
Cu	5.99
O	2.40

(b)

Element	Weight %
Ni	89.60
Cu	9.30
O	1.10

(c)

Element	Weight %
Ni	90.60
Cu	8.00
O	1.40

(d)

Element	Weight %
Ni	65.80
Cu	12.60
O	21.60

Figure 3: EDS spectra and chemical composition of Ni-Cu composite coatings at different Cu particle contents in the electrolyte bath: (a) 0.1 g/L, (b) 0.5 g/L, (c) 1 g/L, and (d) 5 g/L

3.2 Surface Roughness

Surface roughness plays a significant role in determining the coating features, such as the formation of a layer coating and mechanical and tribological properties. In this study, the average surface roughness of pure Ni coating and Ni-Cu composite coating with varying Cu particle content in the electrolyte is depicted in Figure 4. As can be seen, the pure-Ni coating demonstrated the lowest surface roughness, with an average Ra value of 0.36 ± 0.06 µm. In contrast, when the Ni-Cu composite coatings were fabricated at varying Cu particle content levels in the electrolyte bath, specifically 0.1, 0.5, 1, and 5 g/L. These coatings exhibited roughness of 0.52 ± 0.18 µm, 2.11 ± 1.3 µm, 5.94 ± 1.3 µm, and 4.63 ± 1.98 µm, respectively. Therefore, the coarseness of the Ni-Cu composite coating is consistent with the surface morphology images presented in Figure 2 (d, g, j). The coarseness of surface roughness is attributed to the incorporation of Cu particles, which leads to the formation of a hierarchical, broccoli-like structure on the surface coatings. The microscopic surface roughness features also play a significant role in mechanical and tribological performance as well as corrosion resistance. Liu et al. and He et al. fabricated a Ni-W-WS_2 composite coating and found that a hierarchical structure is beneficial for corrosion resistance [4,7]. They suggested that the hierarchical structure functions as an air trap, preventing a corrosive environment and thereby enhancing corrosion resistance. Maharana et al. fabricated Ni-MoS_2 and claimed that a rougher surface reduces the friction coefficient. They suggested that a rougher surface with MoS_2 works as a barrier between mating contacts in reducing friction during the sliding test [16]. In comparison to previous research findings, the hierarchical broccoli-like structure in this study is expected to enhance tribological and corrosion resistance.

However, the surface roughness is decreased when the highest content of Cu is added. This is because mostly the coating structure is not grown in a broccoli-like structure and has more flat coating surface areas compared to others, as shown in the SEM images in Figure 2 (k,l). Furthermore, this finding confirmed the formation of a hierarchical broccoli-like structure, which can be observed when the highest Cu particle content is subjected to the electrolyte bath, up to 1 g/L of Cu particles. Moreover, the graph also showed a more significant standard deviation in the Ni-Cu composite coating compared to the pure Ni coating due to the uneven size distribution of particles and the presence of agglomerated particles on the surface coating.

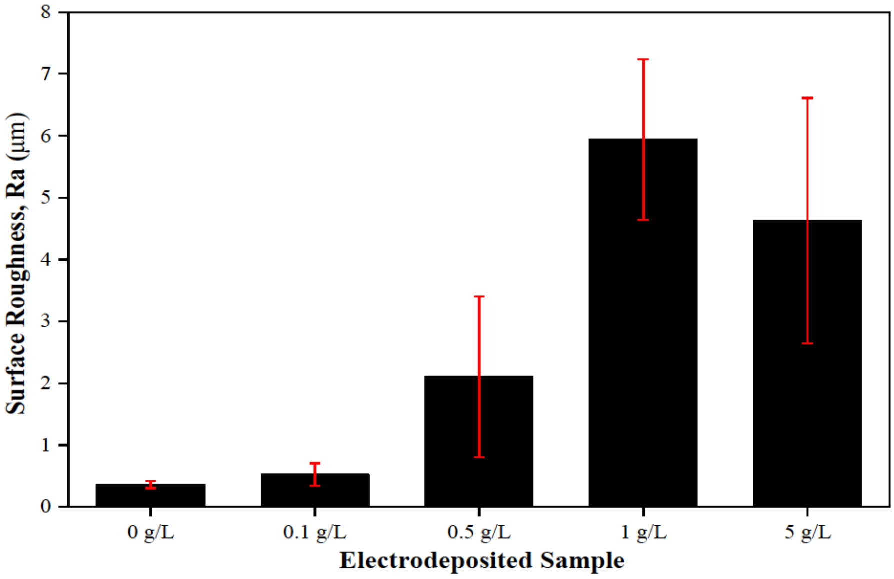


Figure 4: Surface roughness of pure-Ni coating and Ni-Cu composite coating with 0, 0.1, 0.5, 1 and 5 g/L Cu particles in the electrolyte by using direct current (DC)

3.3 Mechanical Properties of Ni-Cu Composite Coating

The microhardness of the pure Ni coating and the Ni-Cu composite coating is shown in Figure 5. The lowest microhardness values, 3.43 ± 0.05 GPa, were observed in the pure Ni coating, as determined by the measurements. Meanwhile, the microhardness values of the Ni-Cu composite coatings increased slightly from 4.02 ± 0.09 GPa to 5.96 ± 0.78 GPa, with an increase of Cu particle content in the electrolyte bath from 0.1 to 1 g/L. Based on the hardness results, it is evident that Cu particles play a significant role in strengthening the Ni-Cu composite coating. Previous studies with similar trends suggested that conductive particles in Ni composite coating strengthen the coating through grain refinement [4,5,7,12]. Therefore, in this study, we believe that the higher conductivity of Cu particles contributed to strengthening the composite coating. They can be part of the surface cathode or referred to as the secondary cathode, which creates new nucleation sites on the surface of the Cu particles and inhibits crystal growth, resulting in grain refinement. Therefore, increasing the number of grain boundaries, which obstructs dislocation motion, led to enhanced hardness. Figure 2 (a,d,g,j) shows the comparison of the bare base of Ni-Cu composite coatings with different Cu contents. As can be seen, the grain size of the bare Ni base decreases as the Cu content increases. It was reported by Mousavi et al. that soft and conductive particles have a significant influence in strengthening Ni composite coating [17]. However, when the Cu particles reached their highest concentration in the electrolyte bath, they agglomerated, and small dispersive grain clusters appeared on the coating surface. Hence, during the indenter-applied load, the coating is unable to withstand the plastic deformation, resulting in a decrease in the coating's hardness. On the other hand, the appearance of cracks also contributed to weakening the coating structure.

A concentration of 1 g/L of Cu particles has been chosen for further investigation in this study due to two potential factors, as determined by an analysis of surface morphology, chemical composition, surface roughness, and mechanical properties. Firstly, the surface morphology of 1 g/L Cu appears to have a roughened surface, suggesting that the coating structure has a broccoli-like structure formation. This coating structure was consistent with previous findings [4,7,8].

Secondly, based on mechanical properties, it has been demonstrated that a 1 g/L concentration of Cu particles exhibits higher hardness than other coatings. This also strongly indicates the presence of Cu particles at 1 g/L in the electrolyte, creating new nucleation sites and refining the grain size, which contributes to strengthening the composite coating. Therefore, 1 g/L of Cu particle is expected to be the optimum concentration for understanding coating growth behaviour and enhancing the hardness of the composite coating, which will be discussed further in the investigation. The study reveals that enhanced hardness is achieved with the incorporation of Cu in Ni composite coatings. The high hardness of the composite coating prevents plastic deformation, thereby resisting abrasive and adhesive wear. Therefore, this hard coating will increase wear resistance and is believed to have long-term durability. Thus, it is suitable for harsh conditions and applicable to a wide range of applications.

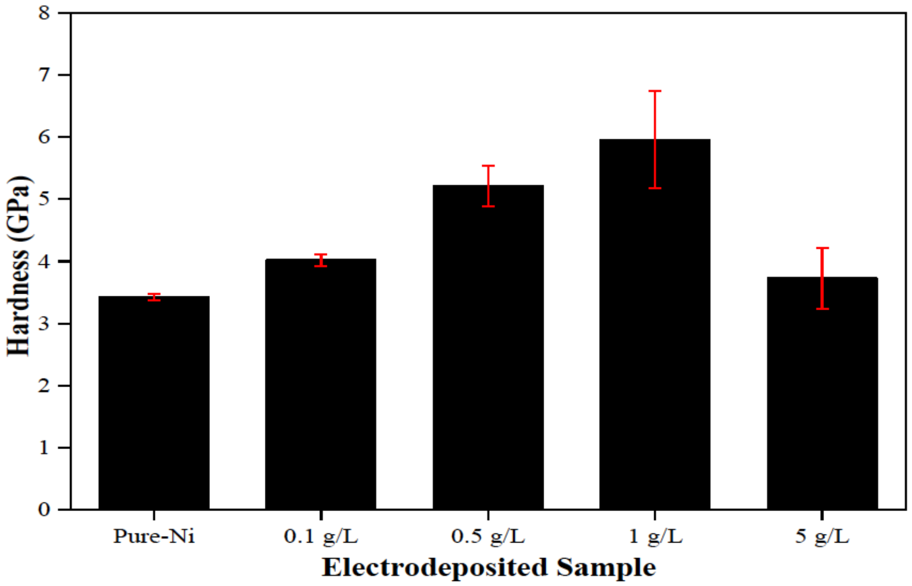


Figure 5: Surface hardness of the pure-Ni coating and Ni-Cu composite coating with 0, 0.1, 0.5, 1 and 5 g/L Cu particles in the electrolyte by using direct current (DC) electrodeposition

3.4 Effect of Copper on Nucleation and Crystallize Size of Ni-Cu Composite Coating (1 g/L)

Further investigations were conducted using XRD to identify the structural changes in the Ni coating resulting from the addition of 1 g/L of Cu particles to the electrolyte bath. In Figure 6, the diffraction peaks were observed at 43.82°, 51.28°, and 75.91°, which correspond to the (111), (200), and (220) orientations of the Ni matrix [18]. The Cu peak near 42.65° was observed once the Cu particles were incorporated into the Ni matrix, and this is consistent with the previous literature [19]. It is evident that the incorporation of Cu particles in the electrolyte significantly increases the relative intensity of the (111) plane and decreases the intensity of the (200) plane.

By incorporating particles into the Ni matrix, this finding reveals that the preferred orientation of the coating during growth was altered [5]. Furthermore, the broader peaks proved the reduction in grain size, which may be attributed to the Cu particles hindering grain growth by initiating new nucleation sites. Meanwhile, it can also be seen that the weak peak of the CuO phase is observed near 64.47°, which is consistent with relevant research [20]. This finding is also consistent with the presence of O in EDS analysis, indicating that Cu may be generated through the partial oxidation of Cu particles during electrodeposition or when the final coating is exposed to air. However, since the wt.% of O is too low and the intensity of CuO is too weak in the XRD spectrum, it suggests that the oxidation effect is minimal and has not significantly influenced the mechanical and tribological properties of this coating. Further investigation to confirm the effects of the CuO phase on coating properties will be discussed in the next section, focusing on the distribution of elemental composition.

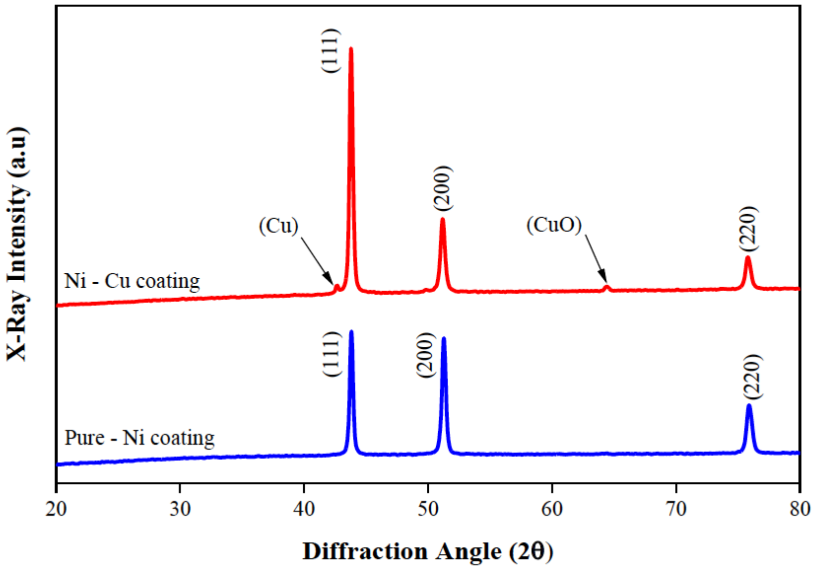


Figure 6: X-ray diffraction pattern of pure-Ni coating and Ni-Cu composite coating (1 g/L of Cu particles in the electrolyte bath)

Table 1 presents the calculated average crystallite size, which further confirms the grain size of the composite coating. The pure Ni and Ni-Cu composite coatings have average crystallite sizes of 22.25 nm and 19.62 nm, respectively. This reduction in crystallite size is also consistent with the previous change in surface morphology in Figure 2 (d,g,j). The hardness of the composite coating is expected to improve due to the decrease in grain size, a topic elaborated upon in the following section.

Table 1: Scherrer's equation parameter of pure-Ni and Ni-Cu composite coating

Sample	2θ	h k l plane	FHWM	L (nm)	Average L (nm)
pure-Ni coating	43.82	111	0.34323	26.08	22.25
	51.28	200	0.40551	22.71	
	75.91	220	0.58615	17.97	
Ni-Cu composite coating	43.80	111	0.34609	25.86	19.62
	51.18	200	0.48614	18.94	
	75.82	220	0.74772	14.08	

3.5 Coating Growth Behaviour of Ni-Cu Composite Coating of 1 g/L Cu Particles in Electrolyte

A comprehensive view of the growth behaviour of a Ni-Cu composite coating (electrolyte bath containing 1 g/L Cu) is presented in Figure 7 through the SEM image and 3D surface topography. The deposition time is set to 0, 5, 10, 20, and 30 minutes. In Figure 7 (a), the morphology of etched carbon steel has a roughened surface, similar to the morphology appearance of activated steel. However, the morphology of the Ni-Cu composite coating appears to change at different times.

As shown in Figure 7 (c), within 5 minutes of deposition, the surface becomes smooth and flat due to the planar growth of Ni. At 10 minutes of deposition, Cu absorption occurs on the cathode surface. Due to the high conductivity of Cu, it enhances current distribution, creating more nucleation sites for Ni ion reduction, as shown in Figure 7 (f). When the deposition reaches 20 minutes, the growth of the Ni-Cu composite coating is shown in Figure 7 (i). During this time, the coating starts to develop smaller protrusion structures. Here, the rise in local current distribution of Cu particles causes Ni ions to reduce at raised regions, resulting in a growing structure that resembles broccoli-like structures. As the deposition time reaches 30 minutes in Figure 7 (m), the irregular and smaller broccoli-like structures grow larger and form hierarchical broccoli-like structures. The denser and larger formation of hierarchical broccoli-like structures is more prominent at this stage.

Furthermore, the size of agglomeration peaks was recorded, as shown in Figure 7 (e,h,k,n). At 5 minutes of deposition, only a few agglomeration peaks were examined, with a width of 4.6 µm and a height of 7.8 µm. However, the number of peaks spotted is few compared to others' deposition times. This agglomeration peak became broader and higher as the deposition time increased. The agglomeration peaks are due to the hierarchical growth of the broccoli-like structures, which reach widths of up to 48.9 µm and heights of 36.0 µm.

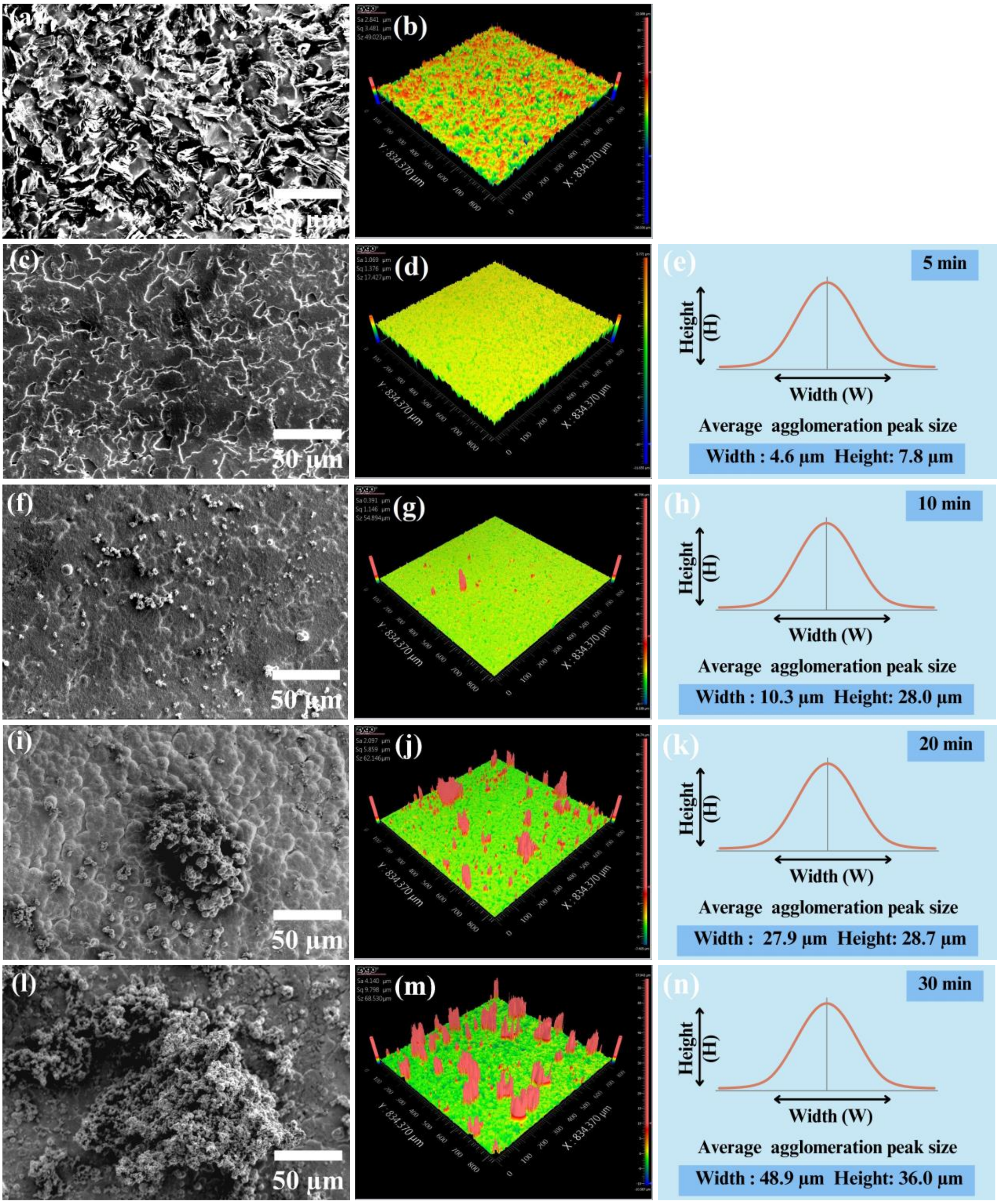


Figure 7: Surface morphologies, 3D surface profile and agglomeration peak size Ni-Cu composite coatings at 1 g/L of Cu particles content in the electrolyte bath at different electrodeposition time: (a, b) 0 min, (c, d, e) 5 min, (f, g, h) 10 min, (i, j, k) 20 min and (l, m, n) 30 mins

3.5 Cross-section Analysis of Ni-Cu Composite Coating of 1 g/L Cu Particles in Electrolyte

Figure 8 presents the cross-sectional image of a Ni-Cu composite coating with 1 g/L of Cu particles in the electrolyte. The coating exhibits a broccoli-like structure and features a strong bonding interface with the substrate. The element distribution maps indicate the successful fabrication of a Ni-Cu composite coating, in which Ni and Cu are uniformly distributed across the coating surface.

However, the presence of O is also detected in the areas surrounding the resin and substrate. The presence of O may be attributed to oxidation reactions that occur during sample preparation.

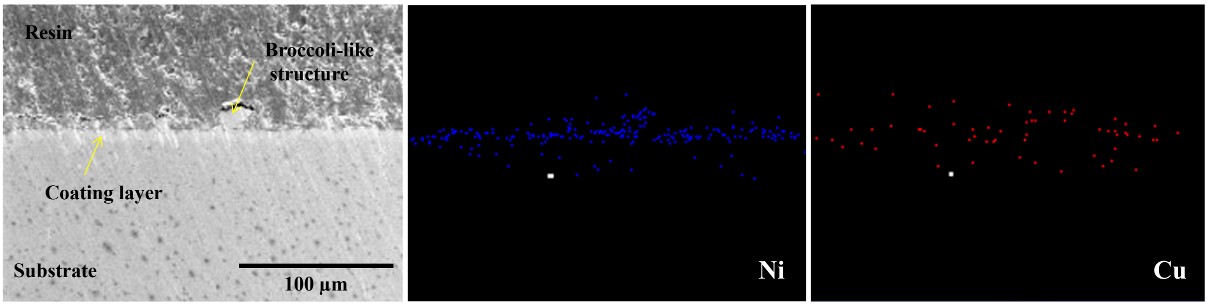


Figure 8: Cross-sectional elemental mapping of Ni-Cu composite coating of 1 g/L Cu particles in electrolyte

4. CONCLUSIONS

Electrodeposition has been employed to fabricate pure-Ni coatings and Ni-Cu composite coatings with the addition of Cu particles on carbon steel substrates. The investigation examined the effect of the concentration of Cu particles in an electrolyte bath on mechanical properties, chemical composition, and surface morphology. Therefore, the results of this study may be summarized as follows:

- The Ni-Cu composite coating structure is significantly influenced by the addition of Cu particles to an electrolyte bath. The Ni-Cu composite coating developed a broccoli-like structure surface at concentrations of 0.5 and 1 g/L of Cu particles. However, 1 g/L of Cu was present in an hierarchical broccoli-like structure in the composite coating without any defects or cracks. In contrast, 5 g/L of Cu particles resulted in degradation of the layer, characterized by gaps and cracks.
- The higher hardness of the Ni-Cu composite coating was achieved when 1 g/L of Cu was added to the electrolyte. This was attributed to Cu particles refining the grain size, as confirmed by the crystallite size determined from the XRD result.
- Based on a growth study, Ni ions were found to preferentially deposit on the substrate and at the Cu particles during the initial 10-minute stage. Subsequently, as time progressed, Ni-coated Cu began to form an accumulated, hierarchical broccoli-like structure and was incorporated into the growing Ni deposits.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work does not require any ethical procedures.

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