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Influence of Glucose on the Microstructure of Electrodeposited Nickel-Al_2O_3 Nanoparticles and the Corrosion Resistance of Low-carbon Steel

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ABSTRACT

The purpose of coating steel surfaces is to enhance their properties, a challenge that continues to attract scientific interest. Selecting an appropriate coating involves choosing the most suitable method and optimizing process conditions to achieve the desired surface characteristics. Within this context, the present study investigates the effect of glucose on the microstructure of nickel coatings electrodeposited on low-carbon steel in the presence of Al_2O_3 nanoparticles. Glucose was selected as an alternative to saccharin, a commonly used additive in nickel electroplating. The coating was applied using electro-deposition in a Watts bath, and its structural characteristics were analyzed using scanning electron microscopy (SEM) and X-ray diffraction (XRD). Corrosion resistance was evaluated via voltammetric measurements in a 3.5% NaCl solution. The results revealed that nickel particles formed along a preferred crystallographic orientation, and the incorporation of glucose altered the particle morphology, leading to the formation of pyramid-shaped structures at a concentration of 2 g/L. Furthermore, the addition of glucose significantly enhanced the corrosion resistance of the coating, achieving a maximum polarization resistance (Rp) of 43.86 kΩ·cm².

Keywords:
Electro-deposition, Steel, Nickel, Glucose, Microstructure, Corrosion Behavior

1. Introduction

Nickel electroplating is one of the most widely used coating techniques in the electroplating industry, with applications in aeronautics, electronics, micro- and nanofabrication, food processing, and jewellery, particularly for decorative purposes [1]. It is highly regarded for its excellent resistance to wear and corrosion [2–5], and has attracted significant interest as a feasible, cost-effective, and practical surface treatment method [6,7]. The quality of electroplated nickel coatings generally

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depends on two main categories of parameters: those related to the plating solution, such as bath concentration, additives, and surfactants, and those associated with the process conditions, including current density, temperature, pH, and bath agitation.

The type of electrolyte and the additives used are critical parameters in nickel electroplating processes. Among the electrolytes commonly employed in industrial applications, the Watts bath is the most prevalent due to its ability to produce high-quality deposits [7]. This bath consists of three primary components: nickel sulfate, nickel chloride, and boric acid. Its formulation can be further enhanced by incorporating various surfactants, which influence the appearance of the coating, such as brightening agents, anti-pitting agents, and surface tension reducers, and help minimize internal stresses within the deposited layer [8].

In nickel electrodeposition, the type of surfactants and additives incorporated into the electrolyte play a crucial role in determining the coating's properties [8]. In recent years, ceramic and organic particles have garnered significant attention due to their unique physical, chemical, and mechanical characteristics [9]. The incorporation of ceramic particles, such as SiC, Al_2O_3, TiO_2, WC, CNT, ZrO_2, CeO_2, SiO_2, B_4C, and Si_3N_4 into nickel coatings has been shown to enhance wear resistance, hardness, and corrosion resistance compared to pure nickel coatings [8–21].

Organic additives are compounds introduced in small quantities into the electro-deposition bath to regulate and enhance the quality of the metal coating [22,23]. The incorporation of various organic additives, such as saccharin, acetylenic and aryl-sulfonic acids, 2-butyne-1,4-diol, thiosemicarbazide derivatives, butanediol, butenediol, pyridine and its derivatives, pyridinium-1-propane-3-sulfonate, and quaternary ammonium chlorides (QACl) can significantly alter the chemical, physical, and mechanical properties of nickel coatings. These additives refine the grain structure and produce diverse surface morphologies, including pyramidal, fibrous, and nodular formations. Depending on the specific additive used, improvements can be achieved in wear resistance, corrosion resistance, ductility, and brightness of the deposited nickel layer [24].

Several studies have investigated the impact of saccharin as an additive on the chemical, physical, and mechanical properties of nickel deposits [1, 4, 6, 24]. Li et al. [1] reported that the addition of saccharin to a sulfate-based electrolyte significantly refines grain size, increases hardness, and alters the internal stress of nickel deposits from tensile to compressive. Cheng et al. [4] found that saccharin reduces the grain size of nanocrystalline nickel as its concentration increases from 0 to 5 g/L, with improved corrosion resistance observed as crystallite size decreases. Rashidi et al. [6] demonstrated that grain size and texture coefficient decrease rapidly with increasing saccharin content, stabilizing at 3 g/L. Ciszewski et al. [22] studied the combined effects of saccharin and quaternary ammonium chlorides in a Watts-type electrolyte, concluding that this combination yields high-quality nickel deposits with improved efficiency. In Ni–Al_2O_3 composite electro-deposition, organic additives enhance nanoparticle co-deposition, prevent agglomeration, and increase particle incorporation [25]. These additives also improve corrosion resistance [26], reduce tensile stresses, refine the microstructure, and enhance both adhesion and surface appearance of the coating [27].

To date, no specific studies have examined the effect of glucose as an organic additive on nickel–Al_2O_3 composite coatings. From an economic perspective, glucose presents a cost-effective alternative to saccharin, with market data indicating a significantly lower price at 3.84 USD/kg for glucose compared to 14.64 USD/kg for saccharin [28].

The objective of the present study is to investigate the influence of glucose as an organic additive in a Watts-type bath on the surface morphology, microstructure, and corrosion resistance of nickel–alumina (Al_2O_3) coatings electrodeposited on low-carbon steel. The selection of Al_2O_3 is supported by previous research, which demonstrates that its incorporation into the nickel matrix forms a Ni–Al_2O_3 composite that significantly enhances the corrosion resistance of the steel substrate [29,30].

Glucose was chosen as the organic additive due to its functional chemical properties and its cost-effectiveness compared to commonly used alternatives such as saccharin.

2. Methodology

Low-carbon steel wires (0.06% C) with a diameter of 4.3 mm and a length of 60 mm were used as substrates (cathodes) for electrodeposition. Prior to coating, the substrates were mechanically polished, degreased with acetone at room temperature for 1 minute, immersed in hydrochloric acid (HCl) solution for 1 minute, rinsed with distilled water, and then dried. A nickel foil (99.9% purity) measuring 75 mm × 15 mm served as the anode. Nickel coatings were deposited using a Watts-type electrolyte composed of 11.9 g/L $NiCl_2 \cdot 6H_2O$, 12.3 g/L NH_4Cl, 4.1 g/L NaCl, and 6.2 g/L H_3BO_3, with the pH adjusted to 4.2. Electrodeposition was carried out at a current of 23 mA for 60 minutes, with the electrolyte temperature maintained at 50 °C. Glucose was added to the electrolyte at concentrations of 2, 4, and 6 g/L. Additionally, alumina (Al_2O_3) nanoparticles with an average size of 80 nm were incorporated at a concentration of 15 g/L. A typical sample after nickel electrodeposition on steel wire is shown in Figure 1, illustrating the uniformity and defect-free nature of the nickel coating on the steel surface.

Fig. 1. Typical sample after electro-deposition of nickel on steel wire

The surface morphology of the nickel coatings was examined using a scanning electron microscope (SEM, VEGA3 TESCAN). Crystalline orientation and phase composition of the nickel deposits were analyzed using X-ray diffraction (XRD, Bruker D8 Advance) with Cu-Kα radiation (λ = 1.542 Å). Corrosion resistance was evaluated through electrochemical testing conducted in a single-compartment, three-electrode cell connected to a PGP-201 potentiostat, operated via VoltaMaster 4 software. A platinum electrode served as the auxiliary electrode, a saturated calomel electrode (SCE) as the reference, and the nickel-coated sample as the working electrode. Tafel polarization curves were recorded to assess corrosion behavior, using a 3.5% NaCl solution as the electrolyte.

3. Results

3.1 Microstructure Observations

SEM micrographs of the electrodeposited nickel coatings under various conditions are presented in Figure 2. Figure 2a shows the surface morphology of nickel deposited on steel without any additive. The coating consists of small, densely packed globules, predominantly around 5 µm in size, with a morphology consistent with previous observations [7,31]. When Al_2O_3 nanoparticles were incorporated into the nickel matrix via the electrolyte, a distinct surface morphology emerged, as shown in Figure 2b. The resulting coating exhibited a heterogeneous structure with the formation of voids. This phenomenon is attributed to the chemical interaction between the Al_2O_3 particles and the nickel matrix during deposition. The presence of voids or pores is a common issue in composite electroplating and can adversely affect the mechanical strength, corrosion resistance, and adhesion of the coating. Due to the inert and ceramic nature of Al_2O_3, weak interfacial bonding with the nickel matrix often leads to the formation of micro-gaps or voids at the Ni/Al_2O_3 interface. To mitigate this issue, the addition of organic additives such as saccharin or other specific compounds has been shown to reduce void formation and improve coating quality [32].

With the addition of glucose as an organic additive to the electrolyte solution, notable changes in surface morphology were observed, as shown in Figures 2c–e. At a glucose concentration of 2 g/L, pyramid-shaped nickel particles began to form a morphology previously reported in the literature [33]. Increasing the glucose concentration to 4 g/L resulted in a coarser surface structure due to particle aggregation, yet the coating remained compact and free of surface voids (Figure 2d). At a higher concentration of 6 g/L, the morphology of the nickel particles shifted significantly, with the appearance of particles exhibiting near-cubic shapes (Figure 2e). These observations suggest that glucose influences the nickel deposition mechanism and the resulting microstructure. Similar effects were reported by Meudre [34], who studied the influence of gelatine-based organic additives on nickel electrodeposition.

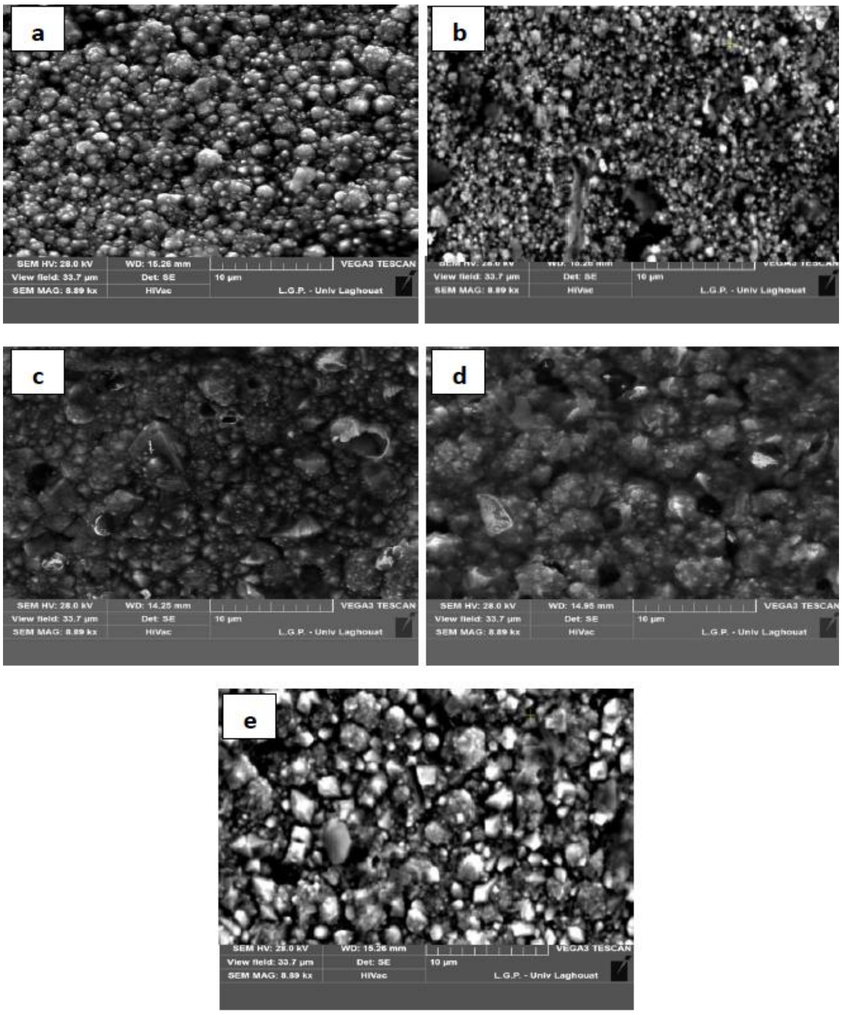

Fig. 2. SEM micrographs of the surface morphology of nickel coatings deposited from: (a) Pure Ni (b) Ni-Al_2O_3 15g/l, (c) Ni- Al2O3 -2 g/l glucose, (d)Ni- Al_2O_3 -4 g/l glucose (e) Ni- Al_2O_3 -6 g/l glucose

3.2 X-Ray Diffraction Analysis

The X-ray diffraction (XRD) patterns for bare steel, pure nickel deposits, and nickel–alumina (Ni–Al_2O_3) composite coatings with varying glucose concentrations (2, 4, and 6 g/L) are presented in Figure 3. The diffraction profiles exhibit characteristic peaks corresponding to the (111), (200), and (311) crystallographic planes of nickel. Peaks associated with Al_2O_3 were not detected, likely due to the nanoscale size of the particles and their relatively low concentration in the electrolyte [35,36]. Across all coatings, the (111) peak displayed the highest intensity, indicating a preferred crystallographic orientation during the electrodeposition process. Dibble et al. [37] similarly observed a strong (111) texture in nickel films deposited on gold substrates with saccharin additives. In the present study, the co-deposition of Al_2O_3 and 4 g/L glucose promoted crystal growth along the (111) and (311) planes, with the (311) peak intensity surpassing that of the (200) plane. This shift suggests a change in preferred orientation and reinforcement content, indicative of crystallographic texture development [28,29]. The combined presence of Al_2O_3 and glucose appears to inhibit columnar grain growth while providing nucleation sites for new grains, a finding consistent with the SEM observations shown in Figure 2d.

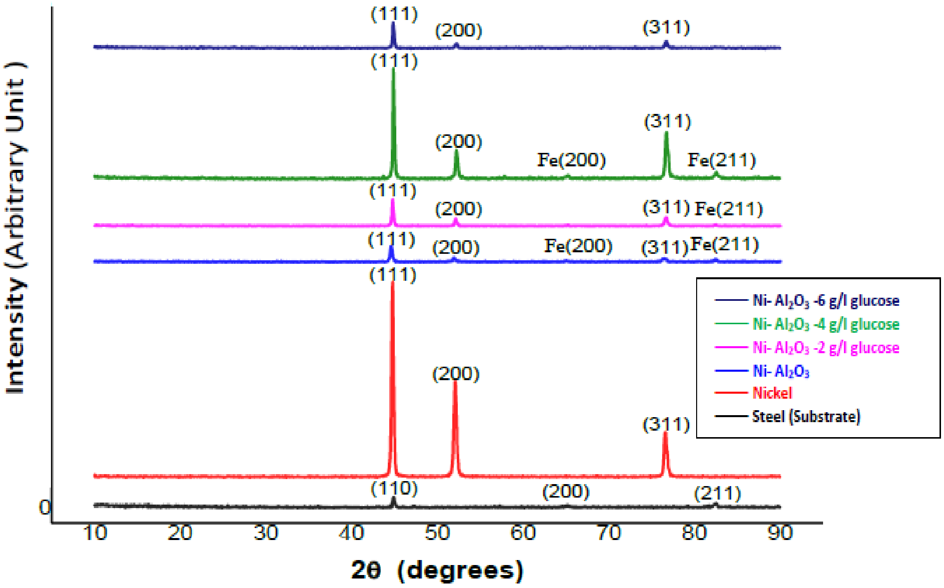

Fig. 3. The XRD patterns of steel, Nickel, Ni- Al_2O_3, Ni- Al_2O_3 -2 g/l glucose, Ni- Al_2O_3 -4 g/l glucose, and Ni- Al_2O_3 -6 g/l glucose

3.3 Corrosion Test Analysis

Tafel polarization curves of nanocrystalline nickel–alumina (Ni–Al_2O_3) coatings, with and without glucose additives, in a 3.5% NaCl solution are presented in Figure 4. Key electrochemical parameters, including corrosion potential (Ecorr), corrosion current density (icorr), and polarization resistance (Rp) were derived from the potentiodynamic polarization curves using Tafel extrapolation and are summarized in Table 1. Analysis of the cathodic branches of the polarization curves reveals no significant differences between coatings with and without glucose. However, the anodic branches show a marked reduction in current densities in the presence of Al_2O_3 and glucose, indicating improved corrosion resistance. Coatings containing glucose exhibit more cathodic behavior and significantly higher polarization resistance compared to those without glucose, confirming the beneficial role of glucose in enhancing the protective performance of the nickel–Al_2O_3 composite coating [9].

In general, both pure nickel and Ni–Al_2O_3 composite coatings enhance corrosion resistance due to the formation of a protective metallic nickel film [8,13,35]. The incorporation of Al_2O_3 nanoparticles into the nickel matrix which particularly at grain boundaries and within grains that can accelerate passivation processes [12]. These ceramic particles act as inert physical barriers, hindering the initiation and propagation of corrosion by isolating the substrate from the corrosive medium, reducing the active corrosion area, and thereby improving overall corrosion resistance [9–11]. This behavior is closely linked to microstructural changes induced by the incorporation of Al_2O_3. Additionally, the wetting properties of electrodeposited Ni–Al_2O_3 composite coatings play a crucial role in their interaction with water and corrosive agents, making them highly relevant for anti-corrosion applications [18]. Among the tested coatings, the sample containing 2 g/L glucose exhibited the best corrosion resistance, with the highest polarization resistance (R_p = 43.86 kΩ·cm²), the lowest corrosion current, and a positive shift in corrosion potential. This behavior suggests an enhanced inhibitory effect of glucose, likely due to its adsorption on the electrode surface, which blocks active corrosion sites [35]. A similar inhibitory effect was previously reported by Li et al. [1] for saccharin in nickel coatings.

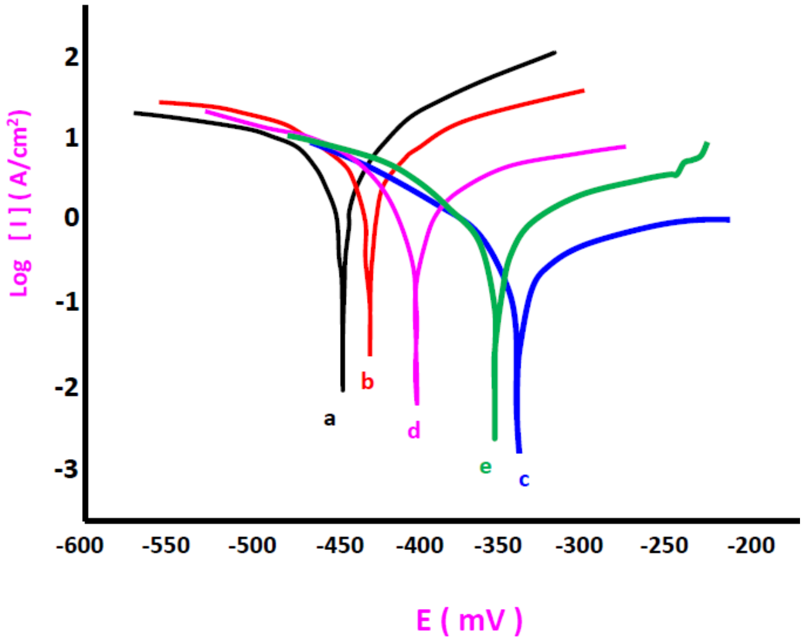

Fig. 4. Potentiodynamic polarization curves obtained for (a) Nickel (b) Ni/Al2O3, (c) Ni/Al_2O_3 +2 g/l glucose, (d) Ni/Al_2O_3 +4 g/l glucose, and (e) Ni/Al_2O_3 +6 g/l glucose, in 3.5% NaCl solution

Table 1
Corrosion data, of the Ni-base, Ni-Al_2O_3, and Ni-Al_2O_3-glucose in 3.5% NaCl solutions

Sample	E_{cor}(i=0) (mV)	i_{corr} (µA/cm^2)	R_p (kΩ.cm^2)
Nickel	- 446.40	10.4030	2.97
Ni/Al_2O_3	- 429.90	8.6380	3.38
Ni/Al_2O_3+2 g/l glucose	-342.40	0.9730	43.86
Ni/Al_2O_3+4 g/l glucose	- 403.20	3.7306	9.94
Ni/Al_2O_3+6 g /l glucose	- 354.60	1.7138	22.25

4. Conclusions

This study aimed to investigate the influence of glucose as an organic additive in a Watts-type bath on the anti-corrosive properties of electrodeposited nickel–alumina (Al_2O_3) coatings. The findings demonstrate that glucose can be successfully incorporated into Ni–Al_2O_3 composite coatings, offering a viable and cost-effective alternative to saccharin. The morphology of nickel particles was found to vary depending on the presence and concentration of glucose: spherical shapes were observed in the absence of glucose, while pyramidal and cubic shapes emerged with increasing glucose concentrations. Additionally, glucose altered the preferred crystallographic orientation of nickel particles, with X-ray diffraction revealing a dominant (111) texture. The combination of ceramic Al_2O_3 particles and glucose significantly enhanced the corrosion resistance of the coated substrates, with the best performance observed at a glucose concentration of 2 g/L. These results highlight the potential of glucose as an effective additive for improving both the structural and protective properties of nickel-based composite coatings.

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