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RESEARCH ARTICLE / ARAŞTIRMA MAKALESI

Corrosion Performance of Electrodeposited Ni Nanocomposite Coatings Reinforced with Metal Oxide Particles

Elektrokimyasal Olarak Çöktürülmüş Metal Oksit Takviyelendirilmiş Ni Nanokompozit Kaplamaların Korozyon Performansı

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Abstract

The properties of traditional nickel coatings were enhanced by preparing Ni nanocomposite coatings reinforced with metal oxide nanoparticles by using electrodeposition technique. The impact of applied current density was investigated by changing SiO_2 nanoparticle concentration. Phase structure study and morphological investigation of the samples were performed by X-Ray Diffractometer and Scanning Electron Microscopy, respectively. Vickers indentation method was utilized to determine the mechanical properties of obtained nanocomposite coatings. Potentiodynamic polarization technique was performed to evaluate electrochemical behavior of nanocomposite coatings. As a result, introducing SiO_2 nanoparticles to Ni matrix improves both mechanical and electrochemical properties of produced nanocomposite coatings.

Keywords: *Nanocomposite Coatings, SiO_2 Nanoparticle, Electrodeposition, Microhardness, Corrosion*

Öz

Geleneksel nikel kaplamaların özellikleri, elektrokimyasal çöktürme yöntemi kullanılarak üretilen metal oksit nanopartiküller ile güçlendirilmiş Ni nanokompozit kaplamalar hazırlanarak geliştirilmiştir. Uygulanan akım yoğunluğunun etkisi, SiO_2 nanopartikül konsantrasyonu değiştirilerek incelenmiştir. Nanokompozit kaplamaların faz yapısı çalışması ve morfolojik incelemeleri sırasıyla X-Işını Difraktometre ve Taramalı Elektron Mikroskobu ile yapılmıştır. Elde edilen nanokompozit kaplamaların mekanik özelliklerini belirlemek için Vickers indentasyon yöntemi kullanılmıştır. Nanokompozit kaplamaların elektrokimyasal davranışını değerlendirmek için potansiyodinamik polarizasyon tekniği uygulanmıştır. Sonuç olarak, Ni matrisine SiO_2 nanopartiküllerinin ilavesi, üretilen nanokompozit kaplamaların hem mekanik hem de elektrokimyasal özelliklerini iyileştirmektedir.

Anahtar Kelimeler: *Nanokompozit Kaplamalar, SiO_2 Nanopartikü, Elektrokimyasal Çöktürm, Mikrosertlik; Korozyon*

1. Introduction

Composite coatings have been employed in a wide range of industrial applications due to their exceptional properties such as improved wear resistance, diminished corrosion tendency, and high hardness. Several techniques, including electroless plating, physical vapor deposition (PVD), chemical vapor deposition (CVD), and electrodeposition, can be used to develop composite coatings. Among these technologies, the electrodeposition approach has the benefits of having lower operating temperatures, low cost, and simplicity of operation, shape flexibility, high production rates, and versatility with varied combinations of attributes by simply adjusting the electroplating settings [1-5].

The use of various materials in industrial applications is expanded by electrodeposited layers with superior properties [6, 7]. A stable solution containing well-dispersed nanoparticles with appropriate electrophoretic mobility and an additive for suspension stabilization is required for the electrodeposition technique. The deposition bath concentration, pH, and dispersion solution are critical factors in this technique [8, 9]. The electroplating technique is among the widely used methods for the manufacture of nano-structured composites due to improved particle-metal bonding and easier control of coating thickness. The coating thickness and composition in the resulting structure can be precisely controlled by variables that influence the applied current density and solution composition [10-11].

To obtain nano/micro composite coatings, solid micro/nano-particles are often introduced to an electrodeposition bath while being vigorously agitated. In contrast to composites containing micro-sized particles, nano-sized particles might stimulate the hardness of the material. Because of their superior qualities, nanocomposite coatings made with nano-sized particles are

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more attractive than those made with micro-sized particles [12-14]. Ni-based coatings reinforced by ceramic nanoparticles such as CeO_2 [11], TiO_2 [15], Al_2O_3 [8], ZrO_2 [16], SnO_2 [17], WC [18], SiC [3], SiO_2 [19], Cr_2O_3 [20] and TiN [21, 22] are typical nanocomposite coatings. Nickel nanocomposite coatings are widely employed in industrial and engineering applications such as consumer technology, chemical, computer, and telecommunications to promote corrosion and wear resistance and to assess magnetic characteristics [23].

SiO_2 is a ceramic compound with excellent physical and chemical properties that is commonly employed as a second-phase additive in electrodeposited coatings based on Ni, Cu, Zn, Ag, and Au [24]. In this study, nanosized silica was employed as second phase for the electrodeposition of nickel-based nanocomposite coatings. This research focuses on the development of nanocomposite coatings and investigates the effect of electrodeposition parameters on the structural, mechanical and corrosion properties of nanocomposite coatings.

2. Materials and Methods

Ni-SiO_2 nanocomposite coatings were prepared using electrodeposition technique on steel substrates. The effects of deposition current density and incorporated SiO_2 nanoparticles concentration on the structural, mechanical and deteriorative properties of coatings were investigated. First of all, SiO_2 nanoparticles were synthesized by hydrolyzing tetraethyl orthosilicate (TEOS) in ethanol with ammonium hydroxide. Firstly, TEOS, ethanol and deionized water were mixed in molar ratio of 5:2:7. Subsequently, condensation reaction was then accelerated by the addition of ammonium hydroxide dropwise as a catalyst. Following the completion of these reactions, the resulting suspension was dried in an oven at 85 °C and then calcined at 500 °C. The resulting nanoparticles ranged in size from 50 to 70 nm.

Then, nickel coatings were deposited on steel substrates using a traditional nickel Watts bath. Watts bath is composed of 0.91 M nickel (II) sulfate hexahydrate ($NiSO_4.6H_2O$), 0.17 M nickel (III) chloride hexahydrate ($NiCl_2.6H_2O$) and 0.48 M boric acid (H_3BO_3) chemicals. Ni-SiO_2 nanocomposite coatings were deposited using a DC power supply with varying current densities of 3, 5 and 7 (A/dm^2) at 45°C. The distance between the nickel anode and the steel cathode was adjusted to be 3 cm. Table 1 depicts the designation of deposited coatings.

X-Ray Diffraction was executed to determine the phase structure of the nanocomposite coatings (XRD, Rigaku, D/Max 2100 PC). The Scherrer equation was used to estimate the crystallite size values, which represent the structural properties of the samples [25]. SEM was utilized to analyze the morphological properties of the samples (SEM-EDS, Jeol, JSM 6060). Surface roughness measurements was performed by Mitutoyo SJ-301 Surftest Surface Roughness device. The microhardness measurements of the samples were performed on the surface of the coatings with a Shimadzu HMV-2 model Vickers microhardness tester at a load of 25 g for 10 seconds. Then, samples' microhardness values were determined by calculating the mean of ten measurements.

The corrosion studies were conducted using a Gamry potentiostat/galvanostat instrument. In addition, the Echem Analyst tool was utilized to collect and analyze electrochemical data. The corrosion experiments were performed using a standard three-electrode cell. The graphite electrode, deposited coatings, and Ag/AgCl electrode used as the counter electrode, working electrode, and reference electrode, respectively, in the three-electrode system. The open circuit potentials (OCP) of the produced coatings were monitored in a NaCl solution for 30 minutes. Potentiodynamic curves for corrosion performance of the samples were generated by sweeping the potential from OCP to 0.6 V versus the reference electrode at a rate of 1 mV/s. SEM investigation was carried out to examine the morphological investigation of the coatings after the corrosion experiments.

Table 1. Nomenclature of the produced coatings.

SiO_2 Nanoparticle concentration (g/l)	Current Density (A/dm^2)	Nomenclature
0	3	S-1
1	3	S-2
3	3	S-3
	5	S-4
	7	S-5

3. Results and Dicussion

Insoluble particles become trapped in the growing Ni layer during the electrodeposition of Ni matrix composite coatings. Indeed, cations are adsorbed on the widely dispersed particles and moved to the cathode surface in the electrolyte. The positive particles are then accumulated on the cathode surface and attacked by the developing Ni layer, eventually forming a composite coating [26]. XRD patterns of coatings produced with increasing SiO_2 nanoparticle content in electrolyte, at constant current density are given in Figure 1. The XRD pattern shows typical 2θ characteristic peaks of nickel phase (JCPDS 70-1849) at 44.40°, 51.80° and 76.42° corresponding to (111), (200) and (220) crystallographic planes of nickel, respectively.

This reveals that nickel coatings have been efficiently grown on steel substrates. The absence of the SiO_2 peaks in the XRD pattern of the samples is due to the low amount and small size of SiO_2 nanoparticles.

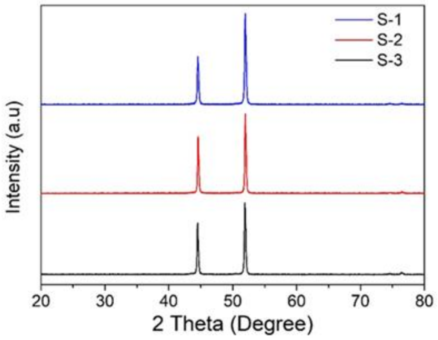


Figure 1. XRD patterns of coatings produced with increasing SiO_2 content in electrolyte, at constant current density and deposition time.

The average crystallite size of the deposited coatings was calculated using the Scherrer equation, which took into consideration the (220) plane peak broadening with the values [26]. The (200) crystal surface dominated the growth of Ni coatings with SiO_2 addition, and the (200) peak intensity was boosted. The crystallite size values of the coatings are 141.9, 135.9, and 114.4 nm for coatings S-1, S-2, and S-3, respectively. It indicated that the size of crystallites was highly dependent on the SiO_2 nanoparticle content of the coatings. As SiO_2 particles are incorporated into the Ni matrix, the average crystallite size of the deposit decreases because adsorbed SiO_2 nanoparticles act as additional nucleation sites and prevent the growth sites of Ni ions in the Watts bath. It was found that the crystallite size values modified in accordance with previous studies [27, 28]. Current density is a critical operating variable in the electrodeposition process because it regulates the electrodeposition rate, chemical composition, and microstructure, all of which have a significant impact on the functional properties of the obtained coatings [29]. In the present study, the effect of current density was investigated at constant particle concentration (3 g/l) with the deposition time of 15 min. Obtained results show that intensity of (200) peak increased with increasing current density as illustrated in Figure 2.

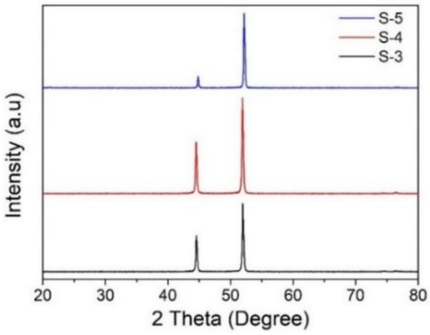
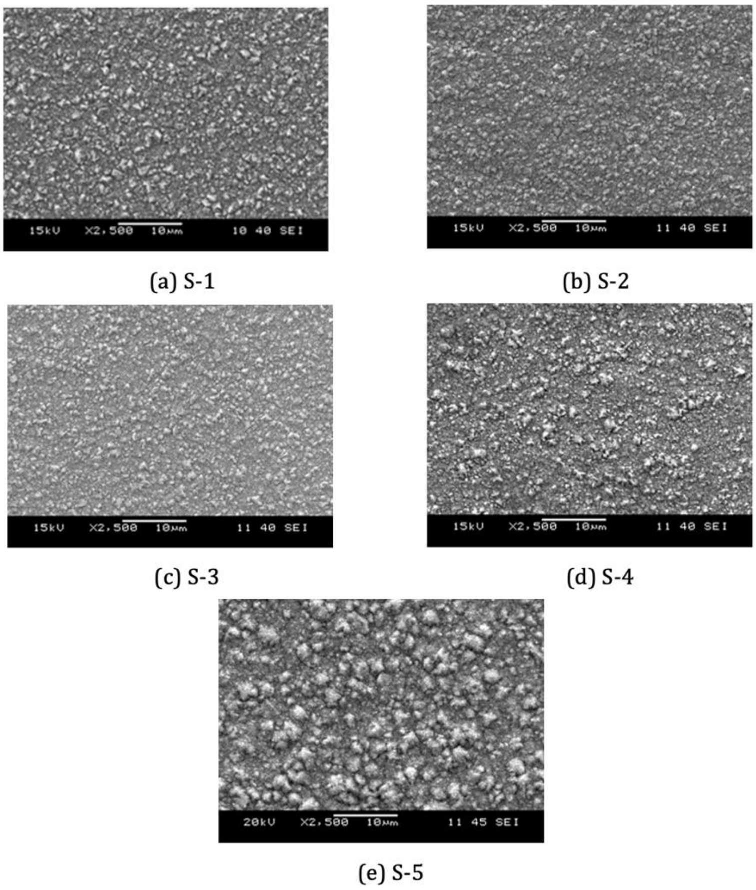


Figure 2. XRD patterns of coatings produced with increasing current density, at a constant SiO_2 content in electrolyte and deposition time.

The inert particle concentration of deposited coatings with increasing current density either declines or raises constantly, which is mainly attributed to metal deposition behavior [29]. The crystallite size of the coatings are 114.4, 118.6 and 125.1 nm for coatings S-3, S-4 and S-5, respectively. According to these findings, increased current density causes an increase in the average crystallite size of nickel matrix nanocomposite deposits. The findings of Aruna et al. are similar to our results in terms of current density and crystallinity relationship [30].

Figure 3 illustrates the surface morphology of Ni-SiO_2 nanocomposite coatings experienced at current densities of 3, 5, and 7 A/dm^2 and SiO_2 nanoparticle ratios of 1 and 3 g/l. All of the produced nanocomposite coatings have homogeneous, compact, crack free and nodular surface structure regardless of the current density, SiO_2 content in the electrolyte and deposition time. The incorporation of SiO_2 nanoparticles was obtained and uniformly distributed into the Ni metal matrix successfully achieved, even for higher SiO_2 concentration in the electrolyte. Similar results were also observed in the study of Beltowska-Lehmanet al. [12]. Surface morphology of coatings alters to finer and more compact in terms of structure especially for lower current density values with the introduction of SiO_2 nanoparticles into the electrolyte solution. As shown in Figure 3, nodular structures of the nanocomposite coatings grow as the current density increases. Increasing the deposition time results in similar structural changes as well.



(a) S-1

(b) S-2

(c) S-3

(d) S-4

(e) S-5

Figure 3. SEM images of Ni-SiO_2 nanocomposite coatings with increasing SiO_2 contents and current density values.

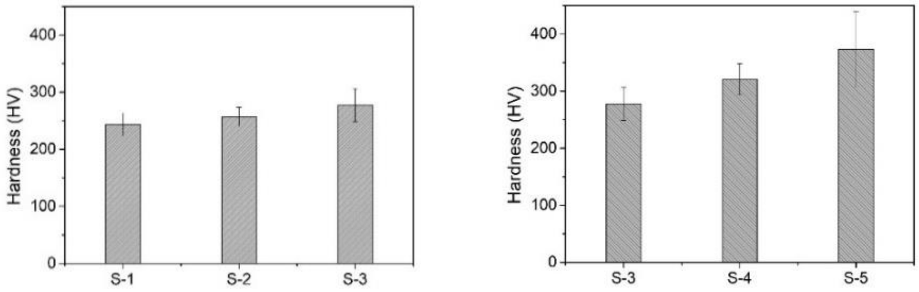
The hardness of nanocomposite coatings is affected by the amount of hard particles incorporated to the composite structure and the hardness of the metal matrix established by the coating parameters [31]. Results of the effect of SiO_2 concentration and current density on microhardness of nanocomposite coatings are given in Figure 4. Average thickness values of the coatings of S-1, S-2, S-3, S-4 and S-5 are 23.4, 25.1, 21.9, 28.5 and 33.7 µm, respectively. The arithmetic average roughness (R_a) value was used to describe surface characteristic of the coatings. R_a values of S-1, S-2, S-3, S-4 and S-5 coatings was measured as 0.16, 0.18, 0.26, 0.32 and 0.33 µm, respectively. Presence of SiO_2 nanoparticles lead to increment of the surface roughness values by increasing current density in comparison to Ni coatings [32]. The hardness values of Ni coatings and nanocomposite coatings deposited at different SiO_2 content were measured as 243 HV, 257 HV and 278 HV for coatings S-1, S-2 and S-3, respectively. Results indicate that hardness of the produced Ni coatings increased with introduction of SiO_2 nanoparticles and increasing SiO_2 concentration in the electrolyte. This increase could be attributed to the influence and strengthening effect of the introduction of inert SiO_2 particles. Moreover, current density also has affirmative effect on the microhardness of nanocomposite coatings. The microhardness values of coatings deposited at constant nanoparticle concentration were measured as: 278 HV, 321 HV and 373 HV, for coatings S-3, S-4 and S-5, respectively. Estimation of Vickers indentation depth is calculated according to the equation of $HV = \frac{1.854xP}{di^2}$ (P: applied load, d_i:size of the indentation). Obtained values of indentation depth are 1.97, 1.92, 1.84, 1.72 and 1.59 µm for S-1, S-2, S-3, S-4 and S-5, respectively.

The time-dependent variation of the open circuit potential (E_{oc}) in the NaCl electrolyte of Ni-SiO_2 nanocomposite coatings deposited with variable SiO_2 concentration is shown in Figure 5(a).

These findings demonstrate that the introduction of SiO_2 nanoparticles into the Ni matrix shifts the E_{oc} toward positive values. In particular, the produced nanocomposite coatings are significantly electrochemically stable than pure Ni coatings in the corrosive medium. Figure 5(b) exhibits the potentiodynamic polarization curves of Ni-SiO_2 nanocomposite coatings electrodeposited at various levels of nanoparticle concentrations. The result of applied deposition current density on corrosion performance of the Ni-SiO_2 nanocomposite coatings is depicted in Figure 6. Table 2 presents data derived from potentiodynamic polarization measurements. It can be seen that corrosion current density (i_{corr}) of the samples declined from 0.267 A/cm^2 to 0.182 A/cm^2 as the particle content increased. The reduction in i_{corr} values of nanocomposite coatings as nanoparticle concentrations increase can be explained by reducing the metallic area subjected to the corrosive environment. In this context, a change in corrosion potential to higher positive values is caused by homogeneous distribution of the inert SiO_2 phase in the structure [23].

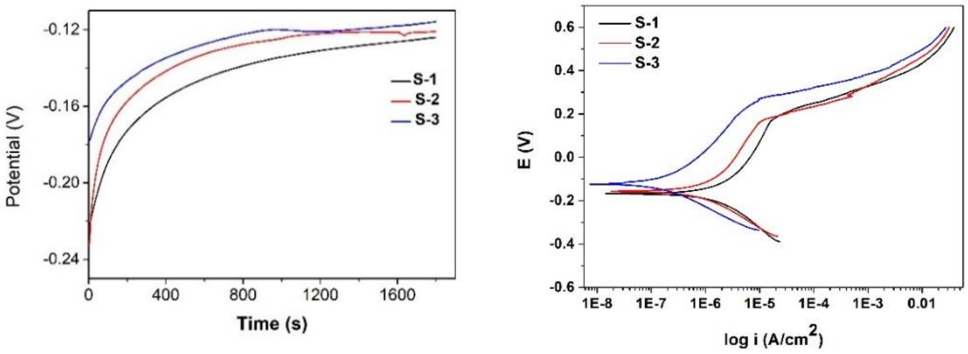
Table 2. Electrochemical parameters obtained from the corrosion experiments of the investigated coatings

Sample Name	E_{corr} (mV)	i_{corr} (µA/cm^2)	Corrosion rate (mpy)
S-1	-167	0.267	0.061
S-2	-157	0.245	0.056
S-3	-124	0.182	0.041
S-4	-154	0.227	0.052
S-5	-161	0.270	0.061



(a) (b)

Figure 4. Microhardness of coatings (a) effect of SiO_2 incorporation and concentration and (b) effect of applied current density.



(a) (b)

Figure 5 (a) The time-dependent fluctuation in the open circuit potential of the produced coatings and (b) polarization curves of the produced coatings with different SiO_2 content.

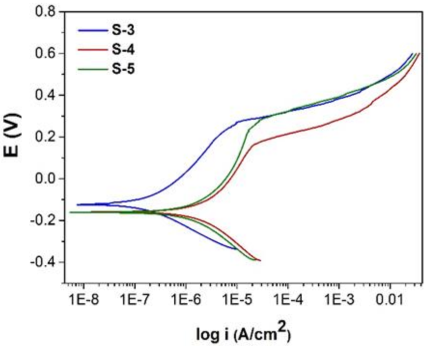
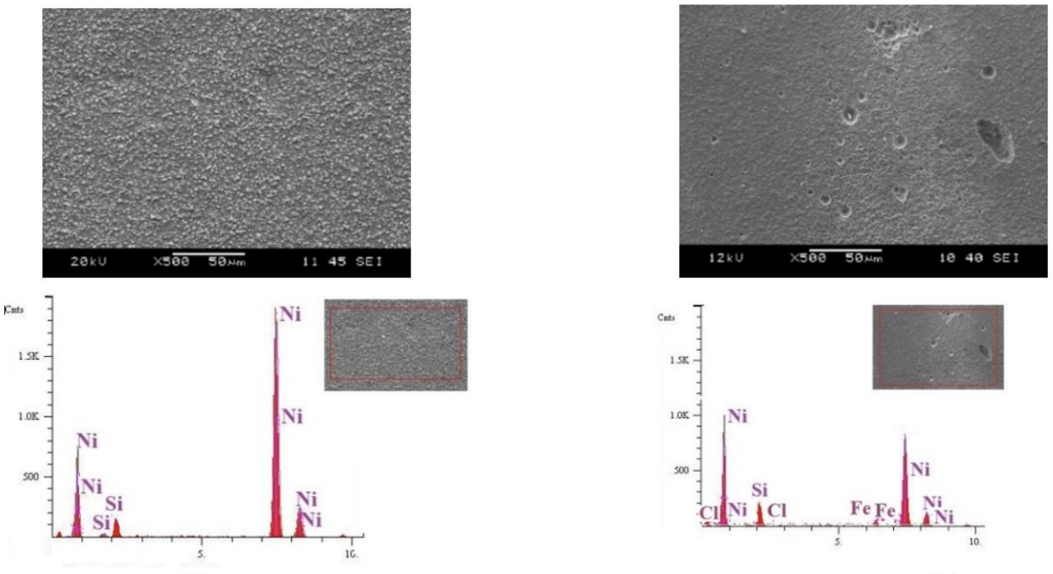


Figure 6. Potentiodynamic polarization curves of produced nanocomposite coatings deposited at various current density values.

Microgalvanic cells are generated at particle/matrix interactions because SiO_2 structures have a larger reduction potential than Ni matrix. SiO_2 nanoparticles perform as inert physical obstacles and enlarge the nucleation regions. They could disrupt the dissolution of Ni and retard the interpenetration of corrosion solution to the substrate. This leads to the enhancement of corrosion resistance.

Besides, uniformly distributed nanoparticles in coating could enhance the inertness and prevent corrosion process [33]. Consequently, Ni-SiO_2 nanocomposites exhibited outstanding electrochemical properties. As a result, uniform corrosion controlled localized corrosion and pitting corrosion as the predominant forms of corrosion [34]. According to the literature, with increasing current density, the hydrogen evolution reaction of the cathode increase which diminishes the deposition of the nanoparticles. This results with the reduced corrosion performance of deposited coatings [35] The corrosion current density (i_{corr}) of the Ni-SiO_2 nanocomposite coatings deposited at 3 g/l SiO_2 nanoparticle concentration is rises from 0.182 A/cm^2 to 0.270 A/cm^2 with an increase in current density from 3 A/dm^2 to 7 A/dm^2, according to the polarization measurements data. This can be attributed to the fact that the corrosion resistance of the S-3 coating is higher than that of coatings deposited at higher current density.

SEM micrographs and EDS spectrums of the coating S-5 (a) before corrosion and (b) after corrosion are given in Figure 7. The presence of iron element that comes from substrate proves the dissolution of the nanocomposite coatings after the corrosion test.



(a)

(b)

Figure 7. SEM images and EDS spectrums of the coating S-5 (a) before corrosion and (b) after corrosion

4. Conclusions

In the present study, the electrodeposition parameters for Ni-SiO_2 nanocomposite coatings have been investigated in terms of structural, morphological, mechanical and corrosion properties. All of the deposited coatings show only nickel phase diffraction peaks on the XRD patterns regardless of the current density, SiO_2 content in the electrolyte and deposition time. All of the deposited nanocomposite coatings have homogeneous, compact, crack free and nodular structure. Ni-SiO_2 nanocomposite coatings prepared utilizing a bath with a higher SiO_2 content also had a greater hardness value. This increase can be linked to the mechanical properties of Ni-SiO_2 nanocomposite coatings being superior to pure Ni coatings. Furthermore, the introduction of SiO_2 nanoparticles enhanced the corrosion characteristics of produced nanocomposite coatings. Therefore, Ni-SiO_2 nanocomposite coatings are candidate coatings for metal protection in the field where corrosion resistance and excellent mechanical properties are necessary.

Ethics committee approval and conflict of interest statement

This article does not require ethics committee approval.

This article has no conflicts of interest with any individual or institution.

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Author Contribution Statement

Funda Ak Azem: conceptualization, experimental design, conducting analyses, literature review, writing, critical review; Isil Birlik: conceptualization, experimental design, conducting analysis, literature review, writing; Tulay Koc Delice: conceptualization, data collection, conducting analysis, literature review, writing; Ramazan Dalmis: conceptualization, data collection, conducting analysis, literature review, writing.

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