

Research Article

Influence of Surfactants on the Characteristics of Nickel Matrix Nanocomposite Coatings

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Nickel-based nanocomposite coatings were prepared from a Watts-type electrolyte containing reinforcement's particles (silicon carbide and graphite) to deposit onto the steel St-37 substrate. The electrochemical plating of the coatings in absence and presence of surfactants and reinforcements particles was carried out to optimize high quality coatings with appropriate mechanical and morphological features. The surfactants such as cetyltrimethylammonium bromide (CTAB), sodium dodecyl sulfate (SDS), and saccharine affected electrodeposition plating and subsequently changed mechanical characteristics. Based on XRD results, the dominant phases in the absence of surfactants were nickel oxide (NiO), nickel, and silicon carbide (SiC), while the main phases in presence of surfactants were nickel (Ni) and SiC. The hardness of the resultant coatings was found to be from 332 to 593 (Hv) depending on the bath parameter and the reinforcements weight percentage (wt%) in the Ni matrix. Microscopic observations illustrated a cluster-like structure which consisted of some fine sphere particulates with average particle size of 65–150 nm. According to elemental mapping spectra, a homogenous distribution of nickel, silicon, and carbon particles appeared into the nickel matrix coating. Finally, the experimental outcomes demonstrated that the surfactants have significant influence on the composition of coatings, surface morphology, and mechanical properties.

1. Introduction

The recent researches in surface engineering were significantly focused on the development of low carbon steels (St-37) surfaces which can meet the industrial request [1–4]. The mechanical properties of many parts of surfaces were promoted by coating of numerous pure metals, ceramics, and alloys which can be deposited with fine grain sizes, for example, Ni, Pd, Cu, Ni-P, Ni-W, and Ni-Fe-Cr [5, 6]. Furthermore, various methods such as electrodeposition, ion implantation, chemical vapor deposition (CVD), laser beam deposition, physical vapor deposition (PVD), plasma, and high-velocity oxygen fuel (HVOF) spraying have been developed [7, 8]. Among these procedures, electrochemical plating is a proper method with remarkable features such as easy maintainability, easy low working temperatures, low cost, and high production rate [9]. In chemical techniques, surfactants have been used to absorb much more fine particles into the matrix, so that the repulsion force between particles

with the same charges can be increased. This, in turn, reduces the agglomeration and provides a solution with more stable particles. It should be mentioned that nanocomposite coatings are valuable for promoting of the surface properties so that they have been revolutionized surface sciences and applications. The use of particles in coatings could decline the problem of created imperfection, such as voids between the particles and matrix interfaces. In addition, the deposition of enough amounts of reinforced particles could lead to generation of stronger and more resistant covers [10].

Electrodeposition of nickel coating has received widespread acceptance as it provides a uniform deposit on irregular surfaces, direct deposition on surface activated, high hardness, and excellent resistance to wear, abrasion and corrosion. Additionally, nickel as an engineering material was broadly used among the electrodeposited materials [11]. Many studies illustrated that deposition of coatings in the presence of fine particles such as hard materials (e.g., SiC [12, 13], Al₂O₃ [12], WC [14], and diamond [15]) or

lubricating particles (e.g., MoS₂, PTFE, and graphite (Gr) [16, 17]) into the nickel matrix might effectively improve the mechanical and tribological properties of the surfaces [9]. Also, numerous researchers have studied synthesis of Ni-based nanocomposites by electrodeposition for instance Ni-SiC [18–20], Ni-P-SiC [21], Ni-Al₂O₃ [22], and Ni-ZrO₂ [22]. The deposition of nanocomposites was affected by many factors including processing parameters (e.g., surfactant, pH value, current density, magnetic field, and waveform), bath composition, and particle content. Meanwhile, by manipulating the processing parameters some noticeable results were acquired [18, 23–25]. It should be noted that the determination of optimum conditions for production of nickel coatings from the reported results is difficult because they are, in some cases, different or paradoxical. For instance, the different results have been reported about effect of reinforcement grain size into the nickel-based coatings [26].

Although different nickel matrix nanocomposite coatings with suitable mechanical and tribological properties were known, but to the author's knowledge, the influence of surfactants (CTAB, SDS, and saccharine) and reinforcements (SiC and Gr) on the characteristics of nickel matrix nanocomposite coating was not investigated. In order to obtain the Ni-based coating with acceptable microhardness, it is necessary to use surfactants to absorb SiC and graphite (Gr) particles into the matrix during plating. In this paper, the main target was the electrodeposition of Ni-SiC-Gr nanocomposite coating onto the St-37 substrate with suitable mechanical and morphological properties. Moreover, the influence of surfactants on the morphological features and distribution of SiC and Gr particles into the Ni matrix was evaluated by XRD, SEM/FE-SEM, EDS, and elemental mapping analysis techniques. Finally, the mechanical properties of all specimens were compared under different circumstances which exhibited valuable results.

2. Experimental Procedures

2.1. Nanocomposite Coating Preparation. Nickel coatings were deposited from Watts bath by direct current (DC) electroplating. Electrochemical bath components were consisted of nickel sulfate (Merck, 99%), boric acid (Merck, 99.8%), and nickel chloride (Merck, 98%). The basic compositions of the bath and the experimental operating parameters are provided in Table 1. SiC nanopowders in the range of 40–100 nm (Hefeikaier Nanometer Energy & Technology, 99%) and Graphite (Gr) particles in the range of 5–150 μm (Merck, 99.8%) were utilized as reinforcements. Meanwhile, the electrostatic adsorption of suspended particles on the cathode surface increased by enhancing of their positive charge [27] and the surfactants such as cetyltrimethylammonium bromide (CTAB, Merck 98%), sodium dodecyl sulphate (SDS, Acros Organics 98%), and Saccharine (Merck 99%) were used. To obtain suitable nanocomposites coatings, all the bath ingredients were agitated (stirring rate = 500 rpm) for 20 min by magnetic stirring. Subsequently, the sonication process was served for 15 min to provide homogeneous

dispersion and to prevent agglomeration of the particles. Low carbon steel (St-37) plates 10 mm × 10 mm × 1 mm as substrate and nickel cylindrical were utilized for electroplating process. Before the electrodeposition, the substrates have been prepared by the following procedures: (a) degreasing using a surfactants solution at 50°C for 5 min, (b) acid pickling in a diluted sulfuric acid solution (10% H₂SO₄) for 1 min, and (c) polishing with silicon carbide metallographic papers. After each preparation step, the substrates have been rinsed by distilled water in order to completely remove the residuals of each stage.

2.2. Characterization Techniques. Phase analyses and structural changes of nanocomposite powders were determined by X-ray diffraction (Philips X-ray diffractometer (XRD), Cu-K_α radiation, 40 kV, 30 mA, and 0.02° S⁻¹ step scan). For qualitative analysis, XRD graphs were recorded in the interval 20° ≤ 2θ ≤ 70° at scan speed of 1°/min. *PANalytical X'Pert HighScore* software was also utilized for the analysis of different peaks. The obtained experimental patterns were compared to standards compiled by the Joint Committee on Powder Diffraction and Standards (JCPDS), which involved card number 04–0850 for Ni, number 029–1128 for SiC, number 01–0646 for Gr, and number 047–1049 for NiO. The surface morphologies of coatings were observed by scanning electron microscopy (SEM, VEGA Tescan easyprobe). X-ray energy dispersion spectroscopy (EDS) and elemental mapping analysis system attached to the SEM were utilized to determine the weight percentage of nanoparticles and distribution of materials. The particle size and thickness of the coated layer were measured by a field emission scanning electron microscope (FE-SEM Hitachi S1831) that operated at the acceleration voltage of 15 kV. Microhardness analysis was performed on polished samples at room temperature using a Vickers microhardness tester (MICROMET3, Buehler Ltd., USA) with Vickers pyramidal diamond indenter. A load of 50 g was applied for 20 s and the final value quoted for the hardness of the deposit was the average of at least three measurements. It should be mentioned that the reported values of the microhardness are representative of the deposited coatings without significant influence from underlying substrate.

3. Results and Discussions

3.1. XRD Analysis. Figure 1 shows the XRD profile of the SiC nanoparticles. As can be seen, the single phase of SiC with high crystalline structure is close to the standard card of SiC (no. 29-1128). Figure 2 illustrates the XRD pattern of Ni matrix coating deposited in absence of reinforcement materials (SiC and Gr) in electrolyte. According to the XRD pattern, it can be observed that after 20 min of plating, the dominant phase was nickel. The XRD profile of the Ni-based coatings in the absence and presence of surfactants are presented in Figures 3(a) and 3(b), respectively. As can be seen in Figure 3(a), the strong peak was related to nickel oxide and it proposes that the surface of the Ni matrix was oxidized during contacting with the plating bath [28]. In addition,

TABLE 1: Electrochemical plating process at different bath compositions, (I) without any reinforcements powders, (II) in absence of surfactants solution, and (III) in presence of surfactants solution.

Sample	Bath composition (g/L)								Operating conditions		
	NiSO ₄ ·6H ₂ O	NiCl ₂ ·6H ₂ O	H ₃ BO ₃	SiC	Graphite	CTAB	SDS	C ₇ H ₅ NO ₃ S	T (°C)	pH	DC (A/dm ²)
I	200	20	40	—	—	5	5	1	45	4.8	4
II	200	20	40	12	1	0	0	0	45	4.8	4
III	200	20	40	12	1	5	5	1	45	4.8	4

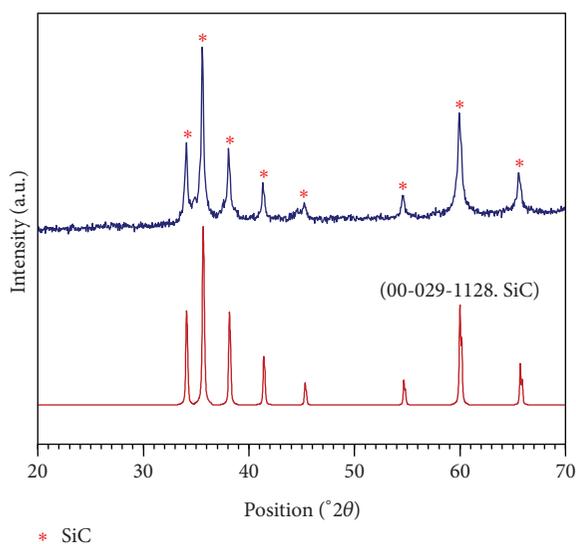


FIGURE 1: XRD pattern of the SiC nanoparticles.

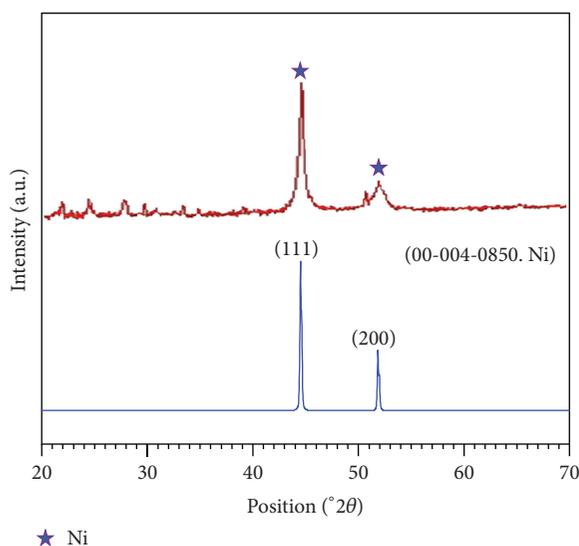


FIGURE 2: XRD pattern of Ni matrix coating without any reinforcement materials (sample I).

the weak signal of nickel was found on the diffraction pattern. Fine peaks of SiC were also identified which can be inferred that the low amount of SiC was absorbed into the nickel matrix. Figure 3(b) shows the XRD pattern of Ni-SiC-Gr nanocomposite coating deposited in presence of surfactants

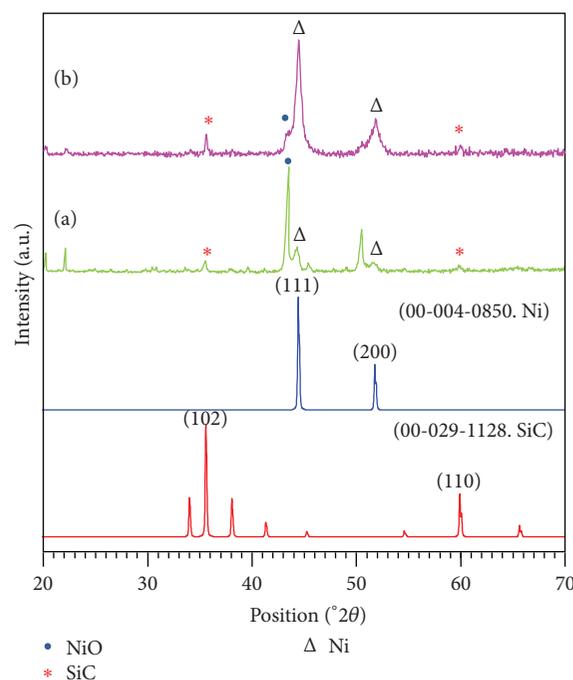


FIGURE 3: XRD pattern of Ni-SiC-Gr nanocomposite coatings deposited; (a) in absence of surfactants (sample II), and (b) in presence of surfactants (sample III).

solution. The profile of the sample confirms the presence of Ni, SiC, graphite (Gr), and NiO phases. The strong peak of nickel shows that the electrochemical deposition was done almost perfectly so that this event shows key role of surfactants in plating of nickel-based coating. The XRD pattern demonstrates typical peaks corresponding to (111) and (200) crystallographic planes of nickel as well as the (102) and (110) planes of SiC. It has been reported that [11] the embedding of SiC particulates in the nickel matrix could modify the Ni texture from the soft [1 0 0] mode to the mixed preferred [2 1 1] orientation. Note that the peaks corresponding to Gr particles could not be completely detected in XRD patterns from nanocomposite coatings due to very low content (1 g/L). The specimen is prepared by surfactants solution, and the intensity of (111) peak is higher than the other planes. This result indicated that the preferred orientation of Ni is (111) plane. It should be noted that (111) peak at $2\theta = 44.508^\circ$ is the strongest peak (relative intensity = 100%) in the standard XRD pattern from randomly oriented polycrystalline nickel (no. 04-0850). The crystallinity (CrI) of

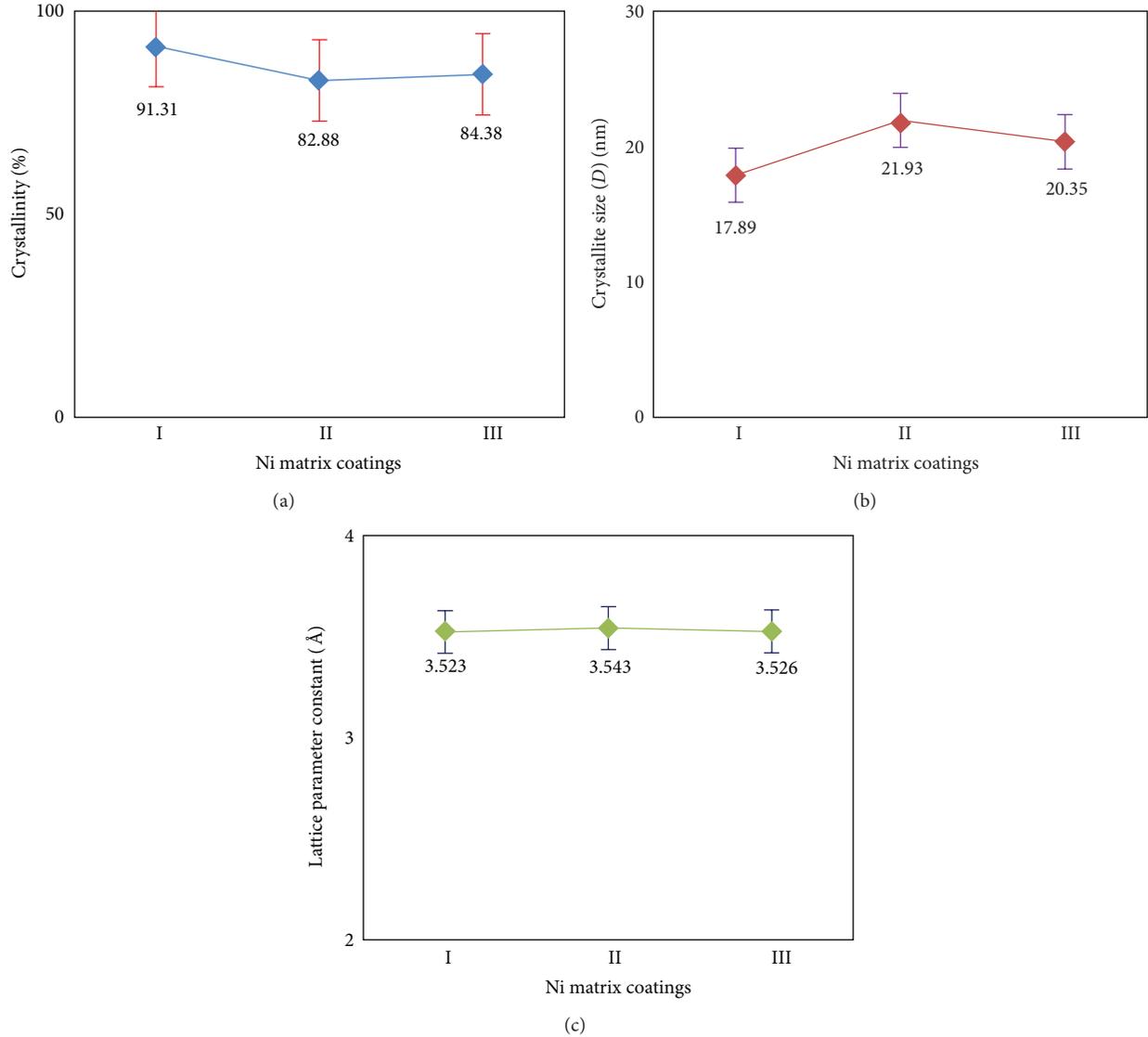


FIGURE 4: Crystallite size (D), crystallinity (%), and lattice parameter constant (α) of nickel in nanocomposite coatings deposited; (a) in absence of reinforcements powders in electrolyte (SiC and Gr) (sample I), (b) in absence of surfactants (sample II), and (c) in presence of surfactants (sample III).

Ni particulates of the sample was determined by using the XRD data according to the following equation [29]:

$$\text{CrI} = \frac{I_{111} - I_{\text{am}}}{I_{111}} \times 100, \quad (1)$$

where I_{111} is the diffraction intensity of (111) plane and I_{am} is the intensity of the measured amorphous peak (200).

The crystallite size (D) for nickel was calculated using Scherrer equation [22] as follows:

$$\text{FWHM} = \frac{K\lambda}{D \cos \theta} \frac{180^\circ}{\pi}, \quad (2)$$

where FWHM is full width half maxima in 2θ degrees, D is the crystallite size in nm, K is constant (usually evaluated as 0.94), and λ is the wavelength of Cu- K_α radiation (0.154 nm).

Also, as part of the structural characteristics, the effect of surfactants on Ni lattice (cubic) constant (a_c) was evaluated by the following relation [30]:

$$d_{hkl} = \frac{a_c}{\sqrt{(h^2 + k^2 + l^2)}}, \quad (3)$$

where the Miller indices (hkl) obtained from the diffraction spectra were identified using JCPDS cards (no. 04-0850) and d_{hkl} is the distance between adjacent Bragg planes. Figure 4 shows the structural features of Ni phase for all the nanocomposite coatings deposited at different bath conditions (see Table 1). From the results of crystallinity (Figure 4(a)), it could be observed that the crystallinity of Ni deposited in absence of any reinforcements is higher than the other coatings (sample I). The crystallinity degree of Ni into

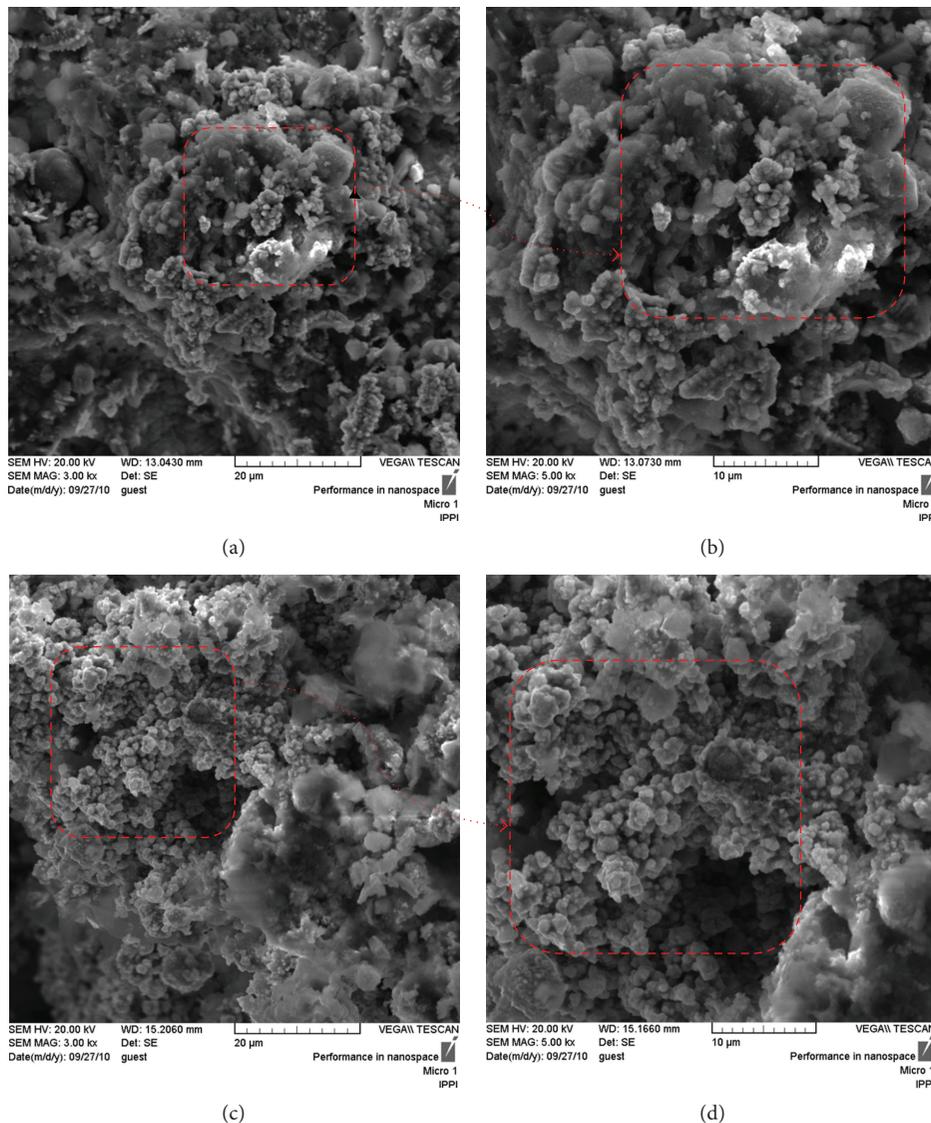


FIGURE 5: SEM micrographs of Ni-SiC-Gr nanocomposite coatings deposited; (a, b) in absence of surfactants (sample II), and (c, d) in the presence of surfactants (sample III).

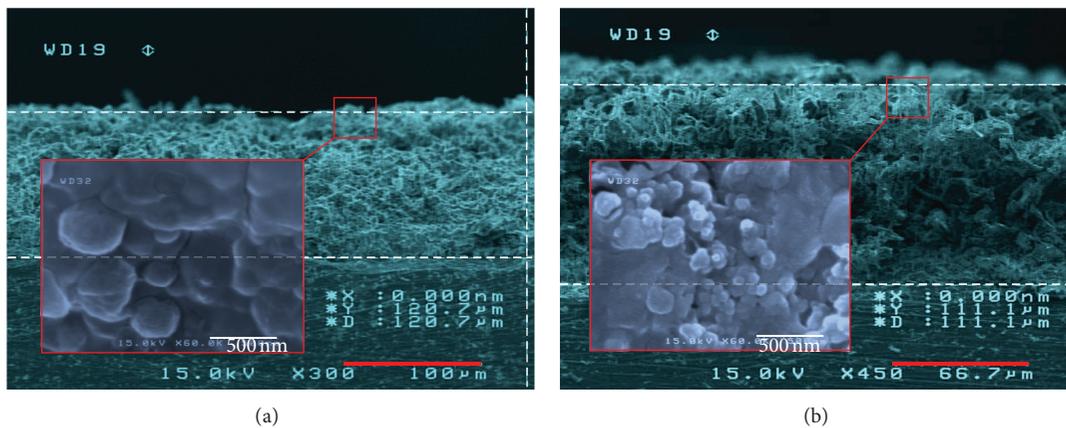


FIGURE 6: FE-SEM image of Ni-SiC-Gr nanocomposite coatings deposited; (a) in absence of surfactants (sample II), and (b) in the presence of surfactants (sample III).

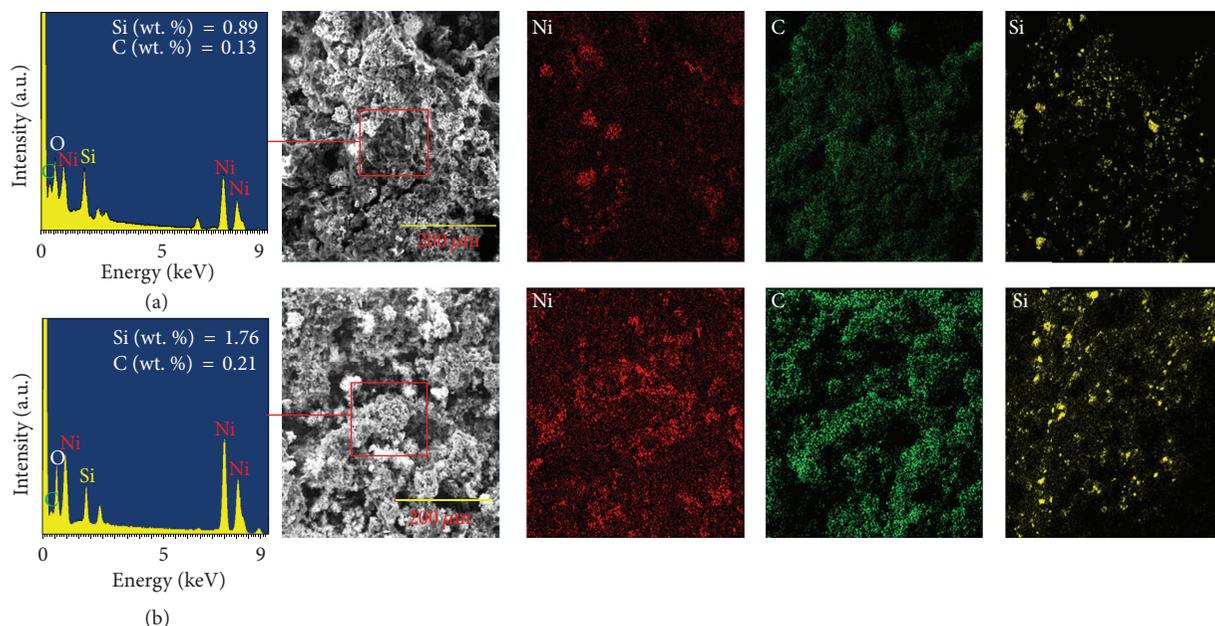


FIGURE 7: EDS and Elemental mapping spectra of Ni-SiC-Gr nanocomposite coatings deposited; (a) in absence of surfactants (sample II), and (b) in the presence of surfactants (sample III).

the coating prepared in absence of surfactants is decreased (sample II). Also, it seems that incorporating of SiC and Gr in electrolyte led to increase of crystallinity degree of Ni; therefore, this indicates that SiC and Gr affected the crystallinity (samples III). The high crystallinity degree of Ni at different circumstances proposes that all of the coatings have appropriate thermal stability at high temperatures [26]. As can be seen in Figure 4(b), crystallite size of the nickel for all the coatings was less than 25 nm. It is revealed that the average crystallite size of Ni is affected in different specimens. Figure 4(c) displays the variation of lattice parameter constant (a_c) as a function of different bath conditions (samples I, II, and III). The lattice parameter values for all the samples are close to the standard value (3.524 Å) which indicates that diverse electrochemical parameters have very little effect on the lattice constant of Ni during plating process. Thus, it could be concluded that the surfactants have very little effect on the lattice constant of Ni during plating process.

3.2. Morphological Features. The surfaces morphology of Ni matrix nanocomposite coatings deposited in absence and presence of surfactants were shown in Figure 5. As can be seen, all the nanocomposite coatings have no regular surface, but it could be observed that some the reinforced particulates with cluster-like structure were dispersed into the matrix. Figures 5(a) and 5(b) display the micrographs of specimen prepared in absence of the surfactants solution which shows the compact reinforcement materials with heterogeneous structure into the nickel matrix. According to Figures 5(c) and 5(d), the surface prepared by the solution containing of surfactants is a relatively homogenous structure which consists of some fine particles. Furthermore, some agglomerates were seen on the surface of the sample. In fact, the used

surfactants during plating led to modification of the surface charge of the particles by the absorbed molecules or ions which are a key factor for preventing of the agglomeration [31, 32]. The cross-sectional images of Ni-SiC-Gr nanocomposite coatings deposited at various bath parameters are shown in Figure 6. As can be seen in Figures 6(a) and 6(b), both coatings deposited at various bath conditions were uniform and homogenous with a good bonding to the substrate. The depositions were controlled to obtain a specific thickness between 50 and 200 μm ; thus, the thickness of coatings in specimens II and III was acquired about 120 and 111 μm , respectively. It seems that the reinforce SiC and graphite particles in the composite coatings act as physical barriers to grain growth and slow down the growth rate (hence thinner composite coatings). While hydrogen evolution at the cathode can also result in coating thickness reduction, such effects are not of electrodeposition from Watts bath. Furthermore, boric acid in the electrolyte is known for suppressing the hydrogen evolution during electrodeposition of nickel from Watts bath. The result is in agreement with other researches [22, 26]. From higher magnification of FE-SEM observations of the coatings surface, it is observed that Ni-SiC-Gr nanocomposite coatings were formed as spherical globules with the average crystallite size of 65–150 nm. It seems that the globules have tendency to adhere onto the clean upper surface of the substrate (St-37).

3.3. EDS Analysis. Figure 7 shows the EDS (energy dispersive X-ray spectroscopy) spectra of Ni-SiC-Gr nanocomposite coating deposited at different circumstances (samples II and III) which confirms the presence of Ni, Si, C, and O elementals in the coatings. There is no specific separation in this analysis between carbon of graphite and carbide

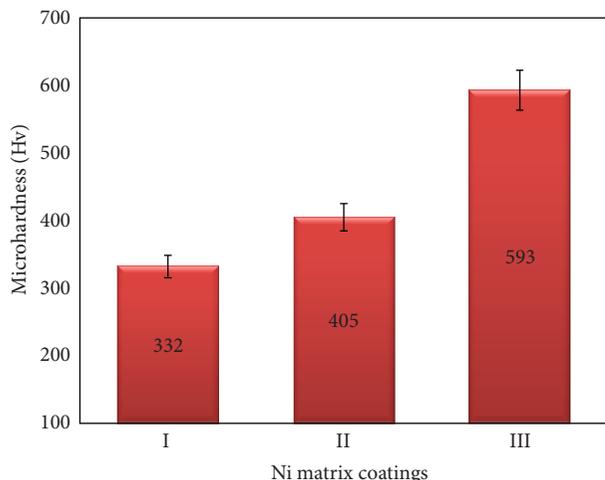


FIGURE 8: Variation of microhardness of Ni-SiC-Gr nanocomposite coatings deposited; (I) in absence of reinforcements powders in electrolyte (SiC and Gr), (II) in absence of surfactants, and (III) in the presence of surfactants.

structures, the displayed carbon signals derived from silicon carbide and graphite sources. Furthermore, it is noteworthy to mention that chemically stable contaminants were not detected and also it was demonstrated that the Ni-SiC-Gr nanocomposite coating onto the St-37 has acceptable purity. As can be seen, the wt.% of SiC and C_g in coating sample III is higher than sample II which indicates that presence of surfactants led to enhancement reinforcements powders contents (SiC and Gr) into the matrix. Also, the distribution of reinforced materials such as SiC and Gr as well as Ni particles was depicted. According to elemental mapping analysis (see Figures 7(a) and 7(b)), the dispersion of particles is uniformly homogenous in the sample, so that the presence of the particles in sample III is more obvious than sample II. It seems that the attendance of reinforcement's particulates resulted in the changing of surface charge on the substrate and affected the deposition rate [33]. Therefore, using of surfactants during electrochemical plating can develop the efficiency of electrodeposition process of nickel-based nanocomposite coatings.

3.4. Microhardness Analysis. The microhardness of nanocomposite coatings as a function of varied bath parameters is presented in Figure 8. Based on this figure, the maximum hardness (593 Hv) is corresponding to the coating deposited in presence of surfactants and reinforcements (sample III). As discussed earlier, this condition also resulted in increasing content of reinforce nanoparticles in the coatings. Thus, the improvement in the hardness of nanocomposite coating at this situation seems to be due to increased content of reinforced particles in the coatings. The minimum hardness of coatings was relevant to the specimen in absence of any reinforcement materials (332 Hv). It reveals that the silicon carbide and graphite particulates have an effective role in mechanical properties and, as a result, promotes the microhardness. In fact, the utilized surfactants cause to absorb more the SiC and Gr particles into the nickel matrix,

and this phenomenon led to an increase of microhardness. It should be mentioned that the SiC particles deposited into Ni matrix played the role of obstacles versus the growth of the Ni grains and the plastic deformation of the matrix [34]. This result is in good agreement with the microscopic observation and XRD analysis.

4. Conclusion

The influence of surfactant and reinforcement powders on the mechanical properties and morphological characteristics of the electrodeposited Ni Matrix nanocomposite coatings was investigated. According to the XRD data, the dominant phases after electrodeposition were nickel, nickel oxide, and SiC. The presence of nickel oxide in the sample deposited in absence of any surfactants confirms that the plating process was performed incompletely. It can be found out that the bath solution without any surfactants is not efficient in deposition of Ni-SiC-Gr nanocomposite coating, while the solution by using the surfactants successfully deposited SiC and Gr particles into the Ni matrix with high crystallinity degree. According to the SEM observations, the nanocomposite coatings displayed a typical cluster-like structure which consisted of some fine sphere particles. Also, from FE-SEM micrograph, the coated thickness was decreased by incorporating of SiC and Gr. Based on EDS spectra, the presence and distribution of Ni, Si, and C particles were uniformly homogenous in the samples. Also, from the mapping analysis system, it can be seen that the presence of the particles in sample III is more obvious than sample II. Eventually, the coating prepared by surfactants solutions illustrated maximum microhardness about 593 HV which is in accordance with the SEM and EDS results.

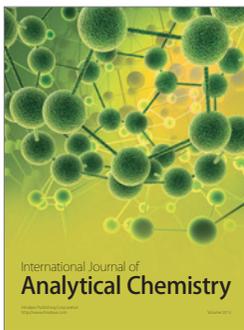
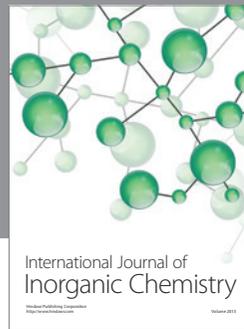
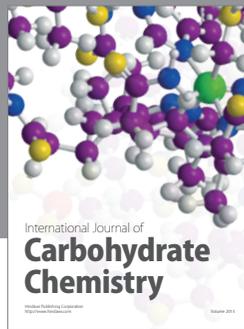
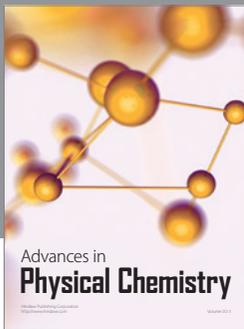
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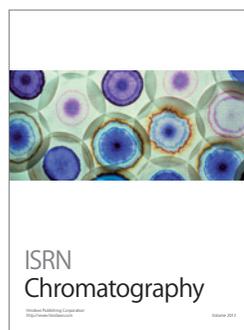
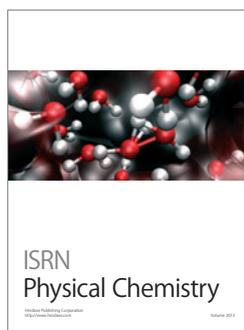
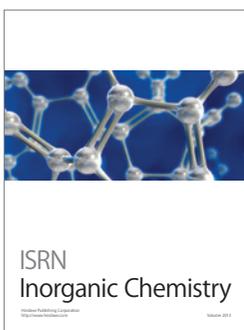
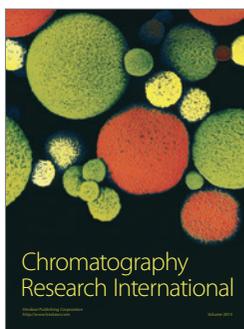
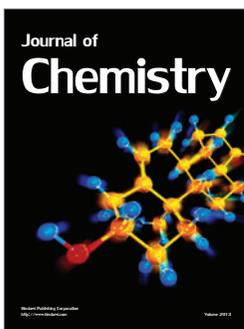
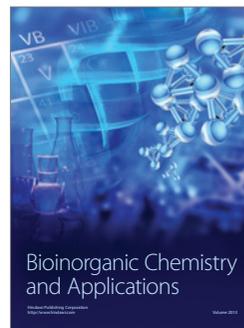
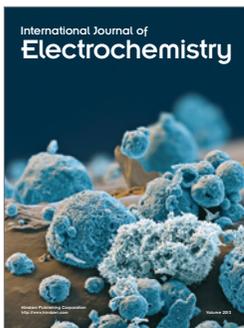
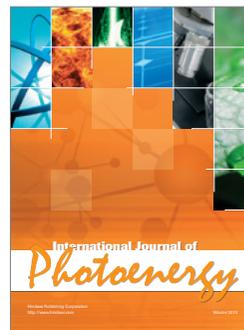
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