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A novel method to enhance silicon incorporation into nickel electrodeposited coatings

Morteza Alizadeh [a, *], Alireza Teymuri [a], Erfan Salahinejad [b], Fatemeh Alijani [a]

[a] *Department of Materials Science and Engineering, Shiraz University of Technology, Modarres Blvd., 71555-313, Shiraz, Iran*

[b] *Faculty of Materials Science and Engineering, K.N. Toosi University of Technology, Tehran, Iran*

* Corresponding author: E-mail address: Alizadeh@sutech.ac.ir (M. Alizadeh)

Abstract

It is known that the addition of silicon to nickel coatings can advantageously improve the coatings' characteristics, especially oxidation resistance. However, the incorporation of Si, because of its relatively low electrical conductivity, into Ni-electrodeposited coatings hardly exceeds 1 wt %. In this work, a new method is pointed out to increase the introduction of Si into Ni-electrodeposited coatings. In this regard, Ni-Si composite coatings were deposited on low-carbon steel by nickel electrodeposition from Watt's baths with the different concentrations of treated and untreated silicon particles. The treated particles were prepared by simultaneous milling of silicon and nickel powders (in 80:20 wt ratio), while the untreated particles were obtained from milling of only silicon particles. The structural characteristics of the produces were studied by scanning electron microscopy, energy dispersive X-ray spectroscopy, and X-ray diffraction. As the most typical finding of the work, it was realized that by using the treated particles, the content of silicon in the deposited composite coating reaches about 9.5 wt %, although it does not exceed 1 wt % when using the untreated particles.

Keywords: Composite coatings; Milling; Electrodeposition; Microstructure

1. Introduction

Nickel coatings are extensively used in industry because of their decorative appearance, good corrosive resistance in aggressive environments, and high catalytic activity in many electrochemical processes [1]. These coatings are frequently deposited on metals surfaces to improve their wear and corrosion behaviors. It has been reported that electrodeposition is an appropriate method to prepare nickel coatings on metals surface since it is a simple, low temperature, and low-cost method [2]. Electrodeposition parameters such as bath composition, pH, temperature, and current density affect the structural, mechanical, and environmental properties of electrodeposited coatings [2-4].

Recently, the electrodeposition method has extensively been used to produce nickel-matrix nanocomposite coatings [1-6]. By the addition of metallic or ceramic powders to the electrolyte bath during nickel electrodeposition, nickel-matrix composite coatings can be fabricated [1,6]. The presence of metallic or ceramic reinforcements in the nickel coatings improves some properties like hardness, wear, corrosion, and hydrogen diffusion resistance [5-8]. Some elements such as Mo, Cu, and Si and ceramic powders like Al_2O_3, SiC, CeO_2, B_4C, and SiO_2, have been used as reinforcements in nickel-matrix composite coatings [1,6,9,10]. These reinforcements refine the nickel matrix grain size and change the texture of the nickel matrix, changing some nickel coating properties [3,6]. Among the mentioned reinforcements, silicon is promising due to potential in protective scale forming on refractory metals and high-temperature alloys [11]. In fact, silicon has beneficial effects on the high-temperature oxidation resistance of heat resistant steels and Nichrome heater alloys. In addition, silicon can improve carburization resistance in high-temperature gas-cooled reactor helium environments [11]. It should be noted that in silicon containing coatings, the formation of siliside phases can occur at high temperatures. Silicide phases have many unique properties such as low density, high melting point, excellent high temperature oxidation, and creep resistance [1].

Thermal spraying, laser cladding, and electrodeposition have been used for the preparation of NieSi coatings [12,13]. Fellner et al. [13] have used the electrodeposition method for codeposition of Ni-Si coating. They found that in the normal condition of electrodeposition, the value of the silicon content in the coating does not exceed one weight percent. They also found that the pretreatment of silicon particles by electroless nickel coating allows the increase of the content of silicon in the coating up to 15 wt percent. Kubisztal et al. [1] used the same method to produce Ni-Mo-Si composite coatings. They have reported that the process of electroless nickel plating on silicon powder affects the content of silicon in the coatings, so that the percentage of silicon in the coating increased from 2.5 to 15 wt%.

The aim of this work is to assess the influence of pretreating silicon powders by ball milling with nickel powders on the silicon content of Ni-Si composite coatings produced by the electrodeposition method. Some properties of the produced composite coatings are also evaluated.

2. Experimental procedure

2.1. *Powder preparation*

In this study, in the first step, silicon powder (Merck,>99.5%) with an average particle size of 50 mm was milled in a planetary ball mill (Sepahan 84D) with tempered steel vial (90 ml) and balls (5 balls of 20 mm and 10 balls of 10 mm in diameter) under a dry atmosphere. The milling process was conducted at the speed of 400 rpm and the ball-to-powder mass ratio of 20:1 at room temperature. It should be noted that to reduce the amount of contamination, the powders obtained from the 3rd milling duration were used for the electrodeposition process. In the second step, silicon and nickel powders (Merck, >99.5%, average particle size: 40 mm) were mixed with 80:20 wt ratio and then mechanically alloyed for 21 h in a planetary ball mill with the above-mentioned conditions.

2.2. *Electrodeposition*

Ni-Si composite coatings were electrodeposited using direct current (DC) from a Watt's Ni bath containing the Si and Si/Ni particles prepared by the milling process on low carbon steel. The composition of the electrolyte bath and deposition parameters are listed in Table 1, where the solution was prepared from analytic grade chemicals and double distilled water.

Table 1. Chemical composition of the bath and conditions of electrodeposition.

Bath composition		Electrodeposition conditions	
Component	Value (g/L)	Parameter	Value
$NiSO_4.6H_2O$	320	Current density (A/dm^2)	2

$NiCl_2.6H_2O$	40	pH	4 ± 0.2
H_3BO_3	50	Temperature (ºC)	50 ± 1
$C_{12}H_{25}NaO_4S$ (SDS)	0.1	Agitation speed (rpm)	250
Silicon particles	0-40		

The silicon and silicon/nickel particles were distinctly added to the electrolyte bath, and ultrasonic waves were applied to the electrolyte bath for 20 min in order to prevent agglomeration of the silicon particles. In addition, prior to electrodeposition, the electrolyte bath was stirred for about 24 h by using a magnetic stirrer at 600 rpm to get de-agglomerated silicon particles. During the composite deposition process, magnetic stirring was also employed to inhibit the precipitation of the particles and to keep the electrolyte fresh. Both of the electrodes were dipped in 500 ml of the test electrolyte dispersions. The anode in this work was a pure nickel with 50 × 30 × 4 mm dipped size and the cathode was a low carbon steel with 20 × 15 × 1.5 mm dipped size. The surface area of the anode was chosen almost five times larger than the cathode to reduce the polarization effect, and the distance between the cathode and anode was about 3 cm. Prior to the electrodeposition process, the surface of the samples was mechanically polished with emery papers up to No. 2500, sonicated in acetone for about 10 min, and then activated in 10% hydrochloric acid for about 1 min at room temperature.

2.3. Characterization

The surface morphology of the prepared coatings was examined using a VEGA3 TESCAN scanning electron microscope (SEM). The composition of each coating was examined by energy dispersive X-ray spectroscopy attached to SEM, over three uniform regions. The

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structural analysis of the treated particles was performed by using X-ray diffraction (XRD, D8 Bruker diffractometer) by Cu target and Kα_1 radiation with 0.15406 nm wavelength. An X-ray diffractometer was operated at a scanning rate of 0.03 min^{-1} for 2θ ranging from 20 to 70°. The XRD quantitative analysis was done by the Rietveld method using the MAUD software package, version 2.3.

3. Results and discussion

3.1. Electrodeposition with the untreated silicon powder

Fig. 1 shows the EDS analysis of the silicon powders milled for 21 h. As it can be seen, there is only one peak which is located at 1.83 keV and is attributed to silicon. The milled silicon particles with different concentrations were used in the electrolyte bath and electrodeposition was done to produce Ni-Si composite coatings. The effect of the concentration of the silicon particles in the electrolyte bath on the content of the silicon particles in the produced coatings is shown in Fig. 2. Each point of the figure has been extracted from the average value of three EDS measurements at different locations on each coated sample. As it can be seen, the weight percent of the deposited particles continuously increases with increasing the silicon particles concentration in the electrolyte (up to 40 g/L). The weight percent of the silicon particles in the deposited coatings from zero in the pure nickel coating increases to about 0.7 in the coating deposited in 40 g/L silicon particles in the electrolyte. The increasing trend of the reinforcement in the deposited coating by increasing the reinforcement concentration in the electrolyte bath is consistent with other researches [6,9,13]. For example, it has been shown that the content of SiO_2 is increased from 1.3 to 1.9% by increasing SiO_2 nano-particles in the electroplating bath from 2.5 to 20 g/L [9].

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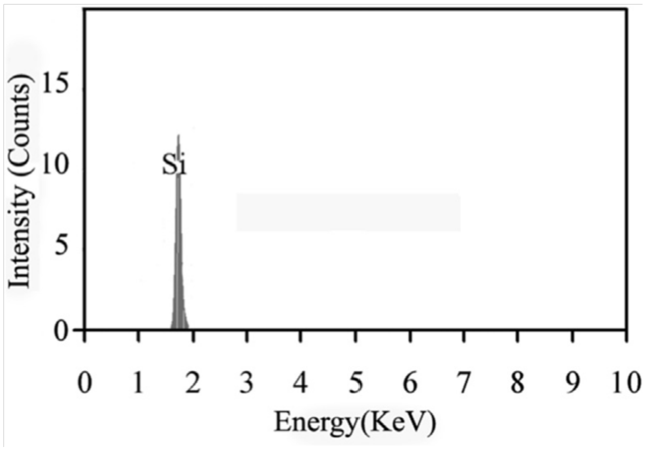

Fig. 1. EDS analysis of the untreated silicon powder after 21 h of milling.

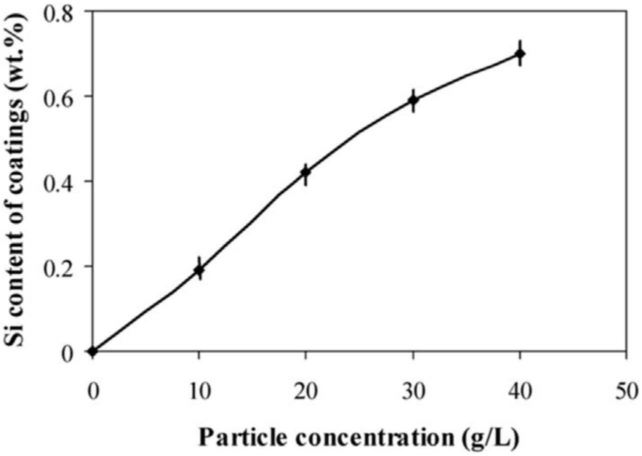

Fig. 2. Silicon particle content in the coatings as a function of the concentration of the untreated particles in the electrolyte bath.

Fig. 3 shows the surface SEM image of the Ni-Si composite coating deposited in an electrolytic bath with the silicon particle concentration of 40 g/L. As it can be seen in Fig. 3a, the coating surface consists of two regions: smooth surface and protruding points. According to Fig. 3b, the smooth surface has a regular pyramidal and compact microstructure. This morphology is similar to pure nickel film morphology which is shown in Fig. 4. Comparing Figs. 3b and 4 shows that the morphology of the coating is influenced by the addition of the silicon particles in the electrodeposition bath. As it is seen, the size of grains in the Ni-Si composite coating decreases due to the presence of the silicon particles. The silicon particles incorporated in the coating act as nucleus for the deposition of nickel atoms in the electrodeposition process [14,15]. In fact, the presence of the silicon particles in the coating presents more nucleation sites available for nickel atoms in comparison to the pure nickel coating. Therefore, when nucleation sites increase in the coating, the crystalline growth is prevented during electrodeposition and the grain size of the coating is decreased [15].

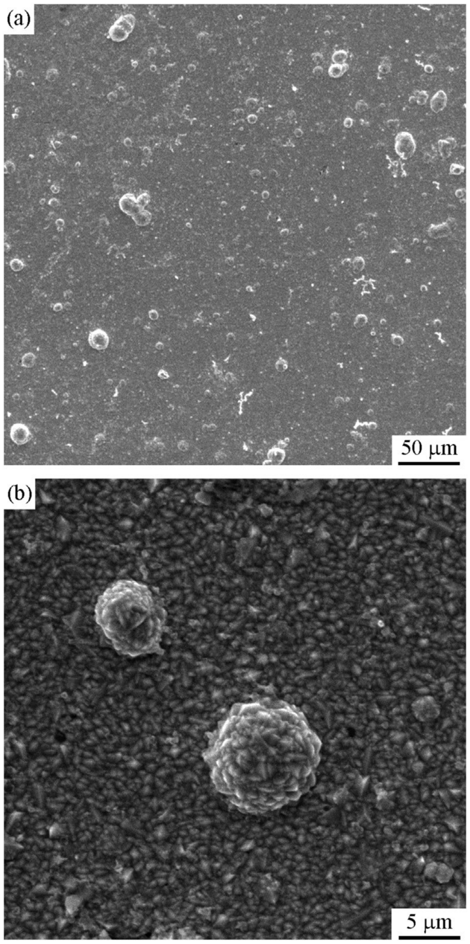

Fig. 3. SEM micrographs of the Ni-Si composite coating deposited in an electrolytic bath with 40 g/L of the untreated silicon particles.

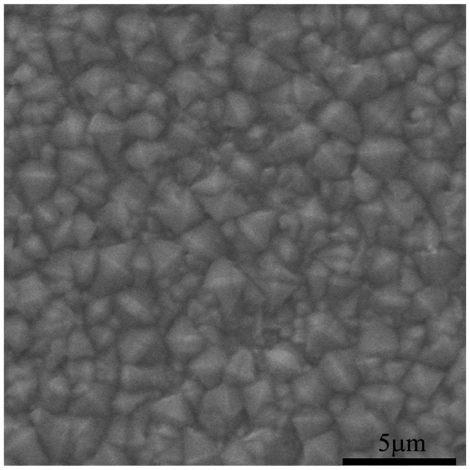

Fig. 4. SEM micrograph of the pure nickel coating.

3.2. *Electrodeposition with the treated silicon powder*

For preparing the new reinforcement particles, the silicon and nickel powders were mixed and then milled for 21 h. Fig. 5 shows the SEM image of the Si-Ni powder after the milling process. As it can be seen, the milled powders have an equiaxed morphology and their size has a wide range. It has been reported that mechanical alloying of powder mixtures has three main stages: cold welding, fracturing, and steady-state condition [16]. At the early stages of milling, the cold-welding mechanism is dominated so the particles are cold welded together. By increasing the milling time, the fracturing of the cold-welded powders occurs. In fact, in the milling process due to the accumulation of strain energy, the milled powders are work hardened and their hardness is increased [17]. In this stage of the milling process, the hardened particles are fragmented and consequently the particle size is reduced. In the next stage, cold welding and fracturing occur simultaneously and a balance may be achieved between their rates. In this stage, according to Fig. 5, the particles show an equiaxed morphology.

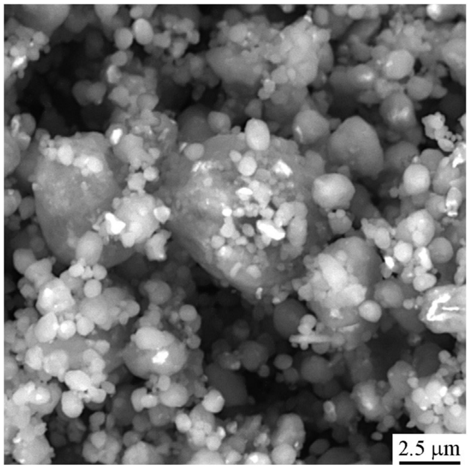

Fig. 5. SEM image of the Si-Ni powder after 21 h of milling.

The XRD profile of the Si-Ni powders milled for 21 h is presented in Fig. 6. This figure shows that only Si and Ni are present and there is no new phase in the XRD pattern. This means that diffusion between Si and Ni elements during milling has not occurred. It has been reported that during milling, severe plastic deformation is introduced to powders and a variety of crystalline defects such as dislocations, stacking faults and grain boundaries are created. The created defects decrease diffusion distances, so that the diffusivity of elements is increased [18]. However, the milling time in this work was not enough for diffusion and alloying; therefore, no new phase appears in the XRD pattern.

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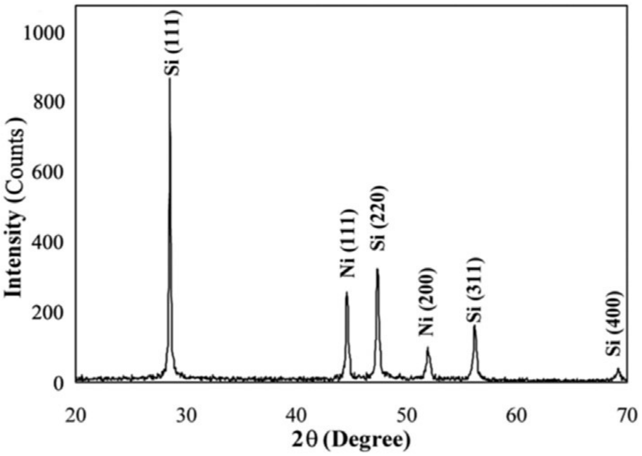

Fig. 6. XRD pattern of the milled silicon and nickel mixture powders after 21 h.

The crystallite size of the milled powders, as one of the important structural parameters, was determined by the XRD technique. The crystallite size of the silicon and nickel phases was determined by the Rietveld method using MAUD software to be about 79 and 64 nm, respectively. The reduction in the crystallite size of the powder particles occurs during milling. It is due to severe collisions between the balls and powder particles, introducing a severe plastic deformation to the powders. The severe plastic deformation creates dislocations and grain boundaries and refining happen [18].

The treated Si-Ni powder was used in the electrolyte bath with various concentrations and electrodeposition was done in order to produce Ni-Si composite coatings. In Fig. 7, the content of silicon in the produced Ni-Si coatings has been plotted as a function of the treated particles concentration in the electrolyte bath. Each point of Fig. 7 has been extracted from the average value of three EDS measurements at different locations on each sample. From this figure, it is inferred that the content of silicon deposited in the composite coating depends remarkably on the treated powder content in the bath. As is seen in Fig. 7, by increasing the treated powders in the electrolyte bath, the silicon content in the coating is increased and reaches about 9.5 wt percent at the concentration of 40 g/L.

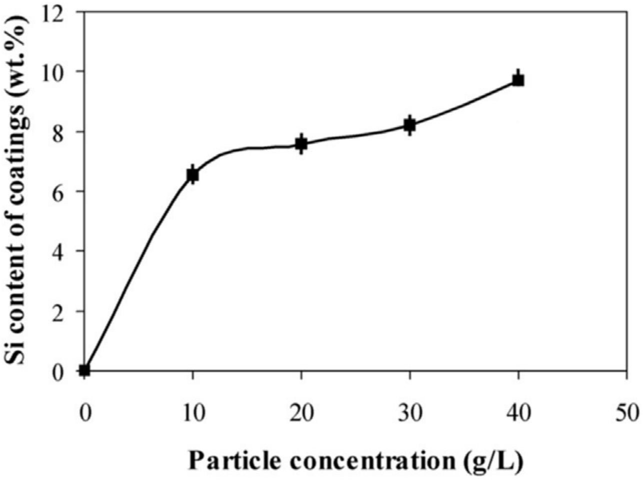

Fig. 7. Silicon particle content in the coatings as a function of the concentration of the treated particles in the electrolyte bath.

A comparison of Figs. 2 and 7 shows that by increasing the silicon particles in the electrolyte bath, the silicon content of the deposited Ni-Si composite coating is increased in both the methods (using the untreated and treated powders). However, it is clear that the use of the treated powder increases the silicon content in the Ni-Si composite coating more than that of the untreated powder. As is seen, the difference between the silicon content in the deposited composite coating in these two methods is significant. For instance, the silicon content in the composite coating deposited in the bath with 40 g/L of the treated powder is about 13 times more than that in the composite coating deposited in the bath with 40 g/L of the untreated powder. Milling of the silicon and nickel powder mixture creates new particles consisting of both the elements by the mechanism of cold welding and fracturing [16]. Fig. 8 depicts the EDS elemental mapping and point spectrum of the 21 h-milled silicon and nickel powders.

As it can be seen from this figure, a particle may have both of silicon and nickel elements. The presence of nickel and silicon in a particle changes the electrical conductivity of the particle. The electrical conductivity of silicon is 4×10^3 Sm^1 while it is 12.84×10^6 Sm^1 for nickel [13]. Since silicon and nickel powders are connected together in the milling process and the produced particles consist of both elements, the electrical conductivity of the particles is increased. The electrical conductivity of the particles affects their ability for incorporation into the matrix layer. In fact, the electrical conductivity of the particles influences the distribution of the electric field and the transport of electroactive species to the cathode surface [13]. In addition, the conductive particles have an effect on the reduction of Ni^{+2} ions on the cathode surface. It is well known that Ni^{+2} ions should be reduced on the cathode surface during the electrodeposition process. When a particle reaches the cathode surface, the reduction of adsorbed ionic species takes place and the particle is incorporated in the metal matrix [19]. The incorporated particles affect the discharge process of metal ions. Depending on the conductivity of the incorporated particles, two states arise. Since the incorporated untreated silicon particles in the nickel matrix have very low electrical conductivity, they act as an obstacle to the current flow on the cathode surface [20]. In this case, according to Fig. 9a, the current cannot pass from nonconductive silicon particle and it has to go around the particle to reach the surface cathode. Therefore, Ni^{+2} ions cannot be reduced on the surface of nonconductive silicon particles and they are only reduced in the vicinity of the untreated silicon particles (Fig. 9b). Thus, it is possible that the silicon particles are not engulfed by nickel ions completely. When the particles cannot be completely engulfed by Ni^{+2} ions, they may sink under gravity or may be disconnected by the turbulent electrolyte [20]. Consequently, the content of the silicon particles in the electrodeposited composite is reduced when the untreated silicon particles are used (see Fig.

9d). In contrast, the current directly passes from the surface of the treated particles which have been incorporated in the cathode surface (Fig. 10a). Therefore, Ni^{+2} ions not only can be reduced in the vicinity of conductive particles, but they can also be reduced on their surface. In this case, the silicon particles are engulfed by Ni^{+2} ions faster than the untreated silicon particles. Thus, the probability of sinking of the particles under gravity and their disconnection under the turbulence electrolyte will be lower than that of the former case (Fig. 10b). Accordingly, it can be deduced that the high conductivity of particles can promote the content of particles into the produced composite coating during the co-deposition process (see Fig. 10d). So that in this work, the content of the silicon particles was increased to about 9.5 wt percent by using the treated silicon particles in the electrolytic bath (see Fig. 7), while it did not exceed 0.7 wt percent by using the untreated silicon particles in the electrolytic bath (see Fig. 2).

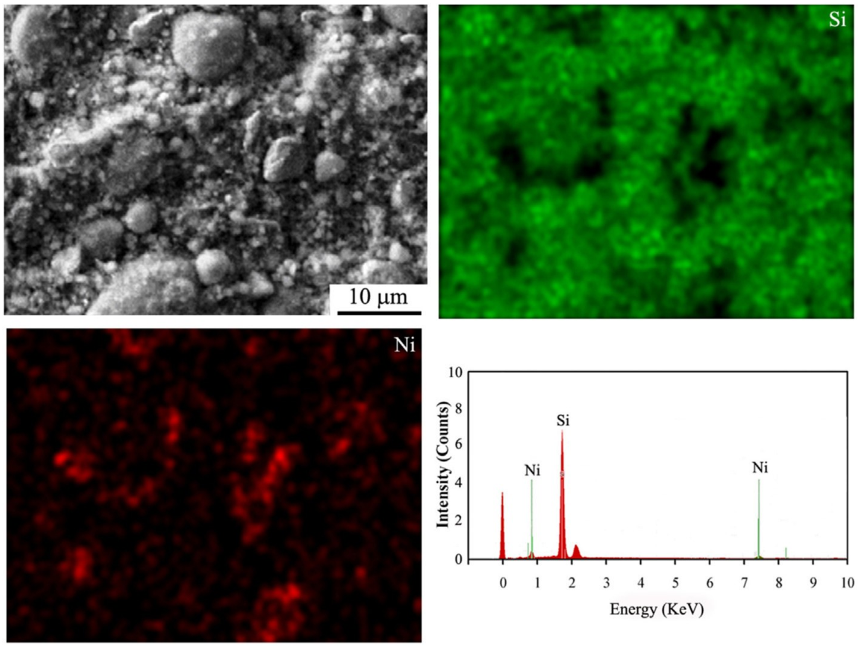

Fig. 8. Elemental X-ray maps and point spectrum of the 21 h-milled silicon/nickel powders.

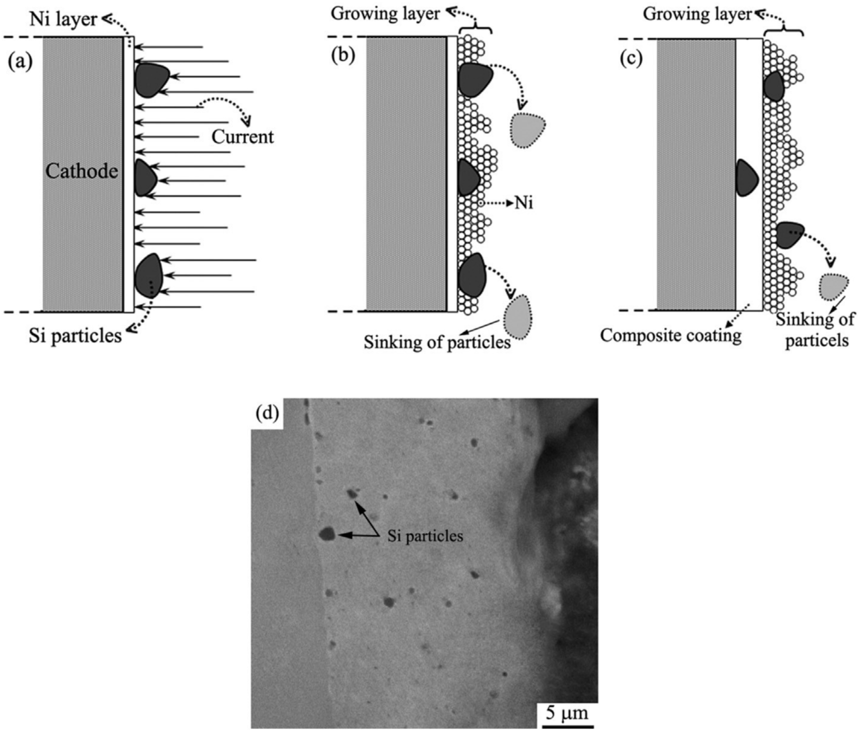

Fig. 9. Electrolytic current distributions in the vicinity of the applied particles (a), schematic views of composite electrodeposition (b,c), and cross-sectional SEM morphology of the prepared composite coating (d) for the untreated particles.

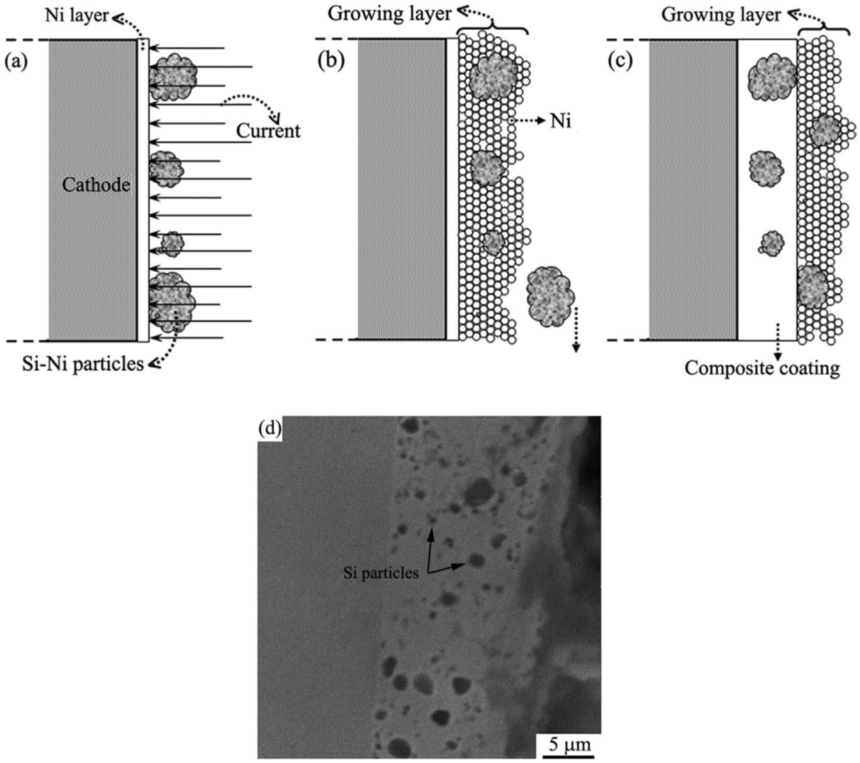

Fig. 10. Electrolytic current distributions in the vicinity of applied particles (a), schematic views of composite electrodeposition (b,c), and cross-sectional SEM morphology of the prepared composite coating (d) for the treated particles.

Fig. 11 presents the surface morphology of the Ni-Si composite coating deposited in an electrolyte bath consisting of 40 g/L of the treated powder in two magnifications. It is clear that the surface morphology is not the same as the pure Ni coating (see Fig. 4). As shown in Fig. 11, with the addition of the Si particles, the grain size is reduced and the morphology is changed to spherical crystals. At the higher magnification (Fig. 11b), the rough morphology can be seen, while it consists of protruding points. It has been reported that by adding the second phase particles to the Ni matrix in the production of composite coatings, the preferred orientation is changed to a random orientation and the morphology of the coating

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is changed [21]. Thus, it can be concluded that the increase of the silicon content in the deposited composite coating has a direct effect on the surface morphology of the coating. It can be seen from Figs. 3, 4 and 11 that the roughness increases with the amount of the particles in the coatings. It has been reported that in the electrodeposition process of nickel matrix composites, at first nickel ions are deposited preferably on the substrate and a thin nickel layer is produced [6,22]. Henceforth, the second phase particles dispersed in the deposition bath and nickel ions are deposited simultaneously on the growing nickel matrix [6,22]. The silicon particles in the electrolyte bath adsorb some positive ions and move directly to the cathode surface under the effect of electric force [20,23]. They then are engulfed by the growing nickel matrix. The present silicon particles in the nickel matrix increase the number of nucleation sites and can also postpone the growth of nickel grains and change the preferred growth orientations of grains [22]. Therefore, the silicon particles can prevent the further growth of the Ni grains and change the coating surface morphology.

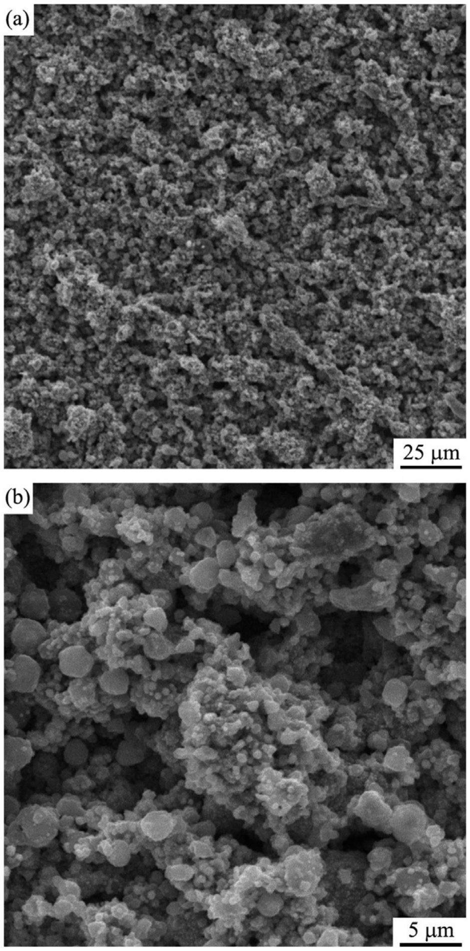

Fig. 11. SEM micrographs of the Ni-Si composite coating deposited in an electrolytic bath with 40 g/L of the treated silicon particles.

Since the composite coating produced by the treated particles has more amounts of the silicon particles, it is expected that the surface morphology of the composite coating be different from the produced composite coating by untreated particles.

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4. Conclusions

In the present work, a novel method was used to enhance silicon incorporation into nickel electrodeposited coatings. Ni-Si composite coatings containing untreated (milled Si) and treated (milled Si þ Ni) particles were electrodeposited on low carbon steel. The outcome of this study can be summarized as follows:

1. No new phase was created after 21 h of milling of the siliconnickel powders, where it consisted only of the silicon and nickel phases.
2. Ni-Si composite coatings with low and high Si contents weresuccessfully produced by electrodeposition.
3. By increasing both the treated and untreated particles in theelectrolyte bath, the content of the silicon particles in the deposited coating was increased.
4. The XRD analysis showed the preparation of a nanostructuredNi-Si coating.
5. The Si content of the coating deposited by the treated particlesreached 9.5 wt %, although it did not exceed 1 wt % while using the untreated particles.
6. By increasing the silicon content in the composite coating, thesurface morphology was changed from a regular pyramidal compact microstructure to a spherical crystal microstructure with a rough surface.

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