



Preparation and Characterization of Ni-diamond Composite Coatings

Using a Modified Sedimentation Co-deposition Technique



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Abstract

Composite coatings are prepared by the co-deposition of inert particles with metal/alloy in order to promote the hardness, wear and corrosion resistance of the coatings. Micro crystalline Ni based composite coatings were prepared by conventional electrodeposition method from a Watt's-type electrolyte containing different contents of diamond particles. A modified sedimentation co-deposition (MSCD) electro-deposition method was used to deposit Ni-diamond (Ni-D) composite coatings having more diamond particles in the Ni matrix. Results show that Ni-diamond composite coating prepared by the MSCD method contains quite high content of diamond (67-97%) as evidenced by EDS analysis. In contrary the traditional electrodeposition method shows low content of diamond (≈ 10 -20%). The hardness and wear resistance of the MSCD Ni-diamond coating are markedly increased in comparison to the conventionally electrodeposited coatings.

Keywords: Composite coatings; conventional electrodeposition; sedimentation co-deposition; corrosion resistance.

1. Introduction

Composite coatings find increasing interest as alternatives of hard chrome coating to avoid the generation of hexavalent carcinogenic chromium ion. Composite plating involves co-electrodeposition of inert particles with metal/alloy which increases the hardness, wear and corrosion resistance of the coatings. The fabrication of micro crystalline Ni based composite coatings was carried out using conventional electrodeposition method from Watt's bath containing nano-diamond particles [1]. The impediment of particles of diamond whose size in the nano scale apparently alters the Ni-W electrodeposited coating properties [2]. The codeposition of diamond particles with uniform distribution was successfully obtained in the electrodeposited Ni-W matrix in nickel sulfamate baths using a rotating disc and wire geometries. The impediment of diamond particles was maximum (about 21.1 vol.%) at a bath loading of 1 g/l and at a

stirring rate of 180 rpm. The microhardness as well as the wear resistance of the coatings was increased [3]. Typical non-conductive diamond and conductive molybdenum disulfide were chosen to fabricate Ni composite coatings. Ni/MoS₂ composite coatings cannot be achieved using this method, high-particle-content Ni/diamond coatings can [4]. Nano-diamond particles were co-deposited on AISI-1045 steel substrates with nickel from a Watt's type bath by conventional electrodeposition technique. Co²⁺ addition to the Ni/diamond plating bath apparently increased the amount and uniformity of dispersed nano-diamond particles in the metal matrix. The produced nano composites increased hardness and wear resistance to a large extent. Co²⁺ ions mostly act as a cationic stimulator, promoting the co-deposition of nano-diamond particles with nickel. This leads to intensify the positive contribution of the incorporated nano-diamond particles [5]. The co-deposition of Ni and nano-diamond powder on Cu

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alloy was performed by the direct current and pulse plating methods. The microhardness of the specimen treated with 40 g/L diamond powder concentration was as high as 611 HV using pulse plating technique with stirring speed below 300 rpm and plating temperature 30-50°C as optimum conditions for Ni-diamond composite plating [6]. The published results proved that the addition of micro-diamond particles to the plating bath shifts the reduction potential of Ni towards more negative values [7]. Ni-B/diamond composite coatings have been prepared using electrophoretic deposition and electroless deposition techniques. These methods produced very high hardness, which is comparable to hard coatings prepared by dry processes [8]. Nickel/surface-modified diamond composite electroplating was prepared as a new application of surface-modified diamond particles. Oxygenated surface diamond powder was treated with (tridecafluoro-1,1,2,2-tetrahydrooctyl) triethoxysilane ($\text{CF}_3(\text{CF}_2)_5(\text{CH}_2)_2 \text{Si}(\text{OC}_2\text{H}_5)_3$), which is a kind of silane coupling reagent, for the introduction of the perfluoro ($\text{CF}_3(\text{CF}_2)_n-$) groups on diamond surface. The surface-modified diamond powder was suspended into Watt's nickel bath, with a surfactant. The wear resistance of the deposited Ni plate was improved by the introduction of surface-modified diamond particle. The surface contact angle with water increased with increasing the amount of the surface-modified diamond particle introduced in the deposited Ni plate [9]. Ni-diamond composite coatings with 1.47–15.6 wt.% particle content were prepared by composite electrodeposition technique in Watt's bath [10]. Interface force per unit area between particle and cathode was calculated to analyze and predict the mechanism of composite electrodeposition. The highest mass fraction of codeposited diamond particles was obtained at medium stirring rate. Diamond particles adsorbing positive charges are direct transported toward cathodic surface due to stirring. Ni/diamond composite coatings having various amount of diamond (up to 57.6 vol.%) were prepared by electrodeposition from a Watt's nickel bath contained dispersed diamond particles with the aid of a redox-active surfactant containing an azobenzene moiety. The microhardness and the wear resistance of this composite coating were about 4.5 and 14 times higher than that of a nickel-only coating. It is demonstrated that the particle size of the nanodiamond affects the texture of the coatings as well as their susceptibility to electrochemical attack in 3.5% NaCl solution [11-13]. Wang, et al published a work on Effect of Colloidal Stability of Diamond Nanoparticles on the Ni-W/Diamond Electro-co-deposition Composite Coatings [14]. More recent work on morphology, hardness, and

wear properties of Ni-base composite coating containing Al Particle was issued by Mousavi et al [15].

Ni-Diamond composite coatings prepared using traditional co-deposition techniques have relatively low diamond particles content. A modified electro-deposition method called sedimentation co-deposition (SCD) was developed. SCD technique depends on keeping diamond particles suspended in the plating bath, allowing the incorporation of more diamond particles in the Ni matrix. Therefore, diamond particles could be easily co-electrodeposited with metal ions on the substrate because of the gravity of diamond particles [16-17]. Nickel-diamond and nickel-cobalt diamond coatings prepared using SCD technique on mild steel substrate and their tribological properties were evaluated. The presence of cobalt increased volume percent incorporation of diamond powder, hardness and wear resistance of the coatings [18]. Ni-diamond composite coatings with high concentration and uniform distribution of diamond particles were prepared using SCD technique from Watts-type electrolyte without any additives. Monolayer of Ni-diamond composite coatings ($\sim 40 \pm 5 \mu\text{m}$) was successfully prepared by the newly developed setup for SCD technique. This set up gave high concentration and uniform distribution of diamond particles of Ni-monolayer diamond composite coatings. Anti-wear and cutting performance was highly better than those prepared via conventional co-electrodeposition (CED) technique and pure Ni coatings. It was shown that the diamond particle content is not only the controlling factor of the wear resistance and cutting performance, the distribution of diamond particles is also very important factor as well [19]. Recent work on the comparison of mechanical properties of Ni-diamond composite coatings fabricated by different methods was published by Hansheng, et al [20].

The present work addresses the preparation of Ni-D composite coating on steel substrate using a newly developed technique based on sedimentation co-deposition (SCD) with the purpose of incorporation diamond particles in the coating. The newly developed method is called modified sedimentation co-deposition (MSCD).

2. Experimental

2.1. Conventional electroplating technique

Carbon steel (CS) samples having dimensions of 100 x 20 x 2 mm were used as cathode. Samples were mechanically polished using emery papers of different grades from 240 up to 1200 so as to acquire mirror-like finish. Polished samples were immersed

in a degreasing path containing 20 g/L NaOH, 20 g/L sodium phosphate, and 20 g/L sodium carbonate at 60°C for 30 min. Degreased samples were washed and then pickling in diluted HCl. Finally, samples were washed in distilled water and air dried. Two pure Ni plates were used as anodes in the electrochemical cell to enable the electro deposition on the two faces of cathode. Electrodepositing was performed in the standard Watt's bath in the absence and in the presence of diamond particles (37-44 µm). The Watt's bath composition and deposition conditions are listed in Table 1. The diamond powder used in deposition is purified by chemically etching with 10 vol. % with H₂SO₄ for 1 hour then cleaning diamond powder with acetone and distilled water before using in the electrodeposition bath. The electrodeposition temperature and stirring rate was controlled using hotplate with magnetic stirrer.

2.2. Modified Sedimentation Co-deposition (MSCD) Technique.

MSCD procedure involved the immersion of the cathode (CS) in a solution containing diamond particles under constant voltage of 6 V for 5 minutes. In this step diamond particles are adherently electrodeposited on the CS substrate. Subsequently, CS samples are transferred to the standard Watt's bath where electrodeposition of nickel was performed for 1 hour as described above. The difference between the MSCD and the previously described SCD lies on the fact that MSCD is performed two steps allows the incorporation of considerably large amount of diamond particles on the substrate surface. In contrary the SCD proceeds in one step with a setup allowing more incorporation of diamond particles in the Ni-diamond composite coating compared to the conventional electrodeposition (CED) method.

2.3. Coating Characterization

2.3.1. Surface characterization

Prior to surface analysis, all coatings were washed in deionized water and ultra-sonicated in acetone for 5min. to improve the images quality. (Thickness gauge 6000-N4) was used to determine the thickness of coating. Field emission scanning electron microscope (FESEM, a Sanxiao cy-828.900 W) was used to examine the surface morphology of coated

samples before and after corrosion testing. The elemental analysis of the coatings was performed using electron dispersive spectroscopy (EDS) unit attached with the FESEM instrument. The phases of the composite coatings were detected via X-ray diffraction using an X'Pert Pro diffractometer (Panalytical). The particle contents in the composite coatings was evaluated using the gravimetrically method. Deposits were stripped in nitric acid which was filtered and the mass of diamond powder in the deposit was estimated gravimetrically.

2.3.2. Mechanical properties

Vickers micro hardness for coatings surfaces were measured using a micro hardness tester under an indentation load of 100 g for 15s at different locations of the sample. The average value of the 6 measurements (except the maximum and minimum values) is quoted as the hardness of the film. The surface profile (Electrometer 224, Digital profile Gauge) was used to measure the surface roughness.

Wear tests were carried out using sand wear method (The Abrasion Rate Measurements) using (IKA RW 20 digital) rotator. For measurement of the abrasion resistance, samples (coated samples by SCD, CED & base metal) were rotated in a mixture of sand/water medium with a ratio 2:3 (Shawki et.al., 1997). The rotation speed was kept constant at 400 rpm and the abrasion effect was measured in terms of weight loss expressed in milligrams.

2.3.3. Electrochemical Testing

To evaluate the electrochemical behavior of the uncoated and coated samples in 0.6 M NaCl solution, Electrochemical impedance (EIS) technique has been used. Tests were carried out using a computerized potentiostat (Autolab PGSTAT 30). Tests were performed using three electrode corrosion cell, the coated sample was used as the working electrode with a 1.0 cm² exposed area, a saturated calomel electrode (SCE) was used as the reference electrode, and a platinum sheet as the counter electrode. EIS experiments were carried out at room temperature over the frequency range between 1 Hz and 65 kHz OCP. The peak to peak amplitude of the sinusoidal voltage signal was 10 mV.

Table 1: Bath compositions and deposition conditions

Chemicals	Parameters
NiSO ₄ ·6H ₂ O (300 g/ L)	Current density (5m A cm ⁻²)
NiCl ₂ (60 g/ L)	Temperature (55°C)
H ₃ BO ₃ (40 g /L)	pH = 5
Diamond powder, 37-44 µm (5,10, 20,40 g/L)	

3. Results and Discussion

CED or MSCD techniques using Watt's bath prepared Ni-diamond composite coatings. The obtained coatings were investigated under FE-SEM (Fig.1a-d). Figure 1 shows the SEM images of Ni-D composite coatings with different magnifications.

Images 1a and b revealed that the Ni matrix prepared by conventional electrodeposition cover the substrate surface while incorporated diamond particles are not seen due to the fact that nickel represents the major constituent of the coating and also the formation of nickel aggregates that hide diamond.

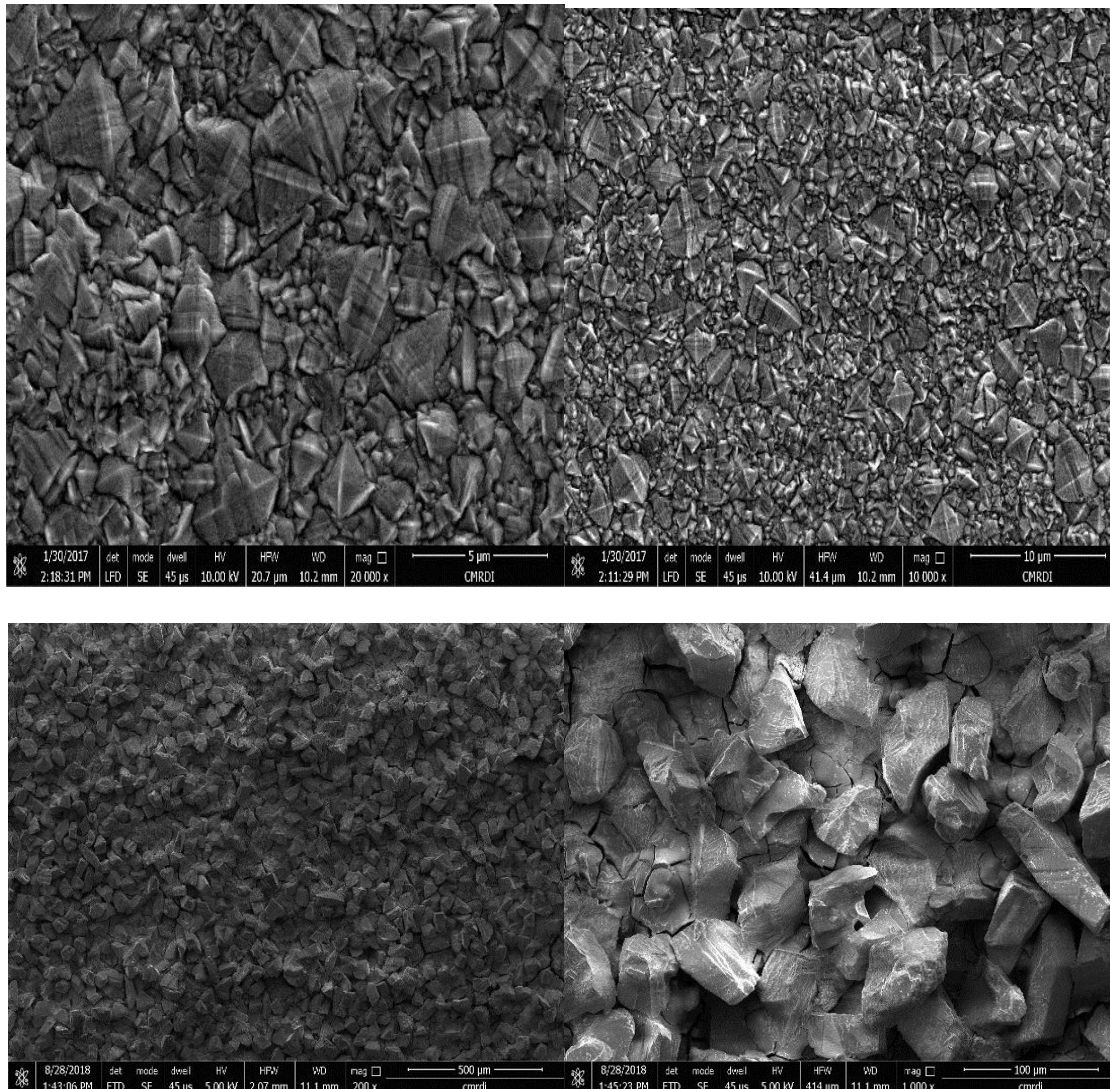


Fig. (1): FE-SEM image with different magnifications of: a and b) Ni-D composite coating prepared by CED at $5\text{mA}/\text{cm}^2$, 60°C , and $10\text{g}/\text{L}$ diamond concentration, c and d) Ni-D composite coating prepared by MSCD, the coating was prepared at $5\text{mA}/\text{cm}^2$, 10 min. and 60°C .

Moreover, Figs.1 c and d show the FE-SEM images at two different magnifications of Ni-D composite coatings prepared by MSCD technique using diamond concentration $10\text{g}/\text{L}$, $5\text{mA}/\text{cm}^2$, 60°C and 10 min. In such a case the diamond particles get larger and could be observed.

The effect of deposition time using MSCD technique on the diamond percent in the coating layer has been investigated. EDS analysis of coated samples after

different times are shown in Fig.2. Interesting findings are prevailed where EDS of analyzed areas shows very high content of diamond ($>97\text{wt.}\%$) and very low content of Ni ($<3\%$) for samples coated in the presence of $10\text{g}/\text{L}$ diamond in the plating bath for deposition time 10 min. Similarly, sample coated for 5 min. shows slightly lower percent of diamond ($\sim 93\text{wt.}\%$) and slightly higher content of Ni ($\sim 7\%$). On the other hand, sample coated after 2 min. shows markedly lower percent of diamond ($\sim 67\text{wt.}\%$) and

relatively high content of Ni (~32%). In contrary, the EDS of analyzed areas of Ni-composite prepared by CED technique shows very low content of diamond (~5 wt.%) for the sample coated in the presence of 10g/L diamond in the plating bath for deposition time 60 min.. Additionally, the effect of deposition time on the coating thickness obtained using MSCD technique is shown in Table 2. The data reveals that the measured thickness of the coating varies in the range 40-60 μm.

The X-ray diffraction (XRD) patterns of Ni-D composite coatings prepared either by CED or MSCD technique operated for 10 minutes are presented in Fig. 3. The diffraction spectra illustrate

five peaks at 2θ of 44.5°, 51.9°, 76.4°, 93.0 and 98.4 which are indexed to Ni (JCPDS File No. 04-0850) corresponding to phases (111), (200) and (220), while the other three peaks at 2θ 44°, 65.5°, and 75.5° which are indexed to diamond (JCPDS File No. 06-0675) and corresponding to phases (111), (000) and (220). XRD results demonstrate that the Ni-D composite coatings were successfully fabricated using MSCD method.

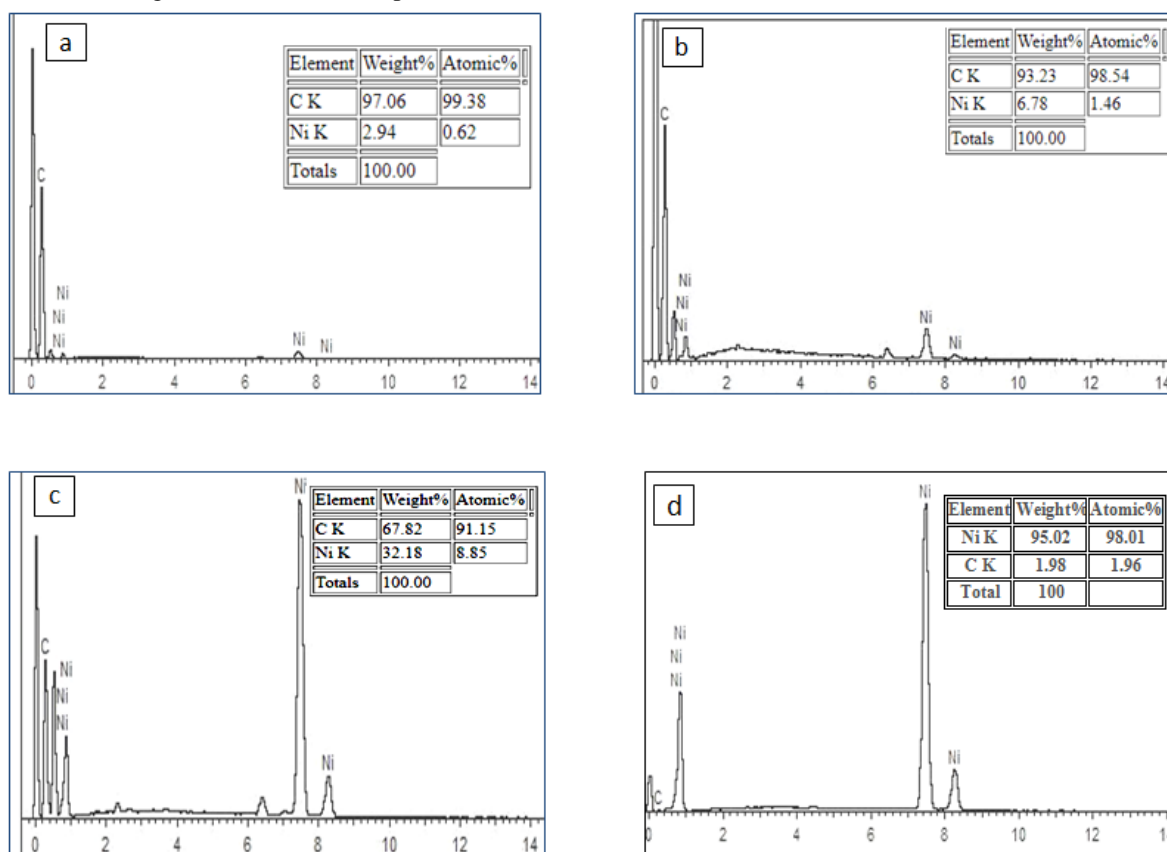


Fig. (2): EDS analysis of Ni-D composite coatings prepared by MSCD technique at different times. (a) 10 min., (b) 5 min., (c) 2 min and (d) CED in the presence of 10 g/l D.

Table 2: The relation between the deposition time and coating thickness of Ni-D composite coating using MSCD technique

Deposition time, min.	Coating thickness, μm
2	20
4	25
5	30
7	40
10	60

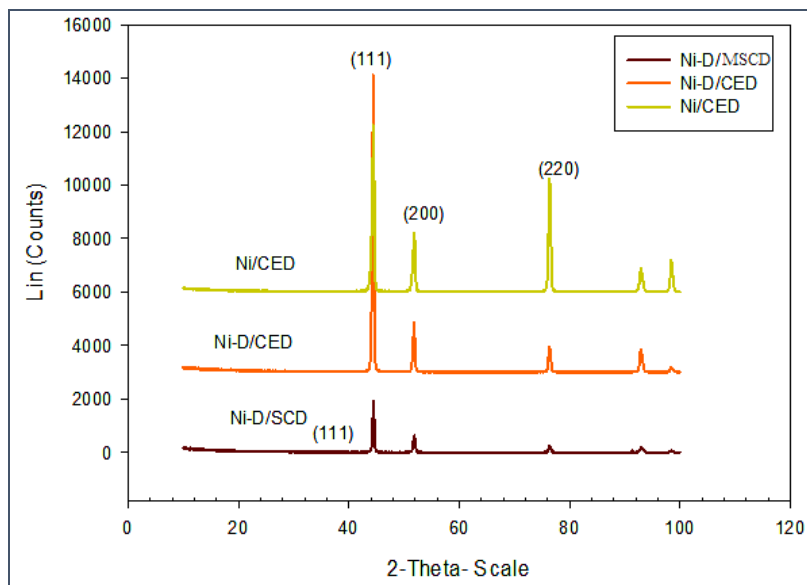


Fig. (3): XRD of Ni and Ni-D composite coatings prepared by CED and MSCD.

Fig. 4 shows the hardness profile of base metal, nickel and Ni-D composite coating prepared by CED and MSCD techniques. Without diamond additions, the electrodeposited Ni fabricated using current density of 0.5 Acm^{-2} exhibits hardness of $220 \pm 10 \text{ Hv}$. The composite coating, prepared by CED in the presence of 10 g/L diamond in the plating bath exhibits hardness of $400 \pm 10 \text{ Hv}$. Alternatively, Ni-D composite coating prepared by MSCD technique exhibits hardness of 1200 Hv in the presence of same contents of diamond in the plating bath (10 g/L). The hardness measured for base carbon steel is $\sim 180 \text{ Hv}$. The large increase in hardness produced by MSCD can be attributed to the large amount of diamond incorporated in the nickel matrix. It is difficult to co-deposit diamond particles into the formed matrix with the conventional electrodeposition method so that the diamond content in Ni-diamond composite coatings prepared is low. In the MSCD process, diamond particles are kept suspended in the electrolyte solution during the deposition. Therefore, diamond particles could be easily co-electrodeposited with metal ions on the substrate because of the gravity of diamond particles. This MSCD fabrication process can significantly improve the content of diamond particles in the deposits.

The surface roughness of Ni and Ni-D coatings was examined by surface profilometer. The surface profile (R_a) of Ni coating is $34 \mu\text{m}$, while the surface profile, R_a , of Ni-D coatings prepared by CED technique lies in the range $37\text{-}44 \mu\text{m}$. On the other

hand, Ni-diamond composite coat prepared by MSCD technique ranged from 160 to $220 \mu\text{m}$ with the diamond concentration in bath 10 g/L . Moreover, surface profile of Ni-diamond MSCD and covered Ni by electroless technique is $34 \mu\text{m}$.

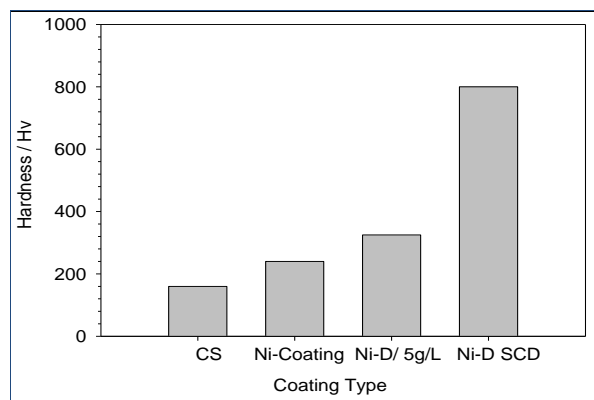


Figure 4: Micro hardness of uncoated carbon steel, Ni- and Ni-D composite coatings; current density 5 mA/cm^2 , 10 g/L diamond and bath temperature 60°C .

The performance of many products and engineering components depends critically on tribological properties of surfaces such as wear and friction. Here the wear testing was performed by using sand wear method for Ni and Ni-D composite coatings (prepared at current density of 0.5 A/cm^2). A tangential motion wear test in sand/water mixture

was used to assess the resistance to sliding and abrasive wear. The relation between the testing time and the sliding distance is illustrated by equation (1). The weight loss of the coated sample was determined and plotted against the sliding distance is shown in Fig. (5).

$$\text{Sliding distance} = \text{Time} \times \text{rpm} \times \text{Circumference}$$

(1)

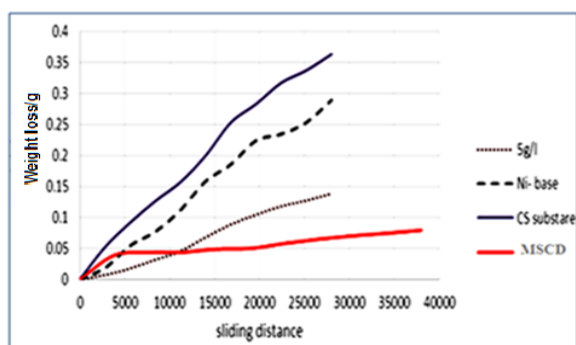


Fig. 5: Relation between sliding distance and weight loss of carbon steel, Ni coating and Ni-D composite coating prepared by CED (5 g/l D) and MSCD (10g/l D)

The wear resistance of the Ni, Ni-D (CED), and Ni-D (MSCD) coatings as a function of the diamond content is shown in Fig. 5. The wear resistance of electrodeposited Ni is slightly increased in comparison to the uncoated substrate. The wear resistance of Ni-D composite coating prepared by CED technique is markedly increased in comparison to either the uncoated substrate or the Ni-electrodeposited coating using CED technique. The wear resistance of Ni-D electrodeposits. Further improvement in wear resistance was achieved by Ni-D coating performed by MSCD technique. Results reveal that the more diamond particles in the Ni-D composite coatings the more increase in the wear resistance. During the MSCD process, particles are adsorbed onto the growing surface film in two successive steps and then are embedded within the electrodeposited metal matrix. The diamond particles are distributed uniformly in the Ni-matrix and embedded into the Ni-matrix by mechanical interlocking with Ni electroplating [16-19].

The corrosion resistance of the carbon steel, Ni, Ni-D (CED), and Ni-D (MSCD) coatings were investigated using electrochemical impedance spectroscopy (EIS) techniques. Nyquist plots are shown in Fig. 6. The capacity of double layer expressed as constant phase element (CPE) of depositions, polarization or charge transfer resistance (R_p) and solution resistance (R_s) were simulated using the equivalent electrical circuit

model (Fig. 8) and are given shown in Table 3. The Nyquist curves are single semi-circular plots whose diameters increase in the following manner:

$$CS < CS-Ni < CS-Ni-40D-CED < CS-Ni-MSCD < CS-Ni-20D-CED \approx CS-Ni-5D CED$$

It is well known that the corrosion resistance of metallic substrates increases with the increase in R_p and decrease in CPE. As predicted in Table 3, the R_p values shown by Ni-diamond composite coatings are quite high compared to Ni coating alone. Ni-5D, Ni-20D and Ni-40D show R_p values of $9.98 \times 10^4 \Omega$, $2.7 \times 10^4 \Omega$ and $1.6 \times 10^4 \Omega$, respectively in comparison to $3.5 \times 10^3 \Omega$ shown by Ni-only coating. On the other hand, the Ni-MSCD 10D shows almost R_p as the Ni-only coating. The same trend is shown by CPE values where the lowest values are shown by Ni-5D, Ni-20D and Ni-40D in comparison to Ni and Ni-MSCD coatings.

The improving corrosion resistance obtained after the incorporation of diamond nano particles may be due to accommodation of these particles to nano and micro porosities or may be attributed to decreasing surface conductivity and the effective metallic area prone to corrosion attack due to incorporation of nano-diamond [1, 11]. The decreased corrosion resistance at high diamond content may be related to the formation of porous coating that allows the penetration of corrosive media into the carbon steel substrate.

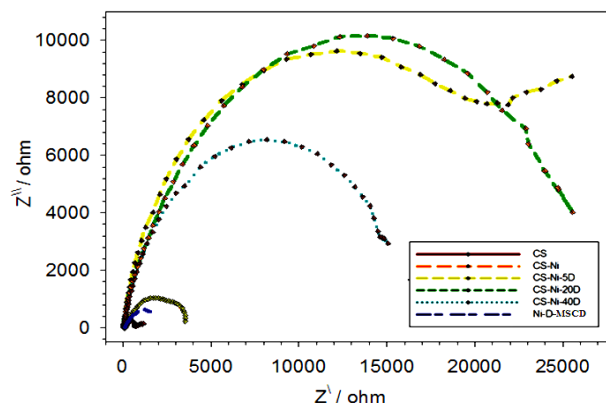


Fig 6: Nyquist curves of Ni-Diamond coatings prepared using CED and MSCD techniques compared with Ni blank coat and CS substrate

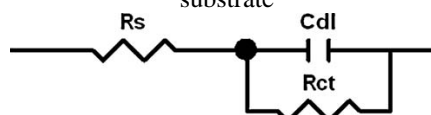


Fig. 7: Equivalent circuit model for the corrosion behavior of the Ni–diamond composite coatings in 3.5wt.% NaCl solution.

Table 3: EIS parameters of tested samples

	$R_s/\Omega.cm^2$	$R_p/\Omega.cm^2$	CPE/F	Normalizing factor (n)
CS	43	6.63×10^2	5.74×10^{-4}	0.85456
CS/Ni	141	3.52×10^3	1.00×10^{-3}	0.67684
CS/Ni/D 5g/l	48	9.98×10^4	3.39×10^{-5}	0.80469
CS/Ni/D 20g/l	37	2.73×10^4	3.51×10^{-5}	0.80943
CS/Ni/D 40g/l	52	1.63×10^4	6.07×10^{-4}	0.86244
CS/Ni/D MSCD	144	3.11×10^3	9.15×10^{-4}	0.69802

4. Conclusions

MSCD was performed to prepare hard Ni-diamond composite coatings. Ni-diamond composite coating prepared by the MSCD method contains quite high content of diamond (67-97%) as evidenced by EDS analysis. The prepared coatings exhibited quite high hardness and superior wear resistance. The hardness of the composite coatings was increased by increasing the concentration of diamond particles because the tendency of diamond particles to co-deposit into Ni-matrix becomes more easily at high concentration. According to the results of the present work, the following remarks can be outlined:

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