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Improvement of Microhardness and Corrosion Resistance of Stainless Steel by Nanocomposite Coating

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Abstract

Stainless steel (AISI 304) has good electrical and thermal conductivities, good corrosion resistance at ambient temperature, apart from these it is cheap and abundantly available; but has good mechanical properties such as hardness. To improve the hardness and corrosion resistance of stainless steel its surface can be modified by developing nanocomposite coatings applied on its surface. The main objective of this paper is to study effect of electroco-deposition method on microhardness and corrosion resistance of stainless steel, and to analyze effect of nanoparticles (Al_2O_3, ZrO_2 , and SiC) on properties of composite coatings. In this paper employed Electroco-deposition process to develop a composite coating with (Ni) matrix and Ceramic oxide particles: Al_2O_3 (135nm), ZrO_2 (40nm), and SiC (80nm) as reinforcements. The coatings were developed with 10 g/L, and 20 g/L concentrations in bath, at four different current densities (0.5, 1, 2, 3 A/dm^2) using Watts bath to study the effect of current density and particle concentration in bath, on structure and properties of the coatings developed. The surface morphology of nanocomposite coating was characterized by Scanning Electron Microscopy (SEM). The hardness of the nanocoating was carried out using Digital Vickers microhardness tester. The corrosion resistance property of nanocomposite coating was carried out in 3.5% NaCl solution used Open circuit potential (OCP) and potentialastic polarization. The results showed the nanocomposites coating have a smooth and compact surface and have higher hardness than the uncoated stainless steel (2.3 times), and also found that the nanocomposite coating improves the corrosion resistance significantly (89.25%).

Keywords: *Stainless Steel, Nanocomposite Coating, Electroco-Deposition ECD, Microhardness, Corrosion Resistance, and Potentialastic Polarization.*

1. Introduction

Stainless steel is environment friendly and abundantly available material that have good corrosion resistance, retains strength even at high temperatures, and easily machined, welded, formed and fabricated [1]. In order to enhance the mechanical properties bulk modification/alloying have been tried but limitations in alloying and adversely effects in its another properties has been reported. Another recent way to improve its mechanical properties is with surface modification by developing composite coating on its surface. The surface coating technique available in this work that Electroco-deposition (ECD) it has several advantages in developing metal matrix composite coatings among other coating processes such as, uniform depositions on complexly shaped substrates, low cost, good reproducibility and the reduction of waste [2]. ECD process has been in use successfully to develop such nanocomposite coatings from the past decades. The second phase can be hard oxide (Al_2O_3,TiO_2, SiO_2) or carbides particles (SiC, WC), etc., embedded in metals like Cu, Ni, Cr, Co and various alloys [3].

According to Guglielmi's model, composite electroplating takes place in two steps. During electrodeposition, solid particles are surrounded with cloud of adsorbed ions and these particles are weakly adsorbed at cathode surface by Vander Walls forces when they approach the cathode in the first step. And in the second step, loosely adsorbed particles get adsorbed strongly on cathode surface by Coulomb force and consequently entrapped within metal matrix. The main drawback of this model is absence of mass transfer effect during ECD process. [4].

One of the common mechanism of co-deposition process consist of five consecutive steps [5] shown in Figure 1, five consecutive steps of co-deposition mechanism are:

1. Formation of ionic clouds on the particles.
2. Convection towards the cathode.
3. Diffusion through hydrodynamic boundary layer.
4. Diffusion through concentration boundary layer.

Adsorption at the cathode where particles are entrapped within metal deposit.

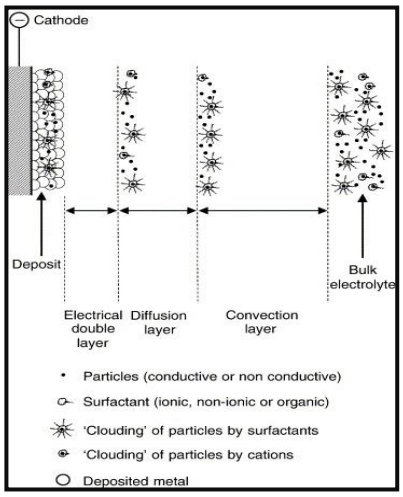

Fig. 1. Mechanism of co-deposition process.

Hashimoto and Abe [6], characterized Zn-SiO_2 composites before and after corrosion test. Zn-SiO_2 composites exhibited better corrosion resistance due to formation of protective corrosion products supported by SiO_2.

Akarapu [7], employed ECD process to develop a composite coating with Cu matrix and Ceramic oxide particles TiO_2 (particle size ~202 nm), Al_2O_3 (particle size ~287 nm) as reinforcements. The coatings were developed with 10 g/l, 30 g/l and 0 g/l (unreinforced) concentrations in bath, at four different current densities (5, 8, 11, 14 A/dm2) with using copper sulfate bath in order to study the effect of Current density and particle concentration in bath, on structure and properties of the coatings developed. The crystallite size was averagely 50-65 nm and a strong (220) texture was obtained in composite coatings and uncoated Cu coatings determined from the XRD data. The composition and surface morphology of coatings were studied by using EDS and SEM. Hardness and Wear resistance of the coatings were determined by using microhardness tester and ball on plate wear tester, improved hardness and wear resistance of composite coatings were observed compared to the unreinforced copper coatings.

Borkar [8], in this work, Nickel composite coatings (Ni-Al_2O_3, Ni-SiC, and Ni-ZrO_2) were successfully synthesized by DC, PC, and PRC techniques to study effect of ECD methods on microstructure, mechanical, and tribological behavior. Ni-CNT composite coatings were also fabricated by pulse ECD method to investigate CNT reinforcement effect on mechanical and tribological property. Ni-Al_2O_3 composites coatings were deposited to analyze effect of nanoparticles on properties of composite coatings.

Bahrololoom and Sani [9] ,at first, Particles reinforcement increases sharply at the beginning with increase in current density till it reaches maximum value followed by sharp decrease. Therefore, hardness of composite coatings mainly increases due to the combined effect of both grain refining as well as of dispersive strengthening.

Saha and Khan [10] ,when electroplating at lower current densities, nickel ions dissolved from anode (i.e. nickel) are transported at low rate and hence there is insufficient time for these ions to absorb on particles resulting in weak Coulomb force between anions adsorbed on particles leading to lower concentration of electrodeposited particles in the composite coatings. On the other hand, at higher current densities, nickel ions dissolved from anode are transported faster than particles by the mechanical agitation which causes a decrease in codeposition of particles as well as hardness of composite coatings. Therefore, selection of optimum current density is important to enhance the concentration of particles in the composite coatings.

2. Experimental Procedure

The schematic diagram of electroco- deposition shown in Figure 2. The nickel composite coatings

prepared by electrodeposition from Watts solution suspended with nanoparticles. The nanoparticles used as reinforcement have (Al_2O_3=135nm, ZrO_2 = 40 nm, SiC=80 nm) particle sizes. Before electrodeposition electrolyte was stirred for about 24 hours using magnetic stirrer (model VS-130SH). All the electrodeposition experiments were carried out at room temperature. A stainless steel plate (with an area of 4 cm^2) and (99.99%) pure nickel plate (with an area of 10 cm^2) were used as cathode and anode respectively, the steps of preparation stainless steel plate may be summarized as follow:

1. Cutting the selected stainless steel (substrate) to the desired dimensions (20mm×20mm×0.5mm).
2. Cleaning the stainless steel (substrate) by using acetone. This step was necessary to be sure to remove any surface oxide and organic impurities.
3. Masking the substrates were leaving free only the surface to be coated.
4. Dipping the masked substrate in distilled water in order to remove the small amount of oxides which might be formed during the exposure to the atmosphere while masking.

Since the substrates were prepared for deposition. After the deposition the tape used as a mask was removed and the samples were rinsed in distilled water and dried. These procedure were necessary to ensure the removal of any residuals of the watts bath, especially any loose adsorbed nanoparticles from the surface. Standard Watts solution consists of $NiSO_4.6H_2O$ (Nickel sulphate hexahydrate), $NiCl_2.6H_2O$ (Nickel chloride hexahydrate), and $H_3.BO_3$ (Boric acid). Table 1 shows content of these chemicals for making of 1 L of electroplating bath. Deposition parameters of Ni-Al_2O_3/Ni-ZrO_2/Ni-SiC and uncoated Nickel coatings are reported in Table 2.

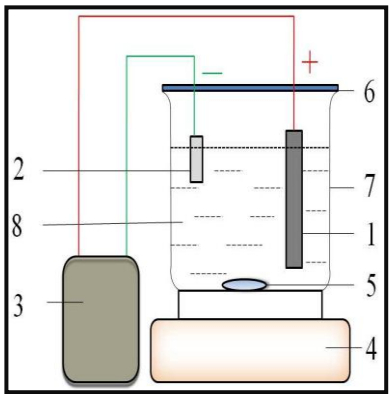

Fig. 2. The schematic diagram of electroco-deposition.

Table 1,
Overview of the composition of chemicals for Watts bath.

Bath composition	
$NiSO_4.6H_2O$	265g/L
$NiCl_2.6H_2O$	48g/L
$H_3.BO_3$	31g/L

Table 2,
Determination of deposition parameters.

Current density	0.5, 1, 2, 3 (A/dm^2)
Dispersion	Al_2O_3/ZrO_2/SiC: 10, 20 (g/L)

The surface morphology of the coatings and distribution of the particles was examined by Scanning Electron Microscopic (SEM) (Tescan Vega 3). Assessments of microhardness of the coated and the uncoated stainless steel were determined by using Digital Vickers microhardness ester (TH-715) with 9.807N load for 10 seconds. The hardness values were taken at 3 different points on the surfaces and average of these values were considered in the results. Open circuit potential (OCP) and potentialastic polarization were used as the techniques for evaluating corrosion parameters of uncoated stainless steel and the composite coatings, the localized corrosion of the specimens were studied in 3.5% NaCl solution.

3. Results and Discussion

1. Scanning Electron Microscope (SEM) Studies

Figures (3-8) shows SEM surface micrographs of the electrodeposited (Al_2O_3, ZrO_2 , and SiC) composite coatings prepared at 10 g/l Al_2O_3 in the bath and current densities 0.5, 1, 2, and 3 A/dm^2.

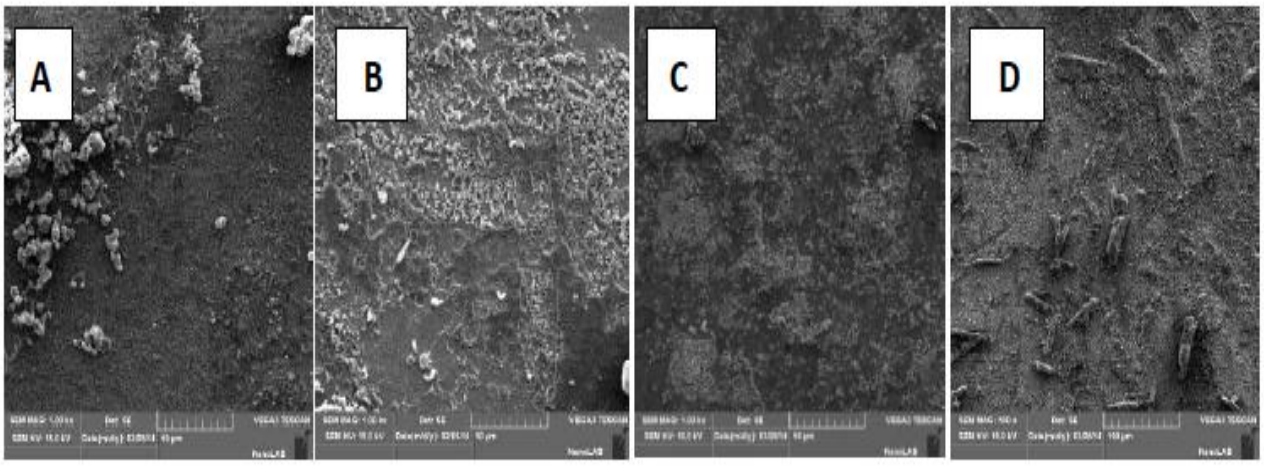

Fig. 3. Surface morphology of electrodeposited Ni-Al_2O_3 coatings at 10 g/L (A) 0.5 A/dm^2 (B) 1 A/dm^2 (C) 2 A/dm^2 (D) 3 A/dm^2.

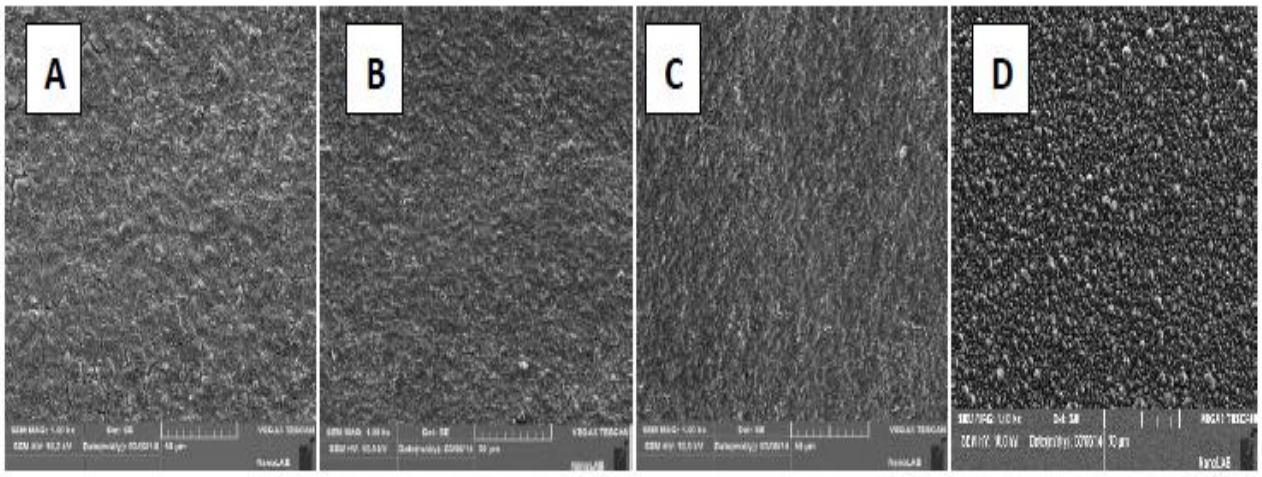

Fig. 4. Surface morphology of electrodeposited Ni-Al_2O_3 coatings at 20 g/L (A) 0.5 A/dm^2 (B) 1 A/dm^2 (C) 2 A/dm^2 (D) 3 A/dm^2.

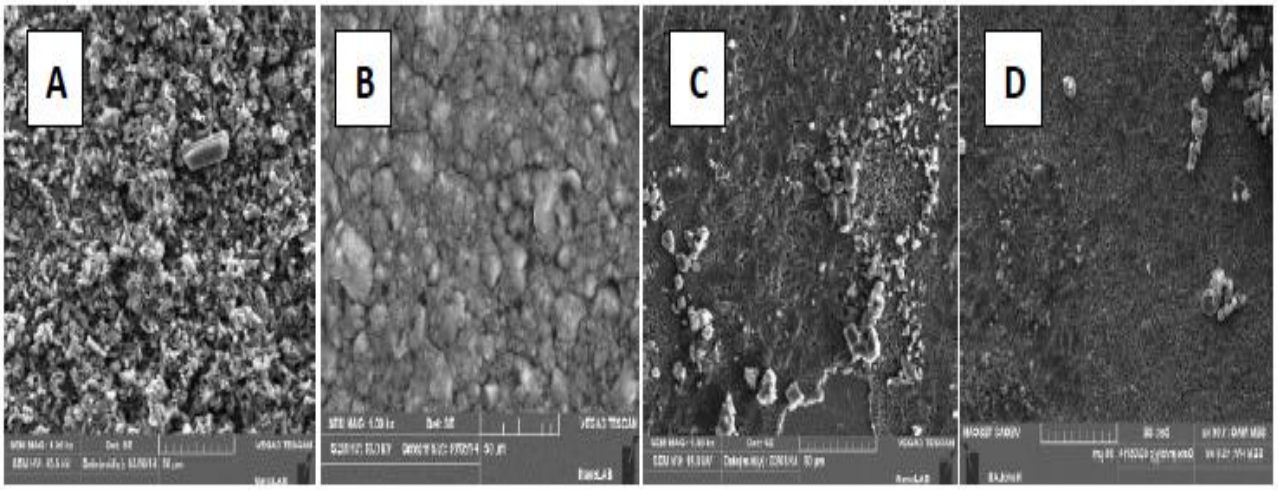

Fig. 5. Surface morphology of electrodeposited Ni-ZrO_2 coatings at 10 g/L (A) 0.5 A/dm^2 (B) 1 A/dm^2 (C) 2 A/dm^2 (D) 3 A/dm^2.

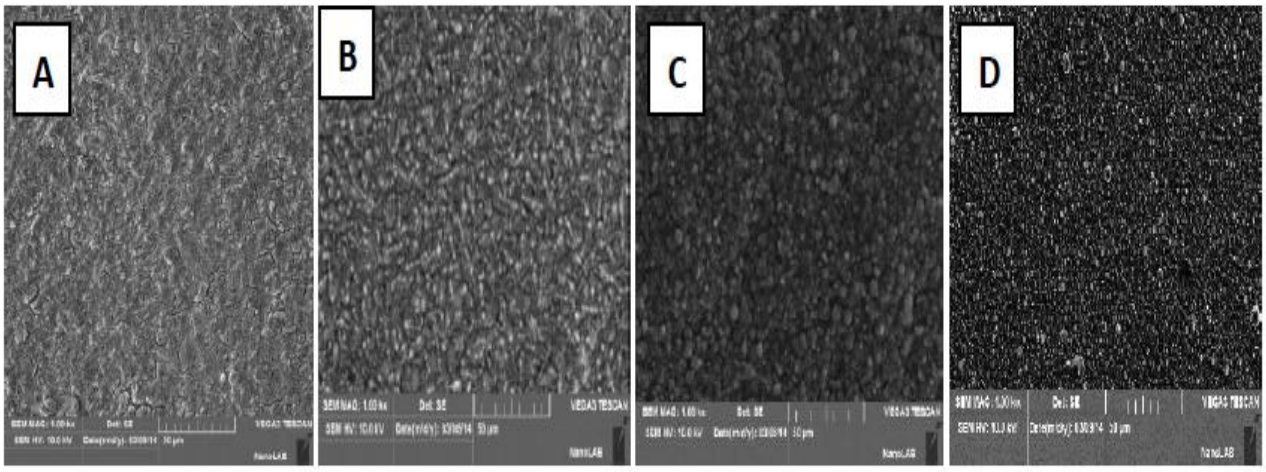

Fig.6. Surface morphology of electrodeposited Ni-ZrO_2 coatings at 20 g/L (A) 0.5 A/dm^2 (B) 1 A/dm^2 (C) 2 A/dm^2 (D) 3 A/dm^2.

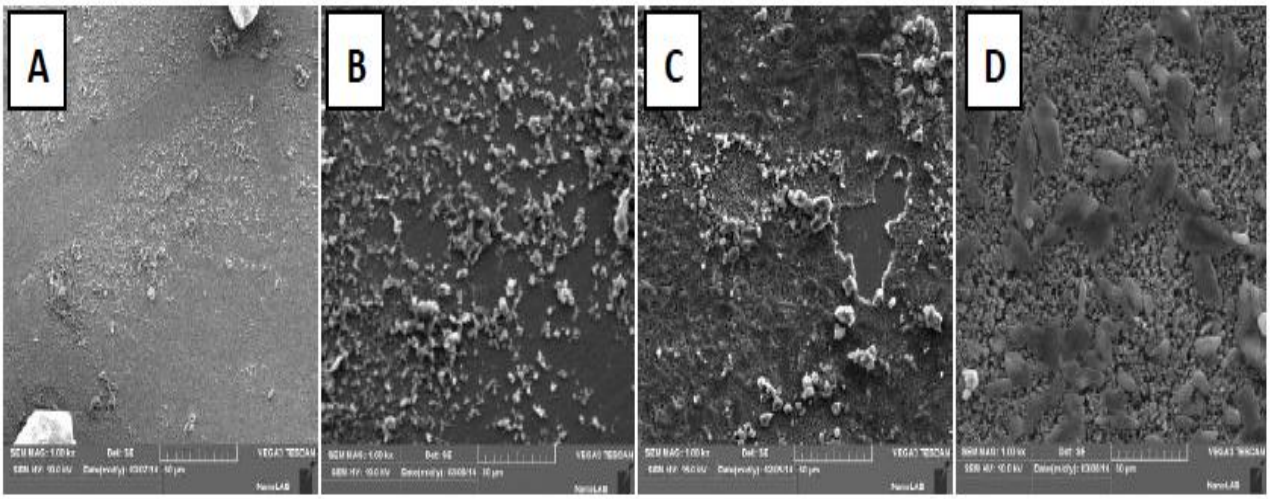

Fig. 7. Surface morphology of electrodeposited Ni-*SiC* coatings at 10 g/L (A) 0.5 A/dm^2 (B) 1 A/dm^2 (C) 2 A/dm^2 (D) 3 A/dm^2.

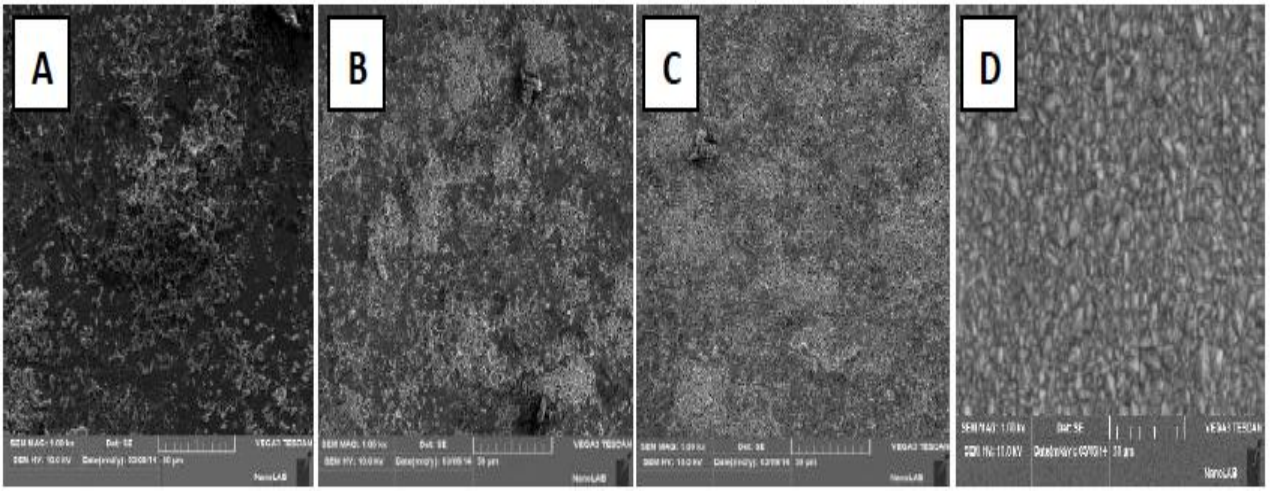

Fig. 8. Surface morphology of electrodeposited Ni-*SiC* coatings at 20 g/L (A) 0.5 A/dm^2 (B) 1 A/dm^2 (C) 2 A/dm^2 (D) 3 A/dm^2.

2. Microhardness Study

The microhardness of the composite coatings were measured by using Digital microhardness tester by applying 9.807N load for 10 seconds in order to ensure that the microhardness values are not affected by the substrate. The effect of current density on microhardness of Ni-Al_2O_3, Ni-ZrO_2 and Ni-SiC composite coatings developed at current densities 0.5, 1, 2, 3 A/dm^2 shown in Figures (9-12). The hardness values obtained for the composite coatings (Ni-Al_2O_3, Ni-ZrO_2 and Ni-SiC) are higher than the hardness values of substrate (pure stainless) 187.6 HV. In all the cases (Ni-Al_2O_3, Ni-ZrO_2 and Ni-SiC) coatings the microhardness values obtained followed the same trend. When the current density increased from 0.5 to 2 A/dm2, the hardness values increased and at 3 A/dm2 a little decrease in hardness values were obtained. In the present study at 2 A/dm2 current density higher hardness values shown in Figure 12.

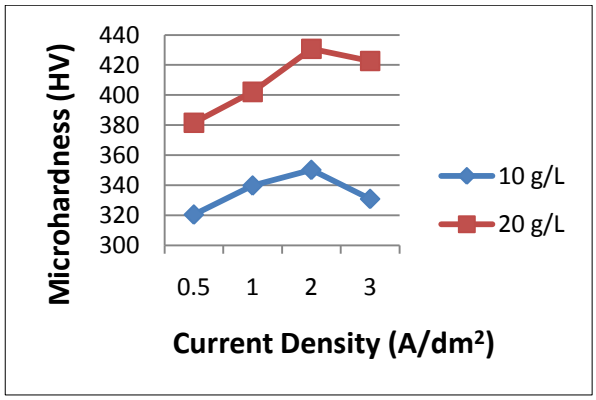

Fig. 9. Effect of current density on microhardness of Ni-Al_2O_3 coating at current densities 0.5,1,2, and 3 A/dm^2.

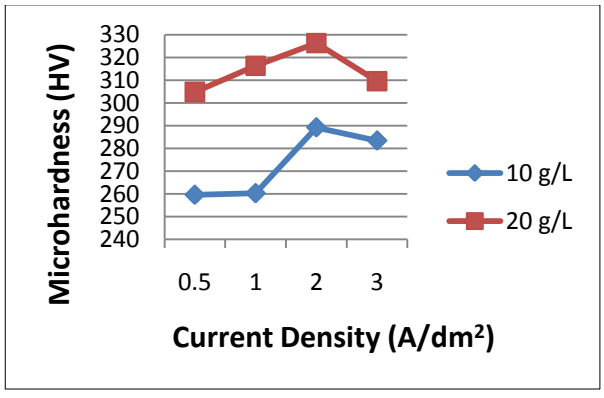

Fig. 10. Effect of current density on microhardness of Ni-ZrO_2 coating at current densities 0.5,1,2, and 3 A/dm^2.

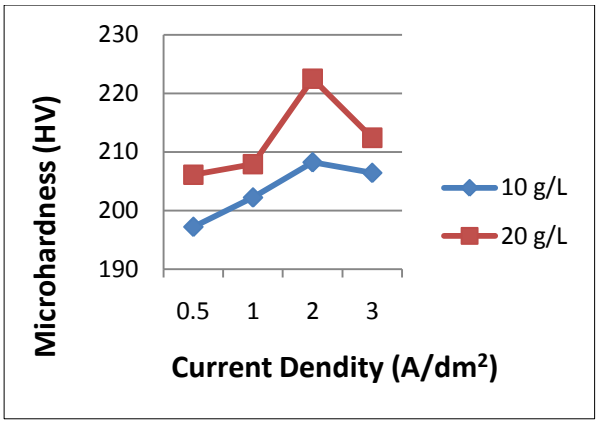

Fig. 11. Effect of current density on microhardness of Ni-SiC coating at current densities 0.5,1,2, and 3 A/dm^2.

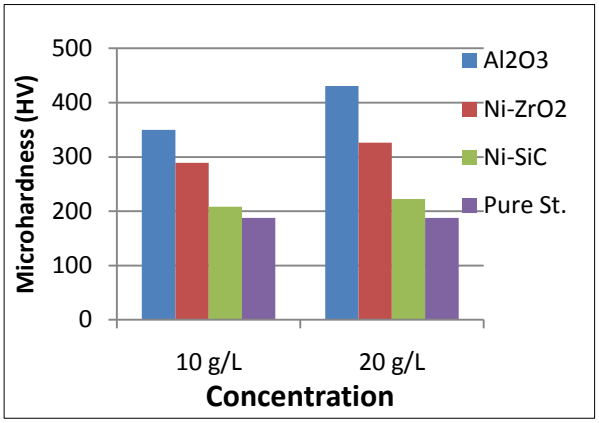

Fig. 12. Microhardness of uncoated stainless steel and nickel composite coatings depostied at 10 and 20 (g/L) at 2 A/dm^2.

3. Corrosion Study

The corrosion bahavior of the composite coatings at defferent conditions were studied in Sodium chloride at room tempature using open – circuit potential and potentiostatic polarization measurements.

- Open Circuit Potential (OCP)- Time Measurements.

The values of the open circuit potential (OCP) measured with respect to SCE for 15 min in 3.5% NaCl at room temperature showed the corrosion behavior of the uncoated and coated sample under equilibrated conditions in the solution. Figure 13 illustrates the OCP – time curve of uncoated stainless steel. The potential is generally changed from initial negative value of -380mV vs (SCE) to the positive direction of -223mV vs. (SCE) and the potential almost remains stable at this value for

more than 15 minutes. The increase in potential in the positive direction in this case may be due to the formation of the stable passive film.

- Potentiostatic Polarization Measurements

Polarization curve is commonly used as a plot of the electrode potential versus the logarithm of current density. The potentiostatic polarization for uncoated stainless steel and composite coatings specimens are presented in Figures (14-17) which show cathodic and anodic polarization curves of uncoated stainless steel and composite coatings specimens in 3.5% NaCl solution.

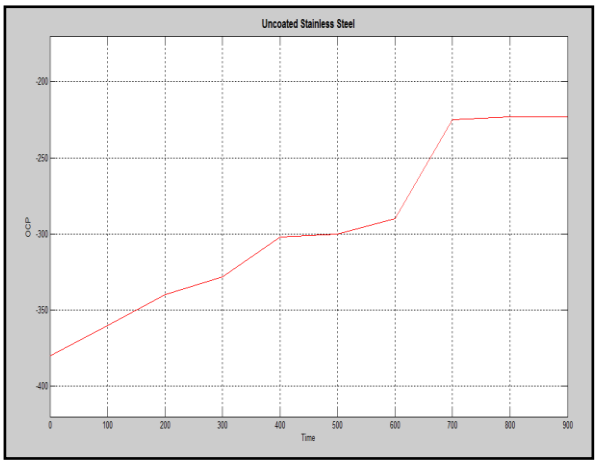

Fig. 13. The OCP – time curve of uncoated stainless steel.

Figure 14 indicates such curve, for uncoated stainless steel; which shows that corrosion potential (E_{cor}) and corrosion current density (I_{cor}) values are (-214.7 mV) and (6.12 µA/cm^2) respectively.

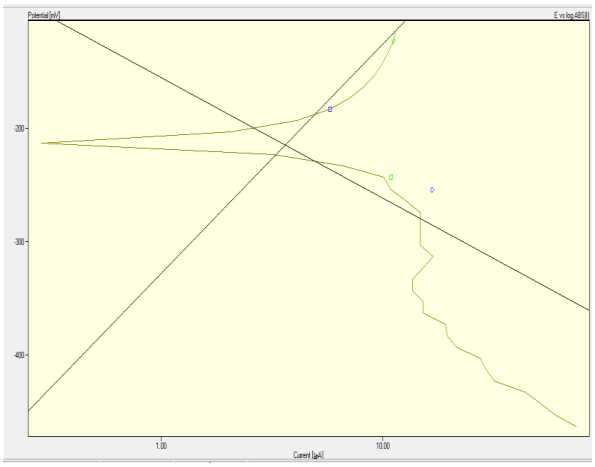

Fig. 14. The potentiostatic polarization for uncoated stainless steel.

Figure (15 a) illustrates the case of Ni-SiC coatings at 20 g/L at 0.5, 1, 2 , and 3 A/dm^2, which show that corrosion potential (E_{cor}) and corrosion current density (I_{cor}) values are (-168.3 mV, -162.1 mV, -159.8 mV, and -154.6 mV) and (4.74 µA/cm^2 , 4.53 µA/cm^2, 4.40 µA/cm^2, and 4.39 µA/cm^2) respectively. The results show the obvious protection to the metal due to the Ni-SiC layer that covers the metal surface. Figure (15 b) illustrates the case of Ni-SiC coatings at 10 g/L at 0.5, 1, 2 , and 3 A/dm^2, which show that corrosion potential (E_{cor}) and corrosion current density (I_{cor}) values are (-195.4 mV, -189.2 mV, -183.5 mV, and -177.9 mV) and (5.92 µA/cm^2 , 5.84 µA/cm^2, 5.66 µA/cm^2, and 4.87 µA/cm^2) respectively. The results show the obvious protection to the metal due to the Ni-SiC layer that covers the metal surface. The results show surface protection to the metal but, the protection is less than the protection provided by Ni-SiC coatings at 20 g/L. The magnitude of E_{cor} is not a parameter that allows characterization of the corrosion phenomenon in a given system; its magnitude is determined by several factors, such as the nature of the metal, the environment or the electronic reactions that take place.

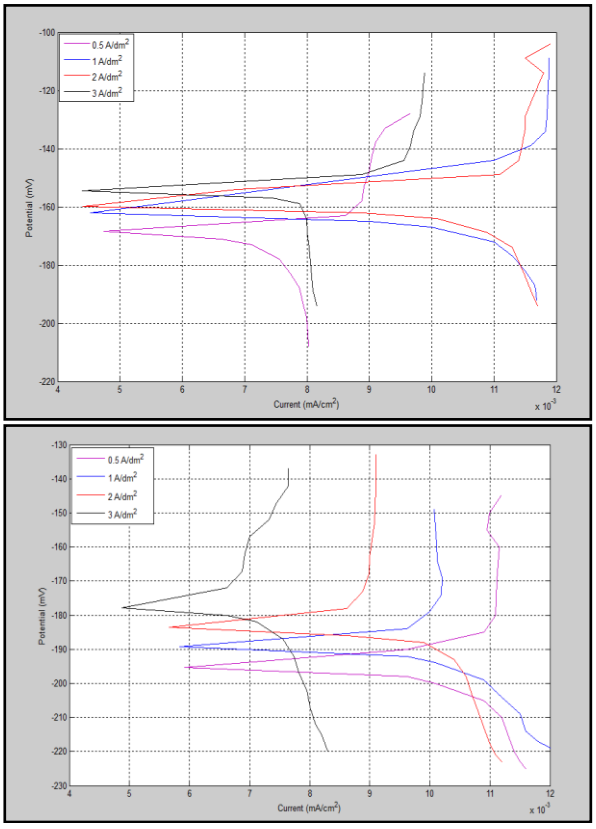

Fig. 15. Potentiostatic polarization behaviour of Ni-SiC coatings at 0.5, 1, 2 , and 3 A/dm^2 a)10 g/L, and b) 20 g/L.

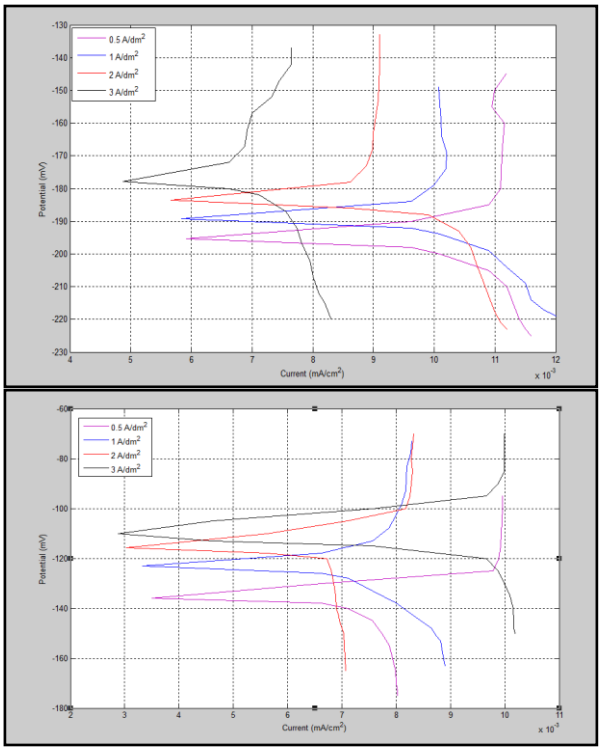

Fig. 16. Potentiostatic polarization behaviour of Ni-ZrO_2 coatings at 0.5, 1, 2 , and 3 A/dm^2 a)10 g/L, and b) 20 g/L.

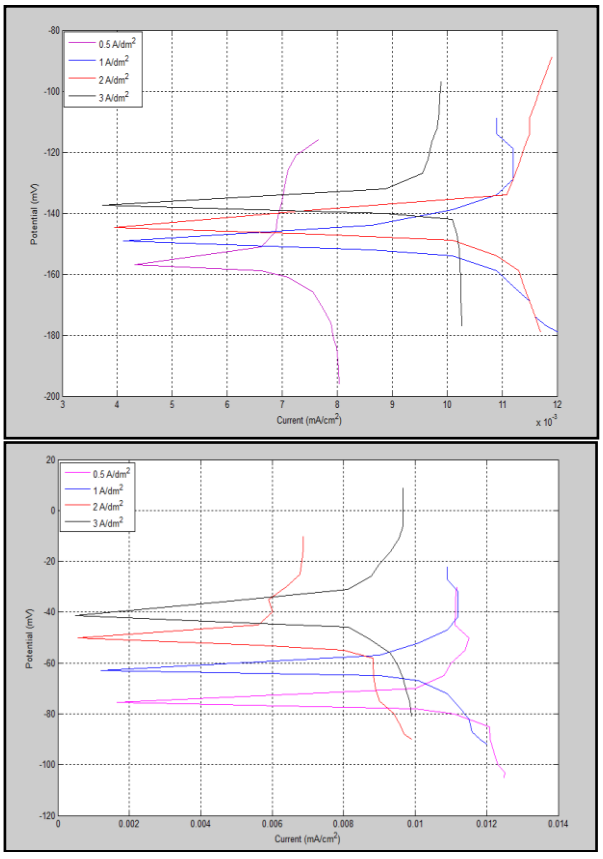

Fig. 17. Potentiostatic polarization behavior of Ni-Al_2O_3 coatings at 0.5, 1, 2 , and 3 A/dm2 a)10 g/L, and b) 20 g/L.

In all the cases (Ni-Al_2O_3, Ni-ZrO_2 and Ni-SiC) coatings the polarization curves obtained have the same behavior at increased concentration of (Al_2O_3, ZrO_2 and SiC) in the nanocoating. The corrosion current decreases and the corrosion potential shifts to a more positive potential resulting in a decreased corrosion rate. This results show the concentration 10 g/L at (Al_2O_3, ZrO_2 and SiC) has the largest corrosion current because of the void space on the surface leading to entering solutions to the metal, causing dissolutions faster than the surface with concentration 20 g/L. From the above results the examination of uncoated and coated stainless steel in 3.5% NaCl solution indicates that excellent corrosion resistance is observed for Ni-Al_2O_3 coatings at 20 g/L and 3 A/dm2. The best value of corrosion rate for uncoated and coated stainless steel are shown in Table 3 and Figure 18.

The efficiency in improvement of current density and corrosion rate (mpy) are due to composite coatings. They can be obtained by using the following relations (1) and (2) :

$$\text{Efficiency in Current Density } (I_{cor}) = \frac{(I_{cor})_{uncoated} - (I_{cor})_{coated}}{(I_{cor})_{uncoated}} \times 100\% \quad \ldots (1)$$

$$\text{Efficiency in Corrosion Rate } (C.R) = \frac{(C.R)_{uncoated} - (C.R)_{coated}}{(C.R)_{uncoated}} \times 100\% \quad \ldots (2)$$

Table 3,
The best corrosion parameters of specimens in 3.5% NaCl.

Type	Ex. No	E_{cor} (mV)	I_{cor} ($\mu A/cm^2$)	I_{cor} %	mpy	C.R %
Ni-Al_2O_3	4	-83.2	1.95	68.13	1.81* 10-1	80.15
	8	-41.2	0.49429	91.92	0.98* 10-1	89.25
Ni-ZrO_2	12	-137.4	3.73	39.05	3.43* 10-1	62.39
	16	-110.0	2.88	52.94	2.7* 10-1	70.39
Ni-SiC	20	-177.9	4.87	20.42	5.23* 10-1	43.65
	24	-154.6	4.39	28.26	4.56* 10-1	50

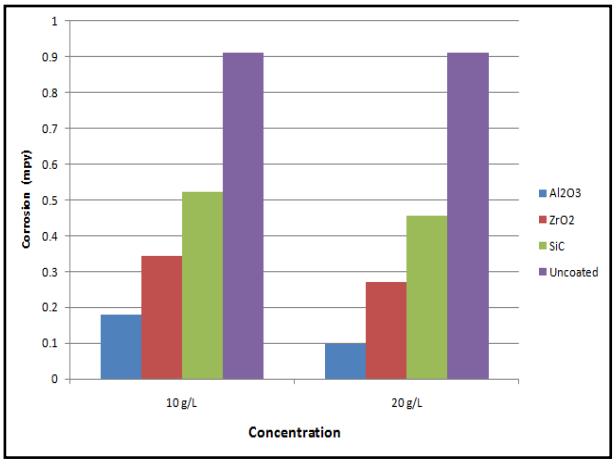

Fig. 18. The best corrosion parameters of specimens in 3.5% NaCl.

4. Conclusions

In the present study, Ni-Al2O3, Ni-ZrO2, and Ni-SiC nanocomposite coatings were developed successfully by using Electroco-deposition process on the Stainless steel (AISI 304) from Watts bath with different current densities and powders concentrations. From the detailed investigation of the results obtained, the following conclusions can be drawn:

1. The microhardness values obtained for Ni-Al_2O_3, Ni-ZrO_2, and Ni-SiC composite coatings are higher than the uncoated stainless steel hardness (HV).
2. The maximum of microhardness at (2 A/dm^2):

- For Al_2O_3, maximum for 10 g/L was 1.87 and 2.30 times increase for 20 g/L.
- For ZrO2 , maximum for 10 g/L was 1.54 and 1.74 times increase for 20 g/L.
- For SiC, maximum for 10 g/L was 1.11 and 1.19 times increase for 20 g/L.

3. The microhardness of the Ni-Al_2O_3, Ni-ZrO_2, and Ni-SiC composite coatings increased with increasing the content of nanoparticle loading in the electrolyte bath due to enhanced dispersion strengthening effects.
4. The corrosion resistance of the composite coatings was higher than the uncoated stainless steel.
5. The optimum corrosion rate achieved at (20 g/L and 3 A/dm^2):

- For Al_2O_3 was 89.25%.
- For ZrO2 was 70.39%.
- For SiC was 50%.

5. References

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تحسين الصلادة ومقاومة التآكل للفولاذ المقاوم للصدأ بواسطة الطلاء بمركب نانوي

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الخلاصة

يُعد الفولاذ المقاوم للصدأ (AISI٣٠٤) من المواد الموصلة للحرارة والكهربائية ومقاوم جيد للتآكل في درجات الحرارة الأعتي ادية، منخفض الثمن ومتوافر بكثرة، لكنه يُعد جيداً من ناحية الخواص الميكانيكية مثل الصلادة ومقاومة الشد. لغرض تحسين خواص السطح مثل الصلادة ومقاومة التآكل مع تعديل السطح باستخدام الطلاء بالمركبات النانوي .في هذا البحث استخدمنا عملية (Electroco-deposition) لوضع الطلاء المركب مع مصفوفة النيكل وجزيئات اوكسيد السيراميك كمعززات على النحو التالي:- (Al_2O_3 (135nm), ZrO_2 (40nm), and SiC (80nm). تم تنفيذ الطلاء في تركيزات مختلفة (10g/L و 20g/L)، وكذلك كثافة التيار مختلفة ($0.5, 1, 2,$ and $3\ A/dm^2$) باستخدام حمام (Watts) من اجل دراسة تأثير كثافة التيار وتركيز الجزيئات في الحمام على بنية الطلاء المنجز وخصائصه. الهدف الاساس من هذا البحث هو تحسين الصلادة ومقاومة التآكل للفولاذ المقاوم للصدأ ، ودراسة تأثير طريقة (Electroco-deposition) على السلوك المجهري والميكانيكي للفولاذ المس تخدم في البحث ، وكذلك تحليل تأثير الجزيئات النانوية على خصائص الطلاء المركب. وتم كذلك دراسة تشكيل سطح الطلاء بالمركب النانوي من خلال المجهر الالكتروني (SEM). وقد اجري فحص الصلادة بواسطة الاختبار الرقمي للصلادة الدقيقة (Microhardness-HV)، كذلك تم اختبار التآ كل في محلول كلوريد الصوديوم (NaCl) بتركيز 3.5% باستخدام طاقة الدائرة المفتوحة (OCP) وطاقة الاستقطاب. أظهرت النتائج ان الطلاء بالمركبات النانوية له سطح أملس مدمج ، وكذلك صلادة عالية بنسبة (2.3 مرة) مقارنة بالفولاذ المقاوم للصدأ غير المطلي، وايضآ وجد ان مقاومة التآكل قد تحسنت بشكل كبير بنسبة 89.25% مقارنة بالفولاذ غير المطلي.