Assessment on the microhardness and corrosion resistance characteristics of Ni-SiC and Ni-MWCNT coatings by pulse reverse electrodeposition technique

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Abstract

The characteristics of Silicon carbide (SiC) and multi-walled carbon nanotube (MWCNT) nano composite coatings by a Pulse Reverse Electrodeposition (PRE) method is investigated in detail to enhance the microhardness (MH) and corrosion resistance characteristics of AISI 304 stainless steel substrate. The electrodeposition nature, dissolution behavior, surface characteristics are assessed by Scanning Electron Microscopy (SEM) with energy dispersive x-ray (EDX), x-Ray Diffraction (XRD), Atomic Force Microscopy (AFM), Microhardness (MH) Test and Electrochemical studies. The coatings are prepared in the watts type bath using pulse reverse electrodepositions (PRE) method of varying the electrolyte deposition parameters in different combinations. Present results clearly reveal that, there is a drastic improvement in the magnitude of microhardness of coated specimens, silicon carbide (SiC) and multi-walled carbon nanotube (MWCNT) composite coatings yield a maximum hike of 91.6% and 168% respectively. Furthermore, the Nyquist and impedance plots clearly depict that, multi-walled carbon nanotube (MWCNT) exhibits higher corrosion resistance characteristics.

1. Introduction

The performances of engineering materials are highly dependent on its surface characteristics such as friction, wear, corrosion and fatigue. In the current industrial scenario, there is a need in developing promising coating combinations and techniques to enhance these surface characteristics.

Electrodeposition process is a technologically feasible and economically superior technique to provide coatings by using different metals to their metal matrix composites due to its low cost and easy control of deposition parameters [1–4]. In electrodeposition processes, the direct current (DC) method is considered as the easiest one to deposit pure metals, alloys and their composites. However, in the recent years, many researchers have focused mainly on electrodeposition coatings prepared by Pulse current and Pulse reverse current methods [5]. This method selects due to the better deposition rate and improvement in microstructural, mechanical and corrosion properties when compared with the classical DC method [6].

Producing Nano crystalline materials by the pulse reverse current (PRC) received considerable attention owing to the flexibility of improving their properties by regulation of pulse parameters [4, 7–10]. Nanoparticles are incorporated into metal and metal alloy deposits to enhance their physical and mechanical properties [11]. Recently, nanomaterial’s like carbon nanotubes (CNTs) have gained significant focus as the incorporating filler to endow the composite coatings with functional properties such as smoothness [12], better hardness [12–18], lower coefficient of friction [13], better wear and resistance [12, 15], corrosion resistance [14, 19], higher compressive strength [16] and good flexibility [20, 21].

Carbon nanotubes (CNTs) [22, 23] possess excellent mechanical characteristics, such as high tensile strength, elastic modulus, thermal and electrical conductivity. The study into the practical applications of CNTs
has been increasing, and metal composites of such Nano sized filler materials provide promising new compositor offering innovative functions.

Nickel is a desirable corrosion resistance, good mechanical properties and high catalytic performance. Ni based composites produced by an electrochemical process are utilisable for this surface material purpose [24]. In recent years interest in Ni based composite coatings electro co-deposition has increased to their unique combination of magnetic, wear and corrosion properties [25]. Nickel based electrodeposited composite coatings can be economically in major markets in construction, automotive, power generation, mining and aerospace fields, would befit from development [26, 27]. Ni is a metal with high hardness, good ductility, and ferromagnetism. It is added to various kinds of metals as a typical alloy element. Therefore, Ni-SiC and Ni-MWCNT composite powder is expected to be a promising coating material for various Ni alloys with SiC and MWCNT composite materials. In many industrial applications, Ni/SiC composite coatings are widely used and significant attention has been given to study the parameter like composite, PH, Temperature, current density and type of currents. Ni-SiC composite coatings resulted in the better corrosion resistance nature with increase in SiC coated [28–30].

The potential for carbon nanotube (CNT) reinforced metal composites having extraordinary specific stiffness and strength represents a tremendous opportunity for the development of new material systems [31, 32] and excellent thermal conductivity, electrical conductivity and mechanical properties [33]. The CNTs have been revealed as effective fillers for reinforcing Ni electrodeposits to improve the hardness, wear resistance and corrosion resistance [34–36]. Further, It is found that the deposited Ni/CNTs exhibited superior mechanical properties and superb additive components [37] due to the incorporation of the uniformly dispersed CNTs in it. Interest in electro co-deposition of nickel based nanocomposite coatings have increased in recent years due to their unique combination of wear, magnetic, electrical, and corrosion properties [25]. Incorporation of SiC and CNTs into a nickel matrix [38] significantly increases in mechanical properties and corrosion resistance character which acts as physical barriers to the corrosion process by filling in crevices, gaps, and micron holes on the surface of nickel coatings. Electrodeposition has been widely performed to produce CNTs metal matrix composites due to its operation in low cost and low temperature and CNTs have been successfully co-deposited into MMC using electrodeposition [39, 40]. CNTs composite coatings contact surface is the worn surface were much smoothing due to the less plastic deformation [41]. Pulse reverse electroplating method is significant differences in microhardness, corrosion, composition, wear and tribological performance were observed [42]. Corrosion is a fundamental difficulties that result in posses environmental issues and also material degradation of engineering steel components. A nano particles were studies as nano materials for the corrosion coatings to improve their corrosion efficiency [43]. In view of these findings, the main aim of the present work is to study and compare the detailed influence of pulse reverse parameters on nano crystalline Ni-SiCs and Ni-MWCNTs alloy composite coating, corrosion and its mechanical properties on AISI 304 stainless steel subtract.

2. Experimental procedure

2.1. Preparations of samples
The substrate of AISI 304 Stainless steel (Size: 10 mm × 30 mm), (Composition (%): C- 0.08, Mn- 2.00, Si- 0.75, S- 0.03, P- 0.045, Cr- 19, Ni- 8.75, N- 0.10 and Fe-Remaining) and (Microhardness—253.6 Hv) used as a cathode in this two top surface of samples prepared for coating and cylindrical area was masked using a polyvinyl chloride (PVC) adhesive tape and the anode was made of a pure Nickel (99.9%) plate (size: 100 mm × 30 mm × 10 mm and Composition% : Ni - 99.25, Mn- 0.15 and Fe-0.19). Before electroplating process, the substrate is polished with Silicon carbide abrasive papers (grade of 80–2500) and then kept in an ultrasonically cleaned in acetone for 15 min. Before deposition substrates were washed in distilled water at room temperature. This was done to clean the sample for impurities.

2.2. Ni-SiC nano coating methodology
Ni-SiC electrodeposition solution was prepared using the analytical reagent and distilled water before the Ni-SiC electrodeposition. The Beta SiC (Purity >95%) nano particle purchased from otto (Chemika—Biochemika—Reagents) the size of particles 50–60 nm and particle morphology is spherical with concentration of 10 gl−1 were dispersed in the electrolyte in presence of sodumdodecyl sulfate after that suspended solution were stirred for 16 h.

Ni-SiC composite coatings were electrodeposited from a nickel Watts type bath under pulse reverse current (PRC) method. The compositions of solution for electrodeposition are shown in table 1. The pretreated SiCs were added into a plating cell containing 250 ml of the solution with pH fixed at 4 and kept at a temperature (50 °C), thermostatically controlled by heater mixer.
2.3. Ni-MWCNT coating methodology
Ni—MWCNT electrodeposition solution was prepared using the same procedure in Ni-SiC composite coating. The MWCNT (Purity > 98%) particle purchased from IENT incorporation, the size of particles 16 nm with concentration of 4 g l\(^{-1}\) and the Ni-SiCs carried out by the same procedure as in Ni—MWCNT. The composition for electrodeposition is shown in table 2.

2.4. Pulse reverse electrodeposition parameters
A Dynatronix, USA (Model: Micro star pulse series DPR 20–30–100 power supply) Pulse rectifier used in this experiment. The deposition parameters belonging to the coating of Ni-SiCs and Ni-MWCNTs are as follows: A magnetic bar stirrer was continuously rotated at 500 rpm upon deposition. The deposition parameters used in this study are given in table 3 for reference.

The microstructure of the Ni-SiCs and Ni-MWCNTs composite films was examined was investigated by means of x-ray diffraction (A RigakuMiniFlex) using Cu-K\(\alpha\) radiation. The surface morphology of coatings was characterized with scanning electron microscopy (\(\sum\) version–Corl Zeiss, Germany, 5.07 Beta). Microhardness of the coatings was determined using a Vickers microhardness Shimadzu tester (HMV 2 T indenter). The microhardness measurement has been carried out using a 100 g load applied for 15 s. The reported hardness is the average of ten different readings. The nickel composite coatings were also analyzed for microhardness and corrosion properties.

The following figure 1 represents the geometry of an uncoated sample, Ni—SiC sample and Ni MWCNT sample for reference.

3. Result and discussion

3.1. Discussion on microhardness
Table 4 and figure 2 clearly represents the variation of microhardness for sixteen samples coated with assured pulse reverse parameters [44, 45]. From close observation the following significant results are identified.

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**Table 1. Bath compositions for SiC reinforced Ni matrixed composite coating production.**

| S.no | Electroplating bath composition                  | Value  |
|------|--------------------------------------------------|--------|
| 1    | Nickel sulfate (Ni\(_2\)SO\(_4\)·6H\(_2\)O) (g l\(^{-1}\)) | 300    |
| 2    | Nickel chloride (NiCl\(_2\)·6H\(_2\)O) (g l\(^{-1}\)) | 50     |
| 3    | Boric acid (H\(_3\)BO\(_3\)) (g l\(^{-1}\))         | 35     |
| 4    | Sodium dodecyl sulfate (SDS) (g l\(^{-1}\))        | 0.2    |
| 5    | SiC nano particle (g l\(^{-1}\))                  | 10     |
| 6    | pH Value                                         | 4      |
| 7    | Temperature (°C)                                 | 50 ± 2 |

**Table 2. Bath compositions for MWCNT reinforced Ni matrixed composite coating production.**

| S. no | Electroplating bath composition                  | Value  |
|-------|--------------------------------------------------|--------|
| 1     | Nickel sulfate (Ni\(_2\)SO\(_4\)·6H\(_2\)O) (g l\(^{-1}\)) | 300    |
| 2     | Nickel chloride (NiCl\(_2\)·6H\(_2\)O) (g l\(^{-1}\)) | 50     |
| 3     | Boric acid (H\(_3\)BO\(_3\)) (g l\(^{-1}\))         | 35     |
| 4     | Sodium dodecyl sulfate (SDS) (g l\(^{-1}\))        | 0.2    |
| 5     | MWCNT nano particle (g l\(^{-1}\))                | 4      |
| 6     | pH Value                                         | 4      |
| 7     | Temperature (°C)                                 | 50 ± 2 |

**Table 3. Electrolyte deposition parameters.**

| S. no | Deposition parameters                  | Value               |
|-------|---------------------------------------|---------------------|
| 1     | Cathodic current density (CCD) (A cm\(^{-2}\)) | 0.3–1.0            |
| 2     | Cathodic current time (CCT) (s)        | 0.01                |
| 3     | Relaxation time (RT) (s)               | 0.1                 |
| 4     | Anodic current density (ACD) (A cm\(^{-2}\)) | 0.02–0.08         |
| 5     | Anodic current time (ACT) (s)          | 0.02–0.1            |
A combination of higher cathodic current density (CCD) and lower anodic current density (ACD), anodic current time (ACT) results in maximum magnitude of microhardness levels. Also the rate of cathodic current density is predominant in deciding the resulting microhardness magnitude. Moreover, incorporation of SiC and MWCNT nano particles significantly enhances the microhardness to a maximum of 91.6% and 168% respectively while compared with the uncoated sample. Further, in all the proposed parameter combination, the same trend is evidenced. However, the rate of variation oscillators from 1.21 to 2.68 times for different samples. Hence, it is evident that, nanocomposite coating reinforced with MWCNT’s out performs in all the cases. However, on economic consideration, it is preferable to use SiC nano particles for most cases. The cost of MWCNTs while compared with SiC particles are quite higher and at the same contest the average hike in the hardness parameter is only about 27%. This can also be achieved by varying the composition of SiC particles to maintain a balanced relationship with cost and microhardness.

The following figure 3 portrays the variation of microhardness over cathodic current density. From the plot, it is evident that, there is no significant change at the lower range of cathodic current density. For better clarity, the raw data fit into a polynomial trend line to predict the realistic effects. The fifth order polynomial function is deployed with an agreeable adjusted R² value of 0.95 and above. The plot indicates that, the altitude of microhardness is higher at 1 A cm⁻², thereafter a saturation phenomenon is evidenced. The subsequent increase in the cathodic current density (CCD) after this optimal zone has a relatively less significant effect over the hardness of coated samples.

Table 4. Designation of Specimen deposited at different parameters with Microhardness.

| Sample No. | CCD (A cm⁻²) | ACD (A cm⁻²) | ACT (s) | Ni—MWCNT (Hv) | Ni—SiC (Hv) |
|------------|--------------|--------------|---------|----------------|-------------|
| 1          | 0.3          | 0.02         | 0.02    | 317            | 306.5       |
| 2          | 0.3          | 0.08         | 0.1     | 326.4          | 322.1       |
| 3          | 0.4          | 0.02         | 0.02    | 344.2          | 330.9       |
| 4          | 0.4          | 0.08         | 0.1     | 349.3          | 334.6       |
| 5          | 0.5          | 0.02         | 0.02    | 380.3          | 350.3       |
| 6          | 0.5          | 0.08         | 0.1     | 462.7          | 357.1       |
| 7          | 0.6          | 0.02         | 0.02    | 484.3          | 371.5       |
| 8          | 0.6          | 0.08         | 0.1     | 493.4          | 385.4       |
| 9          | 0.7          | 0.02         | 0.02    | 541.1          | 408.3       |
| 10         | 0.7          | 0.08         | 0.1     | 544.5          | 418.3       |
| 11         | 0.8          | 0.02         | 0.02    | 581.7          | 422.1       |
| 12         | 0.8          | 0.08         | 0.1     | 632.9          | 428.1       |
| 13         | 0.9          | 0.02         | 0.02    | 671.6          | 482.3       |
| 14         | 0.9          | 0.08         | 0.1     | 670.1          | 480.8       |
| 15         | 1            | 0.02         | 0.02    | 679.7          | 485.9       |
| 16         | 1            | 0.08         | 0.1     | 675.6          | 483.2       |
3.2. Potentiodynamic polarization

The plot shown in Figure 4 depicts the potentiodynamic polarization curves for three different samples (Uncoated AISI 304 stainless steel, Ni-MWCNT coated and Ni-SiC coated). The plot is varied against the logarithm of the corrosion current density and corrosion potential (E). The required (log i) data are extracted from Tafel test close observation of the plot reveals that, the corrosion potential of Ni-MWCNT, Ni-SiC and uncoated sample are in descending sequence. The Ni-MWCNT sample reported a maximum corrosion potential of 0.280.

In addition, the polarization resistance ($R_p$) and corrosion rate are calculated using statistical tools and the final values are reported in Table 5. Hence, it is evident that Ni-MWCNT coated samples process higher polarization resistance and lower corrosion rate. To confirm this decision, electrochemical impedance spectroscopy studies are also performed for validation and the results are discussed in the subsequent section.

As seen from Table 5, reveals the corrosion characteristics obtained by pulse reverse electrodeposion measurement. From close observation, it can be seen that the corrosion current density of the coatings decreased with Ni-MWCNT particles. Also, the polarization resistance ($R_p$) is low for uncoated sample followed by Ni-SiC and Ni-MWCNT particles.
3.3. Electrochemical impedance spectroscopy (EIS)

Intensive investigation of literature revivals that, Electrochemical impedance spectroscopy is a powerful tool to assess the corrosion resistance characteristics. Figures 5, 6 and 7 shows the measured impedance spectra of three specimens as bode plot and nyquist plot respectively.

Bode plot represents the variation of frequency in logarithmic scale versus impedance modulus \((Z_{\text{mod}})\). It can be clearly seen that, Ni-MWCNT coatings have highest impedance modulus of \( \approx 550 \, \text{ohm cm}^{-2}\). Ni-SiC coatings have moderate amplitude and uncoated specimens possess the least one. The shape and pattern of all the three samples in nyquist plot is almost similar. However, the geometrical radius and the ending point is different. This particular phenomenon explains that the corrosion pattern will be similar in all the three cases, however, with a varying time rate following the same style of corroding mechanism. Moreover, higher radius of circles in nyquist plot and higher value of impedance mode in bode plot implies a higher corrosion resistance characteristics. Hence, it is obvious that Ni-MWCNT samples out performance well while compared with all

| Sample       | \( E_{\text{corr}} \) (V) | \( i_{\text{corr}} \) (μA cm\(^{-2}\)) | \( R_p \) (Ω cm\(^2\)) |
|--------------|--------------------------|--------------------------------------|-------------------------|
| Uncoated     | -0.589                   | 0.7179                               | 327                     |
| Ni-SiC       | -0.552                   | 0.3458                               | 356                     |
| Ni-MWCNT     | -0.499                   | 0.0176                               | 710                     |
other samples. Further, the results predicted by electrochemical impedance spectroscopy data and plots strongly support the conclusive derived from potentiodynamic polarization studies.

As seen from table 6, the presence of Ni-MWCNT particles in the coating leads to an increase in $R_{ct}$ values and a decrease in $Y_o$ values. This phenomenon might be due to the fact that, the incorporation MWCNT particles in Ni matrix to fill the pores through the coating at a uniform rate.

### 3.4. Microstructure investigation

The microstructure of the coated specimens were assessed using scanning electron microscope (SEM) as shown in figures 8(a), (c). The images clearly reveal that, Ni-SiC coating has scattered porosity zones along the entire surface. However, Multi walled carbon nanotube (MWCNT) coated samples exhibit a dense structure and thereby the micro-hardness value is enhanced. The micro-structure of Ni-SiC samples reveal a bar shaped geometrical profile with varying lengths. The growth mechanisms of Ni showed to be prone to spherical grains. Co-deposited MWCNTs are clearly observed on the surface as a necklace like network which confirms that MWCNTs in nickel structure resulted in grain morphology modification and the pyramidal grain morphology. Spherical Ni grain growth can be attributed to reduction of nickel ions onto defect zones created by functionalization of MWCNTs [46, 47].

On the other hand, the Ni-SiC and Ni-MWCNT nano composite coating shows smoother, more uniform and compact surface. It was expected that the reduction of the grain size of Ni crystallites was due to the presence of SiC and MWCNTs particles. This can be clearly seen in SEM micrographs of composite deposits. The reduction in the grain size and the change in the structure of nickel matrices could be attributed not only to the presence of the SiC and MWCNTs particles in the Ni matrix, but also to their physico-chemical interaction.

| Sample    | $R_{ct}$ ($\Omega \text{cm}^2$) | $Y_o$ ($\Omega^{-1} \text{cm}^{-2} s^{-1}$) | $n$  |
|-----------|---------------------------------|-----------------------------------------|------|
| Uncoated  | 358.5                           | 0.001 0440                              | 0.7552 |
| Ni-SiC    | 385.5                           | 0.000 4198                              | 0.8076 |
| Ni-MWCNT  | 749.9                           | 0.000 5437                              | 0.8858 |

Figure 6. Nyquist impedance diagrams for Ni-MWCNT, Ni-SiC and Uncoated sample in 1 M of H$_2$SO$_4$.

Figure 7. Equivalent circuit for Uncoated, Ni-SiC and Ni-MWCNT sample in 1 M of H$_2$SO$_4$. 

Table 6. Electrochemical parameters fitted from EIS measurement.
when approaching the catholytic area. This leads to changes in catholyte composition by adsorption desorption phenomena on their surface. It also perturbs the crystal growth by increasing the number of nucleation sites and consequently a reduction in the crystallite size occurs [48].

The influence of MWCNTs and SiC particles on the structure and surface morphology of the nickel coatings were investigated by x-ray diffraction (XRD). The XRD diffractograms for Ni-SiCs and Ni-MWCNTs nano composite coatings are indicated in figures 9(a), (b). The nickel layer deposited in the presence of MWCNTs/SiCs particles shows a completely different crystal orientation from what has been considered previously for pure nickel deposits [49].

The coatings exhibit an FCC lattice structure with dominant reinforcement at (111) plane followed by (200) and (220) planes for Ni-SiC coatings. However, for Ni-MWCNT coated samples (111) plane seems to be highly influencing followed by (222) and (220) planes.

Energy dispersive x-ray (EDX) analysis figures 8(b), (d) reveals that Ni-MWCNT coated samples have carbon (C K), iron (Fe K), Nickel (Ni K), oxide (O K) and Manganese (Mn K) content of 35.15%, 21.32%, 37.53%, 5.24% and 0.76% respectively, while comparing Ni-SiC coatings, the dominance of the carbon content is high in Ni-MWCNT coatings. This clearly reveals that, Ni-MWCNT coatings will yield higher hardness levels due to the presence of higher carbon content.

Figure 8. (a) SEM images of Ni-SiC (b) EDX of Ni-SiC (c) SEM images of Ni-MWCNT (d) EDX of Ni-MWCNT.

Figure 9. XRD pattern of (a) Ni-SiC coating (b) Ni-MWCNT coating.
3.5. Discussion on coating thickness and atomic force microscope (AFM)

The pulse reverse parameters for RPD were optimized to get a better coating thickness was investigated using Dewinter Tech—Metallurgical microscope, vilella’s reagent was used as an etchant in this study. The thickness of coatings was taken at five different geometric points and the mean value is reported in the following table 7 and figures 10(a), (b). The Ni-MWCNT coatings exhibit relatively less coating thickness due to the fine grain structure and subsequent compact packing characteristics.

Further, figures 11(a), (b) portrays the results of atomic force microscope (AFM) analysis. The average surface roughness values of Ni-SiC and Ni-MWCNT samples are 1.8 and 1.08 respectively. As the value of surface roughness increases the contact area is reduced and a ploughing effect will be evidenced. Also, higher surface roughness leads to higher wear rate due to weaker bonding and lower area of contact.

The morphological finding was further confirmed through that the Ni-MWCNT and Ni-SiC coatings. The AFM analysis confirmed that the developed coatings free of defects such as cracks and pores. In the AFM of Ni-MWCNT and Ni-SiC Nano composite surface deformation and tribological properties of coatings are caused by high roughness due to surface flaws and large crystal agglomerates in the Nano composite coatings [50, 51].

![Figure 10. Image of coating thickness of (a) Ni-SiC coating (b) Ni-MWCNT coating.](image)

![Figure 11. AFM images of (a) Ni-SiC (b) Ni-MWCNT.](image)

### Table 7. Microscopic method of coating thickness measurement.

| Sample       | Coating thickness at random locations | Coating thickness (μm) |
|--------------|---------------------------------------|------------------------|
| Ni-SiC       | 08 08 08 08 10                        | 08                     |
| Ni-MWCNT     | 06 06 08 06 06                        | 06                     |

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4. Conclusion

The corrosion resistance characteristics of Ni-SiC and Ni-MWCNT nanocomposite coatings over AISI 304 stainless steel samples are investigated in this research work by pulse reverse electrodeposition technique. Based on the study, the following conclusion is drawn. Microscopic investigation of coated specimens clearly reveals that, pulse reverse electrodeposition is effective in formulation of better coating thickness with the uniform surface distribution. Based on Vickers microhardness test, it is concluded that Ni-MWCNT coating outperformed well in all samples with higher magnitudes of microhardness. Also, it is clear that cathodic current density plays a dominant role in microhardness value. Higher range of cathodic current density yields higher microhardness for all the samples. Both potentiodynamic polarization study and electrochemical impedance spectroscopy reveal that, Ni-MWCNT coated samples possess excellent corrosion resistance characteristics while compared with Ni-SiC samples.

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