



Preparation and properties of electrodeposited Ni-B-V₂O₅ composite coatings

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ABSTRACT

Coatings of Ni-B have gained significant importance in various industries owing to their major role in improving mechanical properties including hardness, conductivity, and wear resistance. Despite all these characteristic features, there is still the need for a lot of modifications. This is to improve the properties of the coating so as to increase their durability and overall performance. The current study is based on development of Ni-B-V₂O₅ composite coating on mild steel substrate through the electrodeposition technique and the investigation of mechanical and anti-corrosive properties of the formed coating. The incorporation of V₂O₅ particles into the composite coating was confirmed by energy dispersive spectroscopy. X-ray diffraction pattern showed amorphous nature of electrodeposited Ni-B matrix, while the crystalline nature improved with the addition of V₂O₅ particles to the composite. Field emission scanning electron microscopy and atomic force microscopic studies clearly indicated that the addition of V₂O₅ particles to the Ni-B coating increased the surface roughness. Further studies reveal increase in the micro-hardness (by 171.11%), and elastic modulus (by 9.4%) in case of the Ni-B-V₂O₅ composite coating relative to the Ni-B coating. The enhanced micro-hardness was attributed to the inclusion of hard V₂O₅ particles into the Ni-B matrix, which in turn, may inhibit the dislocation motion in the composite. An increase in corrosion resistance (by 229%) was also experienced in the electrodeposited Ni-B-V₂O₅ composite coating in comparison to the bare Ni-B matrix, which may be due to the masking of inert V₂O₅ particles on the active region of the Ni-B composite.

1. Introduction

The surface mechanical forces and environmental electrochemical reactions can cause wear and corrosion processes on the surface of mechanical components, respectively. The collective impacts of these two phenomena can lead to many aggressive damages such as rapid deterioration and frequent breakdown of machine components that can cause economic losses and even threaten human life. Several methods have been developed to prevent and control material from wear and

corrosion, such as alloying, heat treatment, and protective coating [1–6]. In all these methods, the coating has developed as a promising solution for increasing the durability and performance of the material as it is simple and cost-effective [7]. The fundamental necessity of a coating is to change the surface properties (hardening, smoothing, surface energy, and chemical reactivity, etc.) of a material to increase wear and corrosion resistance. There are several techniques available for coating techniques including sputtering, CVD, sol-gel, plasma spray, laser ablation, atomic layer deposition, and electrodeposition (ED) [8–11].

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Table 1
Electrolyte raw materials and working parameters for ED coating bath.

Sr. no.	Chemicals	Composition
1	Nickel sulphate hexa hydrate	240 g/l
2	Nickel chloride hexa hydrate	45 g/l
3	Boric acid	30 g/l
4	Dimethylamine borane complex (DMAB)	3 g/l
5	Vanadium oxide	0 and 15 g/l

Sr. no.	Working parameters	Composition
1	pH	4 ± 0.1
2	Temperature	55 ± °C
3	Current	50 mA/cm ²
4	Deposition time	30 min
5	Coating solution stirring	600 rpm

Among all these techniques, ED is more promising for the following reasons: (1) the thickness, morphology and composition of the coating can easily be tuned by changing electrochemical quantities like voltage, and current, (2) it allows a uniform coating to be formed on a large/uneven surface, (3) it is cost-effective, rapid and can be implemented on an economical scale. Furthermore, this technique has been extensively used for deposition of nanocomposites due to its atomic precision, low porosity, high purity, higher deposition rate, to name a few [12–16]. Despite several advantages, literature reports have shown that EDs have not often been used to deposit Ni-B coatings [6,17–19]. Hence, the present work is based on the ED technique to develop Ni-B and Ni-B-V₂O₅ coatings on a mild steel substrate. Recently, Ni-B coating has received significant attention due to its versatile characteristics including good micro-hardness, low porosity, and superior wear resistance, among others. For all these reasons Ni-B coating can be considered as an alternative that has the potential to replace the hard chromium coating in future. However, its practical application also requires resistivity in corrosive environments. Therefore, to increase the durability and in-service performance of such coatings, the demand for further improvements in mechanical and anti-corrosion properties continues [6,20–23]. To achieve all these properties, various methods have been explored such as changing the manufacturing process or incorporating reinforcement materials including oxides and carbides with the Ni-B coating. Previously, the authors group has shown significant progress for anti-corrosive and mechanical properties of Ni-B composite coatings by including second-phase hard materials such as Si₃N₄, Ti₂O₃, and CeO₂ etc. [17–19] Here, the authors have studied mechanical, and anti-corrosive properties of ED Ni-B-V₂O₅ coating in which V₂O₅ has been chosen as the reinforcing material. The curiosity in ED Ni-B-V₂O₅ coating concerns the development of surface coating for industrial applications with higher hardness and better corrosion-resistant property. In addition, detailed elaboration on the developments of the surface structure, morphology, and thermal properties because of the addition of V₂O₅ particles into the Ni-B matrix has also been made.

2. Experimental details

2.1. Coating preparation

Both Ni-B and Ni-B-V₂O₅ coatings were fabricated on a mild steel surface (22 × 2 mm), using electrochemical deposition technique. On circular mild steel Ni-B and Ni-B-V₂O₅ coatings were electrodeposited. The 22 mm diameter and 2 mm thickness were the dimension of steel substrate. Vanadium oxide (V₂O₅), a yellow powder purchased from Sigma Aldrich (Merck) was used for the present study. The size of the V₂O₅ particles was in between 70 and 90 nm. All samples (mild steel substrate) were mechanically ground using SiC paper of various grades, such as 80, 320, 500, 1000, 1200, 2500, 4000 to achieve crystal clean surface finishing. After mechanical grinding, the degreasing of samples

Table 2
Compositional study of ED Ni-B and Ni-B-V₂O₅ coatings.

Coatings	Ni (wt%)	B (wt%)	V ₂ O ₅ (wt%)
Ni-B	84.12	15.88	–
Ni-B-V ₂ O ₅	68.07	26.48	05.45

was done using acetone and then treatment with alkaline water and finally was washed with deionized water. At the end of cleaning, the samples were treated with 20% HCL solution for 1 min and subsequently washed again thoroughly with deionized water. Finally, the samples were put into an oven with compressed air to dry. In electrochemical cell, a plain low carbon steel was used as cathode, while Ni plate was used as anode. The ED was performed for 30 min at 55 ± 2 °C. In order ensure uniformity and to avoid precipitation of insoluble V₂O₅ particles, the electrolyte was vigorously shaken and stirred. Table 1 represents different parameters and solution bath composition for ED of Ni-B and Ni-B-V₂O₅ coatings.

2.2. Analysis of samples

ICP-AES of Thermo, ICAP 6500 (USA) and energy dispersive spectroscopy (EDX) was used to confirm the chemical composition of ED Ni-B and Ni-B-V₂O₅ coatings. X-Ray diffractometer (XRD) of Rigaku, Miniflex (Tokyo) with radiation source of Cu-K α was used to check the phase purity and crystallinity of Ni-B and Ni-B-V₂O₅ coatings. FE-SEM-Nova Nano-450 (Netherland), AFM MFP-3D Asylum research (USA) were employed to check the morphology and surface analysis. Differential scanning calorimeter (DSC, JADE, Perkin Elmer) was employed to analyze the phase changes in coatings during heating. The temperature was varied between 25 °C to 450 °C with argon flow during the analysis. MFP-3D NanoIndenter was employed to determine the elastic modulus of ED Ni-B and Ni-B-V₂O₅ coatings. All the measurements were carried out under a 1 mN indentation force applied through a Berkovich diamond indenter. At least five different reading were taken on two different samples prepared under similar conditions and the average was calculated. Oliver-Pharr method was employed to determine the indentation depth. A 30K BOOSTER potentiostat/galvanostat/ZRA (USA) was employed to determine the corrosion resistance. The linear polarization studies were conducted at room temperature in a 3.5% NaCl solution. In the corrosion study set up, the coating, a graphite rod and a saturated calomel electrode served as the working, counter and reference electrode, respectively. In order to ensure reproducibility, at last two different samples prepared under similar conditions were tested. Corrosion potential and corrosion current were determined by extrapolating the linear portions of the anodic and cathodic portions of the Tafel plot.

3. Results and discussion

3.1. Compositional study

The ED includes the addition of DMAB complex and V₂O₅ particles into electrolyte solution that results in co-deposition of boron (B) along with V₂O₅ particles. Table 2 lists the chemical constituents of ED Ni-B and Ni-B-V₂O₅ coatings. From Table 2 it can be inferred that there is successful co-deposition of B and V₂O₅ particles with the ED Ni-B and ED Ni-B-V₂O₅ on mild steel. Incorporation of V₂O₅ particles in the Ni-B matrix led to variations in the chemical composition and surface morphology of Ni-B-V₂O₅ coating. These observations have been found to be consistent with our earlier studies on ED Ni-B-CeO₂ and Ni-B-Si₃N₄ coatings [17–19]. The co-deposition of inert particles (V₂O₅) into ED Ni-B matrix can be understood using Guglielmi's model [24]. Accordingly, this process is considered as two successive steps. First, adsorption of Ni ions onto V₂O₅ surface takes place, and then loose adsorption of these particles onto the cathode surface occurs. These weekly bound V₂O₅

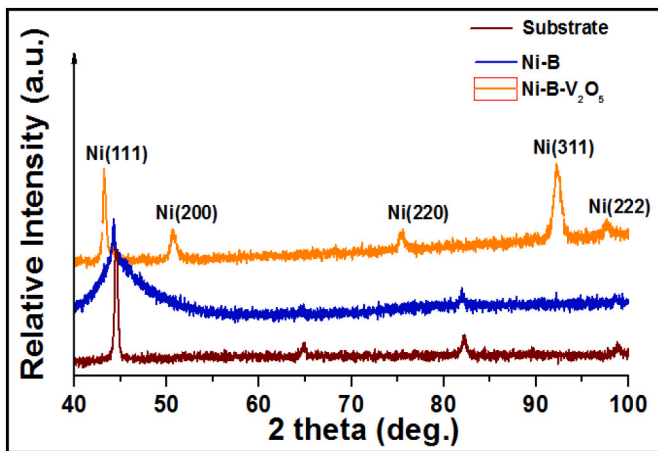


Fig. 1. Comparative study of XRD spectra of mild-steel, ED Ni-B and Ni-B- V_2O_5 coatings.

particles are still remaining surrounded with an ionic cloud of Ni ions. In the second phase, the particles begin to lose their ionic cloud and then bind strongly to the cathode. Subsequently, incorporation of strongly adsorbed V_2O_5 particles in the matrix of growing Ni-B coating takes place. The co-deposition of V_2O_5 particles has led to an increased deposition of B into the coating. The exact reason behind this observation is not known. However, a possible reason might be the deposition of B being assisted by the V_2O_5 particles. In the absence of V_2O_5 , the incorporation of B into the coating is by the adsorption of borane

complexes at the cathode. However, in the presence of V_2O_5 , the borane complexes also get adsorbed on to the surfaces of the ceramic particles, which later get incorporated at the cathode along with the V_2O_5 particles.

3.1.1. XRD spectra

XRD pattern of bare mild steel, ED Ni-B and Ni-B- V_2O_5 coatings is illustrated in Fig. 1. It can be clearly seen that the XRD spectra of the ED Ni-B coating show a clear peak which is single and broad, whereas ED Ni-B- V_2O_5 exhibits several sharp peaks. The appearance of single broad peak reflects the amorphous nature of bare ED Ni-B matrix. Generally, if the boron content in the coating is 4% or more, the coating exhibits amorphous nature. On the other hand, when the boron content is greater than 8%, the single peak widens along with other peaks decreasing [25,26]. In contrast, the sharp peaks, such as Ni (200), Ni (111), Ni (311), Ni (220), and Ni (222) in the composite coating reveal the crystalline nature of ED Ni-B- V_2O_5 . From these observations, it can be concluded entry of V_2O_5 particles in the matrix of Ni-B changes the amorphous structure into the crystalline structure. No conclusive reasons have been found by the authors for this observation. Even the earlier literatures have not given any satisfactory explanation for this observation. A probable reason is that the incorporation of a third element/compound into the Ni-B coating prevents the B to remain in solid solution with the matrix. Rather, it stays at the interface between the third element/compound and the matrix, or in the form of separate clusters. In that way, it improves the crystallinity of the matrix. This requires a further detailed investigation to ascertain the reason. Moreover, the addition of secondary phase particles (V_2O_5) changes the surface roughness, metallicity luster and chemical composition of the

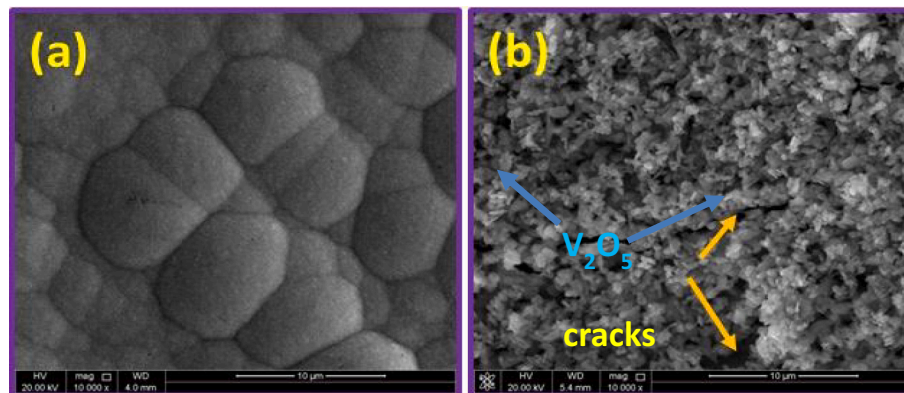


Fig. 2. SEM micrographs depicting surface structure of ED; (a) Ni-B (Image is reproduced from journal article with DOI: <https://doi.org/10.1007/s11837-020-04247-9>). (b) Ni-B- V_2O_5 coatings.

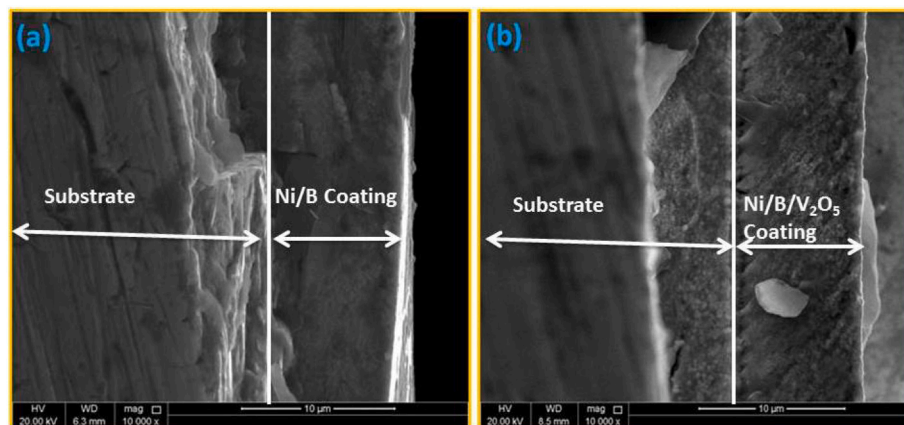


Fig. 3. SEM micrographs depicting cross-section of ED; (a) Ni-B and (b) Ni-B- V_2O_5 coatings.

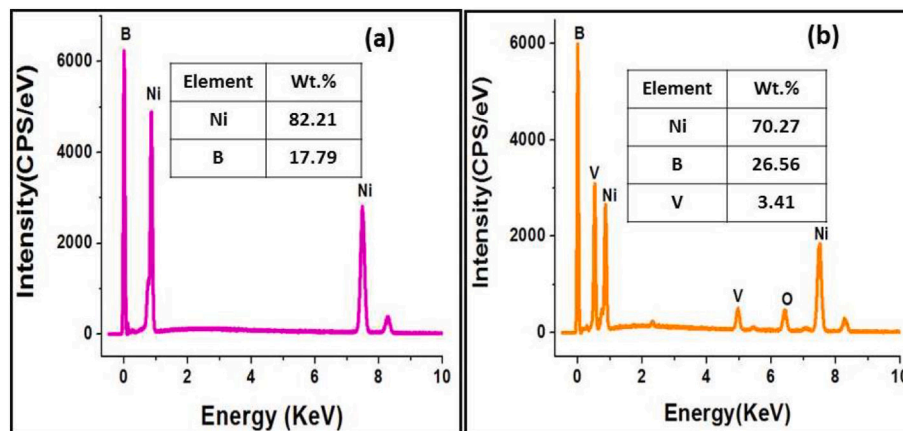


Fig. 4. EDX analysis of ED; (a) Ni-B and (b) Ni-B-V₂O₅ coatings.

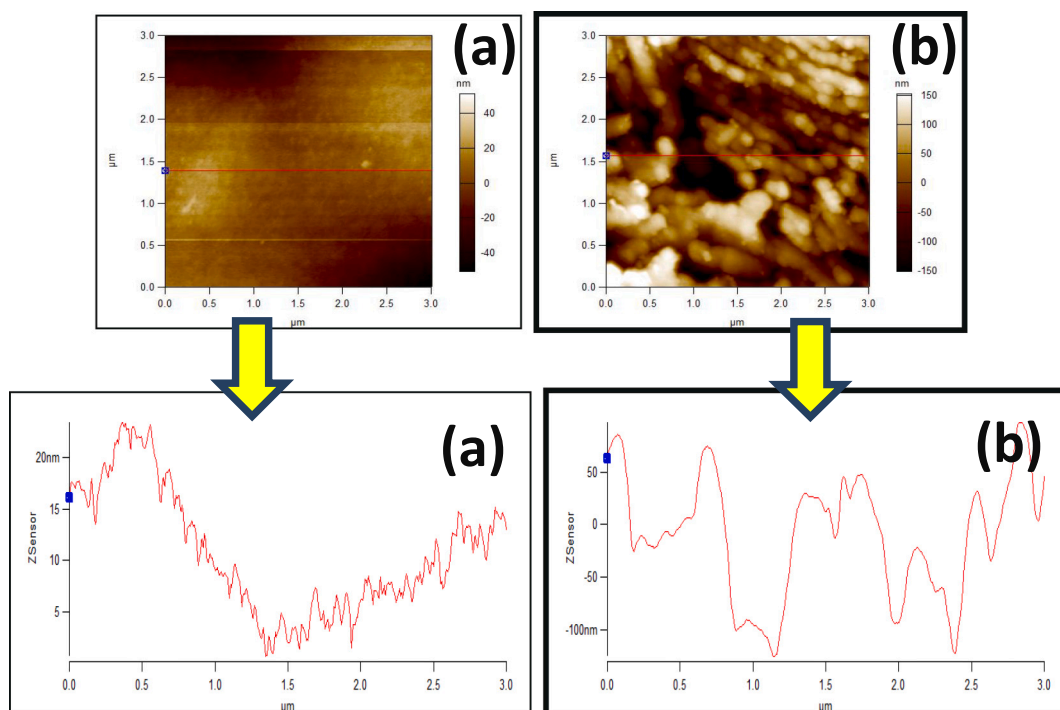


Fig. 5. The roughness of surface profiles of synthesized coatings based on AFM scans of ED; (a) Ni-B coating (b) Ni-B-V₂O₅ coatings.

composite coatings (Table 2). These effects can cause changes in the mechanical and electrochemical properties of Ni-B-V₂O₅ coating.

3.1.2. SEM analysis

Surface morphological analysis of ED Ni-B and Ni-B-V₂O₅ coatings was performed using FE-SEM (Fig. 2). Fig. 2(a) shows densely packed nodular structures in Ni-B coating, which is uniformly and smoothly distributed over the entire surface. It can be clearly seen from Fig. 2(b) that addition of V₂O₅ into Ni-B matrix increased surface roughness of Ni-B-V₂O₅ coating. In our previous studies we have shown that entrance of second-phase particles increases the average roughness of surface [17–19]. Several reasons can cause the increase in the surface roughness of the composite coating such as preferential nucleation, hydrogen evolution reaction, removal of H₂ at the cathode in the presence of these particles (V₂O₅) is difficult, and so on. In addition to increasing in surface roughness, we can observe few micro-cracks on the ED Ni-B-V₂O₅ coating, while there is no crack visible on bare ED Ni-B matrix. Moreover, Fig. 3(b) shows the homogenous distribution of V₂O₅ particles over

the entire surface of Ni-B-V₂O₅ coating.

Fig. 3 depicted the difference between two cross section images of (a) Ni-B and (b) Ni-B-V₂O₅ coatings captured by FE-SEM. It can be inferred that the thickness of Ni-B is quite higher than Ni-B-V₂O₅ coating, the possible reason for that is entered V₂O₅ in matrix of Ni-B. Moreover, Ni-B-V₂O₅ surface is uniformly distributed compared to bare Ni-B.

3.1.3. EDX spectra

Fig. 4(a, b) presents EDX spectra of ED Ni-B and Ni-B-V₂O₅ coatings. The corresponding peaks for elements Ni, and B are presented in Fig. 3 (a). The presence of V, and O peaks with Ni, and B in Fig. 4(b) indicates the successful inclusion of V₂O₅ particles into Ni-B matrix. This EDX analysis has been found to be consistent with our earlier studies on ED Ni-B-CeO₂ and Ni-B-Si₃N₄ coatings [17–19].

3.1.4. AFM analysis

Fig. 5(a, b) presents roughness of surface profiles of ED Ni-B and ED Ni-B-V₂O₅ coatings based on AFM scans. The comparison of roughness of

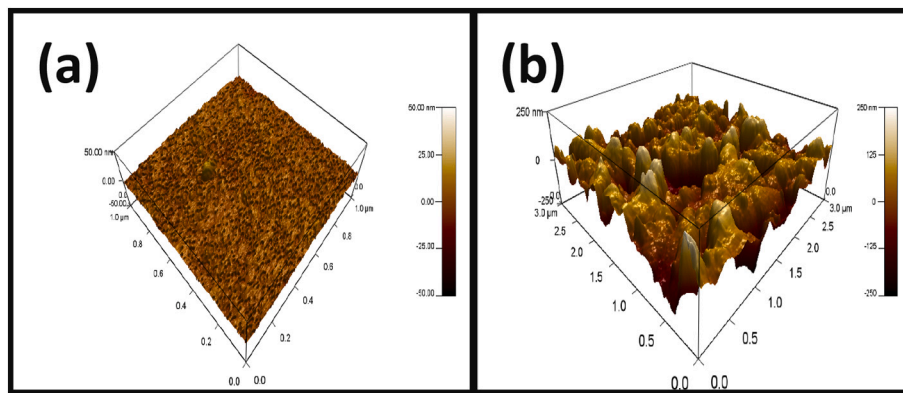


Fig. 6. 3-D AFM images of ED; (a) Ni-B and (b) Ni-B-V₂O₅ coatings.

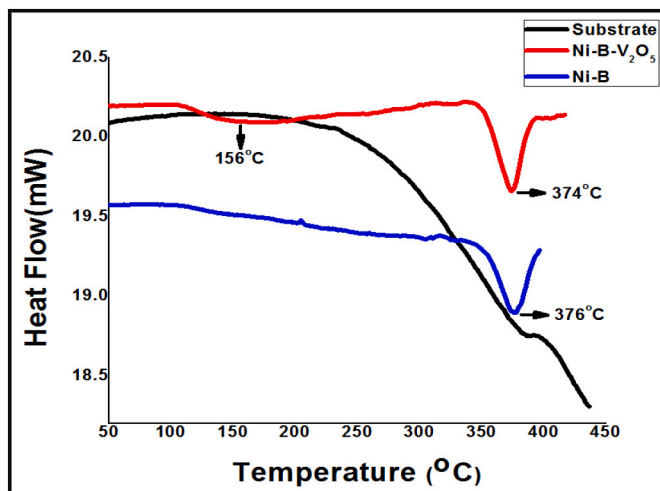


Fig. 7. A comparative study of DSC scans of mild-steel, ED Ni-B and Ni-B-V₂O₅ coatings.

surface profile (Fig. 5) of ED Ni-B coating with that of ED Ni-B-V₂O₅ coating, indicates that ED Ni-B matrix is smoother. The roughness of surface for ED Ni-B coating ranges from about 25 nm to a flat surface. However, the ED Ni-B-V₂O₅ coating was found to be coarser than the ED Ni-B coating and have a value varying between ~52 nm to ~130 nm. In addition, the peaks on the Ni-B-V₂O₅ coating were found to be larger in both height and diameter than those found on the ED Ni-B matrix. From

these results, it can be concluded that the incorporation of V₂O₅ into the Ni-B coating leads to a significant increase in the surface roughness, which may affect the material interaction with the external environment [27,28]. Fig. 6(a, b) shows 3D reconstructions of ED Ni-B and Ni-B-V₂O₅ coating based on 3 μm × 3 μm AFM scans. Fig. 6(b) shows that ED Ni-B matrix has a smooth surface. Conversely, ED Ni-B-V₂O₅ coating on mild steel gave rise to different surface architecture, showing many peaks and valleys.

3.1.5. Thermal analysis

The ED Ni-B and Ni-B-V₂O₅ coatings undergo phase change upon temperature rise that can be best understood using DSC scans. The DSC traces of mild steel, ED Ni-B and Ni-B-V₂O₅ coatings are shown in Fig. 7. The mild steel substrate is taken as a control and can be clearly seen from the DSC trace (Fig. 7) that it does not undergo any phase change. The DSC trace of ED Ni-B coating reveals a single exothermic peak at 376 °C, while two different exothermic peaks are observed for ED Ni-B-V₂O₅ at 156 °C and 374 °C. These two peaks in ED Ni-B-V₂O₅ are attributed to nucleation of two different phases (Ni₃B and Ni₃B₄) of microstructures. The literature reports on DSC scans reveal that the chemical composition of the material has a significant effect on the peak position as well as the number of peaks [29]. Several studies have shown that boron content in Ni-B coating has a significant impact on its thermal properties [22,25,30]. For example, Lee et al. have reported exothermic peak shifts toward lower temperature with increasing boron content in ED Ni-B matrix [25]. From these observations, it can be concluded that the inclusion of V₂O₅ particles into Ni-B matrix leads to chemical compositional changes, and thus, it exhibits different thermal behavior than bare ED Ni-B.

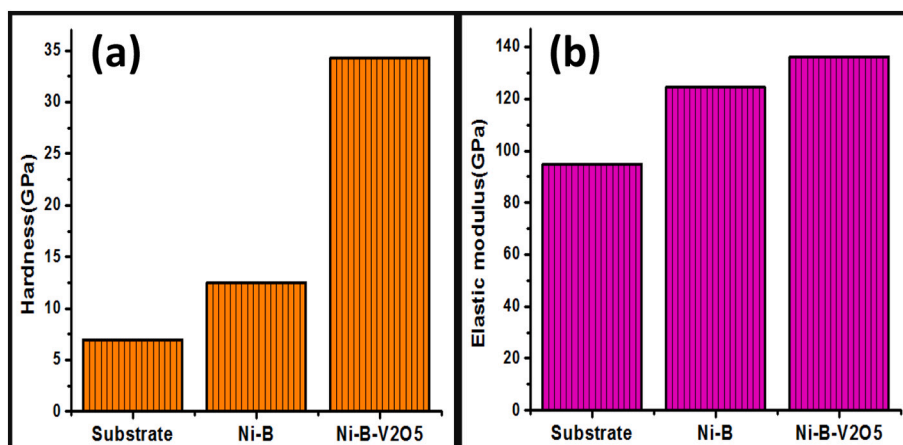


Fig. 8. A comparative study of nano-mechanical characteristics of mild-steel, ED Ni-B and Ni-B-V₂O₅ coatings; (a) hardness and (b) elastic modulus.

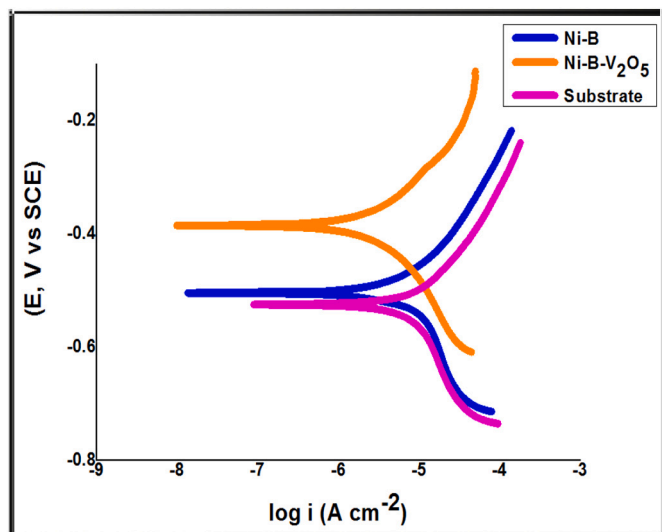


Fig. 9. Corrosion resistance curves of mild steel, ED Ni-B and Ni-B-V₂O₅ coatings in 3.5% NaCl aqueous solution.

Table 3

Calculated values of corrosion parameters of steel substrate, ED Ni-B and Ni-B-V₂O₅ coatings.

Nomenclature	E _{corr} (mV)	I _{corr} (μA)	β a (mV dec ⁻¹)	β c (mV dec ⁻¹)	Protection % (η)
Substrate	-624.0	21.50	70.0	584.7	-
Ni/B	-504.0	16.80	336.5	465.5	27
Ni/BV	-446	8.24	153.5	272.5	89

3.1.6. Mechanical studies

To have a deep knowledge of the mechanical characteristics of the synthesized coatings, the elastic modulus and hardness of the coatings were studied. Fig. 8(a, b) shows comparative studies about hardness and elastic modulus of the mild steel, ED Ni-B and Ni-B-V₂O₅ coatings. Comparing the hardness of the ED Ni-B matrix, which is about 12.70 GPa, with the ED Ni-B-V₂O₅, it can be experienced that the addition of hard V₂O₅ particles into the NiB matrix significantly improved the hardness to about 34.44 GPa as shown in Fig. 8(a). Similarly, the elastic modulus of the ED Ni-B matrix increased from 125 GPa to about 137 GPa with incorporation of V₂O₅ as shown in Fig. 8(b). Quantitatively, the hardness was increased by more than 171% and the elastic modulus also increased by about 9%, which may not a significant increase in elastic modulus. The variation in hardness and elastic modulus (standard deviation) among different reading were observed to less than 6% and 4%, respectively. The observed improvement in hardness of ED Ni-B-V₂O₅ coating may be due to the reinforcement effect of incorporated second phase hard particles such as oxides (Ti₂O₃, CeO₂) and carbides (SiC) as previously reported in several studies [17,31,32]. The improvement in hardness of composite system can be explained by dispersion strengthening effect induced by V₂O₅ particles in Ni/B. These particles hinder the dislocation motion in coating, thereby, improving the micro-hardness of composite coating. In theory, the wear expectancy may be lower if the wearing materials are softer than the wear parts, therefore, the enhanced micro-hardness of the ED Ni-B-V₂O₅ coating in the present study will provide it a better wear resistance property. The mechanical properties of ED Ni-B-V₂O₅ coating have been found to be consistent with our earlier studies on ED Ni-B-CeO₂ and Ni-B-Si₃N₄ composite coatings [17,33].

3.1.7. Anti-corrosion properties

Fig. 9 presents the potentiodynamic polarization curves of mild steel,

ED Ni-B and Ni-B-V₂O₅ coatings immersed in 3.5% NaCl solution. Their corresponding calculated corrosion parameters are also summarized in Table 3. It is quite clear from Fig. 9 and Table 3 that the incorporation of V₂O₅ particles into ED Ni-B coating significantly enhanced corrosion resistance of ED Ni-B-V₂O₅ coating. Clearly, Tafel curve shows that ED Ni-B-V₂O₅ coating has higher E_{corr} (~446 mV) values and lower I_{corr} (~8.24 μA) values than ED Ni-B coatings and bare mild steel. These results have been found according to our previous study on Ni-B-Ti₂O₃ composite coating for corrosion study [34]. In general, E_{corr} is a measure of corrosion resistance of a given material that depends on both intrinsic property of material and corrosion environment [35]. In more detail, the more positive value of E_{corr} indicates that substance will take electrons instead of loss, that is, reduction will more likely to occur. On the other hand, when the E_{corr} falls, it suggests oxidation of substance, thereby, increasing tendency to corrosion. As depicted in Fig. 9, ED Ni-B-V₂O₅ coating has more positive value of E_{corr} (~446 mV) compared to E_{corr} (~504 mV) of ED Ni-B coating and E_{corr} (~504 mV) of steel substrate, suggesting that both Ni-B coating and steel substrate are more prone to corrosion. Moreover, the ED Ni-B-V₂O₅ has also lower value of oxidation current (I_{corr} ~ 8.24 μA) which means that composite coatings have lower corrosion rate. Therefore, a 3-fold increase in overall corrosion resistance was observed in the treatment of Ni-B-V₂O₅ coating on mild steel substrate (Table 3). Several factors determine the coating's effectiveness to prevent corrosion, including the chemical composition of the coating, grain size [36], porosity [37], ability to stop transfer of ions from corrosive solutions to the material [38] and its integrity. The role of inert V₂O₅ particles involved in the Ni-B composite is considered to be a physical barrier that prevents the diffusion of electrolytic ions from the electrolyte to the metal, thereby, decreasing corrosion rate. In addition, it has been reported that addition of an inert substance (V₂O₅) into Ni-B composite coating can passivate active regions on the Ni-B matrix making it less susceptible to corrosion [17]. From these results, it can be concluded that addition of V₂O₅ into ED Ni-B coating significantly improves corrosion resistance.

4. Conclusions

In summary, we have designed the Ni-B-V₂O₅ coating, subsequently used in the surface coating of the mild steel substrate deposited by the electrodeposition technique. It was observed that inclusion of hard V₂O₅ particles into Ni-B matrix led to changes in surface structure, morphology, thermal, mechanical, and electrochemical properties of Ni-B-V₂O₅ coating. The structure of ED Ni-B matrix changes from amorphous to crystalline caused by addition of second-phase V₂O₅ particles. The mechanical properties of composite coating were significantly improved due to the incorporation of V₂O₅ particles into the Ni-B matrix. There is significant improvement in micro-hardness (by 171.11%) of ED Ni-B-V₂O₅ coating compared to ED Ni-B coating which can be explained by the dispersion-strengthening effect induced by hard V₂O₅ particles in Ni-B matrix. A decent increment in elastic modulus of ED Ni-B-V₂O₅ coating was also experienced. Furthermore, corrosion resistance of ED Ni-B coating was increased three folds due to addition of inert V₂O₅ particles. Taken together, this novel ED Ni-B-V₂O₅ coating could be used as a protective coating in machine parts to protect them from wear and corrosion hazards.

CRedit authorship contribution statement

USW, AMSHA and RN designed the work and RN, AKP and AB write down the manuscript. MA, RS and AM commented on the manuscript. USW and AMSHA did few experiments.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence

the work reported in this paper.

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