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Development of electro-co-deposited Ni-Fe(Ti,W)C nanocomposite coatings

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ABSTRACT

The parameters for the electroplating process of Ni-Fe(Ti,W)C nanocomposite on the steel substrate were developed and optimised. For this purpose, the coating process was performed under a direct current using a nickel bath. The coating was fully characterised employing the X-ray diffraction, scanning electron microscopy, energy-dispersive spectroscopy and microhardness tester. The results indicated that the Ni-Fe(Ti,W)C nanocomposite can be coated on the steel with an appropriate structure using the current density and the concentration of 40 mA cm⁻² and 6 g L⁻¹, respectively.

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Ni-Fe(Ti,W)C; electroplating; coating; nanocomposite

Introduction

Surface modification and specialised treatment of materials can improve their physical properties, wear resistance, corrosion behaviour and high-temperature stability of substrates for various industrial applications [1–3]. Nickel-based composite coatings are employed extensively in electronic components, computers, automobiles, textiles industries and petroleum industries for high-pressure valves [4–9]. There are many different techniques for coating the metallic substrates [10–14]. The electrodeposition method is a known technique to produce thin films in the range of a few nanometres in size to thick deposits with large surface areas [6,15–18]. Uniform deposition, low cost, good reproducibility, easy control, versatility, high production rate and reduction of waste can be considered as key advantages of the electrodeposition technique [19–21]. Also, the coating produced by the electrodeposition process is more homogenous and has lower defects compared to that of fabricated with other coating processes [3]. The development of composite coatings based on the hard particles distributed in a metallic matrix has significant potential for engineering applications [8,22,23]. The metal matrix nanocomposites, compared to its bulk counterparts, are more attractive due to their strong properties such as high hardness, improved wear and corrosion resistance, outstanding magnetic property, good electrical and catalytic properties [14]. Currently, nickel-based metal matrix composite coatings have been used in a wide variety of applications, such as hard facing for steel mill rolls and injection moulds due to their enhanced mechanical and chemical properties. The surface modification is usually requested for these materials to improve their corrosion resistance and surface

mechanical properties [24–29]. Recent investigations on nickel coatings have indicated a significant enhancement of properties with the addition of tungsten element [28]. Introduction of W element to the nickel coatings can improve physical and mechanical characteristics including magnetic and electric properties, wear resistance and hardness of the coatings [30]. Such improvements have been observed recently using co-deposition of hard particles such as WC and SiC [22,31]. The purpose of addition of Fe into the carbide composition was to replace a component of Ni-matrix composite coating by a cost-effective material. Thus, the main purpose of the development Ni-Fe(Ti,W)C is to enhance the physical and mechanical properties of Ni-matrix coatings using the cost-effective materials. In addition, to improve the properties of metal matrixes such as Ni, Cr, Co, Re, successful studies were conducted recently using co-deposition of hard particles such as Al₂O₃, TiO₂, SiC, WC, Cr₃C₂, TiC and diamond [6,8,28,32–37].

Song et al. [38] produced a Ni-WC composite coating using the vacuum infiltration casting technique. In the present study, we produced Fe(Ti,W)C carbide as the reinforcement in Ni-matrix nanocomposite coating using the electroplating as a cost-effective method. AISI 304 steel and Aich's alloy brass were selected as the substrate materials due to their applications in hopper lining and the moving part of marine apparatus. More specifically, current density and Fe(Ti,W)C concentration variation were optimised to obtain appropriate coatings.

Materials and methods

The coatings were electrodeposited galvanostatically by applying direct current. Using a digital coulometer

Table 1. Chemical composition of 304 stainless steel and brass substrates.

Substrate	Cr	Ni	C	Mn	Cu	Zn	Sn	P	Co	Fe
AISI 304	18.23	8.024	0.051	1.525	0.233	0.028	0.141	Bal.
Brass	60.66	36.58	1.02	1.74

model BHP 2050, deposition was performed at 50°C. AISI 304 steel and brass Aich's alloy were used as the cathodes. Table 1 shows the chemical composition of AISI 304 steel and brass. An electrode of nickel was used as the anode, in a solution with pH 4.4 ± 0.1 . Before plating, steel and brass substrates (2 cm * 2 cm) as cathode electrode were mounted and connected to the copper wire to conduct electric current. Then the substrates were mechanically polished down to 4000 grit size using abrasive SiC papers and also polished with 0.3 μm alumina. After that, the specimens were cleaned ultrasonically in ethanol for 10 min, and then activated in hydrochloric acid (HCl 15 v/v%) at room temperature for 20 s, washed in distilled water and immediately placed in the electrodeposition bath. The bath composition was $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ (45 g L⁻¹) and H_3BO_3 (40 g L⁻¹). The carbide powder (Ti,W)C including 20 wt-% Fe was used to make composite particles in the solution. Fe element has been added in the powder form. Since Fe(Ti,W)C is the second phase in the Ni matrix, it should occupy less than 50 wt.% of the composite [39,40]. Thus, a middle percentage of Fe (i.e. 20 wt.%) was selected to be able to add a good amount

of (Ti,W)C to the composite to have a total concentration of less than 50 wt.% for Fe(Ti,W)C. A 24 h rest was applied for the prepared solution and it was agitated ultrasonically for 30 min before to the electrodeposition to prevent agglomeration of the Fe-(Ti,W)C particles. Argon gas with low pressure was used to agitate the solution during the coating process. The electrodeposition process was performed in the current density of 20, 40 and 150 mA cm⁻² and the concentration of Fe(Ti,W)C in the solution was 6 and 16 g L⁻¹. Following optimisation of the current density, the experiment was repeated with the concentration of 16 g L⁻¹. The reason for choosing 16 g L⁻¹, which is significantly higher than 6 g L⁻¹, was examining the influence of carbide concentration in our experiment. It was ideal to do the experiment with several carbide concentrations and analyse their results. However, since planning such experiments was practically difficult and expensive, two significantly different carbide concentrations (6 and 16 g L⁻¹) were selected to study the influence of carbide concentration in the experiment. It is worth mentioning that the electrodeposition parameters were selected based on the visual

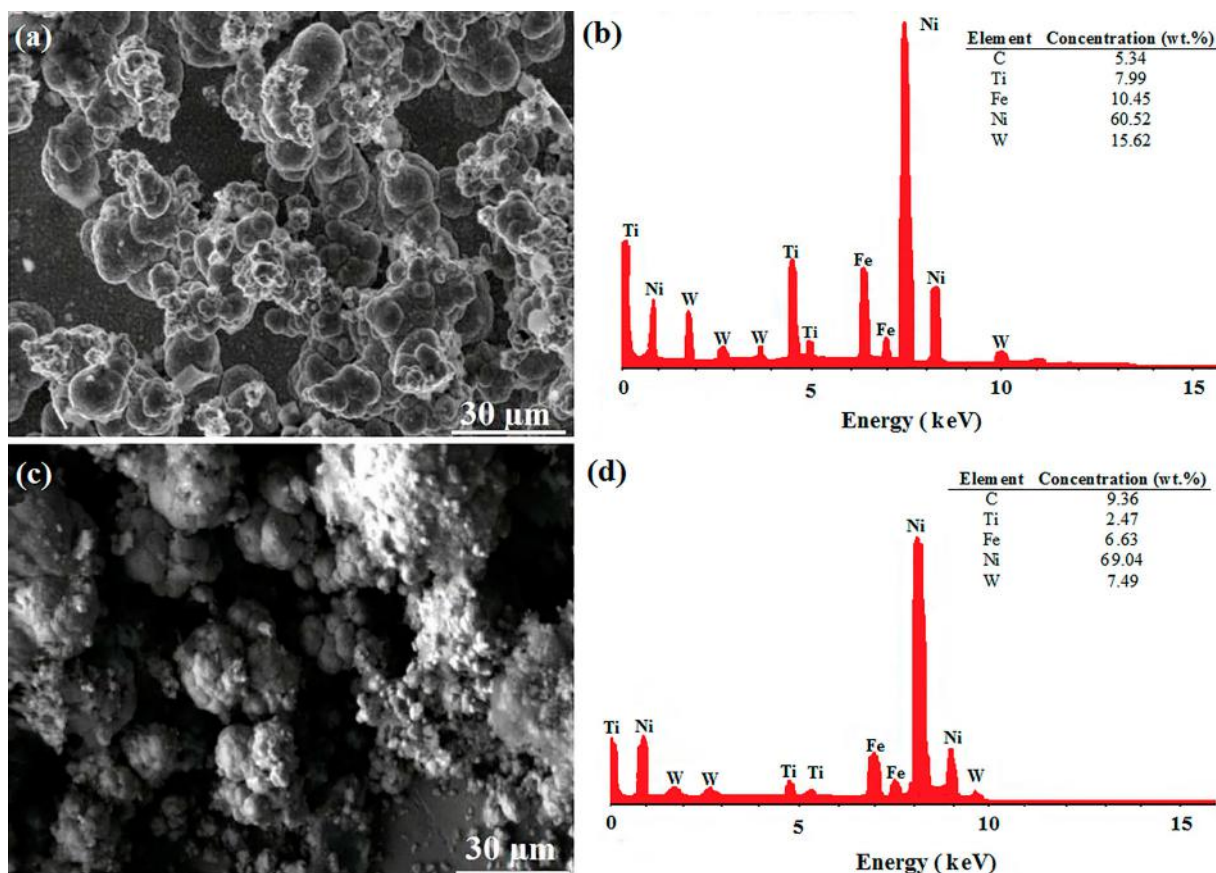


Figure 1. SEM images (a, c) and the EDS analysis (b, d) of prepared coatings in 20 (mA cm⁻²) current density and 6 g L⁻¹ carbide concentration on the steel (a, b) and brass (c, d) substrates.

inspections of the coating quality in terms of thickness and crack formation on the coating surface. The surface morphology of coatings was studied by a scanning electron microscope (SEM, Philips XL30) equipped with an energy-dispersive spectroscopy (EDS). The Vickers microhardness (HV in kgf mm^{-2}) of Ni-Fe (Ti,W)C nanocomposite coatings was measured on their surface using a Reichert microhardness tester under 50 g load for a period of 10 s and corresponding final values were determined as the average of 15 measurements. To evaluate the deposited phases, an X-ray diffractometer (40 kV) with Cu $K\alpha$ radiation was utilised. Using the X'Pert HighScore software, X-ray diffraction (XRD) patterns were analysed. The crystallite size (d) was calculated by software as the following equation (due to lack of lattice strain):

$$d = K\lambda/\beta.\cos\theta \quad (1)$$

where θ is the Bragg diffraction angle, λ the wavelength

of radiation used, β the structural broadening, which is the difference in integral profile width between the standard and sample, and K the Scherrer constant (0.91).

Results and discussion

Figure 1 shows the SEM images (a,c) and the EDS analysis (b,d) of prepared coatings in $20 \text{ (mA cm}^{-2}\text{)}$ current density and 6 g L^{-1} carbide concentration on the steel (a,b) and brass (c,d) substrates.

The SEM photomicrographs of the coating on the steel and brass substrates indicated that the coating on the brass substrate was more porous with inappropriate accumulation, cohesion and connection similar to a spongy configuration. The EDS analysis indicated that the amount of precipitation of carbide (wt.% Fe + wt.% Ti + wt.% W + wt.% C) in the produced coating on the steel substrate is more than that of the brass substrate [20]. Electrodeposition is a

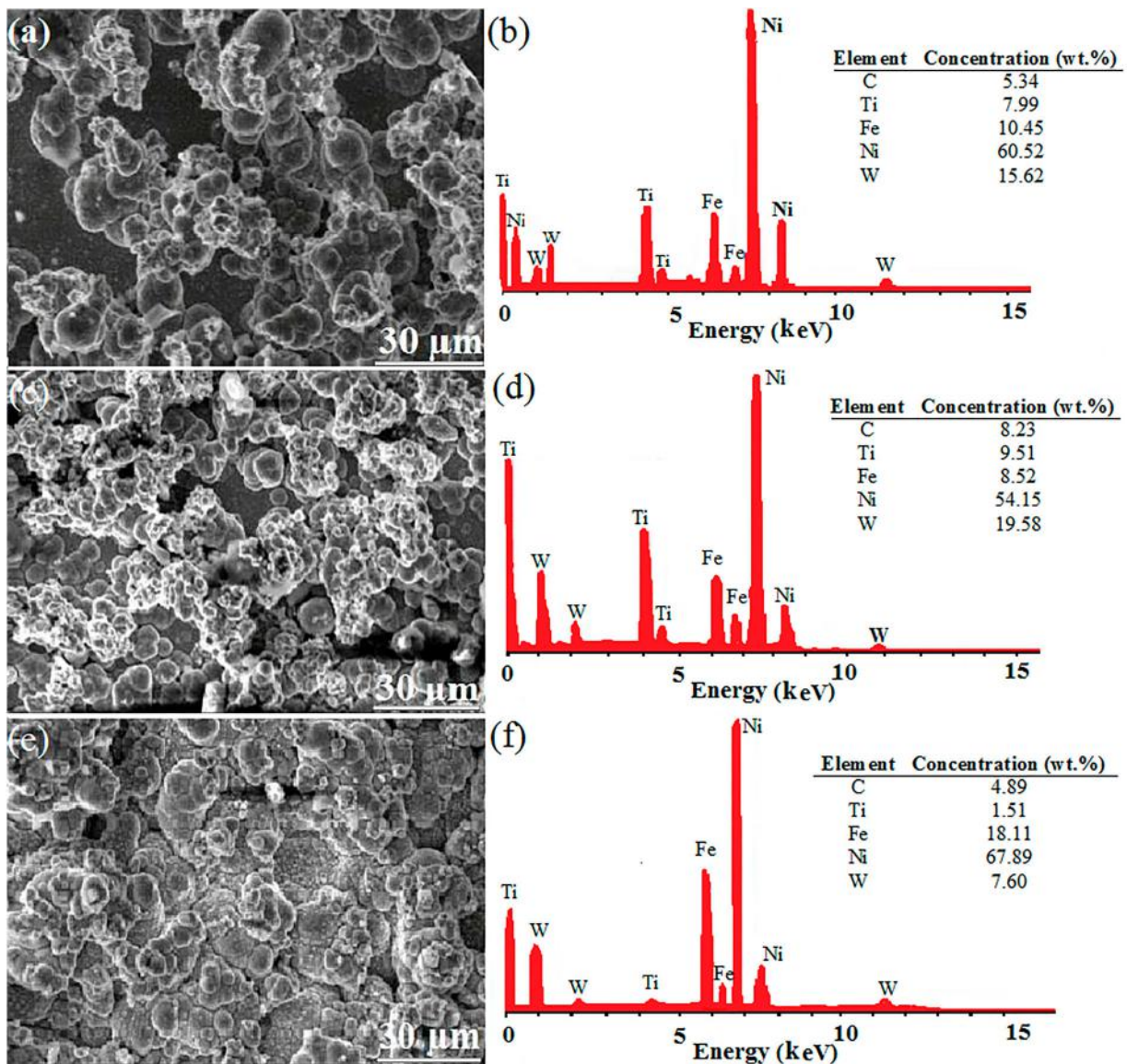


Figure 2. SEM images (a, c, e) and the EDS analysis (b, d, f) of the Ni-Fe-(Ti,W)C coating on the steel substrate with a concentration of 6 g L^{-1} and current density of (a, b) 20 mA cm^{-2} (c, d) 40 mA cm^{-2} , (e, f) 150 mA cm^{-2} .

sophisticated form of dip coating that involves immersing a conductive substrate into a conductive waterborne coating solution. In this process, an electric current is used to incorporate the metal ions into the solution so that these ions form a coherent metal coating on an electrode. In our study, since the capability of steel in the absorbance of electrons from the solution is better than that of the brass substrate, the more accumulation and higher cohesion of the coating materials that prevent the formation of a spongy-shape surface can be observed (Figure 1) [20].

It was found that the physical properties of coatings prepared by the electrodeposition process were strongly affected by deposition parameters such as current density, concentration of electrolyte, pH value of the electrolyte, deposition time and bath temperature [40,41]. Among them, the current density, as a key factor, specifies the physical properties of electrodeposited coatings during the growth process [39]. Therefore, the pH value, the temperature and the concentration of electrolyte were kept constant to investigate the effect of current density. Figure 2 shows SEM photomicrographs and the EDS analysis of the Fe-(Ti,W)C coating on the steel substrate with the concentration of 6 g L^{-1} and current density of 20, 40 and 150 mA cm^{-2} . Comparing the SEM photomicrographs in Figure 2, one may conclude that by increasing the current density, the morphology of the coating becomes smoother and denser. However, increasing the current density results in the fast deposition of nickel and prevents

the adsorption of carbide on the surface. This leads to a decrease in carbide content in the composite [31]. Comparing the results of EDS in Figure 2 confirms that by increasing the current density, the percent of precipitation of carbide increases at the early stage, and then decreases. The first Faraday law explains this fact according to $m = Kq = Kit$, in which m is the mass of precipitated matter, i is the electric current, t is the time to electrodeposition, q is the total charge and K is the coordination constant (electrochemical equivalent). Ni ions could be the carrying agent. When the current increases, there are more ions carrying composite agent in the solution leading to an increase in the amount of precipitated materials [41]. Afterwards, by increasing the current density, the nickel ions are reduced at a faster rate impairing the transportation of carbide. Therefore, the optimum current density for precipitation of carbide on the steel substrate and Fe-(Ti,W)C with a concentration of 6 g L^{-1} is 40 mA cm^{-2} . The highest amount of carbide is incorporated in the mentioned current density compared to others.

Figure 3 shows the SEM photomicrographs (a,c), and the EDS analysis (b,d) of coating in 6 g L^{-1} (a,b), and 16 g L^{-1} (c,d) carbide concentrations and the current density of 40 mA cm^{-2} . According to the SEM photomicrographs, increasing the carbide concentration in the solution from 6 to 16 g L^{-1} resulted in rougher and spongier morphology of the coating. By increasing the carbide concentration, the number of

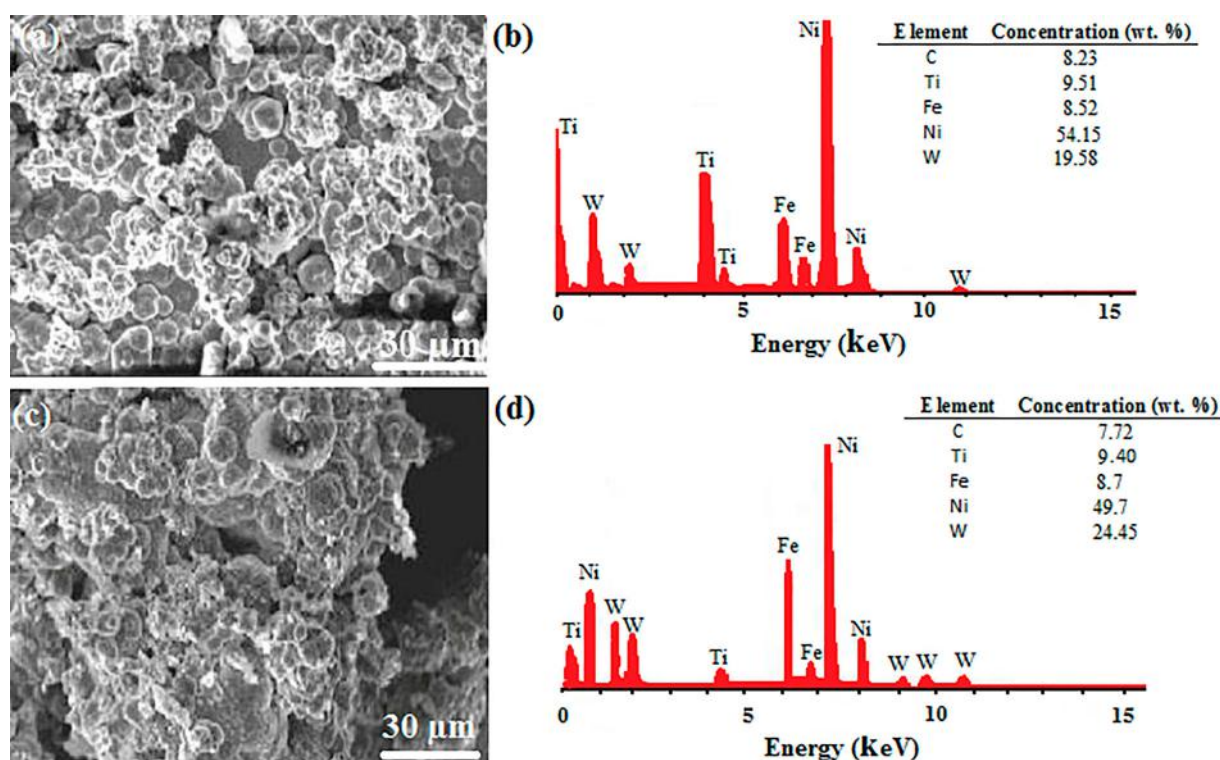


Figure 3. SEM images (a, c) and the EDS analysis (b, d) of coating in 6 g L^{-1} (a, b), and 16 g L^{-1} (c, d) carbide concentrations and the current density of 40 mA cm^{-2} .

voids in the coating layer increases, and the aggregation of Fe-(Ti,W)C (total concentration of Fe + Ti + W + C) also increases. According to the EDS analysis, rising the carbide concentration resulted in an increase in the amount of (Ti,W)C precipitation. The morphology of the deposited carbide can be attributed to the adsorption of suspended particles on the cathode surface as recommended by Guglielmi's adsorption model [40]. The particle adsorption occurs on the surface and the metal starts to encapsulate and incorporate the particles. Similar behaviour has been reported previously for Ni-SiC composites [42]. Moreover, the carbide particles may also be transported by the nickel ions.

Figure 4 shows the XRD pattern of the Fe-(Ti,W)C coating on the steel substrate with the concentration of 6 g L^{-1} and the current densities of 20 , 40 and 150 mA cm^{-2} , respectively (a-c). The grain size of coating was calculated by the Scherrer Eq. and the results are summarised in Table 2. According to this table, the current density of 40 mA cm^{-2} with a carbide concentration of 6 g L^{-1} resulted in lower grain size of nickel. At the current density of 40 mA cm^{-2} , the size of Ni crystallite might be sufficient to encapsulate the particle within the limited time period, whereas the particles stay on the surface of cathode, leading to a higher incorporation of (Ti,W)C in the deposits [31]. The relation between microhardness and current density can be observed in Table 2. Since the deposition rate of Ni is proportional to the current density, the capacity for carbide reception decreases by enhancing the current density, and in turn microhardness decreases. During the hardness measurements, the dispersed particles in the matrix may hinder the dislocation movement and the plastic flow. This conflict to deformation is accompanied by an increased hardness value for Ni-Fe(Ti,W)C coating [42]. According to Table 2, although the carbide concentration of 16 g L^{-1} produces a coating with higher microhardness and lower grain size than the coating deposited with 6 g L^{-1} , increasing the carbide concentration in the solution from 6 to 16 g L^{-1} results in rougher and spongier morphology of the coating according to SEM photomicrographs (Figure 3). The results obtained from EDS and XRD analyses showed that the coating was Ni-Fe matrix and the double carbide nanoparticles of (Ti,W)C. The Ni-Fe coating is employed in many areas such as corrosion, wear, magnetic and electrical applications, and electrocatalytic materials [43]. Fe-Ni alloy electrodeposition exhibits the phenomenon of anomalous co-deposition. Co-deposition is the preferential deposition of the less noble metal, Fe, to the more noble metal, Ni. In other words, the reduction of Ni is inhibited while the deposition of Fe is enhanced when compared with their individual deposition rates [44]. It is reported that Ni-Fe alloy coating is used to decrease the cost production, provide soft magnetic properties,

good electrical conductivity, proper corrosion resistance and special optical properties [44]. Therefore, Fe in the Ni-matrix causes replacement of a component of Ni-matrix composite coating by a cost-effective material. Literature survey also reveals that a variety of particulates such as SiC, TiO_2 , Al_2O_3 and TiC have been added to the electroplating baths to form metal composite coatings [45,46]. Also, the strength and corrosion resistance can be significantly enhanced by introducing second phase ceramic particles into the Ni-Fe matrix [46,47]. Therefore, the results of the present investigation show that the developed Ni-Fe(Ti,W)C coating can full be considered as a cost-effective material with desirable mechanical properties.

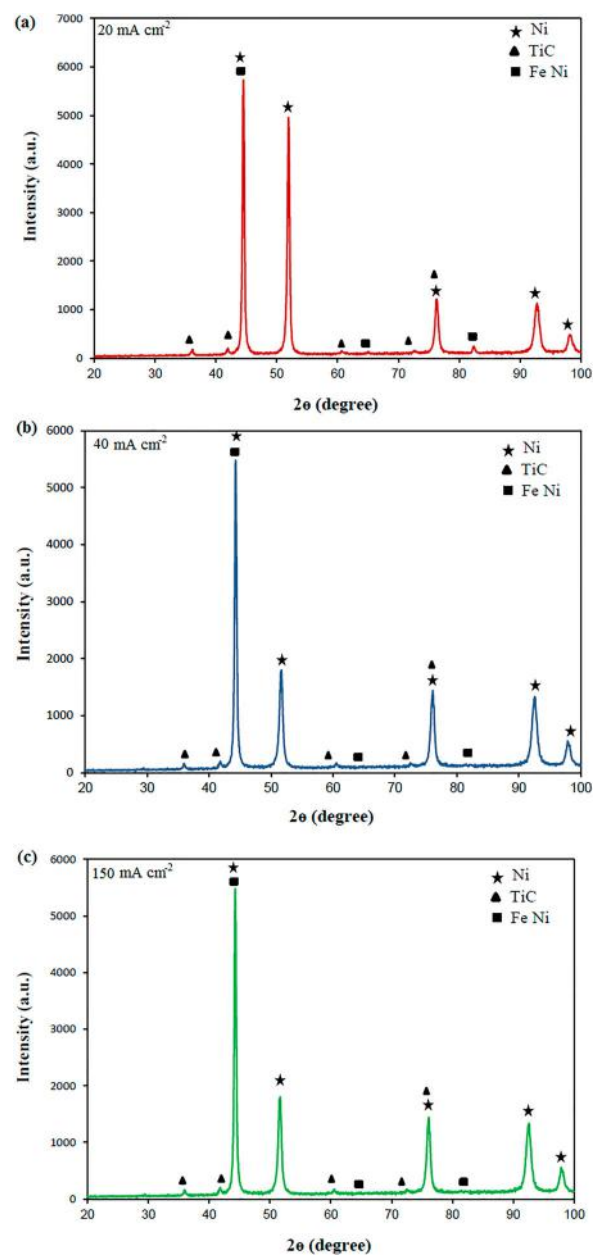


Figure 4. X-ray diffraction pattern of the Ni-Fe(Ti,W)C coating on the steel substrate and with a concentration of 6 g L^{-1} in different current densities of (a) 20 mA cm^{-2} , (b) 40 mA cm^{-2} and (c) 150 mA cm^{-2} .

Table 2. The amounts of microhardness and grain size of the coating in different current densities and different carbide concentrations on the steel substrate.

Current density (mA cm ⁻²)	Carbide concentration (g L ⁻¹)	Microhardness (HV)	Grain size (nm)
150	6	1000	30
40	6	1300	20
20	6	1550	25
40	16	1480	15

Conclusion

Ni-Fe(Ti,W)C nanocomposite coating was prepared using the electroplating technique. The results of SEM imaging indicated that the coating on the steel substrate was smoother and denser compared to that on the brass substrate. The results of microhardness, grain size and morphology examinations confirmed that the current density of 40 mA cm⁻² and the carbide concentration of 6 g L⁻¹ may be considered as the optimised process parameters to obtain the highest microhardness and the least grain size.

Disclosure statement

No potential conflict of interest was reported by the authors.

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