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Electroless Plating of Ni-P-W Coatings Containing Scattered Nb_2O_5 on Sintered NdFeB Substrate

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The ternary Ni-P-W coatings containing scattered Nb_2O_5 particles were deposited on the sintered NdFeB substrates by electroless plating. The effect of experimental processing parameters on the deposition rate was investigated during electroless plating. The coatings performance, such as corrosion resistance, interfacial adhesion strength and micro-hardness, was evaluated by experimental testing. The experimental results indicated that the corrosion resistance of Ni-P-W/Nb_2O_5 composite and the interfacial adhesion strength between Ni-P-W/Nb_2O_5 coatings and NdFeB substrate have been increased with increasing the Nb_2O_5 concentration up to 15gL^{-1}. Increasing Nb_2O_5 concentration over 15gL^{-1} decreases the corrosion resistance of Ni-P-W/Nb_2O_5 composite and the interfacial adhesion strength. Testing of micro-hardness showed that the hardness of Ni-P-W coatings containing scattered Nb_2O_5 composite increases with increase in Nb_2O_5 concentration.

Keywords: *electroless plating, Ni-P-W/Nb_2O_5 composite, NdFeB substrate*

1. Introduction

Electroless plating is an autocatalytic process, which is widely used for the production of uniform films in many industrial applications[1-3]. It has been reported that the Ni-P coatings prepared by electroless plating have some outstanding mechanical and chemical properties, such as good corrosion resistance, wear-resistance and high microhardness etc.[4,5]. On the other hand, the electroless plating has the advantage of its ability to coat interior surfaces of pipes, valves, and other parts. The coatings can be deposited on a variety of materials, such as metals, plastics, glass, and ceramics[6,7]. It has also been reported that the coatings of ternary systems in which a third element has been included into Ni-P coatings, such as Ni-Cu-P, Ni-P-Ag and Ni-B-P systems, possess their comprehensive properties[8-12]. In addition, the Ni-P coatings containing fine dispersed ceramic particles have also attracted a great deal of attention in industrial area[13-16]. Some research results indicated that a combination of dissimilar materials, such as distributed nano-Al_2O_3, SiC, ZrO_2, CeO_2 or Si_3N_4 particles, can produce a coating with good chemical, mechanical, electrical, and optical properties[17-23].

Sintered NdFeB permanent magnet has a substantial drawback in corrosion resistance. The weakness of its poor anti-corrosion property has still limited its working life, although it is widely used in many industrial areas due to its excellent magnetic properties[24,25]. In order to improve its corrosion resistance, the researchers focus their interests mainly on two aspects, namely, adding some alloying elements into the NdFeB magnet during metallurgy or by surface protection technology. However, some investigations indicated that the magnetic performance was decreased simultaneously when the corrosion resistance has been improved by the addition of alloying elements[26]. Therefore, to improve its corrosion resistance through surface protection technology is still an important route to broaden its industrial application. For instance, He Fengjiao and Chen Xing have developed a method for electroplating nickel-tungsten-phosphorus alloy on surface of sintered neodymium-iron-boron magnet[27]. Alia et al.[28] evaluated the anticorrosion characteristics of multilayer titanium nitride ceramic coating deposited on the sintered NdFeB permanent magnets. Song & Yang[25] prepared Ni-P/TiO_2 coating and reported that it has a better corrosion resistance than Ni-P coating. Chen et al.[29] showed a successful multi-layered electroless Ni-P coatings scheme for the protection of sintered NdFeB magnet using multi-layered electroless nickel deposition.

The aim of this work is to prepare Ni-P-W coatings incorporated with scattered Nb_2O_5 particles on NdFeB substrate by electroless plating. The effect of the experimental parameters on the deposition rate during electroless deposition has been investigated experimentally. The coatings' corrosion resistance, the interfacial adhesion strength between Ni-P-W/Nb_2O_5 coatings and NdFeB substrate, and the coatings' micro-hardness have also been evaluated. To the authors' knowledge, this is the first report of Ni-P-W coatings containing scattered Nb_2O_5 on sintered NdFeB substrate. In addition, it is worth noting that there is a disagreement among researchers on the corrosion resistance of electroless coatings. Some studies have revealed that the incorporated particles improve the corrosion resistance whereas other studies contradict such an observation[30-32]. Therefore, more investigations on the performance of composite coatings are a warranty to clarify the situation and to gain an understanding of the underlying mechanism.

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2. Experimental Methods

2.1. Materials and pretreatment

The specimens of sintered NdFeB permanent magnet in the form of 20×10×10 mm were used as the substrate for electroless plating. Its nominally composition was as follows (mass percent): 26.7% Nd; 72.3% Fe and 0.99% B. The Nb_2O_5 powders were provided by Ningxia Orient Tantalum Industry Corporation Limited (China). Its size distribution is from 400 nm to 600 nm. The NdFeB substrate was sequentially polished using silicon carbide papers with different grit before electroless plating. The specimens were degreased in an alkaline solution for 2 minutes after polishing. The alkaline solution was composed of NaOH, Na_2CO_3, and Na_3PO_4•$12H_2O$. Its concentration is 10, 50 and 70 gL^{-1} respectively. The NdFeB specimens were then immersed into deionized water and cleaned in ultrasonic bath for 2 minutes. Finally, the substrate was activated by immersion in a solution containing 25 gL^{-1} $C_7H_6O_6S$•$2H_2O$ and 15 gL^{-1} NH_4HF_2 at 25 °C for 1 minute.

2.2. Preparation of Ni-P-W/Nb_2O_5 coatings

The composition of the electroless plating bath for preparing Ni-P-W/Nb_2O_5 composite coatings is given in Table 1, which has been modified according to references[33-35]. The pH level of the bath was adjusted with NH_4OH to 9. The autocatalytic reaction temperature during electroless plating was maintained at 85 °C. And the deposition time was set as 2 h. The concentration of Nb_2O_5 ceramic particles ranged from 5 gL^{-1} to 20 gL^{-1}. The electroless plating bath was stirred by an adjustable speed motor equipped with a stirring rod and the stirring rate was set as 150 r.p.m. By using the data of deposition time and weight gaining of substrate, the deposition rate was calculated at different plating conditions.

2.3. Characterization

The surface morphologies of composite coatings deposited on the NdFeB substrate were evaluated by a scanning electron microscope (SEM, Model JSM-5610LV, Japan), which has been equipped with GENESIS energy dispersive X-ray spectrometer (EDX). And the EDX was used to determine chemical composition of the coatings. The measurements were carried out at 20 keV accelerating voltage. The composite coatings were also analyzed using X-ray diffraction (XRD, Model D/MAX2200PC, Japan) to identify phases existed in the film. Using a HX-1000TM hardness tester, the coatings Vickers micro-hardness was measured with a load of 0.1 kg for 15 s. The average of diagonal lengths of a square indentation produced by diamond pressing was taken and recorded. In order to obtain the average value of micro-hardness, three indentations were done for each sample.

The coatings corrosion resistance was evaluated by an electrochemical workstation (Model CHI660A, China) which equipped with a conventional three-electrode cell. The coated specimen was used as working electrodes during electrochemical testing. Here, the working electrode is in the form of φ 5 mm×15 mm, and one of the top of the cylinder was exposed in the solutions. The other surface of the coated substrate was enclosed by epoxy resin. A large Pt foil was used as a counter electrode, and Ag/AgCl electrode was chosen as the reference electrode. In order to reduce the possible contamination of chloride ions, the electrolyte was updated for each sample. On the other hand, although some of these tests were done in chloride medium to simulate the seawater medium, the measurements were conducted in 0.5 $molL^{-1}$ H_2SO_4 solutions which can postpone the passivation occurred on an anode. The test temperature was maintained at 25 °C and the polarization curves were recorded with 0.01Vs^{-1} potential scan rate.

The adhesion strength between Ni-P-W/Nb_2O_5 coatings and NdFeB substrate was evaluated by an auto-scratch testing system (Model WS-2005, China). When the coating has been broken away from the substrate, the acoustic emission signal would be detected, and the corresponding load was named as the interfacial adhesion strength between Ni-P-W/Nb_2O_5 coatings and NdFeB substrate. Two times adhesion test was performed for each sample. However, the interfacial adhesion strength of the two times adhesion test for each sample is almost similar. Therefore, one of them was selected to compare with the test of other samples. The thickness of all composite coatings used in this study ranged from 15 to 22 μm.

Table 1. Bath composition used for Ni-P-W/Nb_2O_5 preparation.

Chemicals	Concentration [gL^{-1}]
$NiSO_4$•$6H_2O$	35
Na_2WO_4•$2H_2O$	35
NaH_2PO_2•H_2O	30
$Na_3C_6H_5O_7$•$2H_2O$	62.5
$(NH_4)_2SO_4$	52.5
CH_4N_2S	5×10^{-3}
$C_{12}H_{25}NaO_3S$	5×10^{-3}
Nb_2O_5	5;10;15;20

3. Results and Discussion

3.1. Effect of experimental parameters on deposition rate

Figure 1 shows the effect of experimental processing parameters on deposition rate during electroless plating. The deposition rate was measured as a function of the pH, the bath temperature, the stirring rate and the reducing agent concentration respectively. All electroless plating experiments were conducted based on the bath composition given in Table 1. The experimental points were connected by a mathematical fitting of spline interpolation. In consideration of the possible errors or experimental deviations caused by measurements, the 5% error bars were added on all curves. In order to maintain the pH of the solution at a proper level, to avoid the poisonous effect, and to neutralize acid during electroless deposition, the NH_4OH was added into the solution to replace evaporated ammonia and adjust the pH of the bath during plating. From Figure 1a, it can be seen that with the pH level increasing from 8 to 9, the deposition rate of Ni-P-W/Nb_2O_5 coatings increased correspondingly. However, with the pH

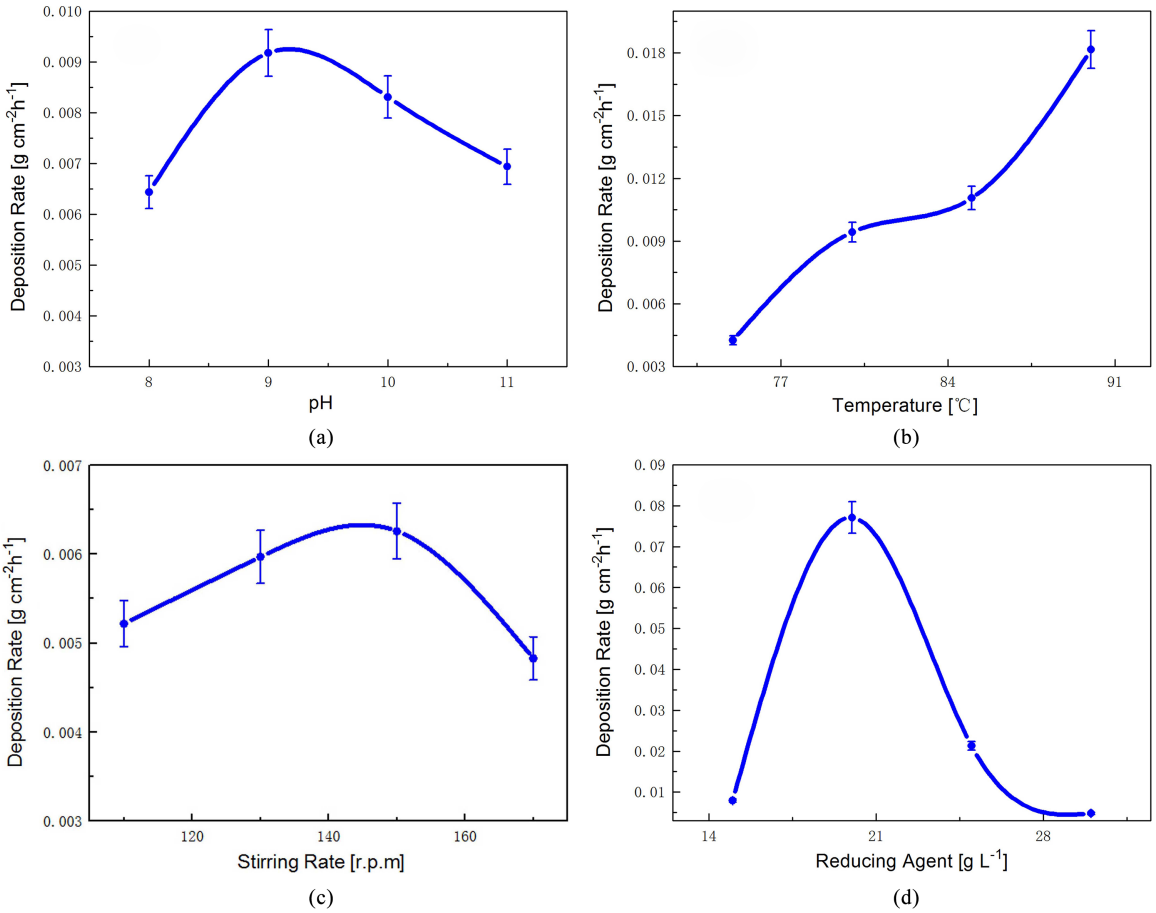

Figure 1. Effect of experimental parameters on deposition rate (a) pH, (b) temperature, (c) stirring rate, (d) concentration of reducing agent.

level increasing from 9 to 11, the deposition rate decreases inversely. It can be explained that the metal ions decrease rapidly with the pH of the bath increasing, resulting in the reduction product atoms decreasing. Thus, the deposition rate decreases correspondingly.

Based on the experimental parameters described in section 2.2, the bath temperature was scheduled in the range of 75-90 °C to evaluate the effect of bath temperature on the deposition rate. While the bath temperature rises from 75 °C to 90 °C, the deposition rate increases correspondingly (Figure 1b). This can be explained that higher temperature speeds up the chemical reaction rate during electroless plating, resulting in the deposition rate increasing.

In order to circulate the hydrogen produced during deposition and to maintain the solution in a uniform state, the solution was physically stirred by an adjustable speed motor. Figure 1c shows the correlation between the deposition rate and the stirring speed during electroless plating. It can be seen that the deposition rate increases obviously while the stirring rate increases from 110-150 r.p.m. As the stirring rate rises from 150-170 r.p.m, the deposition rate decreases inversely. This phenomenon may be explained that the kinetic energy of the metal atoms and the particles was increased exceedingly by a much higher stirring speed, which affected the process of electroless deposition. Correspondingly, the deposition rate has been decreased.

Electroless plating can be carried out via the redox reaction of an oxidizer and a reductant in an electrolyte solution. Sodium hypophosphite ($NaH_2PO_2 \bullet H_2O$) is one of the reducing agents used in the chemical reduction of nickel from aqueous solution. In this study, the concentration of sodium hypophosphite was changed from 15 to 30 gL^{-1}. It can be seen that the deposition rate increases while the reducing agent increases from 15 to 20 gL^{-1}. Increasing the reducing agent concentration from 20 to 30 gL^{-1} decreases the deposition rate (Figure 1d). Due to the fact that the content of hydrogen produced during deposition increases suddenly when the concentration of the reducing agent increases over 20 gL^{-1}, the redox reaction rate has been restrained by the hydrogen bubbles accumulated on the deposition sites, resulting in the deposition rate decreasing.

3.2. Morphology and EDX of Ni-P-W/Nb_2O_5 coatings

Figure 2a and Figure 2b show the morphology (SEM) of Ni-P-W coatings containing scattered Nb_2O_5 particles on sintered NdFeB substrate. The Nb_2O_5 concentration contained in the bath is 20 gL^{-1}. It can be seen that the

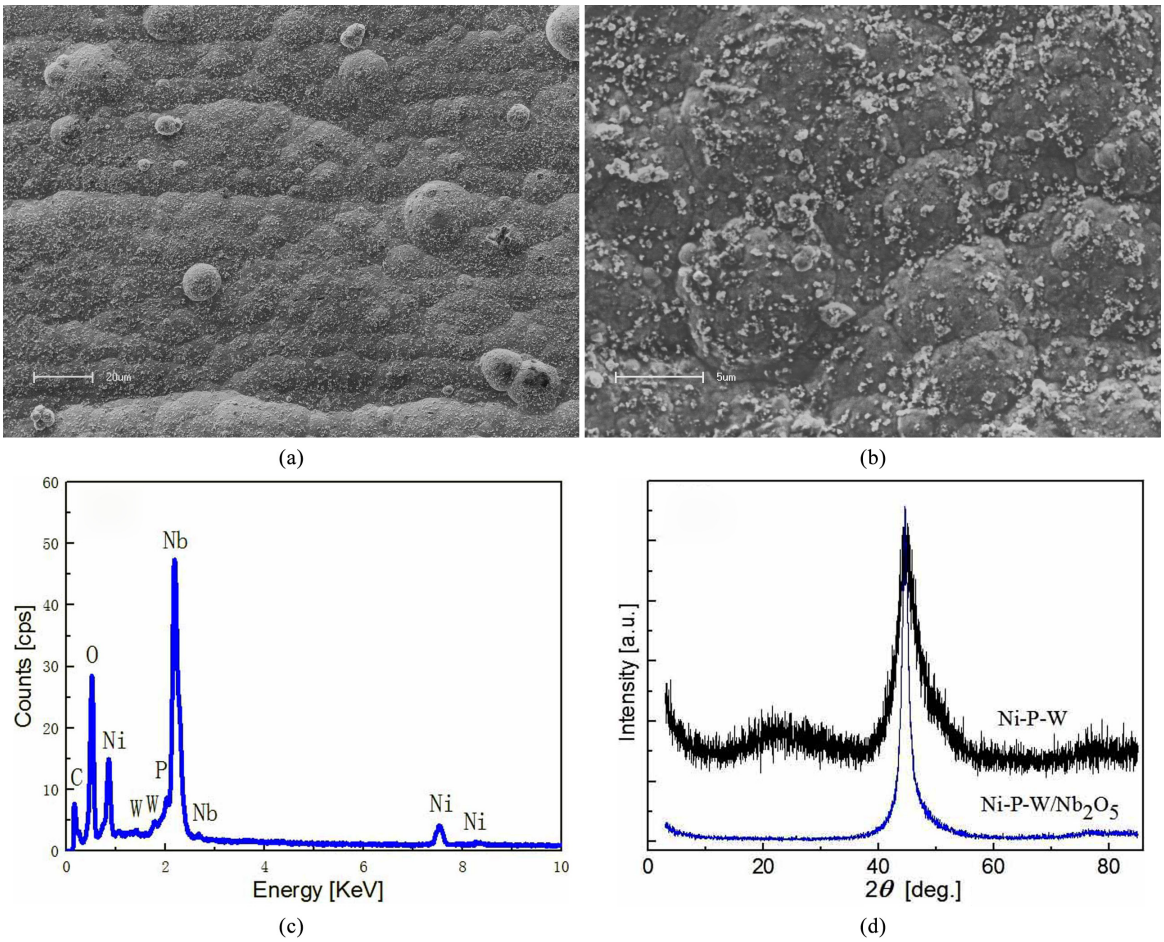

Figure 2. Morphology (a)×500, (b)×3000, EDX pattern (c) and XRD patterns of the coatings (d).

dissimilar Nb_2O_5 particles distributed in the Ni-P-W alloy dispersively, formed a protective coatings on the surface of NdFeB permanent magnet. In order to determine the chemical elements contained in the coatings deposited on the substrate, the EDX analysis was conducted by energy dispersive X-ray spectrometer. As shown in Figure 2c, there are Ni, P, W, Nb and O elements in prepared Ni-P-W/Nb_2O_5 composite coatings except the inclusions of C. Due to the detected site for EDX was located on the Nb_2O_5 ceramic particles, the peaks corresponding to Nb and O elements are more intensive than the others. The neodymium, iron and boron elements forming the sintered NdFeB substrate are not detected through Ni-P-W/Nb_2O_5 composite coatings. It indicates that the substrate was coated completely by Ni-P-W/Nb_2O_5 composite coatings. An anti-corrosion coating was formed on the surface of NdFeB permanent magnet. The X-ray diffraction patterns of Ni-P-W alloy coating and Ni-P-W/Nb_2O_5 composite coating with which contained 20 g/L Nb_2O_5 during the deposition are shown in Figure 2d. The results illustrates that the diffraction patterns of Ni-P-W alloy coating and Ni-P-W/Nb_2O_5 composite coatings coated on the substrates both exhibit a single broad peak centered 2θ at 44.5° which is corresponded to Ni(111) phase[25]. Therefore, both of the prepared Ni-P-W alloy coating and Ni-P-W/Nb_2O_5 composite coatings are possibly amorphous. In addition, there is no indication of Nb_2O_5 phase in the XRD pattern of Ni-P-W/Nb_2O_5 composite coatings. It may be inferred that the Nb_2O_5 peaks are located below the Ni-P-W peaks due to the low percentage of Nb_2O_5 or the particles have been covered by Ni-P-W alloy.

In order to confirm the roughly results described and concluded above, the chemical elements distribution of Ni-P-W/Nb_2O_5 composite coating is conducted ulteriorly by SEM (Figure 3). Here, the Nb_2O_5 concentration contained in the bath is still 20 gL^{-1}. It can be seen that Ni, P, W, Nb and O elements are distributed on the prepared coating. The Nd, Fe and B chemical elements can not be detected. Therefore, the formed Ni-P-W/Nb_2O_5 composite coating covered the surface of NdFeB permanent magnet completely.

3.3. Testing of interfacial adhesion strength

The testing results (Figure 4a) indicate that the acoustic emission signal is a horizontal line during the elasto-plastic deformation stage. With the load increasing, the acoustic emission noise has been detected gradually, implying the coatings began to separate from substrate. Figure 4b gives the relationship of adhesion strength, which is expressed in critical stress, versus Nb_2O_5 concentration in the bath.

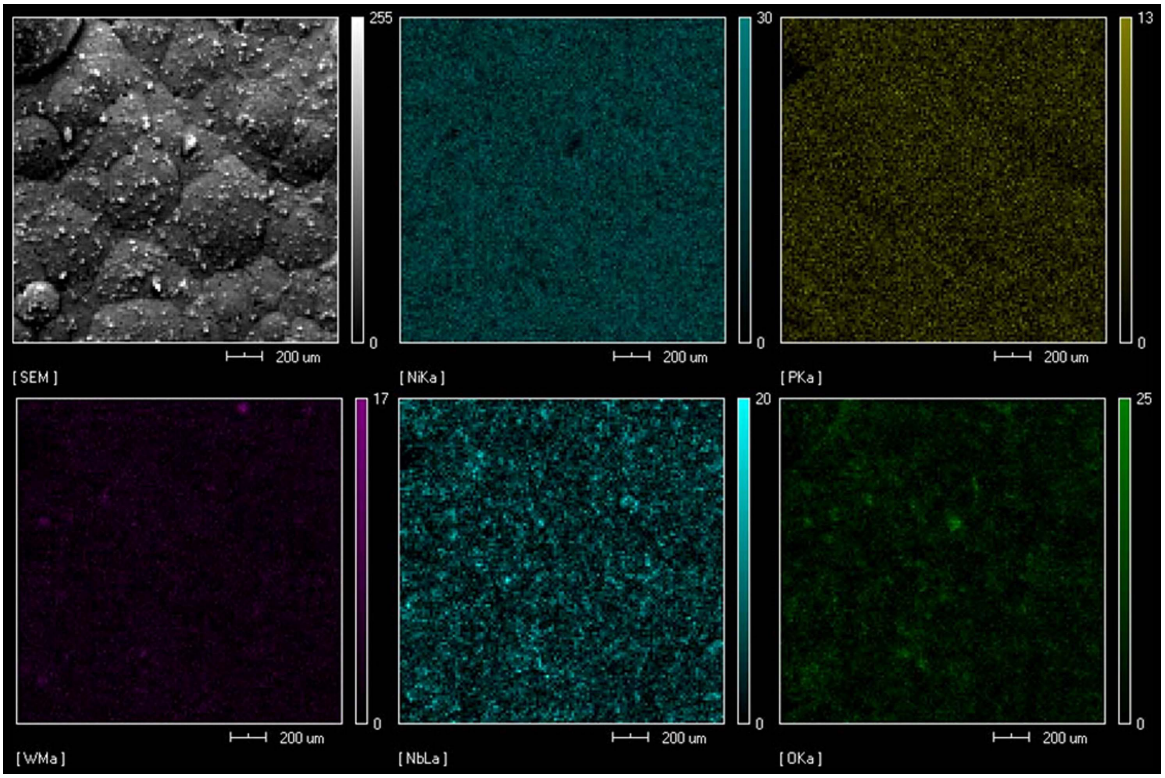

Figure 3. Chemical elements distribution of Ni-P-W/Nb_2O_5 composites (SE).

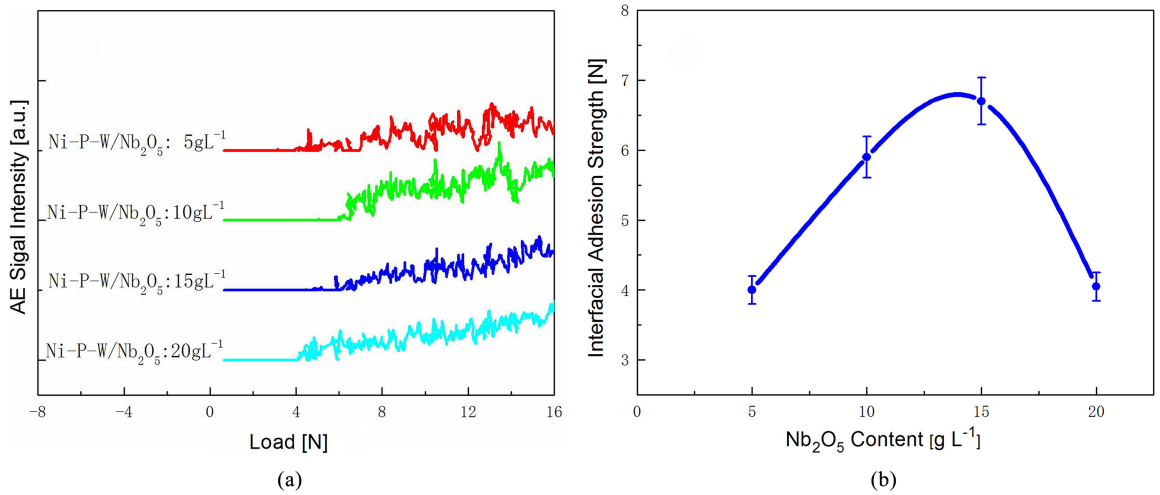

Figure 4. Acoustic emission signal (a) and the relationship of critical stress *vs* Nb_2O_5 concentration (b).

The adhesion strength has been increased with increase in the Nb_2O_5 concentration up to 15gL^{-1}. Increasing the Nb_2O_5 concentration from 15 to 20 gL^{-1} decreases the adhesion strength inversely (Figure 4b). The adhesion strength depends mainly on the properties of involved coatings, substrate and the interface bonding mode. With the Nb_2O_5 concentration increasing, the plasticity of the composite coating was decreased and its brittleness was increased simultaneously, resulting in the adhesion strength between coatings and substrate decreased.

3.4. Corrosion resistance and hardness of Ni-P-W/ Nb_2O_5 coatings

Corrosion involves the destructive attack of metal by chemical or electrochemical reaction with its environment. Usually corrosion consists of a set of redox reactions that are electrochemically in nature. The metal is oxidized to corrosion products at anodic sites: $M \Leftrightarrow M^{+2} + 2e^-$, and hydrogen is reduced at the cathodic sites: $2H^+ + 2e^- \Leftrightarrow H_2$. Theoretically, the current potential relationship can be expressed

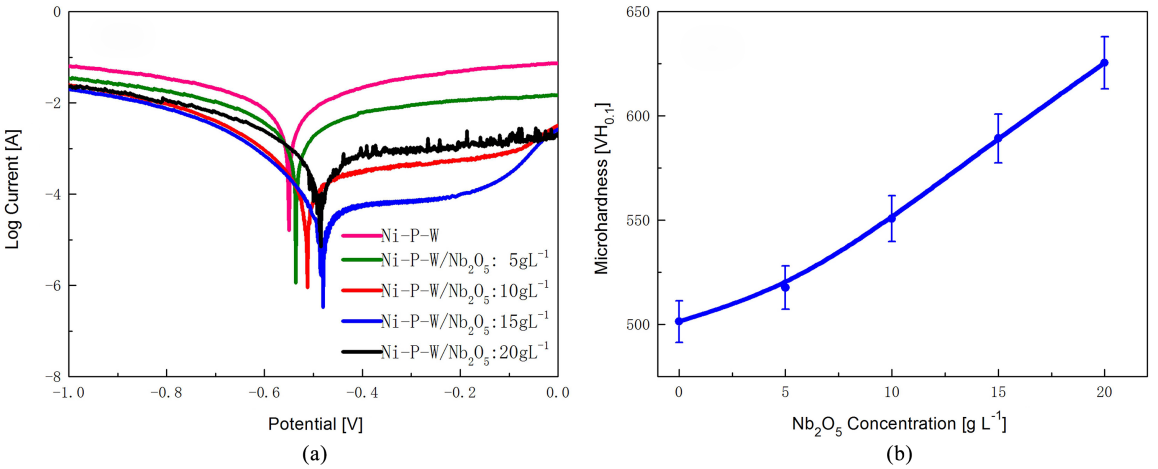

Figure 5. Effect of Nb_2O_5 concentration on corrosion resistance (a) and micro-hardness (b).

as[36]: $E = E_0 + b\log(i/i_0)$. Where E denotes the potential at current i, E_0 stands for the potential at current i_0, b is Tafel slope ("+" for anodic reaction, "-" for cathodic reaction).

The experimental results (Figure 5a) indicate that the corrosion potential and the corrosion current of Ni-P-W/Nb_2O_5 coatings varied with the Nb_2O_5 concentration changing. Increasing the Nb_2O_5 concentration from 5 to 15 gL^{-1}, the corrosion potential changed from -0.536 V to -0.480 V and the corrosion current, which was expressed in log(current), varied from -5.941 to -6.476, resulting in the polarization resistance decrease. When the Nb_2O_5 concentration increases up to 20 gL^{-1}, the corrosion potential moved to the more negative side inversely. Apparently, the working electrodes of Ni-P-W/Nb_2O_5 coatings with the concentration of scattered Nb_2O_5 ranged from 5 to 15 gL^{-1} have more positive potential in comparison with that of the coatings in which the Nb_2O_5 concentration is 20 gL^{-1}. Therefore, the composite coating is more thermodynamically steady as the concentration of scattered Nb_2O_5 ranged from 5 to 15 gL^{-1}. In addition, it is worth noting that the current increases when the Nb_2O_5 concentration is greater than 15 gL^{-1}. It may be caused by the increasing of the weak sites, such as the phase boundary and other defects formed during deposition. The variation in micro-hardness of Ni-P-W/Nb_2O_5 coatings versus the Nb_2O_5 concentration was showed in Figure 5b. The testing results showed that the micro-hardness of Ni-P-W coatings containing scattered Nb_2O_5 composite increases with increase in Nb_2O_5 concentration. Compared with Ni-P-W coatings (without Nb_2O_5 addition), the corrosion resistance (Figure 5a) and the micro-hardness (Figure 5b) were improved by the addition of Nb_2O_5 particles obviously.

4. Summary

An electroless nickel plating technique was used to prepare the Ni-P-W/Nb_2O_5 composite coatings on the sintered NdFeB substrate. The effect of experimental parameters on the deposition rate and the coatings performance, including corrosion resistance, interfacial adhesion strength and hardness, were investigated. Our experiments have shown that the corrosion resistance of electroless coatings can be increased when the Ni-P-W alloy has been incorporated with some moderate particles. Similarly, the adhesion strength between composite coatings and substrate can be also reinforced by a moderate addition of Nb_2O_5 particles. The reason that made the interfacial bonding strength decreasing is the brittleness of the composite coatings increasing while the Nb_2O_5 concentration has been increased over 15 gL^{-1}. Comparatively, the Vickers hardness of Ni-P-W/Nb_2O_5 coatings has been increased with the Nb_2O_5 concentration increasing monotonically.

Acknowledgements

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