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Study on microstructure and properties of electrodeposited Ni–W alloy coating with glycolic acid system

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ABSTRACT

In this paper, Ni–W alloy coatings were prepared by electrodeposition technique with glycolic acid as a complexing agent for the first time. The microstructure and properties of the coatings were characterized and analyzed by employing X-ray diffractometer (XRD), scanning electron microscope (SEM), and X-ray fluorescence spectrometry (XRF). The results show that for the Ni–W alloy coatings with glycolic acid as complexing agent, the crystal grains are fine and compact and the surface is smooth. Compared to the coatings with conventional complexing agent of citric acid, the wear lost rate and corrosion rate decrease by 45% and 35%, respectively, while the current efficiency increases by 60%.

1. Introduction

The electrodeposited Ni–W alloys can be applied in axletree, cylinder, and high-temperature glassy mould [1–3] owing to their intense hardness [4], good corrosion resistance [5], and wear resistance [6]. At present, the complexing agent used extensively for Ni–W electrodeposition is citric acid [7,8]. High W-content in coating can be obtained by this complexing agent; however, the high W-content coatings show poor compactness and a low current efficiency during deposition process [9,10]. Improvement of current efficiency and compactness of coatings has attracted great attention now [11–13]. Our recent study has revealed that the Ni–W coatings using glycolic acid as a complexing agent was denser and smoother and better properties could be expected in the future. Therefore, glycolic acid may be a potential candidate to replace citric acid as complexing agent for Ni–W electrodeposition. But, correlative research has not been reported as yet. Keeping glycolic acid as a complexing agent, a certain direct current was employed to prepare Ni–W coatings, afterwards microstructure, corrosion resistance, wear resistance, and current efficiency were studied. The experimental results provide reference to prepare Ni–W alloy with depositing high efficiency and excellent properties.

2. Experimental

The 45# steel with a demension of 40 mm × 25 mm × 3 mm was used as experimental material. The samples were polished, degreased and activated. Afterwards, Ni–W coatings with 60 μm thickness were prepared by direct current electrodeposition using DF1720SL10A power supply. The chemical composition of plating solution and operating parameters are as followings: $NiSO_4{\cdot}6H_2O$ (12 g/l), $Na_2WO_4{\cdot}2H_2O$ (24–60 g/l), $C_2H_4O_3$ (40–80 g/l), appropriate glycolic acid or citric acid, temperature (55–75 °C), pH (5.5–7.5), and current density (1.2–2.4 A/dm^2). All the chemicals are in analytical grade (A.R.) and dissolved with distilled water. The formula

$$\eta = \eta_W + \eta_{Ni} = \frac{W\% \cdot \Delta G/E_W}{I \cdot t} + \frac{Ni\% \cdot \Delta G/E_{Ni}}{I \cdot t}$$

was used to calculate current efficiency, wherein, I, t, E_W, and E_{Ni}, ΔG, η_W and η_{Ni} represent corresponding current density, time, electrochemical equivalent of W and Ni (0.304 mg/C and 0.318 mg/C), weight gains (mg), and partial current efficiency for W and Ni, respectively.

The contents of Ni and W in coatings were analyzed by ZSX Primus II-type X-ray fluorescence spectrometer. The microhardness of the composite coating was measured in a model MHV-2000 microhardness tester at a load of 0.49 N. The wear-resistance performance was examined under dry sliding condition in air at 25 °C by a ball-on-disk tribometer. GCr15 steel balls with 3 mm diameter were used as a static counterpart in the wear-resistant tests. The rotating velocity, the radius, the load, and the time were 1000 rpm, 5 mm, 3.92 N, and 10 min, respectively. The wearing mass loss was measured with a model CP225D balance (sensitivity 0.01 mg) and the wear resistance was expressed by the mass loss in unit distance. The anodic polarization curves were measured by CHI660B electrochemical testing apparatus with the coatings dipped into 10% H_2SO_4 solution at 25 °C. The saturated calomel electrode was used as a reference electrode and Pt sheet as counter electrode for the testing system, and the scanning rate was 0.5 mV/s. The corrosion rate of coatings in 10% H_2SO_4 solution was also measured. The hardness, mass loss, and corrosion rate were all measured for three times and the average value was taken. The phase structure, morphology, and wear resistance of coatings were observed and analyzed with D/max-3C-type X-ray diffractometer and S-570-type scanning electron microscope, and then compared with those of Ni–W coatings obtained using citric acid as the complexing agent.

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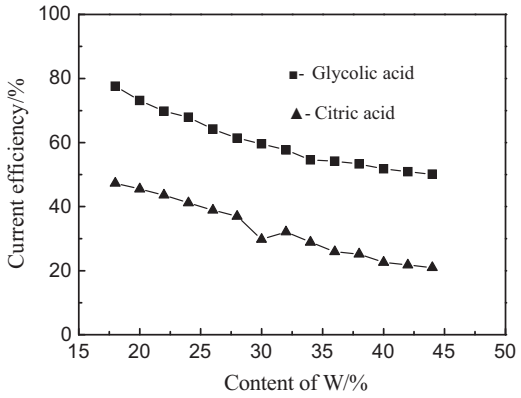

Fig. 1. Relationship between the current efficiency and the W-content in the coatings during electrodeposition process.

3. Results and discussion

3.1. Current efficiency

Fig. 1 shows the current efficiency during electrodeposition using two different complexing agents. It can be seen that the current efficiency decreases with increasing W-content of the coatings prepared by two different systems. However, the current efficiency of coatings by glycolic acid as complexing agent is approximately 60% higher than that of the coatings with citric acid. This may be ascribed to the fact that glycolic acid is a weaker complexing agent than citric acid, resulting in easier deposit of metallic ions on cathodes. In addition, the glycolic acid possesses simple structure and short molecule chains, forming a complex with small volume, which benefits electricity transportation for the complex, thus improving the current efficiency upon electrodeposition [14].

3.2. Morphology of coatings and microstructure analysis

Fig. 2 shows the morphologies of Ni–W coatings with different W-content prepared by two different complexing agents. It can be seen that the surface of the coatings with low W-content is rather rougher and appear some globular nodules. These nodules diminish with increase of the W-content, and then smoother surface appears. As compared, a conclusion could be made that the glycolic acid as complexing agent is beneficial for forming Ni–W coatings with finer grains and smoother surface.

It can be seen from XRD patterns (Fig. 3) that the coatings obtained from two different systems have the same phase structure and the diffraction peaks show similar change trends. As the W-content is low, the coatings exhibit single face center cubic (fcc) structure, attributed to fcc Ni. The corresponding diffracted peaks could be indexed as the planes of (1 1 1), (2 0 0), (2 2 0), and (3 1 1) of Ni alloy, respectively. It can be seen also that the diffracted peaks tend to be broader and shift to larger angles with increasing the W content in the coatings. In this case, the average grain size could be calculated using the Scherrer equation, as shown in Fig. 4. It can be seen that the average grains size of Ni–W alloy coatings decreases with increasing W-content in the two types of coatings. Moreover, it should be noted that as the W content is the same, the grain size in Ni–W coating from the solution with glycolic acid are much smaller than those with citric acid. This should be studied further. On the other hand, as the W-contents of coatings prepared from the solutions with citric acid and glycolic acid are about 45% and 47%, respectively, only a single broad peak could be observed at the 2θ angle in the range of 40–50°, appearing an amorphous structures.

3.3. Properties of coatings

3.3.1. Wear resistance

Figs. 5 and 6 show the relationship between the mass loss and friction coefficients and the W-content of coatings obtained with two different complexing agents. It can be seen that the mass loss and friction coefficients of coatings decrease with increasing W-content. Compared with the coatings with citric acid, those with glycolic acid exhibit smaller mass loss and lower friction coefficients. The mass loss of coatings with glycolic acid is lower by about 45% than that with citric acid. Fig. 7 shows the worn morphologies of Ni–W coatings obtained from the solution with glycolic acid (33 wt% W) and citric acid (35 wt% W) as complexing agent. It can be seen that there exists some light furrows on the worn surface

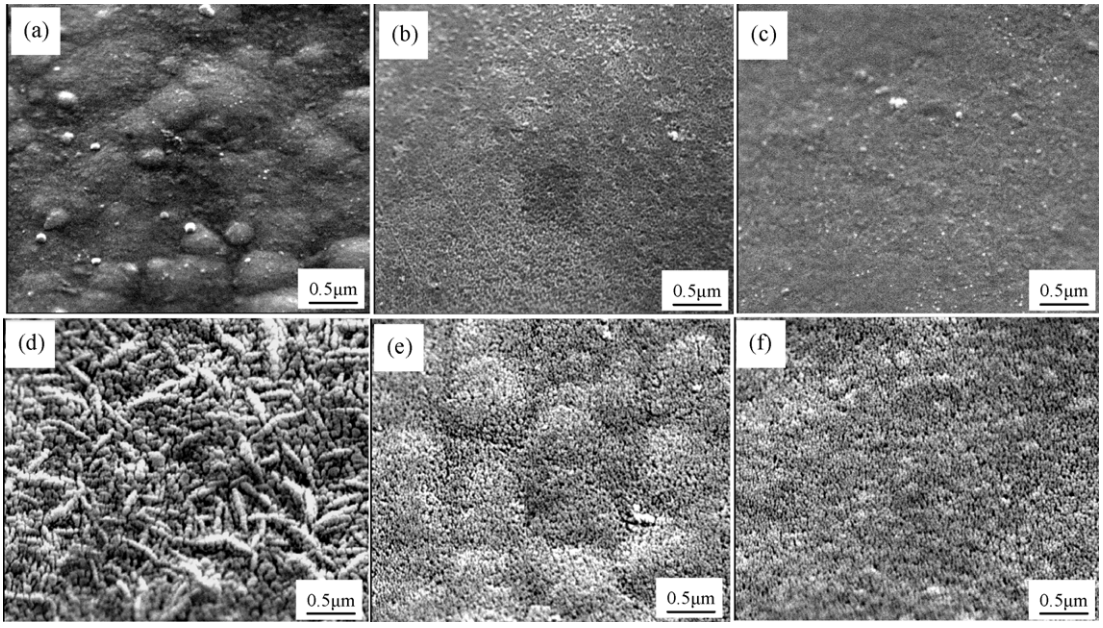

Fig. 2. Surface morphologies of Ni–W coatings with different W-contents.