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A comparative study of the effects of saccharin and β -SiC nano-particles on the properties of Ni and Ni–Co alloy coatings

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ABSTRACT

The effects of saccharin and SiC nano-particles on the properties of Ni and Ni–Co based coatings electrodeposited in modified Watt's baths by sediment co-deposition (SCD) technique were studied. Surface morphologies of Ni and Ni–Co coatings were changed by using saccharin and SiC nano-particles. X-ray diffraction (XRD) results revealed that peak positions and the phase structure of the studied coatings did not change appreciably while the crystallite size and preferred orientation were significantly varied by using SiC nano-particles and particularly saccharin. Although the incorporation of SiC nano-particles had a slight influence on the microhardness of Ni based coatings, using saccharin enhanced their microhardness. However, there was no noticeable difference between the microhardness of Ni–45Co and Ni–45Co/saccharin coatings (containing 45 wt.% Co) while the microhardness of Ni–55Co/SiC nanocomposite coatings (containing 55 wt.% Co) was higher than Ni–45Co and Ni–45Co/saccharin coatings. The potentiodynamic polarization measurements indicated that the corrosion resistance of Ni/saccharin and Ni–45Co/saccharin coatings was better than pure Ni and Ni–45Co coatings. Additionally, the corrosion properties of Ni/SiC and Ni–55Co/SiC nanocomposite coatings were markedly higher than other coatings. Ni–55Co/SiC nanocomposite coatings showed the highest microhardness and corrosion resistance compared to all studied coatings. The unique properties of Ni–55Co/SiC nanocomposite coatings were linked to their alloy matrix and the amount of SiC nano-particles (8.1 vol.%).

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1. Introduction

There is an increasing demand for the production of high performance electrodeposited nickel coatings in different technological applications. During the last decades, several different approaches have been successfully used to enhance the properties of such coatings. The main approaches can be classified as: (i) structural refinement through the use of additive agents [1–5], (ii) electrodeposition of nickel based alloy coatings [6–15], and (iii) electrodeposition of nickel based micro- and nanocomposite coatings containing different particles [16–25].

Additives are mainly categorized in four groups: (i) grain refiners, (ii) dendrite and roughness inhibitors, which are adsorbed on the surface and prevent the growth of dendrite precursors, (iii) leveling agents, which enhance the throwing power of an electrolyte, and (iv) wetting agents or surfactants, which prevent pits and pores from forming in deposits [5]. There are proven results in favor of using organic additives such as saccharin to improve the properties of pure Ni and Ni–Co coatings. The internal stress of coatings is reduced, the crystallite size is refined markedly, the texture and morphology are changed, the surface roughness is reduced, and the brightness and macroscopic quality of coatings are improved by using saccharin [26–28]. Among the eight

decomposition products of saccharin found in different solutions, o-toluene sulfonamide and benzamide were identified in the Watt's bath when saccharin was added to the electrolyte [5]. Therefore, the existence of a certain amount of sulfur and carbon impurities, which is resulted from saccharin in coatings, leads to segregating these impurities along grain boundaries and affecting the properties of coatings. Although these elements play efficient roles in hindering the grain boundaries from sliding, they decrease the ductility of the coatings because sulfur is a critical impurity causing embrittlement.

It is demonstrated that simultaneous codeposition of nickel and cobalt cations produces Ni–Co alloy coatings with better properties in comparison with Ni coatings [6–8]. It was shown that the microstructure of Ni–Co coatings was changed from pyramidal to spherical, and then to a branched structure by increasing the cobalt content in the coatings [6]. It should be also mentioned that the electrodeposition of Ni–Co alloy coatings is classified as an anomalous codeposition in which a less noble metal, cobalt, is deposited preferentially compared to a noble one, nickel [29]. Therefore, the relative content of cobalt in the Ni–Co coatings is higher than that in the electrolyte [8,30,31]. It was illustrated in a previous work that the cobalt content in the Ni–Co alloy coatings was considerably higher than the ratio of $\text{Co}^{2+}/(\text{Co}^{2+} + \text{Ni}^{2+})$ in the electrolyte as the cobalt content was increased from 0 to 45 wt.% between 0 and 16 $\text{Co}^{2+}/(\text{Co}^{2+} + \text{Ni}^{2+})$ in the electrolyte [8]. Furthermore, the microhardness of Ni–Co alloy coatings was

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enhanced markedly by increasing the cobalt content, and after reaching a peak, it was dropped (Co-rich alloys) [8]. Although investigations illustrated that the corrosion resistance of Ni–Co coatings was enhanced and then decreased by increasing the cobalt content [8], the wear resistance of the coatings was improved continuously by increasing the cobalt content [6]. On the other hand, composite coatings have been widely studied over the last decades due to their unique properties. It has been well established that composite coatings containing hard particles show better mechanical properties than common coatings. Surender et al. [16] reported that the presence of WC micro-particles in Ni coatings increased microhardness and wear resistance. Thiemiig et al. [17] stated that Ni/TiO₂ nanocomposite coatings had better microhardness and wear resistance compared to Ni coatings. Pavlatou et al. [18] studied the influence of particle size and deposition current type on the microhardness of Ni/SiC composite coatings and indicated that Ni/SiC nanocomposite coatings electrodeposited by a pulse current had higher microhardness than other composite coatings. Feng et al. [19] reported that Ni/Al₂O₃ nanocomposite coatings electrodeposited by a sediment co-deposition (SCD) technique exhibited better microhardness and friction coefficient compared to the coatings electrodeposited by the conventional electrocodeposition (CECD) technique.

It is confirmed that incorporated particles play a significant part in altering the microstructure and consequently, corrosion rate of composite coatings. In an attempt to reveal the effect of corrosive medium, Ciubotariu et al. [32] reported that the corrosion rate of Ni/Al₂O₃ nanocomposite coatings obtained in a 0.5 M sodium chloride solution was a little higher than those obtained in a 0.5 M potassium sulfate solution. Lekka et al. [33] investigated the corrosion properties of Ni/SiC nanocomposite coatings. Results obtained from salt spray tests indicated that the Ni/SiC nanocomposite coatings had lower corrosion rates in comparison with Ni coatings. Medeliene [34] reported that Ni/SiC composite coatings showed considerable corrosion resistance than Ni/B₄C composite coatings because B₄C particles are conductive materials while SiC particles are not conductive materials. Hu et al. [35] enhanced the corrosion resistance of Ni/SiC nanocomposite coatings by using magnetic fields due to an increase in SiC particle incorporation.

There are many electrodeposited composite coatings containing different types of particles with a variety of sizes which almost have better properties than common coatings [36]; however, nanocomposite coatings have shown surprising behaviors [18,37]. Although the above approaches have been employed to improve the different properties of electrodeposited Ni and Ni–Co coatings, there are few works to compare the influences of saccharin and SiC nano-particles, and to reveal which one is more effective and economical. This study is aimed to investigate the effects of saccharin and SiC nano-particles on the mechanical and corrosion properties of Ni and Ni–Co coatings. The SiC nano-particles were chosen as a reinforcing phase because among various particle types, SiC nano-particles have been considerably investigated in order to obtain enhanced microhardness, at the same time corrosion and wear resistance [37–39]. In addition, the electrodeposition process was performed by using the sediment co-deposition (SCD) technique since the SCD technique is an effective method to promote particle incorporation and consequently, the properties of composite coatings [37]. Since electrodes are positioned horizontally in the SCD technique, not only electrophoretic and hydrodynamic forces but also the gravity force help in the incorporation of particles.

2. Experimental

The electrodeposition of Ni and Ni–Co based coatings was carried out in modified Watt's baths with two different electrolyte compositions containing: (i) saccharin and (ii) SiC nano-particles by the sediment codeposition (SCD) technique (Table 1). The process of preparing a suitable electrolyte for the electrodeposition of nanocomposite coatings containing SiC nano-particles, which had an average size of 20 nm

manufactured by Plasmachem GmbH in Germany, was described in the previous work [37].

Copper plates were used as substrates and a pure nickel plate was used as an anode. Prior to the electrodeposition process, the substrates were polished mechanically by silicon carbide abrasive papers. After cleaning in ethanol and acetone consecutively and washing in distilled water, the substrates were activated in 10% H₂SO₄. At the end of the process, in order to remove loosely entrapped particles from the surface of the nanocomposite coatings, the substrates were washed in distilled water ultrasonically.

The phase structure and surface morphologies of the coatings were studied by a D8 Advance-Bruker AXS X-ray diffractometer and a CamScan MV2300 scanning electron microscope (SEM). The average crystallite size of the coatings was calculated using the Scherrer equation from the achieved X-ray patterns. The chemical composition of the studied coatings was determined using an Oxford energy dispersive X-ray detector (EDX) coupled with SEM. In addition, a MDPEL-M400GL microhardness tester, with the operation conditions of 50 g indentation load and 10 s indentation time, was employed to measure the microhardness of the coatings.

Corrosion investigations were performed in the 3.5% NaCl solution without agitation at 25 °C by using an EG&G-Parstat 2263 potentiostat/galvanostat system. The reference electrode was a saturated calomel electrode (SCE), and the counter electrode was a platinum electrode. The exposed area of the counter electrode was approximately 10 times larger than the working electrode. To determine the open circuit potential (E_{ocp}), specimens were immersed about 60 min in the 3.5% NaCl corrosive medium. After attaining the E_{ocp} , the potentiodynamic sweeping was performed in the potential range of 600 mV for pure Ni, Ni/saccharin, Ni–45Co, and Ni–45Co/saccharin coatings and 250 mV for Ni/SiC and Ni–55Co/SiC nanocomposite coatings with respect to E_{ocp} by a 1 mV/s sweeping rate. The corrosion current densities were calculated according to the Stern–Geary equation [40]:

$$i_{corr} = \frac{\beta_a \cdot \beta_c}{2.303 \times R_p (\beta_a + \beta_c)} \quad (1)$$

where i_{corr} is the corrosion current density, R_p is the polarization resistance, β_a is the anodic Tafel slope and β_c is the cathodic Tafel slope. In addition, the polarization resistance was measured based on the following equation:

$$R_p = \left. \frac{dE}{di} \right|_{E=E_{ocp}} \approx \frac{E}{i} \quad (2)$$

3. Results and discussion

3.1. Surface morphology and phase structure

SEM images of the surface morphologies of studied coatings are shown in Fig. 1. Surface morphologies strongly depend on the chemical composition of coatings. The surface morphology of the Ni coating is changed from pyramidal to nodular by alloying with cobalt (Fig. 1a and e). Although both Ni/saccharin and Ni–45Co/saccharin coatings have colony-like morphologies, it can be seen that the size of colonies in the Ni–45Co/saccharin coating is significantly smaller than the Ni/saccharin coating (Fig. 1b and f). Adsorption of saccharin molecules on the active sites of the cathode surface reduces the growth rate of pyramids in Ni based coatings [41].

Since the electric field has its highest strength around the peaks of pyramids on the growing film surface, the current density is much higher at such regions during the electrodeposition process [5]. As a result, cations are reduced preferentially on the top areas of depositing film during the electrodeposition process. Hence, pyramidal surface morphology is a common topography for ordinary Ni coatings which

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