

Electrodeposited nickel–cobalt composite coating containing nano-sized Si_3N_4

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Abstract

Ni–Co/ Si_3N_4 composite coatings with various contents of Si_3N_4 nano-particulates were prepared by electrodeposition in a Ni–Co plating bath containing Si_3N_4 nano-particulates to be co-deposited. The shape and size of the Si_3N_4 nano-particulates were observed and determined on a transmission electron microscope. The polarization behavior of the composite plating bath was examined on a PAR-273A potentiostat/galvanostat device. The friction and wear behaviors of the Ni–Co/ Si_3N_4 composite coatings were evaluated on a ball-on-disk UMT-2MT test rig. The worn surface morphologies of the Ni–Co/ Si_3N_4 composite coatings wear observed using a scanning electron microscope. It was found that the Si_3N_4 nano-particulates appeared as microspheres of a diameter about 20 nm. The cathodic polarization potential of the composite electrolyte increased with increasing Si_3N_4 concentration in the plating bath, and the addition of Si_3N_4 particulates led to changes in the morphologies of the composite coatings. Namely, the Ni–Co alloy coating was composed of needle-like micro-crystallites, while the Ni–Co/ Si_3N_4 composite coatings were characterized by particulate-like structure and had more compact and fine granular morphologies. At the same time, the morphologies of the composite coatings changed from fibril to granular, which implied that the co-deposited Si_3N_4 nano-particulates at a larger content could agglomerate to some extent. The co-deposited Si_3N_4 nano-particulates were uniformly distributed in the Ni–Co matrix and contributed to greatly increasing the microhardness and tribological properties of the Ni–Co alloy. The microhardness of the composite coatings increased and the friction coefficients and wear rates decreased with increasing content of the nano- Si_3N_4 in the composite coatings. This was attributed to the grain fining and dispersive strengthening effects of the co-deposited hard Si_3N_4 nano-particulates. At the same time, the hydroxylated silicon oxide formed by the tribochemical reaction between the nano- Si_3N_4 of high reactivity and water vapor in the air also contributed to decreasing the friction coefficient.

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1. Introduction

Ceramics or nanocomposites of a metallic matrix containing a dispersion of second-phase particles usually have various novel properties, such as dispersion hardening, self-lubrication, high temperature inertness, good wear and corrosion resistance, and chemical and biological compatibility [1–4]. Electrodeposition is one of the most important techniques for producing nanocomposite and nanocrystal coat-

ings, owing to the advantages including precisely controlled near room temperature operation, low energy requirements, rapid deposition rates, capability to handle complex geometries, low cost, and simple scale-up with easily maintained equipment. For the purpose of increasing the hardness and wear-resistance of various electrodeposited metal- and metal alloy-based coatings, especially, Ni-based coatings, various inorganic particulates including SiC [1,5], ZrO_2 [6], Al_2O_3 [7], and TiO_2 [8] have been tried. It has been found that the incorporation of the nano-particulates in metal matrix improves the mechanical and wear-resistance performance of composites to a certain extent.

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Ni–Co alloy, which possess good adhesion, low-stress, corrosion resistance, and be thermally stable with excellent magnetic properties is a popular choice as recording head materials for computer hard drive industries. However, few has been reported on the incorporation of Si_3N_4 nano-particulates in electrodeposited Ni–Co alloy coating, though it has been known that Si_3N_4 nano-particulates as a filler contribute to greatly improve the friction-reducing and antiwear abilities of some polymeric composites, by way of dispersive strengthening effect and tribochemical function in sliding process [9,10].

Thus Ni–Co/ Si_3N_4 nanocomposite coatings are prepared by the electrodeposition in a nickel–cobalt plating bath containing Si_3N_4 nano-particulates in the present work. The surface morphologies and mechanical and tribological properties of the composite coatings are investigated.

2. Experimental

The plating bath is composed of 200 g L^{-1} NiSO_4 , 40 g L^{-1} NiCl_2 , 40 g L^{-1} CoSO_4 , 30 g L^{-1} H_3BO_3 , $1\text{--}10\text{ g L}^{-1}$ Si_3N_4 , and a proper amount of saccharine. Analytical reagents and distilled water were used to prepare the plating solution. Prior to plating, the Si_3N_4 nano-particulates of a mean diameter 20 nm (Kaier, Hefei, China) were dispersed in the electrolyte in the presence of the surfactant. The electroplating tests were performed on a PAR-273A potentiostat/galvanostat device (EG&G Princeton Applied Research). The bath was stirred by a magnetic stirrer with stirring rate of 150 rpm and heated to 45°C . The experiments were conducted at current density of 30 mA cm^{-2} . A platinum plate of $40\text{ mm} \times 40\text{ mm}$ was used as the anode, while a saturated calomel electrode (SCE) was used as the reference electrode. A rectangular copper plate of a size $10\text{ mm} \times 10\text{ mm}$ and a surface roughness less than $0.05\ \mu\text{m}$ was used as the cathode substrate to be plated. The substrates were sequentially ultrasonically cleaned in ethanol, acetone, and distilled water for 10 min, rinsed with 5% H_2SO_4 , washed in distilled water, and then immersed immediately in the plating bath to allow the electrodeposition of the target composite coatings.

The cathodic polarization curves for the electrolyte at a sweep rate of 0.1 mV/s were recorded. The morphologies of the composite coatings were observed on a scanning electron microscope (JEOL JSM-5600LV) and the composition of the nanocomposite coatings was determined using an energy dispersive spectrometer (EDS) coupled with the SEM. The hardness of the composite coatings with a thickness about $20\ \mu\text{m}$ was measured on a Vickers' microhardness instrument at an applied load of 50 g for 5 s. Five measurements were conducted on each sample and the results were averaged. The shape and size of the nano- Si_3N_4 were observed and determined on a transmission electron microscope (TEM, JEM2010).

The tribological behaviors of the electrodeposited coatings reciprocally sliding against SAE52100 steel ball (ϕ

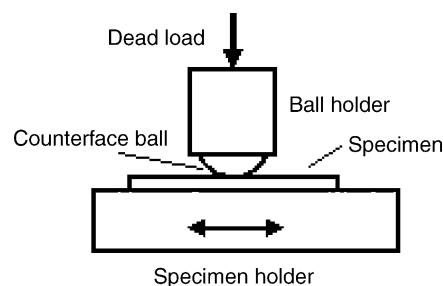


Fig. 1. Schematic diagram of the friction and wear test rig.

3 mm) were examined on a UMT-2MT tribometer (see Fig. 1) in a ball-on-disk configuration. The sliding was performed at an amplitude of 5 mm, a normal load of 1 N, and a frequency of 3 Hz. All the wear tests were performed under unlubricated condition at room temperature and in ambient air (relative humidity 52–56%). The friction coefficient was recorded continuously during the tests.

3. Results and discussion

3.1. TEM observation of Si_3N_4 nano-particulates

Fig. 2 shows the TEM image of the nano-sized amorphous Si_3N_4 particulates (the specific surface area is reported to be $115\text{ m}^2\text{ g}^{-1}$ by the producer). It is seen that they appear as microspheres of a diameter about 20 nm.

3.2. Polarization behavior of Ni–Co/ Si_3N_4 electrolyte

Fig. 3 shows the cathodic polarization behavior of the Ni–Co/ Si_3N_4 electrolyte with different concentrations of Si_3N_4 particulates. It is seen that the addition of Si_3N_4 nano-particulates to the electrolyte causes the reduction potential of Ni–Co to shift towards larger negatives, but the slope of the reduction curve keeps unchanged. The shift to a lower value in the reduction potential is attributed to a decrease in

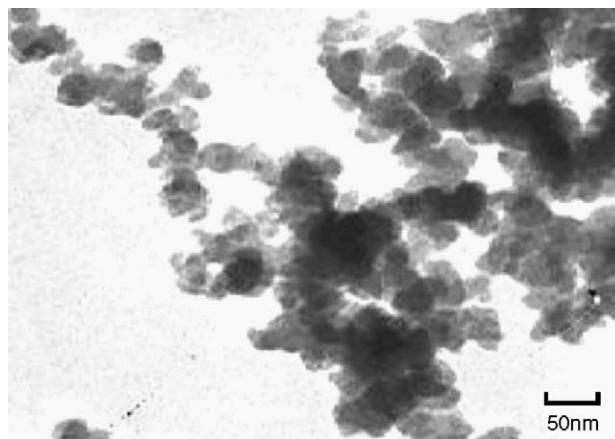


Fig. 2. TEM image of Si_3N_4 nano-particulates.