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Surface & Coatings Technology 200 (2006) 4870 – 4875

SURFACE & COATINGS TECHNOLOGY

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Electrodeposition and characterization of Ni–Co–carbon nanotubes composite coatings

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Received 17 December 2004; accepted in revised form 27 April 2005
Available online 20 June 2005

Abstract

Ni–Co–carbon nanotubes composite coatings were prepared by electrodeposition in a Ni–Co plating bath containing carbon nanotubes (CNTs) to be co-deposited. 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–CNTs composite coatings were evaluated on a UMT-2MT test rig in a ball-on-disk contact mode. The morphologies of the original and worn surfaces of the composite coatings were observed on an atom force microscope (AFM) and scanning electron microscope. It was found that the introduction of the carbon nanotubes in the electrolyte caused the shift towards larger negatives of the reduction potential of the Ni–Co alloy coating, and the co-deposited CNTs had no significant effect on the electrodeposition process of the Ni–Co alloy coating. However, the co-deposited CNTs led to changes in the composition and structure of the composite coatings as well. Namely, the peak width of the Ni–Co solid solution for the composite coating is broader than that of the Ni–Co alloy coating and the composite coating possess higher microhardness and elastic modulus than Ni–Co alloy coating. The co-deposited CNTs were uniformly distributed in the Ni–Co matrix and contributed to greatly increase the microhardness and tribological properties of the Ni–Co alloy coating.

Keywords: Electrodeposition; Ni–Co alloy coating; CNTs; Composite coating; Friction and wear behavior

1. Introduction

The thickness of the magnetic layer used in micro-electrical mechanical system (MEMS) can vary from a few nanometers to a few millimeters, depending on the applications, and the magnetic thin films must have good adhesion and corrosion resistance and low-stress, and be thermally stable with excellent magnetic properties. As a kind of typical magnetic layers for MEMS, electrodeposited Ni–Co alloy coatings have been widely used as recording head materials in computer hard drive industries. Since metal-matrix composite coatings usually have significantly improved mechanical strength, wear resistance and corrosion resistance, and desired chemical and biological compatibilities than the alloy coatings [1–5], it is essential and feasible to improve the comprehensive properties of Ni–Co alloy coatings by introducing third phase reinforcements to generate Ni–Co based composite coatings. This is especially so when carbon nanotubes (CNTs) are selected as the third phase, because carbon nanotubes possess many remarkable properties such as high strength and elastic modulus, good flexibility, and unique conductivity. For example, the Young's modulus of a single-wall nanotube was theoretically estimated to be as high as 5 TPa, and the averaged Young's modulus and bending strength of an isolated multiwalled CNT were measured to be 1.8 TPa and 14.2 GPa. This is why CNTs have been finding tremendous application in the microdevices such as nano diodes, nano transistors, and needle-like tips of atomic imaging machines. The greatly increased wear resistance of hot-pressed Al_2O_3 composite and electroless Ni–P coating by the incorporation of CNTs provides the recent evidences to the successful use of CNTs as a kind of reinforcing agent [6,7].

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doi:10.1016/j.surfcoat.2005.04.037

With those perspectives in mind and with a view 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, of electrodeposition, we anticipate that it would be readily feasible to significantly improve the mechanical and tribological properties of electrodeposited Ni–Co alloy coatings by the inclusion of CNTs as the reinforcing agent. Although CNTs and some kinds of ceramic nano-particulates have been extensively focused on in terms of their effect on the mechanical and tribological properties of electrodeposited and electroless metal-matrix coatings [2,3,7,11], no work has been so far reported on the preparation and properties of electrodeposited Ni–Co–CNTs composite coatings. Thus in the present work, Ni–Co–CNTs nanocomposite coatings were prepared by the electrodeposition in a nickel–cobalt plating bath containing carbon nanotubes. This article deals with the surface morphologies, microstructures, and mechanical and tribological properties of the target composite coatings.

2. Experimental

2.1. Synthesis and purification of CNTs

The CNTs were synthesized by the catalytic decomposition of CH_4 gas in the presence of iron catalyst supported on MgO powder [8,9]. A CH_4/H_2 mixture ($CH_4:H_2=50:1$, V:V) of a pressure 600 Pa was introduced into a quartz chamber heated to 1173 K to allow the generation of the CNTs. The as-prepared CNTs were successively immersed in a concentrated hydrochloric acid and nitric acid, each for 12 h, and cleaned by distilled water to remove the catalyst impurities. After being filtered, the cleaned CNTs were dried at 393 K in an oven. The resultant black powders were heated at 693 K for 1 h in air and refluxed in a dilute nitric acid for 24 h, which gave birth to the target pure CNTs.

2.2. Preparation and characterization of Ni–Co–CNTs composite coatings

Analytical reagents and distilled water were used to prepare the plating solution. The plating baths used to prepare the Ni–Co–CNTs composite coatings were composed of 220 g/l $NiSO_4$, 40 g/l $NiCl_2$, 20 g/l $CoSO_4$, 35 g/l H_3BO_3, 1 g/l carbon tube, and a proper amount of surfactant. The electroplating tests were performed on a 273A potentiostat/galvanostat device (EG and G Princeton Applied Research). The bath was stirred using a magnetic stirrer. The experiments were conducted at 45 °C and current density of 50 mA cm^{-2}. A platinum plate of 40×40 mm^2 was used as the anode, while a saturated calomel electrode (SCE) was used as the reference electrode. A stainless steel plate ($20\times30\times3$ mm) of a surface roughness below 0.3 μm (R_a) was used as the cathode substrate to be plated.

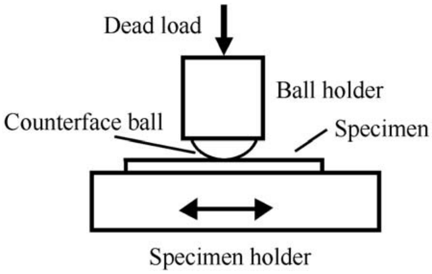

Fig. 1. Contact configuration of the friction pair.

The polarization curves for the electrolyte at a sweep rate of 0.1 mV/s were recorded also on the 273A potentiogalvanostat device controlled by a PC. The surface morphologies of the target composite coatings were observed using an atom force microscope (AFM) which had a sharp Si_3N_4 tip radius approximately 20 nm and a surface scanning area of 10×10 μm^2. The wear tracks of the composite coatings were observed using a scanning electron microscope (JEOLJSM-5600LV). The phase structures of the composite coatings were analyzed on an X-ray diffractometer (Philips X' Pert-MRD). The nanoindentation tests involving loading and unloading courses were conducted on a Nano Test-600 device (Micro Materials, Wrexham, UK), with a trigonal (Berkovich) diamond tip, to determine the hardness of the composite coatings. The approach of Oliver and Pharr [10] was used to realize the tip correction and data reduction, and the computer provides accordingly the microhardness and elastic modulus by Indentatin software and load–unload curve. The load and spatial resolutions of the equipment were 1 mN and 0.1 nm, respectively. Five indentations on different parts of each sample were performed and the averaged hardness values were given in this article. The deviation of the average hardness is less than 5%. The tribological behaviors of the electrodeposited coatings reciprocally sliding against SAE52100 steel ball (ϕ3 mm) were examined on a UMT-2MT tribometer in a ball-on-disk configuration (see Fig. 1). The sliding was performed at an amplitude of 5 mm, a normal load of 0.5~4.0 N, and a frequency of 3.0~5.0 Hz. All the friction and 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. Polarization behavior of Ni–Co–CNTs electrolyte

Fig. 2 shows the cathodic polarization behavior of Ni–Co and Ni–Co–CNTs electrolytes. It is seen that the introduction of the carbon nanotubes in the electrolyte