

# Influence of pulse frequency on the microstructure and wear resistance of electrodeposited Ni–Al<sub>2</sub>O<sub>3</sub> composite coatings

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## Abstract

Nickel matrix composite coatings reinforced with sub-microsized Al<sub>2</sub>O<sub>3</sub> particles were produced by pulse electrodeposition and the effect of pulse frequency on the microstructure, hardness and wear resistance of Ni–Al<sub>2</sub>O<sub>3</sub> composite coatings were investigated. The results showed that the pulse frequency significantly influenced the preferred orientation of Ni–Al<sub>2</sub>O<sub>3</sub> composite coatings; the texture of the coatings progressively changed from a strong (111) preferred orientation to a random orientation when pulse frequency increased. The hardness of composite coatings decreased slightly with the increase of volumetric content of alumina particles. The wear behaviors of composite coatings under dry sliding wear and oil-lubricated wear conditions were different significantly. The wear resistance of Ni–Al<sub>2</sub>O<sub>3</sub> coatings decreased with the increase of incorporated alumina particles under dry sliding wear condition, which was largely influenced by the microstructure of Ni matrix due to the presence of adhesive wear. However, the wear resistance of composite coatings increased with the increase of volumetric content of the reinforcements under oil-lubricated wear condition, which mainly depends on the volumetric content of incorporated alumina particles because the adhesive wear can be avoided under oil-lubricated wear condition.

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## 1. Introduction

Particle-reinforced metal matrix composites generally exhibited wide engineering applications due to their enhanced hardness, better wear and corrosion resistance when compared to pure metal or alloy [1]. Composite electroplating has been identified to be a technologically feasible and economically superior technique for the preparation of such kind of composites [2]. Over the past decades, successful co-deposition of ultra-fine particles such as metallic powder, silicon carbides, oxides, diamond and polymers with metal or alloy matrix have been reported and their corresponding structures and properties were investigated by many researchers [3–6]. The structure and properties of composite coatings depend not only on the concentration, size, distribution, and nature of the reinforced particles, but also on the type of solution used and electroplating

parameters (current density, temperature, pH value, etc.). Among these factors, the type of applied current is one of the most important parameters [7,8].

It is well known that pulse electrodeposition is one of the most effective methods in fabrication of metals and alloys due to its independently controllable parameters and higher instantaneous current densities when compared to traditional DC electrodeposition [9]. The properties of metals and alloys can be controlled and improved by modifying their microstructure when using pulse current. As a result, the effects of pulse electrodeposition parameters on the microstructure and properties of metals and alloys have been reported in many literatures. Jeong et al. have produced nanocrystalline nickel by pulse electrodeposition and investigated the effect of grain size reduction on the wear resistance of pure nickel [10]. Yang et al. investigated the effect of pulse parameters on the morphology and corrosion resistance of nickel deposits and the results showed that electrodeposition of nickel using pulse plating could lower porosity and improve corrosion resistance when

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compared to direct current plating [11]. In addition, pulse electrodeposition of alloys has received considerable attention recently. Brooks and Erb have produced nanocrystalline  $\gamma$ -phase Zn–Ni alloys [12] and found that pulsed electrodeposits presented a smaller grain size and better corrosion properties than the deposits prepared with an equivalent continuous current density [13]. The use of pulse current shifted the polarization curve and greatly affected the composition of alloy deposits [14]. Smooth, bright and nanocrystalline Ni–Cu alloys were obtained by precise control of the pulse parameters. The results showed that the Knoop microhardness for pulse current plated samples was higher than the direct current plated sample and the internal stress was lower for the PC sample [15].

Numerous literatures on pulse electrodeposition of metals and alloys have been reported. However, there are very limited studies focused on the pulse electrodeposition of nickel composite coatings [16–18]. In the present paper, the effect of pulse frequency on the microstructure, hardness and wear resistance of pulse electrodeposition Ni–Al<sub>2</sub>O<sub>3</sub> composite coatings have been investigated.

## 2. Experimental procedures

The plating electrolyte was a Watts-type bath. The composition and the experimental parameters are shown in Table 1. The Al<sub>2</sub>O<sub>3</sub> particle was used as obtained and the average particle size of the Al<sub>2</sub>O<sub>3</sub> particles used in the experiment was about 0.6  $\mu\text{m}$ . Before the co-deposition, the suspended solution was stirred for 6 h, and then was subject to ultrasonic vibration for 30 min. Then the pH of this bath was adjusted to 4 by appropriate additions of HCl or NH<sub>3</sub> solutions.

AISI-1045 steel plate with an area of 0.05 dm<sup>2</sup> on one side was used as the cathode while the other surface of the substrate was blocked with a PVC adhesive tape; the anode was a pure Ni plate. Prior to electroplating, the substrates were mechanically polished to a 0.08–0.12  $\mu\text{m}$  surface finish, and then a sequence of cleanings was performed to remove contamination on the substrate surface. The steel substrates were activated in a mixed acidic bath at room temperature before electroplating. This substrate was placed parallel to a vertically oriented nickel plate at a distance of 0.05 m in the above bath. During the co-deposition process, the bath was slowly stirred by a magnetic stirrer in order to keep the particles dispersed and prevent sedimentation in the electrolyte suspension. After depositing for

3 h, the composite coating was washed in running water. Then, it was cleaned ultrasonically in distilled water for 10 min. The thickness of the produced composite coatings was in the range of 100–120  $\mu\text{m}$ .

The crystal structure of the composite coatings were studied by X-ray diffractometry (Philips X' Pert-MRD). The surface morphology of the coatings were observed using a JSM-5600Lv scanning electron microscopy (SEM) and the percentage of co-deposited Al<sub>2</sub>O<sub>3</sub> particles was evaluated by using energy dispersive X-ray spectroscopy (EDS) analysis tool. Hardness of the coatings was determined using a Vicker's microhardness indenter with a load of 25 g for 10 s. The final value quoted for the hardness of a coating was the average of 10 measurements.

The wear tests were performed on a reciprocating ball-on-disk UMT-2MT tribometer (Center for Tribology, Inc., California, USA) at room temperature with a relative humidity of 45–55% both under dry sliding wear and oil-lubricated wear conditions, respectively. An AISI-52100 stainless steel ball (diameter 3 mm) was used as the counter body. Dry sliding wear tests were performed under a load of 1 N with a sliding speed of 55 mm s<sup>-1</sup>; oil-lubricated wear tests were performed under a load of 20 N with a sliding speed of 110 mm s<sup>-1</sup>. Wear rates of all coatings were calculated on the basis of the volumetric loss, which was measured using a surface profilometer. Wear rates of all the deposits were calculated using the equation of  $K = V/SF$ , where  $V$  is the wear volume loss in mm<sup>3</sup>,  $S$  is the total sliding distance in m and  $F$  is the normal load in N.

## 3. Results and discussion

### 3.1. Effect of pulse frequency on the microstructure of composite coatings

The XRD patterns of Ni–Al<sub>2</sub>O<sub>3</sub> composite coatings prepared by different pulse frequency are illustrated in Fig. 1. As can be seen from Fig. 1, the composite coatings exhibited face-centered cubic (fcc) lattice with different orientation which was influenced by the pulse frequency. It can be clearly observed that the composite coating exhibited obvious (111) preferred orientation at low pulse frequency, and the diffraction intensity of the (200) fiber orientation increased with the increase of pulse frequency. When the pulse frequency increased to 1000 Hz, the composite coating exhibited a random orientation. It indicated that low pulse frequency could produce preferred orientation easier than high pulse frequency. The influence of pulse frequency on the texture coefficient of the composite coatings is tabulated in Table 2. The texture coefficient is expressed as

$$I = 100 \times I_{hkl} / (I_{111} + I_{200} + I_{220} + I_{311}) \quad (1)$$

where  $I$  is the texture coefficient.

As shown in Table 2, the texture coefficients of the composite coatings were affected by pulse frequency. The texture coefficient of the composite coatings prepared under different frequencies was in the order of (111) > (200) > (311) > (220). The texture coefficients of the (200) plane increased while those of the (111) (220) (311) decreased with the increase

Table 1  
Basic bath compositions and electrodeposition conditions

Compositions and conditions	
NiSO <sub>4</sub> ·6H <sub>2</sub> O (g l <sup>-1</sup> )	300
NiCl <sub>2</sub> ·6H <sub>2</sub> O (g l <sup>-1</sup> )	50
H <sub>3</sub> BO <sub>3</sub> (g l <sup>-1</sup> )	40
Sodium dodecyl sulfate (g l <sup>-1</sup> )	0.1
Al <sub>2</sub> O <sub>3</sub> particle (g l <sup>-1</sup> )	20
Temperature (°C)	40–45
pH	4
Current density (A dm <sup>-2</sup> )	3.0
Pulse duty cycle	50%
Pulse frequency (Hz)	10–1000