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Ni–P–SiC composite produced by pulse and direct current plating

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Abstract

This study attempted to incorporate 300 nm SiC particles into a Ni–P alloy matrix by pulse current (PC) and direct current (dc) plating. The effects of bath composition and operating conditions of electrodeposited Ni–P–SiC composite coatings were studied, and the micro-hardness of the deposits was determined. Both plating methods showed that the phosphorus content in the deposit falls with increasing SiC content in the bath, and that the SiC content in the composite coating rises with rising SiC content in the bath. The pulse plating deposit with SiC particles 0.2–1.5 wt.% was higher than direct current plating with SiC particles 0.2–0.5 wt.% in deposits. The wear-proof shows that the tribological behavior of Ni–P–SiC of the PC plating is better than that of the dc plating deposit, due to the higher ultra-fine SiC particles.

Keywords: Ni–P alloy; Composite electroplating; Pulse plating; Direct plating

1. Introduction

Electrodeposition of alloys is especially important for applications to microfabrication technologies, especially to the LIGA molding process, which needs hard microstructure. Ni–P alloys have attracted extensive interest from science and technology due to their beneficial mechanical, chemical and magnetic properties [1,2].

Composite electroplating is a method that involves codepositing fine particles of metallic, non-metallic compounds or polymers in the plated layer to improve material properties such as wear resistance, lubrication and corrosion resistance [3]. During this process, these insoluble materials are suspended in a conventional plating electrolyte and are captured in the growing metal film. The second-phase material may be powder, fiber or encapsulated particles. Generally, the presence of the second phase particles improves micro-hardness, yield strength tensile strength and wear resistance. Significantly improving the application in industry of these materials [4]. The high wear resistance and low cost of ceramic powder has led researchers to investigate and commercialize typical composite materials such as Ni–SiC composites manufactured by electro-codepostion, particularly to protect friction parts [5].

Cheng [6] found that the Ni–P–SiC coatings weight loss of friction decreases with increasing of SiC content in the composite coatings, but increases again after attaining a minimum value with 20 wt.% SiC in the coatings. This phenomenon may be caused by an excessive quantity of SiC particles heaped in the surface, forming a soft constituent. Huichen and Mengxiong [7] found that in dry friction, Si_3N_4 particles in Ni–P–Si_3N_4 composite electrodeposition could reduce the plowing effect between friction pairs and adhesion dropout, enhancing the anti-wear properties of composite electrodeposition.

Although alloy materials such as Ni–Fe and Ni–Co have good hardness at room temperature, these alloys soften as the temperature rises above 200 °C. Ceramic particles can be incorporated into the metal matrix to improve the hardness and anti-wear property of the matrix [8]. Hard particles improve the hardness and tribological properties of the composite Ni layer through dispersive strengthening. The hard inclusions can impede the movement of grain boundaries of Ni grains and slow the grain growth during thermal treatment. Since components of these devices are of the micro scale, the second phase material in the matrix needs to be an order of magnitude smaller; i.e., nanometric [9]. Thus, the deposition of finely dispersed particles in a metal matrix by electrodeposition processes would be inevitable leading to a new generation of composites.

Recently, the availability of ever-smaller particles has expanded potential MMC applications [10]. Well disperse sub-

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micron or nano-size inert particles in a metal matrix can enhance the mechanical properties of composite materials, and are essential composite materials in micro-devices, such as in MEMS (micro-electrical mechanical system). The deposition of finely dispersed particles in a metal matrix by electrodeposition processes correspondingly leads to a new generation of composites. Furthermore, a decrease in size of inert particles in the matrix improves the wear resistance [11].

Pulse plating has been well established in the electrodeposition of metals and alloys, and markedly influences the metal crystallization mechanism. Certain pulse parameters (such as peak current density, duty cycle and frequency) affect adsorption or desorption of a species in the electrolyte and surface diffusion in many more ways than in direct current plating. During the ‘off’ period, the small grain is re-crystallized since its high surface energy makes it less thermodynamically stable than large grains. Hence, as in bubble coalescence, small grains tend to re-crystallize. Additionally, metals deposited by pulsed current exhibit smaller contents of absorbed hydrogen than those produced with continuous current, leading to desorption during the off period [12]. The main aim of pulse plating is to improve deposit properties, including porosity, surface roughness, ductility, hardness, and wear resistance. In recent years, the rectangular waveform of the pulse has been shown to have a higher nucleation rate of the grain in Ni–SiC electro-codeposition than triangular waveform [13]. Additionally, pulse plating has been proven in Ni-electrolyte with nano-Al_2O_3 particles composite plating, which would cause more homogeneous distribution of particles in composite deposits and further enhance the wear resistance [14–16]. A Ni–SiC nanocomposite coating has been developed using pulse current plating and adding sub-micron SiC particles into Watt electrolyte [17], this research shows the micro-hardness values of nano-composites were about two times higher than the polycrystalline Ni–SiC composite.

This study attempted to incorporate SiC particles into an amorphous Ni–P alloy matrix by pulse current (PC) and direct current (dc) plating. Micro-hardness of the deposits, contents of phosphorous and SiC particles in these layers were investigated with respect to the bath composition and operating conditions of electrodeposited Ni–P–SiC coatings processing. Furthermore, the tribological behavior of the coatings with the highest SiC content in deposit that produced by both PC and dc plating were examined.

2. Experimental details

The plating electrolyte was a nickel sulfmate bath. Phosphorous acid was added as the phosphorous element source. The compositions and the range of experimental operating conditions are shown in Table 1. A previous study has shown that higher concentration of SiC in the plating bath would leads to both significant agglomeration and low quantities of SiC particles in the deposition layer [18]. In this study, the concentration of SiC was kept within $20\,g\,L^{-1}$, and surfactant CTAB was added to reduce the SiC particle agglomeration [19]. Additionally, the adsorption of surfactant CTAB on the SiC has been proven to enhance the adhesion force to the cathode, enabling larger particles to be embedded [19]. The average particle size of the SiC used in the experiment was around 300 nm. A nickel plate of 110 mm × 60 mm was used as the anode, and a low carbon steel plate of about half the area of the anode was used as the cathode. The electrolyte bath was stirred by a magnetic stirrer and heated to plating temperature at 60 °C. Each plating solution was mixed by magnetic stirring for 12 h, and subsequently by ultrasonic agitation for 20 min just prior to electroplating. The dc plating used a current density of $10\,A\,dm^{-2}$, and the PC plating had a peak current density of $20\,A\,dm^{-2}$. The duty cycle (i.e., pulse on time/(pulse on time + pulse off time)) was 0.5, and the frequency was 10 Hz. Both plating conditions with the same constant current amount, and with the same average current density. The properties of all composites that plating with 50 μm on steel plate for examined the surface morphology, SiC particle contents and micro-hardness of the cross-section. The current efficiency is well defined as the fraction of current devoted to the deposition of a particular metal. Here, the current efficiency was calculated from dividing the weight of the Ni individually in the Ni–P–SiC coating by the theoretical value of Ni based on the Faraday’s law. The weight of SiC particles is not included in the calculation. For precisely calculate the weight of Ni in the coating, the weight percentage of phosphorous and SiC can be determined using EPMA.

Table 1
Bath composition and operating conditions

Composition and operating conditions	
Nickel sulfmate	$330\,g\,L^{-1}$
Nickel chloride	$10\,g\,L^{-1}$
Boric acid	$40\,g\,L^{-1}$
Phosphoric acid	$10\,g\,L^{-1}$
Cetyltrimethylammonium bromide (CTAB)	$50\,ppm\,L^{-1}$
SiC (0.3 μm)	1, 10, $20\,g\,L^{-1}$
Temperature	60 °C
pH	1.5
Direct current density	$10\,A\,dm^{-2}$
Pulse current condition	
Density	$20\,A\,dm^{-2}$
Duty cycle	1/2
Frequency	10 Hz

After codeposition, all of the 50 μm thick deposit samples were cut and mounted in epoxy resin. Metallographic cross-sections were ground and polished using an alumina suspension. A scanning electron microscope (SEM, Hitachi S-3000) was used to observe the surface microstructure of the deposits. The deposit composition was determined with Electron Probe X-ray Microanalyzer (EPMA). The hardness of the cross-section of the deposited layer was measured with a Vickers micro-hardness tester (TECH FM-100e) with a load of 100 g and tested for 25 s.

Additionally, plating 200 μm thick Ni–P–SiC composite coatings on wear-proof test samples counter surface of the FC-25 cast iron disks in which for examined the tribological behavior through the wear test. The friction and wear tests were carried out on a rotational wear test machine using a ring on disk pair in which is the same as a thrust washer adapter. The steel ring was made of JIS SKD11 steel and was heat treated to attain average hardness HRC 60 ± 1. The stationary iron disk was coated with Ni–P–SiC deposit with a thickness of about 200 μm on the counter surface. Friction and wear tests were carried out in ambient conditions, with temperature 25 ± 3 °C and relative humidity 60 ± 5%, and with no lubrication. The tests were carried out at a speed of $0.269\,m\,s^{-1}$ and under a load of 89.2 N, and the sliding distance was around 965 m. Three replicate friction and wear tests were conducted to minimize data scattering. This study reports the average of the three replicate test results are reported in this work. Detailed polish and treatment process of the test samples were the same as previously work of the Ni–SiC composite by the same study author [21].

3. Results and discussion

Fig. 1 shows the content of SiC particles incorporated into the Ni–P–SiC composite coatings as a function of the bath in both the PC and dc plating. The figure shows that the SiC content in the deposit rises with increasing SiC concentration in the bath at PC and dc plating. Additionally, at the same average