Study of self-lubricant Ni-P-PTFE-SiC composite coating

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It is well known that electroless Ni-P coating has a highly plating capability, high bonding strength, excellent weldability, electrical conductivity, good wear resistance and controllable magnetic properties through suitable heat treatment [1]. Furthermore, the mechanical and tribological properties of Ni-P coating can be improved by the incorporation of different solid particles which are categorized as hard and lubricating one [2–6]. However, the coating hardness is correspondingly decreased with the volume fraction of lubricating particles in the coating and the friction coefficient becomes worse because of the hard particles. To solve the above problem, hybrid composite coatings containing both hard and lubricating particles are receiving more and more interests [7–11].

In this work, electroless composite plating with PTFE and SiC particles is studied to achieve composite coatings with low friction coefficient and good wear resistance. A comparison study of the properties of Ni-P, Ni-P-SiC, Ni-P-PTFE and Ni-P-PTFE-SiC is conducted. Simultaneously, the effects of SiC and PTFE on the properties and microstructure of the coatings are discussed.

The 30 μm coating for each sample was deposited on copper sheet and mild carbon steel under the same process parameters and conditions by electroless nickel plating, respectively. The average size of PTFE and SiC is 0.4 and 4 μm, respectively. Perfluoro plyoxypropylene ammonium iodide (FC134) and hexadecyltrimethyl ammonium bromide (HTAB) surfactants were employed for particles dispersion and surface charge adjustment. Mechanical stirring was used to keep particles from sediment. The main solution composition and experimental conditions for different coatings were listed in Table I.

The micrographs of particles in the coating were analysed using optical microscope. The microstructure and phase transition of the coatings which were stripped from the copper substrate were studied by X-ray diffraction (XRD) and differential scanning calorimetry (DSC). The hardness of these coatings was measured using microhardness tester and the average value of five measurements for each sample was taken as the reported hardness value. Dry sliding wear tests were performed using a ring-on-disc type wear apparatus at an air humidity of 50 ± 10 RH% with a temperature of 22 ± 1 °C. The materials of the ring was GCr15 with the hardness of HV680 and its outer and inner diameter was 30 and 20 mm, respectively. The discs for each samples are prepared by depositing 30 μm coating on $\Phi 30 \times 5$ mm mild carbon steel. The surface roughness of the disc and ring were $R_a = 1.2$ and $R_a = 0.1$ μm, respectively. A normal load of 50 N was used in the wear test and sliding velocity was 50 r/min. The wear loss and normalized wear rate were calculated by the weight loss which was measured by electronic balance with 0.1 mg accuracy. In all wear tests, the average value of three measements in each way is regarded as the final data.

As shown in Fig. 1, it is observed that PTFE and/or SiC particles uniformly distributed in the coatings by virtue of surfactants and mechanical stirring. Moreover, the content of two particles is very large because of FC134 and HTAB. Actually, the distribution of PTFE and SiC can not be uniform if there was no stirring because of the different density of SiC and PTFE.

Fig. 2 shows a typical DSC thermogram of Ni-P-PTFE-SiC coating. At the rate of 20 °C/min, there is a exothermic peak at approximately 346 °C observed in the DSC curve which is associated with the phase transition from the amorphous Ni-P matrix to a mixed structure of polycrystalline Ni and Ni_3P. It can be seen that the introduction of PTFE and SiC particles had little influence on the crystallization temperature (the crystallization temperature of Ni-10 wt%P is 345 °C). This may be due to the low surface energy of PTFE particles and large size of SiC particles.

XRD patterns of Ni-P and Ni-P composite coatings before and after heat treatment are displayed in Fig. 3. It is observed that the as-deposited coatings had amorphous Ni-P structure. After heat treatment at 400 °C for 1 hr, the coatings occurred crystallization resulted from the observation of Ni_3P phase. However, the composite coatings containing SiC or PTFE have relatively weak intensity, whereas, the Ni-P-PTFE-SiC weakest. It indicated that the co-deposited particles impeded the Ni_3P grain growth during the crystallization.

The microhardness of Ni-P and Ni-P composites as a function of heat treatment at different temperature are shown in Fig. 4. It is indicated that the microhardness of the four coatings significantly increased after heat treatment and reached the maximum at 400 °C. This is due to the formation of Ni_3P alloy phase which generates the effect of precipitation hardening. However, the microhardness of the four coatings has a little decrease after heat treatment at above 400 °C because of further growth of the grains at higher temperature. As the above mentioned, the introduction of PTFE or SiC particles

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TABLE I Plating conditions of electroless solution and particles content in the coatings

Plating conditions and particles content in the coatings	Coatings			
	Ni-P	Ni-P-SiC	Ni-P-PTFE	Ni-P-PTFE-SiC
$NiSO_4 \cdot 6H_2O$(g l^{-1})	20–25	20–25	20–25	20–25
$NaH_2PO_2 \cdot H_2O$(g l^{-1})	25–30	25–30	25–30	25–30
$CH_3COONa \cdot 3H_2O$(g l^{-1})	30–40	30–40	30–40	30–40
88%Lactic acid (ml l^{-1})	20–30	20–30	20–30	20–30
Stabilizer (mg l^{-1})	1–3	1–3	1–3	1–3
SiC (g)		6–10		6–10
60 vol%PTFE (ml)			5–8	5–8
HTAB (mg l^{-1})			200	200
FC134 (mg l^{-1})		100–300		100–400
pH	4.6–4.9	4.6–4.9	4.6–4.9	4.6–4.9
T (°C)	89–91	89–91	89–91	89–91
SiC (vol%)		22		10
PTFE (vol%)			20	15

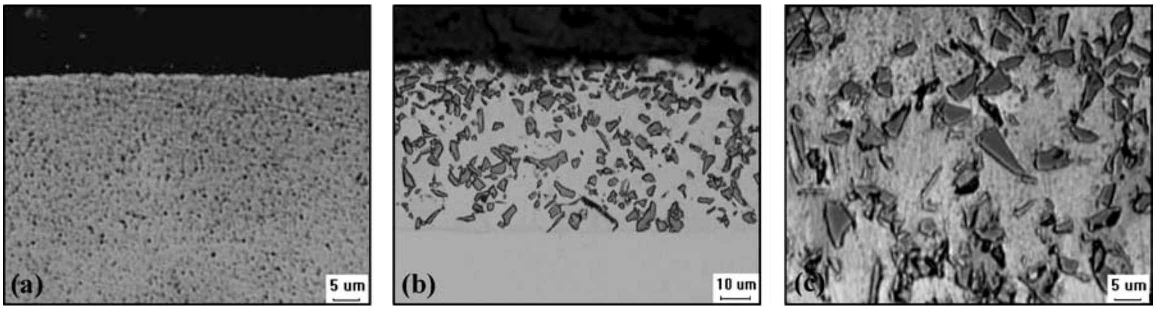

Figure 1 Metallograph of the cross-section of (a) Ni-P-PTFE and (b) Ni-P-SiC (c) Ni-P- PTFE-SiC.

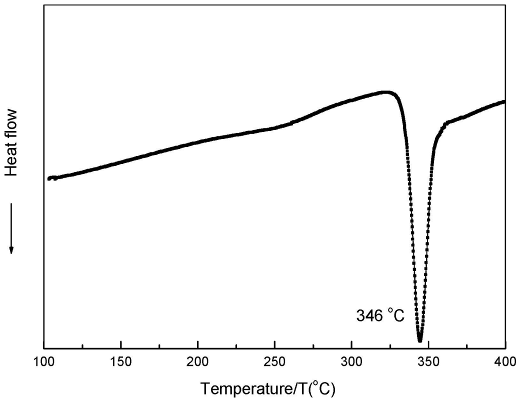

Figure 2 DSC thermogram of the Ni-P-PTFE-SiC at a heating rate of 20 °C/min.

into the Ni-P matrix resulted in a substantial decrease or increase in microhardness. Apparently, the hardness of Ni-P-PTFE-SiC coating exhibits a moderate hardness between Ni-P-PTFE and Ni-P-SiC, depending on the corresponding concentration of PTFE and SiC particles in the coating.

The contact angle, the friction coefficient and the wear rates of Ni-P and Ni-P composite coatings are listed in Table II. To estimate the surface energy, the contact angle of de-ionised water droplet was measured using a OCA15 goniometer. The results tested 3 min later demonstrate that Ni-P-SiC has the highest surface energy. The introduction of PTFE particle into the Ni-P coating significantly increased the contact angle, indicating a decrease in surface energy. A little change of contact angle during three minutes suggested that the surface of the four coatings was compact.

As seen in Fig. 5, the uneven curve with the Ni-P and Ni-P-SiC was caused by the coating spalling along the wear track. The three Ni-P composite coatings-show better wear resistance than Ni-P coating without particles. Among them, Ni-P-PTFE coating has the lowest friction coefficient (Table II), which could be explained by the formation of PTFE film between coating and counterpart during the wear sliding which reduced the friction coefficient. However, the low hardness of Ni-P-PTFE coating make the wear rate higher. The Ni-P-SiC coating demonstrates better load support due to the highest hardness and has the lowest wear rate despite the highest friction coefficient. The Ni-P-PTFE-SiC coating demonstrates a combination of the advantages of Ni-P-SiC in higher load-bearing and Ni-P-PTFE in a low friction coefficient.

In conclusion, the as-deposited coatings have an amorphous Ni-P structure incorporated with uniformly distributed PTFE and SiC particles. DSC thermogram shows the phase transition from the amorphous Ni-P to a mixed structure of crystalline Ni and Ni_3P alloy occurrs at about 346 °C. Heat treatment at different temperature resulted in a increase in microhardness of the coatings which reached the maximum at 400 °C for 1 hr. The Ni-P-SiC coating shows the highest hardness while the Ni-P-PTFE has the lowest friction coefficient. The Ni-P-PTFE-SiC shows a moderate hardness and friction coefficient in between,

TABLE II Main properties of Ni-P and Ni-P composite coatings

		Wear test		Contact angle (°)	
Coatings	Friction coefficient (average)	Load (N)	Wear rate (10^{-3} mg·N^{-1} ·m^{-1})	Beginning	3 minutes later
Ni-P	0.24	50	3.53	95	90
Ni-P-SiC	0.3	50	1.0	88	82
Ni-P-PTFE	0.1	50	2.7	113	109
Ni-P-PTFE-SiC	0.16	50	1.0	103	99

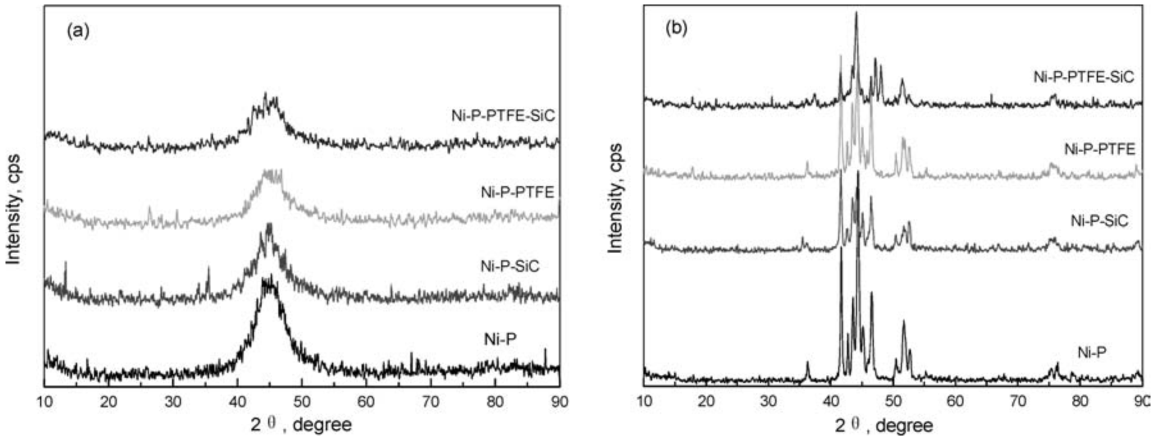

Figure 3 XRD patterns of (a) as-deposited and (b) heat-treated at 400 °C for 1 hr of Ni-P and Ni-P composites.

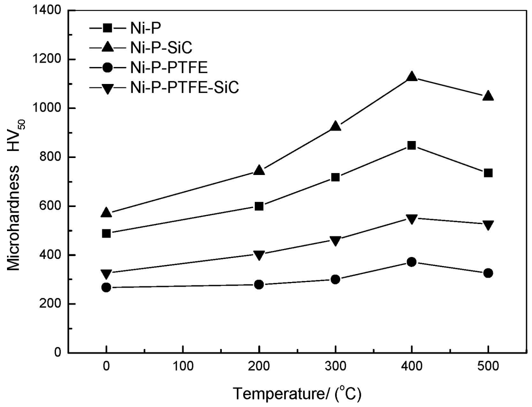

Figure 4 The microhardness of Ni-P and Ni-P composites coatings (at as-deposited, 200 °C, 300 °C, 400 °C, 500 °C for 1 hr, respectively).

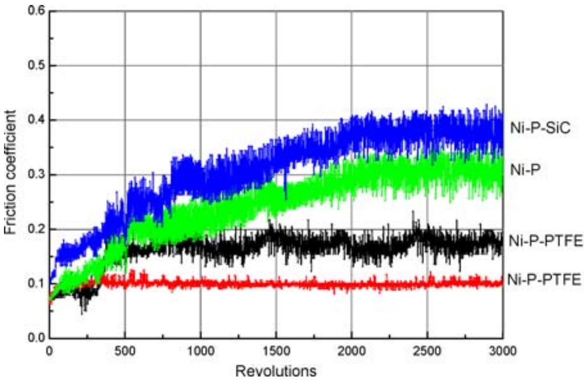

Figure 5 Friction coefficient vs. revolutions for Ni-P and Ni-P composite coatings.

depending on the content of the particles in the coating. In result, the Ni-P-PTFE-SiC composite coating demonstrates a combination of the advantages of the Ni-P-SiC in high hardness and wear resistance, and of the Ni-P-PTFE coating in a low friction coefficient, lower surface energy.

Acknowledgments

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