CODEPOSITION OF NANO-SIZED SiC PARTICLES IN THE NICKEL MATRIX COMPOSITE COATINGS OBTAINED BY ELECTROPLATING

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ABSTRACT

Nano-sized SiC particles were codeposited with nickel by electroplating from a nickel Watts bath and the effects of SiC content in the plating bath and stirring speed on the behaviors of Ni–SiC composite coatings were studied. The researched methods such as: electrochemical method, Scanning Electron Microscope (SEM + EDX), hardness testing… were used. The result shown that the SiC concentration in bath and stirring speed strongly influenced on SiC nano-particulates amount in the coatings, microhardness and corrosion behavior the nano-composite coatings.

Keywords. Electroplating; composite coating; corrosion resistance; microhardness.

1. INTRODUCTION

The incorporation of inert particles during metal electrodeposition has stimulated scientific and technological interests for decades [1 - 3]. Particularly, electrodeposited Ni–SiC composite coating, due to their high wear resistance, have been investigated to the greatest extent and successfully commercialized for the protection of friction parts, combustion engines and casting molds. Recently, interest in electrodeposited nano-composites has increased substantially due mainly to the fact that nano-composite coatings can give various properties, such as wear resistance, high-temperature corrosion protection, oxidation resistance and self-lubrication, to a plated surface [4, 5]. In the present work, nanosized (45 - 55 nm in diameter) SiC particles were employed to prepare nickel matrix composite coatings by electrolytic plating and the effects of plating parameters such as SiC content in the plating bath, current density and stirring speed on the behaviors of Ni–SiC composite coating layers were investigated.

2. EXPERIMENTAL

2.1. Sample preparation

A Watts plating bath with the composition and working condition as showed in Table 1 is prepared from analytical grade chemical and distilled water. Its pH was adjusted by H_2SO_4 5% solution to value 4.0. The commercial SiC powder with an average diameter of 45 - 55 nm (Nanostructured & Amorphous Materials Inc., USA, β-SiC of 98% purity) was used. SiC powders with different concentrations of 1, 2, 4, 8, 12, 16 and 20 gl/L were added to the Watts solution. The magnetic stirring equipment with 2 stirring speed A and B in correspondence with 150 rpm and 250 rpm. was used to keep the stability of suspension.

Before plating, the samples of brass with 2cm^2 area were ground on grit paper 600, cleaned from oils and activated by HCl 5% solution. Electroplating was carried out for 50 minutes and the thickness of the resulting deposits was in the range of 20 - 25 μm.

2.2. Research methods

Electrochemical measurements of nickel coating in testing solution of NaCl 3.5% 1 M were performed on CMS 100 of Gamry Company (USA), three electrodes system was used, in which counter electrode is platinum electrode; reference is Calomel (SCE) and scanning rate 1 mV/s.

The structure and morphography of Nickel composite electroplating coatings were investigated by scanning electron microscope JSM-5410 (Jeol). The SiC amount in deposit was determinated by SEM and EDX JSM-5410. The microhardness of the coating was measured on the polished cross sections and reported as an average of three values (Microhardness Tester MHT-10, Anton Paar).

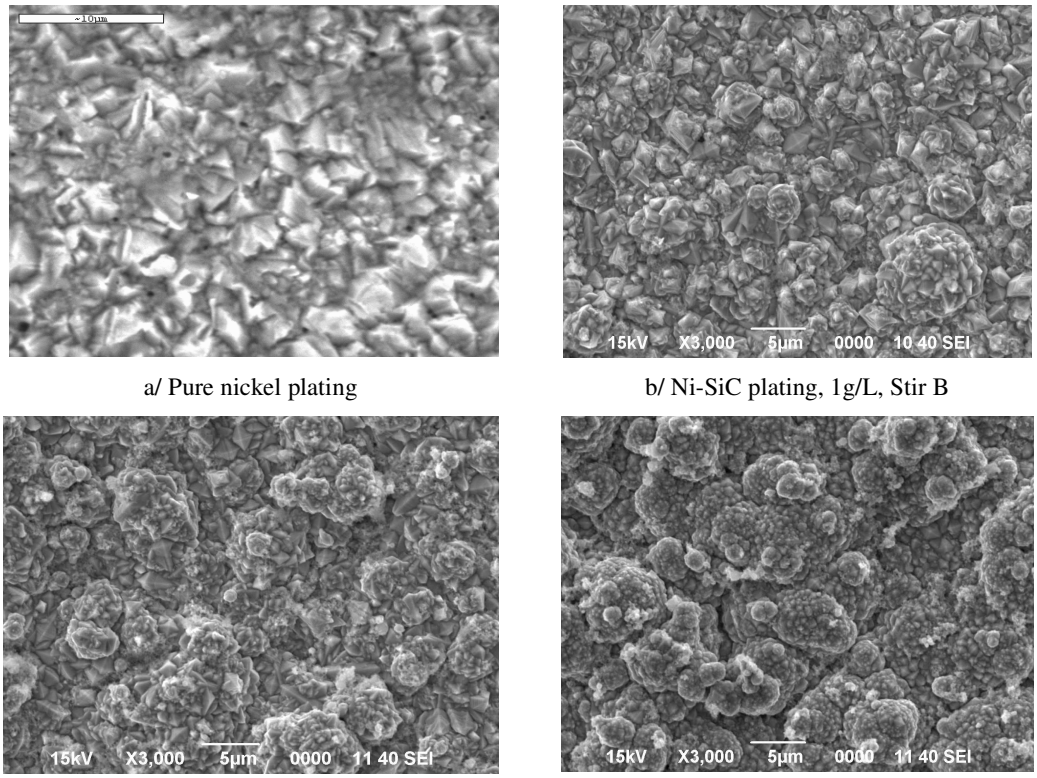
Table 1. The composition of plating bath

Composition	**Working condition**
300 g/l $Ni_2SO_4.6H_2O$	pH = 4
50 g/l $NiCl_2.6H_2O$	T = 55^{o}C
40 g/l H_3BO_4	i_c = 2 – 4 A/dm^2
	t = 50 min
SiC powder, 45 – 55 nm.	Mechnical stirring
	Cathode: Brass

3. RESULTS AND DISCUSSION

3.1. Morphology of plating

The surface morphlogy of the coating is showed on Fig.1. **A** regular pyramidal structure as shown in Fig. l(a) is observed on the surface of the pure nickel film This pyramidal growth is a typical way of field-oriented texture, that is, the preferential growth in the direction of electric field [6]. However, with the increase the of SiC nanoparticles concentration in solution, the size of pyramidal-shaped crystal crystalline decreased gradually, and eventually be substituted by fine grains, the surface became smoother, as shown in Fig. l(b)(c)(d). The influence of SiC particles on nickel crystallization process can be explained that: the deposition process of coating is the combination of crystal nucleus formation speed and crystallization speed. When crystal nucleus formation speed is greater than crystallization speed, the crystalline size of deposits become small. Thus, the dispersion of nano - size SiC particle in solution and adsorbtion on cathode surface strongly influenced on crystal nucleus formation process and inhibited crystallization process of nickel. The grain fining and dispersive strengthening effects become stronger with increasing SiC nano-particulates content in solution.



a/ Pure nickel plating

b/ Ni-SiC plating, 1g/L, Stir B

c/ Ni-SiC plating, 4g/L, Stir A

d/ Ni-SiC plating,20g/L, Stir B

Figure 1. Morphology of nickel pure nickel and nickel compositee platings

3.2. The influence of SiC concentration in the solution and stirring speed on SiC content in the deposit

In order to determine the SiC amount in the deposit, the SEM and zone EDX analysis with the magnification of X2000 was performed and results were shown on table 2 and Fig. 2.

The results showed that in constant stirring rate, the amount of the SiC in the composite coating increases sharply with increasing the SiC nano-particle concentration in the solution and got a maximum value in range 4 - 5 g/L. As the SiC concentration in the solution passes over 5 g/L, the amount of the SiC in the composite coating decreases (Fig. 2). The results can be explained that a higher particle concentration in the electrolyte increases the adsorption, thus resulting in a higher amount of SiC particles in composite coatings. In range 4 – 5 g/L the SiC concentration causing maximum codeposition of SiC and may correspond to steady state equilibrium, where the number codepositing SiC particles equals that number SiC particles approaching the cathode surface. Above this SiC concentration, suspended SiC particles may agglomerate in the plating bath. A similar trend has been reported elsewhere: at high SiC content in the plating bath, the agglomerates themselves will be resistant to incorporate into the nickel matrix and shield the surface of the cathode from theflux of incoming SiC particles [7]. Increasing the stirring rate up to 250 rpm causes to increase the percent of SiC in coating. It can

be explained that: As the stirring speed increases, the stirring speed becomes strong enough to float the SiC particles. So the codeposition of SiC increases as the stirring speed increases up to a certain stirring speed.

Table 2. The dependence of the SiC content in the coating on the SiC concentration in solution and stirring speed.

SiC concentration In solution, g/l	**SiC content in coating, % Weight**		**SiC content in coating, % Volume**	
	Stirring A	**Stirring B**	**Stirring A**	**Stirring B**
1	1,17	1,21	2,98	3,09
2	1,22	1,30	3,11	3,32
4	2,73	3,70	6,96	9,44
8	1,73	1,93	4,41	4,92
12	1,57	1,70	4,00	4,34
16	1,45	1,58	3,70	4,03
20	1,21	1,16	3,09	3,00

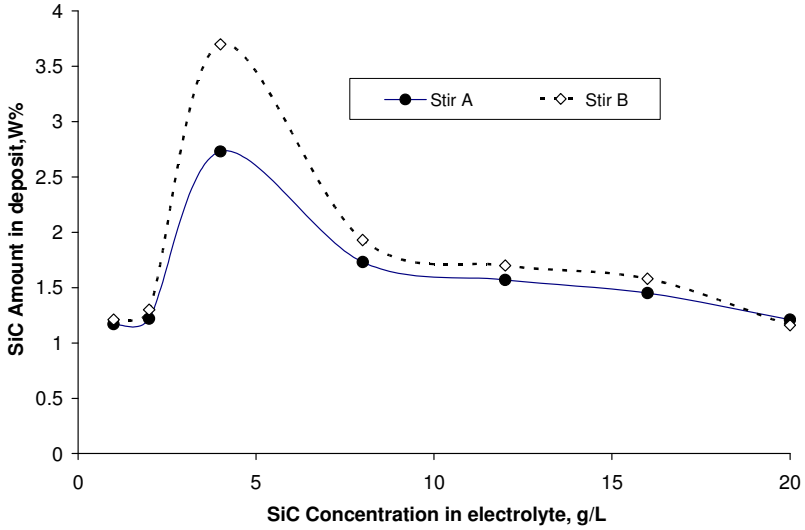


Figure 2. The dependence of the SiC content in the coating on the SiC concentration in solution and stirring speed

3.3. The influence of SiC on the hardness of the deposit

The composite platings with the thickness more than 20 µm were used for hardness evaluation. Obtained results were showed on the Table 3. From this Table, we can see that the hardness of Ni-SiC composite plating is in the range of 318 HV to 522 HV and higher than that of pure nickel coating (212 – 235 HV). Figure 3 shown the relationship between concentration of SiC in solution, stirring speed and microhardness of deposit and fig. 4 expressed the dependence of hardness on amount of SiC in deposit.

The hardness of deposit increases as increasing the amount of inert particles and obeyed also the exponent equation. Probably, the adding of inert particles into deposit was changed the microstructure of deposit, decreasing the size of nickel matrix microcrystalline and widening the area of crystalline boundary. These particles becomethe centers to prohibit the moving of the microstructure site oblique during deformation, therefore the hardness increases [5]. The maximum hardness 522 HV gained when SiC concentration in plating bath 4-5g/L and maximum SiC content in deposit (6,96%W and 9,44%V).

Table 3. The relationship between SiC content and hardness of deposit

SiC concentration In olution, g/l	**SiC content in coating, % Volume**		**Microhardness (HV)**	
	Stirring A	**Stirring B**	**Stirring A**	**Stirring B**
0	0	0	212	235
1	2,98	3,09	347	353
2	3,11	3,32	370	382
4	6,96	9,44	463	522
8	4,41	4,92	434	468
12	4,00	4,34	422	449
16	3,70	4,03	402	413
20	3,09	3,00	318	332

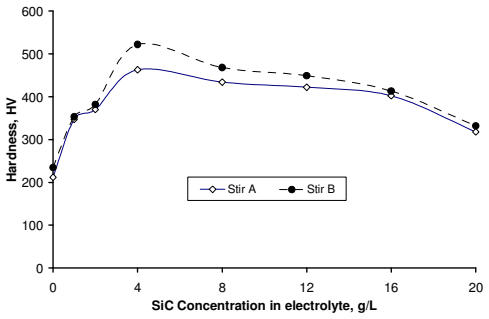


Figure 3. Relationship between concentration of SiC in solution, stirring speed and microhardness of deposit

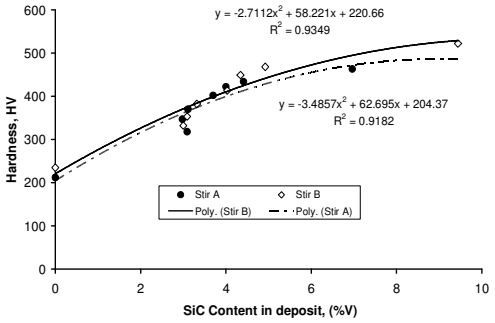


Figure 4. Relationship between content of SiC in deposit and hardness of deposit

4. CONCLUSIONS

Nano - sized SiC particles in plating electrolyte and stirring speed strongly influenced on nickel crystallization process concerning which showed through finer crystall size and content changing of SiC in deposit.

At range 4 – 5 g/L of nano-size SiC powder in plating electrolyte, the maximum amount of SiC in deposit gained and increased as increasing stirring speed..

The hardness of deposit increased when the SiC inert particles amount increased in the deposit. This relationship followed 2-exponent function.

At the nano-size SiC concentration in the solution 4 – 5 g/l, stirring speed 250 rpm, the SiC amount included in the deposit reached 3.70% in mass or 9,44% in volume and the hardness of deposit is maximun 522 HV.

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