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Effect of Bath Temperature on Electrochemical Codeposition of Nickel Silicon Carbide Composite

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Abstract

Ni-SiC composite has been prepared by codeposition of nano SiC particles on nickel matrix by electrochemical codeposition technique. Nickel sulfamate bath was used along with grain modifier saccharine and cationic surfactant Cetyltrimethylammonium bromide (CTAB). The effect of bath temperature was systematically studied and optimized. The result revealed that, 50°C of bath temperature is optimum temperature for the electrochemical codeposition of Ni–SiC. As prepared sample at 50°C showed better surface morphology with uniform distribution of SiC particle with appropriate amount. Elemental Analysis showed the incorporation of nano SiC particle in the ratio of Ni:Si:C in 78:2:20 in atomic weight percentage. Excellent microhardness and low coefficient of friction were observed only in the sample prepared at 50°C bath temperature.

Keywords: Electrochemical Codeposition, Surface Morphology, Micro hardness, Coefficient of Friction

Contents

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1. Introduction

Different methods of producing composite coating with metallic matrix have been described such as thermal spray, physical vapor deposition, chemical vapor deposition, power metallurgy, metal spraying, internal oxidation and electrochemical codeposition [1]. Among them, electrochemical codeposition process became popular in these days due to its low operational cost, high deposition rate [2] and can be conducted at normal pressure and ambient temperature. This method often allows the production of thin foils. Having these advantages, it has been considered as one of the most important techniqueand has been widely used in high-tech industries such as electronic components and computers, automobile industries, paper mills, textile and food industries [3].

Electrochemical codeposition is a process of incorporation of the nano or micro size particles into the growing metal matrix. It is analogous to a galvanic cell but working in reverse way. The pure metallic substrate is placed at cathode whereas material to be plated is kept at anode. Both components are immersed in an electrolytic bath, which

contains one or more dissolved metal salts that permits the flow of electricity. Nano or micro sized particles are also dispersed into the electrolyte, so that, they can incorporated along with growing metal matrix to form composite coating. Ceramic particles like hard oxides (Al2O[4], TiO2[5], ZrO2 [6], Cr2O3[7] etc.), carbides (SiC [8], WC [9], TiC [10] etc.), nitrides (BN [11], AlN [12, 13]), solid lubricants (PTFE [14], graphites [15], MoS2 [16]), TiB2 [17] , diamond, Al flakes [18], nano rods, nano tubes, nano wires [19] are generally used in electrochemical codeposition. Such incorporated particles in metal matrix give low coefficient of friction, high corrosion resistance, improved microhardness, excellent adhesion, good strength, self lubricating and high temperature inertness, along with chemical and biological compatibility [20].

The overall properties of the composite coatings depend upon the amount of incorporated nano particles and its distribution. Whereas, the incorporation of the particles on metal matrix depends upon the parameters like particle characteristics [21], bath composition [22], bath temperature [23], bath pH [24], stirring rate [25] and current conditions [26]. Bath temperature is one of the important parameter for the electrochemical codeposition process. Most of the investigators have studied on different parameters of electrochemical codeposition of composite coatings [27-34]. Zainal et.al studied the effect of temperature on electrodeposition of copper in Tin Selenide film and found that room temperature is suitable condition [23]. Goux et.al also studied the temperature effect on ZnO deposition on metal matrix and found that the temperature less than 34^0C gives thin layer of Zinc hydroxide, whereas, the film transparency and crystallinity was obtained only above 40^0C temperature [35]. Santos et.al had investigated on change of cobalt deposition with the temperature [36]. Kim et al also studied on micro sized SiC particle incorporation for the formation of bilayer Ni-SiC composite coating and highest volume percentage of SiC was obtained at 50^0C [37]. In present study, we have reported the effect of temperature on electrocodeposition of nano sized SiC particles on nickel matrix. The bath temperature was optimized to get highest microhardness and lowest coefficient of friction with agglomerate free, well dispersed nano particles in appropriate amount.2.

2. Materials and Methods

Experiments were carried out in conventional two electrode electrochemical cell, in which the nickel balls in the titania basket was used as anode whereas SUS 304 stainless steel substrate of (4.5×4.5) cm^2 exposed surface area was taken as cathode. The sulfamate bath was used along with grain modifier saccharine and cationic surfactant CTAB. Electrolytic bath composition and operational conditions used during the codeposition process is given in **Table 1**. The temperature of the bath was maintained and regulated by using thermo regulator and varied from 30°C to 60°C. Four different samples were prepared namely (a) S-30, (b) S-40, (c) S-50 and (d) S-60, which were prepared at 30⁰C, 40⁰C, 50⁰C and 60⁰ C bath temperature respectively. Controlled pulse current was supplied by using Pulse Rectifier, Jiasang Electric Company Ltd. All the experiments were carried out in continuous stirring condition by using teflon coated magnetic bar. All reagents used were of analytical grade and obtained from Duksan Pure Chemicals Co. Ltd. Korea. These reagents were prepared in de-ionized water.

Table 1. Bath Composition and Operating Condition

Bath composition		Operating condition	
Ni(NH₂SO₃)₂ (g/L)	300	pH	4
NiCl₂ (g/L)	10	Temperature (^{o}C)	varies (30, 40, 50, and 60)
H₃BO₃ (g/L)	40	Current type	Pulse
CTAB (g/L)	0.2	Duty cycle (%)	50
Saccharine (g/L)	2	Pulse frequency (Hz)	100
SiC (g/L)	20	Current density (mA/cm^2)	60
SiC size (nm)	270	Stirring rate (rpm)	250
-		Time (h)	1

Table 2. Tribological Test Condition

Tribological Parameters	Operating Conditions
Normal load	2N
Sliding Seed	9.42 cm/S
Total sliding Distance	56.52 m
Rotating radius	3 mm
Temperature	RT
Time	10 min
Lubricants	No

In this study, surface morphology, crystal growth and distribution of the incorporated nano SiC particle were characterized by the scanning electron microscopy (SEM), mini-SEM (nanoeye, Co.). Elemental analysis was studied by energy dispersive X-ray spectroscopy (EDS) detector of Oxford data reference system. Vickers Microhardness Tester (Buehler Ltd., USA) was used to measure the microhardness. Here 100 g load was applied for 10 seconds on seven different places of the sample at the cross-sectional area. Then the corresponding final values were determined as the average of seven measurements. Tribological properties including coefficient of friction of the samples were evaluated by ball on disc method using Tribometer (CSM Swiss). The used tribological test condition is given in **Table 2.**

3. Results and Discussion

Surface Morphology

Surface morphology of composite coating was studied by SEM images. The SEM images of samples prepared at different temperatures are shown in **Fig.1**.
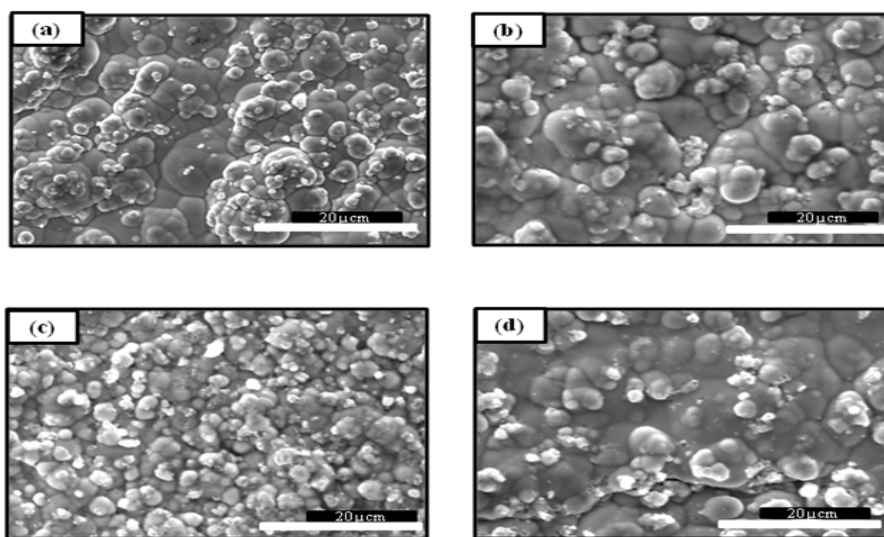

Figure 1. SEM images of (a) S-30, (b) S-40, (c) S-50 and (d) S-60

As can be seen in the **Fig. 1 (a-d),** the surface morphology of samples are found to be different from each other. The **Fig. 1-c** showed the regular surface morphology with large number of crystal growth sites whereas other samples showed irregular surface morphology having some cracks. Sample S-30 (**Fig. 1-a**) showed agglomerated type of surface morphology. Such agglomerate surface is not desirable because it reduces the smoothness of the surface and hence inhomogeniety in the overall properties of coating.. A significant crack is obvious in sample S-60 (**Fig. 1-d**). This may be due to the higher incorporation of SiC particles which leads less compaction of nickel matrix. Thus, the SEM image clearly indicates the effect of temperature on surface morphology. It was found that 50^0C of bath temperature is optimum temperature to get better surface morphology.
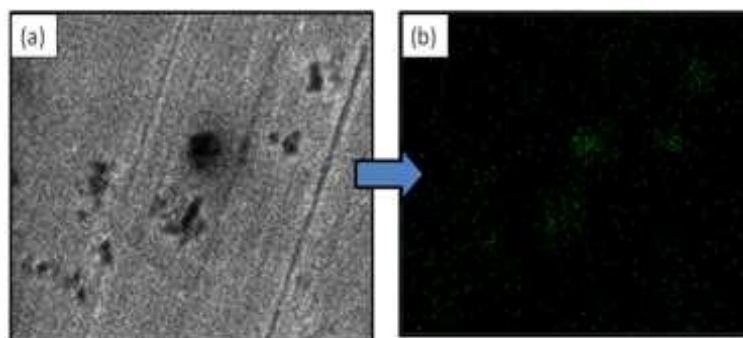
Elemental Analysis

Figure 2. (a) Cross-sectional SEM Micrograph of Ni-SiC Composite Coating
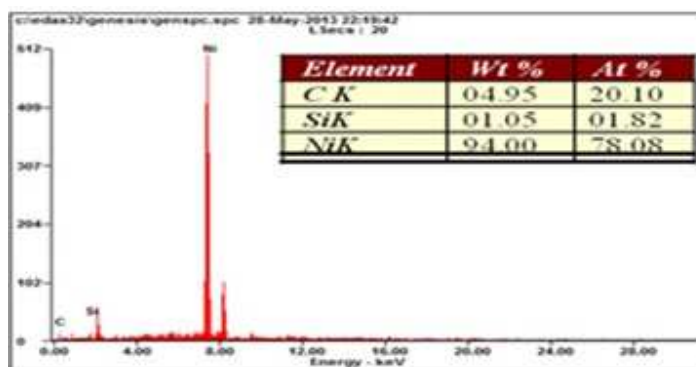**(b) X-Ray Dot Mapping of Silicon**

Figure 3. EDS Spectrum

SEM and EDS mapping image of codeposited sampleS-50 in cross sectional view is shown in Fig. 2 (a), (b). A black particles in SEM showed a presence of silicon particles and it is further evidenced by X-ray dot mapping image. The spots seen in Fig. 2-b indicates the silicon particles. EDS spectrum (Fig. 3) shows that it contains Ni:Si:C in 78:2:20 in atomic weight percentage ratio. The result supports that there is an incorporation of nano SiC particle into the nickel matrix. Cross sectional SEM images of samples are shown in Fig. 4.
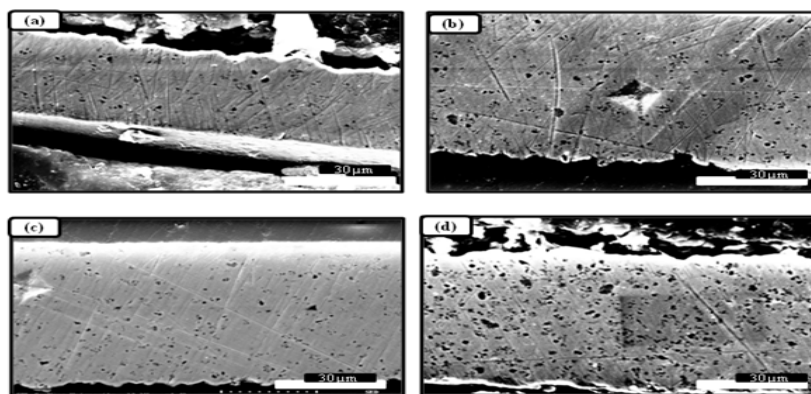

Figure 4. Cross-sectional SEM images of (a) S-30, (b) S-40, (c) S-50 and (d) S-60

In **Fig. 4 (a-d)**, black particles are clearly seen which indicates the nano SiC particles. It exposes that there is a successful incorporation of nano SiC particles along with the metal matrix and are well distributed throughout the sample. Among these four samples, sample S-50 (**Fig. 4-c)** and S-60 (**Fig. 4-d)** have large numbersufficient number of nano SiC particles, however, nano -SiC particle seem to be homogeneously distributed in S-50 (**Fig. 4-c)**. This result reveals that bath temperature has direct effect on the dispersion of nano SiC particles which are responsible for the overall properties of coating sample. Such high amount of incorporation of SiC particles are also not desirable because they show adverse effect on the overall properties due to the agglomeration and less compaction. This result suggests that 50^0C temperature is best condition to get smooth surface with well distributed nano SiC.
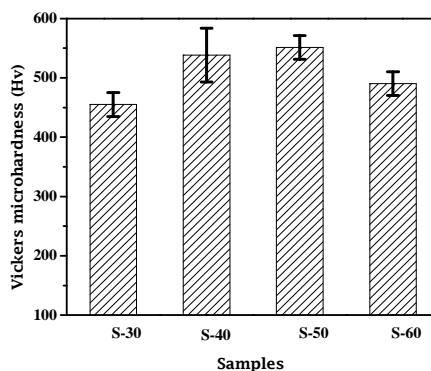**Vickers Microhardness:**

Figure 5: Variation of Vickers microhardness of Ni-SiC coatings as a function of different temperatures

 Fig. 5 shows the bar diagram of microhardness. This diagram indicates that the trend of Vickers microhardness value, initially increases from S-30 to S-50 then it decreases. The sample S-50 has highest vickers microhardness value than that of other samples. It seems that higher value of the microhardness resulted from the higher codeposition of SiC particles with uniformly distribution, which induced the hardening effect by unique dispersion method. This result suggests that the 50^0C temperature is the optimum temperature to produce excellent microhardness.
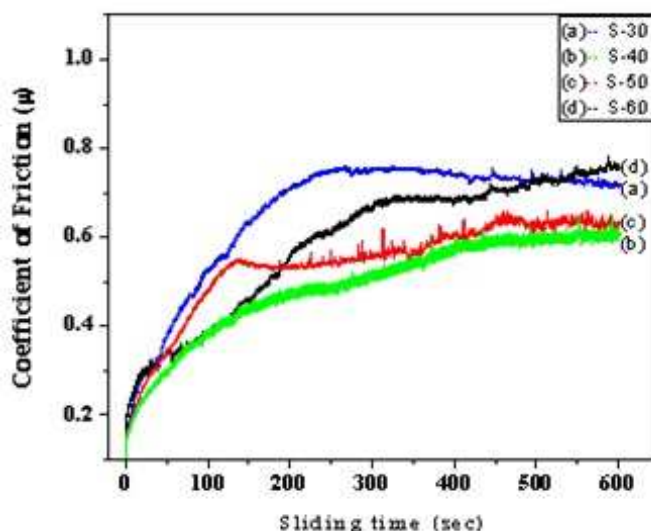
Tribological Test:

Figure 6. Coefficient of friction as a function of sliding time in Sec

Fig.6 shows the plots of coefficient of friction as a function of sliding time for the samples S-30, S-40, S-50 and S-60. Here curve (a) shows the higher coefficient of friction than others. Curve (b) and (d) shows the continuous increase even after 10 minutes while the curve (c) for sample S-50 shows the initial increase of coefficient of friction value upto 350 Sec minutes, then it changes the nature of curve i.e, the plateau indicating the constant value. This result infers that, samples prepared at lower and higher temperature shows the higher value of coefficient of friction. This may be due to the surface roughness and agglomerization of nano particles. Little incorporation of nano particle at lower temperature leads to the higher coefficient of friction while at higher temperature excessive incorporation gives higher coefficient of friction may be due to the wearing of steel ball itself. This fact is also supported by cross-sectional SEM images as shown in **Fig. 4**. From the experiment, it can be resumed that homogeneously distributed nano SiC in appreciable amount sufficient amount is essential condition to reduce the coefficient of friction. Hence, 50^oC bath temperature is favorable condition to bring uniform distribution with lower coefficient of friction.

4. Conclusion

Nano-sized SiC particles were successfully codeposited with nickel by electrochemical codeposition technique from a nickel sulfamate bath. The effects of bath temperature on the deposition behaviors of Ni–SiC composite coating were studied. The following conclusions are derived from the investigation.
   a. The bath temperature 50^0C was found to be optimal temperature to bring smooth and regular surface morphology of composite coating as indicated by SEM image. The temperature lower or higher than 50^oC gives agglomerated and cracked surface.
   b. Elemental analysis showed the incorporation of nano SiC particle along with nickel metal matrix in the ratio of Ni:Si:C in 78:2:20 in atomic weight percentage.
   c. The trend of codeposition of SiC was increased on increasing temperature but homogeneous distribution of SiC particles was observed only in 50^oC.
   d. Highest Vickers microhardness (555 ± 10 Hv) was achieved at 50^0C bath temperature.
   e. The lower and constant coefficient of friction value was observed in sample prepared at 50^0C temperature.
From the results, it can be concluded that the 50^oC of bath temperature is found to be optimum bath temperature for the electrochemical codeposition of Ni-SiC composite to get better surface morphology with improved microhardness that gives anti friction properties.

5. Acknowledgement

6. Refrences

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