



Synthesis and characterization of Ni–Co/SiC nanocomposite coatings using sediment co-deposition technique

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ABSTRACT

SiC nano-particle reinforced Ni–Co alloy matrix nanocomposite coatings were electrodeposited in a modified Watt's bath using sediment co-deposition (SCD) technique. The SiC nano-particle content and the microhardness of the Ni–Co/SiC nanocomposite coatings were investigated as a function of the electrodeposition parameters; such as the deposition current density (i_d), concentrations of the Co (C_{Co}) and the SiC nano-particles (C_{SiC}) in the bath and the electrolyte agitation rate (a_r). The deposited coatings were characterized using scanning electron microscopy (SEM), energy dispersive spectrometer (EDS), X-ray diffraction (XRD) and microhardness testing. $C_{Co} = 50$ g/L and the $C_{SiC} = 5$ g/L, $i_d = 3$ A/dm² and $a_r = 350$ rpm were determined as the optimum electrodeposition conditions to achieve the maximum microhardness. The highest microhardness was obtained for a coating containing 8.1 vol.% SiC nano-particles and 55 wt.% Co. Effects of the electrodeposition parameters on co-deposition behavior of the SiC nano-particles were discussed based on a co-deposition kinetic model. The variations in the microhardness of the coatings were explained based on the nano-structural details.

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1. Introduction

Over the course of the last decades, electrodeposition of metal matrix composite coatings have been fascinated because of their unique mechanical properties [1,2], self-lubricity [3], thermal stability [4], corrosion [5,6] and wear [7–9] resistances. Different types of oxide [5,8,10,11], carbide [2,6], nitride [12,13], graphite [14], diamond [15], polymer [16,17] and metallic [18] particles were used as dispersed phase in electrodepositing the composite coatings.

Principally, properties of the composite coatings are determined by (i) the intrinsic properties of the matrix and the dispersed phase materials, (ii) the size and volume percent of the dispersed phase, and (iii) microstructural features of the composite i.e. distribution of the reinforcing particles and the grain size of the matrix material. The microstructure of the composite coatings is strongly influenced by the electrodeposition parameters such as electrolyte composition, temperature, electrodes arrangement (position and distance), deposition current density and electrolyte agitation. Production of composite coating with the desired microstructure and performance not only requires a proper selection of reinforcing (type, size and shape) and matrix materials, but also necessitates a close control of the electrodeposition parameters. The desired coating properties are achieved at optimized co-deposition conditions.

In recent years, nanocomposite coatings have been produced by incorporation of nano-particles in the metallic matrices to achieve improved properties compared to composite coatings [5,9–11].

At equal volume percent, the nano-particles induce more effective enhancements in the properties of the metallic matrix compared to the micro-sized particles [1,19,20]. Despite their enhanced properties, co-deposition of nanocomposite coatings is more difficult than co-deposition of composite coatings containing coarser (micro-sized) particles mainly due to the particles agglomeration [21].

In conventional co-deposition techniques, the incorporation efficiency of the nano-particles is low. Different methods have been suggested to enhance the nano-particle incorporation in the composite coatings such as using sediment co-deposition (SCD) technique [14,20,22], addition of surfactants [23,24], addition of metallic cations [25,26], changing the applied current mode [27], employing electrolyte agitation using ultrasonic waves [28], changing the electrolyte agitation method [5,29] and applying two step electrodeposition process [30]. In the SCD technique, the electrodes are positioned horizontally inside the electrolyte and unlike the conventional electro-co-deposition (CECD) technique, the gravitational force facilitates incorporation of the particles. In this method, both of the gravitational and electrophoretic forces are acting in the same direction.

Electrophoretic forces are built up between cathode surface and charged particles after adsorption of metallic cations onto the surface of the inert particles. The magnitude of the electrophoretic force is determined by the extent and type of the existing metallic

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cations in the electrolyte. For example, an enhanced incorporation of the nano-particles was reported when the Watt's bath contains the Co^{2+} cations in addition to the Ni^{2+} cations [31]. This leads to the formation of the Ni–Co alloy matrix instead of unalloyed Ni. The Ni–Co alloy deposits have attracted much attention due to their salient features compared to unalloyed Ni deposits [32]. In general, electrodeposition of binary alloys such as Ni–W [33], Zn–Ni [34], Ni–Fe [35] and particularly Ni–Co alloys [31,36–38], instead of single metal, as the matrix material is one of the recent trends in developing the nanocomposite coatings.

Different composite and nanocomposite coatings have been electrodeposited using SCD technique however, according to our knowledge; there are a few publications concerning electrodeposition of the Ni–Co/SiC alloy matrix-nanocomposite coatings using SCD technique [20]. The aim of the present work is investigating the effects of the electrodeposition parameters and characterization of the Ni–Co/SiC nanocomposite coatings deposited using the SCD technique.

2. Experimental details

Electrodeposition of the Ni–Co/SiC nanocomposite coatings was carried out using sediment co-deposition (SCD) technique in a modified Watt's bath. The electrodeposition bath was produced via dispersion of the SiC nano-particles inside an electrolyte which was already prepared using Merck analytical grade reagents. The electrolyte composition and the electrodeposition parameters are given in Table 1. The utilized SiC nano-particles were β -type and were manufactured by Plasmachem GmbH of Germany and had high purity (>99%) and spherical shape with average particle size of 20 nm and specific surface area of $80 \text{ m}^2/\text{g}$. For bath preparation, the SiC nano-particles were added to the electrolyte, little by little. Prior to electrodeposition, the bath was agitated for 24 h in order to overcome wetting problems, to improve dispersion of the nano-SiC particles, to minimize the particle agglomeration, and to ensure the uniform adsorption of the surfactant and cations on the particle surface.

Agitation of the electrolyte was performed by the alternate use of a Hielscher-UP100H ultrasonic disperser and a magnetic stirrer. The ultrasonic dispersion has beneficial effects on disintegration of the agglomerated particles, while the magnetic agitation is an effective approach to keep the nano-particles suspended in the electrolyte. Longitudinal mechanical vibrations were generated by ultrasonic processor and after amplification by a MS7 sonotrode were transferred to the electrodeposition bath. At the beginning, electrolyte was agitated by ultrasonic disperser for 60 min. Then, it was followed by repeated cycles of magnetic stirring (315 min) and ultrasonic dispersion (30 min) for 24 h. During electrodeposition stage, only magnetic stirring was employed.

The substrates were copper plates ($25 \times 25 \times 1 \text{ mm}^3$) which they were positioned horizontally inside the bath, in parallel with and below a pure nickel plate as the anode (Fig. 1). The distance between anode and cathode was 3 cm. Substrates were mechanically polished with silicon carbide abrasive papers of 80–4000 (European FEPA or P-Grading). The polished substrates were ultrasonically cleaned in the ethanol and acetone for 10 min, sequentially and washed in distilled water. After that, they were weighed and activated in 10% H_2SO_4 for 60 s.

Electrodeposition was carried out in a 200 ml glass beaker. The temperature of the bath was maintained at $45 \text{ }^\circ\text{C}$ and the pH was adjusted with H_2SO_4 and NaOH at around 4.3 ± 0.01 . The deposition time was adjusted to attain a coating thickness of around $30 \text{ }\mu\text{m}$. After the electrodeposition process, the coatings were ultrasonically cleaned by distilled water for 5 min; in order to remove partially entrapped nano-particles from the cathode surface.

Table 1
Electrolyte composition and co-deposition conditions.

Electrolyte ingredients	Material grade	Concentration (g/L)
$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$	ME-1.06726	250
$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	ME-1.06717	40
$\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$	ME-1.02556	50
H_3BO_3	ME-1.00165	40
Sodium Dodecyl Sulfate (SDS)	ME-1.12533	0.35
SiC	Plasmachem GmbH	0–20
Deposition parameters		Amount
Type of current		DC
Current density (A/dm^2)		1–4
Temperature ($^\circ\text{C}$)		45 ± 2
Solution pH		4.3 ± 0.02
Magnetic agitation rate (rpm)		200–400

The phase structure and average grain size of the Ni–Co/SiC nanocomposite coatings were determined from the XRD patterns recorded in the Bragg–Brentano configuration using a D8 ADVANCE-BRUKER AXS X-ray diffractometer operated at 40 kV and 40 mA with $\text{Cu K}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$). The XRD patterns were recorded in the angular range $30\text{--}90^\circ$ with step size of 0.03° and step durations of one second. The grain size of the matrix alloy was evaluated from the XRD peak broadening using Scherrer equation. Integral peak width was used to estimate the crystallite size after excluding the instrumental broadening. Instrumental broadening was determined using the XRD pattern of a standard LaB6 sample. Surface morphology of the nanocomposite coatings was investigated using a CamScan MV2300 scanning electron microscope (SEM). An Oxford energy dispersive X-ray detector (EDS) coupled with SEM was used to determine the chemical composition of the coatings and calculate the SiC content of the coatings. Five randomly chosen areas were analyzed (at $500\times$ magnification) and an average value was calculated and reported as the chemical analysis data. Standard deviation of five independent measurements was reported as error bars. The Vickers microhardness measurements were carried out using a MDPEL-M400 GL microhardness tester by employing the indentation load of 50 g and indentation time of 10 s. Microhardness measurements were performed on the polished cross-section and top surface of the coatings. The average value of ten different measurements was reported as the coating microhardness.

3. Results

3.1. Effects of the electrodeposition parameters on the SiC nano-particle content

3.1.1. Deposition current density and SiC nano-particle concentration

Fig. 2 shows the variation of SiC nano-particle content in the Ni–Co/SiC nanocomposite coatings as a function of the deposition current density (i_d) and the SiC nano-particle concentration (C_{SiC}). It can be seen that for a given amount of the C_{SiC} , the SiC nano-particle content in the deposits is increased by increasing the i_d and reaches a maximum at $i_d = 3 \text{ A}/\text{dm}^2$. Beyond this deposition current density, the SiC nano-particle content is decreased. These results confirm previous reports concerning co-deposition behavior of different types of particles [18,39,40] and different sizes of the SiC particles [20,25,26,41].

Moreover, the maximum amount of the SiC nano-particle incorporation is attained at the $C_{\text{SiC}} = 5 \text{ g}/\text{L}$ in the whole range of the

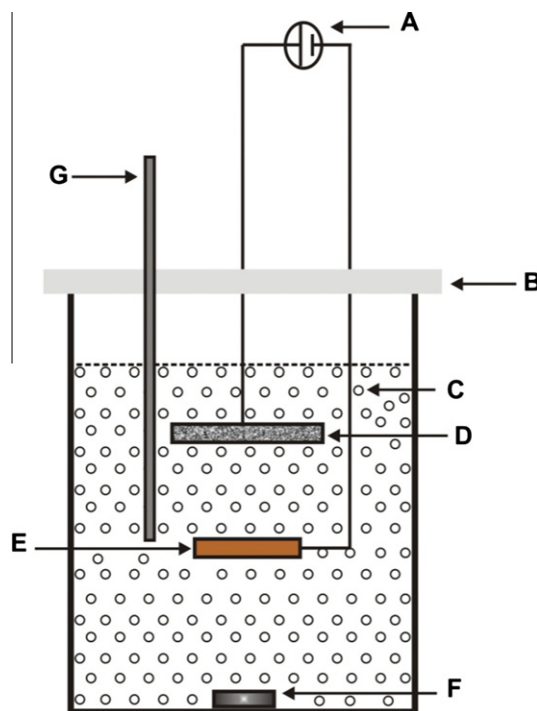


Fig. 1. Schematic illustration of the sediment co-deposition (SCD) setup; (A) DC power supply, (B) epoxy cover, (C) particles, (D) anode, (E) cathode (substrate), (F) magnetic bar and (G) external pH–temperature probe.

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