



# Effect of particle size and co-deposition technique on hardness and corrosion properties of Ni–Co/SiC composite coatings

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## ABSTRACT

Ni–Co/SiC alloy matrix composite coatings were electrodeposited in a modified Watt's bath containing micro and nano sized SiC particles by using conventional electro-co-deposition (CECD) and sediment co-deposition (SCD) techniques. The deposits were characterized using SEM, EDX and XRD analyses, and microhardness and potentiodynamic polarization measurements. The maximum incorporation of the SiC micro- and nano-particles was obtained using the SCD technique at deposition current densities of 2 and 3 A/dm<sup>2</sup>, respectively. It was found that in the composite coatings, incorporation of SiC particles improves the microhardness of unalloyed Ni and Ni–Co alloy matrices. The nanocomposite coatings exhibit higher microhardness values than microcomposite ones. The potentiodynamic polarization measurements in 3.5% NaCl solution revealed that the corrosion resistance of the Ni–Co/SiC nanocomposite coatings is much higher than the Ni–Co alloy and Ni–Co/SiC microcomposite coatings. Moreover, corrosion resistance of Ni–Co/SiC nanocomposite coatings deposited by SCD technique is higher than the ones deposited by CECD technique. Corrosion resistance of the studied Ni–Co/SiC composite coatings was considerably affected by Co content, SiC particle size and content. Hardness enhancement was related to the structural features, and corrosion behavior was discussed based on the formation of corrosion micro cells, diminishing the effective metallic area, and increasing and hindering the corrosion paths.

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## 1. Introduction

Many attempts have been made to find methods for enhancing the surface properties of metal components to protect against corrosion and wear and reducing their costs since all of them are the effective factors in the degradation of industrial parts. Electro-deposition is considered as one of the most important and cost effective industrial techniques for producing protective coatings. It is conducted at normal pressure and ambient temperatures and provides high deposition rate and high throwing power. Electro-deposition of Ni and Ni-alloy coatings has found widespread use in many industrial fields [1,2]. Compared to Ni coatings, Ni based composite coatings provide superior mechanical properties and higher corrosion and wear resistances [3]. Therefore, the interest for electro-deposition of Ni based composite coatings has increased due to their excellent properties.

Composite electro-deposition is a method of codepositing insoluble particles suspended in an electrolyte with metallic cations. Over the past decades, successful co-deposition of different types of particles such as oxide [4–6], carbide [7,8], polymer [9,10], nitride [11,12], diamond [13], graphite [14] and metallic [15] particles has been reported. Among the various composite coating systems produced by

combination of different matrix and reinforcement materials, Ni/SiC composite coatings have been investigated extensively [8,16,17] for exploring the impact of the co-deposition conditions and the properties of the deposited coatings. Adjusting electro-deposition parameters can improve the properties of composite coatings [18]. Recently, more attention has been focused on the synthesis of composite coatings by co-deposition of reinforcing particles in the alloy matrices like Ni–W [19], Zn–Ni [20,21], Ni–Fe [22] and particularly Ni–Co alloys [23–25]. However, only few investigations have been made for the electro-deposition of Ni–Co/SiC nanocomposite coatings [25–29].

One of the great interests in the synthesis of composite coatings is the higher values of incorporated particles. The extent of the particle incorporation not only depends on the deposition parameters such as electrolyte composition, pH values, presence of additives, deposition current type and density, but is also greatly influenced by the co-deposition technique. Sediment co-deposition (SCD) technique is an effective method to increase the particle incorporation [14,30]. Unlike the conventional electro-co-deposition (CECD) technique, in the SCD technique the electrodes are set horizontally in the electrolyte. In the CECD technique particles are suspended in the electrolyte under continuous stirring, but in the SCD technique periodic stirring is applied to allow particles to be suspended in the electrolyte and sediment on the cathode. Therefore, the gravity force provides additional tendency for particle settling in the SCD technique.

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Limited data is available in literature concerning the effect of SiC particle size and co-deposition technique on the properties of Ni-Co/SiC composite coatings [25–29]. Corrosion behavior of composite coatings has been studied extensively [4,8,30–34]. Many published reports have usually focused only on determination of the corrosion rate of electrodeposited composite coatings [4,31–33]. However, a complete understanding of how the microstructural features in conjunction to the various deposition parameters are affected by the corrosion behavior and corrosion mechanisms requires more detailed studies.

In the present work, the CECD and SCD techniques have been applied for electro-deposition of Ni-Co/SiC composite coatings in a modified Watt's bath containing SiC micro- and nano-particles. The Ni-Co alloy was chosen as the matrix material as it has been previously reported to possess promising properties for Ni-Co alloy coatings [35–37]. The effects of particle size, co-deposition technique and deposition current density were investigated on microhardness and corrosion resistance of composite coatings. The goal was to produce Ni-Co/SiC composite coatings with higher microhardness and corrosion resistance.

## 2. Experimental

Electro-deposition of Ni-Co/SiC composite coatings was carried out in a modified Watt's bath by adding SiC micro- and nano-particles to the electrolyte using two different techniques; (i) the CECD technique wherein electrodes were set vertically in the electrolyte and (ii) the SCD technique wherein electrodes were set horizontally in the electrolyte. During the deposition process, the electrolyte was magnetically stirred at 350 rpm agitation rate. Electrolyte stirring was performed continuously in the CECD technique and intermittently in the SCD technique with 10 s resting time after each 300 s stirring time. The schematic illustration of the CECD and SCD setups used in this work is shown in Fig. 1.

The electro-deposition bath was prepared using Merck analytical grade reagents and contained 250 g/l of  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$ , 50 g/l of  $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ , 40 g/l of  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$ , 40 g/l of  $\text{H}_3\text{BO}_3$ , 0.35 g/l of  $\text{NaC}_{12}\text{H}_{25}\text{SO}_4$  and 5 g/l of SiC particles. In preliminary studies, a SiC bath concentration of 5 g/l and a pH of 4.3 were determined as optimum conditions leading to the maximum incorporation of the SiC nanoparticles in the deposited coatings. The micro and nano sized SiC particles used in this study had an average particle size of 20 nm and 10  $\mu\text{m}$ , respectively. The utilized SiC particles had high purity (>99%) and were manufactured by Plasmachem GmbH of Germany. The coatings were deposited on copper plate substrates as cathode and a pure nickel was used as anode by employing

a DC power supply at various current densities (1–4  $\text{A}/\text{dm}^2$ ). The distance between the anode and the cathode was 3 cm. The temperature of the bath was maintained at 45 °C. The pH was adjusted by  $\text{H}_2\text{SO}_4$  and NaOH at around 4.3.

The copper substrates (cathodes) were mechanically polished with silicon carbide abrasion papers and ultrasonically cleaned in ethanol and acetone for 10 min, sequentially and washed in distilled water after the cleaning process. The substrates were activated in 10%  $\text{H}_2\text{SO}_4$  for 60 s before final use. Prior to deposition, the electrolyte containing particles were alternatively subjected to ultrasound waves produced using a Hielscher-UP100H disperser and vigorous magnetic agitation (~700 rpm) using a magnetic stirrer for 24 h before electro-deposition to achieve a highly uniform dispersion of particles. The agitation started by a 60 minute ultrasonic dispersion followed by repeated cycles of a 315 minute magnetic stirring and a 30 minute ultrasonic dispersion (during 24 h), and the last step was the ultrasonic dispersion. After deposition, in terms of removing loosely adsorbed particles from the cathode surface, the coatings were ultrasonically cleaned in the distilled water for 5 min after the electro-deposition process. The deposition time was adjusted to attain a coating thickness of around 30  $\mu\text{m}$ .

The surface morphology and cross section of the composite coatings was investigated by using a CamScan MV2300 scanning electron microscope (SEM). An Oxford Energy Dispersive X-ray detector (EDX) coupled to SEM was utilized to determine the chemical analysis and SiC content of the coatings. Five randomly chosen areas were analyzed (at 500 $\times$  magnification) and an average value was calculated. Standard deviation of five independent measurements is reported as error bars. The phase structure, average grain size and preferred orientation of coatings were determined from XRD patterns recorded in the Bragg–Brentano configuration using a D8 ADVANCE-BRUKER AXS X-ray diffractometer operated at 40 keV and 40 mA with the  $\text{Cu-K}\alpha$  radiation ( $\lambda = 1.5406 \text{ \AA}$ ). Integral peak width was used to estimate the crystallite size of the coatings after excluding the instrumental broadening. Instrumental broadening was determined from the XRD pattern of a standard  $\text{LaB}_6$  sample. The preferred orientation was evaluated by calculation of the texture coefficient. The microhardness of the coatings was measured using a MDPEL-M400 GL microhardness tester equipped with a Vickers indenter under indenting load and time of 50 g and 10 s, respectively. Hardness measurements were performed on the polished cross-section and top surface of the coatings. An average of ten different measurements from the top of each deposit was reported as coating microhardness.

Potentiodynamic polarization measurements were used to investigate the corrosion resistance of composite coatings. Electrochemical measurements were carried out in the non-aerated 3.5% NaCl corrosive medium using an EG&G-Parstat 2263 potentiostat/galvanostat system. A saturated calomel electrode (SCE) was used as the reference electrode and a platinum electrode with an exposed area of about 10 times larger than the working electrode was used as the counter electrode. Potentiodynamic sweeping was performed in the potential range of  $\pm 250 \text{ mV}$  with respect to  $E_{ocp}$  by 1 mV/s sweeping rate. The corrosion current density of the deposits was calculated using the Stern–Geary equation from the polarization measurement.

## 3. Results and discussion

### 3.1. Electro-deposition

Fig. 2 illustrates the effect of deposition current density on the SiC micro- and nano-particle content in the Ni-Co/SiC composite coatings deposited by SCD technique. For a given amount of particle concentration (5 g/l SiC), SiC nano-particle content in the deposits is increased with increasing deposition current density and reaches to a maximum value at 3  $\text{A}/\text{dm}^2$ . SiC nano-particle content is decreased at the higher deposition current densities. The same trend is observed for the

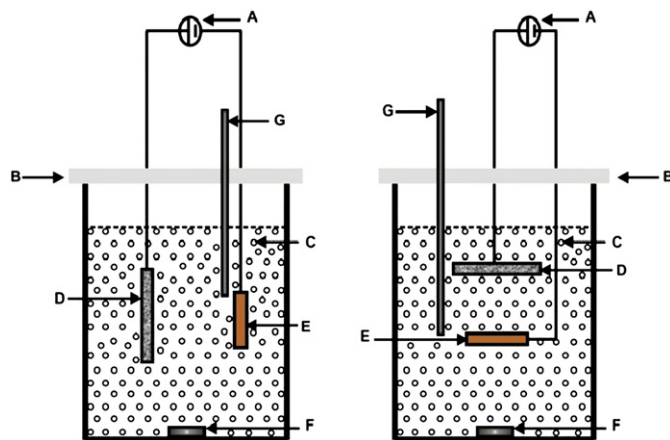


Fig. 1. Schematic image of the co-deposition setups; (A) DC power supply, (B) Epoxy cover, (C) SiC particles, (D) anode, (E) cathode, (F) magnetic bar and (G) external pH-temperature probe.

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