



Novel bilayer Zn–Ni/Ni–Co–SiC nanocomposite coating with exceptional corrosion and wear properties by pulse electrodeposition

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ABSTRACT

This work reports the development of a novel bilayered Zn–Ni/Ni–Co–SiC nanocomposite coating on mild steel (MS) by electrodeposition. The top Ni–Co–SiC nanocomposite layer, when deposited directly on MS (monolayer) shows a hardness of 734 HV and a corrosion current density of $\sim 28 \mu\text{A}/\text{cm}^2$ in 3.5 wt% NaCl. However, the lack of adherence of the coating with MS results in poor wear and scratch resistance. Mild steel is anodic to Ni–Co–SiC nanocomposite and any kind of discontinuity in the coating may lead to severe galvanic corrosion. This problem has been successfully overcome with the incorporation of an anodic (with respect to mild steel) Zn–12%Ni under-layer in between the electrodeposited Ni–Co–SiC nanocomposite coating and MS, which is adherent to both the materials. This bilayer coating has a hardness of 760 HV and corrosion current density of $\sim 19 \mu\text{A}/\text{cm}^2$ in 3.5 wt% NaCl which are slightly better than that of the monolayer. Further, the bilayer coating assembly exhibits exceptionally low volume loss under wear ($\sim 350 \text{mm}^3$) compared to bare mild steel ($\sim 1700 \text{mm}^3$). This Zn–Ni/Ni–Co–SiC nanocomposite bilayer coating has the potential to emerge as hard, wear resistant and anti-corrosive coating which is essential in the field of heavy duty machines.

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1. Introduction

Incorporation of uniformly distributed nano-particles within a metallic matrix up to a certain volume fraction improves the electrochemical and mechanical properties [1–6]. The Ni based electrodeposited nanocomposite coatings reinforced with SiC particles on metallic substrate are known to provide enhanced corrosion protection and tribological properties [7–12]. Studies have shown that further improvement in the properties can be achieved by changing the matrix from pure Ni to Ni–Co alloy [13–15]. The corrosion properties of Ni–Co–SiC nanocomposite coatings have been studied extensively [15–20], but there are not many reports regarding the wear resistance properties of Ni–Co–SiC coatings [13–16]. Any nanocomposite coating beyond a certain thickness and/or amount of incorporation does not exhibit very good adhesion to the substrate resulting in poor wear resistance as the coating tends to crack/peel off from the substrate. There are many factors that affect the adhesion between the electrodeposited

ceramic reinforced nanocomposite coating and the substrate such as residual stress, mismatch of lattice parameters and coefficient of thermal expansion of nanocomposite coating and substrate. In order to improve the adhesion of coating with the substrate, the uniform coating (single layer composite with uniform dispersion of ceramic particles) can be replaced with a functionally gradient composite coating (FGM) or a simple metallic under coat/bond coat which binds well with both the coating and the substrate can be used [21,22]. In addition to that, in case of uniform coating any damage in the coating will provide a path for the corrosive media to come in contact with the substrate causing a failure.

The objective of the present investigation is to improve the wear and corrosion resistance of mild steel (MS) by providing an undercoat which bonds well with both the mild steel (MS) substrate and Ni–Co–SiC coating. In presence of Ni–Co, MS undergoes massive galvanic corrosion causing a catastrophic failure. It would be useful if the undercoat is anodic with respect to the MS. In that case if any discontinuity is present in the Ni–Co–SiC layer, the underlayer will corrode providing cathodic protection to MS.

Zn coating, for a long time, has commercially been used for corrosion protection as a sacrificial anode for steels. However, it is

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found that an addition of Ni to Zn can lower the corrosion rate. It has been reported in the literature that Zn–Ni alloy coating containing about 10–14 wt % Ni with single γ phase ($\text{Ni}_5\text{Zn}_{21}$) gives very good corrosion and wear resistance and this alloy is still anodic with respect to MS [23–25]. Hence, an electrodeposited Zn–Ni coating in between the MS substrate and Ni–Co–SiC nano-composite coating can improve the adherence of Ni–Co–SiC coating with the substrate and can also act as an anodic undercoat which will protect the steel structure by providing cathodic protection until repairing of the Ni–Co–SiC layer is made (buffer period).

The aim of the present investigation is to develop a novel Zn–Ni/Ni–Co–nanoSiC bilayer composite coating on MS by electrodeposition which combines the benefits of adhesion of a cathodically protecting Zn–Ni underlayer with the superior hardness of a Ni–Co–SiC layer which provides resistance to abrasive wear. There have been prior reports on bilayer protective coatings over structural low carbon steels by Sadeghimeresht et al. [26,27] where the top layer is sacrificial and provides cathodic protection, and the middle/underlayer is inert relative to mild steel and provides physical protection. The main drawback of this design is that if the top layer is removed (either by local corrosion or wear) and underlayer is exposed, it leads to severe galvanic corrosion. The uniqueness of the present study is that the top Ni–Co–nanoSiC layer provides physical protection (along with wear) and the middle Zn–Ni layer (undercoat) provides cathodic protection. Therefore, even if the upper layer is mechanically damaged, the underlayer will act as the sacrificial anode protecting the MS till the top layer is repaired or replaced. The damage/discontinuation of the top Ni–Co–nanoSiC layer can be easily detected from the change in open circuit potential.

2. Experimental

2.1. Materials and Methods

Pulse electrodeposition is carried out with Autolab PGSTAT 30 on MS substrates with an exposed area of $2 \text{ cm} \times 1 \text{ cm}$. The MS substrates are polished and pre-treated with 2% HCl solution. After electrodeposition, the as deposited samples are cleaned with distilled water followed by ultrasonic cleaning in ethanol. The bath composition and electrodeposition parameters for Zn–Ni and Ni–Co–SiC are tabulated in Table 1 respectively.

2.2. Materials characterization

Microstructural study and EDS analysis are carried out using

FESEM (FEI Quanta FEG 250) at an operating voltage of 20 kV. Bruker X-ray Diffractometer (D8 Advance) with a cobalt target ($\text{Co } K_{\alpha} = 0.1789 \text{ nm}$) is used for XRD study at an operating voltage and beam current of 40 kV and 30 mA, respectively with a step size of 0.02. Hardness of the samples is measured by Leica Vickers Micro Hardness Tester (UHLVMHT 001) at a load of 25gf with a dwell time of 10 s. The roughness of as deposited samples is studied by the contact mode of Dektak 150 profilometer. The Micro Scratch Tester (MST³) by Anton Paar is used for scratch test by increasing the load stepwise from 30 mN to 30 N at a loading rate of 60 N/min and speed of 2 mm/min. The Rockwell type diamond indenter of 100 μm radius is used for the scratch test. Ball on disc wear test is carried out using DUCOM TR-208-M1 at various normal loads keeping constant disc speed of 10 rpm for 900 s. Test ball is a hardened steel ball of 3 mm diameter. The corrosion resistance of the coatings is studied by the measurement of open circuit potentials at 25 °C. The reference electrode is a saturated Ag/AgCl electrode. A platinum electrode (99.99%, 1 cm^2) is used as the counter electrode. The E_{corr} is monitored for 10 min. The potentiodynamic polarization study is carried out at a scan rate of 5 mV/s in 3.5 wt% NaCl solution.

3. Results and discussion

3.1. Electrodeposition of Zn–Ni alloy coating on MS

Temperature has a significant effect on the Ni content of the electrodeposited Zn–Ni coating [24,25,28,29] and also amount of Ni in the coating plays an important role in determining the corrosion resistance of Zn–Ni coating [23,25,28]. Therefore, Zn–Ni coatings are deposited at temperatures of 25, 35 and 45 °C. The Ni content increases from 7 wt % at 25 ± 2 °C to 21 wt % at 45 ± 2 °C. This is due to the fact that the Fe-group metals are known to be electrochemically inert for ionic exchange reactions. They are generally characterized by very low exchange current densities unlike zinc, which shows high exchange current density. The kinetic cathodic reaction parameters of the iron-group metals are so unfavourable that they destroy the thermodynamic nobleness of these metals with respect to zinc. Increasing temperature increases the transition current density, which in turn enhances the deposition rate of Nickel [18,21]. Fig. 1 shows the BSE micrographs of the Zn–Ni alloy coatings on MS electrodeposited at different temperatures. The composition of coating is found from EDS elemental analysis. The amount of Ni in the coating is found to be 7.1 wt %, 11.80%, and 21.04 wt % when deposited at 25 ± 2 , 35 ± 2 , and 45 ± 2 °C, respectively. According to prior reports Zn–Ni alloy containing 10–14 wt% Ni gives maximum corrosion resistance and

Table 1
Bath Compositions and electrodeposition parameter.

Coating	Bath composition	Concentration (g/L)	Electrodes	Electrodeposition parameters				
Ni–Co–SiC	$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$	300	Working electrode	Zn–Ni on MS (2 cm^2)	Current Density (A/cm^2)	0.3		
	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	40		t_{on} (ms)	1			
	SiC	15	Counter Electrode	Platinum (2 cm^2)	t_{off} (s)	10		
	H_3BO_3	16			Temperature (°C)	25 ± 2		
	$\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$	30			RPM	400		
	CTAB	0.25			Current Density (A/cm^2)	0.06		
	Na-Saccharin	0.2			t_{on} (ms)	0.25		
Zn–Ni [18]	ZnCl_2	81.77	Working electrode	MS (2 cm^2)	t_{off} (ms)	4.75		
	$\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$	213.92						
	H_3BO_3	30.91	Counter Electrode	Nickel (8 cm^2)	Temperature (°C)	35 ± 2		
	NH_4Cl	123.03					RPM	400
	SDS	0.2						
	Gelatin	5						
	Saccharose	10						

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