



Optimum amount of graphene oxide for enhanced corrosion resistance by tin-graphene oxide composite coatings



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ABSTRACT

Incorporation of varying amounts of graphene oxide (GO) into conventional Sn coating electrodeposited over mild steel resulted in significant changes in the corrosion resistance performance of the coatings. An aspect of “optimum” GO amount for best corrosion resistance for the Sn-GO composite coatings was explored. All the Sn-GO coatings exhibited compact and crack free morphology. Incorporation of GO facilitated growth along the low index planes and suppressed it along the high index planes. Corrosion behaviour of the coatings was examined through potentiodynamic polarisation and electrochemical impedance spectroscopy methods. It was observed that with the addition of GO, the corrosion rate initially decreased to a minimum value and then, upon continued addition of GO, it increased to a value that was even higher than the pure Sn coating. In the case of lower GO amounts, the textured growth coupled with impermeability of GO increased the corrosion resistance whereas, at higher GO concentrations, the galvanic coupling between GO and Sn dominated, leading to lowering of the corrosion resistance.

1. Introduction

Coatings have been traditionally used to protect the underlying substrate against corrosion [1–3]. Over the years, substantial efforts have, therefore, been devoted towards engineering the microstructure and morphology of coatings in order to enhance their corrosion resistance performance [4–9]. Nanocomposites fabricated by adding graphene/graphene oxide (GO) into polymers have also been developed extensively as barrier coating material over low carbon steel substrates [10, 11]. Recent studies have illustrated that addition of graphene and GO in conventional protective metallic coatings significantly enhances their corrosion resistance performance [12–18]. This is due to changes in morphology and microstructure of the coatings resulting from the incorporation of graphene/GO in addition to the chemical inertness and impermeability, towards the electroactive media, offered by graphene and GO [19–29]. One important question that has not been addressed yet is: is there an optimum amount of graphene/GO which maximizes the corrosion resistance performance? And what happens when the amount of graphene/GO increases well beyond this optimum value?

The above questions are addressed in the present work. The candidate system chosen is Sn-GO composite coating electrodeposited over mild steel substrate. Sn is widely used as coating material in food industry and as a vital soldering component. Sn coating has also been traditionally used for protecting steel structures against corrosion

[30–34]. Berlia et al. [35] have investigated the electrochemical behaviour of Sn-graphene coatings. They have reported that the addition of graphene in Sn coating results in noticeable changes in the morphology, texture and crystallite size of the coating when compared to pristine Sn coating. These changes, they argue, are responsible for the observed significant improvement in the corrosion resistance of the Sn-graphene composite coating. One aspect that is not addressed by Berlia et al. [35] is how the microstructure and the electrochemical properties of the Sn-graphene composite coating vary as a function of the amount of graphene in the coatings? In the present work, GO was used instead of graphene as used by Berlia et al. [35]. Unlike graphene which is hydrophobic, the hydrophilic nature of the GO [36, 37] makes its dispersion more uniform and stable in aqueous based electrolyte used for the electrodeposition experiment. Stable dispersion of GO is necessary for uniform distribution of GO in the electrodeposited coating. We show here that there exists an optimum with respect to the amount of GO in the Sn-GO composite coating for achieving maximum corrosion resistance performance.

2. Experiment

2.1. GO synthesis

GO was synthesized using the modified Hummer's method [38]

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Table 1
Composition of electrolyte bath and deposition parameters used for electro-deposition of Sn only and Sn-GO composite coatings.

Sample name	Bath composition	Concentration	Operating parameters
Sn only	SnSO ₄	20 g/l	pH 4.0
	C ₂ H ₃ O ₂ Na	20 g/l	Time 20 min
	C ₆ H ₁₁ NaO ₇	140 g/l	Stirring RPM 180
	SLS	0.500 g/l	Temperature 25 ± 2 °C
Sn GO1	Sn only + concentration of GO	0.125 g/l	Current density 6.25 mA/cm ²
Sn GO2		0.250 g/l	
Sn GO3		0.375 g/l	
Sn GO4		0.500 g/l	
Sn GO5		0.625 g/l	
Sn GO6		0.750 g/l	
Sn GO7		0.875 g/l	
Sn GO8		1.250 g/l	
Sn GO9		1.500 g/l	
Sn GO10		1.875 g/l	
Sn GO11		2.500 g/l	

involving the following steps. 2 g of graphite powder (150 mesh, 99.99% metal base), 2 g NaNO₃ and 75 ml of sulphuric acid were taken in a glass beaker under continuous stirring. 6 g KMnO₄ was slowly added into this solution. During this process, the setup was kept under an ice bath and the temperature of the solution was maintained below 5 °C. After the addition of KMnO₄, the solution was stirred continuously for 24 h. After this, the solution temperature was raised to 35 °C and kept for 15 min and then to 95 °C and kept again for 15 min and then cooled to room temperature. The solution was diluted by adding de-ionised water. H₂O₂ (30%w/v) was then added into the solution to terminate the reaction which was characterised by yellow-brown colour of the solution.

2.2. Electrodeposition of Sn-GO composite coatings

For the electrodeposition experiment, a DC power supply with two terminals was used. A mild steel plate with dimensions 2.0 cm × 2.0 cm × 0.5 cm was used as cathode. Platinum foil of dimensions 2.5 cm × 2.0 cm × 0.5 cm was used as anode. The mild steel plate was pickled in 10% HCl solution, thoroughly washed with de-ionised water and then polished using SiC abrasive papers ranging from 80 to 2500 grit sizes. Coatings of Sn and Sn with varying amounts of GO (Sn-GO composite coating) were prepared using a sulphate-based bath whose composition is provided in Table 1. SnSO₄ as Sn²⁺ ion source, sodium acetate anhydrous (C₂H₃O₂Na) as buffer, sodium gluconate (C₆H₁₁NaO₇) as complexing agent and sodium lauryl sulphate (C₁₂H₂₅NaOSO₃; SLS) as surfactant were used. An electrolyte bath of 50 ml was used for each coating. Electrodeposition parameters used for Sn and Sn-GO composite coating are also provided in Table 1.

2.3. Characterisation

UV-Vis spectroscopy was conducted using Perkin Elmer Spectrometer, Lambda 35. Fourier transform infrared (FTIR) spectroscopy profile was obtained using Bruker Tensor 2 (with Pt ATR module)

instrument. X-ray diffraction (XRD) profiles were obtained using PANalytical JDX-8030 diffractometer employing a Cu K_α (λ = 0.154 nm) X-ray source. FEI Tecnai F-30 transmission electron microscope (TEM) operating at 300 keV was used for imaging as-synthesized GO and coating cross-section samples. Nanosurf AFM instrument (Switzerland) in tapping mode was employed obtaining the atomic force microscopy (AFM) images of as synthesized GO. Morphology and composition of the coatings was determined using ESEM QUANTA 200 scanning electron microscope (SEM) coupled with energy dispersive X-ray spectroscopy (EDS) detector operating at 20 keV. Cross-sectional samples of the coatings were prepared using FEI Helios SEM-focused ion beam (SEM-FIB) equipment using Ga ion source for cross-sectional milling at 9.3 nA current and Pt deposited on top (deposition current 0.23 nA) for protection from contamination of coatings by focussed Ga ion beam. Corrosion studies were performed using a CHI electrochemical workstation (CHI Instruments, model no. 604E). Corrosion studies included electrochemical impedance spectroscopy (EIS) and Tafel polarisation. Aerated and neutral 3.5 wt% NaCl solution was used as electrolyte. Electrodeposited sample with 1 cm² of the coating area exposed to the electrolyte was used as working electrode. Platinum foil of dimensions 2.5 cm × 2.0 cm × 0.5 cm was used as auxiliary electrode and a standard Ag-AgCl (sat. KCl) electrode was used as the reference electrode.

3. Results and discussion

XRD profile, UV-VIS spectrum and FTIR spectrum of as-synthesized GO is respectively provided in Fig. 1(a), (b) and (c). In the XRD profile, the high intensity peak at 10.18° 2θ and the corresponding interplanar spacing of 0.87 nm represents (001) plane of GO [39, 40]. In Fig. 1(b), the UV-VIS spectrum shows an absorption maximum at 228 nm and a shoulder peak at 302 nm. The peak at 228 nm corresponds to π-π* transition of C=C and the shoulder peak at 302 nm corresponds to the n-π* transition of C=O present in GO [41, 42]. Formation of GO was also confirmed by the characteristic stretching frequencies observed in the FTIR spectrum (Fig. 1(c)). The C=C (1500–1600 cm⁻¹), O–H (3300–3600 cm⁻¹), C–O (1050–1150 cm⁻¹), C=O (1700–1725 cm⁻¹) troughs are indicative of hydroxyl and carboxyl groups of GO [41, 42]. Representative, TEM bright field image of as-synthesized GO sheet is provided in Fig. 1(d). Representative Atomic force microscopy (AFM) topographical image of as-synthesized GO is provided in Fig. 1(e). Average thickness of the GO sheet determined from several AFM images was 1.9 ± 0.4 nm.

SEM micrographs obtained from Sn and Sn-GO composite coatings are provided in Fig. 2(a–c). It can be observed that all the coatings exhibit similar, compact and crack free morphology. Coating composition values obtained using the SEM-EDS technique are provided in Table 2. A steady increase in the carbon content (wt%) reveals that the increase in GO concentration of the electrolyte bath increased the amount of GO in the composite coatings.

XRD profile of all the coatings are provided in Fig. 3(a–c). Characteristic XRD peaks of only Sn phase are visible in the XRD patterns of Sn and Sn-GO composite coatings. Average crystallite size of the Sn grains was calculated using the Scherer's formula [43, 44] and the full width half maxima (FWHM) of five highest intensity XRD peaks for each coating. The variation in the crystallite sizes with GO content in the coatings is shown in Fig. 3(d). It can be observed that the addition of GO did not produce any noticeable change in the average crystallite

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