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DEVELOPMENT OF NI-SI3N4 NANOCOMPOSITES BY ELECTRODEPOSITION

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ABSTRACT

Nickel–Si3N4 composites were generated by means of Direct current (DC) Pulse Current (PC) and Pulse-reverse (PR) Electrodeposition technique. The corrosion rate was calculated for these composites by electrochemical methods such as polarisation and impedance methods. Composites were characterised by scanning electron microscope (SEM) and X-ray diffractometer (XRD) to evaluate their microstructures. Composites obtained by all these three methods were compared with each other.

KEYWORDS: Pulse Electrodeposition, Nickel-Si3N4 composite coatings, XRD, SEM

INTRODUCTION

Electroplating of composites is a process of depositing fine particles of metallic, non-metallic or polymeric compounds in plated layer along with metal to enhance the mechanical properties [1]. Electrodeposition is considered as one of the simple techniques for developing composites. This method is adopted at low temperature and composites can be generated in small tenure. In Electrodeposition method, inert particles are suspended in a plating bath and these particles are incorporated in the coating during metal deposition. Zinc, nickel, silver and copper are generally used as the base metal. The particles used in these coatings can be Inorganic oxides, carbides and polymers [2–4]. Nanocomposites containing dispersion of second phase particles usually give a variety of novel properties. The emergence of technology revives Electrodeposition techniques for synthesizing a variety of new nanostuctured materials such as nanocomposites, nanowires, and nanomultilayers[5-6].

Pulse electrodepsoition is the new emerging technology in the field of composites. In this method more uniform and higher percentage of the particles can be codeposited with the metal. Pulse electrodepsoition and Pulse reverse electrodepsoition are used to achieve maximum technological applications. In this work Ni-Si3N4 nano composites were generated by different methods such as Pulse electrodepsoition, Direct electrodeposition and Pulse reverse electrodepsoition methods. All coatings were characterised and evaluated their corrosion resistance properties [7-9].

EXPERIMENTAL DETAILS

Ni–Si3N4 composite coatings were developed from a nickel electrolyte containing Si3N4 nano particles. Table 1 provide the constituents and parameters of the bath. Scanning electron microscope (EVO MA18 with Oxford EDS(X-act)) was used to analyze the surface morphology of the samples. X-ray diffraction (XRD) patterns were obtained using an X-ray diffractometer (RigakuMiniflex 600 powder diffractometer). Microhardness was estimated by using Clemex Microhardness tester.

TABLE 1. Electrolytic bath composition

Composition	**g/Litre**
$NiSO_4.6H_2O$	38
Na_2SO_4	66
H_3BO_3	20
Si_3N_4 <50nm particle size	3
Temperature	40^0c
pH	4
Current density (DC)	$1Adm^{-2}$
Current density (PC)	20mA
Duty Cycle (PC)	50%
Current density (PR)	10mA
Duty Cycle (PR)	26%

Corrosion behaviour of the composites obtained by DC, PC and PR method were analysed by using Tafel and Impedance methods. The corrosive medium used for the experiments was 3.5 wt.% NaCl. The corrosion

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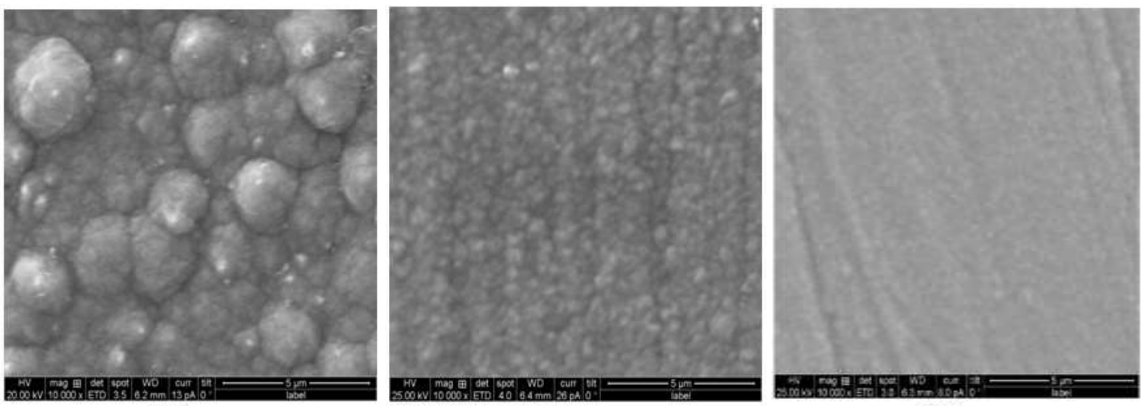
studies were conducted in a simple glass cell by using the Electrochemical System – Compactstat.e10800 from Ivium Technologies, Netherlands at 298 K. The Ni- and Ni–Si3N4 coated steel specimen, platinum wire and a saturated calomel electrode (SCE) were used as working, auxiliary and reference electrodes. 1 cm2 of working electrode was employed for corrosion studies

RESULTS AND DISCUSSIONS

Surface Morphology: SEM images obtained from DC, PC and PR methods are shown in figure 1. composites obtained by PC method shows more uniform and regular development of the crystals. In PR method shows less uniform arrangement of the crystals. In DC method it shows completely disturbed grains compared to PC and PR method. These data shows that, composites obtained by PC method are more suitable one for developing the Ni-Si_3N_4 composites.



DC PR PC

Fig. 1. SEM Images obtained by different methods.

XRD Studies: XRD patterns of the composites obtained by DC, PC and PR method are shown in Fig. 2. The peaks in the diffractogram were indexed using the JCPDS Card no. (70-0989). Crystallite size of the composites was calculated using the Scherrer equation.

$$L = \left(\frac{K\lambda}{\beta \cos\theta} \right)$$

Average crystallite of the deposits obtained from 21 nm, 20 and 19 nm for DC , PR and PC samples respectively.

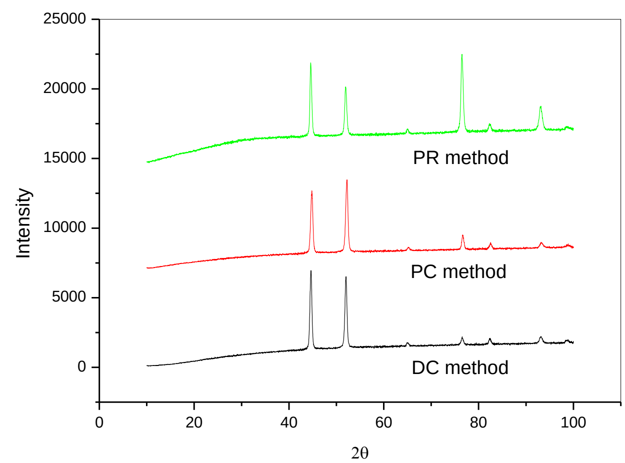


Fig. 2. XRD images of composites obtained by different methods

Microhardness: Vickers microhardness measurements were performed for Ni- Si_3N_4 nanocomposite coatings obtained by different methods. The Ni– Si_3N_4 nanocomposite coatings shows 550 HV, 450 HV and 400 HV for the

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composites obtained by PC, PR and DC method respectively. The increase in the hardness of nanocomposite coating obtained by PC method is due to grain refining and dispersive strengthening effect caused by the dispersion of Si_3N_4 particles in the composite coatings. So microhardness values of the Ni– Si_3N_4 composite coatings were increased in the composites generated by PC method.

Corrosion resistance properties

Tafel extrapolation: The corrosion resistance behavior of the Ni and Ni – Si_3N_4 coatings obtained by PC, DC and PR elctrodeposition were given in Table 2 and Fig.3. The coated specimens of 1 cm^2 area were immersed in 3.5% NaCl solution for the duration of 300 seconds to attain an equilibrium potential. From the table and figure it can be concluded that composites obtain by PC method shows more corrosion resistance than DC and PR method. In PC method more uniform grains with maximum inclusion of nano particles were observed in the coatings and by increases the corrosion resistance property.

Method of Preparation	Conc.of Si_3N_4	Rp in Ωcm^2	R_t in Ωcm^2	C_{dl}	I corr Acm^{-2}	Corrosion rate (mm/y)	ba V/dec	bc V/dec
DC	3 g/L	1061	7.634	0.0424	0.0195	0.1205	0.072	0.177
PC	3 g/L	1749	2.894	0.0370	0.0069	0.0847	0.067	0.198
PR	3 g/L	1393	4.518	0.0400	0.0075	0.1114	0.067	0.214

Table. 2. Electrochemical parameters of the coatings derived from Tafel plots and impedance data

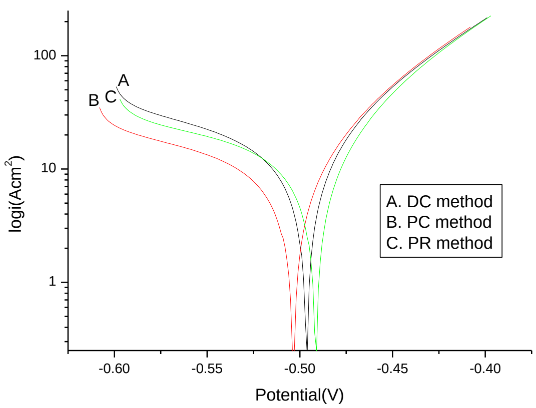

Fig. 3. Tafel Plots for composites obtained by different methods

Impedance Measurement: Nyquist plots for the coatings in a 3.5 wt.% NaCl solution are given in fig. 4. The experiment was carried out separately in the applied frequency range from 1 Hz to 100 kHz. The experimental impedance data was modelled with the electrical equivalent circuit given in Fig.5. Equivalent circuit parameters after fitted to the impedance curve are provided in Table 2. Fig 4 has two relaxation processes in the given frequency range. Coatings obtained from PC method shows higher polarisation resistance and lower double layer capacitance values compared DC and PR method. These data suggests that more corrosion resistance was observed in composites obtained by PC method.

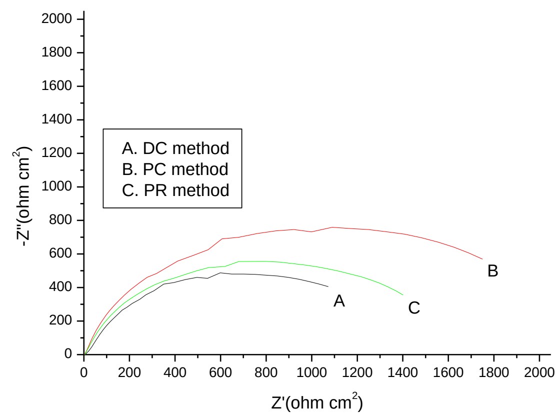

Fig. 4 Impedance Plots for composites obtained by different methods

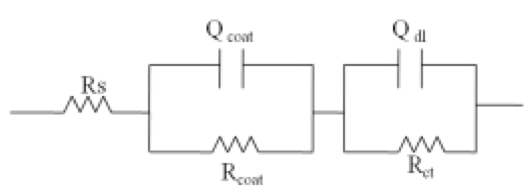

Fig. 5. Equivalent circuit

CONCLUSIONS

- Ni–Si3N4 composites were successfully obtained by the PC, DC and PR method.
- XRD studies indicate presence of Si3N4 particles in the deposit and suggest composites obtained by PC method show more uniform and small grain size.

- Deposits obtained by PC method shows good corrosion resistance compared to deposits obtained by DC and PR method.
- SEM studies reveal that uniform and small grains were obtained in the composites obtained by PC method.

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