



Electroplating of Nickel on Nickel Titanate Modified Mild Steel Surface

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ABSTRACT :

Nickel is a good electrocatalytic metal and nickel electrodes find many applications in different electrochemical fields. The nickel plated electrodes were prepared by electro-deposition technique on mild steel surface modified with in-situ deposition of nickel titanate. The SEM images shows that the nickel plating on nickel titanate modified mild steel shows better adherence than the nickel plating on bare mild steel surfaces. The extent of polarization of the nickel plating on mild steel with nickel titanate was lower than that of nickel plating on mild steel. The incorporation of nickel titanate on mild steel surface before nickel plating enhances physical, chemical and electrochemical properties of the plating film.

Keywords: Nickel plating, Nickel titanate, Mild steel, Corrosion resistance

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1. Introduction

Pure nickel electrodes and nickel coated electrodes find various applications in the field of electroanalytical chemistry. Nickel electrodes can be applied for electrochromic devices,¹⁾ alkaline batteries²⁾ and as electrocatalysts.^{3,4)} Most of these applications are originated based on the redox pair system $\text{Ni}(\text{OH})_2/\text{NiOOH}$.^{5,6)} At a potential range of less than 0.6 V, nickel dissolves from the nickel electrode surface.⁷⁾ Nickel plating is carried out either by electroplating method or electroless plating techniques. The modification of the nickel plating can be achieved by alloying with other metal or by the composite addition. Many pre and post treatment are applied to enhance the plating properties.

Titanate nano particle have caught the attention of researchers due to wide application as capacitors, high performance catalyst electrode for solid oxide fuel cell,⁸⁻¹⁰⁾ metal air batteries¹¹⁾ and gas sensors.¹²⁾ Nickel titanate has been investigated as a tribological coating to reduce friction and wear in high temperature applications.¹³⁻¹⁷⁾

Nickel titanate coating was applied to surfaces due to its high durability and high resistant to oxidizing atmosphere.^{14,18)}

The present study deals with nickel plating on mild steel specimens modified by surface impregnation of nickel titanate. The prepared nickel plated electrodes were characterized by chemical and electrochemical methods. The modified nickel plated electrodes show high corrosion resistance properties, long-term durability and good stability.

2. Experimental Methods

2.1. Substrate

The substrate selected for plating was mild steel. The composition of the substrate is given in Table 1. Rectangular strips of size 3 cm × 1 cm × 0.2 cm were cut from mild steel. These strips were abraded with fine emery papers. Then all the strips were subjected to alkaline cleaning using 5% NaOH solution, acid cleaning using 3% HCl and sensitization using stannous chloride solution (10 g/L SnCl_2 + 40 ml/L conc. HCl) Each process was carried out for 10 minutes successively and the strips were cleaned with water and

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Table 1. Composition of metal specimen (mild steel)

Sl. No.	Elements	Quantity (%)
1	Carbon	0.1
2	Silcon	0.1
3	Manganese	0.7
4	Chromium	0.02
5	Nickel	0.01
6	Molybdenum	0.10
7	Fe	Balance

dried. The dimension of the plating area is 2 cm × 1 cm. The un-required area was covered and insulated using a Teflon tape. These strips were then subjected to nickel titanate impregnation before nickel plating.

2.2. Preparation and incorporation of Nickel titanate on mild steel

For preparing and deposition of NiTiO₃, nickel chloride and titanium tetrachloride were used as the starting material. TiCl₄ (0.2 mol) and NiCl₂ (0.2 mol) are respectively dissolved in 1000 ml of distilled water. An aqueous solution containing ions of titanium and nickel in an equimolar ratio is prepared by admixing 100 ml TiCl₄ solution and 100 ml of NiCl₂ solution (solution 1). A mixed solution is prepared by admixing 20 ml of 30% H₂O₂ solution, 15 ml of 28% aqueous ammonia solution and rest of distilled water (solution 2). The mild steel specimen prepared after pre-treatment procedure was immersed in solution 1. The mixed solution (solution 2) is added drop wise to the aqueous solution containing ions of titanium and nickel in an equimolar ratio (solution 1 with mild steel specimen) with stirring to precipitate complex peroxide corresponding to nickel titanate on metal surface. The specimen is then dried and kept in desiccators. The nickel titanate adhere on the surface of mild steel were scratched out and subjected to XRD analysis using Phillips Analytical X-ray spectrophotometer and average crystalline size was calculated using Scherer equation.

2.3. Ni electroplating bath

The nickel plating is carried out using a nickel plating bath having composition given in Table 2. The Plating conditions are, pH: 4.0, Temperature: 30°C Current density: 100 mA/cm², Deposition time: 10 minutes.

Table 2. The standardized bath used for nickel electroplating

Chemicals	Composition (g/L)
NiSO ₄ .6H ₂ O	300
NiCl ₂ . 6H ₂ O	44
H ₃ BO ₃	30

2.4. Characterization of the nickel plating

The plated substrates were merely observed visually so as to determine visible defects such as blisters, pits, pimples or cracks. The extent of adhesion of the deposit was determined by rubbing the plated substrate using standard metallic brush. The microstructure of the plates were analyzed by Optical microscope (magnification X 20) and Scanning Electron Microscope (SEM) of model HITACHI, S-2400; at a voltage of 15 kV

The corrosion characteristics of the prepared plates were analyzed by polarization technique using a self assembled tri-electrode setup. The polarization study of was carried out in 3 % NaCl solution. The prepared nickel plates with exposed area 1 cm² were used as the working electrode, the SS 316L electrodes were used as the counter electrode. Saturated Calomel Electrode was used as the reference electrode. Rheostat was used to regulate the current. The potential developed due to the current flow is measured using a multimeters.

The long-term stability of the electrodes were noted by dipping them in media like 3% NaCl solution. The potentials were measured with respect to Saturated Calomel Electrode (SCE).

3. Results and Discussions

3.1. Nickel titanate coating on mild steel

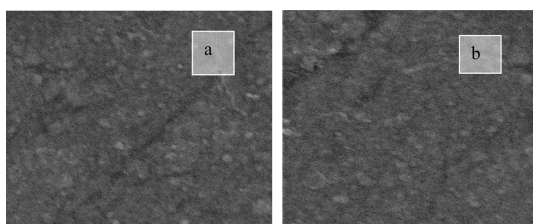
Table 3 shows the XRD analysis data of powdered samples scratched from the nickel titanate impregnated on mild steel surfaces. The peaks in the XRD spectrum were corresponding to nickel titanate with rhombohedral phase. The particle size was calculated using the Scherer formula and found that the particle size of nickel titanate lies between 96-127 nm.

3.2. Physical characterization

On visual observation the nickel plated electrodes shows shiny smooth surface without pores and blistering. The nickel plating on mild steel with and without surface impregnation of nickel titanate was scratched with standard brush. The more peeling was noticed on

Table 3. XRD data of powdered sample collected from the mild steel surface incorporate with nickel titanate

Specimens	Peak at 2θ	Peak corresponding	$\beta \frac{1}{2}$ (radians)	Particle size (nm)
Powder sample collected on the surface of mild steel	37.6	NiTiO ₃	0.001422	96
	34.2	NiTiO ₃	0.001210	112
	45.1	NiTiO ₃	0.001063	127

**Fig. 1.** The SEM pictures (Magnification X 5.0K) of (a) nickel plated electrode and (b) Nickel titanate incorporated Ni plated electrode.

mild steel without impregnation of nickel titanate. This shows that nickel titanate act as a good binder between nickel plating and the mild steel. The nickel plated electrode surface was viewed through optical microscope (magnification X20) and found that nickel titanate modified electrode surface shoes more uniform, clear and void free surfaces compared to conventional nickel plated electrodes. The SEM photos (fig. 1) showed very good bonding between the matrix and particles of nickel and nickel titanate with no voids. The uniformity of the nickel plating was found to be much better by incorporating nickel titanate on the mild steel surface.

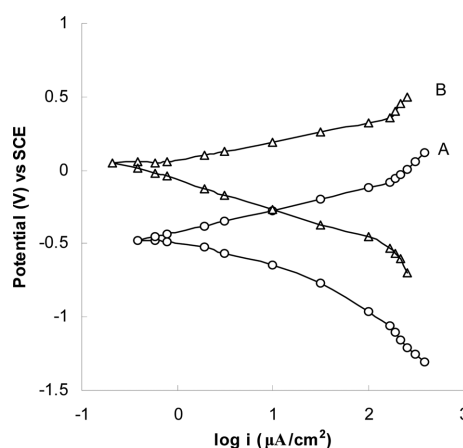
3.3. Electrochemical characterization

3.3.1. Polarizability

fig. 2 shows the polarizability of the nickel plated on mild steel specimen with and without pre impregnation of nickel titanate. The potential of the nickel plated mild steel impregnated with nickel titanate was shifted towards more positive direction compared to nickel plated mild steel without impregnation of nickel titanate. The extent of polarization of the nickel plating on mild steel with nickel titanate was lower than that of nickel plated mild steel without impregnation of nickel titanate. From the Tafel plot the electrochemical parameters were calculated and are given in Table 4. The corrosion potential value of nickel plating changes from -0.480 V to $+0.05$ V and corrosion current changes from $0.340 \mu\text{A}/\text{cm}^2$ to $0.212 \mu\text{A}/\text{cm}^2$ by incorporating nickel titanate on

Table 4. The electrochemical parameters of nickel plated electrode with and without incorporation of nickel titanate

Sample	E_{corr} (V)	i_{corr} ($\mu\text{A}/\text{cm}^2$)	Tafel slope V/($\mu\text{A}/\text{cm}^2$)
Nickel plating on mild steel surface	-0.480	0.340	0.162
Ni plating on mild steel surface modified with nickel titanate	$+0.05$	0.212	0.138

**Fig. 2** The Tafel plots of nickel plated electrodes in 3% NaCl solution A: Nickel plating on mild steel and B: Ni plating on mild steel pre inclusion with nickel titanate.

the mild steel surface. Hence impregnation of nickel titanate on mild steel sample before electroplating will decrease the dissolution of nickel from the nickel plated surface.

3.3.2. Long-term stability

The open circuit potential (OCP) variation of the plated electrodes is shown in fig. 3. The OCP shifted to more anodic direction in nickel plated samples modified with nickel titanate. The anodic potential shift shows the less corrosion rate of the nickel plated mild steel modified with nickel titanate compared to

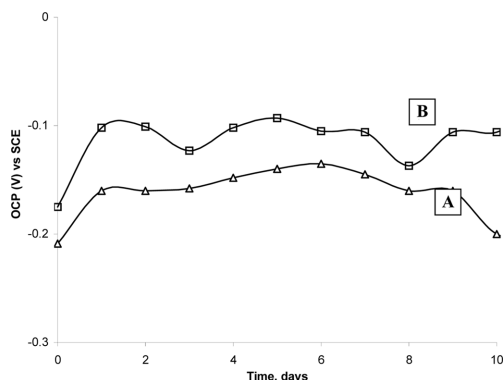


Fig. 3. The open circuit potential decay study of nickel electrodes in 3% NaCl. A: Nickel plating on mild steel and B: Ni plating on mild steel modified with nickel titanate.

nickel plating on pure mild steel surfaces. The nickel titanate enhances the adherence property of nickel plating and hence decreases the dissolution nickel plating when the plated surface was in contact with electrolyte medium.

4. Conclusions

The nickel plated mild steel previously impregnated with nickel titanate shows good adherence, high corrosion resistance and long durability compared to nickel plating on mild steel samples without impregnated with nickel titanate. The nickel titanate act as a good binder between nickel plating and mild steel in nickel plated mild steel surface. More over, the impregnation of nickel titanate on mild steel sample before electroplating will decrease the dissolution of nickel from the nickel plated surface.

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