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Evaluation of Corrosion Resistance of Electroless Ni-P-X ($X=Al_2O_3$ and ZrO_2) Nano-composite Coatings

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ABSTRACT

*The Ni-P-X EL nano-composite coatings are obtained by the immersion of the substrate material (Mild Steel, MS AISI1040) into electroless bath solution having second phase alumina and zirconia particles respectively. Microstructure and basic composition of as-plated and heat treated specimens were analyzed by SEM and EDAX techniques. The SEM results indicated supplement of Al_2O_3 and ZrO_2 nano-particles separately into an EL Ni-P matrix and were confirmed through EDAX analysis. When the coated specimens were heated at 380°C for 1 hour in argon atmosphere, nano-particles turned out to be closely packed which suggest an improvement in corrosion resistance of these EL nano-composite coatings. From the long term immersion corrosion test, it can be concluded that there is a significant improvement in corrosion resistance of deposited samples than un-deposited sam*ples.

Keywords: Electroless coating, Corrosion, SEM, EDAX etc.

I. INTRODUCTION

In process industries it is experienced that corrosion and wear phenomenon results thrashing of plant competency and sporadically a shutdown of all industrial plants. The united effects of corrosion and wear can create ground to a lot higher material failure that can be cause by each process in solitary. In copious applications, it has been observed that surface properties of plant materials such as hardness, corrosion and wear and abrasion resistance can be efficiently enhanced by techniques like carburizing, laser hardening, nitriding, flame hardening, internal oxidation, chemical and physical vapor deposition etc [1,2]. The above mentioned techniques also look more economical options rather than improving the bulk properties of plant materials. Currently, metal deposition processes (eletro- and electroless depositions) are getting munificent attention due to their amazing advantages such as simplicity, uniformity of deposits, low cost, high deposition rate and well-brought-up wear and corrosion resistance properties. In recent times, electroless (EL) coatings have put on extensive popularity in petrochemicals, automobiles, space, nuclear, textile and paper, computer, opto-electronics, food casing, printing and in scientific domain, owing to its capability to produce hard, wear and

friction resistant, antibacterial and corrosion resistant surfaces Electroless (EL) coating is an auto-catalytic procedure in which reduction of metallic ions and coating deposition can be conceded elsewhere through oxidation of reducing agent [3-6]. The research papers [7-12] confer chromate, fluoride, phosphate, stannate, rare earth metals on Mg and its alloys by electroplating and electroless procedures. Moreover there is possibility of deposition of second phase particles by electroless plating for improving hardness, corrosion resistance and abrasive properties of dispersion coatings. Incorporation of second phase particles into elctroless coatings can be of useful in cases where one requires a permutation of corrosion resistance along with tribological properties. The encroachment in coating technology are also apparent from the modern research activities on electroless coatings Ni-P-PTFE, Ni-P-W, Ni-P-Al_2O_3, Ni-P-TiO_2 and Ni-P-ZrO_2 etc. having micro/nano sized particles, which demonstrate improved hardness and wear resistance above Ni-P alloys[1-6]. Therefore, the current study covenants with the synthesis and assessment of corrosion behavior of Ni-P-Al_2O_3 (NiPA) and Ni-P-ZrO_2 (NiPZr) EL nano-composite coatings, with the help of long term immersion corrosion tests in NaCl 5% wt. solution, pH 6.9 and at room temperature.

II. MATERIAL AND METHODS

2.1. Mild steel material

It is incredibly fundamental to attain ready substrate surface correctly for effectual deposition of EL nano-composite coatings on the substrate. In contemporary study mild steel (MS, AISI 1040) having proportions 20 mm × 20 mm × 4 mm (flat coupons) is selected as substrate material for NiPA and NiPZr EL nano-composite coatings. For substrate sample preparation, shaping, parting, milling and surface grinding course of action are adopted. The cleaned sample was then immersed in 1% aqueous solution of $SnCl_2$ (2-3 drops of 1M HCl was added to dissolve $SnCl_2$) for 40 seconds to activate the substrate face. Soon after the substrate sample is erect active by dipping it into a mild hot palladium chloride solution (55 °C, $PdCl_2$) followed by distilled water washing and air dry. Now the well activated substrate sample is dipped into EL bath solution retain at 85 °C and nano composite coating is conceded out of a time of two to three hours [1, 4, 8,9].

2.2. Electroless coating bath unit

It consist a magnetic stirrer (Remi make) with heater and temperature ranges launch 0 to 100 °C with stirrer rate 0 to 400 rpm. A fixed stand is provided for holding and supporting the substrate sample and thermometer. A glass beaker (250 ml volume) roofed with electroless bath (200 ml volume) is positioned on to the heating plate. The stirrer rate and bath temperature are put with help out of speed setting and temperature sensing grip. The purpose of magnetic agitator is to maintain nano-composite particles in suspension keep away from of agglomeration in underneath of glass beaker. Bath composition and working conditions for EL NiPA and NiPZr nano-composite coatings are selected after copious experiments.

2.3 Long term immersion test for corrosion rate evaluation

For immersion testing, individually cleaned, pre-weighed NiPA and NiPZr EL nano-composite coated (as-plated along with heat treated) base specimens are exposed into the solution of NaCl (5% wt., pH 6.9, at room temperature) for 120 days. Subsequent exposed duration, the specimens were cleaned mechanically by distilled

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water and follow by an acetone washing. The weight loss was measured and corrosion rate was calculated in mpy (mils per year) by means of the subsequent formula.

Corrosion rate (mpy) = 534 W / DAT

where symbols W is weight decrease in mg, D is density and have value 7.75 g/cm^3, A is exposed surface area and measured into inch square, T is time of whole exposure duration and measured in hours [3]

2.4 Characterization techniques used for surface coatings

The microstructure and constituent composition of as-plated and heat treated specimens were calculated by the help of SEM and EDAX techniques. The sufficient grain dimensions of the deposit were calculated by using Scherer equation ($t = 0.9\lambda / B \cos\theta_B$) where parameter λ is Cu K_α wavelength (λ=1.54 A^0), B is broadening of full width at half maximum and θ_B is the Bragg's angle by the intense Ni (111) peak (after removal of instrumental broadening cause [8, 9].

III. RESULTS AND DISCUSSION

3.1 Characterization of coatings

From the SEM with EDAX micrographs of NiPA and NiPZr EL nano-composite coated (as-plated NiPZr Figure1) coupons it can be concluded that the SEM micrograph of as-plated coupons predicted the supplement of Al_2O_3 and ZrO_2 nano-particles separately into an EL Ni-P matrix which is confirmed by EDAX analysis and given in Table 1. This makes improvements in metallic polished surfaces. Also the steady distributions of Al_2O_3 and ZrO_2 nano-particles in their entity class (NiPA and NiPZr) on the coated surface are observed with very little porosity. It is suggested that it may improve corrosion and hardness resistance of EL nano-composite coatings.

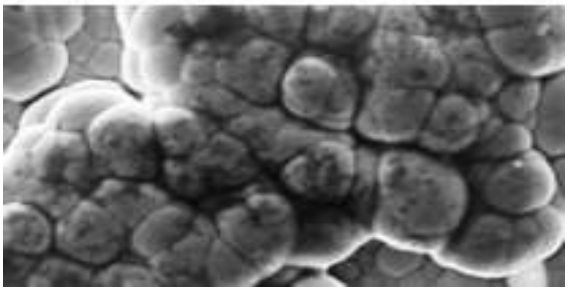

Figure 1: SEM micrograph of NiPZr (as-plated) EL nano-composite coated coupon

Table 1: EDAX analysis of NiPA and NiPZr coatings

Element	Weight %			
	NiPZr		NiPA	
	As-plated	Heat treated	As-plated	Heat treated
OK	3.02	2.58	3.12	2.86
Al K	-	-	1.54	1.21
ZrK	2.21	2.1	-	-
PK	11.87	11.46	11.74	10.87
NiK	80.93	79.88	81.43	79.64
FeK	1.97	3.98	2.17	5.42
Total	100	100	100	100

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3.2 Corrosion immersion experiment

A long time weight loss corrosion experiment for 120 days period was done in a solution of NaCl (5% wt., pH 6.9) on MS, NiPA as well as NiPZr (as-plated, heated) base type coupons. The weight loss (Table 2) test results make obvious decline in corrosion resistance of heated coupons. The NiPZr coated (as-plated, heated) coupons demonstrate weight loss least while MS coupon highest. The corrosion rate in tested coupons can be put in order as NiPZr (as-plated) > NiPZr (heated) > NiPA (as-plated) >NiPA (heat treated) >MS

Table 2: Data on corrosion rate by weight loss test in NaCl (5% wt., pH 6.9) solution

Samples	As-Coated (corrosion rate , mpy)	Heat treated at 380°C corrosion rate, mpy	Without EL Coating corrosion rate, mpy	Pitting corrosion depth mm		Crevice corrosion depth mm	
				As-Plated	Heat treated at 380°C	As-Plated	Heat treated at 380°C
MS	NA	NA	23.82	-	-	0.027	-
NiPA	5.09	6.24	NA	0.013	0.015	0.011	0.018
NiPZr	4.72	4.87	NA	0.011	0.014	0.010	0.010

NA (not applicable)

This is also noticeable after weight loss test (Figure 3) that pitting corrosion emerge more in heated coupons as contrast to as-plated coupons. Performance of NiPZr EL nano-composite against corrosion is better than NiPA coatings .

Figure 3: Photographs after weight loss test of corroded coupon (a) NiPA as-plated (b) NiPZr as-plated

IV. CONCLUSIONS

The SEM examinations illustrate that nano-composite coating is done successfully on base material and as-plated depositions have amorphous nature; while heated materials demonstrate decline in amorphous nature and enrichment in crystallization nature. These transformations propose to elevated hardness in EL deposited heated coupons [13-15]. The long term immersion corrosion experiment in NaCl (5% wt. and pH 6.9) solution on as-plated and heat treated coupons proposed existence of inter-metallics which have protecting character against corrosion with respect to un-deposited coupons. Thus these depositions may accredit good and cost effective option for easy-going corrosive environment, with superior hardness properties .

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