Development of Electroless Ni-P-X (X= TiO_2+ZrO_2) Nanocomposite Platings and its Corrosion Resistance

Manoj Kumar[1], Munna Ram[2], Sulaxna Sharma[3], Awanish Sharma[4]

[1,2,4]Department of Allied Sciences , Graphic Era University, Dehradun (India)

[3]Department of Chemistry, IHET, THDC Tehri, UK (India)

ABSTRACT

The Ni-P-TiO_2-ZrO_2 EL nano-composite platings are obtained by the immersion of the substrate material (Mild Steel, MS AISI1040) into the acidic electroless bath solution having second phase titaina and zirconia particles respectively. The Microstructure and constituent composition of as-plated and heat treated specimens were analyzed by SEM and EDAX techniques The SEM results indicated the addition of TiO_2 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 400°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 than conventional material (MS).

Keywords: Electroless coating, TiO_2 , ZrO_2, SEM ,EDAX and corrosion

I. INTRODUCTION

It is apparent that most work associated to corrosion in pulping section of paper industry, has considered the composition of test liquor and test conditions as those observed in case of kraft pulping. The kraft pulping process uses alkaline sulfide solution mainly sodium hydroxide and sodium sulphide solution for the purpose of delignification of wood/non wood species in order to draw cellulose fibers from them. These fibers in result are made into paper. The cooking alkaline sulfide liquor consist main ions as (sulphide) S^{2-}, (thiosulphate) $S_2O_3^{2-}$, (polysulphide) S_x^{2-} and (choloride) Cl^-. The cooking pulping liquor with higher sulfidity will apparently have higher concentration of these sulfur ions which is expected to result in increased corrosivity of liquor [1-5]To address these major issues of enhanced corrosion, long term corrosion experiments of three months duration were planned in solutions having sulfur containing compounds with their composition matching with those observed in paper mills. In order to analyze the corrosion attack and corrosivity of the solutions, it was planned to use conventional material (MS, mild steel) as well as alternate material (developed electroless Ni-P-TiO_2-ZrO_2 coating).

II. EXPERIMENTAL

2.1. *Materials and Methods*

 ISBN: 978-93-86171-93-1

In this study, mild steel (MS) grade (AISI 1040) having dimensions 1 cm × 1 cm × 0.2 cm (base coupons) is selected as substrate material for NiPTiZr (Ni-P-TiO_2-ZrO_2) EL nano-composite coatings (Figure 1). For substrate sample preparation, shaping, parting, milling and surface grinding are done properly. For nano-composite coated samples, acidic EL plating procedure are taken [6].

Figure 1: Ni-P-TiO_2-ZrO_2 EL nano-composite (as-plated) coupon.

2.2 Electroless Plating Unit and its Composition

The experimental set up planned for EL Ni-P-X (X= TiO_2 + ZrO_2) nano-composite coatings is shown in Figure 2. It consist heater and magnetic stirrer (Remi make) and temperature ranges beginning 0 to 100 °C with stirrer rate ranges starting 0 - 200 rpm. The bath composition and working conditions for EL NiPA and NiPTi nano-composite platings are chosen subsequent to many experiments and pertinent range of parameters are given as; Nickel sulphate 35.2 gpl, Trisodium citrate 48.5 gpl Sodium acetate 22.1gpl, Sodium hypophosphite 23gpl, Synthesized TiO_2 and ZrO_2 2gpl each, pH 5.6 and Temperature 85 ^{o}C[7].

Figure 2: Experimental set up for EL Ni-P-X nano-composite platings

Coating thickness is calculated by using formula

$$\text{Thickness } (t, \mu m) = \frac{W \times 10^4}{D \times A}$$

And here 'W' stands for weight lift up (gm), 'D' is density of deposits (7.75 gm/cm^3) and 'A' is surface area of deposition (cm^2). Plating rate (μm/h) was measured as thickness of plating set down per unit time of deposition [2, 7].

2.3 Characterization techniques used for surface coatings

The microstructure and element composition of as-plated as well as annealed specimens was evaluated via help of scanning electron microscopy (SEM) and energy dispersive X-ray study (EDAX). Their X-ray diffraction (XRD) study was carried out by resources of Cu K_α X- rays for identifying phases present. The adequate grain dimension of deposit was calculated by using Scherer equation ($t=0.9\lambda/ BCos\theta_B$) where parameter λ is Cu K_α wavelength (λ = 1.54 A^0), B is broadening of full width at the half utmost width and θ_B is Bragg's angle using the intense Ni (111) peak (after elimination of instrumental broadening effect) [8].

2.4 Test Solution

The sulphide test solution has the following composition and concentrations: NaOH (sodium hydroxide) =80 gpl, Na_2S (sodium sulphide) =22 gpl (considering 20% sulfidity level), $Na_2S_2O_3$ (sodium thiosulphate) = 5gpl, S_x^{2-} (polysulphide) = 5gpl and Cl^- (chloride) =1.5 gpl.

III. RESULTS AND DISCUSSION

3.1 Characterization of coatings

The SEM micrographs of NiPTiZr EL nano-composite coated (as-plated and annealed, 400 °C Argon atmosphere) coupons are discussed in thesis [1]. From SEM micrograph figures it is clear that EL nano-composite plated surface in all cases have glow surface with steady sharing of titania and zirconia nano-particles. It may increase in corrosion resistance of developed EL nano-composite platings [2, 7, 8].

3.2 Corrosion rate in sulphide solution

A visual examination of corroded coupons, after cleaning, shows no observable pitting attack but uniform and slight crevice attack is visible in case of mild steel sample only. The degree of uniform attack, assessed from corrosion rate, was obtained on the basis of weight loss observed by the samples during the three months test. Corrosion rates experienced by different steels are given in Table-1. One observes that mild steel experiences maximum corrosion rate and corrosion rates on coated coupons are low. Although, heated coupon shows less corrosion resistance than as plated coupon in case of coated coupons. The results will further be analyzed[9-14].

Table-1: Corrosion rates (mils per year) of samples in alkaline sulfide solution

Materials	Corrosion rate (mpy)
MS(Mild Steel)	3.213
Ni-P-TiO2-ZrO2 (As-plated)	0.517
Ni-P-TiO2-ZrO2 (Heated, 400^0C)	0.686

IV. CONCLUSIONS

The current investigations illustrate that NiPTiZr EL nano platings have been successfully deposited on mild steel substrate. The as-plated EL nano-composite coatings have amorphous character; besides, the heated EL nano-composite coated coupon show decrease in amorphous character and enhancement into the crystallization character. These changes have been recommended to pick up in wear, hardness and corrosion resistance. The coated coupons show roughly six times corrosion resistance as compared to mild steel coupon.

REFERENCES

[1] A. Sharma, "Corrosion Investigations in Pulping and Bleaching media", PhD. Thesis, Indian Institute of Technology Roorkee, 2006.

[2] N. Feidstein, T. Lancsek, D. Lindsay and L. Salerno, "Electroless composite Plating" , Metal Finishing, 81, 1983, 35-41.

[3] A. Wensle, "Corrosion and protection of Kraft batch digesters", Technical Association of Pulp and Paper Industry Journal, 79 (10), 1996, 153.

[4] D. C. Bennet, "Cracking of continuous digester-review of history", Proc. of 4th International Symposium on Corrosion in Pulp & Paper Industry, 4, 1983.

[5] A. H. Tuthill (Ed.), "Stainless Steels and Specialty Alloys for Modern Pulp & Paper Mills", NiDi, Reference Book Series no. 11025:138.

[6] ASM Handbook Committee. Electroless Nickel-Plating, Surface Engineering. American Society for Metals. 5, 1994, 291.

[7] M.A. Kumar, R.C. Agarwala and V. Agarwala, "Synthesis and characterization of electroless Ni–P coated graphite particles", Bull. Mater. Sci., 31, 2008, 819–824.

[8] F. A. Esfahani , M. Sarafbidabad, "Evaluation of Corrosion Rate and Surface Oxides Formed of Zr-2.5%Nb as an Implant Material in Ringer's Solution", Indian Journal of Science and Technology, 9(6), 2016.

[9] J. K. Dennis and K. S. Sagoo, "Wear Behaviour of Engineering Coatings and Surface Treatments", Metal Finish, 89, 1991, 111–121.

[10] B. Elsener , M. Crobu , M. A. Scorciapino , A. Rossi, "Electroless Deposited Ni-P Alloys: Corrosion Resistance Mechanism", Journal of Applied Electrochemistry 38, 2008, 1053–60.

[11] S. H. Wang , H. Y. Yang , "Characterization of Ni-P/TiO_2 MMC Coatings Prepared by Electroless Plating Process on Mg-Nd-Zn-Zr Magnesium Alloys", Material Science Forum, 690, 2011, 422.

[12] Anil Kumar, M. G. Ananthaprasad, K. Gopala Krishna, "A Review on Mechanical and Tribological Behaviours of Nickel Matrix Composites", Indian Journal of Science and Technology, 9(2), 2016.

[13] X. Chen, SS. Mao, "Titanium Dioxide Nanomaterials: Synthesis, Properties, Modifications, and Applications", Chemical Reviews 107, 2007, 2891-2959.

[14] A. Sharma and A. K. Singh, "Electroless N-P-PTFE-Al_2O_3 Dispersion Nanocomposite Coating for Corrosion and Wear Resistance", Journal of Materials and Engineering Performance, 23 (1), 2014, 142-151.