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Development and Characterization of Electroless Ni-P-X ($X=Al_2O_3$ /TiO_2) Nanocomposite Platings

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ABSTRACT

The Ni-P-X EL nanocomposite platings are obtained by the immersion of the substrate material (Mild Steel, MS AISI1040) into the acidic electroless bath solution having second phase alumina and titania particles respectively. The Microstructure and constituent composition of as-plated and heat treated specimens were analyzed by FESEM and EDAX techniques The FESEM results indicated the inclusion of Al_2O_3 and TiO_2 nano-particles into an EL Ni-P matrix and were confirmed through EDAX analysis. When the coated specimens were heated at 350°C for 1 hour in argon atmosphere, nano-particles turned out to be closely packed which suggest an improvement in corrosion and wear resistance of these EL nano-composite coatings.

Keywords : Electroless plating, Al_2O_3 , TiO_2 , SEM, EDAX

I.INTRODUCTION

In most industries shield of machine parts, tools and equipment from ecological changes, corrosion, erosion, friction and wear is very vital. For that purpose these should be equipped by hard, tough, wear and anticorrosion materials, yet it is not advantageous in all veneration. Afterward, it is too found that some amazing platings for surface shield is a vital component of design, development and synthesis of new novel materials. In recent times, electroless depositions have attain extensive recognition in oil and gas, aerospace, automobile, mechanical, chemical as well as in scientific domain, owing to its cleverness to produce hard, wear, friction and corrosion resistant surface. The electroless plating is an autocatalytic method. In this method, reduction of metallic ions along with plating deposition can be carried out by oxidation of reducing driving force [1-4]. The present investigational study deals with synthesis of Ni-P-Al_2O_3 (NiPA) and Ni-P-TiO_2 (NiPTi) EL nano-composite platings. The techniques such as FESEM, EDAX and XRD are used for surface morphology and composition of Ni-P-Al_2O_3 (NiPA) and Ni-P-TiO_2 (NiPTi) coatings under as-plated and annealed conditions [5-7].

II.EXPERIMENTAL

2.1. Materials and Methods

It is very essential to get ready substrate surface carefully and appropriately for effective deposition of EL nano-composite coatings on the substrate. In this study mild steel (MS) grade (AISI 1040) having dimensions 2 cm × 2 cm × 0.4 cm (base coupons) is selected as substrate material for EL NiPA and NiPTi nano-composite platings. For substrate sample preparation, shaping, parting, milling and surface grinding procedure are taken. After these processes, substrate surface is mechanically cleaned starting overseas stuffs and corrosion products. Afterward, substrate was rinsed by fresh distilled water as well as with dilute 50 % HCl for 1min to get rid of any contaminated surface layer. The well polished, cleaned sample was engrossed in 1% aqueous solution of $SnCl_2$ (2-3 drops of 1M HCl was added to dissolve $SnCl_2$) for 30 seconds to activate the substrate surface. The substrate sample was subsequently washed by means of distilled water and air dried. Later on the substrate sample was actived by dipping it into a solution of palladium chloride (55 °C, $PdCl_2$) followed by distilled water washing and air dry[3,6-8]. Now the activated substrate sample is dipped in EL bath solution retain at 85 °C and plating is conceded out of a time of two to three hours.

2.2 Electroless Plating Unit

Experimental set up planned for EL Ni-P-X (X=Al_2O_3 and TiO_2 nano-particles) nano-composite coatings is shown in Figure 1. It consist heater and magnetic stirrer (Remi make) and temperature ranges beginning 0 to 100 °C with stirrer rate ranges starting 0 - 400 rpm. A solid stand is provided for holding and supporting substrate sample and thermometer. A glass beaker (250 ml volume) enclosed with electroless bath (200 ml volume) is placed on heating plate. The stirrer rate as well as bath temperature are set with the help of speed setting and temperature sensing grip. The function of magnetic agitator is to keep nano-composite particles in suspension devoid of agglomeration in bottom of the glass beaker. The stirrer speed is fixed after huge numeral of trials to keep away from instability of electroless bath owing to agglomeration of particles. Bath composition and working conditions for EL NiPA and NiPTi nano-composite platings are chosen subsequent to many experiments and appropriate range of parameters are revealed in Table 1. The Sodium Dodecyl Sulphate (SDS) is supplementary in a very small quantity into electroless (EL) bath unit for improved spreading of second phase alumina and titania nano-particles. The significant function of surfactants is to subordinate the surface stress of a liquid and reduce interfacial stress flanked by solid and liquid surfaces. Two sets of relating to 100 ml of EL Ni solution holding necessary quantity of alumina and titania are thoroughly mixed with the help of PTFE magnetic stirrer.

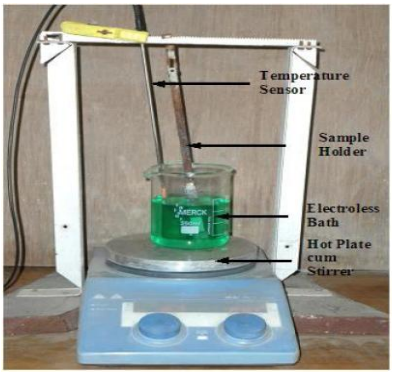

Figure 1: Experimental set up for EL Ni-P-X nano-composite platings

Table 1: EL bath components and their function

S.No.	**Salt/Compound chemical formula**	**Quantity in gram (g) for 100 ml)**	**Function of chemicals**
1	Nickel Sulphate ($NiSO_4$)	3.5 g	Source of Ni^{2+} ions
2	Trisodium Citrate	4.8 g	Complexing agent, prevent uncontrolled release of Ni^{2+} ions
3	Sodium Acetate	2.2 g each	Work as basic buffer in the presence of ammonia, to maintain the pH
4	Sod. Hydroxide/ Acetic acid10% Solution	Added drop wise	Maintain pH of the solution ~5.5
5	Sodium Hypophosphite	2 g	Reducing agent, provide electrons to the Ni^{2+} ions which on accepting electrons get reduced to Ni^0 and deposited on the catalytic surface
6	Sodium Dodecyl Sulphate	0.01g	increase the wettability and surface charge
7	Lead Acetate	0.1mg	Stabilizer
8	Synthesized Al_2O_3	1 g	Work as reinforcement into the matrix

9	Synthesized TiO_2	1 g	Work as reinforcement into the matrix
10	Bath Operating Conditions	-	pH 5.5; Temperature 85-90 ^oC; constant rousing is required
11	Annealing Hotness	Up to 350 ^0C	Understand the consequence of heat conduct on corrosion resistance

Coating thickness, t, is calculated by using formula

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

Here 'W' stands for weight raise (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, 5, 6]. In current work, plating thickness is bringing into being in range of 23 to 30 microns. When plating is over, coupons are washed by means of distilled water and dried in air. To recognize outcome of annealing of EL nano-composite coatings, plated coupons are annealed in furnace meant for 1 hour duration at temperature (350 °C) according to an orthogonal array. Ensuing to annealing, coupons are cooled to room temperature [4,7].

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 middling grain dimension of the 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) [2, 4, 5, 7-10].

III.RESULTS AND DISCUSSION

3.1 Characterization of coatings

The SEM with EDAX micrographs of NiPTi and NiPA EL nano-composite coated (as-plated and annealed, 350 °C Argon atmosphere) coupons are shown in Fig. 2 and Fig. 3. From SEM micrograph figures it is clear that EL nano-composite coated surface in all cases have flush surface with consistent sharing of alumina and titania nano-particles in respective cases. The surfactant sodium dodecyl sulphate (SDS) presence can be responsible for this uniform plating in electroless bath solution. When EL nano-composites are heated at 350 °C for one hour duration, globules of nickel with phosphorus are seen in all cases with embedded alumina and titania particles in their respective cases. The globules turn out to be more packed together, which further condensed the porosity of plating. It may enhance in corrosion and triobological resistance of developed EL nano-composite platings [3, 6, 7-10].

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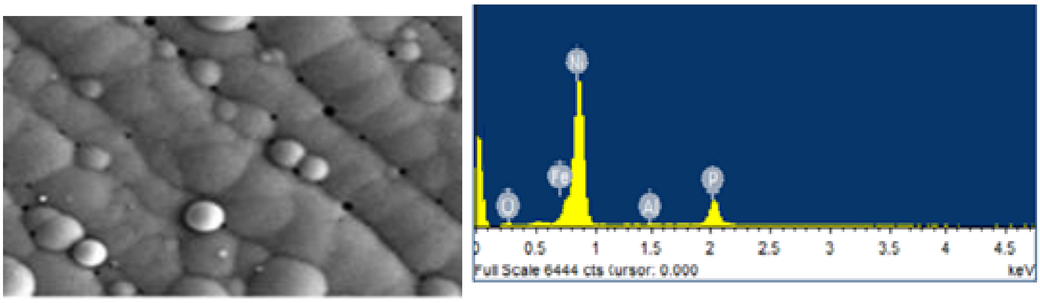
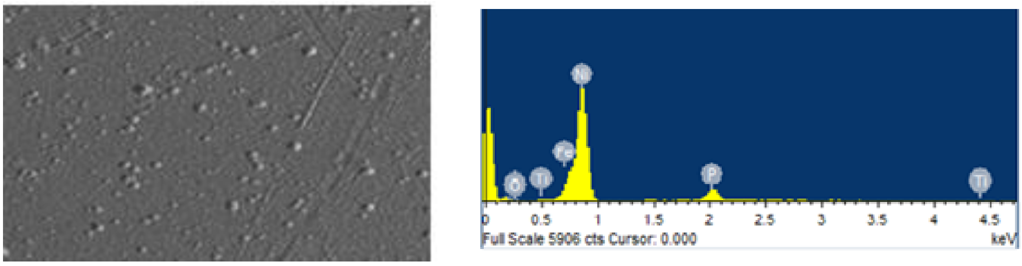


(a) **(b)**

Figures 2: SEM (a) with EDAX (b) micrograph of NiPA (HT) EL nano-composite platings



(a) **(b)**

Figures 3: SEM (a) with EDAX (b) micrograph of NiPTi (HT) EL nano-composite platings

By EDAX data analysis this is inveterate that EL nano-composite coatings (NiPA and NiPTi) under as-plated conditions consisting of 10 gpl of Al_2O_3 nano-particles have 80.45 wt% of nickel, 10.98 wt% of phosphorus, 3.06 wt% of oxygen, 2.95 wt% Fe and 2.56 wt% of aluminum. While 10 gpl TiO_2 nano-particles, have 79.46 wt% of nickel, 11.74 wt% of phosphorus, 3.92 wt% of oxygen, 2.41wt% of Fe and 2.47 wt% of titanium. In NiPTi EL nano-composite coatings P content is slightly higher than others case and thus exhibiting the higher fineness and density of the surface structure. By EDAX analysis this is inveterate that EL nano-composite coatings under annealed conditions consisting of 10 gpl of Al_2O_3 nano-particles have 79.32 wt% of nickel, 10.24 wt% of phosphorus, 3.02 wt% of oxygen, 5.29 wt% Fe and 2.13 wt% of aluminum. While for 10 gpl of TiO_2 nano-particles, have 79.28 wt% of nickel, 11.21 wt% of phosphorus, 3.51 wt% of oxygen, 2.41wt% of Fe and 3.59 wt% of titanium. The SEM/EDAX images of heat treated EL NiPA and NiPTi nano-composite plating shows occurrence of several globules on the surface. It is also evident by EDAX analysis that amount of coating element Ni, P, O, Al and Ti declines on annealing while that of Fe increases, this can be because of diffusion of coating element towards interface of plating and substrate surface and results an increased corrosion of mild steel. However, less amount of Fe for NiPTi EL nano-composite coating in comparison to NiPA EL nano-composite coating put forward a lesser amount of diffusion of second phase TiO_2 nano-particles, which helps to prevent corrosion[1, 2,4,5,7,8-11].

Furthermore grains size of EL nano-composite platings is expected into range between 1.87 to 13.5 nm for Al_2O_3 and 1.76 to 9.8 nm for TiO_2 nano-particles. Decrease in grain dimension for EL NiPTi coatings can be credited to allocation of TiO_2 nano-particles at the boundary of nickel grains. This behavior curbs escalation of nickel (Ni) grain in deposition procedure and results in very fine and flat surface. It is also observed that grains are coarsened due to heat treatment which helps in increasing the wear resistance and hardness of coatings [2,4,7,10-11]

IV.CONCLUSIONS

The current investigations depict that NiPA and NiPTi EL nano platings have been successfully deposited on mild steel substrate. The as-plated EL nano-composite platings have amorphous character; moreover, the annealed EL nano-composite coatings show decrease in amorphous character and enhancement into the crystallization character. These changes have been suggested to pick up in wear, hardness and corrosion resistance.

REFERENCES

[1] W. Bolton, "*Engineering Materials Technology," 3rd Edition. Butterworth-Heinemann, Oxford, 1998, 447.*

[2] Y. De Hazan, F. Knies, D. Burnat, T. Graule, Y. Yamada-Pittini, C. Aneziris and M. Kraak, "Homogeneous functional Ni-P/ceramic nanocomposite coatings via stable dispersions in electroless nickel electrolytes", *J. Colloid Interface Sci., 365,1012, 163-171.*

[3] G. O. Mallory, and B.Hajdu, *Electroless Plating: Fundamentals and Applications Noyes Pubn Bracknell Berkshire UK 1990.*

[4] R.C. Agarwala, V. Agarwala and R. Sharma, "Electroless Ni-P Based Nanocoating Technology—A Review", Synthesis and Reactivity in Inorganic, *Metal-Organic, and Nano-Metal Chemistry, 36(6),2006, 493-515.*

[5] A. Sharma, A.K. Singh, "Corrosion and Wear Study of Ni-P-PTFE-Al_2O_3 Coating: The Effect of Heat Treatment," *Cent. Eur. J. Eng., 4(1), 2014, 80-89.*

[6] J.N. Balaraju, T.S.N. Sankara Narayanan and S.K. Seshadri, "Electroless Ni–P composite coatings", *J. Appl. Electrochem, 33, 2009, 807–816.*

[7] J. Sudagar, J. Lian and W. Sha, "Electroless nickel, alloy, composite and nano coatings–A critical review", *J. of Alloys and Compounds, 571, 2013, 183-204.*

[8] M. Nováka, D. Vojtěcha, T. Vı´tu°, "Influence of heat treatment on microstructure and adhesion of Al_2O_3 fiber-reinforced electroless Ni–P coating on Al-Si casting alloy", *Mater. Charact., 61, 2010, 668-673.*

[9] A. Sharma and A.K. Singh, “Electroless Ni-P and Ni-P-Al_2O_3 Nanocomposite Coatings and Their Corrosion and Wear Resistance”, *J. Mater. Eng. Perform, 22,2013, 176-183.*

[10] J. Novakovic, P. Vassiliou, K. Samara, T. Argyropoulos, “Electroless Ni-P-TiO_2 composite coatings: Their production and properties”, *Surf Coat Tech, 201, 2006, 895–901*.

[11] A. Sharma A, Sulaxna Sharma, S. Sharma, “In-Plant Test for Corrosion Investigations in Digester of a Paper Mill,” *Journal of Materials and Environment Science*, 4(3), 2013, 420-425.