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CRYSTALLINITY ANALYSIS OF COMPOSITE COATING ON Al SUBSTRATE BY ELECTROPLATING METHOD

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ABSTRACT

Ni-P-TiO_2 composite coatings were prepared on aluminium substrate by TiO_2 sol-enhanced electroplating. A systematic crystallinity study has been conducted in order to understand the effect of heat treatment on the substrate coated with TiO_2 solution incorporated into the Ni-P matrix. The crystalline structure of the deposit produced by electroplating method was analyzed using X-Ray diffraction techniques. The parameters like grain size, full width half maximum, height of the peak, position of the peak, area, intergral breath and d-spacing are found and analysed the results for the impact of heat treatment on the deposit coated on the substrates.

KEYWORDS: Electroplating, Ni-P-TiO_2 coating, Crystallinity.

INTRODUCTION

Electrodeposited composite coatings are widely used in automobiles manufacturing, aviation, chemical processes and other industries due to their excellent properties such as high hardness, good wear and corrosion resistance [1]. Recent years, co-depositing nano-particles of a second phase into electroplated coatings has led to the development of nano-composite coatings with improved mechanical properties [2,3]. The second phase nano-particles are dispersed into the metal matrix, providing good hardenability and wear resistance [4,5]. A variety of such composite coatings have been synthesized and reported, including Ni-Al_2O_3 [6], Ni-TiO_2 [7], Ni-ZrO_2 [8], Ni-SiO_2 [9], and Ni-SiC [10].

The crystallite size of the coatings and microstrains present in them are the important parameters determining the quality of coatings. The crystallite size decreases with increasing phosphorous content [11]. The crystallite size and the microstrain in the lattice could be estimated by X-ray line profile analysis. Crystallite size may be influencing the 'peak broadening' more than the strain in the coating [12]. Most of the earlier/current investigations are focused on the effect of process parameters on the behaviour of coating and optimised the process parameters to improve the coating properties. Studies on the influence of heat treatment on the structure of composite coated deposit are relatively scarce. Sol-gel process has been widely applied to prepare uniform nanoparticles [13-15]. Recently, we have developed a novel technique: sol-enhanced composite plating, to synthesize highly dispersive oxide nano-particle reinforced composite coatings [16-20]. In this new method, transparent sol solution containing desirable oxide components is directly introduced into the electrolyte solution at a controlled speed. There is no step of solid particle formation in the processing, therefore no particle agglomeration occurs. This method can lead to a highly dispersive distribution of TiO2 nanoparticles in the coating, resulting in significantly improved mechanical properties.

The objective of this work is to study the parameters such as grain size, microstrain, height of the peak, position of the peak, area, intergral breath and d-spacing and FWHM of the deposits produced by electrodeposited composite coating and its effect on heat treatment were determined using XRD and the data were collected by using X-Pert highscore software.

Experiment: Details and Procedure

In the present study, both Ni-P and Ni-P-TiO_2 composite coatings were electroplated onto Al substrates ($30\times10\times10$ mm^3). Al substrates were precoated with Cu for 2 to 3 microns and

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mechanically polished using SiC paper to a grit of 1200, then degreased ultrasonically in acetone. Before electroplating, the specimens were pre-treated in an alkaline solution containing 50 g/L NaOH and 10 g/L NaH2PO4·H2O at 65oC for 15 min, then activated in acid solution containing 20 g/L citric acid and 60 g/L ammonium citrate at room temperature for 20 s. TiO_2 sol was prepared as follows: 8.68 ml of tetrabutylorthotitanate [Ti(OBu)$_4$] was dissolved into the mixture solution of 35 mL ethanol and 2.82 mL diethanolamine (DEA) [1]. Table 1 consist the details of substrates produced using different electrolyte and the electrolyte information are given above.

Table 1. Details of the substrate

Substrate ID	Description
1	Ni-P
2	Ni-P-TiO_2
3	Ni-P-TiO_2 (Heat treated at 300°C)

The chemicals used for preparing electrolyte are NiSO4·6H2O 250g/L, $NiCl_2 \cdot 6H_2O$ 15g/L, NaCl 15g/L, H_3BO_3 30g/L, H_3PO_4 6g/L, NaH_2PO_2·6H2O 20g/L, Temperature 70±2ºC, pH value 4, Time 30min, TiO_2 sol 0-50 mL/L, Current 50 mA/cm^2. the degree of crystallinity of the deposit was determined using X-ray diffraction (Rigaku, ultima III). The Xray tube was operated at 30 kV, 20 mA. Step scanning technique was employed with a step width of 0.02° (2θ) and with a counting time of 5 s/step in all the cases. Heat treatment was done on muffle furnace with maximum temperature range of 900°C. The coated substrates were maintained at 300°C.

RESULT AND DISCUSSION

Full width at half maximum (FWHM) is an expression of the extent of a function, given by the difference between the two extreme values of the independent variable at which the dependent variable is equal to half of its maximum value. Half width at half maximum (HWHM) is half of the FWHM. The coated substrates were analysed for FWHM, without TiO_2 0.096, after incorporating the composite material into the deposit it is decreased from 0.096 to 0.078 for substrate and it remains same in substrate 3 also. The FWHM value for the deposits are shown in figure 1.

The peak height are shown in figure 2. It was observed that the height of the peak is very high for the substrate coated with Ni-P and it shows the substrate is highly crystalline. For the substrate produced using Ni-P-TiO_2 and

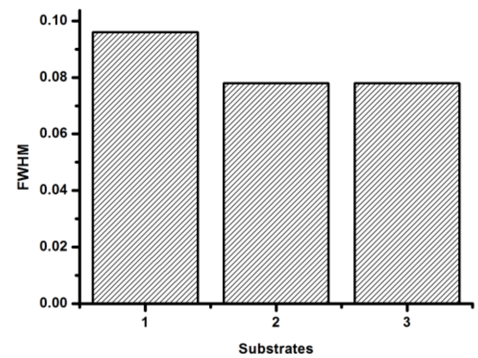

Figure 1

the heat treated substrate showed very less peak height when compared to Ni-P deposit. It depicts that the corrosion resistance of the substrate could be better for the substrate 2 and 3 than the substrate 1.

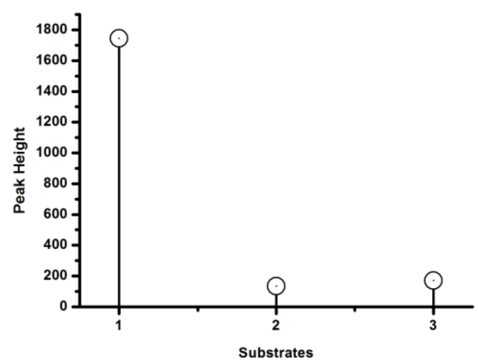

Figure 2

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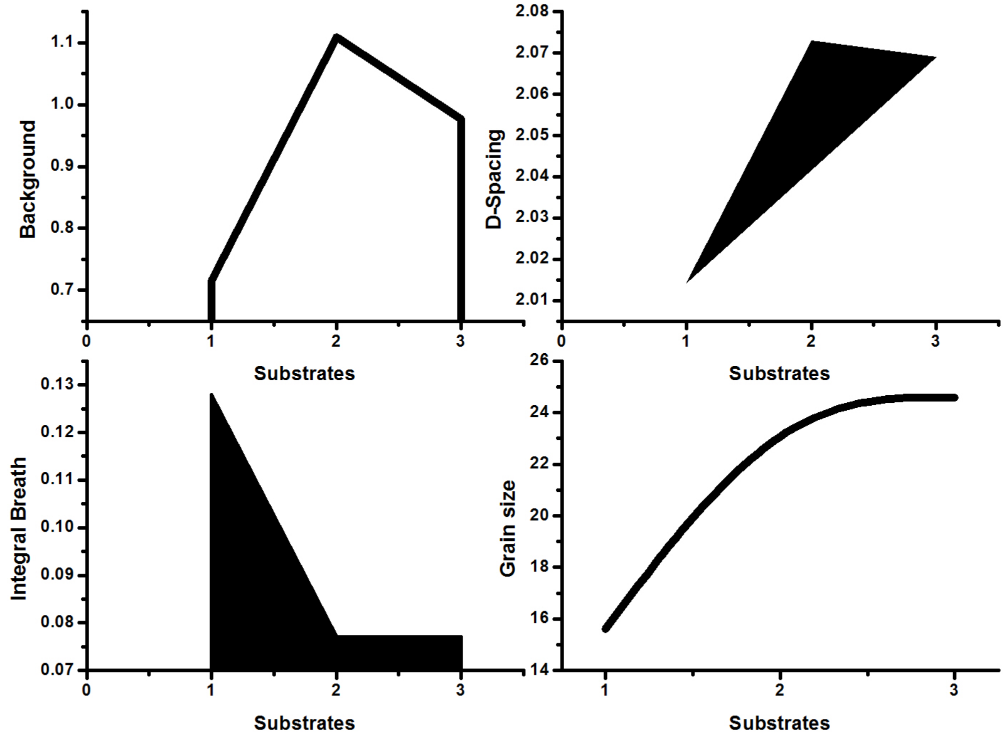

Figure 3

Figure 3 shows the values of backgroung, integral breath, D-spacing and grain size. The grain size of the deposit can be determined
using scherrer equation, for Ni-P substrate the grain size is less, for composite coatings grain size are large when compared to

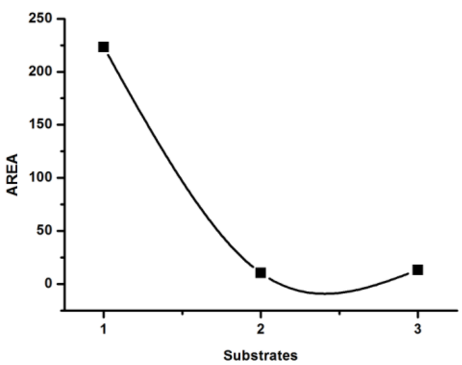

Figure 4

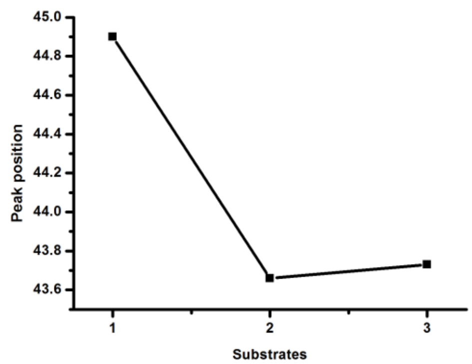

Figure 5

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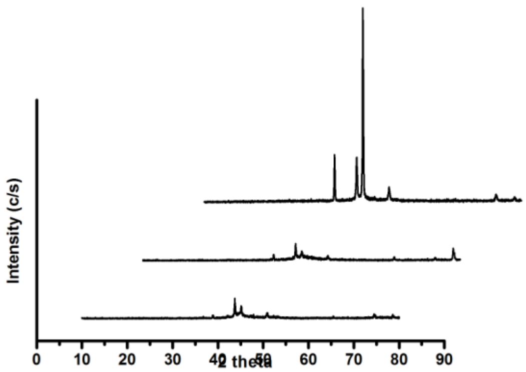

Figure 6

conventional coating. Integral breadth is defined as the breadth of the rectangle having equal height and area as that of the diffraction profile. It is high for the Ni-P deposit and almost same for the substrate 2 and 3, its value is 0.077 for both the substrates. There is no much deviations in D-spacing of the analysed substrates. The d spacing values also show in figure 3. These graphs were generated using origin pro software version 8, panel 4 option was used to generate figure 3. From hall pitch equation, using this grain size, can predicit the surface hardness of the deposits. The area of the peak is shown in figure 4. For 44.96, 43.66, 43.73 for the substrate 1,2,3 respectively. Figure 5 shows the peak position of the substrates. It revealed that there was no deviations in the substrate 2 and 3 but there is a small shift for the substratre 1. Ni-P deposit, the region or the area occupied by the diffractogram is high 223.31, for the substrate 2 and 3 it was 10.43 and 13.29 respectively. The X-ray diffractogram of the substrates are given in figure 6. The data discussed above were determined by using the diffractograms.

CONCLUSION

Ni-P-TiO_2 composite coating was successfully employed on the Al substrate to improve the structural and mechanical properties. From the XRD analysis of the coated substrates, the following conclusions are drawn:

1. The position of the peak is shifted from 44.96 degree to 43.73 degree.
2. The height of the peak is reduced from 1744 to 171.14, it shows that the deposit is changed from highly crystalline to amorphorous. It leads to improve the corrosion resistance of the substrates.
3. Similar to other parameters, there is a change in FWHM and background values of the deposits 0.096, 0.078, 0.078 and 0.7153, 1.108, 0.977 respectively.
4. Integral breath of the deposits doesn't show any changes for TiO_2 incorporated substrates.
5. The grain size of the substrates were increased gradually from 15.62, 19.14 and 24.59.

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