

## EFFECT OF ALUMINA PARTICLE SIZE ON PROPERTIES OF ELECTROLESSLY DEPOSITED COMPOSITE Ni-P/Al<sub>2</sub>O<sub>3</sub>

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### ABSTRACT

*In this study, effect of particle size on morphology, alumina content and hardness of electrolessly deposited composite Ni-P/Al<sub>2</sub>O<sub>3</sub> layers was examined with different sizes of codeposited alumina particles. Results on Scanning Electron Microscopy (SEM), Energy Dispersed Spectroscopy (EDS), X-ray Diffraction (XRD) and hardness measurements showed that as particles size increased composition of NiP matrix did not change remarkably e.g the composition of the obtained matrix ranged from Ni<sub>90</sub>P<sub>10</sub> to Ni<sub>91</sub>P<sub>9</sub>. Meanwhile, Al<sub>2</sub>O<sub>3</sub> content in the composites increased as particle size decreased. As a result, hardness of the obtained layers increased with decreasing particle size and maximum hardness was achieved with particle size of 0.6 μm.*

### I - INTRODUCTION

Autocatalytically deposited Ni-P amorphous alloys have many industrial applications because of their paramagnetic properties, excellent resistance to wear and corrosion, and high hardness [1 - 4]. Codepositing another metallic or non-metallic elements or abrasive/lubricative particles or combination of in binary Ni-P matrix can further enhance these properties. Recently, electroless nickel composite coatings have gained more attention in research community due to their ability to produce coatings that possess improved wear, abrasion and lubrication properties than Ni-P deposits [3-5]. Several particles have been incorporated in the nickel matrix, and among them, the combinations that have received considerable attention are electroless nickel with SiC, B<sub>4</sub>C, Si<sub>3</sub>N<sub>4</sub>, Al<sub>2</sub>O<sub>3</sub> and PTFE and the particles used are of micron size. Codeposition of the particles depends on the size, shape, density, concentration and method of suspension in the bath. In addition, it is very

much dependent on the charge present on the particle [6, 7].

Aim of the present investigation is to study the particle size effect on properties of the deposits. Hence, in this study Ni-P/Al<sub>2</sub>O<sub>3</sub> composites were prepared using alumina powders of sizes 0.6 μm, 8 μm and 15 μm and their surface morphology, structure and hardness were investigated.

### II - EXPERIMENTAL

Mild steel specimens (2.5 cm × 2.5 cm × 0.08 cm) were used for plating electroless NiP and nanocomposite coatings containing alumina particles. Specimens were ultrasonically cleaned in acetone, cathodically cleaned in 10% sodium hydroxide solution at 1.5 A/dm<sup>2</sup> for 5 min. then specimens were thoroughly rinsed with deionized water and immersed in 50 vol.% sulphuric acid solution for deoxidization for 30 s. After deionized water rinse, specimens were transferred immediately to the plating solution.

Electroless nickel bath was used for preparing the Ni-P and composite coatings. Bath contains nickel sulphate 21 g/l, propionic acid 3 g/l and small amounts of lead nitrate and operated at pH 4.0 - 4.5 and temperature  $90\pm 2$  °C. Alumina powders used were of the size 0.8  $\mu\text{m}$ , 6  $\mu\text{m}$  and 15  $\mu\text{m}$  (Acola) and the alumina concentration in solution was kept at 30 g/l. The plating time is 2 hours and the obtained layers have thickness of about 50  $\mu\text{m}$  (thickness determined by electrochemical stripping techniques). After plating, the specimens were taken out then thoroughly rinsed with deionized water and air dried at room temperature. Then these specimens were used for further characterization.

Surface morphology of the obtained electrolessly deposited layers was investigated using scanning Electron Microscopy (SEM). The composition of the alloys was determined by Energy Dispersion Spectroscopy (EDS). Texture and phase formation of the films were analyzed by X-ray Diffractometer (XRD).

### III - RESULTS AND DISCUSSION

Figs. 1 and 2 show SEM images and corresponding EDS analyses of the electrolessly deposited composite Ni-P/ $\text{Al}_2\text{O}_3$  with different particle sizes of 0.6  $\mu\text{m}$ , 8  $\mu\text{m}$  and 15  $\mu\text{m}$ . It can be seen that in all cases, the sedimentation of  $\text{Al}_2\text{O}_3$  on the deposited layers due to high interaction energy between freshly formed Ni-P layer and  $\text{Al}_2\text{O}_3$  particles in the solution do not occur. Since Al signals appear in EDS spectra (Fig. 2), it can be concluded that the  $\text{Al}_2\text{O}_3$  particles are already trapped in the deposited layers.

Composition of the deposited Ni-P alloys and content of dispersed  $\text{Al}_2\text{O}_3$  particles in the layers obtained from EDS analyses are summarized in Tab. 1. Results show that the alumina particle size does not influence on composition of the Ni-P matrix e.g the composition of the Ni-P is in the range of  $\text{Ni}_{90}\text{P}_{10}$  to  $\text{Ni}_{89}\text{P}_{11}$ . Meanwhile,  $\text{Al}_2\text{O}_3$  content decreases with increasing alumina particle sizes. The maximum  $\text{Al}_2\text{O}_3$  content (12%) was

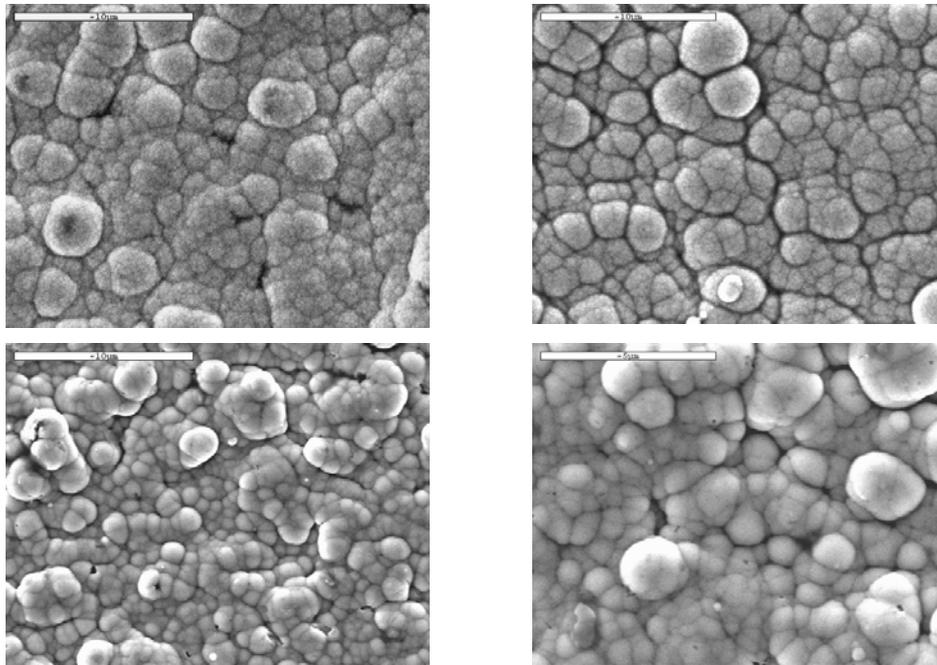


Fig. 1: SEM images of electrolessly deposited composite NiP- $\text{Al}_2\text{O}_3$  with different size of  $\text{Al}_2\text{O}_3$  particles (a) without  $\text{Al}_2\text{O}_3$ ; (b)  $d = 15 \mu\text{m}$ ; (c)  $d = 6 \mu\text{m}$ ; (d)  $d = 0.8 \mu\text{m}$

obtained with particle size of 0.8  $\mu\text{m}$ . This result can be explained based on the formation mechanism of composites. According to Hajdu, the formation of composite coatings consist of 3 steps: (i) transportation of  $\text{Al}_2\text{O}_3$  particles from electrolyte to the surface through convection or diffusion (ii) adsorption of the particles on the

surface and (iii) deposited Ni-P buries the particles [1, 2]. Thus smaller  $\text{Al}_2\text{O}_3$  particles, which have higher specific surface energy compared to that of coarser ones, will tend to adsorb more on the surface. As a result, the amount of  $\text{Al}_2\text{O}_3$  particles in the obtained Ni-P/ $\text{Al}_2\text{O}_3$  composites will increase.

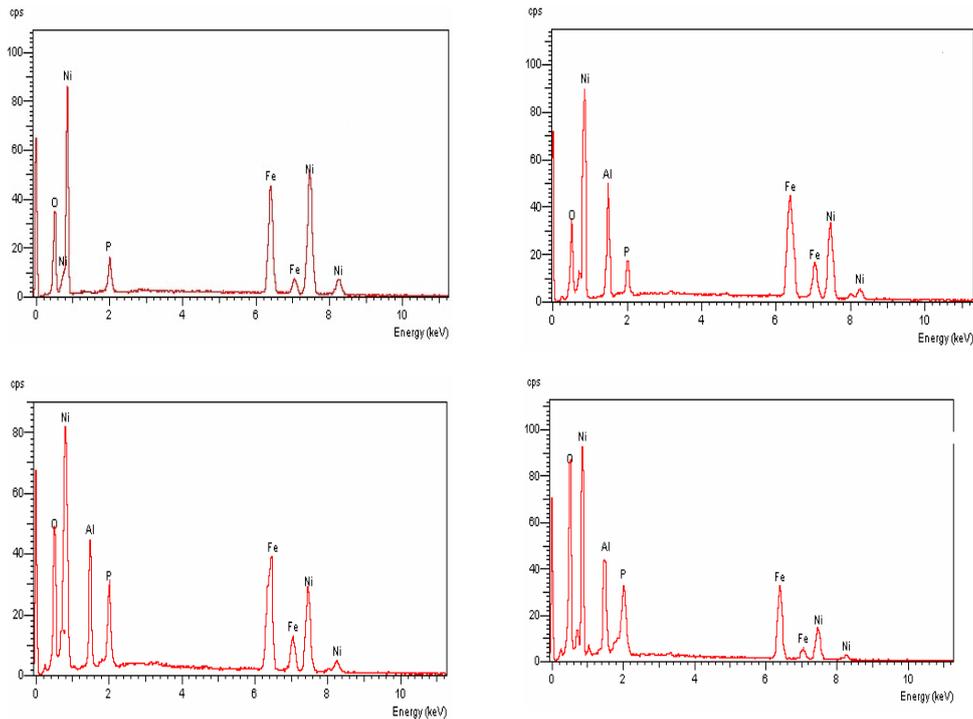


Fig. 2: EDS spectra of electrolessly deposited composite Ni-P/ $\text{Al}_2\text{O}_3$  with different size of  $\text{Al}_2\text{O}_3$  particles (a) without  $\text{Al}_2\text{O}_3$ ; (b)  $d = 15 \mu\text{m}$ ; (c)  $d = 6 \mu\text{m}$ ; (d)  $d = 0,8 \mu\text{m}$

Tab. 1: Composition of as-plated electroless Ni-P and composite coatings determined by EDS analysis

Type of coatings	Ni (wt.%)	P (wt.%)	$\text{Al}_2\text{O}_3$ (wt.%)	Ni-P matrix
Ni — P	89,0	11,0	0	$\text{Ni}_{89}\text{P}_{11}$
Ni — P — $\text{Al}_2\text{O}_3$ (0.8 $\mu\text{m}$ )	79,2	8,8	12,0	$\text{Ni}_{90}\text{P}_{10}$
Ni — P — $\text{Al}_2\text{O}_3$ (6 $\mu\text{m}$ )	71,9	8,1	10,0	$\text{Ni}_{90}\text{P}_{10}$ $\text{Ni}_{91}\text{P}_9$
Ni — P — $\text{Al}_2\text{O}_3$ (15 $\mu\text{m}$ )	82,8	8,2	9,0	

Fig. 3 displays XRD patterns of the composite with alumina particle sizes of 0.8  $\mu\text{m}$ , 6  $\mu\text{m}$  and 15  $\mu\text{m}$ . It can be observed that the obtained Ni-P matrix has semi-amorphous structure, indicated by very low intensity of Ni(111) and Ni(111) signals. This result also

agrees with the EDS analyses, which show that the phosphorus content in Ni-P layer is around 9 - 10%. According to the phase diagram, this composition corresponds to a partly amorphous structure of Ni-P and dispersed Ni crystalline inside the amorphous matrix [1, 2]. This

structure also promotes a high corrosion resistant and high hardness of the Ni-P layer [1, 7]. It is also interesting to note that the  $Al_2O_3$

peak intensities in XRD spectra also decrease with increasing particle size due to the increase of  $Al_2O_3$  content in the composite layers.

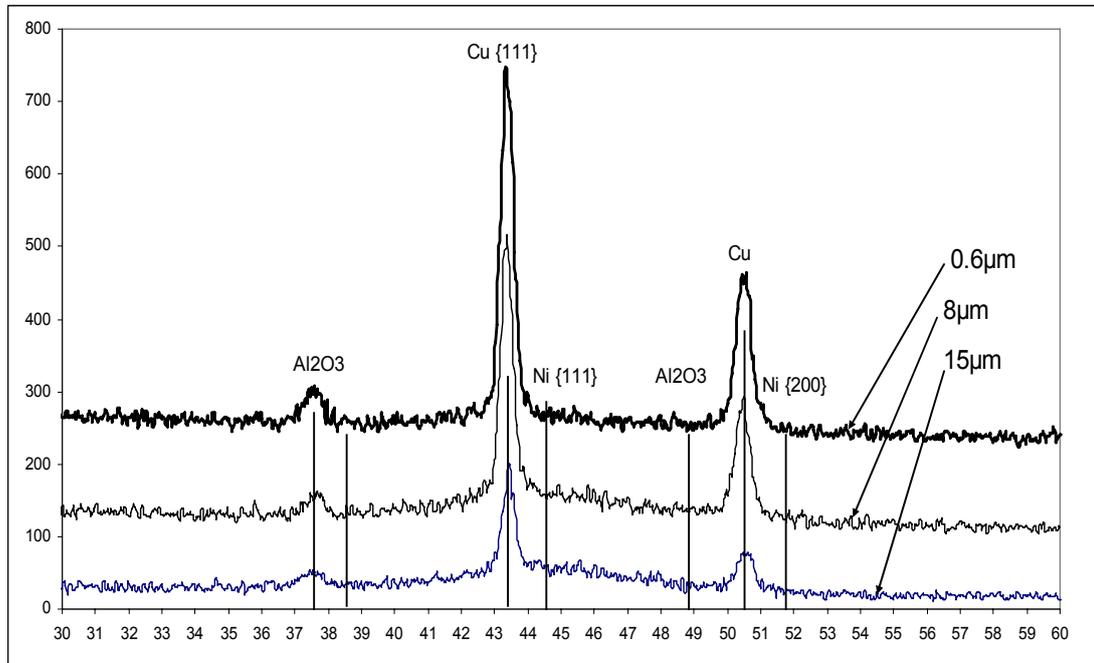


Fig. 3: XRD patterns of electrolessly deposited composite Ni-P/ $Al_2O_3$  with different size (d) of  $Al_2O_3$  particles (a) without  $Al_2O_3$ ; (b)  $d = 15 \mu m$ ; (c)  $d = 8 \mu m$ ; (d)  $d = 0.6 \mu m$

Table 2: Hardness of composite coatings

Type of coatings	HV hardness	HB hardness
Bare steel	216	216
Ni — P	238	226
Ni — P — $Al_2O_3$ (0.8 nm)	266	253
Ni — P — $Al_2O_3$ (6 $\mu m$ )	272	258
Ni — P — $Al_2O_3$ (15 $\mu m$ )	294	279

Hardness of composite layers with different sizes of co-deposited alumina particles was measured and summarized on Tab. 2. Results show that hardness increases with increasing alumina particle size. Since the composition of the matrix Ni-P does not change much as alumina particle size changes (Tab. 1), this result can be explained by the two effects occurring during the deposition process. The first

effect is the decrease of the  $Al_2O_3$  content in composite layers as particle size increases. The second reason is that as particles become smaller, the distribution of alumina particle will be more evenly in the composite layers.

#### IV - CONCLUSION

Effect of alumina particle size on

morphology, particle content and hardness of electrolessly deposited composite Ni-P/Al<sub>2</sub>O<sub>3</sub> layers was examined using alumina particles with sizes of 0.8µm, 6µm and 15µm. Results showed that as particles size increased, the composition of Ni-P matrix did not change remarkably e.g the composition of the obtained matrix ranged from Ni<sub>90</sub>P<sub>10</sub> to Ni<sub>91</sub>P<sub>9</sub>. Meanwhile, Al<sub>2</sub>O<sub>3</sub> content in the composite increased as particle size decreased. As a result, hardness of the obtained layer increased with decreasing particle size and maximum hardness was achieved with particle size of 0.6µm.

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