

A COMPARATIVE STUDY OF THE PROPERTIES OF ZINC-SiO₂ AND ZINC-Al₂O₃ COMPOSITE LAYERS

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Abstract: The aim of this work was the electrodeposition of SiO₂ and Al₂O₃ particles into zinc matrix in order to improve the surface properties. The effect of loading particles in bath on composition, morphology, structure of deposits and their influence on the microhardness, roughness and corrosion resistance of zinc-SiO₂ and zinc-Al₂O₃ composite layers was investigated. Another objective of the present study is to develop the plating baths with suitable compositions and establish the optimum electrodeposition conditions for obtaining good quality zinc-SiO₂ and zinc-Al₂O₃ composites.

Keywords: SiO₂, Al₂O₃, electrochemical deposition, composites.

1. INTRODUCTION

Metallic matrix composites with a wide range of matrix materials such as copper, aluminum, nickel, chromium, cobalt, zinc, etc and second-phase ceramic particles have been produced. The metallic matrix has ductility and toughness properties combined with ceramic characteristics (high strength, hardness) leading to attractive physical and mechanical properties for the new composite material. The reinforced phase includes particulates, whiskers or short fibers [1,2].

The properties of metallic matrix composites can be controlled by the size and volume fraction of the dispersed phase (DP) as well as by the nature and properties of the matrix material. An optimum set of mechanical properties can be obtained when fine, thermally stable ceramic particulates are dispersed uniformly in the metal matrix. [3,4,5]

Metallic matrix composites with ceramic oxides as DP can be manufactured by electrodeposition method obtaining thin-film layers with improved wear resistance, good high-temperature stability and improved friction properties which are important characteristics for industrial applications.

2. EXPERIMENTAL CONDITIONS

The composite layers made using the electrochemical method can be obtained using different methods like: depositing in a centrifugal field, depositing on a centrifugal cathode, direct current or alternative current depositing, depositing with or without the recirculation of the electrolyte, etc[6].

In our experiments we used the direct current electrodeposition technology in order to obtain the composite layers. For obtaining composites with adherence, smoothness, good mechanical and corrosion properties must be respected some experimental parameters such as pH, current density, temperature, stirring, deposition time and electrolyte composition; these parameters are presented in Table 1:

Table 1: The working parameters for electrodeposition of Zn-SiO₂ and Al₂O₃ and composite coatings

Electrolyte composition	ZnSO ₄ ·7H ₂ O=315g ^l ⁻¹ ; Na ₂ SO ₄ ·10H ₂ O=75g ^l ⁻¹ Al ₂ (SO ₄) ₃ ·18H ₂ O=40g ^l ⁻¹
pH	3-4

Temperature (°C)	20
Current density (A·dm ⁻²)	2,3,4
Magnetic stirring (rpm)	300
Electrodeposition time (min.)	60

The surfaces morphologies of the electrodeposits were characterized with ZEISS DSM-960A scanning electron microscopy(SEM) and EDX analysis using Philips XL-30FEG.The microhardness of the samples was analyzed by a CV-400DAT2 NAMICON durimeter with a down force fixed at 50 grams and layers roughness with NAMICON TR 100.

3. RESULTS AND DISCUSSIONS

Detailed investigation on the morphology of the surface of composite coatings deposited at different current densities and different particle loadings in electrolyte has been done. The deposit quality was first identified by visual examination watching to be uniform and adherent. Then we've made the next step-EDX and SEM characterization of the composite layers.

The dispersed phases characterization

The dispersed phase used in the codeposition were SiO₂ and Al₂O₃ microparticles with the average size range between 1-5 μm. The aspect of the used microparticles is shown in Figure 1:

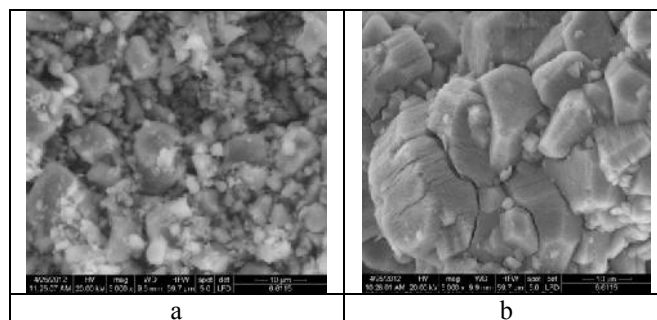


Figure1. SEM analysis of the SiO₂ microparticles(a) and Al₂O₃ microparticles(b)

The EDX analysis and DP embedding in zinc layers

The presence of the dispersed phase in the composite layer has been highlighted by EDX spectra. Both ceramic phases are present in zinc matrix(Figure 2).Oxygen is also present in EDX spectra proving that the second phase is embedded in zinc matrix in the form of oxides.

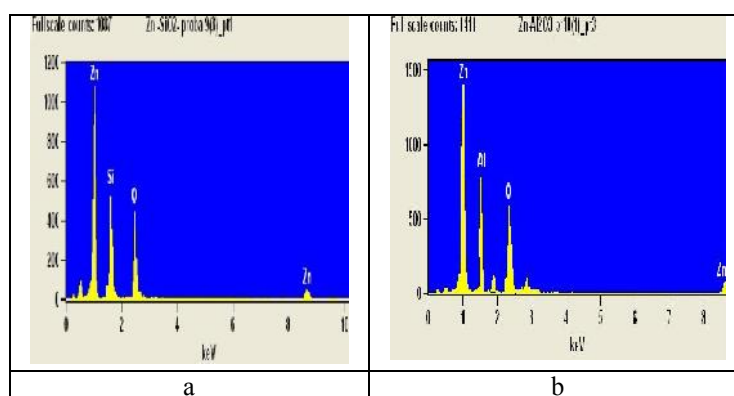


Figure2: The EDX analysys for a)zinc-SiO₂ and b) zinc-Al₂O₃ composites(30gl⁻¹) obtained with the following parameters: 3Adm⁻²,60min,300rpm

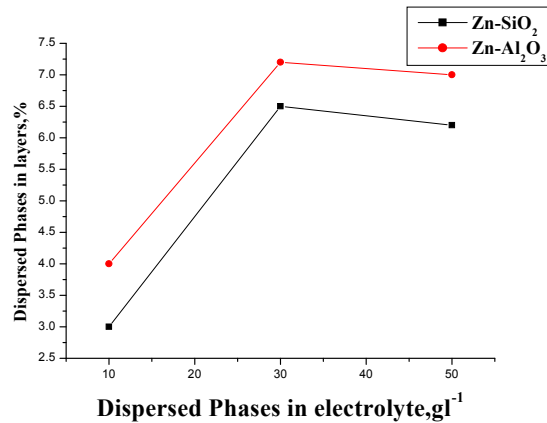


Figure 3. Variation of dispersed phases in electrolyte in accordance with dispersed phases embedded in layers using the following parameters: $3A/dm^2$, 60min, 300rpm, $30gl^{-1}$

It has been observed that the amount of embedded DP increases with increasing concentration of suspended particles in the electrolyte. The highest value for the embedded Al_2O_3 DP in zinc matrix is 6.5% mass percentage obtained for $30gl^{-1}$ particles loading in electrolyte, $3 Adm^{-2}$ current density and electrolyte agitation of 300rpm; as for Zn-SiO₂ composites maximum of embedded phase is 7.2% mass percentage obtained for $30gl^{-1}$ particles loading in electrolyte, $3 Adm^{-2}$ current density and electrolyte agitation of 300rpm. SiO₂ particles are embedded easier in zinc matrix than Al_2O_3 particles.

The microstructure of zinc-SiO₂ and zinc-Al₂O₃ composite layers

The microstructure of zinc-Al₂O₃ composite layers is different compared to pure zinc layer. Oxide particles dispersed in matrix and crystallization of zinc grains in co-deposition process makes relevant changes in depositing structure.

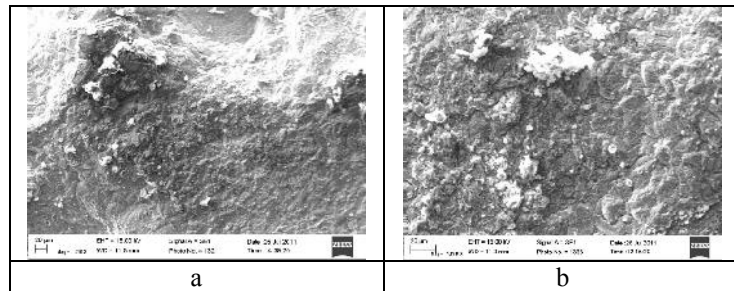


Figure 4: SEM Micrographs of zinc matrix composites obtained at: $3 Adm^{-2}$, $30gl^{-1}$ DP, 60 min, 300rpm. Figure a: Zn-SiO₂ composites and figure b: Zn-Al₂O₃ composites

The SEM analysis indicates an uniform deposit accomplished for $3 Adm^{-2}$, $30gl^{-1}$ DP in zinc matrix, 60 min. deposition time and 300rpm stirring.

Microhardness of the electrodeposited layers

The presence of particles in the deposited layers changed both their structure and properties. As for microhardness a comparative study has been done. The study shows that the microhardness values of composite layers are higher than microhardness values of pure zinc layers (Figure 5):

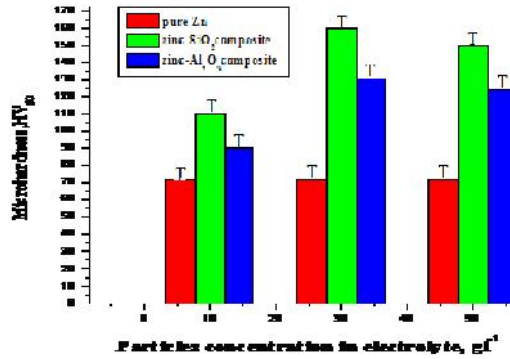


Figure 5: Average microhardness values for samples obtained by electrodeposition according to the particles concentration in electrolyte

The highest average value (158 HV₅₀) is obtained for zinc-SiO₂ at 3Adm⁻², 60min, 300rpm, and 30gl⁻¹ electrocodeposition parameters. At the same parameters, for Zn-Al₂O₃ the highest average value is 131 HV₅₀. Zinc-SiO₂ composite layer is harder than zinc-Al₂O₃ layer.

Roughness of the electrodeposited layers

Another change in properties made by the ceramic oxides dispersed phases is the roughness of the layers.

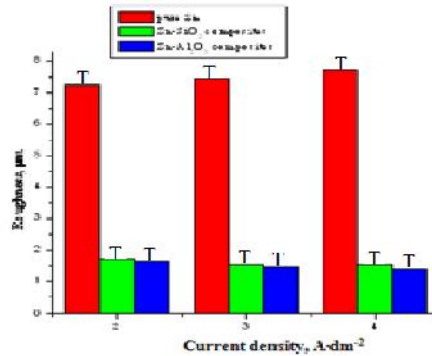


Figure 6: Roughness variation of Zn layers and Zn composite layers for different current densities

In contrast to the microhardness data, the smoothing effect is produced by the amount of DP embedded in layers along with zinc. The lowest roughness average values for composite coatings were obtained at the following parameters: 4A/dm⁻², 60min, 300rpm and 30gl⁻¹ particles in electrolyte for zinc-roughness value of pure zinc layers is higher than roughness values of zinc composites. The smoothest composite surface is obtained for zinc-Al₂O₃ composites which is 1.3µm Ra.

Corrosion comparative study of Zn coatings

The corrosion behaviour of Zn coatings (composite layers and pure zinc layers) was investigated by electrochemical methods in a special facility prepared in order to determine the potentiodynamic corrosions. From the experimental data obtained from measurements we chose representations in the form of Tafel polarization curves. The analysis of the graphical representations made it possible to characterize the corrosion of pure zinc and composite samples made at 3Adm⁻², 60min, 300rpm deposition parameters. The results are given in Figure 7:

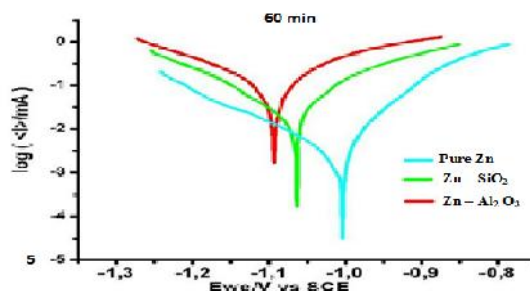


Figure 7: Tafel polarization curves for pure Zn; Zn- SiO₂ and Zn-Al₂O₃, composite layers

Corrosion behavior is appreciated by electrochemical tests showing lower resistance of pure zinc layer compared with Zn composites (corrosion potential of pure Zn is higher than corrosion potential of the composite coatings). The best corrosion resistance is obtained for Zn-Al₂O₃ composite layers.

4. CONCLUSIONS

We have characterized zinc composite coatings where SiO₂ and Al₂O₃ particles are embedded in the metal matrix as dispersed phases. Zinc composites presents good adhesion on substrate and uniform appearance. The composite coatings quality analysis was performed using EDX and SEM analysis which shows that the embedded oxides second phase in zinc matrix depends on their concentration in electrolyte and current density. Microhardness of zinc composites was improved compared with Zn layers. The highest average microhardness value was obtained for Zn-SiO₂ composites.

Zn layers present a large variation of roughness values depending on the amount of embedded particles in layers; the lowest roughness average values were obtained for the following parameters: 4Adm⁻², 60min, 300rpm, and 30g l⁻¹ particles in electrolyte for zinc-Al₂O₃ composites. Pure zinc layers had the highest roughness average values.

The corrosion behavior of composite layers in Zn matrix was studied by potentiodynamic method; the results show an improvement of corrosion behavior of Zn composites compared with pure zinc layers.

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