

# COMPOSITE COATINGS IN ZINC MATRIX WITH SIO<sub>2</sub> IN DISPERSED PHASE OBTAINED BY ELECTRODEPOSITION

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**Abstract:** The study describes the conditions for obtaining zinc matrix composites using  $SiO_2$  as dispersed phase by direct current electrodeposition. Authors made experiments for setting the best codeposition parameters. In this work are presented the results about the influence of the main technological codeposition parameters on the chemical composition, structure and properties of the composite coatings.

Keywords: electrochemical deposition, zinc matrix, coatings composite, SiO2.

# **1. INTRODUCTION**

Metallic coatings are often created by chemical or electrochemical plating methods, heat spraying, cladding and vacuum plating. Composites are combined heterogeneous materials created at least by two phases separated from each other by interfaces[1]. The composite coatings obtained by including the dispersed-phase particles in the metallic matrix has a wide technological interest for many applications. The advantages of creating composites by using electrodeposition techniques are:

- This technology is not very expensive.
- It is possible to regulate precisely the thickness of the coatings.
- It can be obtained composite coatings with matrixes of wide ranges of metals and particles [2].

Electrodeposition is used for improving the mechanical properties such as wear, corrosion improvement and hardness of the coating surfaces. Coatings of zinc are of great practical importance because of their capacity to protect ferrous substrates against corrosion[3,4,6]. In addition for pure zinc coatings, composite coatings of zinc-SiO<sub>2</sub> have attracted the attention of the researchers due to their characteristics concerning of the effect of inert oxide particles content on the crystalline structure and properties of the composites [7,9,10]. Zinc matrix composite coatings are often used for automotive, aerospace and marine parts.

# 2. EXPERIMENTAL CONDITIONS

The quality of the deposit depends on a good correlation between dispersed phase characteristics, bath composition, current density, pH, stirring, etc. The amount of the embedded particles plays an important role in improving the new material properties.

An SP-150 type potentiostat, a magnetic stirring machine and an electrolyte tank were used in order to obtain zinc composite coatings. The schematics of electrodeposition method are shown in Figure 1:

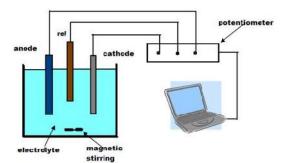


Figure 1: Schematics of the experimental setup

It was used a solution volume of 200 ml electrolyte and the experiments took place at 20° C. As for zinc layers in order to determin the optimal electrodeposition parameters were used steel samples - base metal, substances for the preparation of electrolytic solutions,  $SiO_2$  powder (1-5µm size) as dispersed phase for the coatings.

The active electrodes surfaces were degreased and polished. Concentrations of  $SiO_2$  particles were 10g/l, 30g/l and 50g/l in the electrolyte solution.

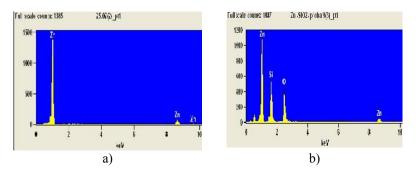
In this work a high purity rolled zinc anode (99,9%) and cathode of steel having an active area of 16 cm<sup>2</sup> were used. The cathode face that did not come in direct contact with the anode was insulated. The working parameters of the deposition are shown in Table1:

$ZnSO_4 \cdot 7H_2O=315g/l;$
$Na_2SO_4 \cdot 10 H_2O = 75g/l;$
$Al_2(SO_4)_3 \cdot 18H_2O = 40g/l$
3 - 4
25
2,3,4
300
60

Table 1: The working parameters for electrodeposition of Zn-SiO<sub>2</sub> composite coatings

# **3. EXPERIMENTAL RESULTS AND DISCUSSIONS**

The EDX analysis shows that  $SiO_2$  particles were present in layers along with zinc. Different  $SiO_2$  percentages reported in the composite coatings are depending on particle concentrations in the bath and current densities; the re-sults are given in Figures 2 and 3:



**Figure 2**: The EDX analisys for a) pure Zn and b) Zn-SiO<sub>2</sub> composites(30g/l) obtained with the following parameters: 3A/dm<sup>2</sup>,120min,500rpm

Figure 2 presents the difference between EDX analysis of the pure zinc layers(Figure a) and composite layers. The presence of oxide particles in zinc matrix is shown in Figure b.

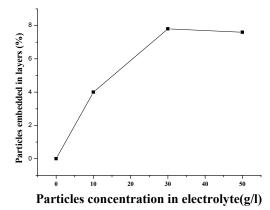


Figure 3: The variation of the particles concentration in electrolyte according to the particles embedded in layers

In Figure 3 has been observed that the amount of embedded particles increases with increasing the concentration of suspended particles in electrolyte. The highest value for the embedded phase volume is 8% obtained for 30 g/l,confirming a good adherence and uniform distribution of DP using the following deposition parameters: current density of 3 A/dm<sup>2</sup>, time 60 min, 500 rpm stirring,temperature  $20^{\circ}$ C. The percentage of the embedded particles in layer is decreasing at 50g/l DP in electrolyte.

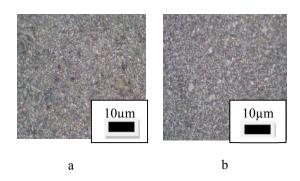


Figure 4:Surface Optical Microscopy (x400) of the coatings: a) Zn pure coating obtained at 3A/dm<sup>2</sup>, b) Zn-SiO<sub>2</sub> composite coatings for 3 A/dm<sup>2</sup> and 30g/l DP in electrolyte

In Figures 4 and 5 is presented the effect of the  $SiO_2$  inert particles on the structure; the structure modifications are highlighted by optical microscopy and SEM analysis.

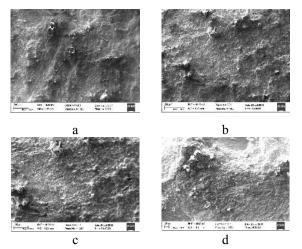


Figure 5:SEM Micrographs of Zn-SiO<sub>2</sub> coatings for different magnitudes obtained at: 3 A/dm<sup>2</sup>, 30g/l DP ,60 min ,500rpm

Using SEM microscopy analysis and optical microscopy(OM) analysis it was observed homogeneous distribution of additional phase in zinc matrix. The presence of  $SiO_2$  in zinc matrix makes modifications of structure(roughness) and mechanical properties(microhardnes).

Study of microhardness is important both for estimating mechanical properties or wear resistance of the composite layers. The microhardness range of the layers is controlled by the amount of DP embedded in zinc matrix. The microhardness Vickers test was made using a CV-400DAT2 NAMICON durimeter.

The microhardness of the electrodeposited layers varies quite large; the results are shown in the chart below:

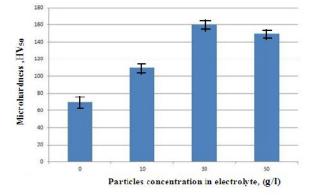


Figure 6: Microhardness ( $HV_{50}$ ) of the samples obtained by electrodepositing according to the particles concentration in electrolyte

It is noted that the average microhardness values of the composite layers are bigger then the average microhardness values of the pure zinc. This increasing of the microhardness values can be attributed to two different phenomenons :firstly, the strengthening effect of the reinforcing phase itself and secondly of the structural modification of Zn electrodeposited crystallites.

Microstructure of  $Zn-SiO_2$  presents a different morphology compared with the pure Zn layers. This depends on the particles volume in electrolyte as well as those particules embedded in composite layers. Surface morphology makes changes in roughness measured by Ra parameter.

There are quite large variations of the roughness values obtained for different current densities. The results are presented in Figure 7:

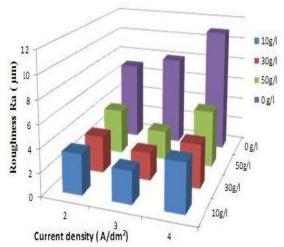


Figure 7: Roughness variation of Zn-SiO<sub>2</sub> composite layers for different current densities and different particle loadings in electrolyte

Values for roughness of the composite layers are lower then the roughness determined for pure zinc. The presence of  $SiO_2$  particles in layers proves a change of properties comparative with simple Zn layers. The particle loading of 30g/l makes the composite surface smoother then 10g/l or 50g/l loading. The smoothest surface is obtained for 3A/dm<sup>2</sup> current density.

### **4.CONCLUSIONS**

The experimental research highlights the conditions for obtaining  $zinc-SiO_2$  composite coatings using electrodeposition method.

The composite coatings quality analysis has been made both qualitatively and quantitatively by using modern methods such as EDX and SEM analysis. These have highlighted inclusion of the dispersed phase and homogeneous distribution of the additional phase for some electrochemical parameters.

It has been stipulated experimental conditions for obtaining the best composite layers from the point of view of the best microhardness and roughness for composite layers.

#### REFERENCES

[1] Schlesinger M.,:Modern Electroplating,5th Edition,ISBN: 978-0-470-16778-6,2010.

[2] ] Hovestad A., Janssen L.J.J.,:Electrochemical Codeposition of Inert Particles in a Metallic Matrix, Journal of Applied Electrochemistry, 25, pag.519,1995.

[3] Khorsand S., Raeissi K., Golozar M.A.: An Investigation on the Role of Texture and Surface Morphology in the Corrosion Resistance of Zinc Electrodeposits, Corrosion Science, 53, pag. 2676-2678, 2011.

[4] Binder L., Kordesch K.,:Electrodeposition of Zinc Using a Multi-Component Pulse Current, Electrochimica Acta,volume 31,Issue 2, pag.255–262,1986.

[5] Zhang Z.,Leng W.H., Cai Q.I., Cao F.H., Zhang J.Q.,:Study of the Zinc Electroplating Process Using Electrochemical Noise Technique, Journal of Electroanalytical Chemistry, volume 578, Issue 2,pag.357–367,2005.

[6] Zhang Q.,Hua Y.,:Kinetic Investigation of Zinc Electrodeposition from Sulfate Electrolytes in the Presence of Impurities and Ionic Liquid Additive [BMIM]HSO<sub>4</sub>, Materials Chemistry and Physics,volume 134, Issue 1, pag. 333–339,2012.

[7] Hashimoto S.,Masaki A.,:The Characterization of Electrodeposited Zn-SiO<sub>2</sub> Composites before and after Corrosion Test, Corrosion Science, volume 36, Issue 12, pag.2125–2137,1994.

[8] Fransaer J., Celis J.P., Roos J.R., Analysis of the Electrolytic Codeposition of Nonbrowian Particles with Metals., Journal of the Electrochemical Society, 139, pag.413,1992.

[9] Kondo K., Tanaka A.O.Z.,:Electrodeposition of Zinc-SiO<sub>2</sub> Composite, Journal of the Electrochemical Society, 147(7): pag.2611,2000.

[10] Khan T.R., Erbe A., Auinger M., Electrodeposition of Zinc–Silica Composite Coatings: Challenges in Incorporating Functionalized Silica Particles into a Zinc Matrix, Science and Technology of Advanced Materials, 2:10.1088/1468-6996/12/5/055005, published 14 September 2011.

[11] Low C.T.J., Wills R.G.A., Walsh F.C.,: Electrodepositing of Composite Coatings Containing Nanoparticles in a Metal Deposit, Surface and Coatings Technology, 201, pag. 371-383, 2006.

[12] Celis J.P., Roos J.R., Buelens C.,: A Mathematical Model for the Electrolytic Codeposition of Particles with a Metallic Matrix, J. of the Electrochemical Society, 134 (6),1402–1408,1987.

[13] Tolumoye J., Tuaweri J., Wilcox G.D., Behaviour of Zn-SiO<sub>2</sub> Electrodeposition in the Presence of N,N-dimethyldodecylamine, Surface and Coatings Technology, 2:10.1016, pag. 5921, 2006.

[14] Lin Z.F., Zhang D., Wang Y., Wang P., A Zinc/Silicon Dioxide Composite Film: Fabrication and Anticorrosion Characterization, Materials and Corrosion, volume 63, Issue 5, pag. 416–420, 2012.

[15] Socha R.P., Fransaer J.,:Mechanism of Formation of Silica-Silicate Thin Films on Zinc, Thin Solid Films, 488,pag.45,2005.