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RESEARCH REGARDING ALUMINIUM ELOXATION FROM PHOSPHORIC ACID ELECTROLYTE

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Abstract: The study presents the influence of production parameters on the aluminium eloxation from H_3PO_4 solution. The thickness and aspect of the deposited Al_2O_3 were determined. The electrodeposition experiments was to determine the optimum conditions for the producing of bright, continuous, adherent and compact Al_2O_3 layers.

Keywords: eloxation, aluminium, Al_2O_3, phosphoric acid, thickness

INTRODUCTION

The electrodeposition of Al_2O_3 layer on the aluminium surface and its alloys presents importance for the protective quality and hardness of the coating and for the economic process. The anodic oxidation of aluminium is used for to improve the corrosion resistance of following layers with paints and for decorative purposes. There has been numerous reports in the application of Al_2O_3 in biodevices, in drug delivery, in the adsorption of volatile organic compounds.

The method is known with alumilit name in U.S.A. and Great Britain and eloxal GS in the majority of E.U., but in our country, is used the eloxation term for the electrochemical oxidation of aluminium.

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The employed acid electrolytes for aluminium eloxation are based on sulphuric acid (Sulka et al., 2002), (De Graeve et al., 2003), (Sulka and Parkola, 2007) and (Belwalkar et al., 2008), oxalic acid (Li and Huang, 2007), phosphoric acid (Chu et al., 2003) and (Araoyinbo et al., 2012), chromic acid (Stojadinovic and Belca, 2004) and acid mixtures (Shingubara et al., 2004) and (Sachiko et al., 2005).
The study presents the influence of production parameters on the aluminium eloxation from H_3PO_4 solution. The thickness and aspect of the deposited Al_2O_3 were determined.

MATERIALS AND METHODS

The reagents used were of analytical grade: H_3PO_4 85% p.a., NaOH p.a. and HCl 36.5% p.a. All solutions were prepared with double distilled water.
The H_3PO_4 electrolyte used for this study of aluminium eloxation had the concentration between 10% – 25%.
Before the electrodeposition experiments, the aluminium electrodes were polished with abrasive paper and then submitted to chemical degreasing in an aqueous solution of sodium hydroxide 20% and activation in HCl 1:1 solution.
The aluminium eloxation was performed by galvanostatic electrolysis, in different experimental conditions for 15 minutes, in an Hull stiplex cell, with a capacity of 267 cm^3. The anode was an aluminium strip, with 99.9% purity. The cathode was a lead strip. The surface ratio of the aluminium/lead electrodes was 1/1. Stirring was done with a magnetic bar. The current density was controlled by a Princeton Applied Research galvanostat, model 173.
The thickness of the deposited Al_2O_3 on aluminium anode was determined by the microscopic method, used a metallographic microscope MMB 2200. Also, in order to determine the current efficiency, the aluminium anode was weighed on an analytical balance before and after each eloxation.
After the aluminium eloxation, the anode was immersed for 5 minutes in a double distilled water at 90 ^oC, in order to improve the corrosion resistance and to decrease the porosity of Al_2O_3 layer.

RESULTS AND DISCUSSIONS

By the passing of electric current through the H_3PO_4 solution, the formed oxygen reacts with the aluminium anode and generates on its surface an

Al_2O_3 layer. There is a partial dissolution of Al_2O_3 in electrolyte solution. Therefore, this reaction leads to pores in the Al_2O_3 deposit.
The physical and protective properties of Al_2O_3 layer depend from the aluminium composition, the nature and the electrolyte concentration and the production parameters.

The effect of current density
The aluminium eloxation was studied at 20 ^oC, using 20% H_3PO_4 solution. The effect of current density on current efficiency and both the thickness and aspect of the deposited Al_2O_3 was studied.
The current densities employed were between 1 and 2.5 A dm^{-2}.
The relationship between Al_2O_3 layer thickness and aspect and the current density is presented in Table 1.
At low current densities, the Al_2O_3 film is dull and porous, while at very high current densities, it becomes pulverous because there is an heating of H_3PO_4 solution by Joule effect which produces a dissolution of Al_2O_3 deposit.

Table 1. The effect of current density on the Al_2O_3 deposit thickness and aspect: 20 ^oC; H_3PO_4 concentration = 20%.

current density (A dm^{-2})	Al_2O_3 thickness (μm)	Al_2O_3 deposit aspect
1	1.14	dull, porous
1.5	1.42	bright
2	1.32	bright
2.5	1.08	pulverous

The optimum current density is 1.5 A dm^{-2} and in this case, the Al_2O_3 layer is bright, adherent and continuous and the current efficiency and the thickness of film are maximum.

The effect of H_3PO_4 concentration
The influence of H_3PO_4 concentration on current efficiency and both the thickness and aspect of the deposited Al_2O_3 was studied at 20 ^oC and at a current density of 1.5 A dm^{-2}.
The H_3PO_4 concentration was varied from 10% to 25%.
The thickness and aspect of the deposited Al_2O_3 layer versus electrolyte concentration is presented in Table 2.

Table 2. The effect of H_3PO_4 concentration on the Al_2O_3 deposit thickness and aspect: 20 ^oC, i = 1.5 A dm^{-2}.

H_3PO_4 concentration (%)	Al_2O_3 thickness (μm)	Al_2O_3 deposit aspect
10	1.16	dull, porous
15	1.26	bright
20	1.54	bright
25	1.62	pulverous

Increasing the H_3PO_4 concentration results in an increase of Al_2O_3 film thickness.

At 10% H_3PO_4 concentration, the Al_2O_3 layer is dull and porous. The H_3PO_4 concentrations, i.e. 15%-20%, favor the formation of bright, compact, adherent and resistant Al_2O_3 deposits.

The 25% H_3PO_4 concentration is not advisable for the aluminium eloxation because leads in the producing of pulverous Al_2O_3 deposit which is not for decorativ aspect. In this case, the dissolution rate of Al_2O_3 film is greater than its forming rate and result pulverous deposits.

The values of current efficiency show an electrolyte concentration of 15%-20% for the producing of bright Al_2O_3 deposits.

The effect of temperature

The aluminium eloxation was performed at a current density of 1.5 A dm^{-2}, using 20% H_3PO_4 solution.

The tested temperatures were: 15 °C, 20 °C, 25 °C and 30 °C.

The dependence of Al_2O_3 deposit thickness and aspect versus temperature is presented in Table 3.

Table 3. The effect of temperature on the Al_2O_3 deposit thickness and aspect: i = 1.5 A dm^{-2}, H_3PO_4 concentration = 20%.

temperature (^0C)	Al_2O_3 thickness (μm)	Al_2O_3 deposit aspect
15	1.34	dull, porous
20	1.51	bright
25	1.68	pulverous
30	1.92	pulverous

The Al_2O_3 thickness layer increases when the temperature increases.

At 15 ^{0}C temperature, a dull and porous Al_2O_3 deposit is obtained which is not good for following protection. The optimum temperature for the aluminium eloxation is 20 ºC and the Al_2O_3 film is bright, continuous and compact. The dissolution process of Al_2O_3 deposit is intensified by the increase of temperature, so that temperatures above 20ºC generate pulverous Al_2O_3 coatings.

CONCLUSIONS

The goal of this set of electrodeposition experiments was to determine the optimum conditions for the producing of bright, continuous, adherent and compact Al_2O_3 layers, at high current efficiency. The analysis of the experimental results can define preferred or optimum production parameters, i.e. the conditions that produce good quality Al_2O_3 deposition, with anticorrosive properties and pleasant aspect.

The study for the aluminium eloxation from H_3PO_4 solution show that:

- The optimum current density is 1.5 A dm^{-2}.
- The optimum H_3PO_4 concentration is 20%.
- The optimum temperature is 20 °C.

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