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The Influence of Input Factors of Aluminium Anodizing Process on Resulting Thickness and Quality of Aluminium Oxide Layer

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Abstract

In order to optimize the technological process of aluminium anodic oxidation, the possibilities of usage of sodium chloride in the electrolyte has been studied, since very small concentration of sodium chloride allows us to reduce concentration of other components of the electrolyte. Also the influence of sodium chloride concentration in the electrolyte on the final thickness and quality of the formed anodic aluminium oxide (AAO) layer has been investigated in this paper. In contrast to common anodizing experiments, in which the influence of only one separate factor at a time is considered, in our research all relevant factors (four chemical factors) were varied simultaneously according to the methodology of statistical experimental design, i.e. design of experiments (DOE). Based on the evaluation of experimentally obtained data by application of mathematical-statistical methods and theory of neural networks, the relationship between the concentration of sodium chloride in the electrolyte and final thickness of the AAO layer was experimentally determined. Thanks to that it was possible to obtain the predictive model which can determine the final thickness of AAO layer. Moreover, the results of this research allows us to reduce the concentration of other components of the electrolyte up to the level of 25 % of commonly used concentration of these electrolyte components designed for the process of aluminium anodic oxidation.

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1. Introduction

Anodic oxidation of aluminium was used for finishing of parts made of aluminium and aluminium alloys since 1923, when it was used as corrosion protection for the first time. Its essence lies in creation of thin layer of aluminium oxide on the surface of a part, which blocks the access of oxygen from the atmosphere and thus to eliminate its corrosive effect [1]. A lot of research was conducted in the area of anodic oxidation of aluminium ever since its first industrial use. Their goal was to clarify the main principle behind the creation of oxide layer on the surface of aluminium part, specify the influences of individual input factors on thickness of oxide layer [2], [3], [4], geometry of oxide layer [4], [5], [6], its properties [7], [8], [9], [10], [11], also how to optimize the process of oxidation [11] and last, but not least, creation of new electrolytes, which could reduce the cost and risks to health and environment [12], [13]. Electrolytes, in which sulphuric acid, oxalic acid and boric acid are the main ingredients, were created thanks to this research. In case of creation of new electrolytes, which would contain lower concentrations of these acids, the addition of sodium chloride seems to be an appropriate solution. So this article is aimed to find optimal operating conditions [14].

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2. Experimental

In order to determine the influence of chemical composition of electrolyte on the resulting oxide layer thickness created during the aluminium anodizing process, an experiment was carried out and statistical analysis of experimentally obtained data was performed. Specifically, the influence of four chemical factors (input factors x_1 , x_2 , x_3 and x_4): the concentration of sulphuric acid, concentration of oxalic acid, concentration of boric acid and concentration of sodium chloride was observed. A circuit diagram for the process of anodizing in a Hull cell is shown in Fig. 1. and the location of the sample during experimental anodizing process is also illustrated in this figure. In the Fig. 1 is shown the sample as the product of experimental anodizing process with deposited AAO layer. The experiment was carried out according to the central composite design based on DOE methodology [15], [16]. Five levels for each input factor were considered during the experimental procedure, as is illustrated in the Table 1. We can see different operating conditions of the experiment, which include the composition of electrolyte (x_1 , x_2 , x_3 and x_4), the electrolyte temperature (x_5), the size of an applied voltage (x_6) and the anodizing time (x_7). The standard operating condition, the center-point, for input factors considered during experimental procedure are defined in Table 2.

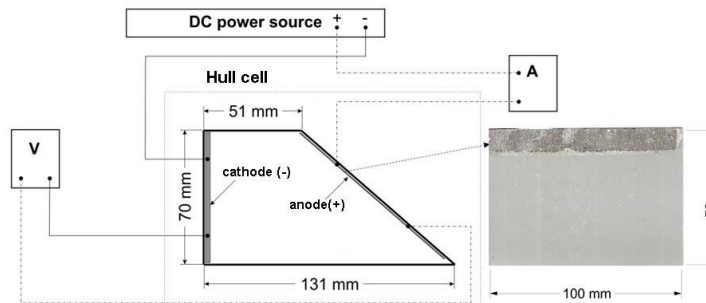


Fig. 1. A circuit diagram for the process of anodizing and location of sample

Table 1. Levels of observed factors.

Coded scale	Natural scale	Factor level				
		-2.83	-1	0	1	2.83
x_1	$\text{H}_2\text{SO}_4 [\text{g}\cdot\text{l}^{-1}]$	12.57	40.00	55.00	70.00	97.43
x_2	$\text{C}_2\text{H}_2\text{O}_4 [\text{g}\cdot\text{l}^{-1}]$	3.76	6.50	8.00	9.50	12.24
x_3	$\text{H}_3\text{BO}_3 [\text{g}\cdot\text{l}^{-1}]$	4.51	10.00	13.00	16.00	21.49
x_4	$\text{NaCl} [\text{g}\cdot\text{l}^{-1}]$	0.12	0.30	0.40	0.50	0.68
x_5	$T [^\circ\text{C}]$	-5.46	11.00	20.00	29.00	45.46
x_6	$U [\text{V}]$	2.34	6.00	8.00	10.00	13.66
x_7	$t [\text{min}]$	1.72	40.00	30.00	40.00	58.28

Table 2. Standard levels of observed factors.

Factor	H_2SO_4	$\text{C}_2\text{H}_2\text{O}_4$	H_3BO_3	NaCl	T	U	t
Level	200 $[\text{g}\cdot\text{l}^{-1}]$	20 $[\text{g}\cdot\text{l}^{-1}]$	50 $[\text{g}\cdot\text{l}^{-1}]$	0 $[\text{g}\cdot\text{l}^{-1}]$	22 $[\text{C}^\circ]$	10 $[\text{V}]$	30 $[\text{min}]$

The samples used in the experiment were cut out from a sheet of alloy EN AW 1050 H24 with dimensions 100x70x1 mm. Before anodic oxidation in a Hull cell, each applied specimen was degreased in a 38 % solution of sodium hydroxide at temperature of 55 to 60 °C for 2 minutes and stained in a 40 % solution of sodium hydroxide at the temperature of 45 to 50 °C for 30 seconds. Afterwards, the surface of each sample was immersed in a 4 % activation solution of nitric acid bath.

3. Evaluation of experiment

At the end of anodizing process, a map of measuring points was created on each sample, where the response - the thickness of aluminium oxide layer was measured. Measuring points were placed on a sample 1 to 9 cm from left edge with distance of 1 cm and 1 to 4 cm from bottom edge with distance of 1 cm. The relationship between chemical composition of the electrolyte and the thickness of the formed AAO layer was determined by implementation of statistical analysis methods [17], which used significant input factors and their interactions at the chosen significance level of $\alpha=10\%$. Table 3 shows significant factors and

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