



Comparative study of mechanical and tribological properties of alumina coatings formed on aluminium in various conditions

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ABSTRACT

Influence of oxalic acid addition to sulphuric acid bath on the mechanical properties of the oxide layers formed on aluminium has been examined. For this purpose two Doehlert experimental designs with three variables (temperature, current density, sulphuric acid concentration) and four variables (oxalic acid concentration, temperature, current density, sulphuric acid concentration) were realized. Four responses were studied namely: growth rate (V_c), Vickers microhardness (D), weight loss after abrasion (W_a) and deflection at failure (D_f) of the anodic oxide layer. A comparative study based on surface responses was achieved. Compared with sulphuric acid bath, it was found that the addition of oxalic acid permits high growth rates, high abrasion resistance and high microhardness but less ductile layers. The observed mechanical properties of the oxide layers can be related to their morphology revealed by SEM observations and their chemical composition determined by GDOES.

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1. Introduction

As weight-saving materials, aluminium and aluminium alloys are becoming increasingly important for both technical and economical considerations [1,2]. However, poor friction property and wear resistance restricted their applications in the industrial fields requiring sliding contact. In order to improve mechanical properties of aluminium, anodizing has been commonly used [1,2].

Anodizing, a surface treatment which originated in the 1930s, is an electrochemical process, consists on converting aluminium into its oxide by appropriate selection of the electrolyte and the anodizing conditions, such as current density, voltage and temperature [3,4]. By adjusting the conditions used in the anodizing process, oxide layers can be produced with almost any desired properties, from thin films used in decorative applications to the extremely hard, corrosion resistant oxides used in engineering applications [5–9]. In the last few decades, the effect of anodizing conditions and the composition of single acid electrolytes, i.e. solution of sulphuric acid, chromic acid, phosphoric acid or oxalic acid, on the properties of the anodic layer such as corrosion resistance, microhardness and abrasion resistance were investigated [5–8]. To improve the properties of the anodic layer and/or to find an alternative of chromic acid anodizing process, mixed acid electro-

lytes such as oxalic acid–chromic acid, nitric acid–sulphuric acid and boric acid–sulphuric acid were implemented [9–11].

In previous works [12,13], we have optimized some of mechanical properties of the anodic oxide layer elaborated on aluminium in sulphuric acid and oxalic acid–sulphuric acid baths using two Doehlert experimental designs. The electrolyte composition, temperature and current density were retained as variables to conduct each study.

The objective of this paper is to investigate the effect of oxalic acid addition to a sulphuric acid bath, on the thickness and the mechanical properties of the elaborated oxide layers, namely: Vickers microhardness (D), weight loss after abrasion (W_a) and deflection at failure (D_f). Comparisons were made using the previously established models obtained from Doehlert experimental design applied to each of the anodizing process [12,13]. Moreover, the morphology and the composition of the anodic oxide layer were examined by scanning electron microscopy (SEM), optical microscopy and glow-discharge optical emission spectroscopy (GDOES).

2. Experimental

2.1. Materials and procedures

Parallelipipedic AA1050 samples $100 \times 25 \times 3 \text{ mm}^3$ were used as the substrate for anodic conversion treatment. Prior to anodizing, samples were mechanically polished to P1000 grade paper

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followed by (i) chemical polishing in a 15:85 (v/v) mixture of concentrated HNO_3 and H_3PO_4 at 85°C for 2 min, (ii) etching in 1 M NaOH solution at room temperature for 1 min and (iii) chemical pickling in 30% (v/v) HNO_3 solution at room temperature for 30 s. Water rinsing was used after each step. Afterwards, samples were anodized in vigorously stirred acid solutions (sulphuric acid and oxalic–sulphuric acid bath) maintained within $\pm 0.1^\circ\text{C}$ of the set temperature for 90 min then washed in deionised water and dried. The used cathodes were also aluminium sheets. Sulphuric, nitric and phosphoric acids are analytical grade chemicals.

2.2. Testing methods

In order to characterize the anodic oxide layer, four tests were conducted. The oxide layer thickness was measured using ELCOMETER 355 Top Thickness Gauge. The Vickers microhardness was carried out using DELTALAB HVS-1000 tester (200 g load for 15 s).

Abrasion tests were conducted using a pin-on-disc machine. Anodized samples with dimensions of $20 \times 20 \times 3 \text{ mm}^3$ were brought into contact with 320 grit SiC paper, fixed on a rotating disc with a constant speed of 20 rpm. The applied normal load was 5 N and the test duration was 1 min.

Deflection at failure of the anodic oxide films on aluminium was measured by performing three point flexure tests on parallelepipedic samples $100 \times 25 \times 3 \text{ mm}^3$ at room temperature. A universal machine [Lloyd instruments LR 50KN] was used for this purpose. Loading speed was fixed at 2 mm min^{-1} and the calibrated distance was 50 mm.

The morphology of the oxide layer was studied from the top side of the layer using a Scanning Electron Microscope SEM (Jeol JSM-6400F and Philips XL30).

The morphology of worn oxide layer surfaces was studied using a LEICA optical microscope.

The distribution of species in the anodic oxide layer was determined by depth profiling using a Jobin Yvon GD Profiler instrument equipped with a 4 mm diameter anode and operating at pressure of 800 Pa and a power of 600 W in an argon atmosphere. The relevant wave-lengths (nm) were as follows: Al, 396.15; O, 130.22, S, 181.73 and C, 156.14. The sputtering layer was 6 μm thick.

Table 1
Doehlert experimental design in coded variables ($k = 4$ and $N_0 = 4$).

No exp.	X_1	X_2	X_3	X_4
1	0.0	0.000	0.000	0.000
2	1.0	0.000	0.000	0.000
3	-1.0	0.000	0.000	0.000
4	0.5	0.886	0.000	0.000
5	-0.5	-0.886	0.000	0.000
6	0.5	-0.866	0.000	0.000
7	-0.5	0.866	0.000	0.000
8	0.5	0.289	0.816	0.000
9	-0.5	-0.289	-0.816	0.000
10	0.5	-0.289	-0.816	0.000
11	0.0	0.577	-0.816	0.000
12	-0.5	0.289	0.816	0.000
13	0.0	-0.577	0.816	0.000
14	0.5	0.289	0.204	0.791
15	-0.5	-0.289	-0.204	-0.791
16	0.5	-0.289	-0.204	-0.791
17	0.0	0.577	-0.204	-0.791
18	0.0	0.000	0.612	-0.791
19	-0.5	0.289	0.204	0.791
20	0.0	-0.577	0.204	0.791
21	0.0	0.000	-0.612	0.791
22	0.0	0.000	0.000	0.000
23	0.0	0.000	0.000	0.000
24	0.0	0.000	0.000	0.000
25	0.0	0.000	0.000	0.000

2.3. Methodology and design of experiments

The Doehlert experimental design [14] was performed to study the effect of the anodizing conditions on the performance of the retained baths and on the aluminium oxide layer properties. As currently used in experimental design, natural variables U_j were transformed into coded variables X_j according to the following relation [15–17]:

$$X_j = \frac{U_j - U_j(0)}{\Delta U_j}$$

where $U_j(0)$ is the value of U_j at the centre of the study domain and ΔU_j is the variation step. Doehlert design requires $N = k^2 + k + N_0$ experiments, where k is the number of the factors and N_0 the number of centre runs. Experiments at the centre are required to conduct statistical tests. For example, Doehlert experimental design in coded variables corresponding to 4 variables is given in Table 1. The delimited zones corresponding to the three and four variables experimental designs. Experiment 1 is the central run for each of the experimental design. Replicates at the central level of the variables (experiments 22–25) were conducted in order to estimate the pure error variance.

A full quadratic model, including interaction terms, was assumed to describe the relationship between each response Y_i and experimental variables X_j :

$$\hat{Y} = b_0 + \sum_{j=1}^k b_j X_j + \sum_{j=1}^k \sum_{h=j+1}^k b_{jh} X_j X_h + \sum_{j=1}^k b_{jj} X_j^2$$

where b_0 is the constant of the model, b_j the first degree coefficients, b_{jh} the cross-products coefficients and b_{jj} the quadratic coefficients.

It is to mention that NEMROD W software [18] was used for data calculation and treatment.

3. Results and discussion

3.1. Variables, study domains and model expressions

In order to compare the performances of sulphuric acid and oxalic acid–sulphuric acid anodizing processes, a Doehlert experimental design was applied for each of them. Four responses were retained:

- growth rate of the anodic oxide layer, V_e , noted \hat{Y}_1 ($\mu\text{m min}^{-1}$),
- Vickers microhardness of the anodic oxide layer, D , noted \hat{Y}_2 (HV),
- weight loss by abrasion of the anodic oxide layer, W_a , noted \hat{Y}_3 (mg),
- deflection at failure of the anodic oxide layer F_r , noted \hat{Y}_4 (mm).

Table 2
Study domains.

Variables	Number of levels	Centre $U_j(0)$	Variation step ΔU_j
<i>Sulphuric acid anodizing process</i>			
U_1 ($^\circ\text{C}$)	5	14	11
U_2 (A dm^{-2})	7	2	1
U_3 (g L^{-1})	3	160	40
<i>Oxalic sulphuric acid anodizing process</i>			
U_1 (g L^{-1})	5	10	8
U_2 ($^\circ\text{C}$)	7	16.5	13.5
U_3 (A dm^{-2})	7	2	1
U_4 (g L^{-1})	3	160	40

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