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Mechanical failure of anodized film of aluminium in bending

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ABSTRACT

The mechanical properties of electrolytic oxide layers elaborated on aluminium substrate in oxalic–sulphuric acid bath were optimized using a four variables doehlert experimental design (bath temperature, anodic current density, sulphuric acid and oxalic acid concentrations). Thickness measurements and flexural tests were conducted. A sudden decrease of the load, indicative of film failure, was observed upon the load–deflection curve. The deflection at failure and the maximum load for each layer were deduced from the corresponding load–deflection curve. The isoresponse curves study and the optimum path study of the three retained responses: thickness, maximum load and deflection at failure, showed that the experimental conditions, where the three optima were found, were opposite. In order to maximize in the same time the three responses, multicriteria optimization using the desirability function was achieved. In so doing, the determined optimal anodizing conditions were: $C_{ox} = 13.6\ \mathrm{g\,L^{-1}}$, $T = 21.4\ ^\circ\mathrm{C}$, $j = 2.48\mathrm{A\,dm^{-2}}$, $C_{sul} = 186.3\ \mathrm{g\,L^{-1}}$, while the corresponding estimated response values were 57.6 μm, 775 N and 4.16 mm for thickness, maximum load and deflection at failure, respectively.

Finally, a morphological study of the aluminium oxide layer after flexure test was conducted using optical microscopy examination.

1. Introduction

Anodizing has greatly extended the application of aluminium and its alloys in products and used where these materials might otherwise not be utilized. The process provides an oxide layer, thick if required, improving the corrosion and abrasion resistances, and overall appearance of aluminium. Thus, anodized aluminium and aluminium alloys have been used for a wide range of applications, including transport, sport sectors and biomedical industry [1]. The use of these materials in transport industry has increased enormously and extended to cars, trucks, planes, ships, etc. [2]. The omnipresence of anodized aluminium in this sector is due to its exceptional surface properties and lighten these structures in order to reduce fuel emissions and to improve fuel economy at affordable prices [1]. Nevertheless, surface damage arises owing to certain mechanical phenomenon, such as friction and deflection [2–6]. Consequently, in many of these applications, the mechanical properties of the anodized layer play a key role [7]. Anodizing [8,9], which 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, temperature etc. The obtained anodic oxide on aluminium is hard and usually accompanied by the risk of brittle failure, especially under surface-concentrated loads from static or cyclic contacts. Consequently, it is imperative to establish a link between anodizing conditions and deformation and fracture properties of the oxide films in order to improve their performances. Tensile testing on the anodized aluminium can be difficult due to possible failure of the oxide layer at the two fixtures. Accordingly, three or four flexure point tests can be used to study the failure of the anodic film on aluminium [3,4].

To the author's knowledge, the literature dealing with the study of the dependence of the mechanical behaviour of the anodized aluminium on the elaboration conditions is not voluminous [10–12]. In previous works [11,12], we have studied the dependence of some properties on the anodizing conditions using the methodology of experimental design.

The main focus of this paper is to improve of the flexural response of anodic layers formed on aluminium in oxalic/sulphuric acid bath. In this study, we are faced with four process variables: bath temperature, anodic current density oxalic and sulphuric acid concentrations. The traditional approach used for optimizing a multivariable system, investigates the influence of each anodizing parameter one at time while keeping the others constant. This method is not only time consuming but also often misses the interactions between the variables. The drawbacks of single variable optimization process can be eliminated by optimizing all the affecting parameters collectively by using Doehlert experimental design [13–18].

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Doehlert experimental design [13] was used to establish the effect of the input variables: bath temperature (*T*), anodic current density (*J*), oxalic (C_{ox}) and sulphuric acid (C_{sul}) concentrations as well as their interactions on three responses namely: thickness (µm), maximal load (F_m) and deflection at failure (D_f) of the aluminium oxide layer. Morphological study of the aluminium oxide layer after flexure tests was conducted using optical microscopy.

2. Experimental

2.1. Materials and methods

Aluminium beams 100 × 25 × 3 mm were used as substrate for anodic conversion treatment. The composition of aluminium is given in Table 1.

Details of the anodization steps were described elsewhere [11,12]. Fig. 1 shows the geometrical parameters of the obtained sandwich beams.

2.2. Testing methods

In order to characterize the anodic oxide layers the following tests were carried out.

2.2.1. Thickness measurement

The thickness of the anodic oxide layer was measured using ELCOMETER 355 Top Thickness Gauge equipped with eddy current probe. The average thickness of 20 measuring points, evenly distributed on both sides, was taken.

2.2.2. Flexure test

Measurements of deflection at failure of the anodic oxide films on aluminium were performed with three points flexure test on the prepared beams (Fig. 2) at room temperature. A universal machine [Lloyd instruments LR 50 kN] was used for this purpose. Loading speed was fixed at 2 mm min^{-1}, whereas calibrated distance was 50 mm. Load–deflection response was then recorded using NEXYGEN software program. The deflection at failure (D_f) and the maximum load (F_m) were then deduced. Fig. 3 shows a typical load–deflection curve of an anodized aluminium specimen. As it can be seen the tested beam showed an initial linear elastic behaviour then a plastic one followed by a sudden decrease of the maximum load magnitude indicative to the aluminium oxide failure.

2.2.3. Surface morphology

The morphology of the oxide layer was studied from the back side of the specimen using a LEICA optical microscope.

2.3. Methodology of experimental design

Doehlert experimental design [13] was used, in order to analyze the influence of the anodizing conditions on the mechanical properties of the anodic layer. Doehlert design offers a number of advantages such as: (i) the number of levels is not the same for all variables, which allows flexibility to assign a large or a small number of levels to the selected variables, (ii) it requires fewer experiments, (iii) it is more efficient than central composite design or Box–Behnken design (the efficiency of any experimental design is defined as the number of coefficients of the model divided by the number of experiments) and (iv) its potential for sequentially where the experiments can be re-used when the boundaries have not been well chosen at first.

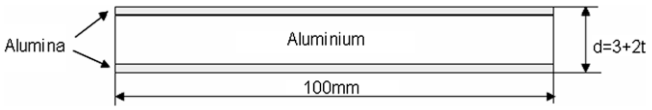

Fig. 1. Geometrical Parameters of a sandwich beam; *t*: thickness of one alumina face sheet, *d*: the total thickness.

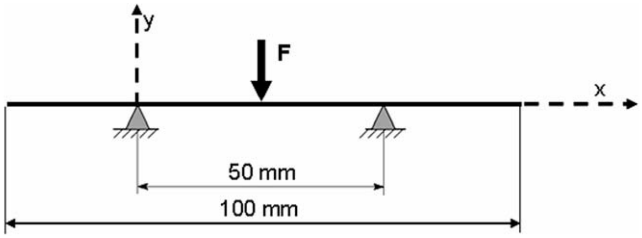

Fig. 2. Three point bending configuration.

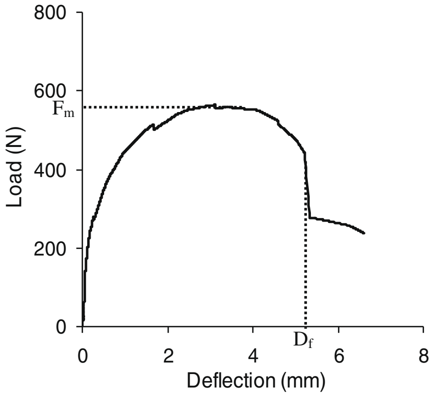

Fig. 3. Typical load–deflection curve of an anodized aluminium specimen elaborated under the following conditions: C_{ox} = 6 g L^{-1}, *T* = 20.4 °C, *J* = 2.82 A dm^{-2} and C_{sul} = 160 g L^{-1}. F_m = 564 N, D_f = 5.2 mm.

The variables U_j under investigation were:

- U_1: the oxalic acid concentration (g L^{-1}),
- U_2: the anodizing temperature (°C),
- U_3: the current density (A dm^{-2}),
- U_4: the sulphuric acid concentration (g L^{-1}).

As currently used in experimental design, natural variables U_j were transformed into coded variables X_j [14–18]. For the Doehlert experimental design construction, centres and variation steps of the retained domain for each variable have been defined as shown in Table 2.

Three responses were studied: Y_1: oxide layer thickness (µm), Y_2: maximum load (*N*) and Y_3: deflection at failure (mm).

In Doehlert matrix, the number of experiments, *N*, for a given number of factors, *k*, is:

Table 1
Chemical composition of the used aluminium (weight %).

Element	Si	Mn	Cu	Ti	Zn	Fe	Pb	Mg	Al
Weight %	0.11	<0.005	<0.005	0.014	0.009	0.37	0.006	<0.005	Balance