

Friction behaviour of anodized aluminium alloy biology essay

[Science](#), [Biology](#)



used for automotive applications M. Guezmila, W. Bensalaha*, A. Khalladia, K. Elleucha, M. Depetris-Weryb and H. F. Ayediaa Laboratoire de Génie des Matériaux et Environnement (LGME), ENIS, B. P. 1173-3038, Université de Sfax, Tunisie. blUT Mesures Physiques d'Orsay- Université Paris Sud, Plateau du Moulon, 91400 Orsay, France. (* Author for correspondence: Tel.: +216 74 274 088; Fax: +216 74 275 595; E-mail address: walidbensalah@gmail.com)

Abstract The paper discusses mechanical and tribological property aspects of thick oxide films formed on two aluminum substrates namely, Al5754 and Al1050A (used as reference) by electrolytic oxidation in acidic solutions. For this purpose, two acidic baths were considered: (i) 160g L⁻¹ sulphuric acid and (ii) 10g L⁻¹ oxalic acid/160g L⁻¹ sulphuric acid mixture. Mechanical/tribological characteristics, morphology and composition of the formed coatings were studied by microhardness analyses, dry sliding tests, Scanning Electron microscopy (SEM), Energy Dispersive X-ray spectroscopy (EDS) and Glow-discharge Optical Emission Spectroscopy (GDOES). It was found that (i) the elaboration conditions have preponderant effects on the mechanical/tribological layer properties and (ii) aluminum and magnesium are oxidized at the substrate/oxide interface, entering the film as Al³⁺ and Mg²⁺ ions. Key-words: Aluminium anodizing; Vickers Micro hardness;

Friction behaviour. 1. Introduction Wrought aluminum-magnesium alloys (5XXX) have been widely used in automotive, shipbuilding and construction industries due to their light weight, physical properties, corrosion resistance and low cost [1-4]. However, their usage is limited because they are subject to damage due to low hardness and high friction. Their surface properties can be enhanced by applying appropriate treatments such as anodizing [5-

7]. Anodizing is a surface treatment process that can generate on aluminum and aluminum alloys, a thick, hard and protective oxide layer [8-10]. It has been reported that the tribological properties of anodic oxide layer depends on its porosity [11-13]. However, this porosity depends strongly on the anodizing operating conditions (cell voltage, electrolyte composition, etc) [14-17]. To the author's knowledge, the literature dealing with the study of the dependence of the friction behavior of the anodized aluminum with the elaboration conditions is not voluminous [11]. In previous works [17-19] dependence of anodized aluminum wear response with anodizing conditions was investigated using the methodology of experimental design. The main focus of this paper is to study the effect of anodizing temperature and current density on the Vickers microhardness and tribological behaviour of anodic oxide layers formed on Al5754 and Al1050A in sulphuric and oxalic/sulphuric acid baths. Moreover, worn surfaces were analyzed using Scanning Electron Microscopy (SEM) coupled with Energy-dispersive spectroscopy (EDS). Glow-Discharge Optical Emission Spectroscopy (GDOES) was used to find out the chemical composition of anodic layers. 2.

Experimental 2. 1. Materials and methods Two aluminium alloys namely: 1050A and 5754H111 were used in this study. Chemical compositions of these materials were given in Table 1. Their Vickers microhardnesses were 28HV and 67HV, respectively. Samples (100×25×3 mm³) of both alloys were mechanically polished to P1000 grade paper to obtain smooth surfaces followed by i) chemical polishing in a 15/85 (v/v) mixture of concentrated HNO₃ and H₃PO₄ at 85°C for 2 min, ii) etching in 1M NaOH solution at room temperature for 1 min and iii) chemical pickling in 30% (v/v) HNO₃ solution

at room temperature for 30s. Water rinsing was used after each step. Afterwards, samples were anodized in vigorously stirred acid solution maintained within $\pm 0.1^\circ\text{C}$ of the set temperature. Two acidic electrolytes were considered for anodizing: (i) 160g L⁻¹ sulphuric acid bath and (ii) 10g L⁻¹ oxalic acid/160g L⁻¹ sulphuric acid mixture. Whatever the anodizing conditions, the anodizing duration was chosen so that to obtain oxide layer thicknesses of 30 μm measured using ELCOMETER 355 Top Thickness Gauge equipped with eddy current probe. It is to mention that, the used cathodes were also aluminum sheets. Sulphuric and oxalic acids are analytical grade chemicals.

2. 2. Testing methods

2. 2. 1. Microhardness measurement Vickers microhardness of the anodic film was determined using a Vickers microhardness tester DELTALAB HVS-1000 (200 g load for 15s as dwell time). The results represented the average of 10 measurements for each sample.

2. 2. 2. Friction test Friction tests were carried out in dry conditions using a pin-on-disc tribometer. Anodized samples with dimensions of 20x20x3mm³ were brought into contact with 100C6 steel ball with a diameter of 6mm. All the tests were performed at the same sliding speed of 100tr/min (0.052m/s). The applied normal load was 1N. Friction tests were performed in ambient air (25-27°C) at relative humidity (RH) of 35-45%. During tests, the variation of the friction coefficient versus time was recorded.

2. 2. 3. Surface morphology The wear tracks were studied using a LEICA optical microscope and a TESCAN VEGA II Scanning Electron Microscope (SEM) coupled with an energy-dispersive X-ray spectroscopy (EDS) for chemical analysis.

2. 2. 4. Glow-discharge optical emission spectroscopy (GDOES) The distribution of chemical 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.

3. Results and discussion

3. 1. Vickers microhardness of the anodic oxide layer: effect of temperature and current density

Figure 1 illustrates the influence of the anodizing temperature on the Vickers microhardness of the anodic oxide layer elaborated under galvanostatic conditions anodic (current density equal to $2\text{A}/\text{dm}^2$). As can be seen, the microhardness decreases with increasing anodizing temperature whatever the electrolyte composition and the substrate are. This fact can be related to the accentuation of the chemical dissolution at the interface oxide/electrolyte, from the top of pores and from the pore-walls. The obtained structure is more porous and with low hardness. Besides, the Vickers microhardness values of anodic oxide layers elaborated in oxalic/sulphuric acid mixture are higher than those elaborated in sulphuric acid bath. These results are predictable and can be attributed to the decrease of aggressiveness of the acid mixture by the addition of oxalic acid [17, 19]. The addition of weak acid (oxalic acid) to sulphuric acid (strong acid) favors the oxide layer growth, minimizes the dissolution reactions at the interface oxide/electrolyte as well as from the pore walls and conducts to compacter layers. Furthermore, the alloying element seems to influence the microhardness of the layer. The values obtained for anodic oxide layers elaborated on Al5754 are smaller than those obtained with Al 1050A. The observed difference can be explained by the presence of magnesium oxide in the layer. As far as we are concerned, further explanation will be given

later. Figure 2 shows the influence of the current density on the Vickers microhardness of the anodic oxide layer elaborated at a temperature of 15°C. The microhardness increases with increasing current density whatever the bath and the substrate are. The increase of current density induces high dissolution of the oxide in the bottom of the pores and favors, thus, the layer growth and its compactness [17, 19]. In the studied domain (1-3A/dm²), the Vickers microhardness values of anodic oxide layers elaborated in oxalic/sulphuric acid mixture are higher than those elaborated in sulphuric bath.

3. 2. Friction coefficient

Before studying different anodic oxide layers, friction behaviour of the aluminium substrates was investigated. The variation of the friction coefficient of the studied substrates versus time is shown in Figure 3. As can be seen, this plot reveals three zones connected to three regimes: friction first increases, then increases and finally achieves a steady state value for the remainder of the sliding distance. The changes of the friction coefficient values can be associated with changes in the wear morphology and the extent of oxidation. These results are in accordance with those of Kim et al. [20]. In fact, they demonstrated the role of oxygen on the wear morphology evolution. Besides, Yerokhin et al.[21] relate this finding to the transition from the wear mechanism of the couple steel/aluminum (ductile) to that steel/oxide film (brittle) formed by oxidation which decreases the friction coefficient. The little difference in the friction coefficient between the two substrates seems to be attributed to the difference in their hardness and/or chemical composition. Figure 4 depicts the friction coefficient evolution vs time for anodic oxide layers formed in sulphuric acid bath at different temperatures. The friction coefficient

increases with temperature for both anodized substrates and was higher for Al1050A whatever the temperature is. At this stage, it is important to mention that a better tribological behaviour is reached with layers formed at low temperatures. These results are predictable and can be attributed to the decrease of dissolution reactions at the interface oxide/electrolyte as well as from the pore walls which favors the oxide layer growth and its compactness. Figure 5 illustrates the friction coefficient of anodic oxide layers elaborated in oxalic/sulphuric acid bath as a function of time for 5 and 25°C. The same conclusions as previously can be emitted in term of dependence of the friction coefficient with anodizing temperature. Considering all the results, the anodic oxide layers formed in oxalic/sulphuric acid bath present the best tribological behaviour whatever the substrate is. The influence of the anodic current density on the friction coefficient evolution for the oxide layers elaborated in sulphuric and mixture acid bathes is shown in Figures 6 and 7 respectively. The friction coefficient decreases as the anodic current density increases. This was reasonable since the specimen's microhardness is higher for high current densities (Figure 2) [9]. Moreover, the current density affects also the shape of the curve; for low current density (1 A dm^{-2}), the friction coefficient reaches rapidly the quasi-stationary levels (0.5-0.7) whilst, for 3 A. dm^{-2} , the same values are obtained when the time is extended to 500s (Figure 6). As previously concluded, the friction coefficient values measured on anodic oxide layers formed on Al1050A are higher than those obtained with layers formed on Al 5754 whatever the current density is. The analysis of figures 6 and 7 shows that anodic oxide layers formed at high current density in oxalic/sulphuric

acid bath present the best tribological behaviour for both anodized substrates. With regards to the obtained results, compared to the anodic oxide layers formed on Al1050A, those elaborated on Al5754 presents less properties in term of microhardness and friction coefficient. This statement seems to be related to the alloying elements. In fact, anodic oxide layer formed on Al5754 contains, with Al₂O₃, the magnesium oxide (MgO) [22]. Zhou et al. [22] demonstrated that Mg²⁺ ions are ejected more rapidly to the oxide/electrolyte interface which permits a uniform distribution through the oxide layer. On the other hand, it was established that the microhardness of MgO is lower than of Al₂O₃ [3]. In order to inspect the presence of Mg through the anodic oxide layers formed on Al5754, chemical analysis was conducted using GDOES (Figure 8a-b). Figure 8a-b shows the presence of magnesium, aluminum, oxygen and sulphur species. The presence of Mg, Al and O can be attributed to the MgO and Al₂O₃ formation [3]. The Mg element seems to be uniform throughout the layer. The existence of sulphur can be explained by the inward migration of sulphate anions through the pores of the coating [23]. In order to study the effect of the anodic oxide layer porosity on the friction coefficient, an enlargement treatment was conducted [13]. This treatment consists in the immersion of anodized samples in 5% (weight) of phosphoric acid at 30°C at different durations. Figure 9 shows the variation of the friction coefficient when the pores are enlarged for 20 and 40min of immersion compared to untreated sample. The quasi-stationary value of the friction coefficient obtained after 20min of immersion is smaller than that of untreated specimen. This finding can be related to the changes of the shape of the pores from irregular round

to regular round, and surface with regular round pores owns better tribological properties [13]. For 40 min of pore-enlargement, the friction coefficient is higher. These results can be attributed to that extended pore-enlargement treatment makes the cell wall thinned, so the capacity of walls to support normal pressure becomes weak. Further enlarging the pores will destroy the anodic porous structure completely.

3. 3. SEM and EDS analysis

Following the sliding test, the possibility of tribochemical reactions at the friction surface is microscopically analysed as shown in figure 10. As can be seen, the morphology of the wear tracks formed on Al7554 is dependent on the anodizing conditions. Figures 10a-c demonstrated that smooth layers were generated by the combined influence of tribochemical reaction including material transfer at the contact surface. The tribo-layer was formed by severe plastic deformation of compacted debris. The positive contribution of this layer is to decrease friction at the contact interface. Figures 10b-d reveal scratches along the sliding direction. The absence of tribo-layers is more probably due to the formation and the destruction of the transfer layer. In fact, the generated wear debris does not adhere to the contact area. Equally, the optical microscopy of the worn surface of the steel ball (Figure 10e) shows degradations. This fact confirms the formation of a transfer layer. Finally, abrasive wear seems to be the predominant wear mechanism. The EDS spectrum shown in figure 11 reveals that the wear tracks was composed of Fe and Mo, originated from the components of the ball, and Al and O elements, the main chemical elements of the coatings. It explains that active tribochemical reaction and material transfer occurred between the mating materials.

4. Conclusion

In this work, Vickers Microhardness and friction

coefficient of anodic oxide layers elaborated on Al 1050A and Al5754 in two different acid electrolytes (160g/L sulphuric acid and 10g/L oxalic acid+160g/L sulphuric acid mixture) were studied. The results of the present investigation can be summarized as follows: Vickers microhardness increases with current density and decreases with temperature whatever the bath and the anodized substrate are, Vickers microhardness values of anodic oxide layers elaborated on Al 1050A are higher than those obtained with Al5754. Vickers microhardness values of anodic oxide layers elaborated in oxalic/sulphuric acid bath are higher than those elaborated in sulphuric bath, the friction coefficient increases with temperature and decreases with current density. The friction coefficient values measured on anodic oxide layers formed on Al1050A are higher than those obtained with anodic layers formed on Al5754 whatever the temperature and current density are. Anodic oxide layers formed in oxalic/sulphuric acid bath present the best tribological behaviour whatever the condition is, GDOES shows the presence of Mg, Al and O due to the MgO and Al₂O₃ formation, SEM and EDS analysis of wear tracks reveal the existence of tribochemical reactions and material transfer between the mating materials. Acknowledgements This work was supported by the Ministry of Higher Education and Scientific Research-Tunisia.

References