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Features in aluminium alloy grains and their effects on anodizing and corrosion

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ABSTRACT

Distinct chemically and mechanically induced features, evident in the grains of the AA5083-O aluminium alloy, and their effects on anodizing and corrosion have been studied. The grain distinct features are orientation dependent and are ridge-like. These features were found after anodizing in Barker's reagent and after micro-trimming with a diamond knife (during the ultramicrotomy process). The grain features were also found to have a relationship with the anodic films formed on the alloy. Distinct striations (associated with the formation of the ridge-like features found after anodizing) were observed on the surface of the alloy after the removal of the anodic layer (using a solution containing 20 g Cr_2O_3 + 30 ml HPO_3 in 1 l of deionised water at 60 °C). Further etched regions, after the removal of the anodic layer, revealed subsurface micro-layers which appear to have significant influence on the corrosion, anodizing and mechanical (at micro-scale) behaviour of the alloy.

1. Introduction

Anodizing is widely employed as a surface coating technique for aluminium alloys [1]. The effect of the conditions of anodizing (such as applied current density, voltage, type of electrolyte, and temperature) [1–9] and intrinsic material factors (such as the presence, types and distributions of second phase particles, alloying elements, and grain orientation) [6,10–18] on the anodizing behaviour and morphology of the anodic films formed on aluminium alloys have been widely researched. With respect to self-ordering of pores, works on the influence of the extent of induced or inherent stress on the anodizing behaviour of aluminium alloys and works on the growth of porous oxide films on defined aluminium crystals have been reported [19–22]. Other factors, such as the state of the surfaces of aluminium substrates [23,24], grain sizes, type of annealing pre-treatment [25], cold deformation [4] and crystal orientations [26–28], have also been reported to have significant influence on pore ordering and pore sizes. In contrast, Fan and co-workers reported that the domain of pore ordering can extend across the grain boundaries of the substrate [29]. This downplays the effects of grain sizes and/or orientation on the formation of porous anodic layer.

However, it has since been established that anodizing can be used to produce optical anisotropy on the surface of aluminium under polarized illumination [30]. The optical anisotropy was said to be due to variations in the inclination of pores as distinctively formed on the different crystals of the aluminium with different orientations as observed by Franklin and Stirland [31]. This led Kato and Ueda [32] to investigate the influence of the aluminium substrate orientation on the anodic films. Aluminium single crystals of crystallographic orientations (100), (110) and (111) were used for the investigation and they concluded that the substrate orientation affected the anodic film and optical reflectance with the (111) plane giving the lowest reflectance, and also that different variations were observed under different anodizing conditions. Similarly, Saetre and co-workers [33] further strengthened the theory that the optical properties of the anodic film formed on aluminium depend on the crystallographic orientation of the grains. Takahashi and Murakami [34] found that grain orientation affected the shape of oxide film and striations were observed along a certain crystallographic direction. This finding is equally reported by many other researchers, who have also revealed that distinct crystallographic orientations produce different regularities in the arrangement of nanopores that are formed on aluminium substrates (e.g. the reports by Beck and co-workers [27,28] that showed that aluminium crystals with (100) orientation produce the best ordering).

Generally, when certain aluminium alloys are anodized in selected media (e.g. Barker's reagent) grain dependent interference colours are produced when the anodized alloys are viewed under polarized light with an optical microscope. The optical anisotropic effect produced by the oxide has been attributed to the chemically induced surface features of the oxide film [35]. Chemically induced features are also observed when certain alloys are subjected to chemical polishing and electropolishing [36,37]. Although there have been suggestions linking the optical anisotropy effect and the chemically induced features of the anodic oxide layer to the surface profile of the substrate [30,35,37], there is no clear established link to the knowledge of the authors.

Understanding that the chemically-induced features are produced from anodizing, chemical and electrolytic polishing processes, and that these processes are controlled corrosion processes, the observed

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Table 1
Compositional analysis of the major alloying elements in the AA5083-O alloy used in this study.

Element	Mg	Si	Mn	Fe	Zn
wt. %	3.92	0.03	0.44	0.23	0.01

chemically induced features should therefore have a link with the corrosion mechanism of the aluminium alloys. Additionally, it has been observed previously that distinct mechanically induced ridge-like features appear on individual grains when an aluminium alloy is ultramicrotomed. These ridge-like features have been suggested to be associated with the slip bands in the alloy [38]. All of these features (the chemically induced and the mechanically induced) definitely resulted from the intrinsic characteristics of the individual grains of aluminium alloys when they are processed.

Therefore, the aim of this work is to investigate the grain-distinct mechanically and chemically induced ridge-like features and how they affect anodizing and corrosion processes. An established relationship between the intrinsic grain features and the anodizing behaviour and the corrosion mode of the AA5083-O alloy would be of immense benefit in providing useful information for the surface finishing of the alloy and in predicting the corrosion susceptibility (including the service life) of the alloy. The observations in this work would be of particular importance for the transport sector where the AA5083-O alloy finds numerous applications [39]. Additionally, the observations can be extended to the understanding of the corrosion and anodizing behaviours of aluminium alloys in general. Lastly, this work also shows data that explain and shed more light on the mechanism of the formation of the anodic oxide layers (formed on AA5083-O in Barker's reagent) that give colour contrasts when viewed under polarized light.

2. Experimental procedure

The data presented in this work were obtained from a cold rolled AA5083-O alloy (8 mm thickness), with hardness value of 81 HV/0.3, supplied by TWI. The compositional analysis of the major alloying elements in the alloy as determined by inductively coupled plasma-atomic emission spectroscopy is presented in Table 1. The cut samples (1.5 cm × 4 cm) from the alloy were polished to a 1 μm finish, using SiC papers and a 1 μm diamond paste, with an alcohol-based lubricant. After polishing, the samples were degreased with ethanol and rinsed with deionised water before drying in a cool air stream.

Selected mechanically polished and dried samples of AA5083-O alloys were electropolished in 25% perchloric acid solution in ethanol at temperatures below 10 °C. After electropolishing, the samples were rinsed in ethanol followed by a rinse in deionised water. The electropolished samples were then anodized in Barker's reagent (2.5% fluoroboric acid solution) using 20 V at room temperature for 120 s. All anodized samples were immediately rinsed in deionised water before drying in a cool air stream.

The Barker's reagent-formed anodic oxide layer was dissolved in a solution containing 20 g Cr_2O_3 + 30 ml HPO_3 in 1 l of deionised water for examination of the resulting surface features after anodizing. The dissolution was carried out at a solution temperature of 60 °C for 6 min.

Corrosion immersion test was conducted at ambient temperature in naturally aerated 3% NaCl + 10 ml HCl solution for 2 h and also in 3.5% NaCl solution for 7 days.

A multimode 8 (Bruker, Santa Barbara) atomic force microscope, operating in Peakforce Tapping mode using a Pt-Ir coated SCMPIT probe (Bruker, nominal spring constant 2 N/m, nominal resonance frequency of 80 kHz), was used to obtain images of the surface of the AA5083 alloy after anodizing in Barker's reagent.

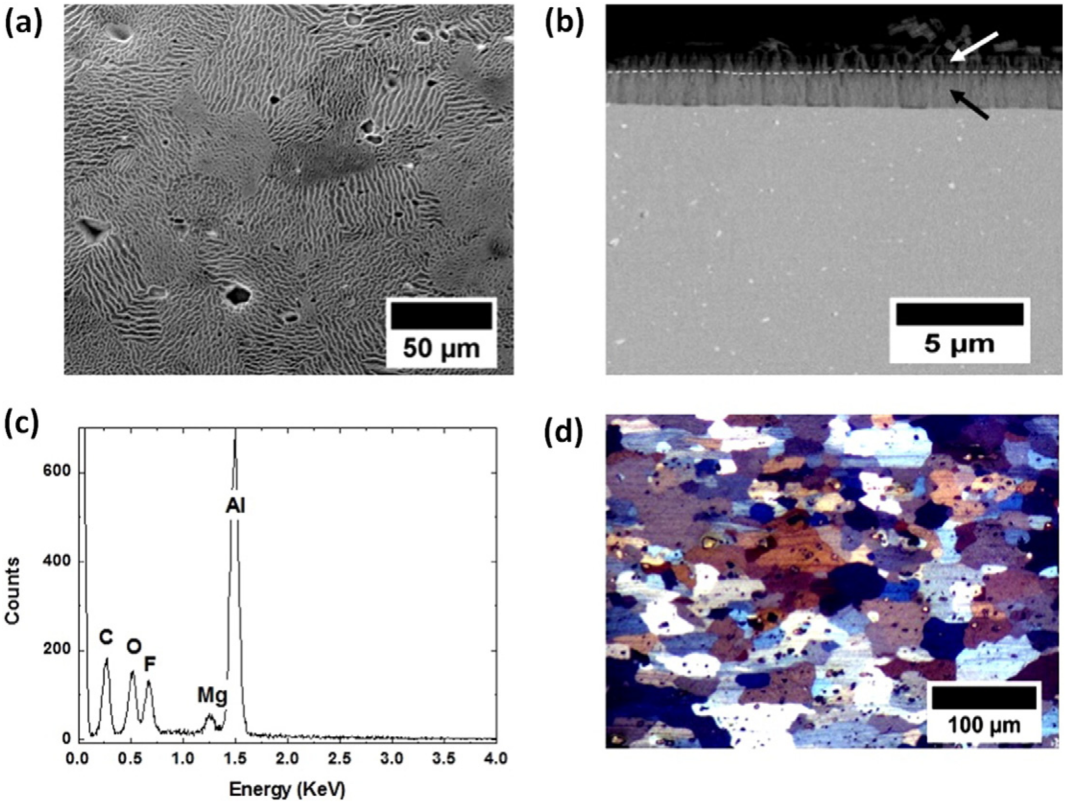

Fig. 1. (a and b) SE micrograph displaying grain dependent chemically-induced, ridge-like features on the (a) surface of the AA5083-O alloy and (b) cross-section of the anodic oxide formed on the alloy after anodizing in Barker's reagent at 20 V for 2 min. (c) EDS spectrum of a spot in the oxide layer.(d) Optical micrograph of the Barker's reagent-anodized surface of the AA5083-O alloy (obtained under polarized light) displaying grain-distinct contrasts.