



## Regular Article

# Effect of prior sputter deposition of pure aluminium on the corrosion behaviour of anodized friction stir weld of dissimilar aluminium alloys



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## ABSTRACT

The effectiveness of the anodic oxide layer formed by a novel processing technique (pre sputter-deposition prior to anodizing) for the corrosion protection of friction stir welds of dissimilar aluminium alloys has been investigated. Two categories of corrosion attacks were observed: a pre-anodizing; and a post-anodizing attack. The novel processing technique prevented the pre-anodizing attack whilst the post-anodizing attack was significantly minimised, and the galvanic current density values between the two alloys were reduced to insignificant values.

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The protection of friction stir welds of similar and dissimilar aluminium alloys has attracted significant interest in recent years. This is due to the fact that friction stir welding is becoming a preferred choice for welding aluminium alloys and the weld may be sensitised. Several methods of corrosion protection and minimisation have been proposed which include post weld heat treatment [1–3], laser surface melting [4, 5], plasma electrolytic oxidation [6], and the use of low melting point metals during the welding process [2]. In our work we have chosen to consider anodizing as a means of protecting friction stir welds of dissimilar aluminium alloys against corrosion. This is because anodizing is an established process for protecting aluminium alloys against corrosion in addition to its other advantages including wear protection, improved adhesion to organic coatings and adhesives etc.

Normal anodizing of the dissimilar weld in 4 M H<sub>2</sub>SO<sub>4</sub> solution produces boundary dissolution as well as non-uniform oxide growth rate across the weld zones [7,8]. Recently, we developed a novel processing technique for the anodizing of friction stir welds of dissimilar aluminium alloys. The novel processing technique involves pre sputter-deposition of 1 µm pure aluminium on the weldment of the dissimilar aluminium alloys prior to anodizing. This newly developed technique prevents the dissolution at the boundary between the two dissimilar aluminium alloys in the thermomechanically affected zone and also minimises the differential oxide growth rates that occur in the zones of the dissimilar aluminium alloy welds when the weldment is anodized [7].

In this article, we have gone a step further to investigate how effective the oxide layer formed by the newly developed method is in the corrosion protection of the friction stir weld of dissimilar AA5083 and AA6082 alloys compared with the oxide layer formed on the substrate by the normal anodizing technique.

The compositions of the AA5083-O and AA6082-T6 aluminium alloys used in this investigation are given in Table 1. The alloys were friction stir welded using a 2-part MX-Triflute™ tool with a pin of diameter to length ratio of 1:0.8 at a rotating speed of 400 rpm and a traverse speed of 300 mm/min.

The welded samples were sequentially polished up to 1 µm surface finish followed by the sputter-deposition of 1 µm thick pure Al on selected welded samples. Anodizing was then carried out on the welded samples with sputter-deposition and without sputter-deposition in 4 M H<sub>2</sub>SO<sub>4</sub> solution using a constant voltage of 15 V for 5 min at room temperature. Galvanic measurements in naturally aerated 3.5% NaCl solution were conducted between the un-anodized couple of AA5083-O and AA6082-T6 alloys, the anodized couple of the alloys without prior sputter-deposition, and the anodized couple of the alloys with prior sputter-deposition. Further corrosion resistance investigation was carried out by immersing the samples in a solution comprising 20 g Cr<sub>2</sub>O<sub>3</sub> + 30 ml HPO<sub>3</sub> in 1 l H<sub>2</sub>O at 60 °C for 12 min. This solution and method is usually used for the removal of anodic oxide films formed on aluminium alloys. However, in this case, this method has been adopted to examine the resistance of the anodic oxide films formed on the dissimilar weld regions (with and without sputter deposition of pure aluminium) to dissolution. After the immersion testing, the tested samples were rinsed in deionised water and then dried in a cool air

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**Table 1**

Compositions of the AA5083-O and AA6082-T6 alloys used in this study (in weight %) as determined using the ICP-AES.

	Al	Mg	Si	Mn	Fe	Zn	Trace elements
AA5083-O	95.22	3.92	0.03	0.44	0.23	0.01	Balance
AA6082-T6	97.45	0.74	0.44	0.40	0.33	0.05	Balance

stream. The microstructures of the tested samples were then investigated using scanning electron microscopy.

The corrosion resistance of the anodized friction stir weld of AA5083 and AA6082 alloys with prior sputter-deposition of 1  $\mu\text{m}$  thick pure Al is compared with the anodized weld without prior sputter-deposition. Displayed in Fig. 1a is a low magnification image of the weldment showing the weld zones in the friction stir weld of AA5083-O (positioned on the advancing side, AS) and AA6082-T6 (positioned on the retreating side, RS) aluminium alloys. The weld zones are the thermomechanically affected zone of the AA5083-O alloy (represented as the MID-TMAZ); the thermomechanically affected zone of the AA6082-T6 alloy (represented as RS-TMAZ); the stir zone (SZ); the heat affected zone (HAZ); and the flow arm (FA). PM represents parent metal. In the thermomechanically affected zone (TMAZ) of the weld, there is a boundary of mismatch between the materials from the AS of the weld and the materials from the RS of the weld [9]. Highly active  $\text{Mg}_2\text{Si}$  particles are distributed in this boundary region [10], resulting in the increased susceptibility of the weld to corrosion along this boundary and also causes boundary dissolution during anodizing in 4 M  $\text{H}_2\text{SO}_4$  solution.

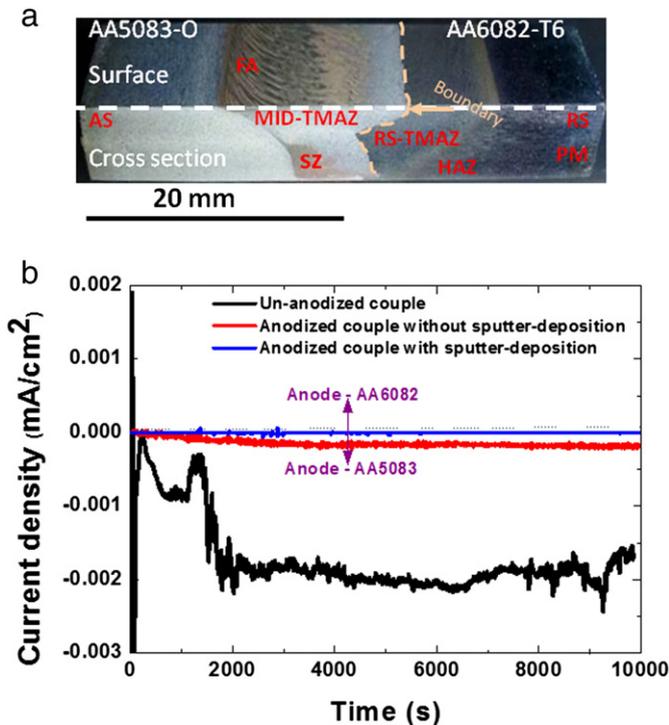
Fig. 1b displays galvanic current measurements between the un-anodized couple, the anodized couple without prior sputter-deposition, and the anodized couple with prior sputter deposition of the AA5083-O and AA6082-T6 aluminium alloys. Negative current density values indicate that the AA5083-O is the anode of the couple and vice versa. Thus, the curves clearly show that irrespective of the surface treatment, the AA5083-O alloy is anodic to the AA6082-T6 alloy. However, anodizing

of the weld reduces the galvanic current density flow between the alloys from approximate  $0.002 \text{ mA/cm}^2$  to near zero values. Importantly, the aluminium alloys with sputter-deposited pure aluminium prior to anodizing show improved corrosion resistance compared with the one anodized without prior sputter-deposition of pure Al. This is evident in the curve of the galvanic current values of the aluminium alloys with prior sputter-deposition being closer to the zero mark compared with the one without prior sputter-deposition.

Further comparative corrosion studies by etching the anodized welded samples in a solution comprising 20 g  $\text{Cr}_2\text{O}_3$  + 30 ml  $\text{HPO}_3$  in 1 l  $\text{H}_2\text{O}$  were carried out on the welded couple anodized without prior sputter-deposition (Fig. 2a and b) and the couple anodized with prior sputter deposition (Fig. 2c and d). The results displayed in Fig. 2, show that after 12 min of etching, the oxide layer on the MID-TMAZ region (the TMAZ of the AA5083-O alloy) had been dissolved to the extent that the matrix is now being attacked, whereas the RS-TMAZ (the TMAZ of the AA6082-T6 alloy) showed little or no attack. This etching attack on the MID-TMAZ is caused by the etchant (a post-anodizing attack). Interestingly, the MID-TMAZ region of the welds without prior sputter-deposition of pure Al before anodizing showed more pronounced attack compared with the MID-TMAZ region with prior sputter-deposition of pure Al before anodizing. Also, preferential boundary dissolution is observed in the anodized weld without prior sputter-deposition. This was not observed in the weld with prior sputter-deposition. In our previous works [7,8,10], we have shown that this boundary dissolution is a result of the preferential dissolution of the  $\text{Mg}_2\text{Si}$  phases distributed along the boundary.

The preferential etching of the anodized AA5083-O zone (the MID-TMAZ) is majorly because the barrier layer formed on the AA5083-O zone is not as protective probably due to the higher Mg content in this region compared with the AA6082-T6 zone. Saenze de Miera and co-workers [11] in their work on the behaviour of second phase particles during anodizing reported that Mg-rich second phase particles are readily oxidised, but the presence of Mg in Mg-rich second phase particles hinders the formation of a stable oxide. It is therefore logical to infer that the reason for the less protective nature of the oxide formed on the AA5083-zone may be because it has a higher Mg content, which also causes the AA5083-O zones to oxidise more speedily [7]. The barrier layer on the AA6082-T6 alloy is much more protective possibly owing to the fact that it has less Mg content compared with the AA5083-O alloy. However, more investigations need to be carried out to prove this point. In particular, further investigations to determine the effect of varying sputter-deposited thicknesses of pure Al on the anodizing and corrosion behaviour of the anodized weldment of the dissimilar aluminium alloys is necessary.

The effect of prior sputter-deposition of pure aluminium on the weld prior to anodizing is further shown in Fig. 3, which displays the surfaces of the anodized HAZ of the AA6082-T6 alloy without prior sputter-deposition (Fig. 3a and b), and the one with prior sputter-deposition (Fig. 3c and d). Certain regions, in the HAZ of the AA6082-T6 alloy without prior sputter-deposition, where the population density of the second phase particles is relatively high (particularly at the grain boundaries) [7], have been evidently attacked (Fig. 3a and b). The HAZ of the weld with prior sputter-deposition before anodizing did not show any trace of etching attack after the 12 min exposure to the etching solution. The features of this attack (in Fig. 3) suggest that they occurred at the onset or before anodizing, immediately after the samples were introduced into the 4 M  $\text{H}_2\text{SO}_4$ , since the etchant only dissolved the anodic oxide but did not attack the underlying alloy (this is a different case from Fig. 2 where the sub-surface attack on the AA5083-O alloy regions is caused by the etchant and not a pre-anodizing attack). The formation of a uniform barrier layer would have prevented the preferential dissolution (pre-anodizing attack). This is also true for the boundary dissolution observed, which is also a pre-anodizing attack. Prior sputter-deposition prevents this form of attack as the deposited pure Al uniformly forms a barrier film that prevents the exposure of the



**Fig. 1.** (a) Low magnification image displaying the zones in the friction stir weld of dissimilar AA5083-O and AA6082-T6 alloys; (b) galvanic current density curves of the un-anodized couple of the AA5083-O and AA6082-T6 alloys and the anodized couple of alloys with and without prior sputter deposition.

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