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Effect of new sealing treatments on corrosion fatigue lifetime of anodized 2024 aluminium alloy

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ABSTRACT

The effect of two sealing processes, i.e. an usual hydrothermal sealing and an innovating sealing process called (B1 + B2), on fatigue behavior of anodized AA2024 was studied in air for as prepared and pre-corroded samples. Pre-corrosion exposure corresponded to salt-spray tests or continuous immersions. For salt-spray tests, the best corrosion resistance was related to the (B1 + B2) sealing and, for continuous immersions, to the hydrothermal sealing. Fatigue life tests in air on pre-corroded samples revealed that anodized samples presented a decrease in fatigue life more pronounced than anodized and sealed samples in relation with a lower corrosion resistance; fatigue crack initiation was localized on pits issued from the degreasing and pickling steps. Independent of the sealing process, the fatigue behavior of the anodized and sealed samples depended on the pre-corrosion exposure. Corrosion fatigue tests induced an additional decrease in fatigue life for both sealing treatments. Crack initiation occurred preferentially on pits issued from degreasing and pickling but also on pits issued from interaction between cyclic loading and corrosive media, in relation with a mechanical damage of the sealed anodic film. The differences in sealed layer morphology could explain the difference in fatigue resistance between the sealed anodic films.

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1. Introduction

Due to its high-strength/weight ratio and good mechanical properties, 2024 aluminium alloy is widely used in the aeronautic industry. Nevertheless, the structural integrity of aging aircraft structures can be affected by corrosion such as pitting corrosion, intergranular corrosion, exfoliation and stress corrosion cracking [1–7]. Anodic oxidation is used extensively to improve the corrosion resistance of aluminium alloys [8]. It is often selected for the excellent adhesion of the protective oxide layer to the substrate. The anodic film is composed of a compact inner layer and a porous outer layer which can be sealed to improve the corrosion resistance [9,10]. Despite the benefits obtained in terms of corrosion protection, the anodic film, which is hard and brittle compared to aluminium substrate, has a detrimental effect on the fatigue life of the base material [3–6] in particular by promoting crack initiation [11–14]. From a microscopic point of view, fatigue cracks of hard anodized samples initiated in the coating in high stress regions while they initiated at the interface between coating and substrate in low stress regions [12]. Rateick and coworkers have shown that anodization of wrought AA6061-T6 and AA6061-T651 gave rise to an appreciable reduction in the fatigue strength [13]. In the worst case, the retained fatigue strength was only 40% of the uncoated material fatigue strength, corresponding to a fatigue life decrease of 60%. However, under stresses less than about 100 MPa, the retained strength began to increase, approaching that of the uncoated material [13].

Camargo and coworkers have related the reduction in the axial fatigue life of AA7050-T7451 to tensile residual stresses obtained at the end of the anodization process [15]. Cree and coworkers have demonstrated that fatigue crack growth rate on anodized AA2024 can be significantly enhanced in the presence of thin anodic film. This can be explained in terms of reduced plasticity induced closure assisted by surface cracking of the brittle oxide film ahead of the propagating fatigue crack [16].

The fatigue life decrease was sometimes related to the global anodization treatment including an initial degreasing step followed by a pickling step and finally the anodization process. During pickling treatment, according to aluminium alloy microstructure and pickling conditions, several pits could be produced due to galvanic coupling between intermetallic particles and aluminium matrix promoting the dissolution of intermetallic particles or surrounding aluminium matrix [17–19]. Shahzad and coworkers have confirmed this deleterious effect of pickling process on pitting corrosion resistance of AA2214 [17]. Fractographic examinations showed that crack initiation has been associated with constituent particles Mg_2Si and Al_7Cu_2Fe. The microscopic aspect of fracture surface for anodized specimen has shown a dual micro-mechanism of failure, with almost all crack initiation sites starting from pits, while very few were found to start from anodic film itself. The decrease in fatigue life could also be attributed to the increase number of crack initiation sites and to the simultaneous propagation of several cracks.

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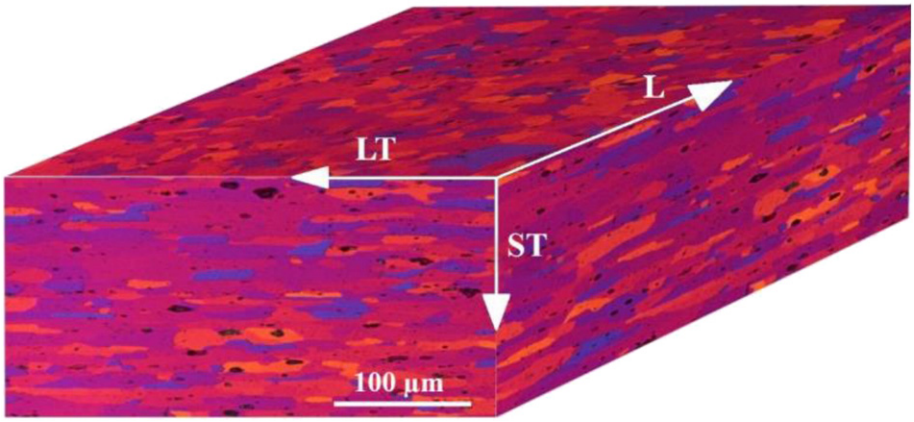

Fig. 1. Optical view of a 2024-T351 rolled plate after electrochemical etching.

It is also of great interest to consider the corrosion fatigue behavior of anodized aluminium alloys because, in service, aircraft structures can be exposed to an aggressive environment while being submitted to cyclic stresses. In literature, some works concerned the corrosion fatigue behavior of anodized aluminium alloys. Tu and coworkers have investigated the corrosion fatigue behavior of un-anodized and anodized AA2024-T3 in 3.5% NaCl solution under constant deflection-bending conditions [20–21]. They recorded the variation of E_{corr} during corrosion fatigue process, which was helpful to reveal the corrosion fatigue mechanism. Results revealed a non-significant variation of E_{corr} until specimen fracture under low-frequency corrosion fatigue conditions for un-anodized specimens; E_{corr} dropped rapidly when approaching the later stage of the corrosion fatigue fracture under high-frequency conditions. Concerning the anodized specimens, a slow drop in E_{corr} was detected from the beginning of the corrosion fatigue process until a much more fast drop at a later stage prior to fracture; this contrasted sharply with the nearly constant E_{corr} of the un-anodized specimen which was recorded for most of its corrosion fatigue life. This behavior could be explained by the breaking of the anodic film and the imperfect recovery of the surface film showing that the anodic films appeared to be readily crackable. Concerning the effect of sealing process on the fatigue behavior of anodized 2xxx series Al-alloys, there are only a few studies and the scientific knowledge about the effect of the sealing process on the corrosion fatigue behavior of these alloys is even less abundant despite the fact that many aluminium alloys are used after anodization and sealing.

In this paper, the effect of pre-corrosion damage on fatigue life and the corrosion fatigue behavior of an anodized and sealed AA2024 alloy was studied. According to the recent REACH environmental law which leads to totally ban chromium (VI) based compounds which are dangerous to health, two new alternative sealing processes were investigated, i.e. a hydrothermal sealing and a new sealing process based on a trivalent chromium process developed by Socomore consisting in a first conversion bath (SOCOSURF TCS) followed by a second passivation bath (SOCOSURF PACS), called (B1 + B2) in this work.

The comparison of the fatigue behavior of different surface states obtained at different steps of the conversion process allowed for identification of the critical parameters responsible for the strong decrease in fatigue lifetime in relation with surface treatments. The fracture modes were explained on the basis of the sample microstructure and the stress corrosion susceptibility of the different surface states.

Table 1
Average grain sizes of AA2024-T351.

Planes	L-LT	LT-ST	L-ST
Average length of grains (μm)	260 ± 126	245 ± 141	85 ± 30
Average width of grains (μm)	166 ± 78	68 ± 32	64 ± 25
Slenderness factor	1.6	3.6	1.3

2. Materials and methods

2.1. Material and geometry of the samples

The material studied in this paper was a 2024 aluminium alloy (Al base, 4.5% Cu, 1.3% Mg, wt%) provided under a 1 mm thick rolled sheet (T3 state). Treatment T3 consists of a heat-treatment at 495 °C (±5 °C), water quenching, cold working and tempering at room temperature for four days. The corrosion tests and electrochemical measurements conducted in this work were performed on this T3 state, because it corresponds to that used in the surface treatment activities of the industrial partner. However, due to the thickness (1 mm) of the rolled sheet, it was not possible to machine normalized cylindrical fatigue specimens with this sheet. Therefore, a T351 state, provided under a 60 mm thick rolled plate, was also considered in this work. The main difference between T3 and T351 states is the value of the strain during the cold working before tempering, which is more precisely controlled for T351 state than for T3 state. Furthermore, as said previously in introduction, most of the works have highlighted the predominant role of coarse intermetallic particles on anodic film characteristics. These particles, formed during solidification processes, are not significantly different in terms of nature and distribution for T3 and T351 states and it is reasonable to consider that the corrosion behavior of anodic films formed on a T3 or T351 substrates are similar. Therefore, the authors assumed that, it was possible to extrapolate the corrosion behavior observed for T3 samples to T351 samples. In order to be concise, only the microstructure of T351 state was detailed in the following paragraph.

Fig. 1 shows an optical microscope (OM, Olympus PMG3) observation of the elongated grain structure of the 2024-T351 rolled plate under polarized light. Before observation, the sample was mechanically abraded with up to 4000 grit SiC papers, polished up to 1 μm with diamond paste on the three characteristic planes and then etched. Electrochemical etching was conducted with HBF_4 (3.5 vol.%) in distilled water for 80s (twice 40s) under 20 V at room temperature. Due to the rolling process, a strong anisotropy of the microstructure was observed. The average grain sizes in L-LT, L-ST and LT-ST planes (L: rolling direction, LT: long transverse direction, ST: short transverse direction) are presented in Table 1.

A combination of optical microscope (OM) and scanning electron microscope (SEM) observations revealed a multiphase microstructure of the alloy consisting mainly in intragranular precipitates of second-phase Al_2CuMg particles (S phase particles) and Al-Cu-Mn-Fe particles. The size of these coarse intermetallic particles was between 5 and 30 μm. These observations present fair agreement with the results found in the literature [6,22]. An image analysis software (Image J) allowed to determine the surface density of coarse intermetallic particles from SEM observations. It was equal to 2.5 ± 0.6%, 4.8 ± 0.2% and

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