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# Effect of trivalent chromium process on fatigue lifetime of 2024-T3 aluminium alloy

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## ABSTRACT

### Keywords:

Aluminium alloy  
Fatigue lifetime  
Conversion coating  
Crack initiation

The effect of a new alternative trivalent chromium conversion process on fatigue lifetime of a 2024-T3 aluminium alloy was investigated. The decrease in fatigue-life induced by coating process was related to the dissolution of coarse intermetallic particles during the deoxidation pre-treatment preceding the conversion layer growth.

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## 1. Introduction

Thanks to its high strength/weight ratio, good mechanical properties and corrosion resistance, 2024 T3 aluminium alloy (AA) is widely used in the aeronautic industry. Nevertheless, its heterogeneous microstructure increases its susceptibility to localised corrosion such as intergranular corrosion, local preferential dissolution or pitting corrosion for example. It was usually shown that local dissolution and pitting corrosion were related to major alloying elements, i.e. copper and magnesium, which are involved in the formation of intermetallic coarse particles (IMCs) [1–6]. These particles, e.g. Al<sub>2</sub>CuMg (S phase) and Al Cu Mn Fe type, can be anodic or cathodic and even change of polarity compared to the aluminium matrix [7,8] leading to galvanic coupling processes and preferential dissolution or pitting corrosion [9,10]. As discussed later in this paper, these forms of corrosion are regularly observed during conversion processes.

Until now, conversion processes based on hexavalent chromium were used extensively and successfully to improve the corrosion resistance of 2xxx series AA [11–14]. However, the recent REACH regulation (Registration, Evaluation and Authorisation of CHemicals) has decided to ban hexavalent chromium for health and environment considerations. So aircraft manufacturers have developed new conversion methods based on Trivalent Chromium Process (TCP).

According to the relative novelty of this type of surface treatment, literature data are very rare or even nonexistent on the effect of these new conversion coatings on fatigue properties of aluminium alloys. However, on some aspects, a parallel can be drawn here with anodisation process, which has been more largely studied. Indeed, despite the benefits obtained in terms of corrosion protection, the anodic films have

a detrimental effect on the fatigue life in particular by promoting crack initiation [15–18].

One explanation for this result was related to the surface pre-treatment and particularly to the deoxidation step. During deoxidation step, dissolution of the matrix around IMCs was observed in several works, depending on deoxidation conditions (i.e. composition of the desmutting bath, immersion time, temperature...) and nature of IMCs [19–23]. Concerning the influence of anodised coatings on fatigue behavior, Shahzad et al. [19] and Priet et al. [24] have shown for AA2214 and AA2024, respectively that all crack initiation sites under fatigue solicitations started from pits formed during the deoxidation step whereas very few started from anodic coating. However, in the complete absence of pitting corrosion during surface treatments, concerning specifically anodised samples, fatigue cracks initiate in the coating in high stress regions whereas they initiate at the interface between coating and substrate in low stress regions [16,25]. The roughness of the treated surfaces, by TCP or anodising, could play a role too as usually seen in numerous works on fatigue [22,26–28].

In this work, the effect of a new alternative TCP on fatigue behavior of AA2024 T3 was studied in ambient air. To determine the role of the deoxidation step on fatigue lifetime, fatigue tests have been performed on samples only deoxidised by comparison with samples completely treated.

## 2. Material and experimental procedures

### 2.1. Material

The material studied was AA2024 cold rolled 3 mm thin sheet with

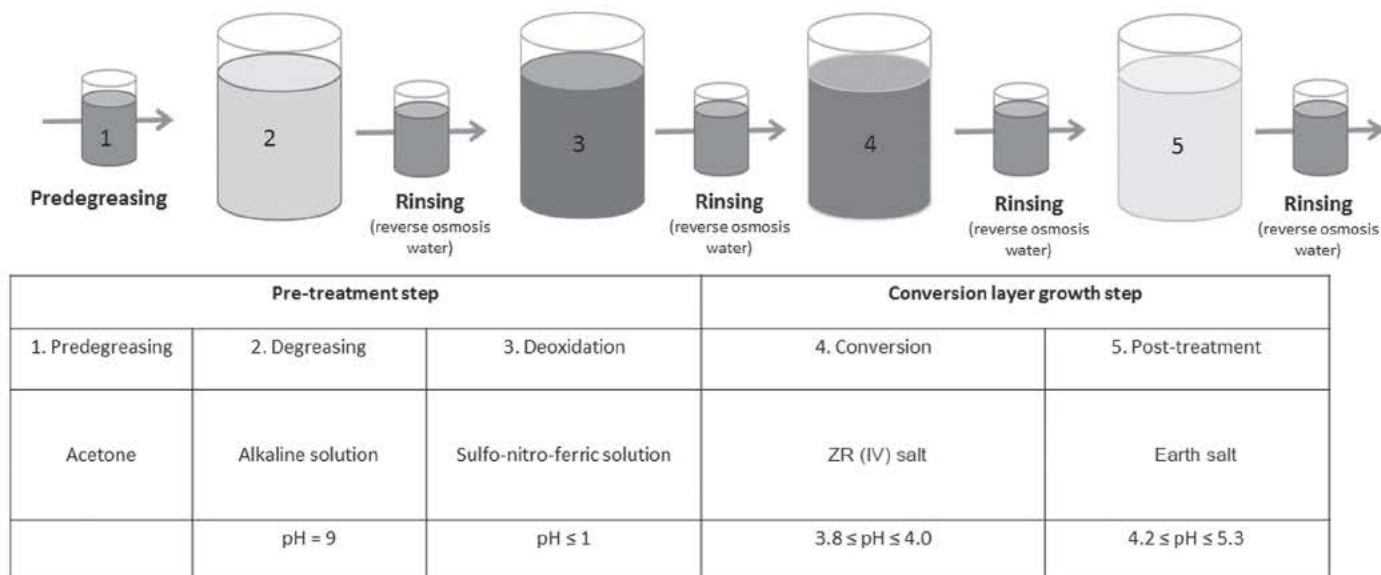
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**Table 1**  
Chemical composition of AA2024 (weight percent).

| Elements | Cu  | Mg  | Mn   | Zn   | Fe   | Si   | Ti   | Cr   | Zr   | Ni     | Al   |
|----------|-----|-----|------|------|------|------|------|------|------|--------|------|
| wt%      | 4.4 | 1.4 | 0.51 | 0.17 | 0.15 | 0.08 | 0.02 | 0.01 | 0.01 | 54 ppm | Bal. |



**Fig. 1.** Characteristics of the different TCP steps.

the chemical composition given in Table 1. The alloy was provided in the T3 metallurgical state, i.e. hot rolled, cold rolled, solution heat treated, water quenched, stress relieved and naturally aged at room temperature.

## 2.2. TCP characteristics

The whole TCP, developed by Socomore, is schematically presented in Fig. 1. This process included a first step of pre treatment followed by a second step of conversion layer growth. The TCP conditions were detailed in a previous paper [29]. Finally, a non contact 3D Surface Profiler was used in optical interferometry mode to measure the surface topology of the treated samples.

## 2.3. Tensile and fatigue tests

To determine the relevant stress levels to apply for fatigue tests, preliminary tensile tests were conducted at 25 °C on uncoated and coated flat dog bone tensile samples (2 mm thickness, 3 mm width, 20 mm gage length) machined in the L TL plane along the L direction of the cold rolled 3 mm thin sheet at a constant strain rate of  $10^{-3} \text{ s}^{-1}$ .

Stress controlled uniaxial fatigue tests were performed at 25 °C, in ambient air. Fatigue life tests were performed using a 20 Hz sine wave with a stress ratio of  $R = 0.1$  on "hourglass" fatigue samples (2 mm thickness, 3 mm width at the mid length, 32 mm gage length) machined in the same plane and direction in the thin sheet than tensile samples.

Before the experiments, all tensile and fatigue samples were ground, including edges. The edges and the heads of samples were protected by silicone during surface treatments.

## 2.4. Optical and transmission electron microscopy observations

An Optical Microscope was used to determine the grain size after an electrochemical etching. Scanning Electron Microscopy (SEM) coupled with Energy Dispersive X ray spectroscopy allowed to characterize

IMCs. Finally, a SEM with a Field Emission Gun and a Transmission Electron Microscope (TEM) were used to observe intergranular pre precipitates and, both intergranular and intragranular hardening pre precipitates, respectively. The details of sample preparation are given in previous paper [29].

## 3. Results and discussion

### 3.1. Microstructures

AA2024 T3 microstructure has been already characterised finely in a previous work [29]. Only main results are reminded here. Average grain sizes of about 22  $\mu\text{m}$  in the longitudinal (rolling) direction and 19.2  $\mu\text{m}$  in the long transverse direction were measured (Fig. 2a). SEM observations showed two types of IMCs, i.e.  $\text{Al}_2\text{CuMg}$  particles (mean surface area close to 2.9  $\mu\text{m}^2$ ) and Al Cu Mn Fe type IMCs (mean surface area close to 6.8  $\mu\text{m}^2$ ). At a finer scale, 200 nm dispersoids were also visible (shown by black arrows in Figs. 2b and 2c); they were composed of Cr, Zr or Mn. No intergranular precipitation was visible as well as Guinier Preston Bagaryatsky (GPB) zones [30]. However, hardness values (under a 200 g loading) close to 143 HV compared to 123 HV for a freshly treated material led to assume the presence of GPB zones due to natural ageing.

### 3.2. Effect of TCP on the tensile properties of AA2024 T3

The tensile mechanical characteristics of uncoated and coated AA2024 T3 are summarised in Table 2. No significant difference was observed between uncoated and coated samples mainly due to the low thickness of the conversion layer, i.e. 135 nm [29]. On the basis of these tests,  $YS_{0.2\%}$  value was determined close to 380 MPa.

### 3.3. Effect of surface treatments on fatigue lifetime

Fatigue life curves are presented in Fig. 3. It was first of interest to note that the fatigue lifetime was quite similar between deoxidised and

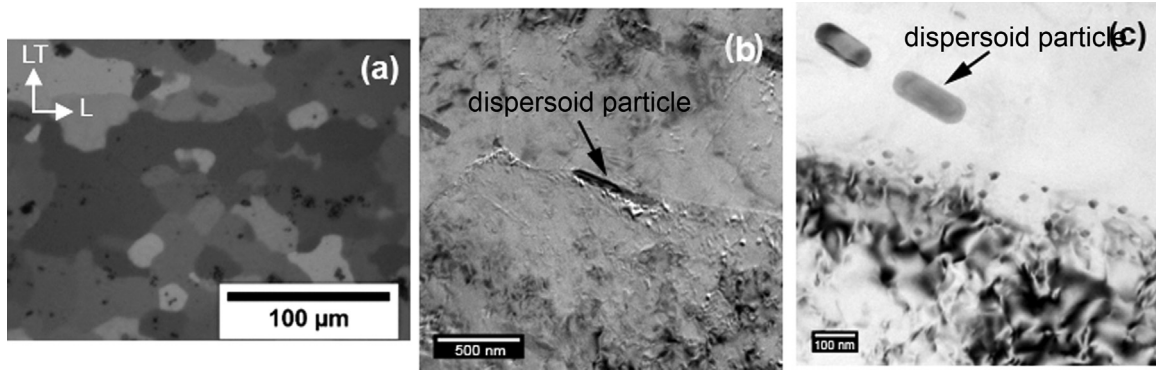


Fig. 2. Optical (a) and TEM (b, c) observations of AA2024-T3.

**Table 2**  
Tensile properties of uncoated and coated samples of AA2024-T3.

| Tensile properties | YS(MPa) | UTS (MPa) | $\epsilon_f$ (%) |
|--------------------|---------|-----------|------------------|
| Uncoated 2024-T3   | 380     | 580       | 18.3             |
| Coated 2024-T3     | 380     | 580       | 17.8             |

coated samples and substantially lower compared to uncoated samples. This result suggested that deoxidation process was the main step of the TCP responsible for the fatigue lifetime decrease.

The second noticeable result was that no effect of deoxidation or complete TCP was seen on fatigue life curves at high stress levels. It was necessary to reach intermediate and low stress levels to observe a decrease of fatigue lifetime due to surface treatments, i.e. around one order of magnitude at 320 MPa. This result suggested that, at high stress levels, for uncoated, deoxidised and coated samples, the same type of surface defects were responsible for crack initiation contrary to the lower stress levels for which new initiation site(s) was(were) probably effective for deoxidised and coated samples. This (these) site(s) was (were) identified on the basis of fracture surfaces observations by SEM and roughness measurement/mapping.

**Table 3**  
Roughness of uncoated, deoxidised and coated surfaces of AA2024-T3.

|                 | Uncoated samples | Deoxidised samples | Coated samples |
|-----------------|------------------|--------------------|----------------|
| Roughness (RMS) | 7 nm             | 14.7 nm            | 7.5 nm         |

### 3.4. Effect of surface treatments on fatigue crack initiation

At first, roughness measurements were performed for uncoated, deoxidised and coated samples (Table 3). According to literature data [31], the average roughness of the coated sample was close to the initial roughness of the uncoated and polished material. On the contrary, the roughness of the deoxidised sample was significantly higher, twice that of both uncoated and coated samples. Therefore, the coating step seemed to reduce the average roughness of deoxidised samples. However, strong local variations were observed, for both deoxidised and coated samples, in relation with the reactivity of S Al<sub>2</sub>CuMg particles, during deoxidation step [9,10,22,29] leading to local dissolution processes (Fig. 4). Harvey et al. have shown that these phenomena could be compared to corrosion processes occurring in NaCl solutions [32]. In deed, S Al<sub>2</sub>CuMg particles are more reactive than Al Cu Mn Fe particles

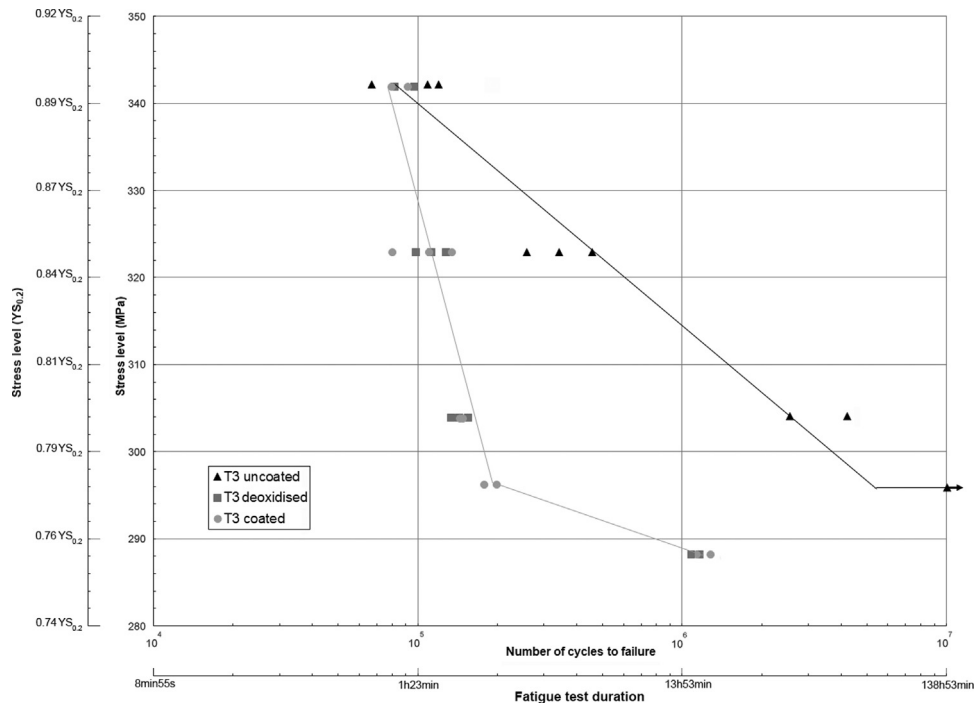


Fig. 3. S-N curves of uncoated, deoxidised and coated samples of AA2024-T3.



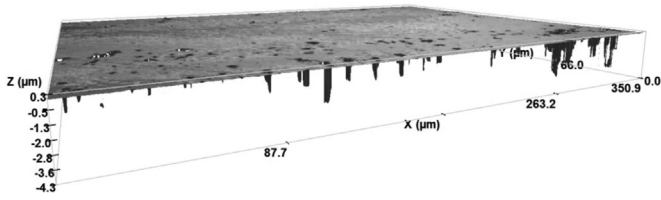


Fig. 4. Roughness map of a coated sample.

because their potential is more negative than this of the aluminium matrix. Changes in their composition during corrosion processes lead to an inversion in polarity that induces a dissolution of the surrounding matrix and finally the removal of the particles from the surface. Concerning Al Cu Mn Fe particles, only the dissolution of the matrix around the particles was observed due to their higher potential by comparison with the matrix.

The fact that fatigue life curves of deoxidised and coated samples were similar while the surfaces of the corresponding samples presented a different average roughness suggested that the average roughness was not a first order parameter responsible for the decrease in fatigue lifetime. A more local approach of the surface state, considering in particular the removal of S Al<sub>2</sub>CuMg particles from the sample surface, had to be considered.

SEM observations of fracture surfaces after fatigue tests are presented in Figs. 5 and 6 for uncoated and deoxidised/coated samples, respectively. Independently of the surface treatment and the applied stress level, the fracture surfaces presented three distinct zones characterised by different fracture modes: the crack initiation zone followed by the crack propagation zone and then the ductile final fracture with dimples (Fig. 5a). A crystallographic propagation, i.e. quasi cleavage fracture, characterised by river patterns, was observed on the fracture surfaces (Fig. 5b) associated with striation patterns (Fig. 5c). This crystallographic propagation is generally associated to environment and more particularly to water vapor and/or hydrogen [33 36]. In deed, depending on environmental conditions and material, water vapor or hydrogen resulting from the chemical dissociation of the adsorbed water vapor molecule on the fresh surfaces at the crack tip can assist crack propagation. In this work, crack initiation was always localised on IMCs for uncoated samples, regardless of the stress level, due

to the localisation of plasticity on these particles, leading to their shearing (Fig. 5d).

Concerning the deoxidised and the coated samples, quasi cleavage propagation and ductile finale fracture were not modified by comparison with uncoated specimens. Only crack initiation sites were modified for intermediate and low stress levels as already suggested by S N curves (Fig. 3), whereas, for high stress levels, IMCs were always preferential crack initiation sites. Indeed, according to the roughness map, the removal of S Al<sub>2</sub>CuMg particles during deoxidation process could explain the premature crack initiation as seen in Fig. 6. Fig. 6a presents an observation of one lateral face of a deoxidised fatigue sample. Local dissolution processes induced by IMCs clusters were clearly visible as well as the preferential initiation of a secondary crack from these surface topology irregularities. This result was also observed under the fracture surface of coated samples as highlighted by Fig. 6b. Once again it is possible to observe, for a secondary crack, a preferential crack path along local dissolution zones confirming the role of the deoxidation step on the decrease in fatigue lifetime. It could be possible that IMCs reactivity was exacerbated after machining and mirror polishing of samples, which could be confirmed by Kelvin probe Force Microscopy [37]. Nevertheless, the premature crack initiation on surface defects induced by deoxidation processes localised on IMCs can explain the decrease of the fatigue lifetime for both deoxidised and coated samples at intermediate and low stress levels and the relative low scattering of fatigue lifetime values measured in this work.

#### 4. Conclusions

The effect of a new alternative TCP on fatigue lifetime of AA 2024 T3 was investigated. A noticeable decrease in fatigue lifetime was observed for deoxidised and coated samples at intermediate and low stress levels suggesting a modification of crack initiation sites. According to surface map and fracture surface observations, it was shown that deoxidation treatment was the main responsible for this decrease in fatigue properties that could be explained by the removal of S Al<sub>2</sub>CuMg particles from surface material during the deoxidation step. Finally, at high stress levels, crack initiation took place on IMCs whereas, for lower stress levels, surface topology irregularities due to the deoxidation step was more detrimental.

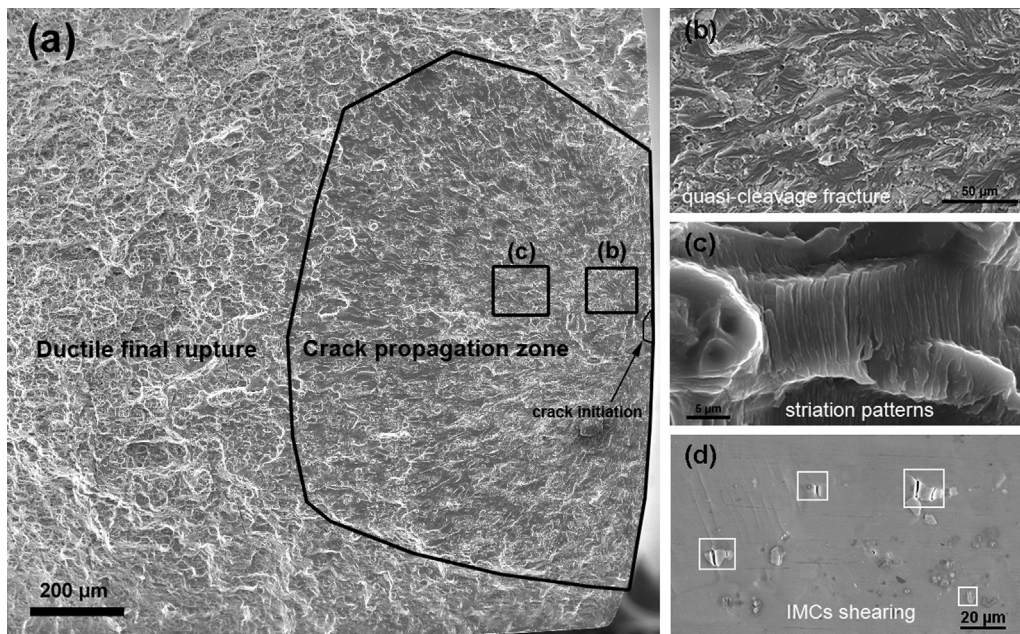


Fig. 5. Rupture surface observations by SEM of uncoated AA2024-T3 after fatigue tests. Global view (a), crack propagation zone: rivers (b) and striation patterns (c) and shearing of IMCs on sample lateral surface (d).

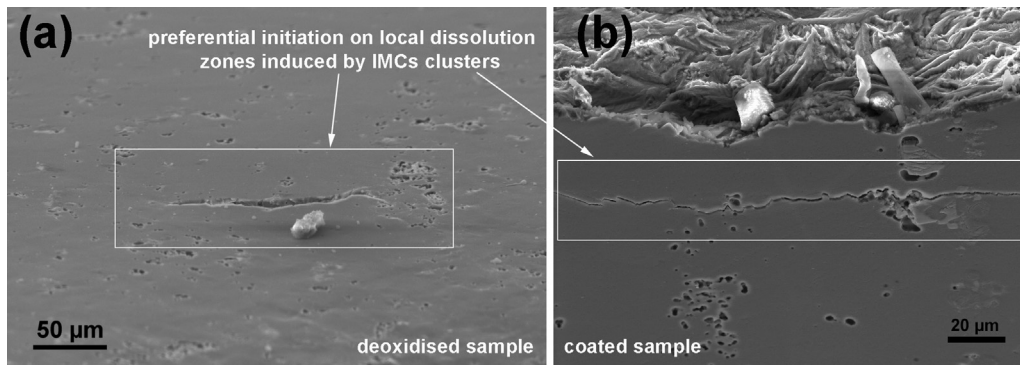


Fig. 6. Fracture surface observations by SEM of deoxidised (a) and coated (b) AA2024-T3 after fatigue tests.

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