Synergistic action of fatigue and corrosion during crack growth in the 2024 aluminium alloy

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Abstract

This study was undertaken with the aim of identifying the domains where synergistic interactions between mechanical, environmental and microstructural parameters may occur during corrosion fatigue crack growth in the aluminum alloy 2024, as well as the rate controlling factors. Corrosion fatigue crack propagation tests have been carried out at different frequencies and/or waveforms, grain orientations and tempers, in air, distilled water and 3.5% NaCl solution at free potential. In saline solution in the S-L orientation, crack growth rates are shown to decrease with decreasing frequency. In addition, at high frequencies, these growth rates are substantially higher than in air and in distilled water. This enhancement is accompanied by a typical change in the shape of the da/dN curve. It is demonstrated that this enhancement, that is only observed in metallurgical conditions of high sensitivity to stress corrosion cracking, is controlled by the loading rate, and not by the duration of exposure to the corrosive medium. The behaviour is discussed on the basis of a competition between passivation and anodic dissolution and/or hydrogen embrittlement. It is finally proposed that the fatigue crack growth enhancement observed is related to a crack-tip embrittlement mechanism by hydrogen produced by anodic dissolution in relation with film rupture periodicity.

Keywords: frequency; stress corrosion cracking; slip dissolution; passivation; hydrogen embrittlement

1. Introduction

Corrosion and fatigue of aluminium alloys are major issues in the service life assessment of aircraft structures and in the management of aging air fleets. The prediction of residual life is all the more complex that these processes can interact to promote damage, especially during crack propagation [1]. More precisely the fatigue data derived from accelerated laboratory tests can be extrapolated to actual service lives which extend over several decades with a high level of confidence provided that the environmentally-assisted mechanisms are fully understood. In particular, the influence of frequency has to be correctly taken into account since the load frequency selected for fatigue crack propagation testing is generally higher than the load frequency withstood in service conditions in order to shorten the duration of experimental investigations. Now low frequencies may promote
synergistic effects between mechanical loading and environmental exposure at the crack tip, and as a consequence enhance Fatigue Crack Growth Rates (FCGRs) [2-4]. Therefore accelerated tests may provide irrelevant data for structure design as soon as time-dependent corrosion processes at the crack tip may affect propagation rate. However literature also contains some evidences that this tendency is not systematically observed in some particular systems [5-10]. This indicates that the propagation domains, in terms of frequency and load levels, where detrimental synergistic effects between fatigue damage and corrosion may take place at the crack tip still need to be clearly identified.

The study presented here was precisely undertaken in order to identify the domains of possible synergistic interactions between fatigue and corrosion in aluminium alloys, to identify the controlling factors and improve the understanding of environmentally-assisted mechanisms. In this aim, the 2024-T351 aluminum-copper alloy was selected. Most of the fatigue crack propagation tests reported in this paper were conducted in the S-L orientation, which is known to be the most susceptible to environmental attack in this alloy. The fatigue crack propagation behaviour observed in saline solution is compared to the behaviour exhibited in air and in distilled water. The influence of load frequency and/or waveform is examined into details.

2. Material and experimental techniques

The material of this study is a 2024 aluminium alloy with nominal composition Cu 4.5%, Mg 1.4%, Mn 0.60%, Fe 0.13%, Si 0.06%, Ti 0.03%, Zr + Ti 0.03%, Al balance, in the temper condition T351. The average grain size is 90 μm, 210 μm and 820 μm in the short transverse S, long transverse T and longitudinal L direction, respectively. The microstructure revealed by etching with Keller Reagent of the alloy and observed with a light microscope is presented in Figure 1. Compact Tension specimens W=40 mm, thickness B=10 mm, have been machined in the S-L orientation from a 50 mm thick plate provided by EADS IW and manufactured by CORUS. The crack plane is located at mid thickness of the plate where a higher sensitivity to stress corrosion cracking is expected due to the slower cooling rate and the resulting precipitation structure [11].

![Figure 1. Microstructure of the 2024-T351 alloy used in the study.](image)

Fatigue crack propagation tests were performed under load control using constant amplitude cyclic loading, i.e. under increasing ΔK. Precracking was initiated in air at 10 Hz to a crack length of about 0.6 mm, using a sinusoidal waveform at the same loading amplitude and load ratio as used for the experiment. For specimens tested in a 3.5% NaCl aqueous solution, a final crack growth increment of about 1 mm was conducted in the aqueous environment under full test conditions as recommended by ASTM E647 [12]. Three typical environments have been considered in this study: laboratory air (20-30°C), distilled water and a saline solution composed of distilled water with 3.5% NaCl addition (pH = 7). For crack propagation tests in air, crack length was derived from compliance measurements. The load ratio R was set at 0.7 for all the tests in order to limit crack closure effects.

Corrosion fatigue experiments in aqueous solution were carried out at free corrosion potential. Specimens were immersed in water with 3.5% NaCl without loading for 24 hours prior to testing [12]. During corrosion-fatigue tests,
CT specimens were fully immersed in a Plexiglas cell mounted on the fatigue machine and non metallic grips were used to avoid galvanic corrosion between the specimen and the cell. A pump connected to the cell ensured a constant flow of the aqueous environment. Crack length was measured optically by mean of a low magnification travelling microscope on one varnished side of the specimen. Measurements carried out by means of a Saturated Calomel Electrode during corrosion fatigue crack propagation tests at 0.1 and 10 Hz, provided potential values ranging from -600 to -700 mV/SCE. Crack closure has not been measured but it is not likely to occur at R = 0.7. Indeed, no crack closure has been detected in air at this load ratio during the fatigue tests in air reported in this study. After the test specimens were cleaned in an ultrasonic bath, then in acid nitric during a few minutes according to ASTM G1 recommendation [13]. Fracture surfaces were observed using a Scanning Electron Microscope (SEM) JEOL 6400.

3. Results

The Fatigue Crack Growth Rates (FCGRs) \( \frac{da}{dN} \) measured at different frequencies under sinusoidal signals in 3.5% NaCl solution are presented in Figure 2 as a function of \( \Delta K \), and compared to data obtained in air and distilled water. Two characteristic behaviours controlled by frequency can be identified. The first type of behaviour is characterised by FCGRs higher than those measured in air and distilled water, especially at low \( \Delta K \) values. In addition they do not depend on frequency in the frequency range 2.5-10 Hz. Finally the Paris law exponent is close to 2, regardless of frequency, while the exponent value in air and distilled water is about 4. The second type of behaviour is observed at frequencies lower than 1 Hz. In this regime the propagation is significantly slower than at 2.5 Hz and higher frequencies. Meanwhile the Paris law exponent value shifts from about 2 at high frequencies to 4, a value close to the one observed in air in the explored \( \Delta K \) range. Finally, when frequency is decreased from 1 to 0.1 Hz, the Paris law exponent does not much vary while FCGRs progressively decrease and become comparable to those obtained in air.

![Figure 2](image-url)
Fracture surfaces produced in air and in saline solution at different frequencies basically present the same appearance, with a transgranular, cleavage-like cracking mode for high values of $\Delta K$ (Figure 3). At lower $\Delta K$ values however, although the same cleavage-like appearance is still prevailing, subtle differences can be noticed in the distribution of flat, large and smooth facets, as pointed out by the small dark arrows in Figure 4.

![Figure 3](image1.png)  
(a) (b)  
Figure 3. Fatigue fracture surfaces, (a) air (b) 3.5% NaCl solution. Large arrow: macroscopic crack growth direction ($\Delta K = 6$ MPa$\sqrt{m}$, Sinusoidal waveform, 10 Hz, R=0.7, S-L orientation).

![Figure 4](image2.png)  
(a) (b)  
Figure 4. Fatigue fracture surfaces. Sinusoidal waveform (a) air (b) 3.5% NaCl. Large arrow: macroscopic crack growth direction (10 Hz, R=0.7, S-L orientation, $\Delta K = 3$ MPa$\sqrt{m}$).

![Figure 5](image3.png)  
Figure 5. Influence of the loading frequency on the FCGR of the alloy 2024-T351 for a sinusoidal waveform (R=0.7, S-L orientation).

![Figure 6](image4.png)  
Figure 6. Different waveforms used for fatigue crack propagation tests in saline solution.
4. Analysis and discussion

The results presented above have revealed an influence of frequency on the fatigue crack growth rates of the 2024 alloy, associated with characteristic transitions in da/dN curves and changes in power law exponent, as schematically represented in Figure 5. It is noteworthy that, in the present case, the maximum FCG enhancement in saline solution as compared to air is observed at higher frequencies. Indeed, FCGRs are expected to increase with decreasing loading frequency, since more time will be available for time-dependent reactions and mechanisms, or to be frequency independent if the environmentally-assisted process is saturating [3,14]. The analysis of this unexpected behaviour is conducted by investigating the rate-controlling parameters of this frequency effect and the environmentally-assisted mechanisms that may be consistent with the observed phenomena.

4.1. Rate controlling factors

The influence of frequency can be attributed to two distinct factors, namely the rising time (RT), which in first approximation governs crack tip deformation rate $\dot{\varepsilon}_{CT}$, and the cycle period $T$, which determines the total duration of exposure to the corrosive medium of the plastically deformed material at the crack tip. Additional tests have been specially designed and performed in order to identify the respective contribution of RT and $T$ to the observed FCGR enhancement observed at high frequencies under sinusoidal signals. Saw-tooth waveforms have been considered since they allow a modification of RT without changing the cycle period $T$ with respect to sinusoidal signal (Figure 6). A loading cycle consisting in a high RT – low unloading time is defined as a positive saw-tooth signal (Figure 6b), while a negative saw-tooth signals is defined as a signal with a low RT and high unloading time (Figure 6c). Negative and positive saw-tooth signals have been applied at two frequencies: 1 and 5 Hz. The results are presented in Figure 7 (a) and (b) for 5 Hz and 1 Hz, respectively. At 5 Hz (Figure 7a) no influence of RT on FCGRs is noticed. Indeed sinusoidal, positive and negative saw-tooth waveforms results into comparable FCGRs. The results obtained under negative saw-tooth signal, corresponding to a very short RT (0.05s), suggests that the effect of corrosion on FCG enhancement as a function of RT is saturating at 5 and 10 Hz under sinusoidal cyclic loading. In the case of the positive saw-tooth signal, the RT is actually identical to that corresponding to a 2.5 Hz sinusoidal waveform while the cycle period is longer. This indicates that RT is the main controlling parameter and that the cycle duration $T$ has no influence on FCGR [15].

At 1 Hz (Figure 7b), FCGRs measured under negative saw-tooth waveform are similar to those obtained at high frequencies and higher than those measured at the same frequency under sinusoidal signal. Moreover the value of the Paris law exponent is close to 2 in both cases, and not 4 as observed at 1 Hz under sinusoidal waveform. This statement once again confirms that RT is the main parameter governing the transition between the two characteristic behaviours represented in Figure 5. Under positive saw-tooth waveform, the propagation is slower than under sinusoidal waveform at the same frequency while the value of the Paris law exponent is unchanged and close to 4. This means that RT might also presumably be responsible for the slight decrease of FCGRs observed at 0.1 Hz as compared to 1 Hz. The examination of this table confirms that a short RT (typically RT<0.5s) in saline solution results into a Paris law exponent close to 2, while for higher values of RT the Paris law exponent is similar to that observed in air or distilled water.

Finally two other waveforms have been considered (Figure 6 d-e) in order to investigate the possible influence of the duration of immersion in solution on FCGR enhancement. A long immersion time (9.9s) has been introduced at the minimum or at maximum load in a 10 Hz triangular waveform signal. Thus in both cases the loading and unloading rates correspond with a 10 Hz triangular waveform cycle, while the total period, and as a consequence the exposure duration, is the same as for a sinusoidal loading at 0.1 Hz. The comparison of the behaviour exhibited under these trapezoidal signals and sinusoidal signals at 0.1 and 10 Hz is presented in Figure 8 indicates that the behaviour observed under trapezoidal waveforms is actually extremely similar to that observed at high frequencies under sinusoidal signal, and as a consequence FCGRs are higher than at 0.1 Hz under sinusoidal signal and/or at short RT. Moreover the da/dN curve presents almost the same slope as at high frequencies under sinusoidal signal. This shows that the introduction of a hold time at minimum and maximum load in order to lengthen the immersion duration has no influence on FCGRs and that the typical change in behaviour observed in Figure 2 is mainly controlled by the RT.
Figure 7. Fatigue crack propagation rates for waveforms (b) and (c) in saline solution as compared to air at (a) 5 Hz (b) 1 Hz.

Figure 8. Fatigue crack propagation rates for waveforms (d) and (e) in saline solution as compared to air.

It is however worth noticing that the influence of waveform and rising time observed here in a 2024 alloy is opposite to that reported by Selines and Pelloux [16] in a 7075 alloy. Indeed the first author noticed a FCG
enhancement in a 3% NaCl solution at low strain rates under sinusoidal and triangular waveform, while a square waveform produced FCGR data similar to those produced in air. The latter authors observed a FCGR enhancement as the rising time was increased in salt water, regardless of the waveform. They suggest that this behaviour might be accounted for by the formation of an oxide layer at the crack tip that would hinder plastic blunting by limiting dislocation emission and presumably by this means promote crack growth by cleavage. The present results seem however consistent with those presented by Piasick and Gangloff [17] who also noticed a characteristic transition in the da/dN curve corresponding with a FCGR enhancement between salt water on the one hand, water vapour or moist air on the other in the case of a 2090 alloy. According to these authors, this transition occurs at different ΔK values according to the considered environment (air / water vapour or salt water). Meanwhile these authors also noticed a decrease in FCGRs when the loading frequency was reduced [9]. These authors claim that this type of frequency effect would be unique to this alloy; the results presented here show that this is not the case, even though this frequency effect is more pronounced at high ΔK values in the 2090 alloy. In any case, the results presented here demonstrate that the shift in Paris law exponent value from 4 to 2 and the associated FCGR enhancement is controlled by a synergistic effect between cyclic plastic deformation at the crack tip and the action of the corrosive medium. However the rate controlling process responsible for this abrupt transition in a narrow RT range and the cracking mechanism need to be elucidated.

4.2. Environmentally-assisted cracking mechanism

The detailed analysis of the cracking mode as influenced by environmental effects is beyond the scope of the present paper. However the question of the mechanisms responsible for the observed FCGR enhancement must be raised in order to evaluate the consistency with the observed influence of RT. According to literature [4,18], three main mechanisms can be considered for corrosion-fatigue crack growth in aluminium alloys, namely slip dissolution, surface energy reduction and hydrogen embrittlement at the crack tip. Surface energy reduction is actually the mechanism proposed to account for the action of ambient air in the upper part of the FCGR regime considered here [19]. In particular it has been shown that water vapour adsorption induces a FCGR enhancement without significant change in the propagation mechanism which is characterised by a 4th power law dependence of da/dN versus ΔK, while a power law exponent close to 2 is generally associated with a hydrogen-assisted mechanism [20]. This suggests that hydrogen might also be involved in the behaviour characterised by a 2nd power law reported in the present study. The question is then to examine to what extend this assumption is consistent with the observed RT effect.

According to Ford [7], the slip-activated dissolution process responsible for stress corrosion cracking in these alloys might still be operative and even enhanced during corrosion-fatigue crack growth. Thus, at low ΔK values, for a fixed frequency, FCGR would be primarily controlled by the dissolution rate. At high ΔK values, where the mechanical rupture of the crack tip film might be induced by intense slip deformation at the crack tip, the periodicity of oxide film rupture would become short as compared to the oxide incubation and growth rate. In this regime FCGR will be governed by the early stage of oxide growth. This regime might be furthermore strongly influenced by the presence of anions such as chlorides in the electrolyte. The possible role of dissolution could be supported in the present investigations by the absence of FCGR enhancement in distilled water. However no clear evidence of extensive crack advance by dissolution was found on fracture surfaces. The main effect of saline solution on fracture mode in the synergistic domain resides in the larger fraction of large smooth and flat facets which have been associated with an intergranular crack path [21], as shown in Figure 9. However, since these facets, while much less numerous, are also present in air, where no electrolyte is present at the crack tip, they do not represent a signature of dissolution. It is also worth noticing that these facets are much less numerous when the same alloy is tested in metallurgical sates corresponding with a much lower sensitivity to stress corrosion cracking, i. e. in the L-T orientation in the T351 temper or in the S-L orientation in the T851 temper [22]. Thus it is suggested that the formation of these intergranular facets is promoted in saline solution and that the synergistic domain is related to severe dissolution in the vicinity of grain boundaries as during stress corrosion cracking.

Therefore the periodic sequence dissolution/passivation/film rupture seems to play a key role in this type of frequency effect, in relation with crack tip strain rate, even though, as pointed out Piasick and Gangloff [17], hydrogen embrittlement might also at least partly account for the observed FCGR enhancement. Indeed the dissolution of bare surfaces at the crack tip may also promote hydrogen production and entry, which would be
consistent with the observed FCGR enhancement. However the results of the present study are not, at a first glance, fully consistent with such an analysis predicting a FCGR enhancement in saline solution when increasing the crack tip strain rates. Indeed, for a fixed high frequency, the higher FCGR enhancement is observed at low ΔK values, and as a consequence at low crack tip strain rates. The point is however that at high ΔK values the higher “mechanical” damage might also compete with environmentally-assisted processes.

Figure 9. Percentages of flat facets measured on fracture surfaces produced in the S-L orientation and reported in a da/dN-ΔK graph.

It is therefore proposed that the effect of short RT on FCGRs described here would be primarily related to localized dissolution and concomitant hydrogen production during cyclic plastic deformation. Hydrogen atoms would be then uptaken into the process zone at the crack tip where the embrittling process takes place. Conversely, for higher values of RT, crack tip surfaces would be passivated during a large amount of the plastic deformation. As a consequence, the hydrogen production and/or entry would be so reduced that hydrogen embrittlement could not induce significant effect on FCGRs with respect to mechanically induced damage. Therefore, the negative frequency effect will be presumed to be due to passivation of the crack tip. Furthermore, as the slope 2 regime disappears and the FCGRs sharply decrease when frequency is decreased from 2.5 to 1 Hz under sinusoidal signal, the hypothesis of passivation requires that a film forms at the surface of the crack tip in less than about 500 ms (rise time at 1 Hz). Repassivation kinetics of bare surfaces have been measured by Kim and Pyun [23] on abraded surfaces of pure aluminium in 0.5 M Na2SO4 solution with different concentrations of chloride ions as a function of applied potential. The results obtained under open circuit potential and a concentration of 0.01M NaCl indicate that the potential rises very rapidly within about 2s and then reaches a steady state value which depends on pH and which corresponds with the thickening of the surface layer. The value estimated in the present study seems consistent with these findings, although the role of metallurgical factors, in particular copper, on the repassivation kinetics should be precised. This process is actually similar to that originally proposed by Gangloff and co-workers [9,24] to account for frequency effects in 7XXX alloys. As mentioned above, the occurrence of these synergistic effects is untimely related to severe localized dissolution processes along the crack path in relation with metallurgical parameters such as grain orientation and tempering [22]. Such a relation between crack tip hydrogen embrittlement, that is directly responsible for the degraded crack propagation resistance, and dissolution, that does not directly control environmentally-assisted crack growth but is necessary to hydrogen production, has been recognised by Holroyd and Hardie [25] in 7XXX alloys. However differences in dissolution mechanisms between the present alloy and alloys from the 7XXX series might at least partly account for the opposite influence of loading waveform.
5. Summary and Conclusion

The aim of this study was to explore the possible synergistic interactions between mechanical, environmental and microstructural parameters during corrosion fatigue crack growth in the 2024 aluminium alloy. A corrosion-enhanced fatigue crack propagation regime, characterized by a 2 power law exponent in the da/dN curve, has been evidenced in a 3.5% NaCl solution at R=0.7, in the temper T351, in the S-L orientation, with waveforms having a short load rise time. If any of those parameters is modified, the regime disappears and the crack growth behaviour is similar to that observed in air and distilled water. This suggests that the occurrence of this synergistic domain is related to a high sensitivity to stress corrosion cracking. Consistently, although the crack path is mostly transgranular in this regime, evidences of intergranular cracking, which is the prevailing fracture mode in stress corrosion cracking, have been noticed. Finally it has been demonstrated that the FCGR enhancement is controlled by the load rise time, and not by the duration of the exposure to the corrosive solution.

The observed fatigue crack growth enhancement has been attributed to a hydrogen embrittlement process at the crack tip. However, a severe localized dissolution, related to the stress corrosion cracking sensitivity, is required to produce hydrogen at the crack tip. The effect of rise time has been analysed along a competition between crack tip strain rate on the one hand and slip dissolution and repassivation kinetics on the other hand.

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