



# Modelling of corrosion fatigue crack initiation on martensitic stainless steel in high cycle fatigue regime



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## ABSTRACT

This paper presents an analytical model for assessing the corrosion fatigue crack initiation life on a martensitic stainless steel X12CrNiMoV12-3 in high cycle fatigue regime (between  $10^5$  and  $10^7$  cycles). Based on in-situ electrochemical measurements during corrosion fatigue tests in NaCl aqueous solution, the corrosion fatigue crack initiation mechanism was identified. Two main stages were investigated: (i) the fracture of the passive film by slip bands and (ii) the free dissolution of the metal developing fatigue crack initiation from a critical corrosion defect. The depassivation stress threshold corresponds to the median fatigue strength at  $10^7$  cycles for fatigue corrosion tests. For an applied stress range less than this threshold, the depassivation phenomenon was not observed at  $10^7$  cycles and no crack initiation occurred. The proposed model takes into account the depassivation process induced by the slip bands emergence at the specimen surface and the corrosion rate under cyclic loading. The experimental results are compared to the proposed model taking into account mechanical and electrochemical material parameters.

## 1. Introduction

The practical importance of fatigue failure in structural materials has motivated many works towards assessing the physical reasons for material sensitivity to corrosion fatigue (CF) and providing adequate damage models for engineers. In particular, developing an analytical methodology for predicting corrosion fatigue crack initiation (CFCI) [1–8] and corrosion fatigue crack growth [7,9,10] for structures is a key issue. A model to compute the fatigue life of material (to crack initiation) under corrosion fatigue should take many variables into consideration: chemical nature of the environment, stress/strain levels, stress ratio and loading frequency. Most investigators agree that CFCI occurs at the surface around corrosion defects [11–14]. The effect of corrosion on fatigue life of stainless steels is most closely related to the passive film resistance [11,15]. Much study has been focused on the effect of the microstructure, the thickness and the chemical composition of the passive films, on corrosion behaviour of metallic alloys, using surface analyses techniques [16–18]. But the mechanical behaviour of passive layer (thickness lower than 10 nm) stay to be difficult to characterise experimentally.

Several CFCI models were proposed and can be classified into:

1. competition between pit growth and short crack propagation [1,2],
2. preferential dissolution of plastically deformed material [3,19,20,8,14],
3. local rupture of the passive film by slip bands [4–6,21–24] and
4. lowering of surface energy due to adsorption of specific species from the environment [7].

All of the above mechanisms involve a synergetic contribution of mechanical loading and electrochemical behaviour of tested material. In order to accurately predict the CFCI life, it is necessary to consider synergetic effects between cyclic loading (stress level, load ratio, loading frequency), material (passive layer) and corrosion parameters [22,12,15,8]. Recent advances in local electrochemical characterization techniques have allowed to develop research works at the microscopic scale [25]. These techniques are used in stress corrosion cracking (SCC) tests to capture, in-situ, the temporal electrochemical behaviour [26,27]. Experimental evidence show that pit growth rate is influenced by static stress in SCC tests [28,27,29] and by cyclic stress [6,30,31]. In HCF tests, the magnitude of synergetic effects is of major influence the pit initiation (depassivation) stage. This phenomenon can be evaluated by the use of in-situ global free potential measurement during CF test [32,11,33]. That allows to characterize the depassivation and corrosion

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Nomenclature			
AES	Auger electron spectroscopy	$\Delta\epsilon^P$	plastic strain range: $\Delta\epsilon^P = \Delta\epsilon_{\max}^P - \Delta\epsilon_{\min}^P$
HCF	high cycle fatigue	$\sqrt{\text{area}}$	square root area of the defect projected onto a plane perpendicular to the maximum normal stress
CFCI	corrosion fatigue crack initiation	SCE	saturated calomel electrode
CF	corrosion fatigue	$E_{\text{free}}$	free electrochemical potential
PSB	persistent slip band	$I_{\text{corr}}$	current value achieved during activated corrosion period ( $N > N_d$ )
$N_f$	number of cycles to fatigue crack initiation	$i_{\text{corr}}$	current density, the exposed surface is 1 cm <sup>2</sup>
$N_d$	number of cycles to depassivation	$A_s$	aqueous solution with 0.1 M NaCl and 0.044 M Na <sub>2</sub> SO <sub>4</sub>
$N_c$	number of cycles to develop a corrosion critical defect with regard to fatigue crack initiation	$\rho$	volumetric mass
$R$	loading ratio	$F$	Faraday constant
$f$	loading frequency	$M$	molar mass
$\Delta\sigma$	stress range: $\Delta\sigma = \sigma_{\max} - \sigma_{\min}$	$\delta_s$	slip band height
$\Delta\sigma_{\text{th}}$	threshold value of $\Delta\sigma$ for depassivation	$h_s$	slip band spacing
$\Delta\sigma_{\text{eff}}$	effective stress range: $\Delta\sigma_{\text{eff}} = \Delta\sigma - \Delta\sigma_{\text{th}}$	$b$	burgers vector
		$e$	corrosion defect thickness

activity at the surface of the specimen during cyclic loading.

In the case of stainless steels, CF crack initiation is promoted by cyclic mechanical damage of the protective passive layer induced by the emergence of slip band. Passive films can act as obstacles to dislocation activity [21,15]. Consequently, the PSB spacing will be small because slip localization is inhibited [34,35]. By raising the dissolution rate, or by making the oxide film thinner, the barriers to slip are reduced, slip localization is favoured and PSB spacing increases [22]. The matrix dislocations tends to be transferred more easily into the PSB, further accelerating the formation of PSBs causing local ruptures of the passive layer [34]. Once the passive film damaged, the metal is directly exposed to the aqueous environment, resulting in preferential anodic area for local corrosive dissolution. This results in geometric corrosion defects leading to corrosion fatigue crack initiation [36,37,13]. The stress intensity will become high enough to initiate a crack in the metal [38,14,8]. An important requirement for this crack initiation scenario is that the material, in the given aqueous environment, must be in the passive domain without any cyclic loading. An efficient way to monitor passive film failure is to perform in-situ measurements of the free potential. Previous studies have shown that a sudden drop in the potential occurs when the mechanical cyclic loading (fatigue) is applied to the material [11]. When considering the CF processes on stainless steel, it should be noticed that both characteristic times of the cyclic loading and passivation processes influence the overall mechanical-corrosion synergetic effects. Indeed, if the passivation process occurs at a higher rate than the mechanically induced passive film damage, no CF effect is expected. On the contrary if the passivation process is too slow, localized dissolution process can take place leading to fatigue crack initiation.

In this study we investigated the effect of aqueous corrosion (0.1 M NaCl + 0.044 M Na<sub>2</sub>SO<sub>4</sub>) on the CFCI mechanism of a martensitic stainless steel X12CrNiMoV12-3 used for aeronautical applications. An analytical CFCI model is proposed considering the coupling between corrosion parameters and cyclic loading in the HCF regime.

## 2. Material and methods

### 2.1. Material

The material used in this study is a martensitic stainless steel X12CrNiMoV12-3 used in aeronautical structures. The chemical composition of this alloy, in weight percent, is given as follows: 0.12C, 12Cr, 3Ni, 1.6Mo, 0.3V and Fe balance. Microstructural investigations showed a typical equiaxed martensite lath structure with residual austenite (less than 1% in the volume). The martensite lath dimension is around 2–3 μm. Prior austenite average grain dimension is around 100 μm. The 0.2% offset yield strength, and elongation to failure of the

alloy are 1040 MPa and 15% respectively.

### 2.2. Experimental methods

For crack nucleation tests, 8 mm cylindrical specimens were machined from rolled bar and mechanically polished to obtain a surface finish with arithmetic roughness of about 0.1 μm. Uniaxial fatigue tests were performed under load control, using a resonant electromagnetic fatigue testing machine at 120 Hz (Vibrophore type). This frequency was chosen so that failure between 10<sup>5</sup> and 10<sup>7</sup> cycles could be reached in a reasonable testing time. Moreover, this loading frequency is typical for HCF test, even if it is higher than what is usually used for corrosion fatigue studies [32]. The stop criterion was a loading frequency drop of 0.7 Hz, corresponding to a technical fatigue crack with a typical surface length of 5 mm and a depth of 2 mm. In order to investigate a possible frequency effect, additional CF tests were performed at 10 Hz on a servo-hydraulic fatigue testing machine.

CF testing were carried out at room temperature (≈20 °C) under an aerated 0.1 M NaCl and 0.044 M Na<sub>2</sub>SO<sub>4</sub> aqueous solution (A<sub>s</sub>) (with pH ≈ 6 and [O<sub>2</sub>] ≈ 7 ppm) at the free potential. This aqueous media was optimized to reproduce the same morphology of corrosion defect observed at the surface of real components using a thin-layer cell [39]. The addition of sulphate limited the increase of the anodic potentials with the inhibitive effect of SO<sub>4</sub><sup>2-</sup> anion on pitting corrosion [17,40]. To perform corrosion fatigue tests in the passive domain of the tested material, the (A<sub>s</sub>) aqueous solution was selected. In this case, the prospective depassivation and pitting of the material under cyclic loading (fatigue test) is stress-assisted mechanisms. An electrochemical corrosion cell was developed for corrosion fatigue tests. This cell allows *in-situ* fatigue testing in an aqueous corrosive environment and include three electrodes: a saturated calomel electrode (SCE) as the reference maintained at a constant distance (3 mm) from the specimen surface, a platinum counter electrode (CE) and the specimen as the working electrode (WE). Specimens were electrically insulated from the frame of the fatigue testing machine. The whole gauge section (area of interest) of the specimen was electrochemically monitored using a potentiostat device (Versastat4). During the CF tests, the free potential (OCP) value was monitored. Electrochemical impedance spectroscopy (EIS) analyses were carried out every 30 min (2.16 × 10<sup>5</sup> cycles at 120 Hz). Usual equivalent circuit modelling of the EIS data was used to extract physical properties of the electrochemical system by modelling the impedance data in terms of an equivalent electrical circuit. A solution resistance,  $R_s$ , in series with a constant phase element,  $CPE$ , in parallel with a charge-transfer resistance,  $R_{ct}$ , was considered, in particular to investigate the high frequency domain of electrochemical impedance measurements [39,41,42]. The high frequency range provides information on the passive film. The EIS investigation was performed in

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