# IN SITU CHARACTERIZATION OF LOW FREQUENCY CORROSION FATIGUE AT MESOSCOPIC AND MICROSCOPIC SCALES

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This study consists in analysing and formalizing the relationship between Stress Corrosion Cracking (SCC) and low frequency (< 0.1 Hz) Corrosion Fatigue (CF) of a 316 L type austenitic stainless steel in aqueous 30% $MgCl_2$ at 117°C, with an emphasis on both crack initiation and propagation. The effect of frequency on time of failure and crack growth rate is examined. This kinetic evaluation of the damage is completed by accurate morphological observations of cracking. An optical method called In Situ Surface Observation Technique (ISSOT) has been brought into focus for monitoring the damage in situ.

## INTRODUCTION

The stress corrosion cracking (SCC) and corrosion fatigue (CF) damages result from the combined effects of static, quasistatic or cyclic stresses and a deletorious environment. The main two stages of these damages are called initiation and propagation. They must be treated experimentally under morphological and kinetic aspects and at the various scales; microscopic, mesoscopic and macroscopic. This is necessary for the understanding and the quantification of the mechanisms involved.

It is generally agreed that fatigue cracks initiate preferentially or near surface discontinuities such as: non metallic particles (1, 2), twin boundaries and slip steps (1), brittle phases, precipitates, pits (2,3), grain boundaries (4). In corrosion fatigue, a critical pit size criterion has been used by several authors. They applied elasto-plastic fracture mechanic in terms of stress intensity factor threshold considering short or long crack concept (5, 6, 7).

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Principal issues to be addressed in order to quantify experimentally initiation process and to propose a criterion through an initiation law are : Evaluation of the morphological features of the initiation site; Identification of its history (from initial surface state to the growth of surface flaw); Localization of the critical site on the material microstructure; Measure of the critical size and of the time at the transition surface defect/microcracking; Quantitative evaluation of the plasticity around the initiation site. An In Situ Surface Observation Technique (ISSOT) has been proposed to obtain such a quantitative informations on crack initiation (8).

The interface between SCC and CF has been characterized from fractographic investigation. The various crack growth modes indicate clearly the propagation is either more time dependent or more cycle dependent. Typical SCC patterns are present on CF fracture surface for low frequency and low stress intensity factor. These patterns have been compared with the crystallography of transgranular SCC studied previously (9).

## Experimental

Corrosion-fatigue tests have been performed on austenitic stainless steel 316L in hot chloride solution ($MgCl_2$ 117°C). The alloy composition is in weight % : C:0.02, Cr:16.92, Ni:12.07, Mo:2.62, Si:0.39, Mn:1.68, S:0.018. The average grain size is about 100 mm. Cylindrical smooth specimens of 5 mm diameter and 15 mm gauge length are used. Triangular push-pull loading tests ($R = -1$, $\Delta\sigma/2=200$ MPa) have been carried out at frequencies ranging between $6\ 10^{-5}$ Hz and $5\ 10^{-2}$ Hz, the corresponding strain rates beeing respectively, $2\ 10^{-6}\ s^{-1}$ and $2\ 10^{-3}\ s^{-1}$.

## Results and discussion

### Initiation stage.

The morphological approach of the initiation consists in identifying surface flaws - the ones which lead to crack initiation and the ones which don't lead to crack initiation. One can distinguish the pre-existing defects inherent to surface preparation and those which are forming during CF testing in either pit form or localized dissolution along slip bands. On the system studied herein a single flaw process for crack initiation operates and the initiation site is always a corrosion pit.

The use of ISSOT has shown the crack initiation time is lower compared to the one usually measured by macroscopic metrology. The sizes of the surfaces defects resulting in crack initiation and the crack length "2a" have been measured by means of ISSOT. The crack length corresponding to a detected change in macroscopic behavior is about 850 µm. This represent the size of about 8 grains diameters of the polycrystal and 15% of the critical crack length for ductile fracture $a_f = 3$ mm. The in situ monitoring at a mesoscopic scale of the sample surface during a CF test at 1.5 $10^{-3}$ Hz showed that crack initiation occurs after 10 cycles, i.e. 2% of number of cycles at fracture Nr, whereas the variation of the elongation only intervenes at 60% of Nr. In addition, crack initiation corresponds to a 10 µm diameter critical flow at this frequency. As a matter of fact, the critical flaw size for crack initiation decreases strongly from 100 µm to few µm when the frequency increases from 6 $10^{-5}$ Hz to 5 $10^{-2}$ Hz. This indicates that crack initiation criterion is not only based on a critical stress concentration but also on the kinetics of initiation site growth and short crack propagation.

Propagation stage.

The kinetic and morphological aspects of propagation stage have been studied by using the ISSOT and fractographic observation of fracture surfaces. The figure n°3 shows the different modes of propagation that are observed according to the loading frequency and to the relative crack depth $a_r$ defined as the ratio crack depth-critical crack size for ductile rupture. For lower and intermediate values of $a_r$, the crystallographic transgranular mode typical of SCC is reduced with increasing frequency. The ductile transgranular mode which is typical of pure fatigue appears for a frequency between $10^{-4}$ Hz and 5. $10^{-3}$ Hz for high values of $a_r$. These observations clearly show the transition between "time dependent" and "cycle dependent" cracking as it is described in figure n°4. The domain called pseudo SCC correspond to crystallographic transgranular patterns with fatigue striations and a transition transgranular-intergranular. The physical processes of propagation are similar in this case to the pure SCC inferred from slow strain rate tests or constant load tests. In the corrosion fatigue domain, intergranular mode predominates and a transition intergranular-crystallographic transgranular appears. The pure fatigue domain is characterized by a transgranular ductile propagation with typical fatigue striations.

This representation frequency-relative crack length permit to define **Φ** as a fractional area of crack that is undergoing either pseudo SCC or CF or PF. **Φ** can be written as a function of frequency into the following form :

$$\Phi_{\text{p-SCC}} = [1 - H(f - f_1)] + [H(f - f_1) - H(f - f_2)]\,[-0.6 - 0.46 \log f] \quad \text{..........(1)}$$

$$\Phi_{CF} = [\,1.6 + 0.46 \,\text{Log}\, f\,][\,H(f - f_1) - H(f - f_2)\,] + [\,0.91 + 0.7 \,\text{Log}\, f\,][\,H(f - f_3) - H(f - f_2)] \quad \text{..................(2)}$$

$$\Phi_{PF} = [\,1.9 + 0.7 \,\mathrm{Log}\, f\,][\,H(f-f_2) - H(f-f_3)\,] + H(f-f_3) \quad (3)$$

With $0 \leq \Phi \leq 1$ , $\Phi_{p\text{-}SCC} + \Phi_{CF} + \Phi_{PF} = 1$ , $f_1 = 3\ 10^{-4}$ Hz ,

$f_2 = 2.\ 10^{-3}$ Hz , $f_3 = 5.\ 10^{-2}$ Hz and $H(x - x_0) = \left| \begin{array}{l} 0 \text{ for } x < x_0 \\ 1 \text{ for } x \geq x_0 \end{array} \right.$ .

Average crack growth rates associated to these different propagation processes have been measured. They vary from $10^{-8}$ m/s (f = 6.4 $10^{-5}$ Hz) to 4 $10^{-8}$ m/s (f = 5 $10^{-2}$ Hz), the crack extension per cycle decreasing respectively from $10^{-4}$ m/cycle to 2 $10^{-7}$ m/cycle. The crack propagation rates inferred from SCC tests are similar than the one obtain in CF for the lowest frequencies.

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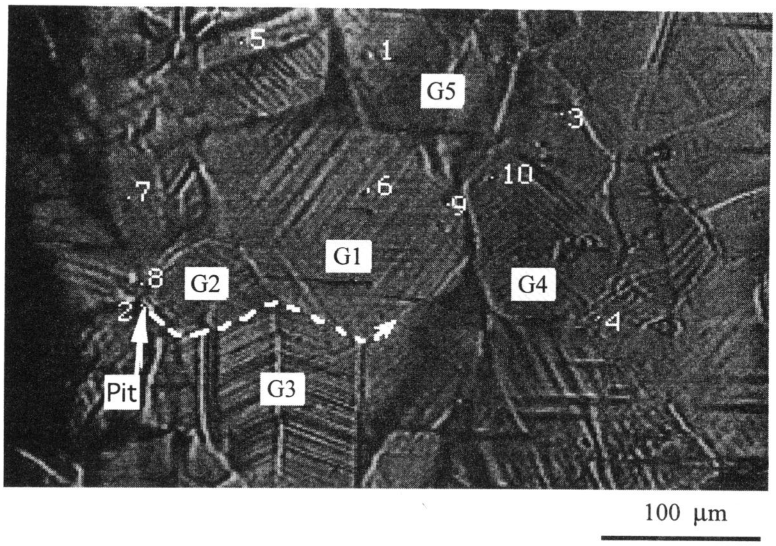
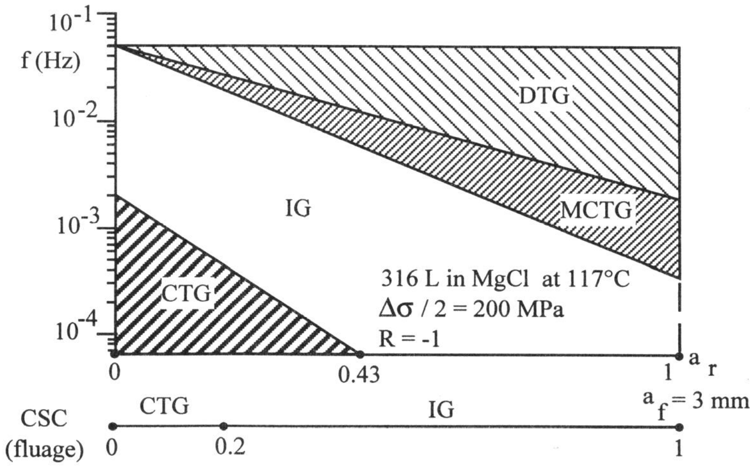
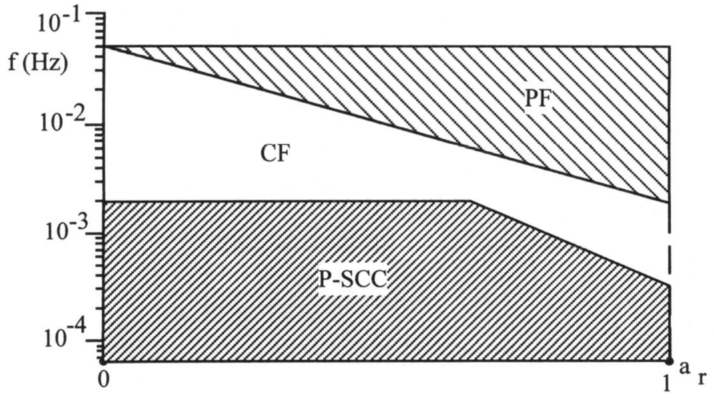


Figure 1 In situ monitoring of crack initiation and propagation during corrosion fatigue test (The crack path is indicated by the dotted line, see also ref. 8).



CTG : Crystallographic transgranular
IG : Intergranular
MCTG : Mixte crystallographic transgranular
DTG : Ductile transgranular

Figure 2 : Influence of frequency and relative crack size (ratio crack length-critical crack length for ductile fracture $a_f$= 3 mm) on crack propagation modes



316 L in $MgCl_2$ at 117°C
$\Delta\sigma / 2 = 200$ MPa
R = -1

P-SCC : Pseudo stress corrosion cracking
CF : Corrosion fatigue
PF : Pure fatigue

Figure 3 Illustration of the interface between stress corrosion cracking, corrosion fatigue and pure fatigue as a function of frequency and crack size