Micro mechanical study of the stress corrosion cracking of the 316L austenitic stainless steel in chloride medium

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Abstract :
Because of the synergetic actions of a corrosive medium and mechanical loading which occur in stress corrosion cracking, modelling this kind of damage on actual industrial structures can be very challenging. However, when studying one single couple material / medium, it is possible to only consider the mechanical aspect of damaging. Establishing this sort of purely mechanical simulation implies to have extensive information on the behaviour of the propagating cracks. Several types of tests are commonly used to gather experimental data on crack propagation, among which are tests conducted on fracture mechanics sample (CT, WOL…) and slow strain rate tests (SSRT) on smooth tensile specimens. The first category of tests is mainly used to obtain the propagation rate of long cracks and the so-called K_{ISCC}, the mode I stress intensity factor below which no propagation can be detected for constant displacement tests. The SSRT are preferred to study initiation, mechanisms and short cracks propagation (below K_{ISCC}). However, as the analyses of the cracking of these tensile tests are essentially statistical, it is somewhat difficult to separate the behaviour of a single short crack from mechanisms resulting from cracks interaction. For instance, for alloy 600 in primary water [1][2], the transition of crack growth rate from a slow to a fast regime for cracks attaining the K_{ISCC} can be interpreted as a mechanism existing for the single crack [3] or as a result of a “natural selection” within the cracks population.
In order to obtain experimental data on the propagation of one single short crack, we have developed SSRT on micro-notched tensile specimens. This new kind of sample allows the initiation and the propagation of one single crack at notch’s tip. The experiments were conducted for an austenitic stainless steel (316L) in a model chloride medium (30% $MgCl_2$ at 117°C). The use of interrupted tests permits us to obtain crack propagation rate in function of the global loading. Modelling these tests, as well as WOL tests, with the finite elements method gives us access to the local mechanical parameters, as strain or strain rate, needed in global stress corrosion cracking simulations. Besides, the predominance of crack branching in SSRT compared to WOL tests has been investigated. The use of EBSD (electron back scattering diffraction) showed the roles of twin and grain boundaries in branching, as well as the confined plasticity on the crack path, revealed by the creation of subgrains boundaries.

Keywords : stress corrosion cracking, austenitic stainless steel, crack growth rate, branching, EBSD.

Introduction

Modelling the Stress Corrosion Cracking (SCC) of engineering systems is a complex task, because of the multiple interactions between various parameters (electrochemical, mechanical, metallurgical…). Several models were proposed to describe and predict SCC damage. These models often describe elementary mechanisms at the microscopic scale

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(dislocation, surface film…) and are not well adapted for macroscopic scale simulations. One of the first developed model, Ford 'slip dissolution model' [4], proposes a crack propagation rate as a function of crack tip strain rate and metal dissolution and re-passivation kinetics. This model has shown to have predicting capabilities, provided it was previously fitted to experimental results, but does not render some characteristic features of SCC cracks, such as propagation discontinuities, branching or brittle aspect. The purpose of this present study is to bring some data to establish a macroscopic mechanical model, taking account specific features of SCC in our model material/environment system.

Tests used to collect experimental data on crack propagation fall in two categories.

- Tests performed on fracture mechanics samples (Compact Tension: CT, Wedge Opening Load: WOL…) give information on mechanical parameters affecting crack propagation rate but make use of fatigue pre-cracked specimen, thus considering initially long straight cracks. Linear fracture mechanics is used to analyse these tests, leading to the concept of K_{ISCC}, the applied Mode I Stress Intensity Factor K_I below which stress corrosion cracking does not occur.
- Slow Strain Rate Tensile (SSRT) tests on smooth samples allow the study of SC cracks from initiation to final sample fracture. By mean of statistical techniques [5], it is possible to derive crack propagation rates, but these results apply to a whole population of small cracks, and it is difficult to separate the role of crack interactions in the overall propagation rate .

For this study, we have used micro-notched SSRT specimens to study the initiation and propagation of isolated small cracks. These tests were compared to conventional tests performed on WOL samples. Experimental data are analysed from a mechanical point of view and the role of strain rate or the use of K_I in analysing SSRT tests, are discussed.

Experimental and results.

<u>Material and methods</u>

Tests were conducted on standard AISI 316L stainless steel (table 1), re-austenitized prior to experiments (1050°C for 1h and water quenched). The average grain size is 80 µm. Some delta ferrite remains after the heat treatment (about 2.5% volume fraction). The corrosive medium is a 30 wt% $MgCl_2$ boiling solution (117°C). All tests were carried at free potential.

Two kind of mechanical tests were conducted:

- Slow Strain Rate Tensile tests (SSRT) on micro-notched specimen. These notches are machined with a diamond wire saw, which produces notches with a 40 µm root radius without significant surface strain hardening. Several notch lengths from 50 to 300 µm are used. Tests are carried out at an "apparent" strain rate of 2.5×10^{-7} s^{-1} and interrupted at approximately 2% elongation (about 250 MPa) taken on the conventional stress-elongation diagram (fig. 1).
- Constant displacement tests on WOL samples. The specimens are fatigue pre-cracked and wedge-loaded to have an applied K_I=13.5 MPa√m. Samples are then placed in the chloride medium. K_{ISCC} is calculated when crack propagation has stopped under constant opening.

Table 1: Chemical composition (wt%) of AISI 316L.

C	Mn	P	S	Si	Ni	Cr	Mo	N
0.03	2	0.045	0.03	1	10-14	16-18	2-3	0.10-0.30

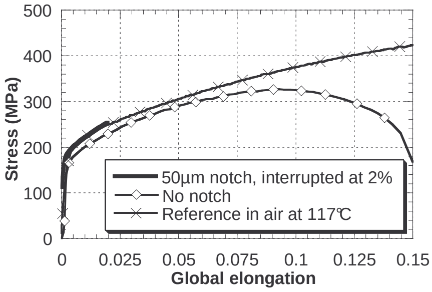


Figure 1.a: stress - elongation diagram of the SSRT specimens

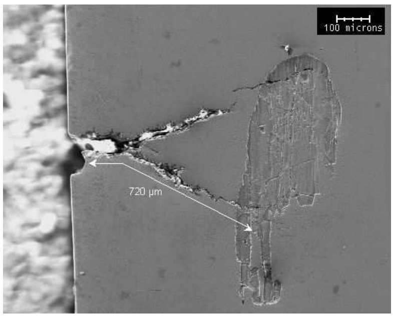
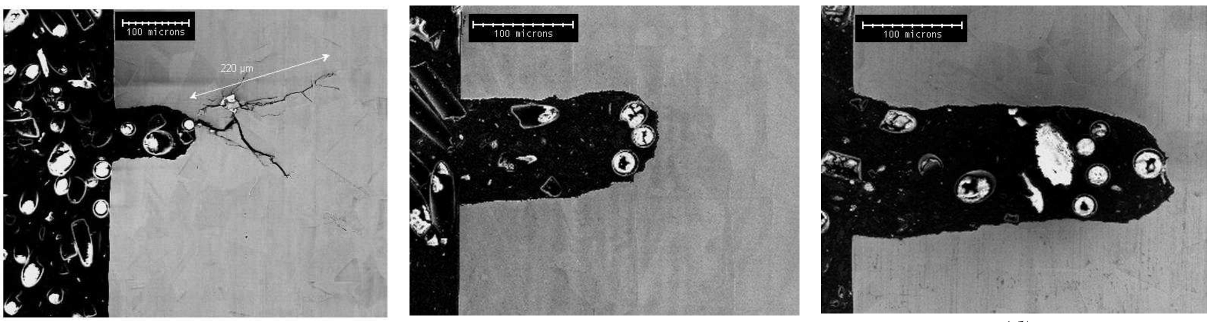


Figure 1.b: 50 µm – notched specimen after interrupted SSRT test.

<u>Results: initiation and propagation.</u>

Micro-notches are used as strain and stress concentrators and thus promote the initiation and propagation of isolated cracks at notch's root. Besides, they allow to control local mechanical parameters as strain rate or stress by adjusting their depth, without changing the duration of the interrupted test. This allows some parametric studies of the influence of the local mechanical parameters on crack initiation and propagation.

The size of cracks strongly depends on the size of the notch. Longest crack are obtained for shortest notches (50 µm) with an average length of 700 µm. For medium notches (125 µm), cracks length is about 230 µm. No crack initiations could be observed for longest notches (200 and 300 µm). Considering that initiation occurs at yielding (175 MPa), average crack propagation rate is thus of $2x10^{-9}$ $m.s^{-1}$ for 125 µm notches and $1.0x10^{-8}$ $m.s^{-1}$ for 50 µm notches. It should be noted that the cracks on SSRT tests are strongly branched. Figure 2 shows the most significant cracks profiles.



(b) *(c)* *(d)*

Figure 2: Notched specimens after interrupted SSRT test (a): 125 µm notch (b) 200 µm notch (d) 300 µm notch

Tests performed on WOL samples have given a K_{ISCC} value of about 11MPa√m. This value was obtained after 48h, and gives us a crack growth rate of $1.7x10^{-9}$ $m.s^{-1}$.No significant crack branching was observed.

<u>Determination of the Stress Intensity Factors.</u>

SSRT tests were first analysed by using linear elastic fracture mechanics, to evaluate mode I Stress Intensity Factors (SIF) applied to the experimental cracks. SIF were determined numerically by Finite Element Method (FEM) analysis to properly account for sample geometries. The defect was modelled as a notch with a straight crack normal to the tensile direction. Simulations were carried out for a linear elastic behaviour (Young's modulus: 190 GPa), in 2D plane strain.

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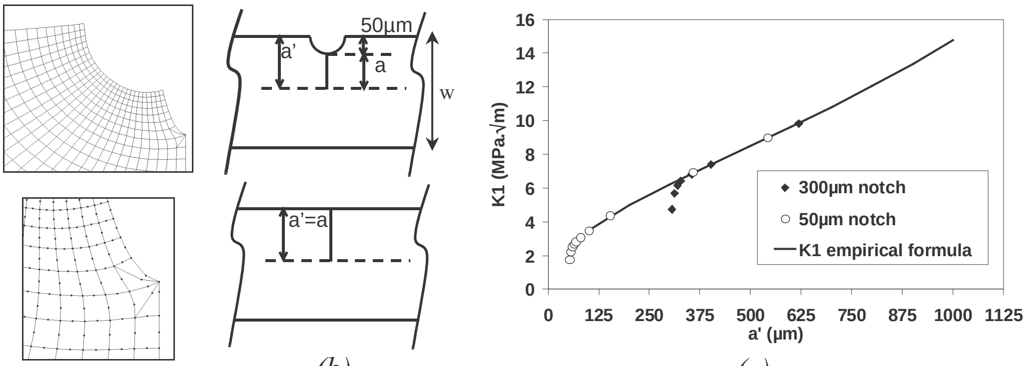
Calculations show that in linear elasticity, a straight crack emerging from a notch has the same SIF as a straight crack of the same total length (notch + crack). The influence of the notch becomes negligible 20 µm ahead of its root (fig. 3). SIF for a straight crack in this configuration can be calculated with empirical formulae, such as Equation 1 [6]. This configuration offers the possibility to control the applied SIF in SSRT test by choosing the notch length.



(a) (b) (c)

Figure 3 : (a) Geometry and crack tip mesh used to compute SIF (b) Geometry of the defect (notch + crack) (c) Effect of the notch on the value of K_I, linear elasticity, for 175 MPa.

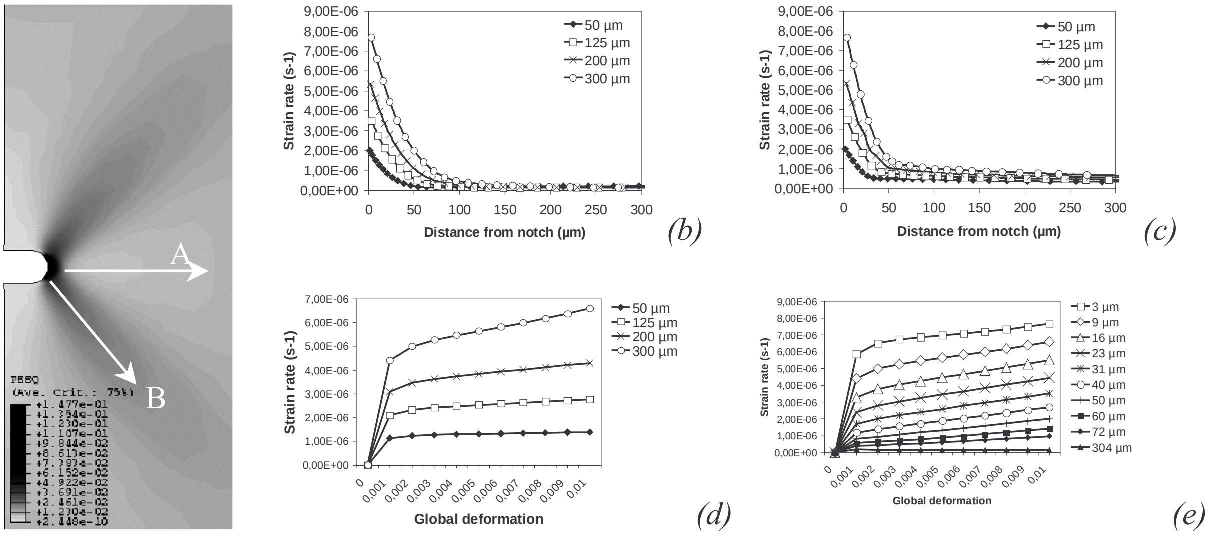
$$K_I = \left(1.99 - 0.41\frac{a}{w} + 18.7\frac{a^2}{w^2} - 38.48\frac{a^3}{w^3} + 53.85\frac{a^4}{w^4}\right)\sigma\sqrt{a}$$

Equation 1: Empirical determination of K_I (Gross (1964) and Brown (1966))

The determination of experimental values of K_I for interrupted SSRT tests were done on a simplified geometry, considering a straight crack. The lengths chosen to calculate the SIF were measured for the deepest fracture's branches normal to the tensile direction (pure mode I loading), with an applied load of 250 MPa. The K_I values were about 16 $MPa.m^{1/2}$ for the 50 µm – notched specimen and 9.5 $MPa.m^{1/2}$ for the 125 µm – notched sample. A more accurate calculation has been carried out taking crack branching into account, for a 250 µm branching crack propagating from a 100µm deep notch (fig 6). The K_I value for an applied load of 250 MPa is about 7 $MPa.m^{1/2}$. This shows the effect of branching on stress relaxation. These results clearly show that propagation occurs in SSRT tests below the measured K_{ISCC}.

FEM simulation and analysis

FEM simulations were carried with a plastic deformation law to determine the effect of strain hardening on crack initiation and propagation. The elasto-plastic models used are time-independent, time being introduced in our simulations by assigning to the simulated total elongation, a numerical value corresponding to the experiment total duration.

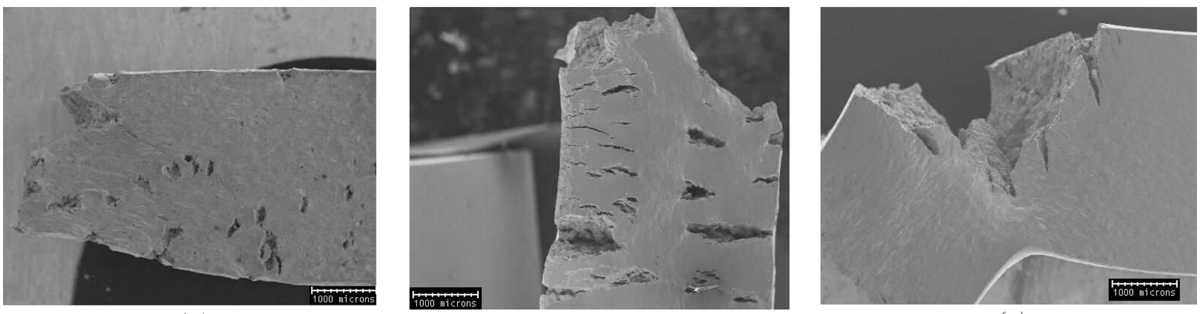


(a)

Figure 4: (a) Equivalent strain concentration at a 125 µm notch. 1% global elongation.
- Strain rate versus distance from notch, for several notch lengths calculated for 1% of total elongation and 2.5x10^{-7} s^{-1} elongation rate (b) in the A direction (c) in the B direction
- Strain rate versus total elongation for 2.5x10^{-7} s^{-1} total elongation rate (d) as a function of the notch length, 10 µm away from the tip in direction A (e) as a function of the distance from a 300 µm deep notch in direction A

<u>Local strain rate</u>

The influence of notch length on local strain rate for SSRT tests was investigated. A classical Ramberg-Osgood deformation law was used and significant precision on strain rate was obtained by imposing short time increments (0.01% of total elongation). The results are given for a total elongation rate of 2.5 x 10^{-7} s^{-1}. Figure 4 (b) and (c) show the evolution of the local strain rate with the distance from the root. A sharp strain rate gradient is present at the notch tip, with the local strain rate asymptotically reaching the remote applied strain rate. The extent of the zone of high strain rate gradient roughly corresponds to the grain size of our material. The local strain rate rapidly increases during the first 0.2% of total elongation and then increases only slightly, depending of the notch length (fig. 4 (d) and (e)).
These results should be put in regard to those obtained with SSRT tests on smooth samples for different global strain rate (fig. 5), showing SCC strain rate sensitivity. The higher the global strain rate is, the most numerous the cracks are, but the less profound they are. The strain rate at the longest crack seems to be to high to permit the initiation of a crack.



(a) (b) (c)

Figure 5 : Broken smooth SSRT samples at different global strain rate (a) 10^{-5} s^{-1} (b) 10^{-6} s^{-1} (c) 10^{-7} s^{-1}

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Crack propagation

Crack propagation was simulated by debonding nodes of the model's mesh along a predefined crack path. The crack propagation rate was here considered as constant, to compare WOL and SSRT test on the same basis. The crack rate has been chosen equal to $1.7x10^{-9}$ $m.s^{-1}$ corresponding to WOL and more or less to 125 µm notch SSRT test crack average velocity. The deformation law is an elasto-plastic law with incremental plasticity, to take account the relaxation after mesh debonding.

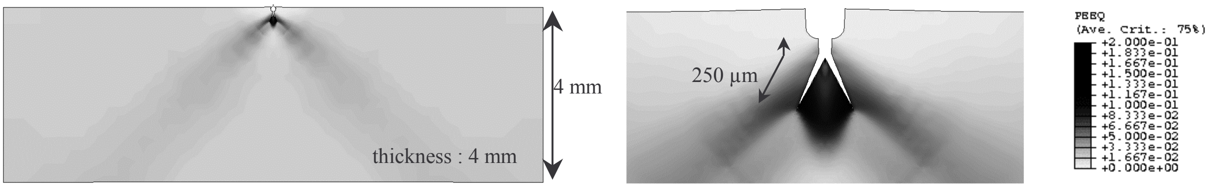

Figure 6: Plastic zone created by a propagated branched crack in a 125µm notched SSRT sample (after 40h at $2.5x10^{-7}$ s^{-1} , constant propagation rate of $1.7x10^{-9}$ $m.s^{-1}$)

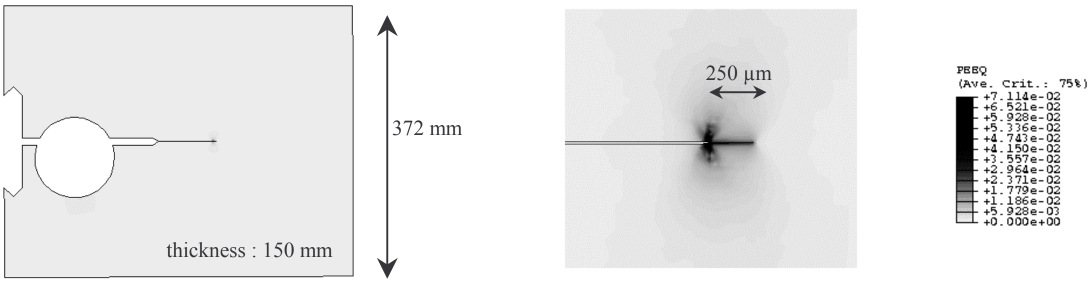

Figure 7: Plastic zone created by a propagated straight crack in a WOL15 sample (after 40h, intial applied K_I=13.5MPa√m, constant propagation rate of $1.7x10^{-9}$ $m.s^{-1}$)

It first appears that deformation values are much higher for SSRT test (fig. 6). Moreover, for SSRT test, the extent of the plastic zone during propagation is not negligible at all compared to the sample dimensions, as it is for WOL tests (fig 7). For WOL tests, we are in the case of confined plasticity, and using linear fracture mechanics makes sense. For SSRT, the extension of plastic zone invalidates the concept of K_I. It does not describe properly the stress field at crack tip and calculated values make little sense. Thus, K_I should not be used for comparing tests on fracture mechanics samples (as WOL samples) to those on tensile tests, or even for comparing different kind of tensile tests, or any mechanical test producing significant strain hardening.

Deformations at crack tip are much higher in SSRT test than in WOL test (fig. 8). After cracking, values do not change for SSRT tests and only slightly increase for WOL test. However, the amount of deformation at the crack locus constantly rises with crack advance for SSRT test, whereas it tends to decrease for WOL test. This diminution of the strain at the moving crack tip could explain the crack arrest for WOL tests. The continuously increasing straining, as well as the extent of the plastic zone in SSRT tests seems to be the reason of the branching.

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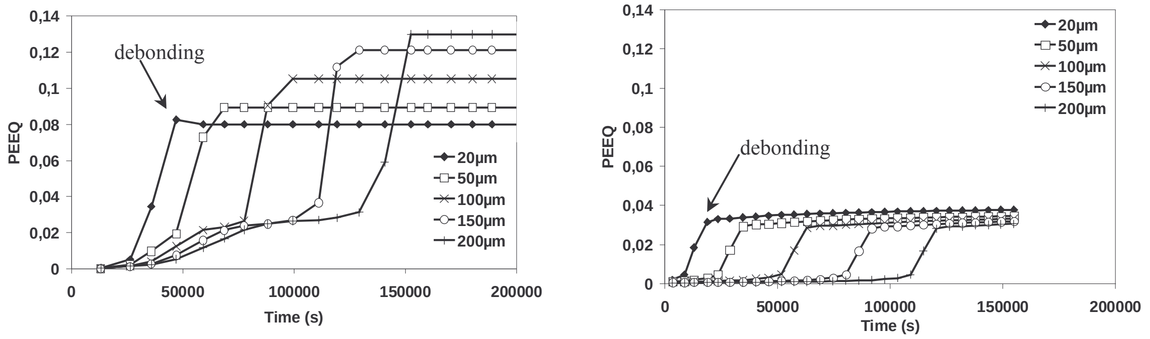
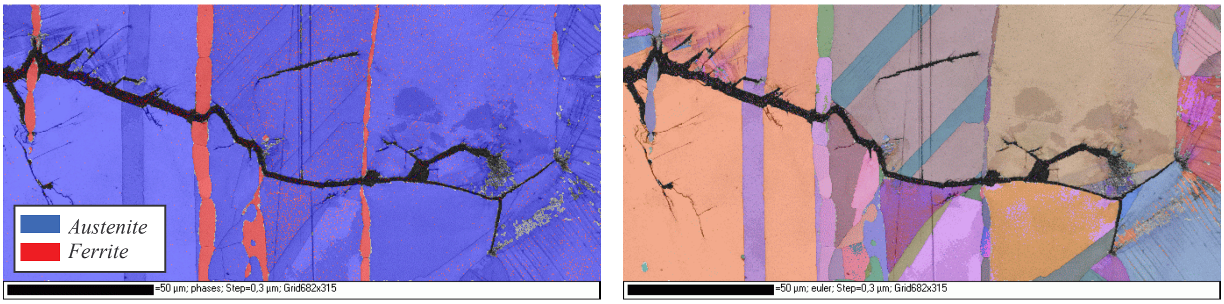


(a) *(b)*

Figure 8: Equivalent plasticity for several points on crack's path during propagation (a) 125µm notched sample, branching crack (b) WOL sample. Distance are taken from the notch root for SSRT sample and from fatigue crack tip for WOL sample.

EBSD analysis

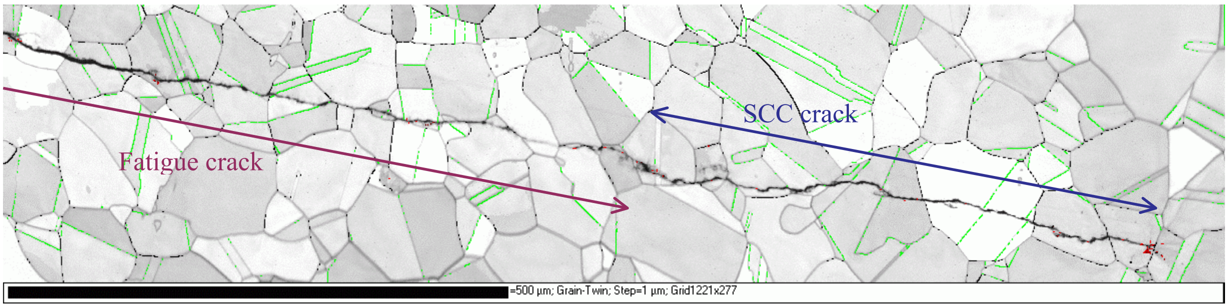
EBSD has recently been evoked as a mean of investigation of SCC crack, essentially for intergranular corrosion or cracking [7][8]. The capabilities of the technique as a mean or revealing plastic deformation, especially around propagated crack, were proofed [9][10]. The technique has given us the opportunity to analyse plastic deformation around our transgranular SCC cracks, with a spatial resolution down to 50 nm using a Field Emission Gun SEM.



(a) *(b)*

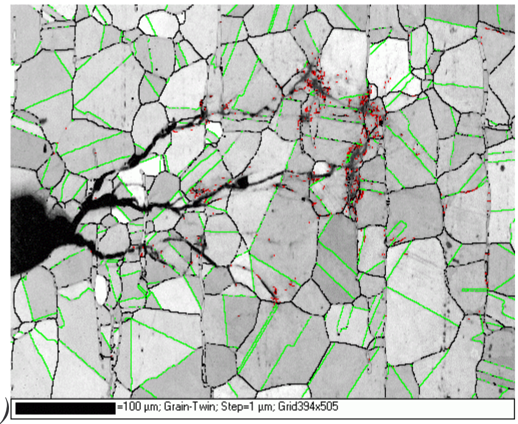
Figure 9: EBSD map of a branch of SCC crack from SSRT sample (interrupted 2% global elongation). (a) Phases (b) Grains coloured according to their orientation.

We have analysed several cracks profiles to reveal the material microstructure and to understand its role on the propagation and branching of the crack. Plastic deformation heterogeneities was evidenced by plotting misorientations boundaries between 2° and 12° (from the limit of background measurement noise to the value defining a grain boundary).



(a)

Grain boundary (>12°)
Misorientation boundary (2° - 12°)
Twin boundary



(b)

Figure 10 : Comparison between WOL (a) and SSRT (b) tests propagated cracks.

It first comes out that grain and twin boundaries play an important role on the crack behaviour. The propagation through a grain is more or less straight and bifurcations occur at boundaries. Even if branching can appear inside a grain, it preferentially takes place at grain boundaries (fig. 11). Besides, despite the fact that the fracture mode is essentially transgranular in this system, intergranular cracking can appear and promote further branching (fig 11).

Some residual ferrite is present in our material, with an average amount of 2.5%. Because of this small quantity, this delta ferrite seems to have here only a minor influence on the propagation of the crack. Ferrite can causes some strain localization, because of deformations incompatibilities with the austenitic matrix but cracks eventually propagate through it with little influence other than bifurcation or branching as a regular grain boundary (fig. 9 & 11).

Finally, the EBSD technique allows to compare the cracking of SSRT and WOL samples from a crystallographic point of view. It appears that plasticity around cracks is more intense in SSRT compared to WOL tests, as revealed by the density of misorientation boundaries. This higher straining seems to be one of the major reason of the predominance of branching in SSRT: the constantly rising deformation of the sample allows the activation of several slip systems at the edge or the tip of the crack, leading eventually to the creation of multiples cracking paths. In this material where the stacking fault energy is low, this deformation can even produce twining at crack tips (fig. 11). The amount of deformation in the two kind of samples can be correlated to the FEM simulations already presented (fig 6 & 7).

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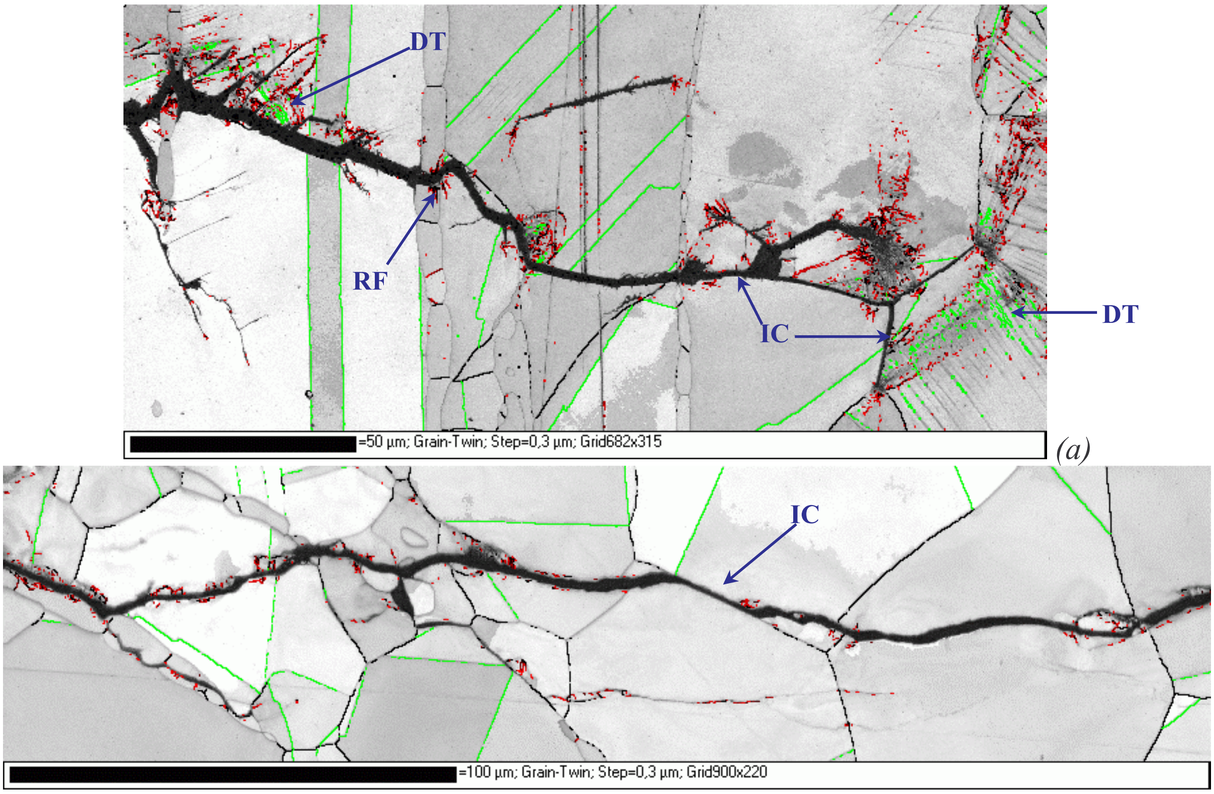

(b)

Figure 11: Comparison between propagated cracks in WOL (a) and SSRT (b) tests. DT: Deformation Twin, RF: residual Ferrite, IC: Intergranular Cracking

Discussion

The comparison of the two kinds of tests shows the influence of the mechanical loading on cracking behaviour. The difference of straining around crack as calculated by FEM and measured with EBSD demonstrates that these tests cannot be compared on the basis of linear elastic fracture mechanics. The concept of K_I is irrelevant for highly strained tests, as SSRT tests, but still valid for fracture mechanics tests. The high deformation in SSRT samples seems to be the major reason of branching.

The strain rate plays a major role in SCC of this material/environment system. The upper limit of strain rate for SCC can be determined from testing smooth SSRT samples, but may be interpreted in various ways, from time shortage for crack initiation, to blunting of initiated cracks or kinetics limits in terms of embrittlement mechanisms. Tests on micro-notched samples tends to prove that this strain rate limit exists in the mechanisms. The absence of initiation for long notch and the shorter crack for medium notch could be interpreted by the blunting of the initiated cracks. However we can interpret the low difference of propagation rate between WOL and SSRT tests from a mechanistic point of view, and consider that it exists a upper limit in propagation rate, due to embrittlement kinetics. As strain rate is high enough to sustain the propagation of multiple cracks, branching occurs.

Finally, observing the strong influence of microstructure on propagation, one could discuss the validity of isotropic FEM simulations. It is so important to recall that these simulations represent the average behaviour of SCC cracks. Moreover, crystal plasticity simulations would have here little practical interest and are much more time an resource consuming.

Conclusion

Mechanical studies of SCC have to go further than standard linear fracture mechanics. The exclusive use of concept of SIF tends to prevent relevant comparisons between different and complementary mechanical tests. Moreover, besides being inappropriate in general plasticity, such as in SSRT tests, the concept of K_{ISCC} is challenged even in fracture mechanics tests [11]. Our present studies focus on non linear fracture mechanics, especially energetic concepts such as contour integrals, and on the influence of local parameters such as the strain rate at the crack tip.

EBSD has proven to be a valuable tool in studying SCC. The technique is still under development for precise strain characterisation, but already offers some information on the influence of the microstructure. By offering a great spatial range of analysis at a good resolution, the method is complementary to high resolution techniques, such as TEM.

Finally, besides the objective to obtain propagation law intended to be incorporated in larger scale simulations, this kind of study could provide critical assessment of mechanistic SCC models at the single crystal or dislocation scale.

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