STRESS CORROSION CRACKING INVESTIGATION IN ZIRCALOY-4 USING DIGITAL IMAGE CORRELATION

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Summary Stress corrosion cracking is a synergetic process between environmental effects and mechanical solicitations. A model system is studied: pre-cracked and pre-oxidized Zircaloy-4 samples are submitted to a mechanical tensile load in halide aqueous media. An innovative Digital Image Correlation technique is developed and performed while the test is running, in order to measure the evolution of the crack length and the stress intensity factors, but also to control the stress intensity factor during the crack propagation. The results suggest that once a critical anodic polarization is prescribed, the crack growth rate depends on the stress intensity factor but also on its time derivative. Indeed, a threshold effect is obtained on both the stress intensity factor, meaning that plasticity must increase for the dissolution reaction to occur, and its rate, meaning that time for plasticity to produce new dislocations must not exceed the characteristic duration of the repassivation process. A phenomenological crack propagation model is proposed and identified from the experimental measurements.

INTRODUCTION

Stress Corrosion Cracking (SCC) is a synergetic issue resulting from the combination of both corrosion process and mechanical damage. Mechano-electrochemical interaction quantification is fundamental to understand the mechanisms involved in the SCC process. Although zirconium alloys are prone to passivation which protects them from corrosion, Farina et al [1] have shown that anodic polarization enhances SCC of Zircaloy-4 in aqueous halide media. On a phenomenological point of view, SCC of zirconium alloys in aqueous halide solutions is characterized by two different cracking modes [2]: intergranular and transgranular. On the one hand the intergranular propagation is generally associated with dissolution processes and takes place just after the initiation of the cracks. On the other hand, transgranular propagation occurs for a given stress intensity factor magnitude (K_I). In that context, the main objective of the present study is to combine local electro-chemical and mechanical measurements to estimate the crack tip coupling between chemical and fracture mechanics and to suggest a crack propagation model identification.

EXPERIMENTAL REQUIREMENTS TO PERFORM SCC TESTS

Mechanical localization of the SCC damage is obtained using fatigue pre-cracking of the specimens. To avoid too small crack initiation, the crack length has to be produced with a good accuracy. In the present case, 2.5 mm long fatigue pre-cracks are performed in 8 mm wide samples. A specific experimental procedure is performed to limit the development of plasticity at crack tip during this stage. By combining real time Digital Image Correlation (DIC) K_I extraction and the *load-shedding* procedure [3] to ensure a "constant K_I" fatigue propagation, the Fracture Process Zone is controlled at crack tip. K_I and the crack tip position are extracted with the Williams' series formalism [4] [5]. The description of this method is given in paper [6]. This procedure enables to control the pre-crack residual stress intensity factor (K_I^f) which can have effects on SCC threshold.

Localization of the electrochemical damage at crack tip is performed using anodic polarization to produce local dissolution in this area. A preliminary potentio-dynamic investigation shows that the pitting potential of a thermo-oxidized surface (40 hours at $470°C$) of Zircaloy-4 is shifted by $+100mV$ in the anodic direction compared to the non oxidized surface. Consequently, an anodic polarization at the pitting potential of the non oxidized metal, on an oxidized specimen, is about to produce the local dissolution of the new bare surfaces created at crack tip due to dislocations emergence. In order to avoid any formation of undesirable pits on the fracture surface, pre-oxidation of the specimens is carried out after fatigue pre-cracking.

CRACK PROPAGATION MODEL IDENTIFICATION ON SCC TESTS

SCC tests are performed on fatigue pre-cracked and oxidized Zircaloy-4 samples. A specific cell is designed allowing in-situ electrochemical measurements and also image acquisition of the in-plane specimen surface, which can be used for DIC. De-aerated aqueous solutions of NaCl or KBr ($0.5M$) are used.

First of all constant load tensile tests reveals that the crack growths only if the solution is enough agressive to avoid repassivation which protect nude metal surface at crack tip (NaCl solution). On one hand, in that case where the stress intensity rate ($\dot{K}_I$) is under $0.02MP.\sqrt{m}.s^{-1}$, the crack propagation rates are found to be very low ($da/dt < 10^{-6}m.s^{-1}$). On the other hand, Slow Strain Rate Tensile Test (SSRT) are performed for different pre-crack samples on both NaCl or KBr

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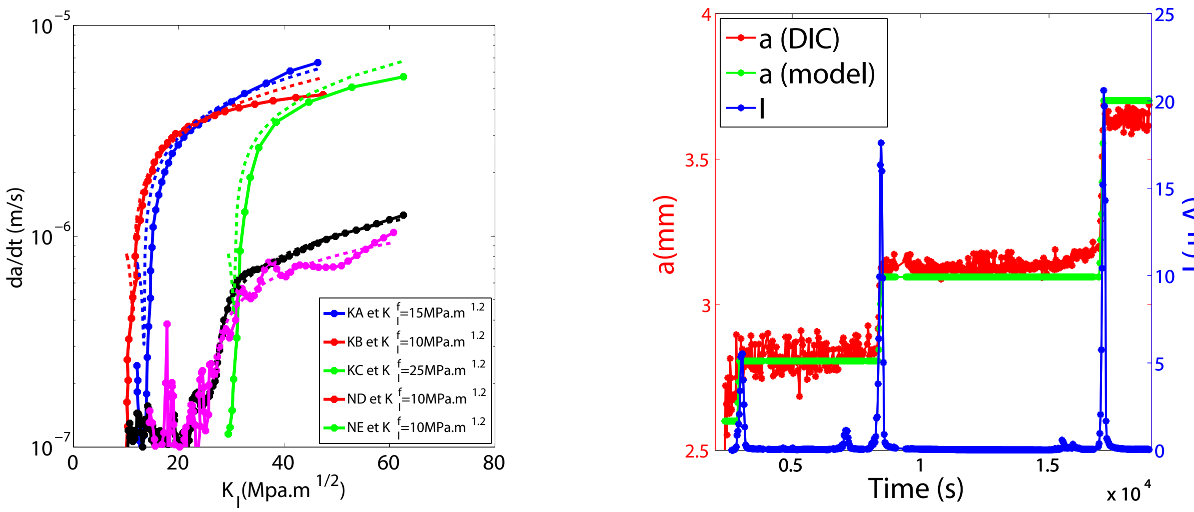
aqueous solutions and show quite high crack velocities ($da/dt > 10^{-6} m.s^{-1}$) with $\dot{K}_I > 0.02MP.\sqrt{m}.s^{-1}$. Two different crack propagation stages with two different crack growth rates are evidenced. The second stage appears above a given stress intensity factor magnitude (K_I^s). A phenomenogical propagation model is suggested with a power law dependence on the difference between K_I and a stress intensity magnitude threshold K_I^s :

$$\begin{cases} \frac{da}{dt} = \alpha_m \left({K_I}^2 - {K_I^s}^2 \right)^{1/n_m} & \text{if} \quad K_I \geq K_I^s \quad \text{and} \quad \dot{K}_I \geq \dot{K}_I^s \\ \frac{da}{dt} = 0 & \text{if} \quad K_I \leq K_I^s \quad \text{and} \quad \dot{K}_I \leq \dot{K}_I^s \end{cases} \tag{1}$$

This crack propagation model gives two thresholds which corresponds to the stress intensity magnitude and rate (K_I^s and $\dot{K}_I^s$). K_I^s can be easily identified as the vertical asymptotic value of the da/dt vs K_I curves given by Figure 1 (a). Into NaCl solution, this threshold is reproductible whereas in KBr solution, K_I^s values are more variable and seems to correspond to K_I^f (fatigue pre-crack stress intensity factor magnitude). Thus in this solution, K_I^s is lower and depends on the loading history because the crack velocity is enhanced once K_I overtakes the previous maximal stress intensity magnitude reached.

A specific SCC test is thus performed to validate this model. Using Load-shedding procedure developed to apply different given K_I magnitudes, the loading history dependance can be identified because the crack length estimation can be predicted by the model (equation 1) is verified. The figure 1 (b) shows how the crack growth is enhanced with this loading path and how the two crack length predictions (experimental and predicted) are in good agreeements. Besides, electrochemical dissolution current measurement shows peaks as soon as the crack propagates. Additionnaly, we can estimate with electrochemical analytical estimation that dissolution is a governing parameter.



(a) Identification on SSRT tests

(b) Validation on a specific SCC test

Figure 1. Crack propagation model identification and validation

CONCLUSIONS

The present work shows that a SCC phenomenological crack propagation model has been identified in the case of the Zircaloy-4 in aqueous halide solution. A threshold effect is obtained on both the stress intensity factor, meaning that plasticity must increase for the dissolution reaction to occur, and its rate, meaning that time for plasticity to produce new dislocations must not exceed the characteristic duration of the repassivation process.

References

[1] SB Farina, GS Duffo, and JR Galvele. Stress corrosion cracking of zirconium and Zircaloy-4 in halide aqueous solutions. *Corrosion Science*, 45(11):2497–2512, 2003.

[2] SB Farina and GS Duffó. Intergranular to transgranular transition in the stress corrosion cracking of Zircaloy-4. *Corrosion Science*, 46(9):2255–2264, 2004.

[3] ASTM. E647-95, in “1995 ASTM Annual Book of Standards,”. *ASTM*, 3:578, 1995.

[4] M. Williams. On the stress distribution at the base of a stationary crack. *ASME Journal of Applied Mechanics*, 24:109–114, 1957.

[5] S. Roux, J. Réthoré, and F. Hild. Digital image correlation and fracture. *Journal of Physics D: Applied Physics*, 42:214004–214024, 2009.

[6] E. Durif, J. Réthoré, A. Combescure, M. Fregonese, and P. Chaudet. Controlling stress intensity factors during a fatigue crack propagation using digital image correlation and a load shedding procedure. *Experimental Mechanics*, pages 1–11, 2011.