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Fatigue crack propagation in gaseous hydrogen environment in low alloy steel

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Abstract

Fatigue crack propagation in low alloyed steel (3.5Ni-1.5Cr-0.5Mo-V) used for turbine generator of nuclear plant is studied under 4 bar hydrogen atmosphere in comparison to ambient air and high vacuum. Tests are conducted on CT specimens and the variation of the fatigue crack growth rate da/dN with respect to the amplitude of the applied stress intensity factor ΔK is explored in a wide range and especially in the near threshold domain. The propagation behaviour under hydrogen atmosphere is shown similar to that obtained in air in the low rate range, i.e. when the maximum of the stress intensity factor K_{max} is lower than a critical level of $16 \text{ MPam}^{1/2}$ with higher crack growth rate than in high vacuum. This environment effect is related to the presence of residual water vapour in both gases. For K_{max} higher than $16 \text{ MPam}^{1/2}$, much faster growth rates under hydrogen atmosphere in comparison to air and vacuum are observed and related to hydrogen assisted intergranular propagation combining fatigue and sustained loading damage. The results are discussed on the basis of micrographic observations supporting the involved mechanisms.

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Keywords: fatigue crack propagation; low carbon steel; martensitic stainless steel; hydrogen, environmentally assisted propagation; crack closure.

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1. Introduction

A large number of modern turbo generators are equipped with hydrogen cooling systems, hydrogen being used as a generator rotor cooling medium since it has a high heat conductivity as compared to that of many gases. However, despite the number of advantages, hydrogen is known to induce a degradation of the mechanical properties of metals and metallic alloys commonly designated as “hydrogen embrittlement” and especially in high-strength steels [1]. In a gaseous hydrogen environment, these steels have been shown to exhibit a decrease in ductility under monotonic loading [2] and enhanced fatigue crack growth rates under cyclic loading [3–5]. The involved mechanisms depend on various variables such as mechanical strength, microstructure, slip mode, stress-intensity factor, time and temperature, which can affect the growth rates, the crack path and the fracture surface appearance. In this context, the knowledge and the understanding of the fatigue crack propagation behavior and its incidence upon in-service components constitute a great deal especially for the structural integrity of nuclear plants. Then, the present research program is focused on the fatigue crack propagation under a 4 bar pressure of hydrogen in comparison to air and high vacuum. Crack closure is determined in order to evaluate the effective stress intensity factor range ΔK_{eff} considered as the driving force for fatigue crack propagation. The results are analyzed and discussed on the basis of SEM observations of fracture surfaces and of existing modelling.

2. Experimental

The generator rotor material is a quenched and tempered low martensitic alloy steel type 3.5Ni-1.5Cr-0.5Mo-V. The determination of the fatigue crack growth data relating the crack growth rate da/dN to ΔK , and of the threshold stress intensity factor range ΔK_{th} , is performed on CT40 (40mm width and 8 mm thick) specimens in accordance to the ASTM recommendations [6] under three different environments: (i) ambient air, (ii) high vacuum (5×10^{-4} Pa) and (iii) 4 bar purified hydrogen atmosphere containing some 60 ppm of residual water vapor (partial pressure of 24 Pa). Tests in air and in high vacuum are carried out at room temperature on a standard 10 kN hydraulic testing machine at 20 Hz under load control and at a load ratio $R=0.1$. Tests in hydrogen atmosphere are conducted at the same frequency and R ratio on a testing facility especially designed to operate under high-pressure gaseous environment [7]. The crack length is either measured with a traveling optical binocular microscope or by means of potential drop technique through a computerized system. For crack closure measurements, the compliance method is used as described in [8] with numerical data acquisition and processing. All the specimens are pre-cracked in laboratory air at $R=0.1$ and at a constant ΔK range of 13 MPa \sqrt{m} up to a length of 2 mm. The threshold tests are then performed using a load shedding procedure with load steps of 7%. The threshold is evaluated for a crack growth rate of 10^{-10} m/cycle. After reaching the threshold, the load is increased by steps of up to ΔK amplitude of 13 MPa \sqrt{m} ; then the load amplitude is kept constant up to the end of the test.

3. Results

3.1. High vacuum

Tests performed in high vacuum provide a reference for fatigue crack propagation in an inert environment which will help in the analysis of the specific effect of the gaseous environments. The variations of da/dN in high vacuum with respect to ΔK and ΔK_{eff} , are plotted in Fig. 1a in a log-log diagram. A progressive increase of da/dN with increasing ΔK is shown, the slope of the curve being at the same time progressively decreasing. A similar trend is observed for da/dN with respect to ΔK_{eff} after crack closure correction, the contribution of crack closure being only detected for ΔK range lower than 12 MPa $m^{1/2}$ with a progressive increase of the crack closure shielding effect when the threshold is approached. The closure correction results in the disappearance of the change of slope in the lower rate range and hence the absence of any well marked threshold knee as commonly observed in the near threshold domain in ambient air at low R ratio.

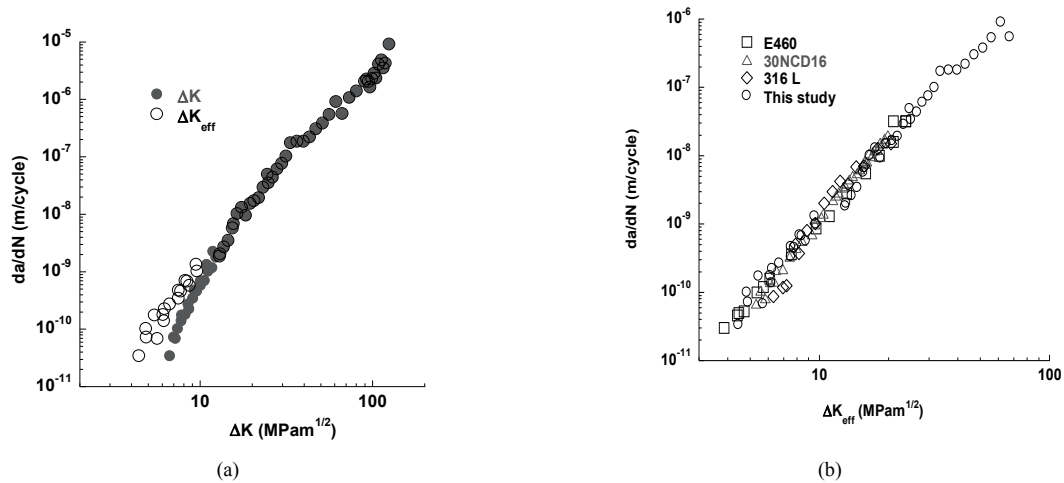


Fig. 1. (a) Fatigue crack propagation in high vacuum da/dN vs ΔK and ΔK_{eff} ; (b) comparison of intrinsic fatigue crack propagation in high vacuum for the studied steel and three high strength steels [9].

In Fig. 1b are plotted the effective crack propagation data in a da/dN vs ΔK_{eff} diagram for four different steels tested in comparable conditions, high vacuum of about 5×10^{-4} Pa, frequency of 20 to 35 Hz, load ratio $R=0.1$, for crack growth rates ranging below 10^{-7} m/cycle. All the data fall in a narrow scatter band along a straight line corresponding to a power law with an exponent close to 4 which is in accordance with a stage II intrinsic propagation well described by Petit et al [9].

3.2. Ambient air

The fatigue crack propagation in air is compared to that in high vacuum in da/dN vs ΔK and da/dN vs ΔK_{eff} diagrams respectively in Figs 2a and 2b. A substantial environment effect is observed in air (Fig. 2a), that is more pronounced in the low rate range with da/dN in air of about one order of magnitude higher than in vacuum.

At higher ΔK , the detrimental effect of ambient air is progressively diminishing when da/dN increases, and at very high ΔK the curves in air and vacuum are merging to gather when the environment effect practically vanishes. After closure correction (Fig. 2b), the detrimental effect of air is still present and even more accentuated in the near threshold domain, demonstrating that closure does not explain the differences due to environment. The effective threshold can be evaluated at 10^{-10} m/cycle in both environments and compared to the nominal threshold:

$$\begin{aligned} \Delta K_{th,air}(10^{-10} \text{ m/cycle}) &= 5.7 \text{ MPa}\sqrt{\text{m}}, & \Delta K_{th,vac}(10^{-10} \text{ m/cycle}) &= 7.5 \text{ MPa}\sqrt{\text{m}}. \\ \Delta K_{eff,th,air}(10^{-10} \text{ m/cycle}) &= 3.2 \text{ MPa}\sqrt{\text{m}}, & \Delta K_{eff,vac}(10^{-10} \text{ m/cycle}) &= 5.5 \text{ MPa}\sqrt{\text{m}}. \end{aligned}$$

The closure correction results in a reduction of the threshold range of 44% in air and of 27% in vacuum. Such an accentuated shielding effect in air can be explained in term of an enhanced oxide induced closure contribution as initially described by Suresh et al [10]. In this last environment, the closure contribution can be related to plasticity or surface roughness induced closure.

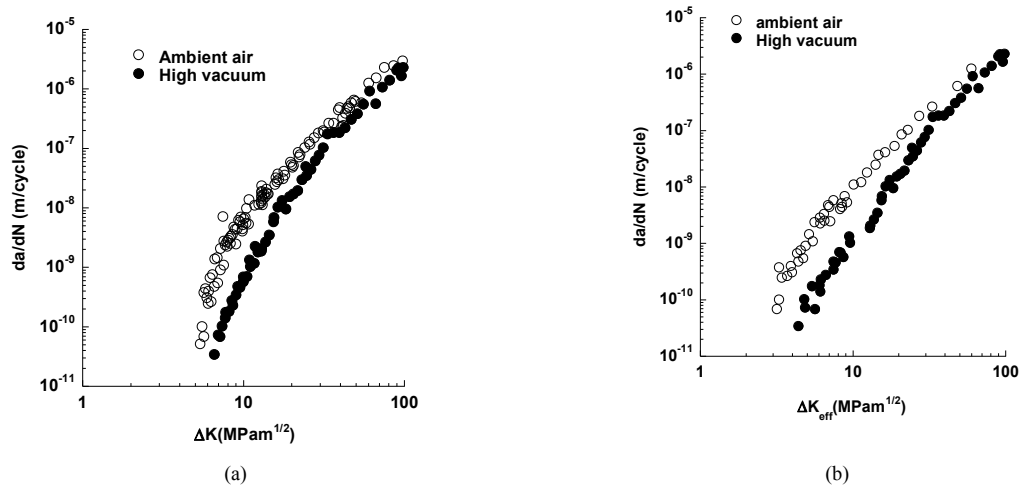


Fig. 2. Influence of ambient air on fatigue crack growth. (a) da/dN vs ΔK ; (b) da/dN vs ΔK_{eff} .

The fracture surface morphology in the near threshold regime in air (Fig. 3a) supports the existence of a transgranular cleavage-like fracture mode typical of a stage II propagation with a crack plane normal to the load axis and a mode I crack opening; localized intergranular areas are in accordance with previous observations on the same alloy tested in air at low R ratio [11] and are more present at ΔK about $14 \text{ MPa}\sqrt{\text{m}}$ (Fig. 3b); the same stage II mechanism is still operating at higher ΔK as shown in Fig. 3c at $\Delta K = 50 \text{ MPa}\sqrt{\text{m}}$, the growth rate being in accordance with the spacing between the ductile striations covering the surface without transgranular facets. Such striations are characteristic of a stage II fatigue crack propagation in ambient air which are not observed in high vacuum, even if a similar stage II crack growth mechanism prevails in both environments and leads to only slightly higher growth rates in air. Following the initial work of Meyn [12], similar observations have been performed on various metallic alloys [9].

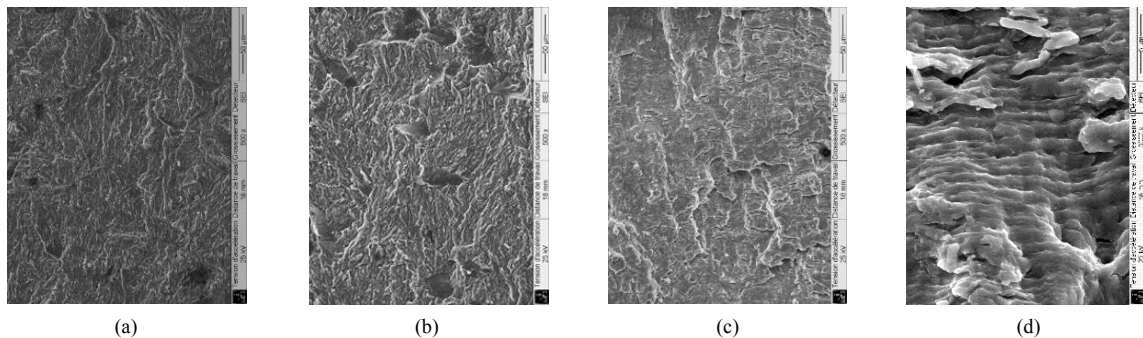


Fig. 3. Micrographic aspect of fracture surface in ambient air (a) near the threshold; (b) $\Delta K = 14 \text{ MPa}\sqrt{\text{m}}$; (c) $\Delta K = 50 \text{ MPa}\sqrt{\text{m}}$; (d) magnification of striations. (Propagation from bottom to top).

3.3. Gaseous hydrogen

The results obtained under 4 bar gaseous hydrogen environment are plotted in Fig. 4 in comparison to data in vacuum and in air. Both diagrams, da/dN vs ΔK in Fig. 4a and da/dN vs ΔK_{eff} in Fig. 4b, let appear two different regimes: for ΔK lower than $14 \text{ MPa}\sqrt{\text{m}}$ (i.e. $K_{max} < 16 \text{ MPa}\sqrt{\text{m}}$), crack growth in hydrogen is similar to that in air,

which means much faster than in vacuum, suggesting a similar environment effect in both gaseous atmospheres. For ΔK higher than $14 \text{ MPa}\sqrt{\text{m}}$ (i.e. $K_{\text{max}} > 16 \text{ MPa}\sqrt{\text{m}}$), da/dN in hydrogen is substantially enhanced compared to ambient air. It can be also noticed that, at increasing ΔK , when the effect of air environment is progressively vanishing, conversely, the effect of hydrogen is increasing.

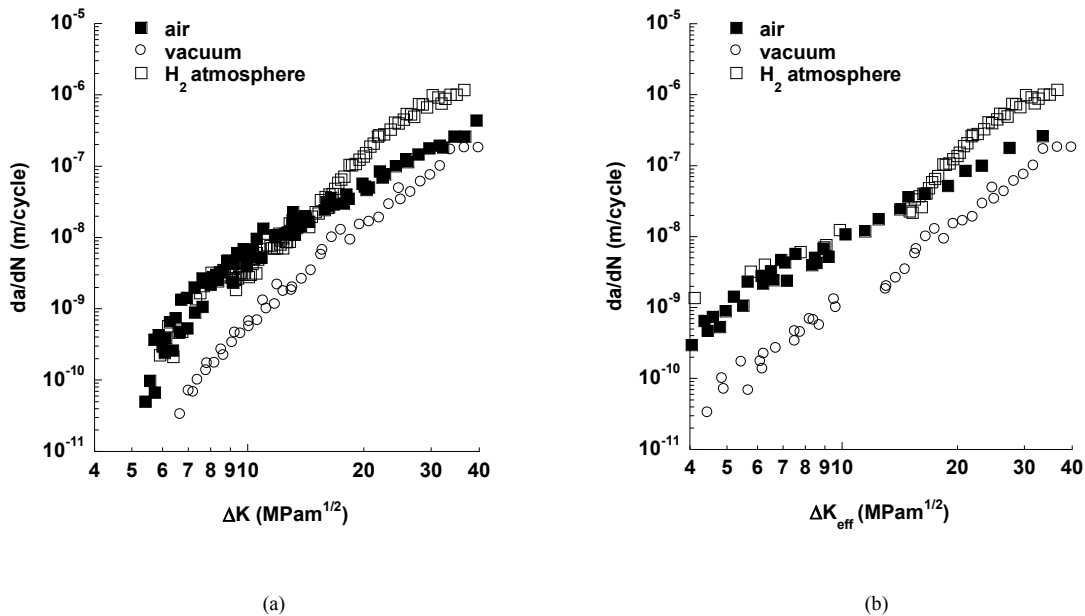


Fig. 4. Fatigue crack growth diagram for tests in air, vacuum and hydrogen environment at $R=0.1$ and 20Hz ; (a) da/dN vs ΔK ; (b) da/dN vs ΔK_{eff} after crack closure correction.

SEM observations of the fracture surface in hydrogen in the near threshold regime (Fig. 5a) appear very similar to that in air (Fig. 3a) with a transgranular cleavage-like fracture mode typical of a stage II propagation with a crack plane normal to the load axis and localized intergranular facets again in accordance with previous observations on the same alloy comparatively tested in hydrogen and ambient air [11]. On the basis of the similitude of crack growth data and of the surface morphology, the same water vapor adsorption assisted stage II propagation mechanism is assumed to be operative in both gaseous environments. This assumption is supported by the presence in the 4 bar hydrogen of a substantial amount of residual water vapor with a partial pressure $P_{\text{H}_2\text{O}}=24 \text{ Pa}$. Such low partial pressure of water vapor in nitrogen gas has been shown sufficient to induce a similar near threshold environment effect as in air [12]. A comparison of the fatigue crack growth behavior in the present alloy with that of construction steel E460 is given in Fig.6. Typically the fatigue crack growth behavior in air and in vacuum as well in terms of da/dN vs ΔK (Fig. 6a) as da/dN vs ΔK_{eff} (Fig. 6b) is identical for the two alloys in all the explored rate range and thus can be analyzed in term of atmospheric water vapor adsorption [9, 13-16].

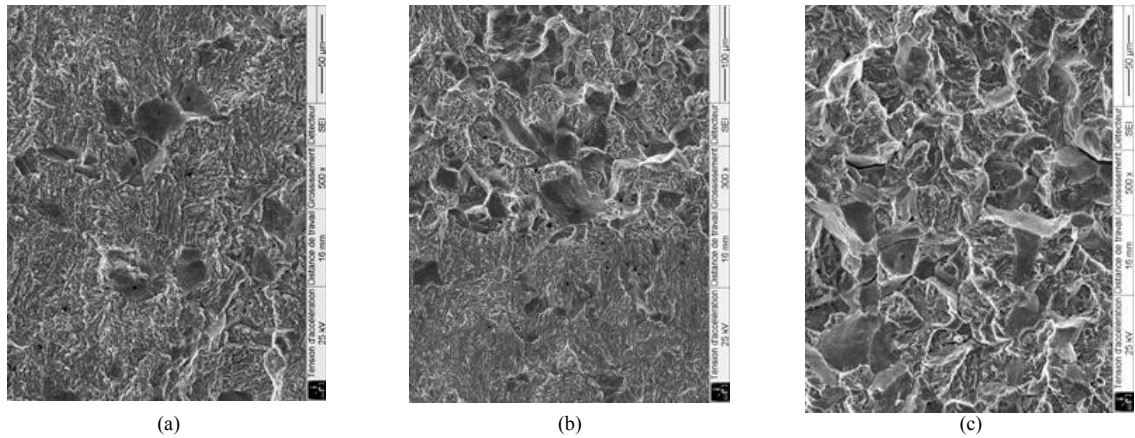


Fig. 5. Crack surface morphology under 4 bars hydrogen atmosphere; (a) in the near threshold domain; (b) $\Delta K = 14 \text{ MPa}\sqrt{\text{m}}$ with a sharp transition in the crack propagation mechanism; (c) $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$. (Propagation from bottom to top).

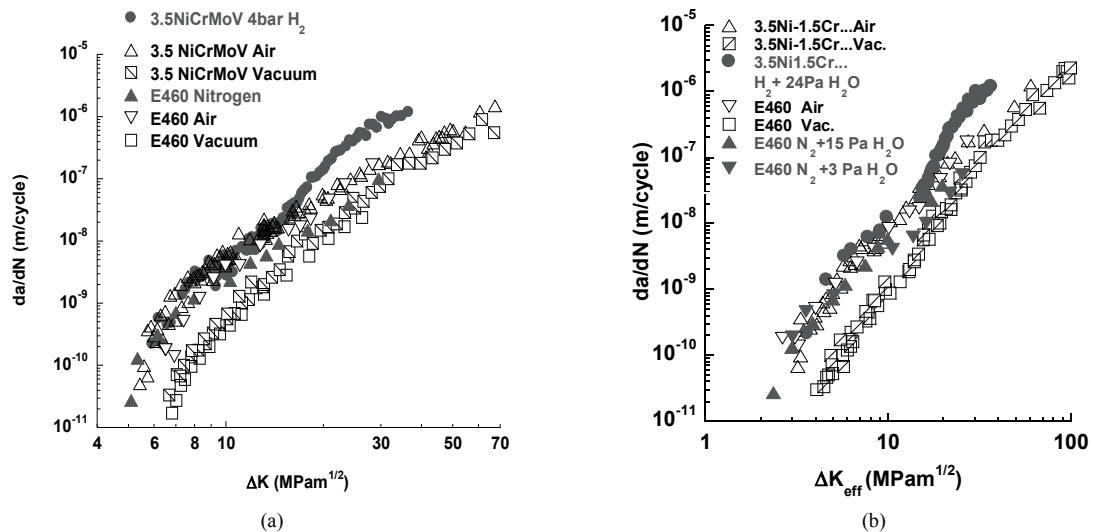


Fig.6. Comparison of the influence of environment on 3.5NiCrMoV in hydrogen with 24Pa of water vapor and E460 steel in Nitrogen with 15Pa and 3 Pa of water vapor, with reference high vacuum ($<5 \cdot 10^{-4} \text{ Pa}$) and ambient air with $\sim 150 \text{ Pa}$ of water vapor; (a) nominal curves; (b) effective curves.

In 4 bar hydrogen with a partial pressure of 24 Pa of water vapor, for ΔK lower than $14 \text{ MPa}\sqrt{\text{m}}$ ($K_{\text{max}} < 16 \text{ MPa}\sqrt{\text{m}}$) the behavior is identical to that in air and, near the threshold, is also identical to that of the E460 steel in nitrogen containing water vapor at a partial pressures of 15 Pa and 3 Pa (Fig. 6b). In addition the fracture surface morphologies under hydrogen and in air are very similar with a predominant transgranular stage II regime as previously observed in the E460 alloy [17]. Consequently, the environment effect of the 4 bar hydrogen in the near threshold region at $R=0.1$ is similar to that in air and in nitrogen with traces of H_2O , and has to be attributed to a mechanism related to the adsorption of water vapor molecules at the crack tip and not at the hydrogenous atmosphere. At higher $\Delta K \sim 14 \text{ MPa}\sqrt{\text{m}}$ ($K_{\text{max}} \sim 16 \text{ MPa}\sqrt{\text{m}}$) the fracture surface in hydrogen shows in Fig. 5b an abrupt change from trans- to inter-granular crack path associated to accelerated da/dN . This result is in accordance with previous results of Sun et al. on 15-5PH alloy [7]. At $\Delta K = 30 \text{ MPa}\sqrt{\text{m}}$ (Fig. 5c) an intergranular propagation

prevails with 10 times faster propagation rates than in air or vacuum. Crack propagation in hydrogen gas has been widely studied under monotonic loading [18]. Time dependent crack growth called HEAP (Hydrogen Environment Assisted Propagation) are typically observed for stress intensity higher than a threshold which namely depends on the hydrogen pressure. The same mechanism can be considered for fatigue crack propagation in hydrogen gas [19] for K_{max} higher than a critical threshold for stress corrosion cracking which appears to be about $16 \text{ MPa}\sqrt{\text{m}}$.

On-going investigations are performed in order to identify precisely the different processes involved in these gaseous environments taking into account test frequency, load ratio, gas pressure ...

4. Conclusions

This study on the influence of 4 bar gaseous hydrogen atmosphere on fatigue crack propagation in low alloyed steel (3.5Ni-1.5Cr-0.5Mo-V) in comparison to ambient and high vacuum leads to the following conclusions:

- 1) For K_{max} lower than $16 \text{ MPa}\sqrt{\text{m}}$, the crack propagation under 4 bar hydrogen is similar to that in ambient air; the acceleration of the crack growth rate da/dN and the lower threshold range ΔK_{th} in comparison to high vacuum are related to the detrimental effect of residual water vapor adsorption at the crack tip; this is consistent with previous results on E460 construction steel tested in Nitrogen with partial pressure of water vapor of 15 Pa and 3 Pa.
- 2) At $\Delta K \sim 14 \text{ MPa}\sqrt{\text{m}}$ ($K_{max} \sim 16 \text{ MPa}\sqrt{\text{m}}$) the fracture surface in 4 bar hydrogen shows an abrupt change from transgranular to intergranular crack path associated to accelerated da/dN which is in accordance with a critical threshold for stress corrosion cracking at about $16 \text{ MPa}\sqrt{\text{m}}$.
- 3) At higher ΔK , the propagation becomes much faster than in air and vacuum; the associated intergranular propagation mechanism seems to be related to the superimposition of a cyclic fatigue component $(da/dN)_{fat}$ and of a subcritical time dependent component as observed under sustained loading $(da/dN)_{scc}$ resulting from hydrogen assisted mechanism.

References

- [1] R P Gangloff: in Encyclopedia of Comprehensive Structural Integrity, edited by I. Milne, R.O. Ritchie, B.L. Krihaloo, Vol. 6, Elsevier Pergamon pub. (2003), 31-101.
- [2] I. Moro, L. Briottet, P. Lemoine, E. Andrieu, C. Blanc, and G. Odemer: Mater. Sci. Eng. A, 527, (2010) 7252–60.
- [3] G. Schuster and C. Altstetter: Metall. Trans. A, vol. 14A, (1983) 2085–90.
- [4] Y. Murakami, T. Kanezaki, Y. Mine, and S. Matsuoka: Metall. Mater. Trans. A, 39A (2008) 1327–39.
- [5] Y.S. Ding, L.W. Tsay, M.F. Chiang, and C. Chen: J. Nucl. Mater., vol. 385 (2009) 538–44.
- [6] E647-00, Standard Test Method, *ASTM International*, 2000.
- [7] Z. Sun, C. Moriconi, G. Benoit, D. Halm, G. Henaff: Metall Mat Trans A, Vol. 44-3 (2013), 1320
- [8] K. Vor, C. Gardin, C. Sarrazin - Baudoux and J. Petit: Engineering Fracture Mechanics, Vol.99 (2013), 266.
- [9] J. Petit, G. Henaff, C. Sarrazin-Baudoux, "Environmentally assisted fatigue in gaseous atmosphere", in J. Petit, P. Scott (eds.), *Comprehensive Structural Integrity, vol.6 Environmentally-Assisted Rupture*, Elsevier, (2003) 211-280.
- [10] S. Suresh and R.O. Ritchie, "Near threshold fatigue crack propagation: a perspective on the role of crack closure", in: "Fatigue Crack Growth Threshold Concepts", D. Davidson and S. Suresh eds., TMS Aime Pub., 1984, pp. 227-262.
- [11] A.T. Stewart: Eng. Fract. Mech., 1980, vol. 13, pp. 463–78.
- [12] D. A. Meyn, Trans ASM, 61 (1968), pp.52-61.
- [14] S.P. Lynch: Hydrogen Effects on Material Behavior and Corrosion Deformation Interactions, TMS, Warrendale, PA, 2003, pp. 449– 66.
- [15] F.J. Bradshaw, C. Wheeler, "The effect of environment on fatigue crack growth in aluminium and some aluminium alloys", Applied Materials Research, vol. 5 (1966) 112.
- [16] J. Petit, "Some aspects of near-threshold crack growth : microstructural and environmental effects", in D.L. Davidson, S. Suresh (eds.), *Fatigue Crack Growth Threshold Concepts*, Philadelphia, PA, Metallurgical Society of AIME, 1984, pp. 3-24.
- [17] A. Bignonnet, D. Loison, R. Namdar-Irani, B. Bouchet, J.H. Kwon, J. Petit, "Environmental and Frequency effects on near-threshold fatigue crack propagation in a structural steel", in: "Fatigue Crack growth Threshold Concepts", D. Davidson and S. Suresh eds., TMS Aime Pub., 1984, pp. 99-113.
- [18] R.A. Oriani: Ann. Rev. Mater. Sci., 8 (1978) 327–57.
- [19] C.D. Beachem: Metall. Trans., 3 (1972) 437–51.