SCC BEHAVIOUR OF PIPELINE MICROALLOYED STEELS

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ABSTRACT

The internal cracking of a microalloyed ferritic-pearlitic API X-65 steel produced in standard NACE HIC tests was evaluated. Most of the cracks were associated to the centre segregation of the plate. Tensile tests, fracture toughness and stress corrosion cracking characterisation tests were performed on specimens which had been submerged in normalised NACE environments. Slow displacement rate fracture tests were also performed in aggressive acid solutions obtained by cathodic polarization at different current densities.

INTRODUCTION

The operating conditions of pipelines used as long distance oil and gas transportation have become more severe in recent years, and hence the material properties required have been also more stringent. Nowadays, microalloyed ferritic steels with a high strength, toughness and good weldability are used in these applications. Likewise, as the development of oil and gas fields containing H_2S has also grown in recent years, satisfactory mechanical behaviour in the presence of hydrogen is a critical factor to be taken into account, requiring the selection of a material able to resist hydrogen induced cracking (HIC) and stress corrosion cracking (SCC) [1].

In these kinds of applications, hydrogen enters the steel due to corrosion in wet H_2S environments and is trapped around non-metallic inclusions in the steel. The hydrogen gas pressure is the driving force of HIC, and the existence of other microstructure factors, such as centre segregation of P and Mn and low transformation phases, are also important aspects to consider. This embrittlement is known to increase with the steel strength. The way in which diffusive and trapped hydrogen interacts with the microstructure of the steel subjected to a sustained mechanical load is also a very important factor that needs to be accurately ascertained when designing structural elements subjected to these kinds of services.

EXPERIMENTAL PROCEDURE

The steel used in this study was an API X-65 structural steel made in ACERALIA (Spain), which was furnished as a plate with a nominal thickness of 23 mm. The steel was treated with calcium in order to proceed to the spherodization of the inclusion population. Table 1 presents the chemical composition of the steel. This grade of steel has a banded, fine grain, ferritic-pearlitic microstructure, obtained by cooling in air after a thermo-mechanical hot rolling process (Figure 1).

Tensile tests were performed on transversal specimens. Fracture mechanics tests were determined using compact specimens with a thickness of 20 mm. All these latter specimens were pre-cracked in fatigue using a load ratio of $R= P_{min}/P_{max} =0.1$. The J-R curves were obtained using the single specimen method proposed

in the ESIS P2-92 standard. A COD clip gauge mounted on the mouth of the specimen notch was used for indirect determination of the stable crack growth based on the measurement of the specimen compliance during several unload/reload sequences. Also, colour marking were made to locate the crack fronts at two different intermediate stages of the tests: at certain load level, the test was stopped and the crack front marked, with coloured paint in the first stage and with transparent lacquer in the second one. The two coloured crack fronts together with the final one allow correction, by means of linear interpolation, of the crack growth values calculated using the compliance method.

TABLE 1
Chemical composition of the steel (weight per cent)

C (%)	Si (%)	Mn (%)	S (%)	P (%)	Cu (%)
0.066	0.285	1.31	0.002	0.011	0.32
V (%)	Mo (%)	Nb (%)	Ti (%)	Al (%)	N((%)
0.065	0.082	0.047	0.006	0.036	0.0061

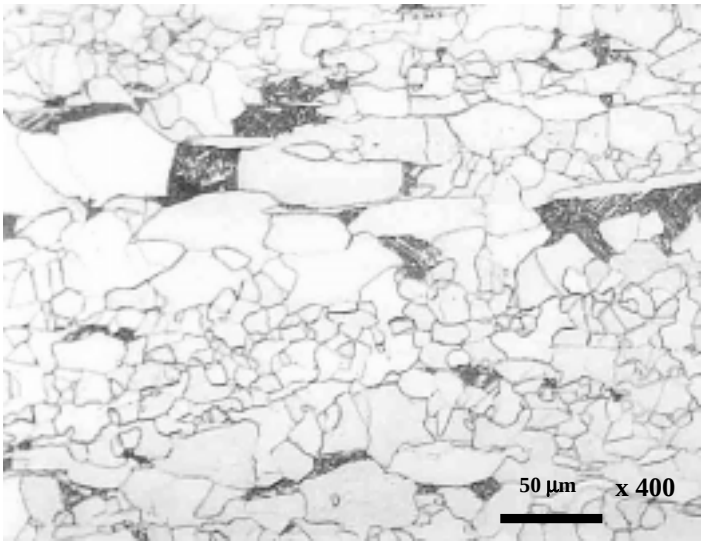


Figure 1: Microstructure of the API X-65 steel

The evaluation of hydrogen induced cracking (HIC) was carried out by means of the 96 hours NACE TM.0284-87 test [2]. In order to ascertain the effect of pH on the hydrogen embrittlement of the steel, two different solutions were used, corresponding to pH 5 and pH 3.5. Due to the wide variations usually obtained in these tests, cracking ratios of nine sections of each one of the three pieces tested were determined and a probabilistic evaluation was performed with all these data. The appearance of cracking was also located along the specimen thickness.

Tensile and fracture mechanics specimens (previously pre-cracked in fatigue) were also immersed in the pH 3.5 medium during 96 hours and subsequently tested. Slow displacement rate fracture tests, using different velocities, were also performed on the pre-cracked compact specimens in the presence of an aggressive environment obtained by cathodic polarization at different current densities in a solution of 1N of H_2SO_4. According to reference [3], the entry of hydrogen in the H_2S solution and when a cathodic current density of 5 mA/cm^2 is applied is equivalent. By applying a suitable analytical methodology [4,5] based on the EPRI procedure [6] for characterising the behaviour of cracked specimens in elastic-plastic regime, it is possible, at any one time, to know the crack length, a, and the J integral values. Finally, all the tested specimens were evaluated under an scanning electron microscope.

RESULTS

Table 2 gives the average results of the crack length ratio (CLR) obtained in the two NACE mediums: No crack were observed in any section when using the pH 5 solution, but significant internal cracking was detected after 96 hours in the pH 3.5 solution. A probability curve that includes all the 27 results obtained in the pH 3.5 medium was represented in Figure 2 [7]. A very high dispersion was obtained in this test: no crack was detected in some sections (CLR)=0, while important cracking was measured, at the same time, in other sections (CLR>50%). This is an indication that the hydrogen content in the steel during the test was near the threshold hydrogen concentration value for cracking [8]. It was also seen that these cracks were mainly located at mid-thickness, and are thus associated to the typical centre segregation of the plate.

TABLE 2
Average results of NACE TM.0284-87 test

NACE medium	pH 5	pH 3.5
CLR (%)	0	11

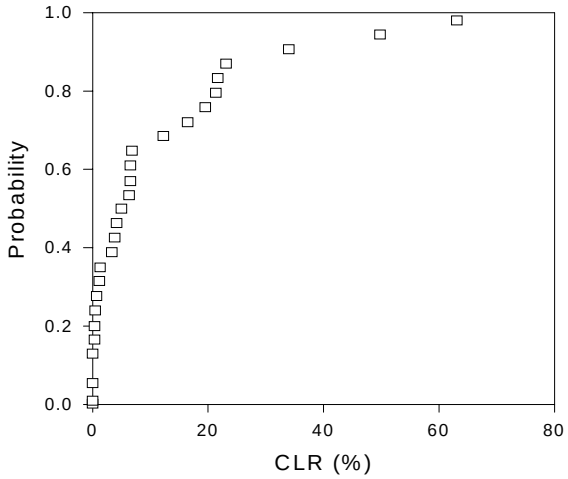


Figure 2: CLR probability curve (NACE pH3.5)

Table 3 shows the tensile mechanical properties at room temperature obtained with the as-rolled steel and after it had been immersed during 96 hours in the pH 3.5 NACE solution. All the properties have decreased slightly, except the specimen area reduction, Z, which reveals a significant loss of ductility due to the presence of internal cracking. This cracking was clearly visible in the centre of the broken section and was associated to centre segregation.

TABLE 3
Tensile properties of the steel

	σ_{ys} (MPa)	σ_u (MPa)	El (%)	Z (%)
As-rolled	450	530	32	76
After NACE test (3.5 pH)	427	514	29	45

Figure 3 represents the J-Δa curve of the as-rolled steel and the results of two different tests obtained after being immersed during 96 hours in the pH 3.5 NACE solution. The results are nearly coincident. A

threshold J value ($J_{0.2}$) of 190 kJ/m^2 was obtained. Only a slightly higher J-Δa curve was observed in the specimen in which a central delamination was initiated as a result of central cracking induced in the previous NACE test. Central delamination induces a change from plane strain to plane stress, which is responsible from the increase in the toughness of the steel. A dimple fracture micromechanisms was always observed in all these tests, meaning that the presence of residual (trapped) hydrogen has modified neither the macroscopic nor the microscopic mechanical behaviour of the steel (Figure 4) .

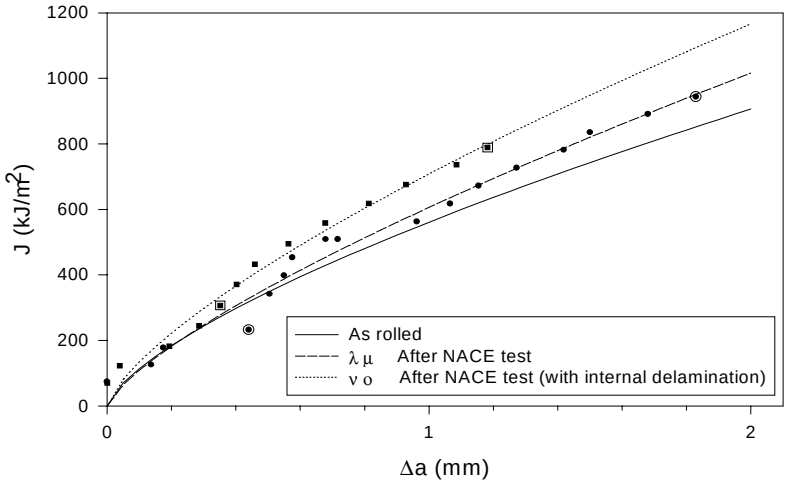


Figure 3: J-Δa curves. As-rolled and after being immersed during 96 hours in the pH 3.5 NACE solution

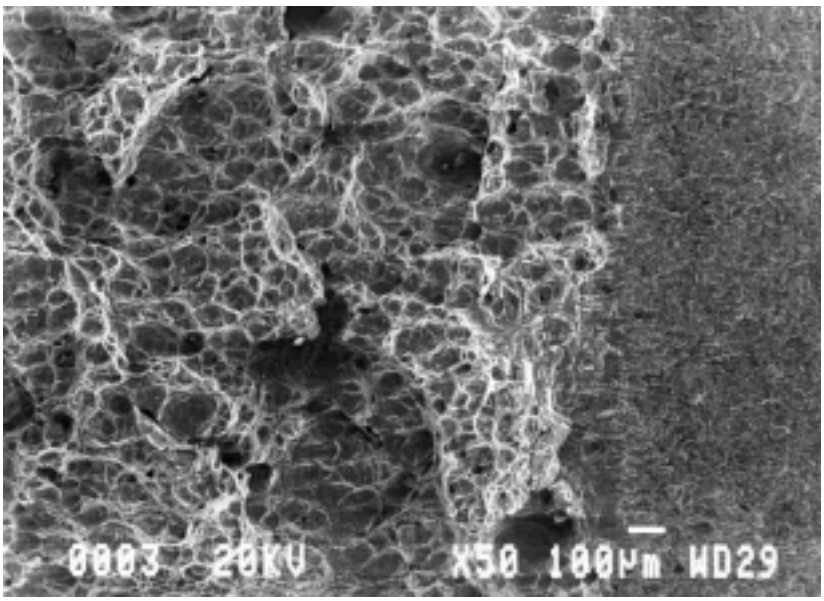
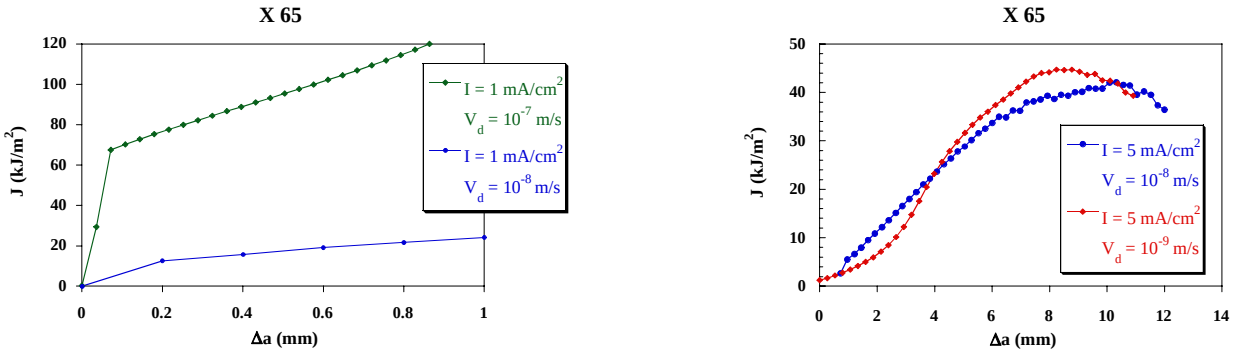


Figure 4: Ductile fracture of CT specimen after being immersed during 96 hours in the pH 3.5 NACE solution (propagation from the fatigue precrack).

In contrast, the J-Δa curves obtained in the slow displacement rate fracture tests performed in the presence of an aggressive environment obtained by cathodic polarization at different current densities were quite different. The steel shows in those tests a high susceptibility to HIC processes. Figure 5 shows the J-Δa curves for all the tests made under different conditions and Table 4 presents the threshold J values ($J_{0.2}$) of all the tests performed.



(a) (b)

Figure 5: J-Δa curves. Slow-rate fracture tests, cathodically polarized.

TABLE 4
Threshold J values. Slow-rate fracture tests, cathodically polarized

	X 65	
$J_{0.2}$ (kJ/m^2)	I = 1 mA/cm^2	I = 5 mA/cm^2
$V_d = 10^{-7}$ m/s	75.4	.....
$V_d = 10^{-8}$ m/s	14.6	8.9
$V_d = 10^{-9}$ m/s	.....	4.1

As can be seen in this figure, for a same current density of 1 mA/cm^2 but different displacement rates, $V_d = 10^{-7}$ m/s and $V_d = 10^{-8}$ m/s (Figure 5(a)), the J values obtained in the last case were much lower. Thus, the displacement rate has a great influence, affecting the HIC response of this steel. However, for higher current density levels; for example 5 mA/cm^2, the J values obtained at two different displacements rates ($V_d = 10^{-8}$ m/s and $V_d = 10^{-9}$ m/s) show similar results as can be appreciated in Figure 5(b). Finally, for a same displacement rate, for example $V_d = 10^{-8}$ m/s and different current densities, as can be 1 mA/cm^2 and 5 mA/cm^2, the J values are quite similar. Thus, the effect of current density is not important when a slow displacement rate is provided.

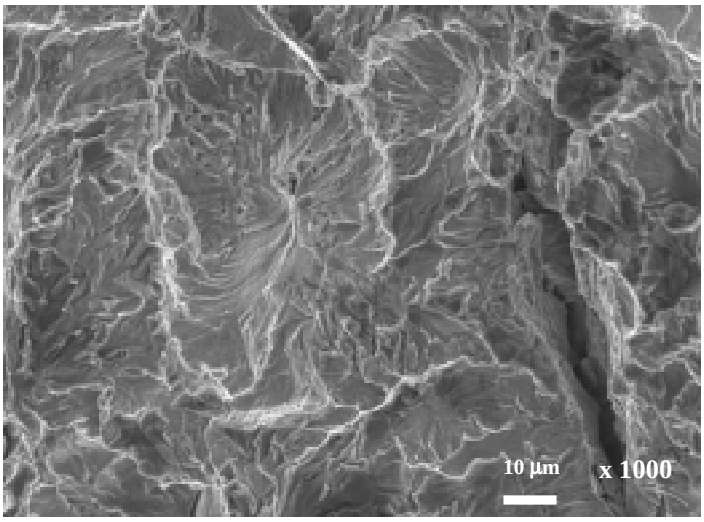


Figure 6: Cleavage fracture in slow-rate test under 5 mA/cm^2 and $V_d = 10^{-9}$ m/s

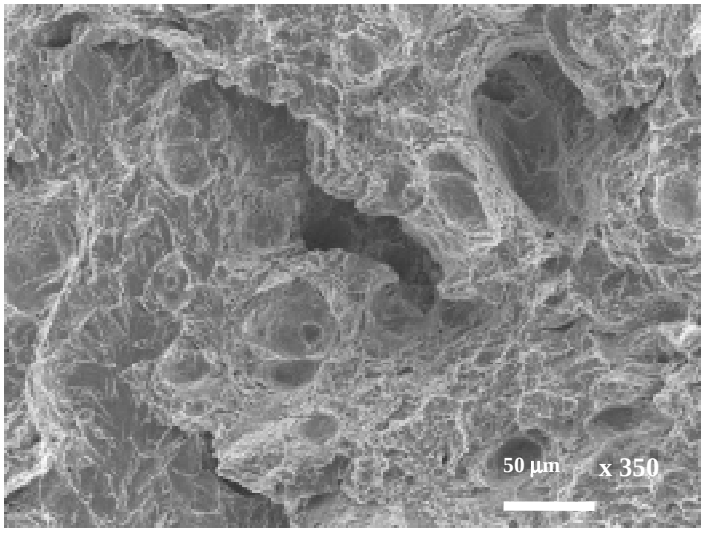


Figure 7: Mixed fracture in slow-rate test under 1 mA/cm^2 and $V_d = 10^{-7}$ m/s

The micrographic observation of this material corresponding to the results which were presented in Figure 5(b) shows a brittle fracture micromechanisms (cleavage) with some tearing, which increases with the mechanical solicitation. Figure 6 shows a typical cleavage fracture obtained in this conditions. The test made under 1 mA/cm^2 and with a displacement rate of 10^{-7} m/s, presented in Figure 5(a), shows a mixed propagation mode composed by cleavage and microvoids micromechanisms. Figure 7 shows a typical fracture surface obtained in this test.

CONCLUSIONS

Highly dispersed results were obtained in the NACE test performed in pH 3.5 medium, the hydrogen content associated at this pH value hence being considered to be near the threshold hydrogen concentration for this steel. Internal cracking was mainly located at mid-thickness, and was consequently associated with the typical center segregation of the plate.

The J-Δa curve of the as-rolled steel and after being immersed during 96 hours in the pH 3.5 NACE solution present nearly coincident results, meaning that the presence of trapped hydrogen has modified neither the macroscopic nor the microscopic mechanical behaviour of the steel. In contrast, the J-Δa curves obtained in the slow-rate fracture tests performed in the presence of an aggressive environment obtained by cathodic polarization at different current densities were much lower.

An explanation for the differences obtained in both sets of tests is the effect of hydrogen provided by the two methods. In NACE environment, the samples are damaged by hydrogen but this situation does not change the global resistance to cracking, when the sample is loaded later on. In the cathodic charging processes the effect of hydrogen is localised at the crack tip region and fracture micromechanisms change and toughness is severely reduced when time is allowed for hydrogen diffusion (very low displacement rates).

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