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Hydrogen induced cracking (HIC) testing of low alloy steel in sour environment – Impact of time of exposure on the extent of damage

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ABSTRACT

The selection of materials for application in the oil and gas industry in environments containing hydrogen sulphide (H_2S) is an important matter for the safety of equipments. In the presence of H_2S, hydrogen atoms originating from the cathodic corrosion reaction can diffuse into the steel, resulting in cracking mechanisms such as hydrogen-induced cracking (HIC) or sulphide stress cracking (SSC).
For the SSC cracking mode, a diagram defining different regions of environmental severity was established in the 1990's. The requirements for SSC testing of steels in a given environment are well defined and accepted by the industry. On the other hand, fit-for-purpose (FFP) testing for HIC remains a subject of debate. The most common practice consists in 96 hours exposure under 1 bar H_2S. But this test environment is extremely severe, and is often considered to be too conservative for the selection of steels for use in mildly sour environments. However, a universally applicable FFP test method for HIC has not yet been established. In addition to pH and H_2S partial pressure, test duration is another major parameter for HIC testing. Therefore, this experimental study was launched to investigate the impact of time of exposure on HIC. Experiments consisted of HIC exposure tests based on the NACE TM0284-2003 standard method. Experiments were conducted in different nodes of the pH – pH_2S diagram respectively between pH 6.5 and 3.5, and between 10 mbar and 1 bar of H_2S. For each condition, tests with different times of exposure were realised, from only a few hours and up to three months. Characterisation of HIC consisted in ultrasonic testing. Permeation experiments were also performed in selected pH – pH_2S conditions. Permeation transients were analysed for calculation of diffusion coefficients. Modelling of hydrogen profiles in HIC specimens was then realised, and compared with the results of HIC immersion tests.
For each test condition, it was possible to draw the evolution with time of the extent of HIC. Depending on the environment, the minimum duration necessary to initiate HIC was extremely variable, from a few hours in the most severe conditions to several weeks in low severity conditions. The extent of cracking at equilibrium also seemed to vary with pH and H_2S.
A good correlation was found between HIC and permeation results, confirming that HIC is strongly linked with the increase of hydrogen concentration in the steel.
These results confirm that low severity conditions require longer exposures to reach equilibrium levels of internal hydrogen. This must be taken into account for FFP HIC testing in mildly sour environment. With that aim, permeation experiments could represent an interesting technique to assess the time to reach a certain level of hydrogen in the material.

KEYWORDS

"Hydrogen induced cracking", "HIC", "hydrogen permeation", "hydrogen sulphide", "H_2S"

INTRODUCTION

Hydrogen damage is one of the major causes of steel equipment failures in the oil & gas industry [1-7]. It is caused by the dissolution of hydrogen in the steel, as a result of the cathodic reduction of H^+ which accompanies the anodic oxidation of iron in acid media. The reduction of water molecules by cathodic overprotection is another potential source of reduced H^+. Several failure modes can occur in service, for example:

- hydrogen induced cracking (HIC), corresponding to internal cracks generated by the recombination of hydrogen to gaseous molecules at certain appropriate traps in the steel, like MnS inclusions of pearlite bands. This failure mode is strictly internal, and does not require an external stress [2,8-14].
- Hydrogen stress cracking (HSC), also designed as sulphide stress cracking (SSC) when H_2S is present: this cracking mode is generated from the surface of the steel and requires an applied stress [15-23].

The presence of H_2S is known to have an aggravating influence.

The selection of steels for oil and gas environments containing H_2S thus requires appropriate testing, to ensure resistance to cracking in the field conditions. NACE MR-0175/ISO 15156 Part 2 [24] describes most of the testing methods used for SSC and HIC evaluation.
For SSC, the pH - pH_2S severity regions were established on an empirical basis, and are well accepted by the industry. On the other hand, a similar diagram for HIC resistance does not exist yet. In addition, NACE MR0175/ISO 15156 Part 2 does not place any lower H_2S limit below which an assessment of HIC resistance is unnecessary. For severe conditions (i.e. 1 bar H_2S), HIC testing methodology and acceptance criteria are the subject of a large consensus. On the other hand, mildly sour testing is still under debate, especially concerning the time of exposure under moderate H_2S partial pressure. A recent study by the European Pipeline Research Group (EPRG) Corrosion Committee [11] proposed a preliminary HIC severity diagram, based on testing five different sweet service steels. One of the main conclusions of this study was the necessity to use long term (2 to 4 weeks) immersion tests for mildly sour conditions.
However, testing for durations up to several weeks is a major drawback for a rapid fit-for-purpose assessment. Permeation measurements could represent an interesting alternative in addition to standard HIC immersion tests. As illustrated recently [25], the diffusion parameters (subsurface concentration C_0 and diffusion coefficient D) for hydrogen in steels exposed to sour environment could be obtained from permeation experiments through thick steel membranes.

Starting from these insights, an experimental study was launched with the objective of studying the time dependence of HIC for different pH – pH_2S conditions. The test methodology was in accordance with NACE TM0284-2003, and several tests with different durations were performed for each condition. HIC cracking was evaluated using ultrasonic testing.
Additionally, hydrogen permeation experiments were realised, using 10 mm thick membranes made of the same steel as for the HIC tests. From the permeation transients, the diffusion coefficient D and the hydrogen subsurface concentration C_0 were determined for different pH – pH_2S. These parameters were then used to calculate the concentration profiles in standard HIC specimens, and the results were compared with HIC tests.

1. EXPERIMENTAL

1.1. Tested Material

The steel used for this study was an API X65 TMCP steel, coming from a sweet service LSAW pipe 42 inches diameter and 1 inch (25.4 mm) thick. Its chemical composition (Table 1) was analysed by optical emission spectrometry (OES), except for carbon and sulphur, analysed by a chemical method. The microstructure consisted mainly of ferrite with a portion of pearlite, with a banded structure particularly pronounced in the centre line.
This material is the same as steel "P" of ref. [11]. Some results of this previous study were used in this paper. The same steel was also used for the permeation study reported in ref. [25].

Table 1: Chemical compositions (wt %) of tested steel.

C	Mn	Si	P	S	Cr	Ni	Mo	Cu	Nb	V
0.09	1.56	0.28	0.014	0.001	0.05	0.03	0.01	0.02	0.04	0.05

Specimens for HIC experiments were sampled following the NACE TM0284-2003 standard test method. The sample dimensions were 100 mm long, 20 mm wide and 24 mm thick (0.5 to 1 mm were removed from the inner and outer surfaces). All faces were machined to obtain a finish equivalent to that achieved using 320 grit SiC paper.

Specimens for permeation experiments were 70 mm x 70 mm plates, with a thickness of 10 mm or 0.5 mm. The surface finish was the same as that of HIC samples.

1.2. Test solutions and exposure conditions

All solutions were prepared according to EFC16 document [26], solution A, and thus contained 5% sodium chloride and 0.4% sodium acetate. The bubbling gas is either pure H_2S, or a mixture of CO_2 with H_2S (1 or 10%).
After a thorough deoxygenating step, the solution was saturated by bubbling gas at ambient pressure. The pH was then adjusted to desired level through addition of deaerated 1N hydrochloric acid or deaerated 1N sodium hydroxide NaOH. The solution was then poured into the test cell.

For HIC experiments, standard glass vessels were used. All HIC tests were performed following the general NACE TM0284-2003 standard, including three replicates for each condition and a volume / surface ratio greater than $3mL.cm^{-2}$. pH measurements of the test solution were performed with a period of 48 hours or less, and adjustments were made if the value was not within -0.1 / + 0.3 pH units of the desired level.
After each test, all specimens were inspected using ultrasonic method. The analysis was performed with a ULTRAPAC system (Euro Physical Acoustics SA) and a 15MHz transducer (1/4" diameter). The system was calibrated according to a well-established specification. For each specimen, the total area of the defects was calculated from the UT scans, and the crack area ratio (CAR) was determined as the ratio between the area of the defects and the total area of the specimen in the short transverse plan. The values reported in this paper are the average of three replicates.

Permeation experiments were conducted with a Devanathan-Stachurski [27] cell (Figure 1) that was designed specially for thick specimens. The exposed surface area was 19.6 cm^2. The experimental set-up consisted of two identical electrolytic cells separated by the steel membrane. The charging surface was left at the corrosion potential. The exit surface was coated with Pd [28-30] and held in a 0.1 M NaOH solution at a potential of +300 mV vs. the saturated calomel electrode (SCE). Thus all the hydrogen atoms diffusing through the membrane were oxidised, this oxidation current providing a measure of the hydrogen permeation flux.

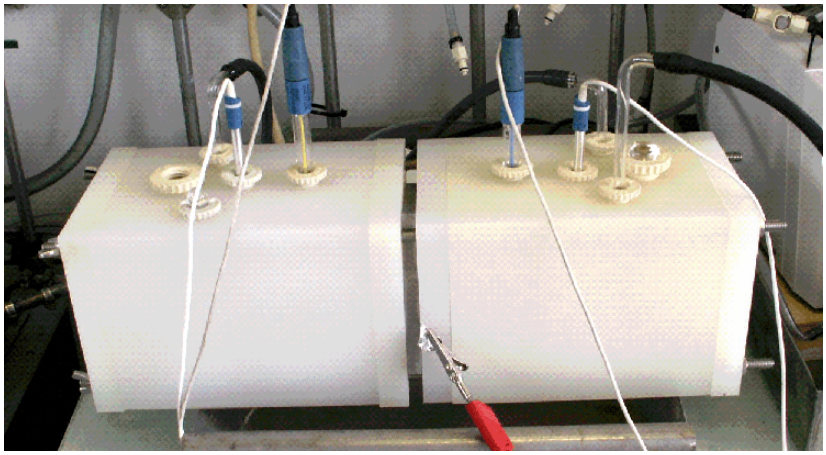


Figure 1: Devanathan-Stachurski cell for permeation measurements on thick specimens.

2. RESULTS AND DISCUSSION

2.1. Permeation data

As described in details in previous papers [25,31], great care must be taken for the interpretation of hydrogen permeation measurements in H_2S environments. For too thin steel membranes, the rate determining step is the charging mechanism at the entry face, and the permeation flux becomes independent of the membrane thickness. For a correct evaluation of the hydrogen sub-surface concentration and of the diffusion coefficient, it was shown for the steel of this study [25] that membranes thicker than 3 mm were necessary.

Three experiments were performed on 10 mm thick steel membranes exposed to 900 mbar CO_2 and 100 mbar H_2S at pH 3.5, 4.5 and 5.5. All the results were then analysed in order to determine the diffusion properties of hydrogen in this steel. Considering one-dimensional diffusion though a membrane of thickness L, the diffusion coefficient was determined from the analysis of the permeation transients $J(t)$. Under these conditions, the ratio between the permeation transient $J(t)$ and the steady-state flux J_{ss} corresponding to the second Fick's law depends on the diffusion coefficient D and on the membrane thickness L [32,33] :

$$\frac{J(t)}{J_{ss}} = 1 + 2\sum_{n=1}^{\infty}(-1)^n \exp\left(-n^2\pi^2 \frac{D\times t}{L^2}\right) \quad (1)$$

Once the steady-state is reached, the hydrogen sub-surface concentration at the entry face C_0 can be determined with the first Fick's law of diffusion from J_{ss} and the diffusion coefficient D:

$$J_{ss} = D \times C_0 / L \quad (2)$$

An example of experimental result is given in Figure 2. An excellent agreement is found between the experimental data and calculations with the Fick's laws of diffusion. Table 2 reports the values of diffusion coefficient and subsurface concentration calculated from the experimental results under 100 mbar H_2S and for different pH. When pH decreases, hydrogen diffusion becomes faster (increase of diffusion coefficient), and the equilibrium hydrogen concentration increases. However, it is worth pointing out that the orders of magnitude of changes of diffusion parameters are far from the changes in hydrogen concentration in the test solution: from pH 5.5 to 4.5, hydrogen concentration in the test solution increases tenfold, while hydrogen subsurface concentration and diffusion coefficient are only multiplied by 1.4 and 2 respectively.

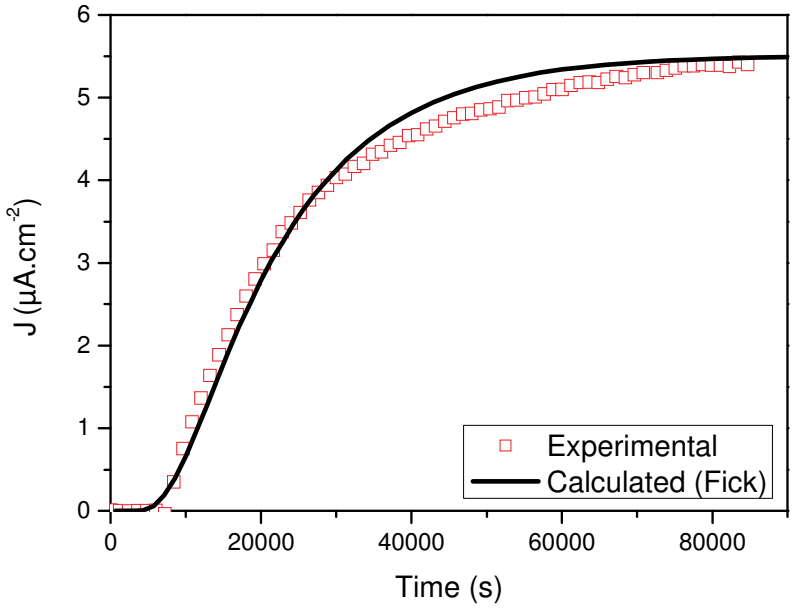


Figure 2 : Hydrogen permeation transient measured for a 10 mm steel membrane exposed to test solution at pH 5.5 under 900 mbar CO_2 and 100 mbar H_2S. Comparison with ideal Fickian behaviour.

Table 2 : Model parameters obtained by fitting experimental results (10 mm thick membranes at 100 mbar H_2S) with the first and second Fick's laws of diffusion.

pH	**3.5**	**4.5**	**5.5**
D ($cm^2.s^{-1}$)	2.7×10^{-5}	1.7×10^{-5}	7.5×10^{-6}
C_0 (ppm)	1.47	1.35	0.97

Another set of interesting permeation transients was obtained at pH 5.5 and with varying H_2S partial pressure (Figure 3). Unfortunately, these experiments were performed with thin

(0.5 mm) membranes, and it was therefore impossible to extract diffusion parameters. Nevertheless, an interesting trend is observed: under 1 bar H_2S, the permeation current decreases rapidly after the initial rise following immersion. After a few hours, the level of permeation current is 4 times less under 1 bar H_2S than under 100 mbar H_2S. Similar trends have already been observed by Kimura et al. [34]. These authors observed that above a certain H_2S partial pressure, corrosion rate of steel was decreasing, thus leading to a decrease in permeation rate (Figure 4). This decrease in corrosion rate might be caused by a more stable and protective iron sulphide layer.

According to this experimental result we might expect that exposure to pH 5.5 and 1 bar H_2S should be less severe than pH 5.5 and 100 mbar H_2S. This will be verified through long term HIC experiments described in the next part of this paper.

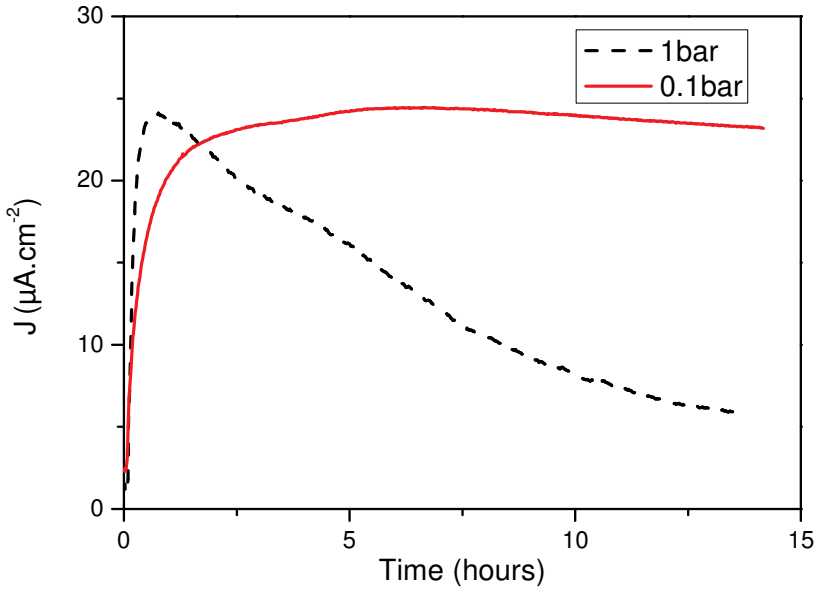


Figure 3 : Hydrogen permeation transient measured for 0.5 mm steel membranes exposed to test solution at pH 5.5 and 1 bar or pH 5.5 and 100 mbar H_2S.

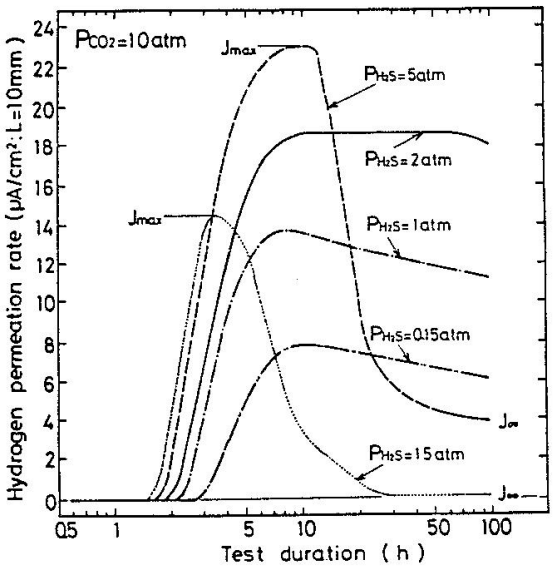


Figure 4 : Hydrogen permeation rate through 10 mm thick API X70 steel membranes in synthetic seawater and at different H_2S partial pressure [34].

2.2. HIC experiments

A great number of HIC experiments were performed. Experimental parameters were pH (varied from 5.5 to 3.5), H_2S partial pressure (10 mbar to 1 bar) and time of exposure (5 hours to 1080 hours). The range of experimental parameters is presented in Table 3.

Table 3 : Range of test durations (hours) of HIC experiments at different pH and pH_2S.

	10 mbar	**100 mbar**	**1 bar**
pH 5.5	96 – 336	24 – 96 – 336	24 – 96 – 120 192 – 336
pH 4.5	96 – 336	24 – 48 – 96 – 336	5 – 8 – 14.5 – 24 44 – 96 – 120 – 336
pH 3.5	96 – 336 – 720 – 1080	24 – 48 – 96 – 336	24 – 96

Figure 5 illustrates ultrasonic evaluations (time-of-flight representation) obtained on HIC triplicates after different time of exposure under 1 bar H_2S and at pH 4.5. HIC extent clearly increases with the time of exposure. We can also notice that most of the cracks are located near the centre line, where the banded pearlite structure was the more pronounced.

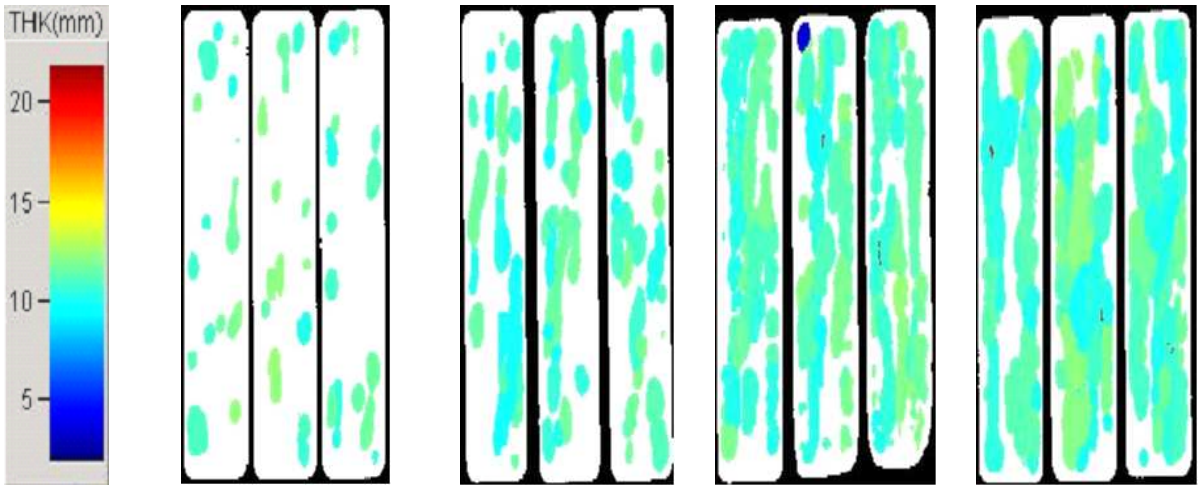


Figure 5 : US-scans after different times of exposure under 1 bar H_2S at pH 4.5.

From all these experimental results, the evolution of CAR with time was analysed. For this purpose, two different representations were adopted. Figure 6 presents CAR evolution at different pH and at constant H_2S partial pressure. Figure 7 presents CAR evolution at different H_2S partial pressure and at constant pH.

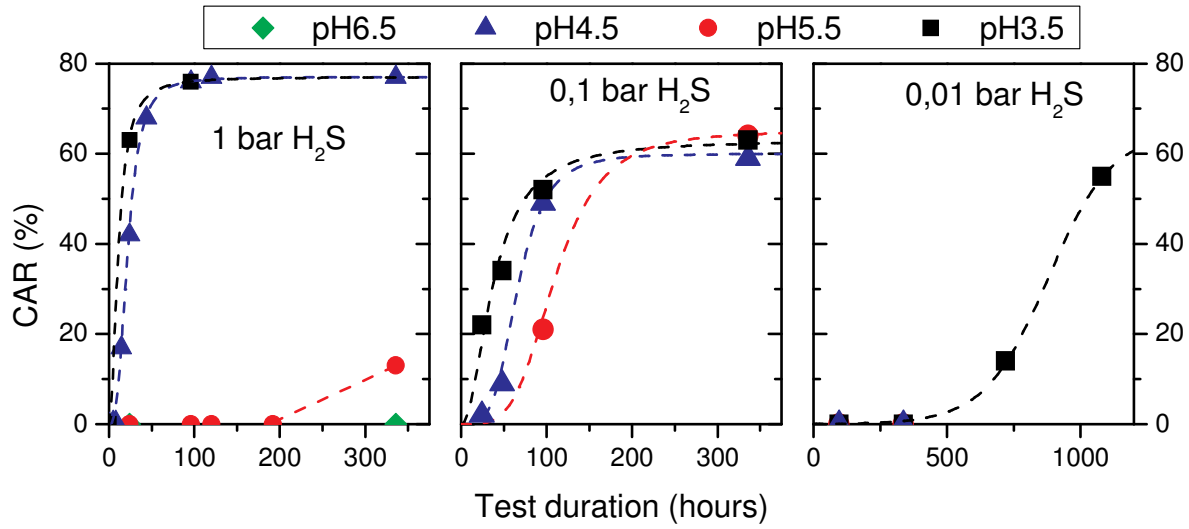


Figure 6 : Evolution of HIC extent with time of exposure. Impact of pH at constant pH_2S.

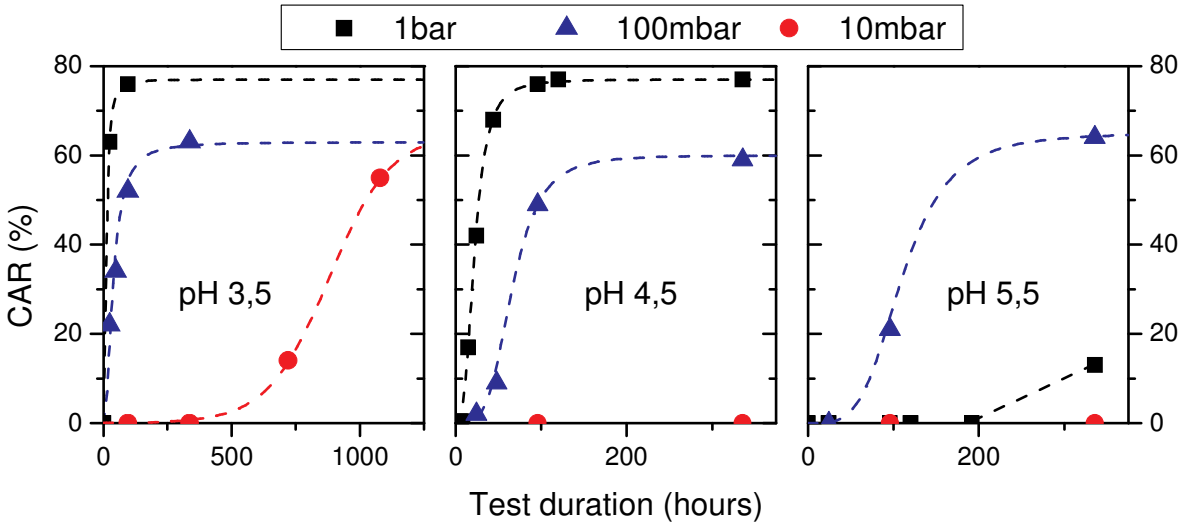


Figure 7 : Evolution of HIC extent with time of exposure. Impact of pH_2S at constant pH.

As already illustrated in a previous paper [11], pH and H_2S partial pressure have a significant impact on the time necessary for HIC to initiate.

At constant H_2S partial pressure (Figure 6), increasing the pH leads to a delayed HIC initiation. This effect is particularly marked under 1 bar H_2S: at pH 4.5, cracks are observed after only a few hours immersion, whereas more than 200 hours are necessary at pH 5.5. The same tendency is observed for the results under 100 mbar H_2S.

It also seems that H_2S partial pressure has a more pronounced influence than pH on the maximum extent of HIC: indeed, under 100 mbar H_2S, the same CAR values are observed for the longer immersion times for the tests at pH 5.5 to 3.5. However, this conclusion requires further work on a range of sweet service materials.

Another interesting result was found for the experiments at pH 5.5 and under 0.1 and 1 bar H_2S. HIC experiments revealed that exposure under 1 bar H_2S at pH 5.5 was far less severe than 0.1 bar H_2S at pH 4.5 (Figure 7). Under 1 bar H_2S, more than 200 hours exposures were

necessary before cracks were observed, while under 0.1 bar H_2S, cracks were detected after only 96 hours exposure. This behaviour is quite unexpected: indeed, it is usually agreed that pH and H_2S partial pressure have a monotone impact on severity, i.e. increase of severity when the pH decreases and H_2S partial pressure increases.

3. DISCUSSION: HYDROGEN DIFFUSION VS. HIC

The objectives of the discussion are to check how permeation data could help for the understanding of HIC evolution results.
The first part of the discussion focuses on experiments under 100 mbar H_2S, for which both permeation on thick membranes and HIC tests are available. Diffusion coefficients determined from permeation experiments were used for modelling hydrogen profiles in HIC specimens, which could then be compared to the results of HIC exposure tests.
The second part of the discussion concerns the unexpected results at pH 5.5, for which cracking occurred much more rapidly under 100 mbar H_2S than under 1 bar H_2S. An explanation to this result could be proposed from permeation data.

3.1. Modelling of hydrogen profiles in HIC samples

For the particular set of experiments under 100 mbar H_2S, permeation measurements allowed us to determine the diffusion coefficients of hydrogen in the steel (Table 2). For the comparison with HIC results, these diffusion parameters were used for the determination of hydrogen concentration profiles in HIC samples at different immersion times. Those calculations were performed using a finite element model software (Comsol multiphysics). The geometry of the samples was considered parallelepipedic (20 mm wide and 24 mm thick) with an infinite length. Hydrogen diffusion was modelled by a pure Fickian behaviour using experimental C_0 and D values. Hydrogen was supposed to enter from all four lateral faces of the samples.

Figure 8 illustrates the evolution of hydrogen concentration with time in the section of a HIC sample at pH 5.5 and with 100 mbar H_2S. Similar calculations were performed for the diffusion parameter determined from the experiments at pH 4.5 and 3.5 (Table 2).

Then, since all ultrasonic evaluations of HIC samples revealed that cracking was located near the mid-thickness, the analysis of hydrogen profile was restricted to the centre line, i.e. at a depth of 12 mm. Evolutions with time of concentration profiles in the centre line are presented in Figure 9. Obviously, hydrogen concentration increases more rapidly at pH 3.5 than at pH 5.5, and the final equilibrium level is also higher.

Finally, for the analysis of this modelling towards HIC, we considered a critical hydrogen concentration, above which cracking might occur. From a previous study using the same steel [11], this threshold hydrogen concentration was found to lie between 0.9 and 1 ppm. This threshold value was reported in Figure 9. For each time increment, we also calculated the average hydrogen concentration in the centre line. The corresponding results are plotted in Figure 10.

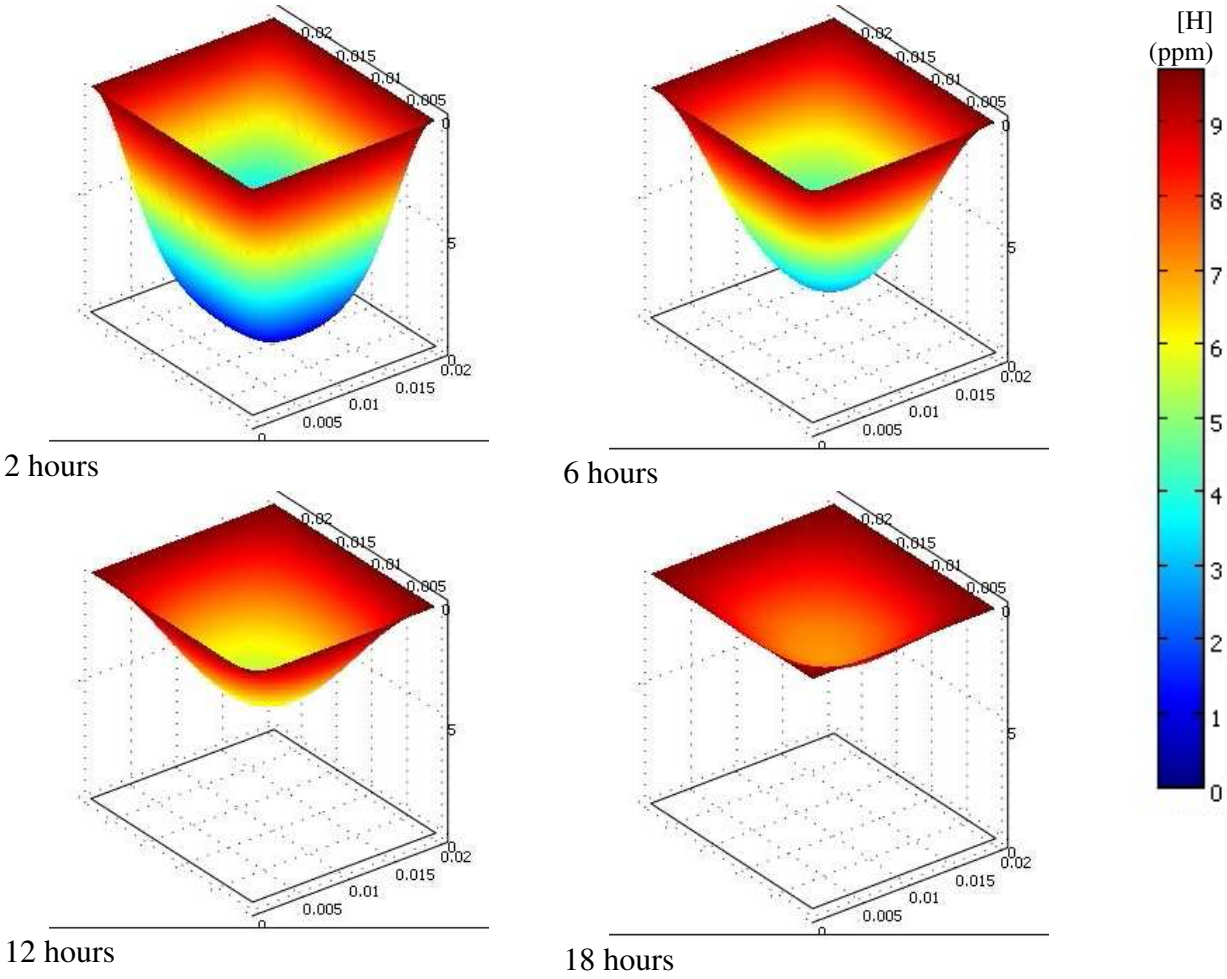


Figure 8 : Calculated hydrogen concentration profile in the 20 mm x 24 mm section of a HIC sample exposed at pH 5.5 and 100 mbar H_2S (D = 7.5 x 10-6 cm².s-1 ; C0 = 0.97 ppm).

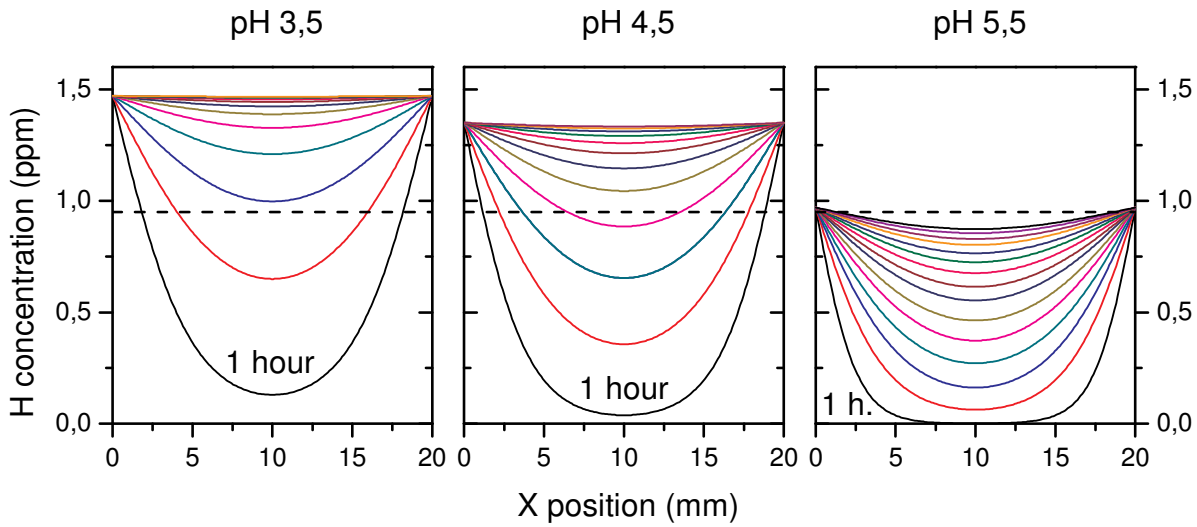


Figure 9 : Modelling of hydrogen concentration profiles in the centre line (Y = 12 mm) of HIC specimens in test solution under 100 mbar H_2S and at different pH, using diffusion constants of Table 2 (time increment between each profile = 2 hours).

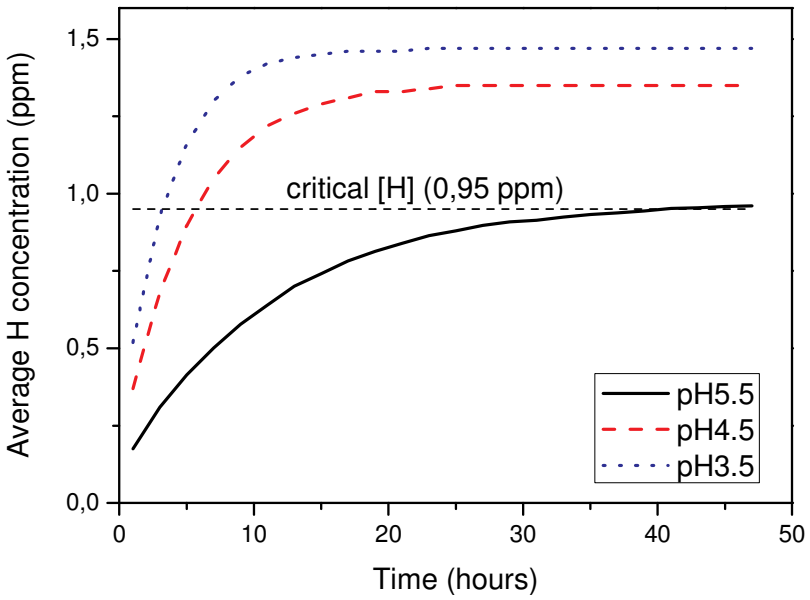


Figure 10 : Average hydrogen concentration in the centre line (Y = 12 mm) of HIC specimens in test solution under 100 mbar H_2S and at different pH, using diffusion constants of Table 2 (time increment between each profile = 2 hours).

For the discussion of time to initiate HIC, comparison between Figure 10 and Figure 6 (results at 100 mbar) is extremely interesting. Modelling of hydrogen diffusion in the centre line of HIC specimens (Figure 10) shows that the average concentration reaches the critical cracking level (0.95 ppm) after 3 hours at pH 3.5, after 7 hours at pH 4.5, and after more than 40 hours at pH 5.5. On the other hand, the evolution of HIC extent with time (Figure 6) reveals that cracks are observed after only a few hours immersion under 0.1 bar H_2S at pH 3.5 and 4.5, while it takes between 24 and 96 hours at pH 5.5. These values are in very close agreement with the timescales obtained from permeation data and diffusion modelling.

This result suggests that permeation measurements might represent an interesting method for the optimisation of HIC testing procedures. From permeation data, it is possible to determine diffusion coefficients, which can then be used for calculations of hydrogen profiles in HIC samples, and thus give an indicative value of the minimum time of exposure necessary to reach a critical hydrogen concentration level. Additionally, when permeation data shows hydrogen subsurface concentration below the critical level, it might become useless to perform HIC tests.

3.2. Evaluation of severity from permeation experiments

The second interesting comparison between HIC and permeation tests lies in the experiments at pH 5.5 and under 100 mbar and 1 bar H_2S. HIC experiments revealed that exposure under 1 bar H_2S at pH 5.5 was far less severe than 0.1 bar H_2S at pH 4.5 (Figure 7). Under 1 bar H_2S, more than 200 hours exposures were necessary before cracks were observed, while under 100 mbar H_2S, cracks were detected after only 96 hours exposure. At the same time, permeation current measured at pH 5.5 under 1 bar H_2S rapidly decreased well below the values under 100 mbar H_2S (Figure 3). This decrease might probably be explained by the build-up of a more protective iron sulphide layer at 1 bar H_2S, and a reduced corrosion rate and hydrogen reduction reaction. Consequently, hydrogen charging in the metal takes more time under 1 bar H_2S, and the time to reach the critical hydrogen concentration is strongly delayed.

This result suggests that permeation measurements could be used in order to define pH – pH_2S severity regions as already suggested by Duval et al. [35].

CONCLUSIONS

These results confirm that HIC assessment of sweet service linepipe steels in mild sour environments requires precautions. For the lower H_2S partial pressure and high pH combination, more than 4 weeks might be necessary before HIC initiation. Therefore, it might become necessary to adopt long term immersion tests for the qualification of sweet service steels in terms of HIC resistance.
For this purpose, permeation measurements might be an interesting and complementary experimental technique. The determination of diffusion coefficients can be used for calculations of hydrogen profiles in HIC samples, and give a correct evaluation of the minimum time of exposure necessary to reach a critical hydrogen concentration level. Additionally, when permeation data shows hydrogen subsurface concentration below the critical level, it might become useless to perform HIC tests.

Another important result is the fact that pH and H_2S partial pressure do not always have a monotone impact on severity, i.e. increase of severity when the pH decreases and H_2S partial pressure increases. Indeed, some results of this study showed faster cracking under 100 mbar H_2S than under 1 bar H_2S (at pH 5.5). This is probably linked with the build-up of a more protective iron sulphide layer under 1 bar H_2S. Once again, this result which required more than 2 weeks HIC testing could have been predicted from 15 hours permeation measurements in both environments. Permeation technique could therefore represent an interesting means to investigate the impact of environmental parameters on HIC.

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