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Hydrogen induced cracking (HIC) assessment of low alloy steel linepipe for sour service application–Laboratory testing

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Report on behalf of the EPRG Corrosion Committee

Abstract

As a first step, a literature survey was undertaken on Hydrogen Induced Cracking (HIC) phenomena. Material and metallurgical parameters were investigated. In addition the various testing environments and criteria used for fit-for-purpose (FFP) testing were assessed. The literature sources suggested a variety of methodologies that can be used for FFP HIC testing. However, a universally applicable FFP test method was not identified. Therefore, an experimental study was launched to establish a suitable methodology and to better define the key parameters involved in HIC phenomena. The steel used for these studies was an API5L X65 sweet service grade. Experiments consisted of HIC exposure tests based on the NACE TM0284-2003 Standard method. Parameters studied were pH (3.5 to 5.5), H₂S partial pressure (10 to 100mbar), and time of exposure (96 hours to 1 month). Comparisons between one-sided and six-sided exposure of HIC specimens were also undertaken. Characterisation included hydrogen content measurement for diffusible hydrogen (glycerol method). HIC cracking was evaluated both by ultrasonic testing and metallographic examination in accordance with the NACE TM0284-2003 Standard.

From the HIC exposure tests, a good correlation was found between diffusible hydrogen and HIC. When the diffusible hydrogen concentration was below 1ppm, no HIC was found. Above 1ppm diffusible hydrogen, all specimens exhibited severe HIC.

For specimens tested at a level of 10mbar H₂S no HIC was observed after 96 hours exposure. But tests with longer exposure (4 weeks) did result in HIC. This suggests that the 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. Considering the impact of the environment, H₂S partial pressure seemed to have a much greater impact than pH on the extent of HIC.

These results suggest that a “regions of environmental severity” diagram, similar to that developed for sulphide stress cracking in the 1990’s and later incorporated into NACE MR0175/ISO 15156, can be developed for the HIC resistance of steels. This relates the HIC resistance to the two-dimensional environmental severity defined by the partial pressure of H₂S and the pH. This diagram will enable the mapping of HIC according to cracking susceptibility and help in defining the ‘HIC sour service’ limits for line-pipe steels.

Keywords

HIC, sour environment, steel

Introduction / Background

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), sulphide stress cracking (SSC) and stress-oriented hydrogen-induced cracking (SOHIC). The presence of liquid water necessary for initiation of these mechanisms can occur even if the gas temperature in the pipeline is above the water dew point, i.e. due to the lower temperature of the pipe wall compared to the gas temperature or where the reduction of gas temperature along the pipeline results in the water vapour of the gas to condense. Due to the possible sudden, unforeseeable nature of such cracking mechanisms, this type of damage is regarded as more dangerous than weight-loss corrosion.

The classification of sour service carbon/low alloy steel materials within regions of environmental severity (Figure 1), as already defined for sulphide stress cracking (SSC) in ISO 15156-2 [1], could provide a tool for accurate assessment of HIC susceptibility. However, it needs to be recognised that the pH – pH_2S severity regions for SSC and HIC do not necessarily match. Thus, Figure 1 is suitable for SSC resistance only and a similar plot still has to be developed for HIC and related mechanisms. In addition, ISO 15156-2 does not place any lower H_2S limit below which an assessment of HIC resistance is unnecessary. The definition of sour service in ISO 15156-2 is not restricted to conditions above 3.5 mbar H_2S , as was the case in the previous editions of NACE MR0175, which considered only SSC resistance. Therefore, for the definition of the boundaries in Figure 1 for HIC, environmental conditions with H_2S partial pressure at and below 3.5 mbar (i.e. region 0) have also to be taken into account. As the standard HIC test is performed under severe conditions it is frequently not an adequate method to qualify material for mildly sour conditions. There is a strong need for new or modified test conditions/methods with respect to the different operating requirements (fit-for-purpose testing).

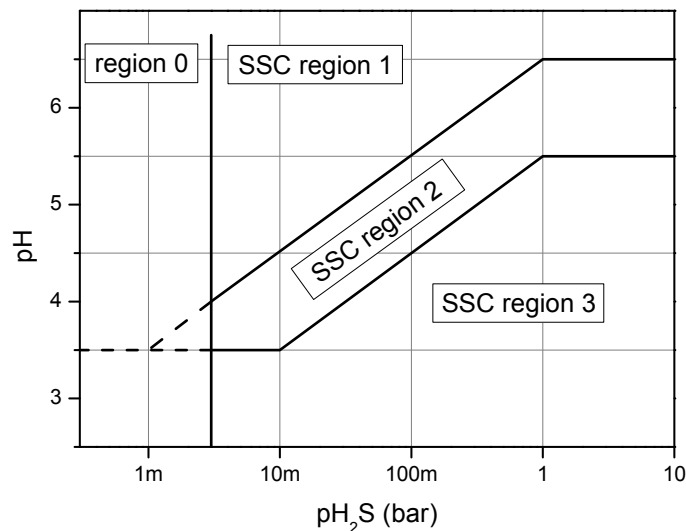


Figure 1: SSC regions of environmental severity according to ISO 15156-2.

As a first step of this study by the EPRG Corrosion Committee, a literature survey was undertaken on Hydrogen Induced Cracking (HIC) phenomena [2]. Material and metallurgical parameters were investigated; in addition the various testing environments and criteria used for fit-for-purpose testing (FFP) were assessed. The literature sources suggested a variety of methodologies that could be used for FFP HIC testing. It was stated that hydrogen permeation measurements in general can provide a quantitative measure for differentiating HIC

performance. Field measurements of the maximum concentration of dissolved hydrogen in steel could also be helpful for FFP classification. However, a universally applicable FFP test method was not identified.

The clarification of the environmental boundary conditions is considered to be the key to FFP testing. A HIC matrix, similar to the ISO 15156-2 definition of SSC regions dependant on H₂S partial pressure and pH, can be obtained by hydrogen permeation measurements. To perform FFP laboratory testing, the test environment must represent the specific operating environment. This can be obtained by the variation of the test severity using modified test set-ups (e.g. the modification of number of exposed specimen sides, H₂S content, specimen type). For acceptable HIC testing, the transferability of the results to the actual field conditions must be achieved. It has been demonstrated that the surface concentration of diffusing hydrogen atoms on operating pipelines reaches a peak value within a few days, such that in order to match the operating environment the test environment must reach the same peak value. It was finally stated that full-scale test results do not necessarily correlate with laboratory test results. In particular, tests involving six-sided hydrogen charging can not be correlated with the performance of the pipes under service conditions. During the laboratory test, the hydrogen activity remains constant over the specimen thickness, whereas under service conditions, there is a linear decrease from the inside to the outside of the pipe wall.

Starting from these insights, an experimental study was launched based on laboratory tests derived from the above mentioned techniques. The objective was to establish a suitable methodology and to better define the key parameters involved in HIC phenomena. A reference steel type (API5L X65 non sour service) was studied. Experiments consisted of HIC exposure tests based on the NACE TM0284-2003 Standard method [3]. Parameters studied were pH (3.5 to 5.5), H₂S partial pressure (10 to 100mbar), and time of exposure (96 hours to 1 month). Comparisons between one-sided (more related with field operating conditions) and six-sided exposure of HIC specimens were also undertaken. Characterisation included hydrogen content measurement for diffusible hydrogen by the glycerol method. HIC cracking was evaluated both by ultrasonic testing and metallographic examination to NACE TM0284-2003.

Material tested during the study

The material selected for the experimental program was a non sour service API5L X65 grade. Specimens were extracted from a LSAW pipe, 42 inches diameter and 1 inch thick. No welds were included in the tested specimens, only the base metal was tested. The chemical composition was analysed by optical emission spectrometry (EOM), except for carbon and sulfur, which were analysed by a chemical method (Table 1).

Table 1: Comparison of chemical compositions (wt %) of tested material.

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	0,040	0,050

The microstructure was examined using a DM4000 Leica optical microscope, after polishing with a 0.05µm finishing suspension and etching with a 2% Nital (nitric acid - ethanol) solution. The corresponding microstructures at the mid wall location are presented in Figure 2.

The microstructure is a typical ferrite – pearlite type. The grain size is between 10 and 30µm and the microstructure is highly oriented in the rolling direction.

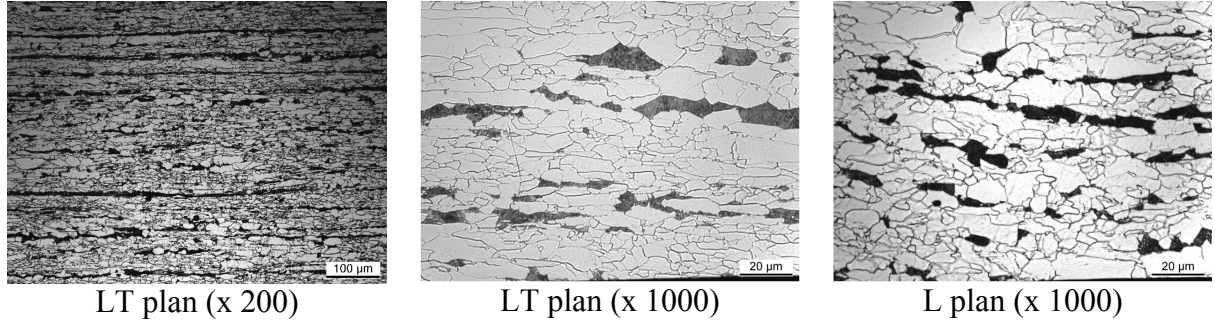


Figure 2: microstructure (LT plan) of the tested steels.

Table 2 presents the tensile properties in the direction longitudinal to rolling, measured according to NF EN10002-1. The inclusion contents analysed according to ASTM E45 method A are presented in Table 3.

Table 2: Mechanical and microstructural properties of tested material.

$R_{p0.2}$ (MPa)	R_m (MPa)	A (%)
523	649	24

Table 3: Inclusion content of tested material, according to ASTM E45 method A.

Type A (sulfides)		Type B (aluminates)		Type C (silicates)		Type D (oxides)	
Thin	Heavy	Thin	Heavy	Thin	Heavy	Thin	Heavy
0	0	0	0	0	0	0.5	0.5

Experimental methods

Test solution and exposure conditions

In order to compare all sides and 1-side exposure, a polypropylene test vessel was designed to contain three 6-sided and three 1-sided specimens. Specimens of the ‘all-sides exposure’ type were fully immersed in the test solution. Specimens for the 1-sided exposure were exposed on a single side through a 100mm x 20mm windows in an IFP tailor made test vessel (Figure 3).

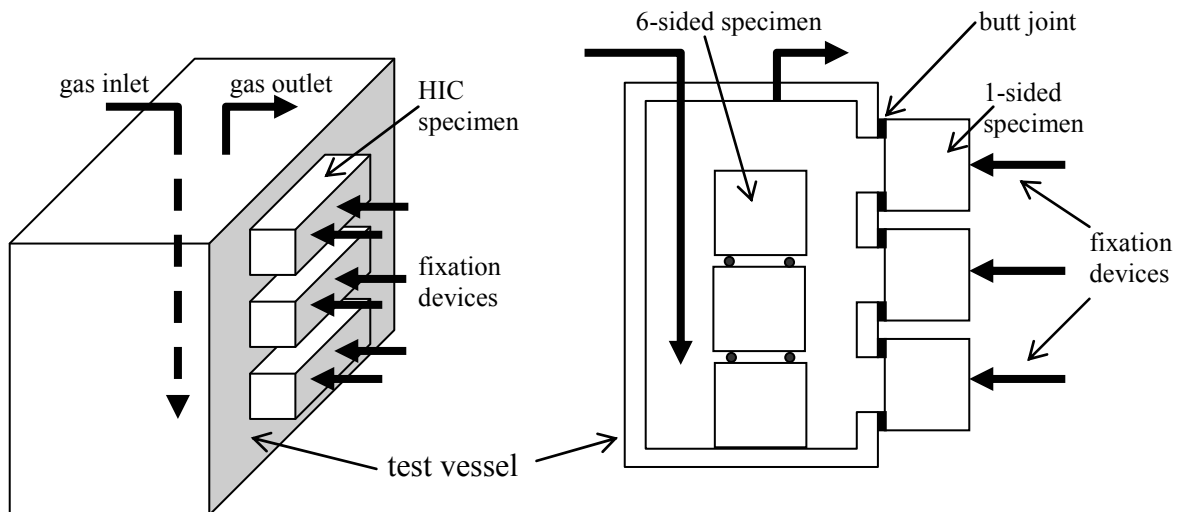


Figure 3: Test vessel design for 1-sided exposed HIC specimens.

The experimental parameters studied were pH (3.5, 4.5 and 5.5) and H₂S partial pressure (10, 35 and 100mbar). The impact of the exposure time was also evaluated: longer exposures were performed for the "low severity" condition (i.e. 4 weeks at 10mbar H₂S and pH 3.5), while the higher pH_{H2S} were tested for 2 weeks and 96 hours.

All HIC tests were performed following the NACE TM0284-2003 recommendations, including:

- three replicates for each condition,
- volume / surface ratio greater than 3mL/cm²

The test solutions were prepared according to EFC16, using sodium acetate buffer, and the pH was controlled and adjusted during the experiments.

HIC specimens preparation

HIC specimens for 6-sides exposure were sampled following the NACE TM0284-2003 standard. The sample dimensions were 100mm in the longitudinal direction, 20mm wide and the same thickness as the base material (0.5 to 1mm were removed from the inner and outer parts of the surfaces). All faces were machined to obtain a finish equivalent to 320 grit.

Specific specimens for 1-side exposure were also prepared. Such specimens were 106mm long by 26mm wide. The face exposed to the test solution was machined to obtain a finish equivalent to 320 grit.

HIC evaluation by metallography and ultrasonic testing

Two methods were used for the determination of HIC extent.

The first consisted of the standard NACE TM0284-2003 practice, with three equidistant cuts along the length and metallographic examination of the cut faces.

For the other method, ultrasonic inspection was used. 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.

Hydrogen concentration

The determination of diffusible hydrogen concentration in the steel specimens was performed using the following procedure. Immediately after removal of the specimen from the test solution, it was rinsed with tap water and wiped with paper, then immediately immersed in 45°C glycerol under a collector tube. The time gap between specimen sampling and immersion in glycerol was always less than 2 minutes. The diffusible hydrogen was then measured by the displacement of glycerol after one week immersion at 45°C. The experimental error associated with free hydrogen measurement is 0.1mL. The repeatability was evaluated by comparing three identical specimens. The standard deviation was always less than 20%.

Results and Discussion

Hydrogen concentration

Table 4 summarizes the diffusible hydrogen content measurements performed after all tests. The hydrogen content is expressed in mass ppm, i.e. in mg of hydrogen per kg of steel. The impact of pH is illustrated in Figure 4 for 1-sided and 6-sided exposure and for various pH₂S and exposure times.

Table 4: Diffusible hydrogen (mass ppm) measured by the glycerol method after each experiment.

		10mbar		35mbar	100mbar	
		1-sided	6-sided	6-sided	1-sided	6-sided
5.5	96 hours	0.1	0.7	/	0.5	1.5
	2 weeks	0.3	1.0	/	0.8	1.5 - 2.1
4.5	96 hours	0.1	0.7	/	0.4	1.3
	2 weeks	0.4	0.9	1.1	0.6	1.5 - 1.2
3.5	96 hours	0.3	0.8	/	0.7	2
	2 weeks	/	/	1.5	/	1.7
	1 month	/	1.0	/	/	/
2.7	2 weeks	/	0.9	/	/	1.3

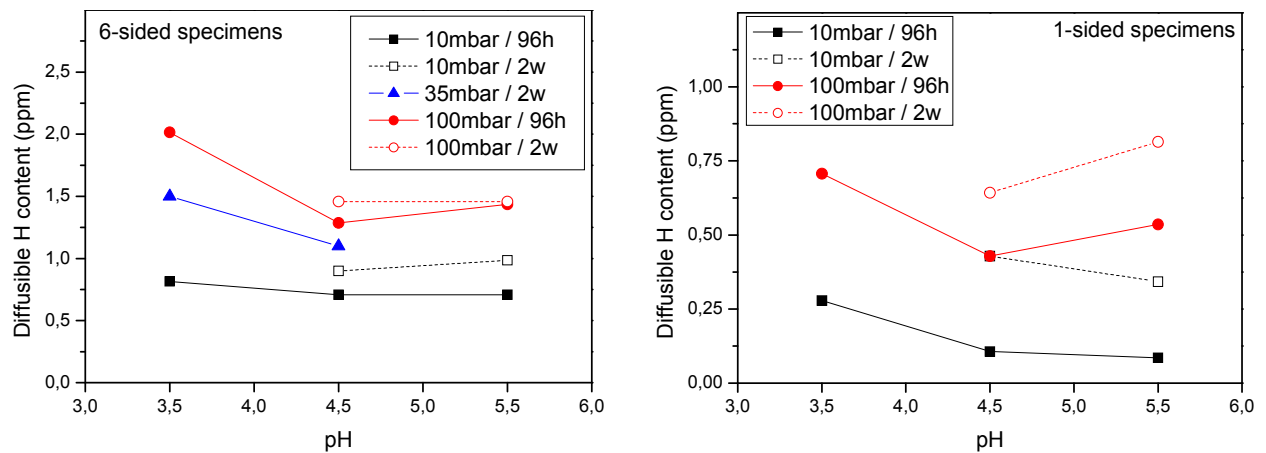


Figure 4: Impact of pH on diffusible hydrogen content (glycerol method) after HIC experiments of 96 hours and 2 weeks. Comparisons between 1-sided and 6-sided exposures.

The following features were apparent from the hydrogen content measurements:

- For the 6-sided specimens exposed to 100mbar H₂S, the hydrogen content hardly increased when the immersion time was increased from 96 hours to 2 weeks; it is therefore assumed that the equilibrium was already reached after 96 hours.
- On the other hand, for the 1-sided specimens, the hydrogen content increased significantly when the immersion time was increased from 96 hours to 2 weeks. There are two possible contributory causes for this: 1/ a slower hydrogen entry by 1 face instead of 6 faces; 2/ hydrogen effusion by the 5 non exposed faces. As a result, the hydrogen content in the 1-sided specimens after 2 weeks was two to three times lower than in the 6-sided specimens. Considering the concentration gradient in the 1-sided specimens, one would theoretically expect a concentration at equilibrium a half that in the 6-sided specimen for a purely transverse diffusion and with a perfect extraction of

hydrogen at the exit face. The actual case is more complicated, as hydrogen might also leave the specimen from the lateral faces in the 1-sided specimen. It is therefore difficult to estimate if the concentration in the 1-sided specimens reached after 2 weeks is far from equilibrium or not. A better representation of field conditions would be achieved with 1-sided specimens of larger dimensions, to minimise effusion from lateral faces. Protection of lateral faces by a suitable coating could have the same effect.

- The most significant influencing parameter with respect to the hydrogen content of the steel was the H_2S partial pressure rather than the pH, as illustrated by the diffusible hydrogen content graphs: the H content – pH curves are rather flat, while the increase in diffusible H is significant when the H_2S partial pressure is raised from 10mbar to 100mbar. A pH influence is only seen when low values are reached, i.e. pH 3.5. A recent study by Hara et al. [4] investigated the impact of pH and pH_2S on the diffusible hydrogen content in X65 linepipe steels. It was found that the effect of pH was one third smaller than that of pH_2S , which is in good qualitative agreement with the findings of this study.

HIC extent

CAR values from US tests and CLR values from metallographic cuts are reported respectively in Table 5 and in Table 6.

Table 5: CAR determined from US scans for all test conditions.

		10mbar		35mbar	100mbar	
		1-sided	6-sided	6-sided	1-sided	6-sided
5.5	96 hours	0	0	/	0	21
	2 weeks	0	0	/	0	62 - 66
4.5	96 hours	0	0	/	0	49
	2 weeks	0	0	32	0	61 - 56
3.5	96 hours	0	0	/	0	52
	2 weeks	/	/	75	/	63
	1 month	/	14	/	/	/
	2 weeks	/	0	/	/	48

Table 6: CLR determined by metallographic examinations according to NACE TM0284-2003.

		10mbar		35mbar	100mbar	
		1-sided	6-sided	6-sided	1-sided	6-sided
5.5	96 hours	0	0	/	0	6
	2 weeks	0	0	/	0	34 - 38
4.5	96 hours	0	0	/	0	13
	2 weeks	0	0	21	0	35 - 33
3.5	96 hours	0	0	/	0	44
	2 weeks	/	/	46	/	38
	1 month	/	11	/	/	/
2.7	2 weeks	/	0	/	/	21

Before discussing in details the influence of different experimental parameters, a comment should be made on the comparison between metallographic and ultrasonic methods for HIC evaluation. Figure 5 presents the correlation graph between CAR and CLR for all the

experiments. Each point in the graph represents the CAR and CLR (average of three cross-sections) measured for one individual specimen. Even though no strict linear correlation was found between CAR and CLR for the specimens with HIC, the quality of the information was the same with both techniques. Therefore, the discussion of the results of this work is based only on CAR values.

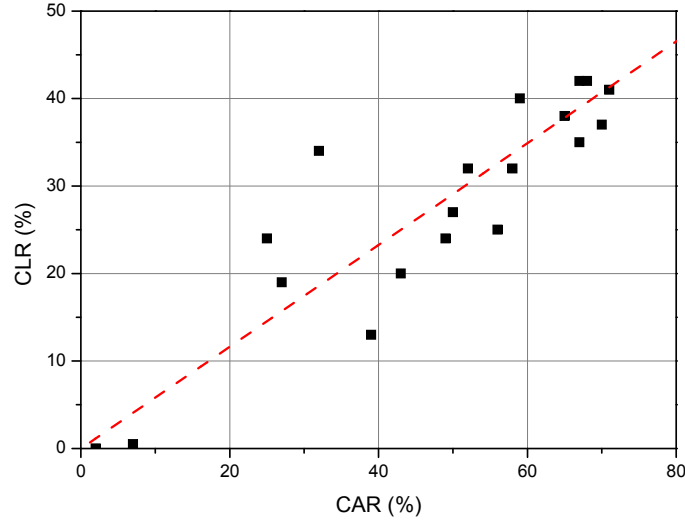


Figure 5: Correlation graph between CAR and CLR for all specimens after HIC experiments.

The impact on CAR of pH and of H_2S partial pressure are compared in Figure 6. The level of H_2S partial pressure was found to be extremely important. Indeed, decreasing the partial pressure of H_2S from 100mbar to 10mbar drastically decreased the risks of HIC. On the other hand, the impact of test solution pH seemed to be less pronounced. For the tests under 100mbar H_2S , only the higher pH with the shorter exposure time resulted in a decrease of CAR. Thus, contrary to the case for H_2S partial pressure, there does not seem to be a clear pH threshold above which no HIC is expected in the pH range 3.5 – 5.5. Even though no tests were performed at high pH and high H_2S during the present study, similar experiments were described in the literature [5], and CAR values above 5% were still observed after 4 days testing at pH 6.2 under 1bar H_2S , on a steel grade close to the one used in the present work.

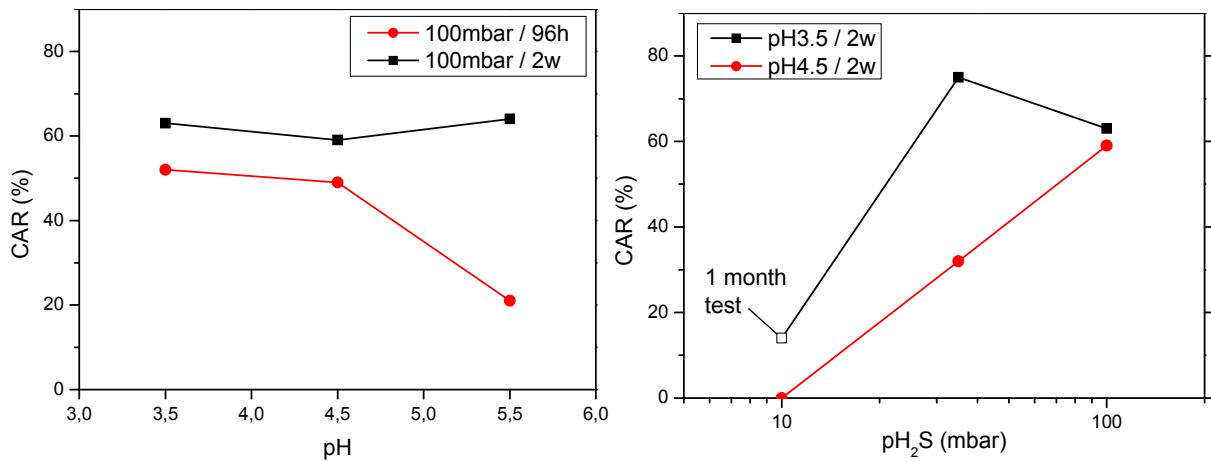


Figure 6: Impact of pH and H_2S partial pressure on CAR for different experiments.

The following conclusions about the HIC results can be drawn:

- None of the 1-sided specimen exhibited cracking, as determined by UT or metallographic examination.
- When HIC was observed, all the cracks were located near the mid wall region of the specimen.
- Considering solely the experiments on the 6-sided specimens tested in solutions with 100mbar H_2S , the crack ratios increased with the increasing acidity (lowering pH) of the test solution.
- As was observed for hydrogen content, H_2S partial pressure seemed to have a greater impact than pH.
- For the tests under 100mbar H_2S , increasing the test duration resulted in an increase in crack ratio, the greater for the high pH values. For the low pH_2S (10mbar) and at pH 3.5, increasing the test duration from 96 hours to 4 weeks did result in cracking.
- For all of the tested specimens, UT and metallographic techniques were always in agreement considering the occurrence or the absence of cracks.

HIC and Hydrogen concentration

In order to check if there was a correlation between the extent of HIC and the amount of hydrogen in the material, the correlation graph between CAR and diffusible hydrogen was drawn for all the experiments (Figure 7).

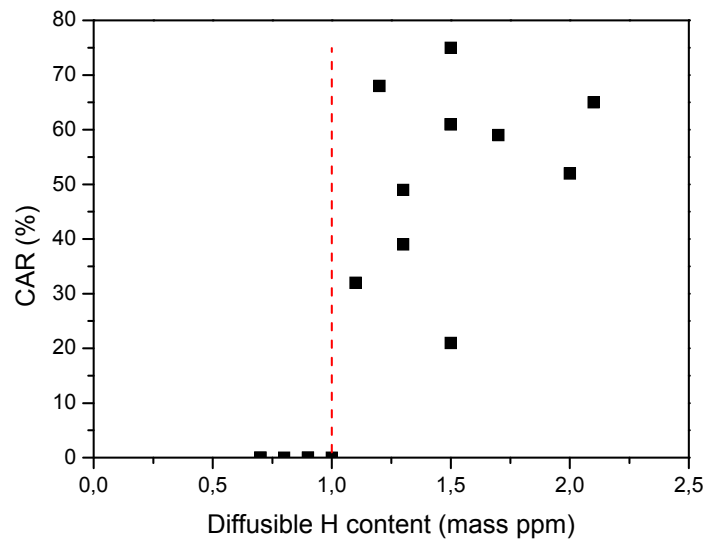


Figure 7: Correlation graph between CAR and diffusible hydrogen for all HIC experiments.

Though there is no strict correlation between CAR and H content, it is still possible to distinguish a threshold hydrogen concentration:

- below 1ppm of diffusible hydrogen, none of the specimens suffered HIC,
- above 1ppm, all specimens exhibited high HIC, with CAR values from 10% to 75%.

These results are in good agreement with previous tests using the same diffusible hydrogen content methodology. Revie et al. [6] studied the HIC resistance of 8 steel grades exposed to buffered solution from pH 1.1 to 5.9 saturated with H_2S . They determined a threshold hydrogen concentration above which HIC occurred. For 4 steel grades out of 5, this threshold hydrogen concentration varied from 0.2 to 0.9mL/100g (standard pressure and temperature),

which corresponds to 0.2 to 0.8ppm, in excellent agreement with the 1ppm limit found for the steel of this study.

Another study recently reported by Hara et al. [4] on HIC evaluation of X65 linepipe steels gave similar CAR / hydrogen concentration trends: for most of the steels studied (8 types of X65 linepipe) the critical hydrogen concentration was between 0.6 and 2ppm.

It is therefore possible to consider that diffusible hydrogen content might be a good indicator of the risks of HIC. However, it seems that the critical hydrogen concentration is not "universal", but rather depends on the steel considered. Establishing clear acceptance criteria for diffusible hydrogen concentration would require further investigation. In particular, experiments on full size pipe samples and investigations on the impact of small levels of HIC on pipe integrity must be addressed.

HIC severity diagram

From these results, a preliminary HIC severity diagram was created in a similar manner to the SSC regions of ISO 15156-2. The following convention was adopted in order to classify CAR values into different categories:

- CAR < 1% no or little HIC
- $1\% \leq \text{CAR} < 20\%$ light to medium HIC
- CAR $\geq 20\%$ strong HIC

It has to be pointed out that these values were chosen essentially for an average distribution of the results of the present study in the different categories. At this stage, it was not the purpose to propose "acceptance criteria".

For comparison, the SSC regions of ISO 15156-2 are also indicated on the graphs (Figure 8).

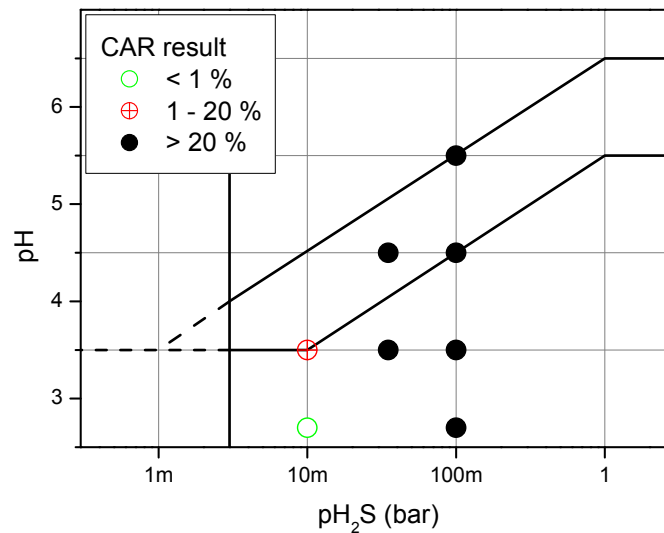


Figure 8: Representation of CAR results in the pH-pH₂S diagram. Exposure time was 2 weeks for all experiments, except at pH 3.5 – 10mbar (4 weeks).

From the results obtained during this study, a preliminary attempt for the definition of HIC severity domains was proposed.

One of the more important conclusions concerns the impact of pH₂S, which always had a greater influence on HIC than pH. This confirms previous findings [5], but is also in contradiction with the well established thoughts that HIC is only linked with the hydrogen activity in the system, and therefore with the test solution pH.

At this stage of the investigation, it seems reasonable to propose incorporating a lower threshold for H_2S partial pressure in a HIC severity diagram, below which HIC is not a practical concern. From the results of this work, the only indication is that this lower H_2S partial pressure threshold could be below 10mbar. More experiments with lower pH_2S and long exposure times are required in order to give a more precise definition of this part of the diagram. But for such low severity conditions, the reasonable exposure time for FFP testing has to be addressed. At the same time, the relevance of 6-sided exposure to field conditions is an open topic.

The opportunity to incorporate a diagonal threshold line in a HIC diagram is also questionable. The SSC diagonal limit of slope 1 between (1mbar H_2S ; pH 3.5) and (1bar H_2S ; pH 6.5) does not seem to be well adapted to HIC. However, if no diagonal threshold was used, the impact of pH would then be completely discarded. This option could be excessive, it was seen that pH did have an impact on HIC, albeit a small effect. The diagonal threshold for HIC could link (3mbar H_2S ; pH 3.5) and (100mbar H_2S ; pH 6.5). During this work, however, no experiments above this diagonal line were performed. More detailed testing will be needed, including an investigation of the impact of test duration. Concerning the latter, it has to be recalled that the tested steel did not suffer HIC after 4 days exposure at pH 3.5 and 10mbar H_2S whereas 4 weeks exposure in the same conditions resulted in 14% CAR. HIC in low severity conditions is therefore not "instantaneous", and several days or weeks might be necessary to initiate cracking. This could be related to low permeation rates observed at low pH_2S such it takes more time to reach the critical hydrogen content for HIC. For this reason and at this very first stage of HIC domains proposal, it is recommended that HIC tests in low H_2S conditions should be extended to at least 1 month exposure. For more severe conditions, 2 weeks exposure could be acceptable.

These preliminary recommendations still require further investigation. Within the present work, only a few experiments were performed in 'regions 0-1', as defined below (Figure 9). A wider range of steels will also have to be tested. Additionally, full scale experiments will be necessary for a better definition of acceptance criteria, especially in the case of US testing, and also to re-define the necessary exposure times of HIC tests.

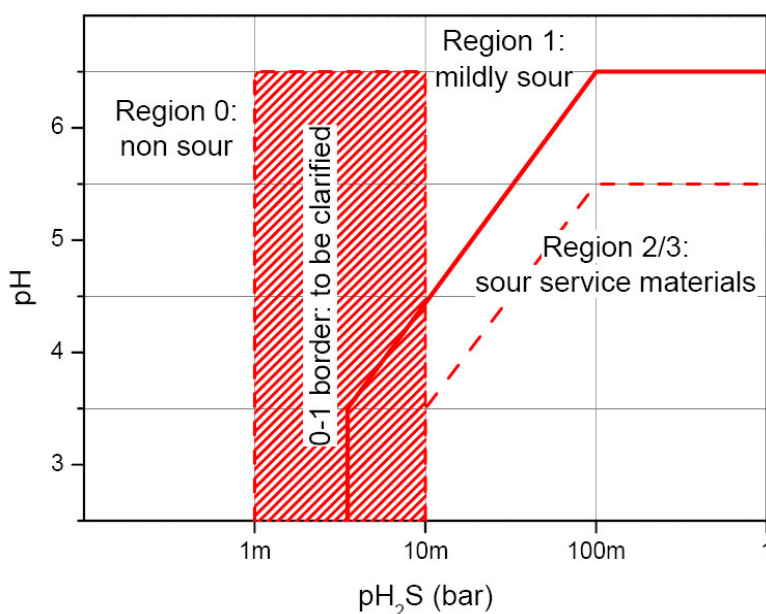


Figure 9: Proposal of HIC severity regions.

At this stage, a very preliminary HIC severity diagram could be proposed, with four distinct regions (Figure 9):

- Region 0: non sour. No specific steels are required. With a less conservative option (and with more data), an alternative H_2S threshold could be chosen between 1 and 10mbar.
- Region 1: mildly sour region. At the present stage of investigations, it is recommended that longer term (e.g. 4 weeks) tests are required to demonstrate suitability in terms of HIC resistance in this region. If it was confirmed by further investigations on different steel grades that no HIC was found in region 1, then it might be possible to put Region 1 together with region 0.
- Region 2: transition region. It is recommended to perform 'application specific' HIC exposure tests for the qualification of steels for use in these conditions.
- Region 3: sour region. It is mandatory to use sour service materials.

Conclusions

A test methodology has been developed for the assessment of the HIC resistance of linepipe steels for 'mild sour environments'. These tests are generally in accordance with NACE TM0284-2003, with the following features:

- Testing period of two weeks, although longer term tests were required to initiate any cracking for lower severity conditions, e.g. 4 weeks at 10mbar H_2S and pH 3.5.
- Six-sided exposure, to ensure attainment of equilibrium levels of hydrogen within the testing period (with one-sided testing, equilibrium could not be ensured).
- The test solution parameters varied were pH (buffered values of 3.5, 4.5 and 5.5) and H_2S partial pressure (10mbar, 35mbar and 100mbar)
- Measurements of the diffusible hydrogen were made in samples at the end of the HIC test.
- HIC cracking was evaluated using both ultrasonic testing and metallographic examination in accordance with NACE TM0284-2003.

One reference steel type (API5L X65) was evaluated, and the following were noted:

- A good correlation was found between diffusible hydrogen and the extent of HIC.
 - For a diffusible hydrogen level of below 1ppm no HIC was observed.
 - For a diffusible hydrogen level of above 1ppm all samples exhibited high extents of HIC, with CAR values of between 21% and 75%.

These values are in good agreement with previously published values and indicate that diffusible hydrogen may be a good indicator of HIC resistance.

- H_2S partial pressure had a far greater influence on the extent of HIC (and indeed the levels of diffusible hydrogen) than did the pH. Increasing the partial pressure of H_2S and, to a lesser extent, decreasing the pH resulted in an increased risk/extent of HIC.

From these results a preliminary HIC severity diagram, synonymous to the SSC severity diagram in ISO 15156-2, has been proposed (Figure 9) with the following Regions:

- Region 0, non sour, where no special precautions are required.
- Region 1, mildly sour. Until further experiments are made available, longer term (e.g. 4 weeks) tests are required to demonstrate suitability in terms of HIC resistance.
- Region 2, transition, where HIC testing with a test period of two weeks is sufficient to qualify materials.
- Region 3, sour, where sour service materials must be used.

It should be recognised that this HIC severity diagram has been generated on testing on a single material such that further testing is required on a range of 'non sour' materials to confirm the limits of the regions, and especially the boundary between regions 0/1 and 2/3.

A problem that has been identified during this study is that the requirement in ISO 15156-2 for an acceptance criteria of 'no cracking' for HIC testing under application specific environmental conditions would be extremely difficult to conform to, even under the most mild sour conditions, and hence requires further study. One possibility is to investigate this in a further Phase of the EPRG programme, with a view to generating acceptance criteria that could be applied to the more mild sour conditions frequently associated with 'application specific' test conditions. This would also enable further refinement of the HIC severity diagram.

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