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PERFORMANCE OF PIPELINE STEELS IN SOUR SERVICE

M. Elboujdaini and R.W. Revie
CANMET Materials Technology Laboratory, 568 Booth Street, Ottawa, Ontario, Canada K1A 0G1

ABSTRACT

The demand for steel for the production of pipelines to transport gas and oil containing hydrogen sulphide prompted the development of steel that is resistant to hydrogen induced cracking (HIC).

During the past two decades, combined research efforts in the areas of product and process metallurgy have made it possible to satisfy most of the main requirements for grades X-42 and X-60 microalloyed steel for mildly acidic (pH = 5) H_2S environments. Building on the experience acquired in the area of microalloyed steel for a mildly acidic (pH ~ 5) H_2S environment, the industry launched a program to develop steel that would satisfy new requirements for H_2S-resistant pipelines under NACE conditions (TM0177, pH~3). In order to develop these steels, it was necessary to define qualitatively and quantitatively the specific effects on H_2S resistance of the multiple intrinsic parameters of the product itself as well as those resulting from the process.

In this paper, data will be presented that have made it possible to relate the HIC performance of steels to chemical content, inclusion levels and thermomechanical treatment parameters.

INTRODUCTION

Hydrogen-induced cracking (HIC) of carbon steel and low-alloy steels can occur in acidic environments (wet H_2S). It is known [1] that HIC develops when hydrogen concentration (C_o) in the steel matrix exceeds the threshold hydrogen concentration (C_{th}). C_o depends on alloy composition, H_2S partial pressure, and pH; C_{th} depends on inclusions and segregation in the matrix [2-4].

Wet H_2S is acidic and corrodes steel, charging it with hydrogen that can cause cracking and blistering. HIC of pipelines, tubing and other steel structures used in sour environments (wet H_2S) have been reported for many years. In terms of a simple test, hardness was identified as the main indicator that correlated well with pipeline steel failures under tensile stress (SSC). NACE cited HRC 22 as the maximum hardness compatible with acceptable resistance to SSC [2]. Currently, purchases of pipe for sour environments verify this hardness limitation. However, there have been occasional unexpected occurrences of HIC of pipelines with hardness values well below HRC 22.

In sour service, H_2S reacts with steel in the presence of moisture to form iron sulphides and hydrogen. The atomic hydrogen formed at the reacting surface can diffuse into the steel where it may cause embrittlement and/or accumulate at the inclusion/matrix interface and build up pressure leading to cracking [5]. The diffusion of hydrogen to inclusions in the matrix occurs because of (i) the presence of incipient microcavities at the inclusion/matrix interface or (ii) the presence of residual stress at the inclusion/matrix interface caused by shrinkage and rolling. Two typical types of HIC cracks are shown in **Figure 1**, namely, blister cracks and centre-line cracks. Blister cracks are those hydrogen-induced cracks that are formed near the surface so that the hydrogen pressure is able to lift the material; thus, blisters are observed on the surface (**Figure 1a**). The formation of blister cracks is related to the type and distribution of nonmetallic inclusions in the steel. Elongated Type II MnS inclusions as well as planar arrays of other inclusions are the predominant initiation sites for cracking [4].

Since inclusions are elongated and/or aligned in the longitudinal (rolling) direction, the cracks also run along the longitudinal direction. It should be recognized that the role of inclusions in HIC behavior may vary depending on the service conditions, such as environmental factors and the stress applied to the steel [6-9].

The present study was carried out to investigate the relationship between nonmetallic inclusions and HIC; and to correlate measurements of inclusion parameters with results of SSC and HIC behavior.

EXPERIMENTAL PROCEDURE

Materials

The chemical compositions of the linepipe steels used in this study are presented in **Table 1.** Wall thickness was approximately ~ 10 mm.

Hydrogen-Induced Cracking Tests

HIC tests were carried out according to NACE standard TM-0284 [10]. Three test coupons were cut from each pipe with the following dimensions: 100 mm long x 20 mm wide by wall thickness from parent metal at 0°, 90° and 180° from the weld, with length dimension of the coupon parallel to the rolling direction (**Figure 2**). Coupons were not flattened before the specimens were cut. The coupons were immersed for 4 days in H_2S-saturated solution, with initial pH of 5. Following the test, metallographic sections were prepared to determine the amounts of cracking as well as the orientation of cracks (**Table 2**). In addition, the quantity of diffusible hydrogen in the specimens exposed for the 96 h was determined by immersion in glycerol [11]. The volume of hydrogen evolved is then converted to standard temperature and pressure [STP] per 100 g of sample after 72 h. Coupons were examined for cracks or defects by ultrasonic C-scan before and after hydrogen charging using an automated Ultrasonic Flaw Imaging System. The amount of HIC was determined by measuring cracks on metallographic sections of the tested coupons.

Sulphide Stress Cracking Tests

Smooth tensile specimens with a gage length of 25.5 mm and gauge diameter of 6.35 mm were tested following NACE standard TM-0177 [12]. The test solution (5% sodium chloride, 0.5% acetic acid, and initial pH of ~ 3) was prepared and deaerated with nitrogen for 24 hours. The deaerated solution was passed into the cell, and the flow of H_2S was started. After a few minutes, stress was applied through the Cortest apparatus. The H_2S flow was continued for the duration of the test, 720 h or time to failure, whichever is less. Specimens were tested at different stress levels to determine the threshold stress B i.e. the maximum stress at which the specimen did not fail within the 720 h test period.

Quantitative Metallography of Inclusions

Metallographic specimens were prepared from sections perpendicular to the rolling direction for many of the linepipe steels, and were examined in the as-polished condition by optical microscopy. Quantitative measurements of inclusion length and volume fractions were obtained using a Leco 2001 Image Analysis System. The inclusion populations in 200 microscopic fields were measured on each metallographic sample to obtain statistically significant data on volume fraction and size of the nonmetallic inclusions present.

RESULTS AND DISCUSSION

Sulphide Stress Cracking and Hydrogen-Induced Cracking of Steels

The sulphide stress cracking results for the maximum threshold stress (σ_{tth}) together with the yield strengths (YS) and the σ_{th} /YS ratios for the various steels are given in **Table 3**. Values of crack thickness ratios (%CTR) for unfailed SSC specimens, after immersion for 720 h, as well as values of %CTR for the unstressed HIC coupons are also given in **Table 3**. The ratios $_{th}$ /YS are plotted against YS and the results are shown in **Figure 3**. The plot indicated that the σ_{th} /YS ratio increases in general with increasing YS, and therefore, $_{th}$/YS is a measure of intrinsic resistance to SSC. The %CTR values, for both the SSC and HIC reported in **Table 3** are plotted against YS, as shown in **Figure 4**. The HIC data follow a linear correlation (represented by a straight line), whereas the SSC data follow a different trend, a bell-shaped curve with a maximum at yield strengths in the range 400-525 MPa.

The NACE standard test TM-0177 for SSC is consistent in that similar specimens exhibited similar behavior with a well-defined threshold stress σ_{th} representing resistance to SSC. Nevertheless, the study directed attention towards a possible source of error in the test. The H_2S-saturated solution has an initial pH value of ~ 3, but corrosion of SSC specimens caused an increase in pH (to values as high as 5 for some steels) after 720 h (1 month). Therefore, during the course of the tests, the aggressivity of the solution may decrease with time to a variable extent depending on the alloy and its corrosion behavior. Accordingly, it is difficult to make significant comparisons between different steels. Cracks might have begun to propagate in the highly aggressive solution typical of the initial stages of testing, only to be deactivated as the pH increased in later stages of testing. Comparisons between different steels would be more significant if solution pH could be held more constant during the tests. Possibly, the development of an alternative test solution with better buffering characteristics would be a more convenient approach.

Also, there is a basic drawback to metallographic evaluations arising from the nature of the cracking examined. HIC is usually unevenly distributed, in groups of varying shape and size, some relatively large, such that the three metallographic sections specified at each of 3 locations in the pipe circumference are often too small a sample to represent adequately the HIC behavior of the whole pipe. Reproducibility between coupons from the same part of a pipe was only fair. Therefore, a more rapid and simple method of evaluating HIC is needed. In such a method, a much larger volume of sample that is more representative of the whole pipe would be examined.

The basic limitation of the metallographic evaluation of HIC could be overcome by using the ultrasonic C-scan test, which is rapid and sensitive to determining cracks in samples. However, quantitative data on cracking, such as crack length ratio, are not obtained by this method. **Figures 5** and **6** show a typical output from a C-scan test carried out after the steel is exposed to H_2S-saturated solution in the HIC test. The metallographic examination confirmed the findings indicated by C-scan. Among the three methods of evaluation (metallography, hydrogen evolution and C-scan) the C-scan was by far superior with respect to speed and the amount of information provided, and it compares very well with the susceptibility of the steel to HIC.

Regarding the various locations over the pipe circumference, it is prudent that more testing should be performed to determine the variability of SSC resistance around the pipe circumference. Another sample location that should be looked at more critically is the through-thickness location of SSC samples that are taken from the middle 6.4 mm of the wall thickness, according to NACE standard TM-0177. This location will not necessarily have the same susceptibility for initiation and propagation of cracks as the metal at the inner pipe surface, which is in direct contact with the sour environment. For thicker walled pipes, it would be useful to compare the SSC behavior of Asurface@ and Acentre-thickness@ specimens.

Diffusible Hydrogen (C_o) and Threshold Hydrogen Content (C_{th})

The factors C_{th} and C_o, representing the hydrogen embrittlement (HE) behaviour of steel materials, might be interpreted in terms of threshold hydrogen permeability representing the HE susceptibility of the material and hydrogen permeability representing the amount of hydrogen penetrating the steel matrix from the environment, respectively.

Determining the amount of hydrogen absorbed by a coupon during a HIC test is a relatively simple procedure. Many researchers have reported a correlation between absorbed hydrogen and the amount of HIC [13, 14]. Absorbed hydrogen can provide a better estimate of susceptibility to HIC than the small slices used in metallographic evaluations. In addition, the reproducibility of hydrogen contents was superior to that of HIC determinations. The amount of absorbed hydrogen was shown to correlate with HIC data obtained by metallography **(Figure 7)**. Ikeda et al. [13] have shown that there is a critical concentration of dissolved hydrogen in a steel, termed C_{th}, that must be reached before HIC can occur. Low HIC resistance material is, therefore, characterized by a low threshold value. For example, a steel with C_{th} = 0.3 mL (STP)/100 g has low HIC resistance, but over 2 mL (STP)/100 g has high HIC resistance (e.g., Ca treated steels) [10, 15].

The use of the C_{th} of a steel as a measure of its resistance to HIC would be a more rational approach than that used in the present NACE standard test. There would be less difficulty in ranking steels in their susceptibility to HIC; even more important, C_{th} value of a steel can be related directly to the performance of that steel in a pipe containing a corrosive sour environment. In such pipe, permeation methods can be used in the field to determine the maximum concentration of dissolved hydrogen in the wall, C_0; HIC should only occur when C_0 C_{th}.

Significance of Inclusions

Scanning electron microscopic (SEM) examination indicated the presence of cracks, as shown in **Figure 8**. The EDX microanalysis revealed that the inclusions in cracks are manganese sulphide. The present investigation shows that MnS inclusions are the predominant initiation sites for cracking. It appears that MnS inclusions provided sites for hydrogen to accumulate, leading to higher HIC susceptibility. The results of the quantitative metallographic determination of inclusions using image analysis are reported in **Table 5** and plotted in **Figure 9**. The results indicate that the total length of cracks per unit area (CLR) correlates well with the total inclusions per unit area. However, three steels, A, G and B, exhibited anomalous results. Steels A and G showed much less cracking than that predicted by the correlation with inclusion measurements, and steel B showed much more. Steels A and G are the only semi-killed steels, and their behaviour is consistent with the general observation that semi-killed steels are less susceptible to HIC than fully-killed steels [16]. The elongated inclusions in steels A and G are predominantly silicates which are more plastic than type II MnS at rolling temperatures. This should result in fewer microvoids at the inclusion/matrix interfaces in the semi-killed steels. The most likely explanation for the high incidence of cracking in steel B is its heavily banded microstructure compared with the other steels **(Figure 10)**. This is illustrated by comparing the HIC results for steels B and C. The two steels have similar inclusion measurements **(Figure 9)** , but CLR for B is much higher than C, which has a uniform microstructure as shown in **Figures 10a** and **10b**, respectively. It has been established that heavily banded microstructures provide a low fracture resistance path for easier crack propagation [10, 17].

The study demonstrated that there is a good correlation between HIC and inclusion measurements. Some anomalous results are found which illustrate the effects of other factors (other than inclusions) in HIC behavior and emphasize that the correlation with inclusion measurements is valid only when other factors remain constant.

Quantitative Measurement of Inclusions

Quantitative metallography of inclusions was investigated by image analysis. The steels investigated were: WC-1, G-2; AM-1, PC-1 and CTR-2 (see **Table 1**). The summary of C_{th} and pH_{th} for all steels is given in **Table 6**. Cracking was observed below pH 5.3 in all but two of the steels. Data on volume percent, average size and length of inclusions are also reported in **Table 6**. The results are correlated with threshold hydrogen concentration and plotted in **Figures 11** and **12**. Both volume fraction and total length per unit area correspond closely to the threshold hydrogen concentration. It is clear from these figures that the steel with minimum inclusions has the highest threshold hydrogen concentration and vice versa. The correlation is reported in **Figure 13** as ratio of inclusion volume fraction to that of the WC-1 steel [highest volume fraction (0.387 in **Table 6**)] versus the ratio of hydrogen threshold concentration to that of AM-2 steel [highest hydrogen threshold concentration (~ 2 mL, in **Table 6**)]. A linear relationship exists between threshold hydrogen concentration ratio and the quantity of inclusions in the steel.

Metallurgical Factors Influencing Hydrogen Damage and Cracking

The development of new steels that are resistant to HIC and SSC are now studied in both the laboratory and the field. Actually it is possible to relate the HIC performance of steels to chemical content, inclusion levels and thermomechanical treatment parameters. By controlling these parameters, it is now possible to produce H_2S-resistant line pipe steels. In future, it may be possible to use steels of X-70 Grade and higher.

Effect of Rolling Processing

Industrial and laboratory results had indicated that a given type of steel would behave differently in the HIC test depending upon whether rolling ended in the austenite or intercritical region. A significant deterioration in HIC performance of the steel was observed with a decrease in the final rolling temperature below the transformation line. Strain hardening of ferrite contributes to an increase in dislocation density, thus changing the diffusion kinetics of hydrogen and its distribution in the steel during HIC testing.

HIC resistance can change with the metallurgical processing method used in steel production. Highest susceptibility is associated with hot rolled and final rolling temperature. Normalizing, quenching and tempering have been shown to increase HIC resistance.

Effect of Chemical Segregation

It has been established that certain elements, such as carbon (C), manganese (Mn) and phosphorus (P), tend to segregate during solidification in the continuous slab casting process. This explains the banded structure classically observed in pipeline steel produced by controlled rolling [18].

Studies have shown that, where Mn exceeds 1%, there is a dramatic increase in manganese content in the banded regions [19-22]. Microprobe analysis reported that, within the banded regions, it is possible to achieve local levels of Mn and P of the order of 2 and 6 times higher, respectively, than the base metal. This leads to the local formation of bainite/martensite structures that interfere with HIC performance, particularly at the mid-thickness of the plate. The HIC performance on rolled samples shows better behavior for samples from skin-milled than samples from the mid-thickness. This was directly related to the fact that segregated banded structures are more extensive in samples from the mid-thickness.

Based on casting and rolling results reported in the literature [23, 24], it has been established that a decrease in phosphorus content improves resistance to HIC in steel. In addition, numerous studies have demonstrated an intensification in phosphorus and manganese segregation with increasing carbon content [25]. In some cases, copper (Cu) addition is also beneficial in reducing HIC susceptibility. Cu additions greater than 0.2 wt% reduce hydrogen absorption from sulfide corrosion in the intermediate pH (pH 4.5 - 5.5) environments [26].

Effect of Cleanliness on Steel

Susceptibility to HIC is highly dependent on the impurity content of the material. The effective resistance to HIC requires the lowest possible level of sulphur [27], combined with spheroidization of inclusions, generally achieved through calcium-silicon treatment. The Ca treatment must be applied under highly controlled conditions in order to ensure that the amount of calcium introduced into the steel is sufficient for spheroidization of aluminum and sulphur inclusions. Ca addition helps to improve HIC resistance through sulfide inclusion shape control by breaking up the normally elongated inclusions to a more spherical shape, i.e., reducing the inclusion aspect ratio. This shape is less conducive to the initiation of the planar blisters typical of HIC [15]. This requires an approximate Ca/S ratio in excess of 2 in order to prevent the formation of MnS. However, when S contents are very low, over-treatment by Ca (i.e., Ca/S _ 5), can lead to the formation of calcium sulphide (CaS) inclusions, which are particularly detrimental to HIC performance.

Effect of Precipitation Heat-Treatment

Significant improvement in resistance to HIC in the precipitation state, control-rolled condition can be achieved. The susceptibility to HIC cracking can drop significantly. Precipitation treatments can reduce considerably the quantity of hydrogen absorbed onto steel. The benefit of the precipitation treatment has been attributed to partial restoration of ferrite which, in the untreated, control-rolled condition, is characterized by a high dislocation density induced by strain hardening in the two-phase region. It was also remarked that precipitation treatment causes a structural change that enhances the HIC performance of the material as follows: spheroidization of the pearlite and smoothing out of locally segregated microstructures of bainite/martensite phases.

Requirement for H_2S-Resistant Linepipe Steels

Steels that are more resistant to hydrogen damage cracking in H_2S containing environments:
Managing the spheroidization treatment (injection of calcium-silicon) and casting to ensure low inclusion content;

- Adapting casting conditions to minimize chemically-induced segregation during the solidification process;
- Deoxidation state of the steel, Mn/S ratio, rolling temperature, degree of substitution of Mn or S by other elements. In general, the deformability of sulphides decreases as the degree of substitution of either Mn or S increases;
- The balance of carbon and manganese has an overriding effect on resistance to HIC;
- The lower the susceptibility parameter, the better the resistance of the steel to HIC;
- The structures in steel with high HIC resistance are much more homogeneous; i.e., absence of banded structures.

CONCLUSIONS

1. The ratio σ_{th}/YS is a good index for the intrinsic resistance of various steels to SSC.
2. HIC can occur only in an operating sour-gas pipeline if the maximum hydrogen concentration C_0 in the pipe wall equals or exceeds C_{th}, the threshold hydrogen concentration for HIC.
3. The pH_{th} values can be used to rank the steels with respect to HIC resistance.
4. Ultrasonic C-scan provided the most rapid and informative method for evaluating HIC in a test coupon. Metallographic evaluations are time-consuming and poorly reproducible.
5. There is good correlation between inclusion measurements, threshold hydrogen

concentration (C_{th}), and HIC. The elongated MnS inclusions are primarily responsible for cracking. Lower volume fractions of inclusions corresponded to higher C_{th}^{H}, and hence, higher resistance to HIC.
6. The microstructure may also increase HIC susceptibility by providing paths of low resistance to crack propagation.

Metallurgical studies have made it possible to distinguish the effects attributable to each of the various parameters governing the resistance of linepipe steels to hydrogen damage cracking. This knowledge paves the way for future development of high grade steel; i.e., X80 and beyond.

ACKNOWLEDGEMENTS

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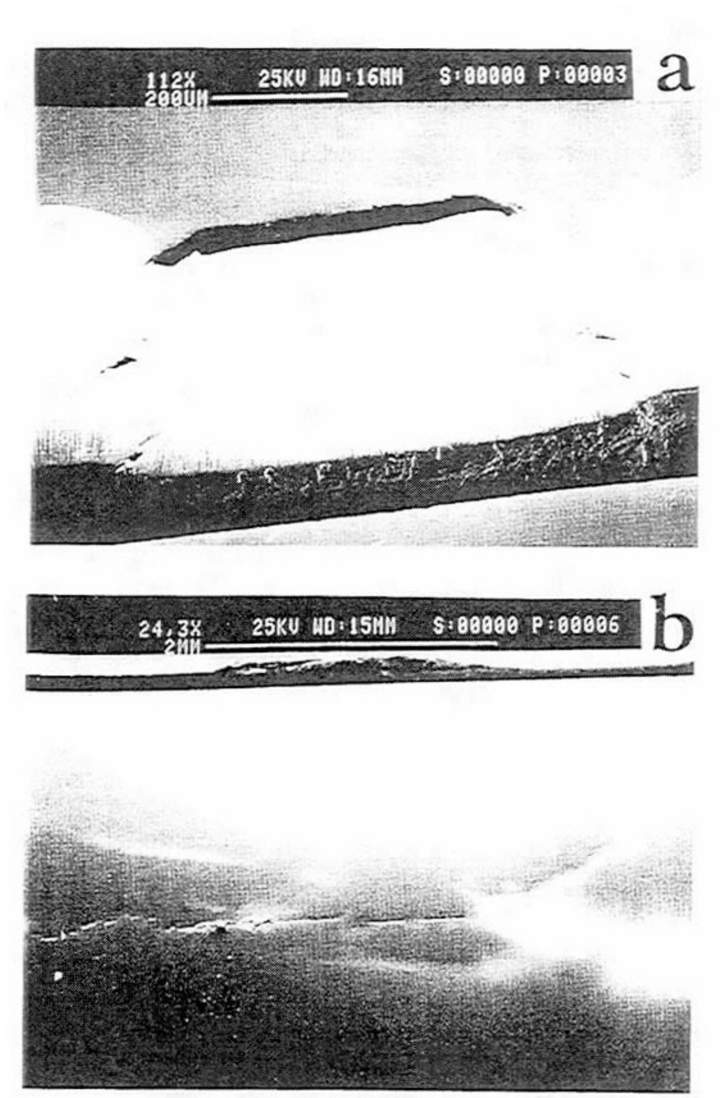

Figure 1. Two types of hydrogen-induced cracking (HIC):
a) Blister crack; and b) Centre line cracks

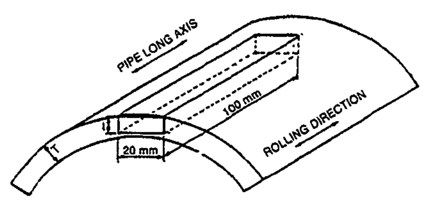

Figure 2. Extraction of HIC samples from pipe material, and orientation of test coupons.

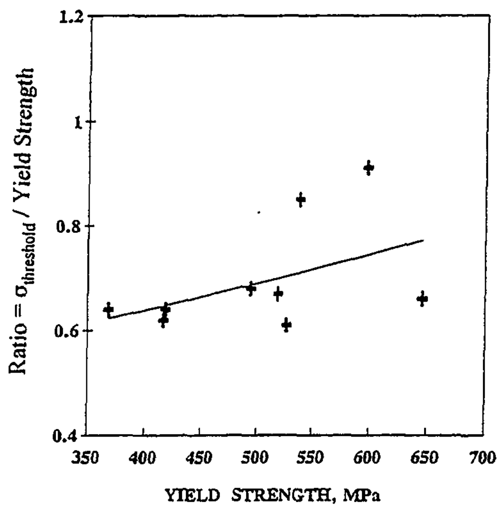

Figure 3. Data from sulfide stress cracking (SSC) tests in NACE Standard TM-0177 solution

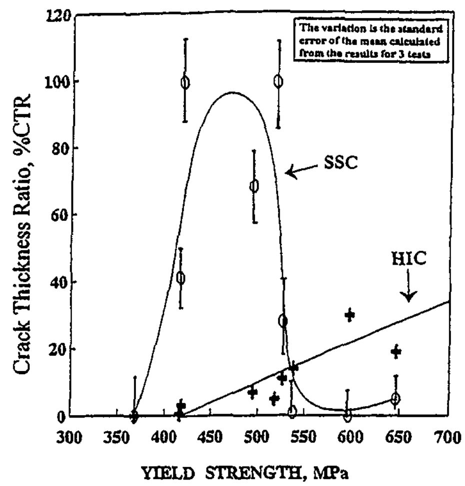

Figure 4. % CTR values on unfailed SSC specimens after testing for 720 h and on unstressed HIC coupons. Both plotted against yield strength.

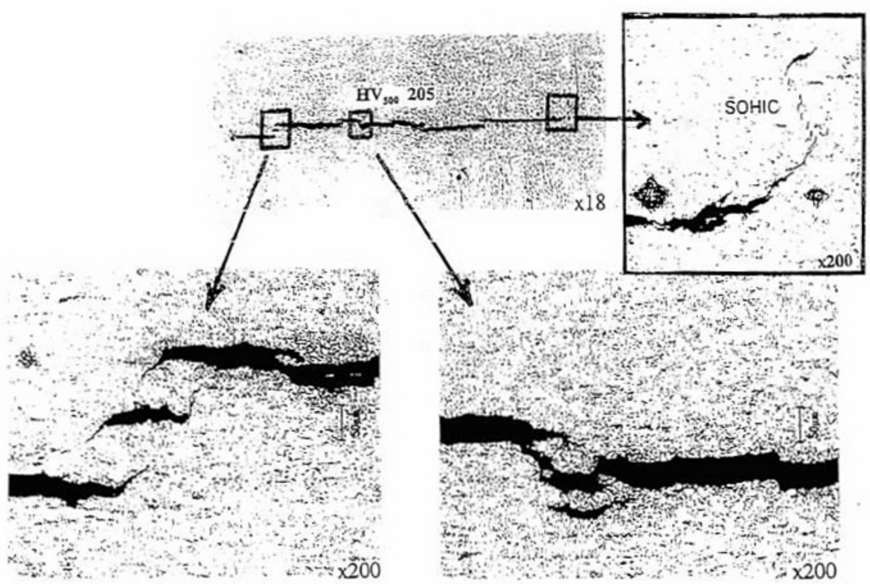

Figure 5. Optical micrographs showing cracks of SSC specimen after testing in NACE Standard TM-0177 solution.

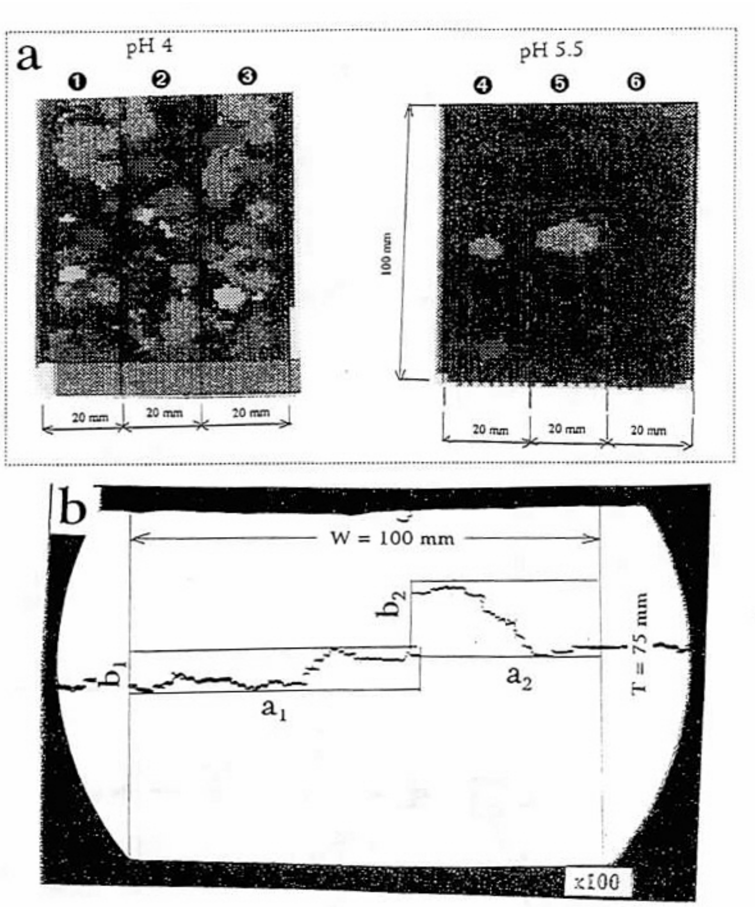

Figure 6. a) Ultrasonic C-scan image of 6 coupons tested in NACE Standard TM-0284 solution: (i) 3 coupons with significant cracks, and (ii) 3 coupons free of cracks

b) *Metallographic examination of HIC obtained on cracking coupon #❸.*

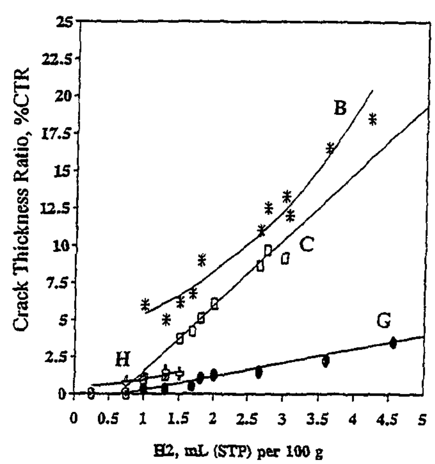

Figure 7. Diffusible hydrogen at 45°C related to % CTR for 4 pipes B, C, G, and H.

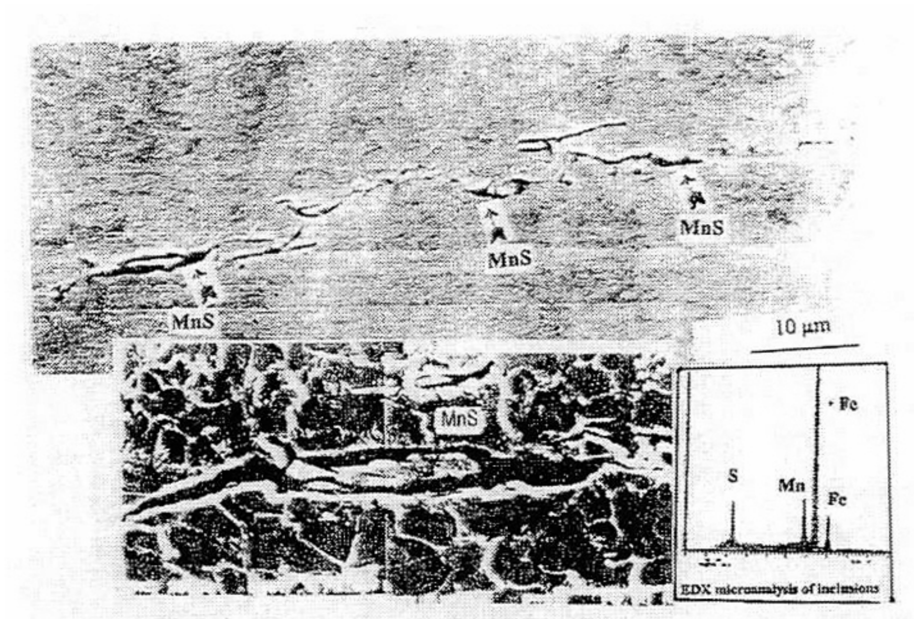

Figure 8. SEM micrograph of crack showing elongated nonmetallic inclusion (MnS).

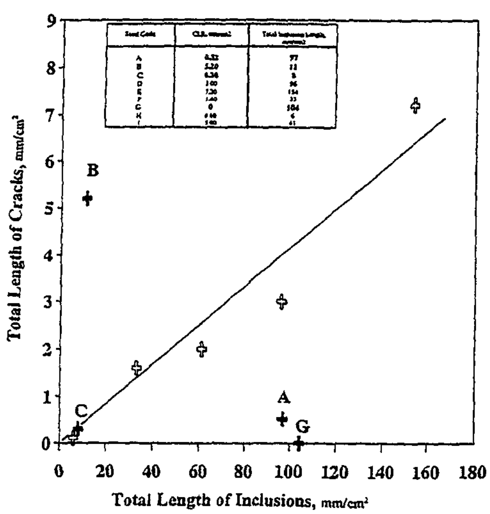

Figure 9. Plot of the effect of the total length of inclusions on total length of cracks (CLR) per unit area.

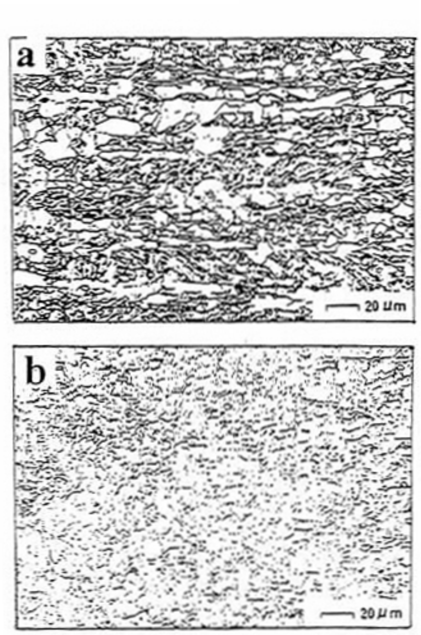

Figure 10. (a) Photomicrograph of steel B with a banded microstruture; and (b) Photomicrograph of steel C with a uniform microstruture.

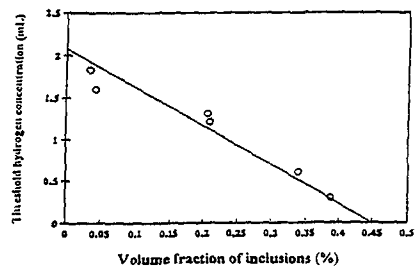

Figure 11. Effect of the volume fraction of inclusions (%) on threshold hydrogen concentration for cracking C_{th}).

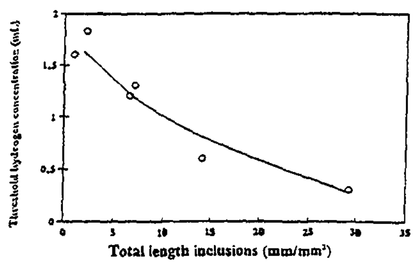

Figure 12. Effect of the effect of the total length inclusions on threshold hydrogen concentration for cracking (C_{th}).

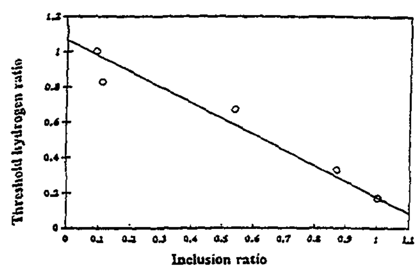

Figure 13. Effect of the effect of inclusion ratio on threshold hydrogen concentration for cracking (C_{th}).