

Short Note

THE EFFECTS OF HYDROGEN ON THE FRACTURE TOUGHNESS OF STAINLESS STEEL

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تأثيرات الهيدروجين على خشونة كسر الحديد غير القابل للتآكل

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تمت في هذه الدراسة إجراء التجارب على نوعين من الحديد الغير قابل للتآكل (UB50, 410) باستعمال ثلاثة طرق مختلفة ، وذلك بتطبيق المواصفات الإنجليزية (5447) لقياس خشونة الكسر (K_{IC}). الطريقة الأولى شملت :
— الطريقة الكهروكيميائية للحصول على درجة عالية من التشبع بالهيدروجين.
— طريقة محلول جمعية التآكل الأمريكية من درجة الحرارة العادية.
— طريقة محلول جمعية التآكل الأمريكية من درجة 50 درجة مئوية.
ومن هذه الطرق الثلاثة تبين التالي :
— ارتفاع وانخفاض خشونة الكسر بالنسبة للحديد UB50, 410 على التوالي باستعمال الطريقة الأولى.
— إنخفاض خشونة الكسر لنوعين من الحديد باستعمال الطريقة الثانية.
— ارتفاع وانخفاض خشونة الكسر للحديد UB50, 410 على التوالي باستعمال الطريقة الثالثة.

INTRODUCTION

A duplex stainless steel UB50 and 410 martensitic stainless steel were exposed to hydrogen using three different methods. Cathodic charging with hydrogen in electrochemical cell, H₂S NACE solution which consisted of 5 weight percent (w%), NaCl, 0.5 w% acetic acid, and balance of deionized water at room temperature, and NACE solution at 50°C.

The effect of the methods on the fracture toughness showed that cathodic charging resulted in increasing and decreasing the fracture toughness of UB50 and 410; respectively. The H₂S NACE solution at room temperature resulted in decreasing the fracture toughness of both. Stainless steels and H₂S NACE solution at 50°C resulted in increasing and decreasing the fracture toughness of UB50 and 410; respectively.

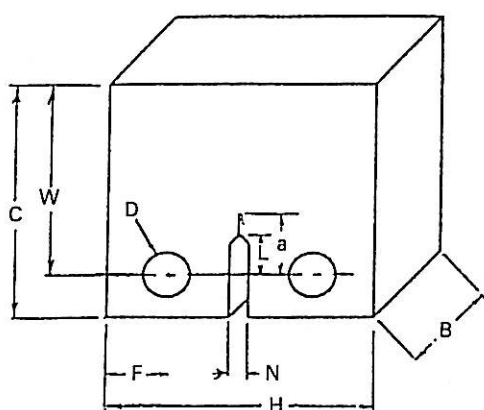
EXPERIMENTAL PROCEDURE

Samples of two materials (UB50 and 410 stainless steel) were machined into crack growth specimen of

dimensions shown in Fig. 1, [4]. The material chemical compositions of both alloys are given in Table 1. A fatigue precrack of length approximately $\frac{1}{2}w$ was placed in each sample prior to hydrogen cracking. The fatigue parameters used were 37-40 Hz, a maximum tensile load around 9 KN and a R-ratio of 0.1. The maximum load was chosen so that it would not exceed 70% of the expected value of K_{IC} .

The samples were cathodically charged with hydrogen in an electrochemical cell at constant current of 1.6 Amp. in 0.5 M H₂SO₄ with 1 g/l of thiourea added as a hydrogen recombination poison as shown in Fig. 2. Argon was bubbled through the solution both prior to and during the charging to remove any dissolved oxygen which might adversely affect the test. The precracked fracture toughness specimen was used as cathode and a platinum electrode was used as the anode. Direct current power source was connected to both electrodes to provide a constant current during charging operation. Another method of hydrogen charging was used which is exposing samples of both steels (UB50, 410) to a saturated H₂S NACE solution

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Dimension	Symbol	Measurement (mm)
Net width	W	25.40
Total width	C	31.75
Height	H	30.48
Hole diameter	D	6.35
Hole distance	F	5.08
Notch width	N	1.65
Effective notch length	l	6.75
Effective crack length	a	Measured after pre-crack

FIG. 1. Dimensions of test specimen (CTS).

Table 1. Compositions of Alloys

Elements	C	Si	Mn	S	P	Cr	Ni	Fe	Mo	Cu	Co
UB50	0.029	0.47	1.56	0.006	0.016	20.74	6.6	bal	2.4	1.4	—
AISI 410	0.13	0.33	0.4	0.006	0.002	13.2	0.38	bal	0.03	0.06	0.02

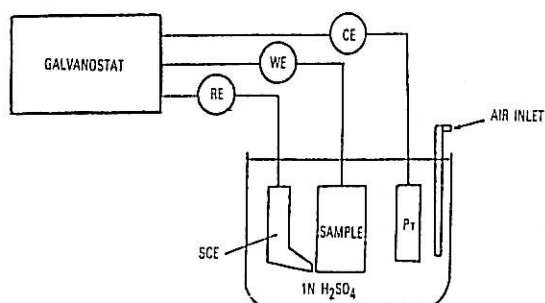


FIG. 2. Schematic diagram of hydrogen charging in electrochemical cell.

at 50°C and room temperature. For comparison the NACE solution was saturated by bubbling H₂S gas through the solution until the solubility limit has been reached.

Attempts were made to keep the pH of the solution low enough to maintain a high concentration of H⁺ ions near the cathode producing a high partial pressure of H₂ gas near the sample. In the first test a four liter jug

was used as the cell and the charging time was thirty days. However, at the end of 30 days, the pH of the solution was nearly 0.30. This indicated high concentration of H⁺ ions to generate H₂ gas throughout the entire test.

All samples were tested to determine the K_{IC} values within 15 minutes from the time they were removed from the electrochemical cell. A load vs strain curve was generated for all tests. The load was measured by the testing machine and the strain was measured from the clip gauge attached to the sample. The clip gauge allowed the actual strain of the sample to be measured instead of measuring the cross head position which would have included the strain of the test pins and attachments. The K_{IC} value was determined according to British standard 5447. A line was drawn through the origin of the load vs displacement plot with a slope 5% less than the slope of the elastic region. The load where this line intercepts the plot is called P_Q. K_Q is determined from P_Q and the geometry of the specimen. If the test data conform with all requirements outlined in the standard; the K_Q becomes the K_{IC} which is the fracture toughness. If the requirements are not met then only a K_Q value can be reported. All the results obtained in this study are K_Q values except one result, because in most instances, the value of P_{max}/P_Q was greater than 1.10 making the test invalid for K_{IC} determination.

RESULTS

The results obtained using L.E.F.M. method and the stated conditions as well as P_{max}/P_Q values, pre-cracking length (a), specimen width (w) values, K_Q and K_{IC} values are listed in Table 2.

The values are plotted against curves generated from previous work done using H₂S environments (Fig. 3).

DISCUSSION

In order to reveal the embrittling effect of hydrogen uptake on the change of materials properties and the possible different response of the different stainless steels to hydrogen entry, the effects of precharging on the fracture toughness of both steels (UB50 and 410) was determined. All the specimens were machined with the notch and crack parallel to the longitudinal (rolling) direction.

Table 2. Fracture Toughness Results and Testing Conditions

Sample No.	Test Conditions	P_{max}/P_0	a/w	K_{IC} and K_{IC0} (MPa $\sqrt{m}^{1/2}$)
1B UB 50	4 litre cell cathodic charged for 11 days in H ₂ SO ₄ + 1 gr l ⁻¹ thiourea solution	1.05	0.48	50
R UB 50	4 litre cell cathodic charged for 30 days in H ₂ SO ₄ + 1 gr l ⁻¹ thiourea solution	1.06	0.49	55
3B UB 50	4 litre jar, specimen immersed in saturated H ₂ S NACE solution for 6 days at room temperature	1.2	0.57	46
3A UB 50	4 litre jar, specimen immersed in saturated H ₂ S NACE solution for 6 days at 50°C using water bath	1.16	0.50	51 (K _{IC})
1A UB 50	4 litre jar, specimen immersed in saturated H ₂ S NACE solution for 6 days at 50°C using water bath	1.16	0.50	54
410 (4)	4 litre cell cathodic charged for 6 days in H ₂ SO ₄ + 1 gr l ⁻¹ thiourea solution	1.06	0.51	63
F 410 (2)	4 litre jar, specimen immersed in saturated N ₂ S NACE solution for 6 days at room temperature	1.02	0.51	60
410 (3)	4 litre jar, specimen immersed in saturated H ₂ S NACE solution for 6 days at 50°C using water bath	1.02	0.55	72
UB 50	4 litre cell cathodic charged for 13 days in H ₂ SO ₄ + 1 gr/l thiourea solution	—	—	—

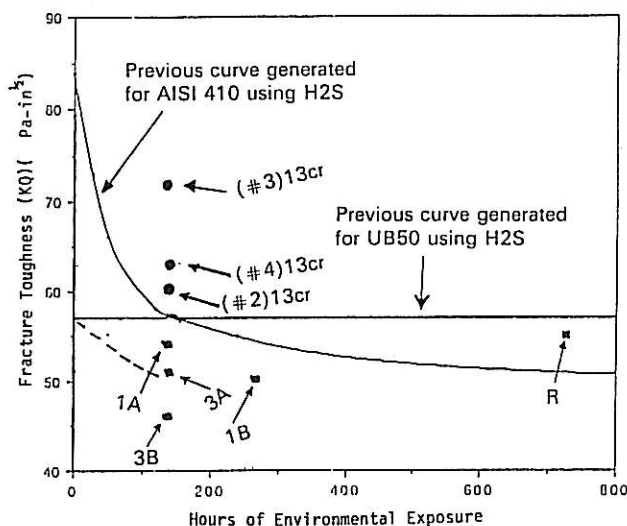


FIG. 3. Comparison of data from this study with results obtained. Descriptions of samples are given in Table 2.

Table 2 lists all values of K_Q of both stainless steel. These values are plotted against curves from previous study or work done using H_2S environment (Fig. 3).

The 410 martensitic stainless steel was actually very tough in air ($K_Q = 87 \text{ Mpa/m}^{1/2}$), but its toughness dropped sharply in six days after immersion in H_2S NACE solution. Further pre-charging for up to 30 days results in entirely brittle fracture behaviour of the AISI 410 giving its K_{IC} of around $50 \text{ Mpa/m}^{1/2}$ [1]. All the results obtained concerning 410 are K_Q values because in most instances the requirements of BS 5447 are not satisfied making the test invalid for K_{IC} determination.

The results performed on 410 samples in two different conditions (H_2S NACE solution at room temperature and 50°C) using CT specimen correlate very well with the previous results conducted with H_2S environment Fig. 3.

Electrochemically charging with hydrogen (410 #4) has the same effect as using a reaction with H_2S at room temperature (410 #2) to embrittle the alloy. Increasing the temperature while using H_2S to charge the sample had a little effect on the fracture toughness: on the sample (410 #3) exposed to H_2S NACE solution at 50°C for six days the fracture toughness value ($72 \text{ Mpa/m}^{1/2}$) is below the line constructed from that taken using an H_2S environment at room temperature, Fig. 3.

The method of hydrogen charging has relatively little effect on the fracture toughness of 410 while the bulk precharging of H_2S had a significant precharging effect on the 410. The temperature also has an effect on the fracture toughness of 410 in a H_2S environment.

The electrochemical charging of specimens (IB UB50 and R UB50) shows a small decrease and no change in the fracture toughness respectively. The base-line fracture toughness value of UB50 before any environment exposure was close to the $52 \text{ Mpa/m}^{1/2}$ obtained in the previous study [1] Fig. 3. This gives good indication that the electrochemical hydrogen charging has a little if any effect on the fracture toughness of UB50 (specimens R UB50, IB UB50). The fracture toughness however, is reduced after charging with H_2S NACE solution at room temperature ($46 \text{ Mpa/m}^{1/2}$).

The H_2S content of a saturated H_2S NACE solution at 20°C is approximately 2700 ppm and at 50°C this value dropped to 1300 ppm.

The decrease in H_2S content of the solution at higher temperature as mentioned above causes the samples exposed to H_2S NACE solution at 50°C to have a higher fracture toughness for UB50 as shown in Table 2.

Another explanation for this phenomenon is that increased diffusion at higher temperature [2, 3] had a greater effect than the lower H_2S concentration.

In case of AISI 410 stainless steel, Table 2, the same effect of temperature was observed as for UB50, i.e. when the temperature increased the fracture toughness increased.

CONCLUSION

Due to time constraints only a limited number of the tests were performed during this study.

1. Electrochemical hydrogen charging lowers the fracture toughness of both 410 and UB50 stainless steels. The effect of the electrochemical on AISI 410 was slightly lower than the effect observed in a previous study, but higher for UB50 than observed in a previous study.
2. The effect of the electrochemical hydrogen charging appears to be slightly higher than the effect of the H_2S NACE solution, for both steels (UB50 and 410).
3. The drop in fracture toughness of UB50 was relatively small compared to the decrease observed with the AISI 410 stainless steel.
4. The effect of H_2S NACE solution at 50°C has a significant effect on the fracture toughness of both stainless steel (410 and UB50). Increasing the temperature, from room temperature to 50°C , increased the fracture toughness of both steels.

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