Fracture toughness characterization of hydrogen embrittled Cr-Mo steel

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ABSTRACT

Since pressure vessels for petroleum use are operated at high temperature high pressure hydrogen gas, it is a special concern whether a crack at a stressed region grow by internal hydrogen embrittement (I.H.E.) mechanism during shutdown low to temperature ambience. In this study, fracture mechanics tests were conducted to clarify how hydrogen assisted crack of 2.25Cr-1Mo steels grows under I.H.E. condition. Hydrogen was pre-charged inside of the steel by high pressure, high temperature hydrogen autoclave and tests were conducted at room temperature air ambience. As a result, for the majority of steels tested, the stress intensity factor at hydrogen crack growths by I.H.E. mechanism were very low at initiation ($=K_{IH}\sim30MPa\cdot m^{1/2}$)) and grows faster if an active, rising load was applied. The old 60's made generation steel, which has higher temper embrittlment susceptibility, exhibited a higher crack velocity and resulted in fast fracture (K_{IC-H}) during rising loading. On the contrary, if a load was applied for a static, fixed crack mouth displacement manner (i.e. falling load condition), the crack velocity significantly decreased and finally stopped to give a higher threshold stress intensity factor ($=K_{th}$) except for higher strength steel. Higher strength steel (; Enhanced 2.25Cr-1Mo grade material) tended to continue to propagate despite under fixed displacement condition and falls on to a very low K_{th}.

KEYWORDS

I.H.E., Hydrogen charging, 2.25Cr-1Mo steel,K_{IH}, K_{IC-H}, K_{th}

INTRODUCTION

In the pressure vessel with overlay or attachment of stainless steel inside of the wall, a high stress may be arised at discontinuous and complex structure area due to thermal expansion mismatch between stainless steel

(SS) and base Cr-Mo steel. Furthermore, when pressure vessel cooled down to ambient temperature, hydrogen atoms absorbed inside of the Cr-Mo steel rather accumulates between interface of SS and Cr-Mo steel than degassing outside of the wall which may cause disbonding or initiation of hydrogen embrittled cracking. Figure 1 shows the calculation example of shutdown procedure. It is shown from the analysis that the crack at welded structure driving force arises at stainless structure welded area. Therefore, special attention should be paid whether the crack at the interface of stainless steel and Cr-Mo base initiates and penetrates through wall, which may cause final collapse of the entire vessel. In this study, fracture mechanics tests were conducted to investigate how hydrogen assisted cracking of 2.25Cr-1Mo steels grows by I.H.E. mechanisms examining loading method, materials toughness level and steel strength.

NOMENCLATURE

RL :Rising load

CD :Constant Displacement

K_{IH} :Stress intensity for onset of subcritical crack growth

K_{th} :the threshold intensity factor for hydrogen charging environment.

$K_{IC\text{-}H}$:material toughness measured in the hydrogen charging environment.

MATERIALS

Table 1 shows chemical composition of steels tested. Impurities Si, P and Sn were intentionally added to simulate the old temper embrittled steel controlling J-factor=(Si+Mn)x(P+Sn)x10^4 wt% level. After hot rolling, those heats were subjected to the quenched and tempered heat treatment + PWHT at 690°C for 8hrs. followed by step cooling. Table 2 shows mechanical properties. Low J steel is the new generation made steel with high fracture toughness at room temperature. Mid J and High J steels were temper embrittled by step cooling. The compact tension specimen were machined and Ni/Au were plated to prevent hydrogen degassing from inside of the steel. Hydrogen were charged in autoclave at 420°C, 12MPa for 48hrs followed by water quenching to room temperature and preserved in liquid nitrogen container until fracture mechanics test in air environment.

CRACK GROWTH BEHAVIOR

Effect of loading condition

Slow rising load (:RL) and constant displacement(:CD)loading method (Figure 2) were applied on hydrogen crack growth testing. Load was controlled by Crack Mouth Opening Displacement(CMOD) with a speed of 0.00003mm/sec. efficacious for hydrogen embrittlement [1)]. Crack was monitored by D.C. potential drop method. Figure 3 shows typical result of RL+CD test. Crack initiation was occurred after 3hrs of a rising load test. Continuous propagation was observed during rising load applied. After CMOD kept constant, the crack growth rate decreased and finally stopped after 12hrs.. Figure 4 shows the repetitional RL and CD test. Subsequent imposing of rising load obviously enhances crack growth, whereas crack growth was deactivated by keeping CMOD constant. Finally, a remarkable increase in crack growth observed in the later RL stage and resulted in fast fracture.

K and da/dt relation

Typical relationship between K and crack growth rate (da/dt) for 610MPa tensile strength steel is shown in Figure 5. Under RL condition, crack start to grow rapidly at low initiation point K_{IH}, and continue to propagate at relatively constant speed of 10^{-4}mm/sec order of magnitude for K_I level ranging from 30 to 80MPa·$m^{1/2}$. After CMOD kept constant, K_I and da/dt relationship can be drawn as steeper falling line, which finally reaches threshold slightly decreasing from holded K_I. Since repetitional RL+CD result indicate that crack stops at any holded K_I level ($<K_{IC-H}$), threshold K_{th} may exist in between K_{IH} and K_{IC-H} affected by the holded CMOD value.

Effect of temper embrittlement

K_{IH} were measured by RL condition for a temper embrittled old and newer high toughness heats and were plotted as a function of its material's FATT in Figure 6. Regardless of FATT of its heat, majority of steels exhibits low initiation sensitivity of cracking; K_{IH}=30MPa·$m^{1/2}$. K-curves for Low J and Mid J steels are compared in Figure7. Initiation occur at low K_I point in two heats, but cracking resistibility is higher for low J steel, whereas Mid J steel cracking propagates in low K_I and led to K_{IC-H}. Fracture appearances are compared in Figure 8. Intergranular cracking was dominant in Mid J steel whereas LowJ steel heat exhibits almost quasi-cleavage with some intergranular fracture surfaces. Although difference in fracture surfaces recognized, the role of hydrogen on the initiation kinetics should still to have to be studied.

Effect steel strength level

Crack growth measurement of RL+CD tests were conducted for enhanced and annealed heat and results are shown together in Figure 9. Three heats show entirely different cracking characteristics respectively where the enhanced heat exhibited a very aggressive propagation under long term CD condition, on the other hand, the only little cracking was observed in the annealed heat. These tendency indicates that the higher strength steel is susceptible to delayed type cracking under static CD condition falls on to lower K_{th} and lower strength steel as well as was already suggested by the previous studies.[2)]

SUMMARY

Crack growth characteristics for a variety of 2.25Cr-1Mo steels were clarified and summarized. Schematic illustration of K and da/dt relationship is shown in Figure 10.

1. Imposing of active rising load obviously enhances crack initiation and growth rate, whereas crack growth was deactivated by keeping CMOD constant.
2. Crack start to grow rapidly at low initiation point K_{IH}, and continue to propagate at relatively constant speed and as K_I level increases, fast fracture occurs at K_{IC-H}.
3. Initiation of cracking KIH did not make much difference in temper embrittled and new high toughness steel. But cracking resistibility is higher for low J steel, whereas Mid J steel propagates cracking in low K_I and reached K_{IC-H}.
4. The higher strength steel is susceptible to delayed type cracking under static CD condition falling on to lower K_{th} and lower strength steel as well as was already suggested by the previous studies.

REFERENCES

1) J.Watanabe, T.Ishiguro, T, Iwadate and K.Ohnishi(1987), *Hydrogen Embrittlement of 21/4Cr-1Mo and 3Cr- 1Mo- 0.25V-B Pressure Vessel Steels*",presented at API/MPC Task Group Meeting on Materials for Pressure Vessels May 1987

2) W.E.Erwin and J.G.Kerr(1982);WRC Bulletin 275,2

Table1 Chemical compositions(wt%) and mechanical properties of steels tested

Steel	C	Si	Mn	P	S	Cr	Mo	Sn	Sb	J-factor	FATT ℃	TS MPa
Low J	0.14	**0.08**	**0.55**	**0.005**	0.0007	2.42	1.08	**0.010**	0.0011	95	**-76**	613
Mid J	0.15	**0.25**	**0.55**	**0.015**	0.0013	2.39	1.03	**0.024**	0.0009	312	**6**	623
Annealed	0.15	0.24	0.53	0.013	0.0013	2.45	1.02	0.022	0.0006	270	7	**552**
Enhanced	0.16	0.28	0.40	0.009	0.0150	2.16	1.01	0.004	0.001	88	4	**668**

J-factor=(Si+Mn)X(P+Sn)x10^4

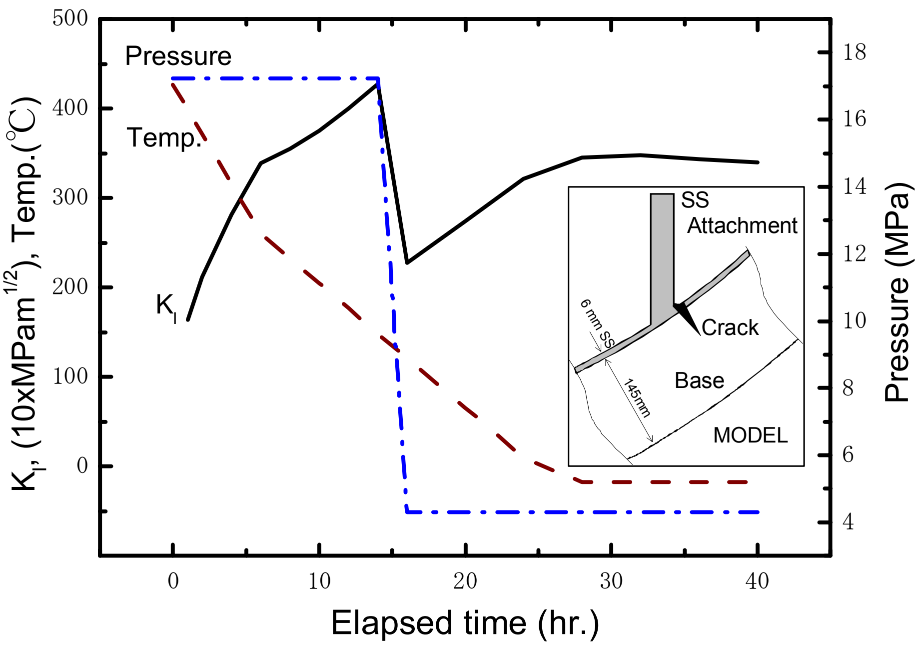


Figure 1 Example of shutdown temeperature, pressure and calculated result of crack loading at a bottom shell section with a stainless attachment

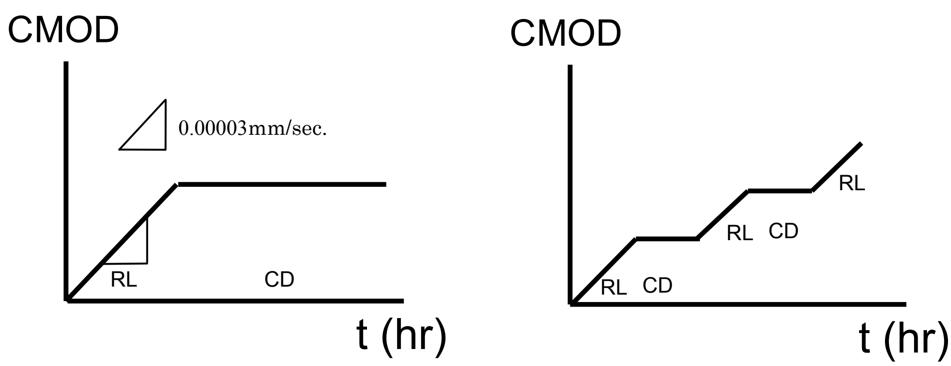


Figure 2 Loading pattern for hydrogen embrittlment fracture mechanics test

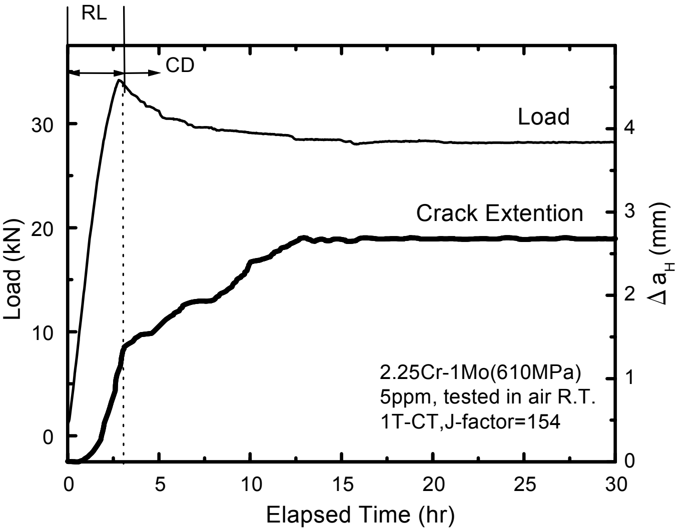


Figure3 Typical crack growth behavior of RL+CD test

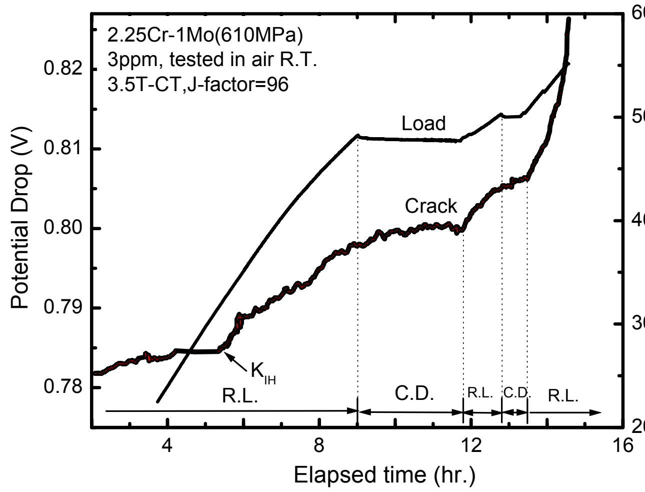


Figure 4 Crack growth behavior of repetitional RL and CD loading

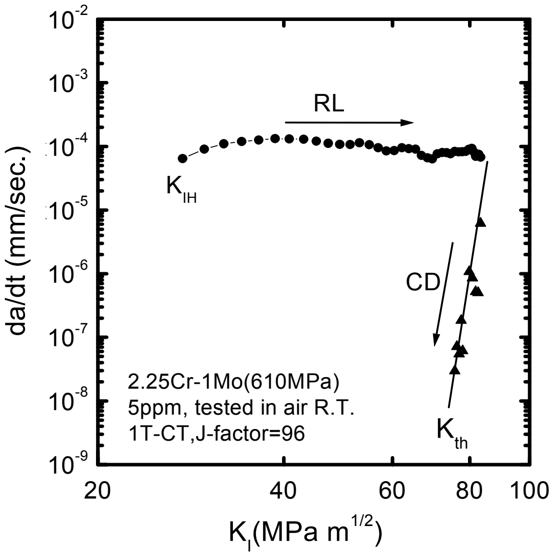


Figure 5 K and da/dt relationship of a RL+CD test

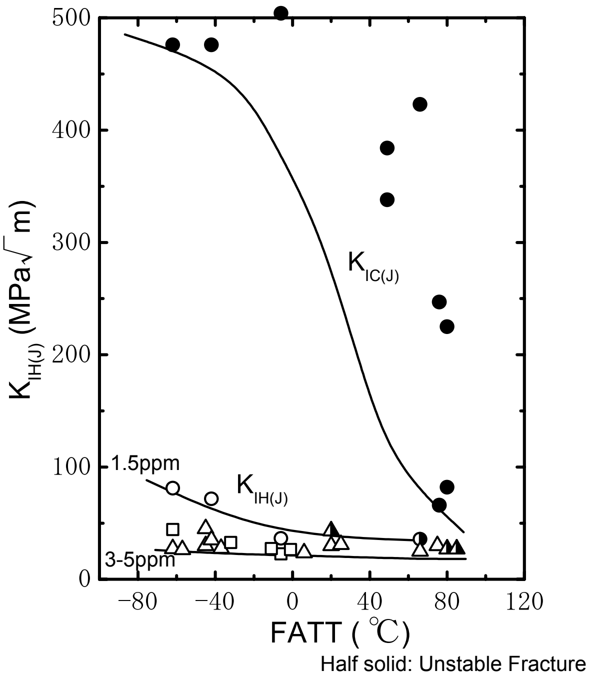


Figure6 K_{IH} and K_{IC} for a variety of temper embrittled steels

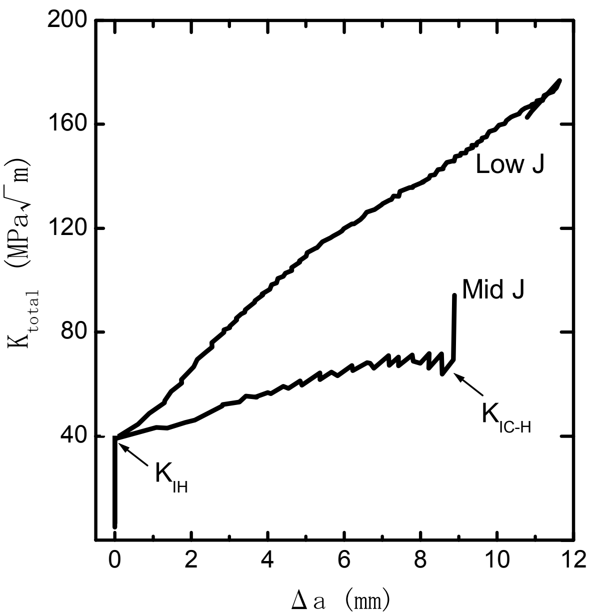
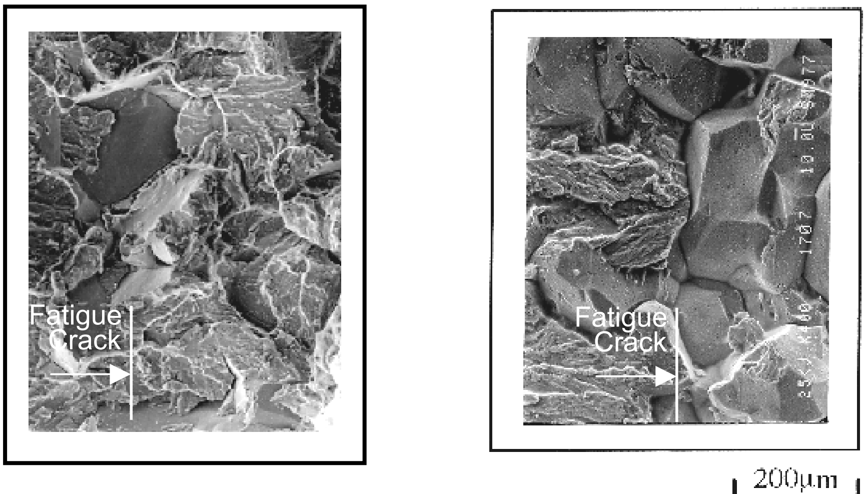


Figure 7 K-curves of Mid J and Low J heats



(a) (b)

Figure 8 Fractography of Low J (a) and Mid J (b) heats

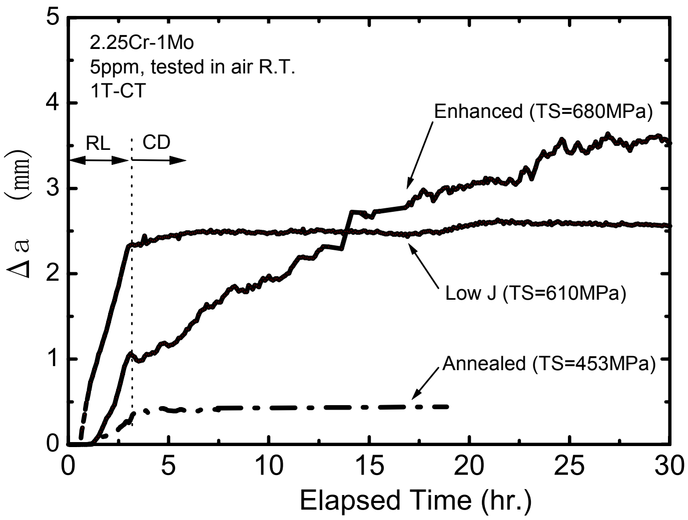


Figure 9 Crack growth measurement of RL+CD tests for various strength steel

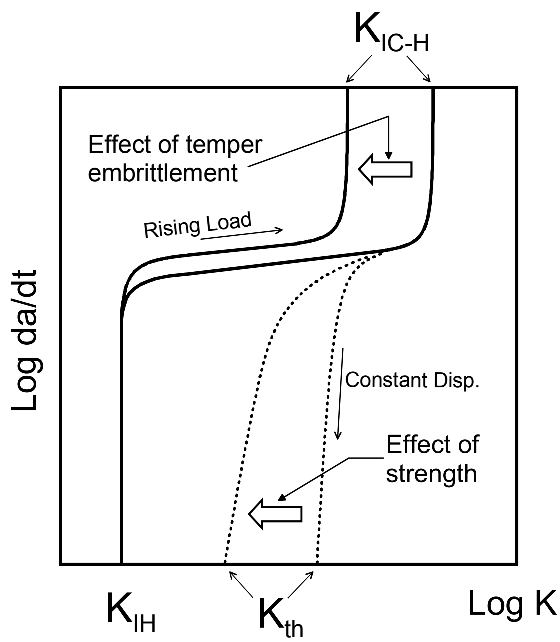


Figure 10 Schematical illustration of I.H.E. crack growth curve of 2.25Cr-1Mo steel