EFFECT OF SPECIMEN SIZE AND HYDROGEN EXPOSURE CONDITION ON  $\,K_{I\!H}\,$  TEST

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The critical stress intensity factor for hydrogen assisted cracking,  $K_{IH}$ , is one of key parameters for plant components used in hydrogen service. In order to define the minimum required size of test specimen in  $K_{IH}$  test, the size effect was studied in the paper for 2.25 Cr-1 Mo steel. It was concluded that  $K_{IH}$  can be measured by the Holding Load and Fractography method proposed by one of the authors using CT specimen as small as 1/2T size, provided that crack extension is measured at the center of the thickness of the specimen and the take-out time from an autoclave after exposing hydrogen is shorter than 40 sec.

### INTRODUCTION

For the integrity of plant components used in hydrogen service, assessment of hydrogen assisted cracking is one of important problems (1). In order to measure the critical stress intensity factor for hydrogen assisted cracking,  $K_{IH}$ , the size of test specimens in  $K_{IH}$  test is desirable as small as possible in a practical application. However, there are a few study on the specimen size effect on  $K_{IH}$  test.

In the study  $K_{\rm IH}$  test was conducted for 2.25 Cr-1 Mo steel by using holding-load and fractogrphy (HLF) method proposed by Ohtsuka and Yamamoto (2) and the effect of specimen size and the condition of hydrogen charge on  $K_{IH}$  was discussed.

## **EXPERIMENT**

### Test Specimen

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The material used in the study was 2.25 Cr-1 Mo steel. The chemical composition and the mechanical properties of the material are shown in TABLE 1 and TABLE 2, respectively. The following three types of compact tension (CT) specimen with different sizes shown in Figure 1 were used in the study.

2R type: W =50mm,  $a_0$  =25mm,  $B_0$  =25mm, and  $B_N$  =20mm 4R type: W =25mm,  $a_0$  =12.5mm,  $B_0$  =25mm, and  $B_N$  =20mm 6R type: W =25mm,  $a_0$  =12.5mm,  $B_0$  =12.5mm, and  $B_N$  =10mm

Each specimen was exposed to hydrogen in an autoclave at the temperature of 693 K and pressure of 9.8 MPa for 48 h. The time to take out the 4R and 6R specimens from the autoclave was 40 sec, while that of 2R specimen was 4 min. In order to minimize the amount of diffused hydrogen the specimens were preserved in liquid nitrogen until just starting of  $K_{IH}$  test.

### **Experimental Procedure**

The  $K_{I\!H}$  test was conducted at room temperature by using the holding-load and fractogrphy (HLF) method proposed by Ohtsuka and Yamamoto (2). In the HLF test several specimens for each type were held at different load levels for 8 h. After the test the specimen was broken at liquid nitrogen temperature and the distribution of crack extension on the crack surface of the specimens was measured by using scanning electron microscope. Comparing the relation between stress intensity factor,  $K_I$ , at the holding load and crack extension  $\Delta a_H$  at the center of the thickness of the specimen during the test,  $K_{I\!H}$  was defined as the extrapolated value of  $K_I$  where  $\Delta a_H$  reaches to zero.

TABLE 1- Chemical Composition of the Material Used (wt. %).

С	Si	Mn	P	S	Cr	Mo
0.13	0.22	0.53	0.010	0.011	2.40	1.09

TABLE 2- Mechanical Properties of the Material Used.

Yielding Stress MPa	UTS. MPa	Elongation %	Reduction of Area %
473	628	23.7	76.2

In order to estimate the hydrogen content in the specimen, some of the specimen were soaked in glycerin, and the discharged hydrogen from the specimen was measured as a function of time (Hujii and Nomura (3)).

# EXPERIMENTAL RESULTS AND DISCUSSION

### Result of HLF method

Figure 2 indicates the distribution of crack extension during the holding load test through the thickness of CT specimen. In the figure Z and  $B_N$  are the distance from the center of thickness and the net thickness at the root of side grooves of specimen, respectively. The figure shows that the crack extension near the surface during the holding load test is extremely smaller than the midsection of the specimen due to discharge of hydrogen from the surface. The fact justifies measuring of crack extension at the midsection of the specimen in the HLF method.

Figure 3 indicates the relationship between  $K_I$  and  $\Delta a_H$  during the holding load test. From the figure the values of  $K_{IH}$  for 2R, 4R and 6R specimen were estimated as 129, 92 and 95 kgf/mm<sup>3/2</sup> (40, 29 and 30 MPa  $\sqrt{m}$ ), respectively. That is, the value of  $K_{IH}$  for the smallest specimen type, 6R, was nearly equal to that of 4R. However,  $K_{IH}$  for the largest specimen type, 2R, was larger than the former two specimens. It was expected before the test that apparent value of  $K_{IH}$  will increase as the size of the specimen decreases due to the decrease of residual hydrogen in the specimen during the test, but the result was contrary.

# Cause of the Difference of Hydrogen Assisted Crack Extension

In order to study the cause of the above mentioned result the discharged hydrogen from 2R and 6R specimens was plotted as a function of elapsed time in Figure 4. The figure shows that the sum of discharged hydrogen from the 2R specimen during the holding load test which is nearly equal to the total content of residual diffusible hydrogen in the specimen before the test is less than that of the 6R specimen with smaller size.

Because the take-out time of the 2R specimen from the autoclave was longer than the 6R specimen, it is assumed that the amount of discharged hydrogen from the former specimen in the process of taking out would be more than the latter specimen and the content of residual hydrogen before the test be less than the latter one as in Figure 4. Therefore, the difference of the content of residual diffusible hydrogen in the specimen before the test is considered to be the reason of the reversal result in Figure 3.

Although the slope of the  $K_I$  vs.  $\Delta a_H$  relation of the 6R specimen with the smallest size is larger than that of 4R specimen in Figure 3, both specimens have the nearly same value of  $K_{IH}$ . The result proves that  $K_{IH}$  can be measured by the HLF method using CT specimens as small as 1/2T size (6R specimen), provided that crack extension is measured at the center of the thickness of the specimen and the take-out time from an autoclave after exposing hydrogen is shorter than 40 sec.

### **CONCLUSIONS**

- (1) The value of  $K_{IH}$  can be measured by the Holding Load and Fractography (HLF) method using CT specimens as small as 1/2T size, provided that the take-out time from an autoclave after exposing hydrogen is shorter than 40 sec.
- (2) Crack extension near the surface of a specimen during the test is extremely smaller than the midsection of the specimen due to discharge of hydrogen from the surface.

### **SYMBOLS USED**

 $a_0$  = pre-fatigued crack length of CT specimen (mm or m)

 $B_0$  = nominal thickness of CT specimen (mm or m)

 $B_N$  = net thickness at the root of side grooves of CT specimen (mm or m)

 $K_I$  = stress intensity factor (kgf/mm<sup>3/2</sup> or MPa  $\sqrt{m}$ )

 $K_{IH}$  = critical value of  $K_I$  for hydrogen assisted cracking (kgf/mm<sup>3/2</sup> or MPa  $\sqrt{m}$ )

W =width of CT specimen (mm or m)

Z = distance from the center of thickness of CT specimen

 $\Delta a_H$  =crack extension due to hydrogen assisted cracking (mm or m)

### **REFERENCES**

- (1) Subcommittee on Hydrogen Embrittlement, "Susceptibility of 2 1/4 Cr-1 Mo Steel to Hydrogen Embrittlement Cracking", Materials Division, The Japan Pressure Vessel Research Council, 1989.
- (2) N. Ohtsuka and H. Yamamoto, ASTM STP 1085, 1990, pp.69-88.
- (3) T. Hujii and K. Nomura, J. of Iron and Steel, Japan, Vol.70, No. 1, 1984, pp.104-111 (in Japanese).

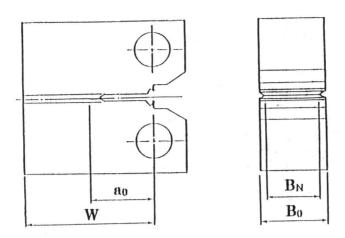


Figure 1 Configuration of CT specimen

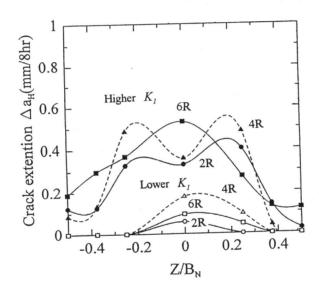


Figure 2 Through-thickness distribution of crack extension during the holding load test

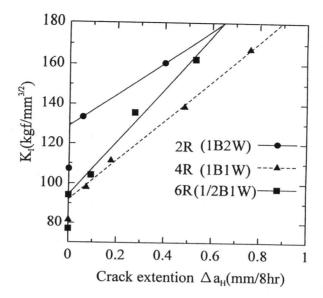


Figure 3 Relationship between  $K_I$  and  $\Delta a_H$  during the holding load test (1 kgf/mm<sup>3/2</sup> = 0.31MPa  $\sqrt{m}$  m)

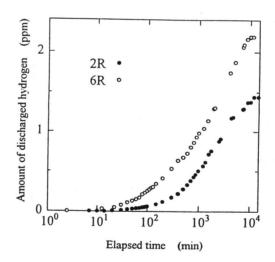


Figure 4 Discharged hydrogen from 2R and 6R specimens as a function of elapsed time