

# CRITICAL PHENOMENON IN HYDROGEN ASSISTED CRACKING IN HIGH STRENGTH STEEL

H. Yatabe<sup>1</sup> and K. Yamada<sup>2</sup>

<sup>1</sup>Fundamental Technology Laboratory, Tokyo Gas Co. Ltd.  
1-6-25 Shibaura, Minato-ku, Tokyo 105, JAPAN  
<sup>2</sup>Department of Mechanical Engineering, Keio University  
3-14-1 Hiyoshi, Kohoku-ku, Yokohama 223-8522, JAPAN

## ABSTRACT

Not only intergranular (IG) crack but also quasi-cleavage (QC) crack play a vital role in the hydrogen assisted fracture process of Q/T ASTM A490 steels. Fractographic analysis of the fracture surface of the specimen revealed that at the beginning of a fracture process a QC crack was developed at the site of a micro-defect, such as a non-metallic inclusion inherent in the engineering materials, and grew to critical size, triggering the fatal unstable growth of an IG crack, involving a change of the propagation mode from QC to IG cracking. A particular fracture toughness value associated with hydrogen degradation can be obtained from the event of unstable growth of an IG crack during the fracture test. This fracture toughness value,  $K_{IH}$ , can be explained as the critical condition for the onset of unstable growth of an IG crack or crack growth resistance of the hydrogen-damaged grain boundary. The generality of the statement was confirmed by further experiments employing a specially prepared unnotched specimen, having a fatigue precrack that triggered the onset of IG crack growth in the same manner as the QC crack. Furthermore, the discussion is extended to the essential nature of the hydrogen assisted fracture process by comparing the cracking behaviour in notched and unnotched specimens.

## INTRODUCTION

The delayed fracture or hydrogen-related fracture behaviour of high strength steels has been mostly concerned with the emphasis on either the crack propagation behaviour of the large crack from the viewpoint of fracture mechanics [1~7] or hydrogen transport phenomena in the matrix material [8~12]. These studies have made important contributions to the knowledge of fracture prevention of structural components from premature failure under hydrogen attack. However, little attention has been paid to the essential nature of the delayed fracture: What is the critical condition for the brittle fracture? How does the crack behave in matrix materials subjected to a combination of applied stress and hydrogen? These questions associated with the mechanisms of crack propagation under the influence of hydrogen remain unclear.

Fractography employing a SEM has steadily developed as a useful tool to trace the history of crack initiation and propagation in the fracture surface, especially of the crack propagation in a subsurface section of the specimen undetectable by surface observation. This fractographic analysis has successfully explained

the contribution of the QC crack as the Griffith crack to trigger the onset of growth of the fatal IG crack in unnotched specimens of the Q/T ASTM A490 and AISI 4340 type high strength steels [13~17]. This suggests that the role of the QC crack in unnotched specimens is the same as the extremely sharp notch in the ordinary delayed fracture specimens. This poses the question whether the development of the QC crack in the unnotched specimen, which has no macroscopic stress raiser, is a necessary condition for the commencement of growth of IG cracks, the critical propagation of which leads to the fracture of high strength steel specimens.

To examine the role or the properties of the IG and the QC crack propagation in hydrogen related fracture of high strength steels, the cracking behaviour of notched specimens, unnotched specimens, and unnotched specimens having a fatigue precrack will be investigated. An examination of the role of QC crack may be a key issue in understanding the fracture mechanisms of high strength steels under the influence of hydrogen.

## EXPERIMENTAL PROCEDURRE

The material employed was ASTM A490 high strength bolt steel. It was machined into smooth round tensile specimens with gauge length diameters of 5 mm and was water-quenched from the temperature of 850 • C and tempered at 200 • C for 2 hours in a vacuum of about  $10^{-4}$  torr followed by grinding and buff polishing to obtain a fine smooth surface at the gauge area of the specimens. The average diameter of the prior austenite grains was 18 $\mu$ m and the mechanical properties obtained at laboratory environment are shown in Tab.1.

TABLE 1  
MECHANICAL PROPERTIES OF QUENCHED AND TEMPERED SPECIMENS

Material	UTS (MPa)	Fracture Stress (MPa)	0.2%Proof Stress (MPa)	Reduction in Area (%)	Micro-Vickers Hardness (100g)
ASTM A490	1750	1260	1340	50	474

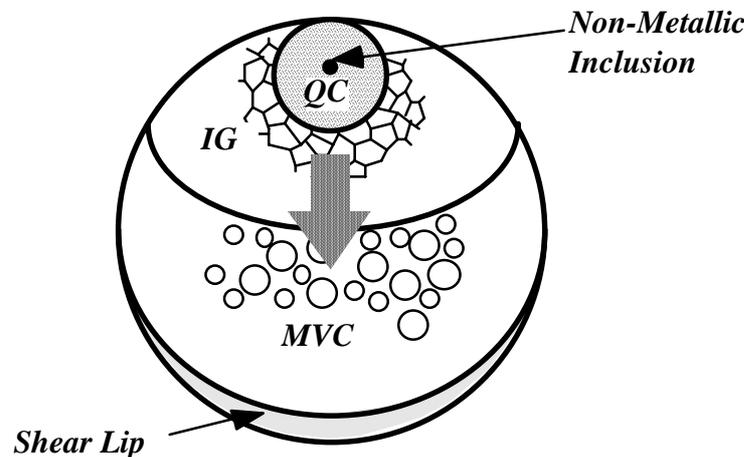
A fatigue precrack was formed by applying a push-pull cyclic load with a frequency of 10Hz in air to the specimen having a micropit as a starter notch. This micropit was made by electro-discharge machining, having a dimension of 150 $\mu$ m x 80 $\mu$ m in the surface and in-depth direction, respectively. Respective stress level and a number of stress cycles applied to the specimen were chosen as  $\sigma = 300$ MPa, which corresponds to 115~120% of the endurance limit of the material, and  $N = 1.0 \times 10^4 \sim N = 2.0 \times 10^5$  to have a controlled length of the fatigue precrack. This preparation of the precracked specimen was carried out before a vacuum annealing of 200 • C 2 hours to reduce the influence of plasticity at around the fatigue crack tip as much as possible.

The specimens were subsequently hydrogen charged cathodically through a gauge area of the surface using a platinum anode in 1N H<sub>2</sub>SO<sub>4</sub> solution at a current density of 500 A/m<sup>2</sup> under ambient temperature. The rest of the specimen surface was covered with a shielding tape to protect it from H<sub>2</sub>SO<sub>4</sub> attack. A conventional delayed fracture test was carried out using a 3-ton creep testing machine under conditions in which mechanical loading began with concurrent hydrogen charging. Tensile fracture tests of specimens which had been hydrogen charged for 24 hours under no applied stress were carried out under a constant strain rate of  $\dot{\epsilon} = 1.0 \times 10^{-5} \sim 7.0 \times 10^{-4} \text{ s}^{-1}$  under continuing hydrogen charging conditions. An observation of the morphology of crack propagation and a measurement of the depth of QC and IG facets from an outer surface to a furthest end of the crack were made on a SEM photograph.

## RESULTS AND DISCUSSION

### *The QC Crack and the Stress Raiser in the Unnotched Specimen*

Since no cracking behaviour can be detected by surface observation in the delayed fracture of unnotched specimens, fractographic analysis is useful to trace the crack propagation behaviour in a subsurface section of the specimen. A crack usually begins at a non-metallic inclusion followed by the propagation of the QC crack, the propagation of the IG crack, and the propagation of the crack developing a dimple pattern, due to micro-void coalescence (MVC) [13~17] as shown in Fig.1.



**Figure 1:** Schematic fractograph illustrating the cracking process in a smooth specimen; where QC = Quasi-Cleavage Crack, IG=Intergranular Crack and MVC=Micro-Void Coalescence. An arrow shows the direction of crack propagation.

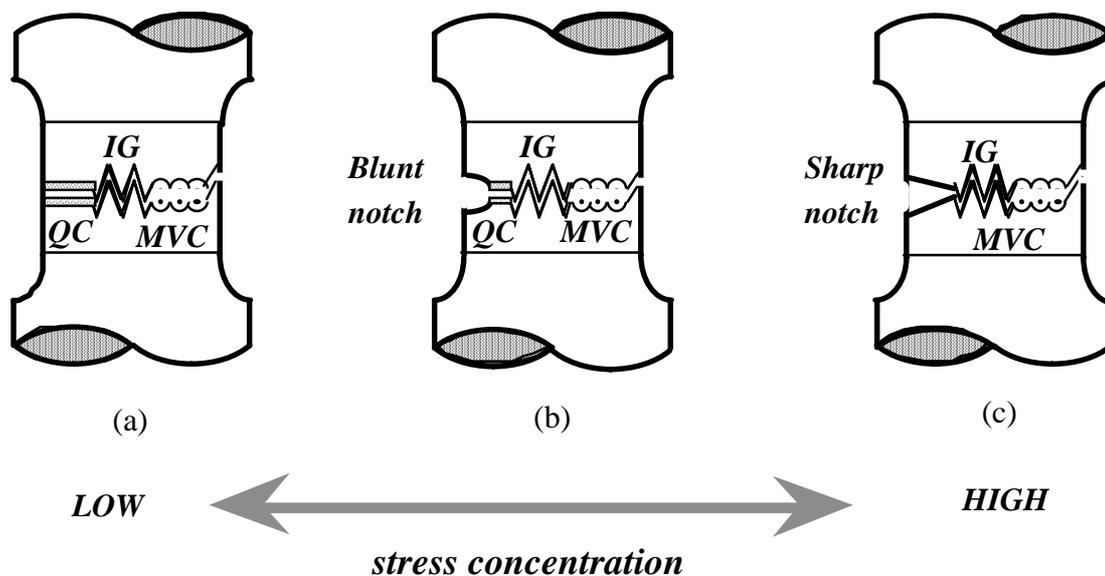
On the other hand, in sharply notched specimens having a stress concentration factor  $K_t$  larger than 7, no appreciable amount of a trace of the QC facets was observed in the fracture surface near the notch root, i.e., the IG crack started from an immediate bottom of the notch root without the help of additional stress concentration due to QC crack [16]. However, when the  $K_t$  of the notched specimen was less than 7, the development of a QC crack was needed at the onset of growth of the IG crack, such as the case typically observed in the fracture surface of the unnotched specimen.

Although a contribution of QC cracking to the onset of growth of IG cracks is still being debated, a comparison of the fracture appearances which were brought about by the same condition of the delayed fracture as shown in Fig. 2, strongly suggests that the commencement of growth of IG crack does not occur solely but does occur at QC cracks or at a notch root which should build up a high stress concentration. This may well explain the reason for the development of QC cracks, which triggers the onset of growth of IG cracks in the unnotched and in the bluntly notched specimen, and also the reason why the IG crack appears just at the root of the extremely sharp notch.

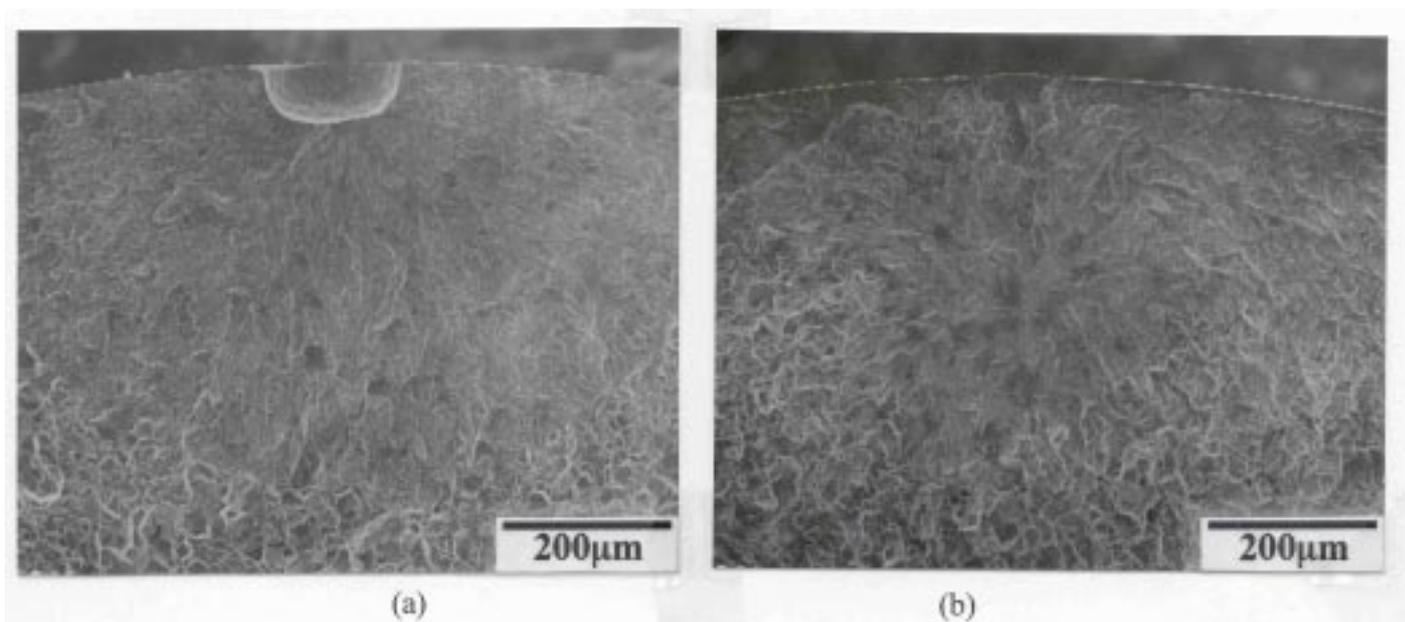
Accordingly, we may conclude that no IG cracks commence their growth without the presence of an appropriate stress raiser, such as a QC crack or a extremely sharp notch that promotes the hydrogen condensation at either the crack tip or the notch root in the hydrogen charged high strength steel.

### *The Condition for the Commencement of Growth of IG cracks under Hydrogen Environment*

To examine the critical behaviour of QC cracks as a stress raiser required for the commencement of growth of IG cracks in the delayed fracture of unnotched specimens, various lengths of fatigue precracks were brought into the specimen acting as an extremely sharp notch to simulate a mechanistic aspect of QC cracks. A similar SEM fractographic analysis of a fracture surface of the above specimens as described in the previous chapter would give a clear explanation to the necessary condition for the onset of growth of IG cracks in the delayed



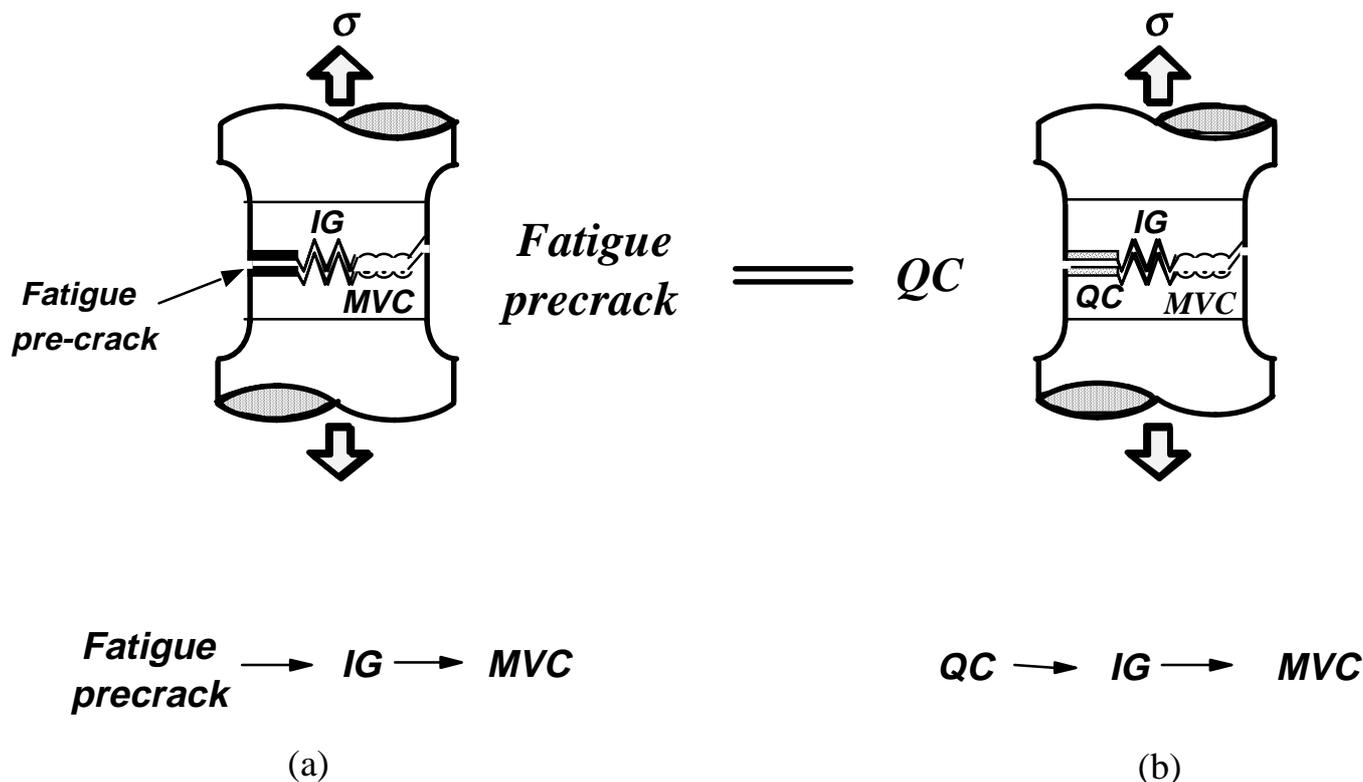
**Figure 2:** Characteristic fracture process of a hydrogen charged high strength steel in (a) an unnotched specimen, (b) a bluntly notched specimen, and (c) a sharply notched specimen



**Figure 3:** SEM fractographs of the fracture surfaces of (a) a fatigue precracked and (b) an unnotched specimen which were fractured at  $\sigma = 365\text{MPa}$ , after 101min.,  $\sigma = 350\text{MPa}$ , 240min., respectively, under the hydrogen charging condition of  $500\text{A/m}^2$ .

fracture of the high strength steel. Characteristics of the appearance of the fracture surfaces of the unnotched specimens with the fatigue precracks were compared to those of QC cracked specimens, both of which had been subjected to an identical condition of delayed fracture tests.

Figure 3(a) shows a SEM photograph of the fatigue precracked specimen, while Fig. 3(b) shows that of the QC cracked specimen. It should be noted in Fig. 3(a) that no appreciable trace of the QC facet is observed along the narrow band between the fatigue crack and the IG facets. No significant differences are observed between the two fracture surfaces shown in Fig. 3 except the fatigue cracked and the QC cracked facets, both of which occupy the area in front of the IG facets. In other words, the IG crack started from the immediate tip of the fatigue precrack.



**Figure 4:** Schematic illustrations showing the crack propagation process of unnotched specimens with (a) a fatigue precrack (b) a QC crack

Although the discussion is limited to the mechanistic aspect of the delayed fracture, the above mentioned results well account for the fact that the role of the fatigue precrack as a stress raiser in the development of the IG crack is equivalent to the QC crack as illustrated in Fig.4. This may lead to the conclusion that the development of IG cracks always requires a trigger crack such as a QC or a fatigue precrack which should build up the extremely high stress concentration to promote the hydrogen condensation and also that there is no essential difference in the cracking process of delayed fracture between the sharply notched and the unnotched specimens except that a period of the QC crack initiation is needed for the unnotched specimen.

Furthermore, if we study delayed fracture behaviour employing only a sharply notched specimen which may be regarded as the standardised test specimen simulating the geometry of a root of bolt screw under hostile environments, then we definitely lose finding a critical matter to understand an essential mechanism of the fracture behaviour of the high strength steel under hydrogen environment.

#### ***The Critical Stress Intensity Factor for the Commencement of IG Crack***

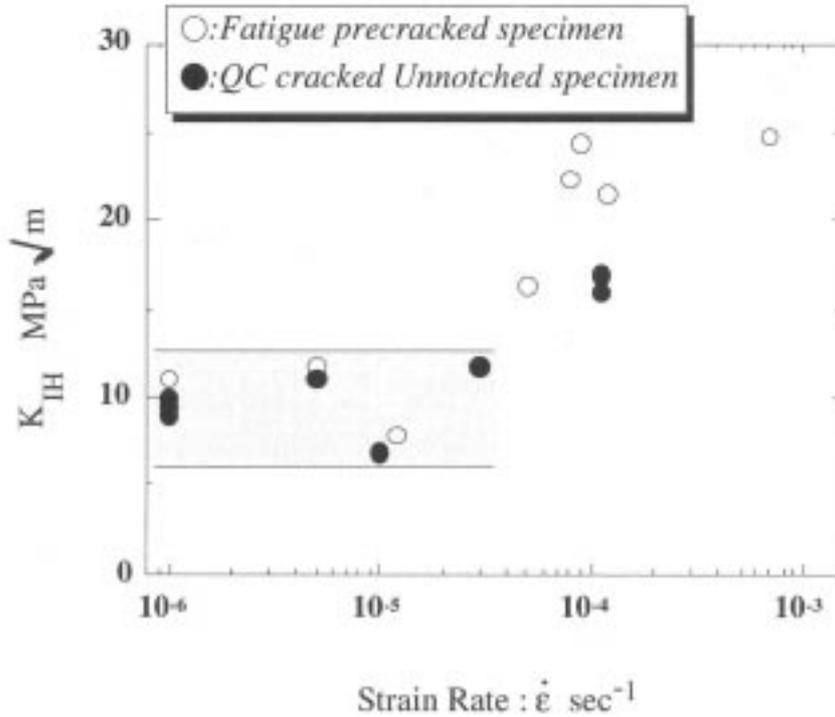
Two types of unnotched specimens, one has a fatigue precrack and the other has a QC crack, were prepared for conventional fracture tests with various constant strain rates. The QC cracks in unnotched specimens were formed by preliminary hydrogen charging with a current density of  $500\text{A/m}^2$  for 24 h, details of that were given in elsewhere [11,15,16]. Fatigue precracked specimens were treated in the same hydrogen charging condition as the QC cracked specimens to give an identical history of hydrogen treatment. The fracture tests of the two kinds of specimens brought out common features of crack propagation, though the onset of unstable growth of the IG crack began from different types of trigger cracks. The onset of this IG crack growth may be regarded as a critical event that is directly related to the magnitude of crack growth resistance of a damaged grain boundary due to hydrogen penetration.

An attempt to evaluate the stress intensity factor at the onset of unstable crack growth using the following equation should give the critical stress intensity factor to the unstable growth of an IG crack,  $K_c$  or  $K_{IH}$ , the crack growth resistance of the hydrogen damaged grain boundary.

$$K_c = \alpha \sigma_f (\pi L)^{1/2} \quad (1)$$

Where  $\alpha$ : geometrical constant ( $\alpha=1$  for the present geometry),  $\sigma_f$ : the fracture stress,  $L$ : the critical crack length.

The substitution of particular values obtained from the fracture tests of the fatigue precracked and the QC cracked unnotched specimens into the Eq. (1) gives a particular value of  $K_c$  which can be regarded as the fracture toughness value of the hydrogen charged high strength steel,  $K_{IH}$ . This  $K_{IH}$  value involves two different critical cracks, the QC crack and the fatigue precrack, both of which triggered the onset of the unstable growth of IG crack.



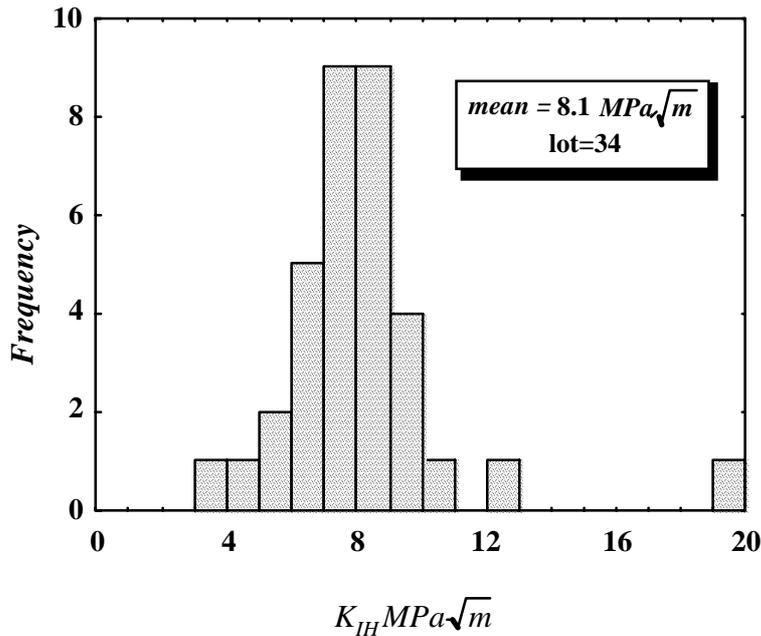
**Figure 5:**  $K_{IH}$  under hydrogen charging of fatigue precracked and QC cracked specimens (ASTM A490)

The results of  $K_{IH}$  values are shown in Fig. 5 with a parameter of various strain rates ranging from  $10^{-6}$  to  $10^{-3} \text{ s}^{-1}$ . At higher strain rate ranges ( $> 1.0 \times 10^{-5} \text{ s}^{-1}$ ) the  $K_{IH}$  have scattered values which would be explained by unstable behaviour of hydrogen diffusion and condensation at the crack tip with respect to the higher strain rate loading, while at lower strain rate ranges ( $< 3 \times 10^{-5} \text{ s}^{-1}$ ) the  $K_{IH}$  remained well within a scatter band of  $8 \sim 12 \text{ MPa}\sqrt{\text{m}}$ .

These values were compared to the results of the published data as follows. The  $K_{IH}$  values of the AISI 4340 steel having a yield strength level of about 1600 MPa are about  $20 \text{ MPa}\sqrt{\text{m}}$  [18] which are slightly higher in value than the present data. However, a good coincidence of values was found with the results of Banerji [19], McMahan [6], Aoki [20], and Nakamura [21]. This implies that the  $K_{IH}$  values of  $8 \sim 12 \text{ MPa}\sqrt{\text{m}}$  which correspond to the crack growth resistance of the hydrogen damaged grain boundary, can be regarded as the most probable  $K_{IH}$  value of the hydrogen damaged high strength steel.

#### **Reliable Values of $K_{IH}$ and the Significance of QC Cracks**

If the onset of critical growth of IG cracks in the delayed fracture of high strength steel begins simply with a trigger crack, such as a QC crack or a fatigue precrack which develops hydrogen condensation at the crack tip [3,22] as above described, all the critical  $K$  values calculated from the fracture stress and the critical QC crack length of the delayed fracture tests of unnotched specimens of ASTM A490 should also be classified into the same  $K_{IH}$  category as shown in Fig. 6.



**Figure 6:** Histogram of  $K_{IH}$  obtained from delayed fracture tests of ASTM A490

A mean value of the histogram is calculated as  $8.1 MPa\sqrt{m}$  which coincides well with the previous results obtained from the tensile fracture tests of both hydrogen charged fatigue precracked and QC cracked unnotched specimens.

From the study mentioned above, it seems that we can conclude that one of the most essential matters in the delayed fracture of high strength steel is the initiation of a QC crack rather than the growth of an IG crack, which has been well known as a critical feature in the delayed fracture phenomenon. In conventional delayed fracture tests employing sharply notched specimens that simulate the geometry of the bottom of a bolt screw, the stage of QC crack initiation often disappears so that only the growth of IG crack has been high-lighted on the fracture surface, at the expense of the importance of the development of a QC crack.

## CONCLUSION

- (1) The most essential nature of the behaviour in delayed fracture of high strength steel is the development of a QC crack rather than the development of an IG crack.
- (2) A stage of the development of a QC crack disappears in the delayed fracture of high strength steel if the specimen has a high stress concentration factor due to the extremely sharp notch or fatigue precrack geometry.
- (3) The fracture toughness value,  $K_{IH}$ , of a hydrogen charged high strength steel such as ASTM A490, is about  $8 MPa\sqrt{m}$  which may be regarded as crack growth resistance of the hydrogen damaged grain boundary and which corresponds well with a number of published data.

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## Figure and Table Captions

Table 1 Mechanical properties of quenched and tempered specimens

Figure 1: Schematic fractograph illustrating the cracking process in a smooth specimen; where QC = Quasi-Cleavage Crack, IG=Intergranular Crack and MVC=Micro-Void Coalescence. An arrow shows the direction of crack propagation.

Figure 2: Characteristic fracture process of a hydrogen charged high strength steel in (a) an unnotched specimen, (b) a bluntly notched specimen, and (c) a sharply notched specimen

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specimen which were fractured at  $\sigma = 365\text{MPa}$ , after 101min.,  $\sigma = 350\text{MPa}$ , 240min., respectively, under the hydrogen charging condition of  $500\text{A/m}^2$ .

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