# **Critical Aspects of the Crack Development in the Delayed Fracture of Structural Steels**

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**ABSTRACT.** A mechanistic aspect of the susceptibility to the delayed fracture was studied with an emphasis on the critical behaviour of the subsurface growth of Quasi-Cleavage (QC) and Inter-Granular (IG) cracks. The materials employed were 0.35%C plain carbon steel S35C and boron added bolt steel Bolten110N which were quenched and tempered to have various levels of yield strength ranging from 500~1400MPa. These were put into sustained load fracture test with cathodic hydrogen charging. The delayed fracture strength was evaluated by the threshold stress ( $\sigma_{th}$ ) at the elapsed time of  $10^4$  minutes. Fractographic analysis shows us that QC-IG-MVC (Micro-Void-Coalescence) cracking process can be essential aspects in the delayed fracture of steels. A low susceptibility to delayed fracture in low strength steels can be explained by the absence of IG crack in the crack growth process where the crucial blunting occurred at the crack tip.

#### INTRODUCTION

There still remains a lot of discussion about the issue of premature fracture of structural components due to hydrogen degradation under corrosive environments. Since this sort of fracture that developed considerably below the yield strength level, is quite well known for high strength steels [1~7], the material selection involving high strength steels would meet with highly complicated problems. Pipeline steels for crude oil and natural gas etc, still encounter a difficult problem of premature fracture in SCC (Stress Corrosion Cracking)[8], hydrogen induced cracking (HIC)[9,10] associated with the hydrogen degradation [11] of the materials even in the case of using low strength steels. An intergranular (IG) type of fracture is notable in high strength steel while in low strength steels no appreciable trace of IG fracture is noticeable in the hydrogen related fracture [11,12]. According to these evidences of the fracture morphologies, there still remain unknown issues on the crack development in either high and low strength steels under hydrogen attack.

To examine a previously mentioned issue, some types of structural steels such as boron added steel and plain carbon steel were prepared as particular specimens having various levels of yield strength ranging from 500MPa to 1400MPa. These specimens were then put into sustained load fracture tests with concurrent hydrogen charging in order to study the mechanistic aspects of the hydrogen effects on the crack initiation and propagation characteristics.

#### **EXPERIMENTAL PROCEDURE**

The materials employed in this study were a boron added bolt steel Bolten110N and a plain carbon steel S35C with chemical compositions as shown in Tab. 1 and with mechanical properties as shown in Tab. 2 respectively.

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Materials	С	Si	Mn	Р	S	Ni	Cr	Mo	Cu	В	Al
S35C	0.38	0.19	0.70	0.015	0.009	0.06	0.14	0.01	0.06	0.0002	0.002
Bolten110N	0.20	0.16	0.77	0.012	0.010	0.03	0.63	_	0.02	0.0022	0.066

Table 1 Chemical compositions of specimens

 $(xx \neq 0/)$ 

These materials were machined into unnotched shape with a gauge length and a mid-section diameter of 10mm and 5mm respectively.

To obtain martensitic structure with an average prior austenite grain diameter around  $20\mu$ m, the Bolten110N specimen was first austenitized at 1223K for 10 minutes in electro-furnace, followed by oil quenching. The S35C steel was austenitized at 1173K for 10 minutes in electro-furnace, followed by oil quenching to arrange a quenched martensitic structure. These specimens were mechanically polished, and then tempered at 473K, 523K, 573K, 623K, 673K, 773K, 873K and 973K for 1 hour in vacuum furnace with 1.3x10<sup>-4</sup>Pa to have a variety of strength levels as shown in Tab. 2.

Magnitudes of mechanical properties in the Tab. 2 are averaged results of at least two specimens. The threshold stresses of the delayed fracture were estimated from the test results of 5-10 specimens as shown later in Figs. 1 and 2. The levels of the threshold stress were determined as the maximum stress at which the fracture did not occur at the elapsed time of  $10^4$ .

The specimen surface was covered with a shielding tape to prevent damage from an attack by  $H_2SO_4$  except the gauge area. Sustained load type fracture tests were carried out using a 3 ton creep-testing machine under the conditions, in which the mechanical loading began with hydrogen charging in  $H_2SO_4$  solution having 0.05M concentration and 500A/m<sup>2</sup> current density during the test. A trace of crack path on the fracture surface was then examined using scanning electron microscope (SEM).

#### **RESULTS AND DISCUSSION**

#### **Results of Delayed Fracture Test**

A delayed fracture test with the above hydrogen charging condition was carried out for all kinds of specimens as shown in Tab. 2. Figure 1 shows the relationship between the

fracture stress and the time to fracture under hydrogen attack using quenched and tempered S35C specimens. The results of Bolten110N specimens are also shown in Fig. 2. These fracture curves level off at about  $10^3$  minutes so that applied load was discontinued at  $10^4$  minutes to estimate the threshold stress. Threshold stress ( $\sigma_{th}$ ) was then determined as the maximum stress at which the fracture did not take place after  $10^4$  minutes.

Materials	Tempering temperature (K)	Yield strength (MPa)	U.T.S (MPa)	Micro-Vickers hardness (P=500g, 30points)	Threshold stress (MPa)	Elasped loading time (minutes)
S35C	473	1330	1630	494	150	10 <sup>4</sup>
	573	1224	1471	443	350	10 <sup>4</sup>
	673	1170	1307	406	500	10 <sup>4</sup>
	723	1071	1179	362	650	10 <sup>4</sup>
	773	970	1096	334	600	10 <sup>4</sup>
	973	650	697	215	475	10 <sup>4</sup>
Bolten110N	473	1205	1372	440	600	10 <sup>4</sup>
	523	1204	1367	434	350	10 <sup>4</sup>
	573	1271	1308	446	250	10 <sup>4</sup>
	673	978	1197	368	400	10 <sup>4</sup>
	723	785	998	302	450	10 <sup>4</sup>
	973	462	707	186	410	10 <sup>4</sup>

Table 2 Mechanical properties of the specimens



Fig.1 Delayed fracture curve of hydrogen charged specimen (S35C).



Fig.2 Delayed fracture curve of hydrogen charged specimen (Bolten110N).

To have an extensive understanding on the nature of delayed fracture, the threshold stress ( $\sigma_{th}$ ) was normalized by the yield strength ( $\sigma_y$ ) as shown in Fig.3.



Fig.3 Relationship between resistance of delayed fracture and yield strength.

The results show that the normalized delayed fracture strength decreases with the increase in the yield strength, and a remarkable change in fracture susceptibility can be seen at about the yield strength level of 1000MPa. Then, to examine the detailed aspects of crack development during the fracture process in all range of yield strength, fractographic analysis was made with SEM.

**Difference in Morphology of Crack Development in High and Low Strength Steels** It has long been considered that the characteristic feature of crack growth under hydrogen attack are influenced by the yield strength level, such that IG crack is noticeable in high strength steels and that no IG crack is appreciable in low strength steels [3]. A characteristic feature of crack development in the fracture surface gave us a notable information that the QC crack first appears in the subsurface crack growth process in both high and low strength steels rather than the expected IG crack development as shown in Fig.4.



Fig.4 Illustration of the morphology of crack propagation in delayed fracture.

This QC crack mode continues to grow up to the transition point where the growth mode changes into IG mode with the unstable growth in high strength steel specimen, while in low strength steel specimen, no transition to IG crack was identified in the crack growth process.

Accordingly, it can be concluded that the susceptibility to delayed fracture is related to the fact that the high susceptibility prefers the IG crack growth triggered by the QC crack resulting in a fast fracture while no IG crack appears in the low susceptibility case. To elucidate the mechanistic aspects of the susceptibility in delayed fracture, a role of the development of QC crack, which triggers the onset of IG crack growth, was carefully examined in the following chapter.

#### Crack Tip Blunting and IG Crack Development

QC cracks in low strength steels show a particular mode of crack growth different from that in high strength steels. The fractographic analysis revealed as shown in Fig.4 that the QC crack grows with a coalescence of a number of neighboring QC cracks under a relatively long period of time followed by a development of MVC not the IG cracks

leading to fast fracture. The QC cracks should remain as non-propagation cracks (NPC) when the applied stress is not enough to boost further crack coalescence to fast fracture.

From the mechanistic viewpoint the most probable reason for the QC crack to propagate or to remain as NPC is a role of plasticity at the crack tip, namely, the crack tip blunting. If a substantial plasticity is built up at the crack tip in low strength steels, the hydrogen diffusion and concentration should be decreased to a certain extent [12] compared with the high strength steels, which keep a sharp crack tip configuration with little plastic deformation. A reduction in the stress intensity and reduced hydrogen accumulation at the crack tip result in the difficulty of the onset of IG crack growth from the QC crack front.

Bolten110N tempered at 473K showed a slightly different crack propagation behaviour that the QC crack developed at the beginning continues to grow along the QC like facet, QC' facet having a rough and uneven surface, not the IG facet leaving appreciable plastic deformation.

The question arises as to whether the crack tip blunting actually behaves as we expected.

A configuration of the tip of QC crack was then examined on the specimens which had already been loaded for  $4.32 \times 10^3$  minutes to produce QC crack in the specimen under sustained load at the stress level of 150MPa and 600MPa for S35C specimen and 250MPa and 600MPa for Bolten110N specimen, both of which correspond to the respective threshold stress of the delayed fracture. A metallographical examination on the cross section of the gauge section of specimen was made after mechanical polishing to see the crack tip configuration as shown in Fig.5 (a)(b)(c)(d).



Fig.5 The optical micrographs of a crack shape.

These micrographs show that two different crack tip morphologies of the initial QC

cracks have developed in both types of materials tested. The crack tip is very sharp with no appreciable plasticity in S35C steel tempered at 473K as shown in Fig.5 (a) and also Bolten110N tempered at 573K in Fig.5(c). On the other hand, in the cases of S35C steel tempered at 773K and Bolten110N tempered at 473K, both crack tips show substantial plastic deformation at the tip and the flank of the crack as shown in Fig. 5(b)(d). From these evidences, the reason for the difficulty for the onset of IG crack growth from the QC crack in the low strength steel may be explained that the crack tip blunting is easily built up in the case of S35C having low strength level and this may suppress the hydrogen accumulation near the crack tip [13] resulting in the disappearance of IG crack growth which should be triggered by the QC crack in the crack growth process. This explanation is also applicable to the case of crack tip blunting of Bolten110N tempered at 473K though it has a high yield strength.

#### Model of Crack Development in Delayed Fracture of Steels

Materials	Tempering temperature (K)	Yield strength (MPa)	$\begin{array}{c} \text{Resistance of} \\ \text{delayed fracuture} \\ (\sigma_{th}/\sigma_y) \end{array}$	Crack propagation	Crack shape
S35C	473	1330	0.11	QC→IG→MVC	sharp
	773	970	0.62	QC→MVC	blunt
Bolten110N	473	1207	0.50	QC→QC'→MVC	blunt
	573	1271	0.20	QC→IG→MVC	sharp

Table 3 An essential aspect in the crack growth process

This table3 gives the obtained results of crack propagation analyses in a simple model. It is obvious that the initial crack is always the QC crack irrespective of the strength level of the specimens and also that the basic manner of crack growth process consists of the QC crack, IG crack, and MVC in the delayed fracture. However, in the case of low susceptibility case, the IG cracking process may disappear due probably to the crack tip blunting resulting in difficulty of the hydrogen concentration and of the high stress intensity.

The model of the sequence of crack growth "QC-IG-MVC" can be considered as an essential aspect in the crack propagation mechanism regardless of the level of the yield strength in steels under the delayed fracture process. This model of crack growth fully appears in high susceptibility case while the IG crack disappears in low susceptibility case. This event of disappearance of IG crack in low susceptibility case can be well explained by the role of plasticity around the QC crack.

### CONCLUSION

A mechanistic aspect of the susceptibility to delayed fracture of structural steels was studied using unnotched type of specimens with an emphasis on the subsurface crack development mechanisms using various types of steels which have yield strength levels ranging from 500MPa to 1400MPa. Results obtained are summarized as in the followings:

(1) The delayed fracture strength remarkably decreases at the particular strength level about  $\sigma_y$ =1000MPa in S35C and Bolten110N steels. The development of the subsurface QC crack is common and fundamental phenomenon in the crack growth process in high and low strength steels under hydrogen attack.

(2) The crack growth process "QC-IG-MVC" is an essential aspect to the fracture of unnotched specimen under hydrogen attack. This model of crack growth fully appears in the high susceptibility case, while the IG crack disappears in the low susceptibility case. This difference can be well explained by the critical event of crack tip plasticity.

(3) The susceptibility to delayed fracture in steels can be explained from the mechanistic aspects of crack tip blunting depending on the capability for plastic deformation under hydrogen attack.

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