INITIATION OF CRACKS AT DELAYED FRACTURE OF A HIGH STRENGTH STEEL

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INTRODUCTION

Delayed fracture of high strength steels is sensitively dependent on minor alloying element as well as microstructures. Although metallurgical means for improvements are still remained for extension, a unified view on hydrogen embrittlement is also required to minimizing the efforts.

Most theories previously proposed with hydrogen embrittlement of steels deal with stability of an incipient crack [1 - 4]. While it is well accepted that kinetics of delayed fracture is governed by mobility of hydrogen in steels, the steps resulting in the formation of incipient cracks and their linking or propagation should be revealed. Experimental difficulties exist for providing decisive evidences for various models.

In the present study, results obtained by an acoustic emission technique for the investigation of the incubation period of delayed fracture [5] are discussed together with new findings on the role of nitrogen on delayed fracture of a high strength steel [6].

ACOUSTIC EMISSION INVESTIGATION OF INCUBATION PERIOD

Delayed fracture tests were conducted with a steel the chemical composition of which is shown in Table 1. The macrostructure is martensite tempered at 573°C to give tensile strength of 1500 MPa. The delayed fracture test was in 0.1 N HCl solution by a cantilever type bending using V-notched specimens at constant loads. The specimens were 10 mm x 10 mm in cross section and the notch depth was 2 mm with root radius of 0.1 mm. The initiation of a macroscopic crack and its growth were monitored by an electric resistance technique across the notch root. Increase in the resistance indicating the initiation of the crack growth from the notch root take place after a long incubation period. The fraction of incubation period to the total fracture time amounted to 99.8%.

Although there revealed no apparent crackings in a specimen during incubation period, many acoustic emissions were detected as shown in Figure 1. The acoustic emission instrument employed was designed so as to detect acoustic emissions in the frequency range of 100 kHz ~ 2 MHz with amplification as high as 100 db. A coincidence gate system was devised to detect acoustic emissions generated only at the notch root area and also made possible to separate the longitudinal wave mode from the transverse one. The apparatus equipped a wave form monitoring device by means of a transient recorder connected to main amplifier. The frequency response of the apparatus was governed by that of the transducer, which covered from 100 kHz to 2 MHz.

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The acoustic emissions generated in the incubation period were mostly transverse waves and those generated in the crack growth stage were like wave forms as shown in Figure 2a while those of the latter were by random wave forms as shown in Figure 2b.

In order to discern the process leading to the generation of packet-like acoustic emissions, those generated during continual tensile testing were stage of deformation and just after the start of stress relaxation or plastic deformation is a source of such packet-like acoustic emissions and similar events take place at incipient period of delayed fracture.

When specimens subjected to delayed fracture test were examined, incipient daries at the notch root where hydrostatic stress is maximum. Thus it formation. Actually, packet-like signals following random amplitude ones packet-like signals, release of elastic energy may occur in the motion of dislocation groups.

Under a concept that hydrogen reduces the frictional stress of dislocation, Seachem has proposed a "Hydrogen Assisted Cracking" model [7]. In this context, in the present study is related to the formation of incipient cracks or leads to accumulation of plastic strain to a critical value.

**Effect of Nitrogen and Related Intergranular Fracture**

A feature of delayed fracture was obtained by investigating the effects of minor alloying elements. The amount of soluble nitrogen in aluminum in steels having similar compositions of Table 1. Delayed fracture 9.2 mm/s in 3% NaCl solution were conducted. Figure 3 shows that amount of soluble nitrogen. The amount was experimentally determined by a hot-extraction method [8] which detects nitrogen converted into NH₃. Temperature according to the states of nitrogen in the steel. Soluble in the present case as nitrogen except as TiN, BN and not always below 673 K, but also in higher temperature range. Trend was obtained at tests in 0.1 N HCl.

Associated with the increase in the amount of soluble nitrogen, the fracture mode at delayed fracture changed from transgranular to intergranular mode. In transgranular testing of the same steels at room temperature, nitrogen produce intergranular fracture in impact testing of a steel when however, neither nitrogen nor hydrogen solely produced intergranular embrittlement is enhanced by the coexistence of nitrogen along grain boundaries.

It has been shown that temper embrittlement of a high strength steel reduces the critical stress intensity at delayed fracture which is featured by intergranular fracture mode [10]. Such a cooperative relation was attributed to the decrease in cohesive force along grain boundaries. An important point was whether the cooperative relation between temper embrittlement and hydrogen embrittlement is a simple superposition of each effect or there exists any interaction. For the case of temper embrittlement, intergranular fracture takes place irrelevant of the existence of hydrogen. So that the present case can be regarded to suggest the interaction between nitrogen and hydrogen.

**DISCUSSION**

A characteristic of delayed fracture is that macroscopic cracking takes place after a long incubation period, and steps leading to crack initiation are of primary importance.

It has been well accepted that hydrogen concentrates at notch root where hydrostatic stress builds up by triaxial constraint. It is due to accommodation of elastic strain energy or in more general terms to make uniform chemical potential of hydrogen [11]. Hydrogen concentration C under hydrostatic stress \( \sigma_h \) is given as

\[
C = C_0 \exp \left( \frac{\sigma_h V_H}{RT} \right)
\]

where \( C_0 \) is hydrogen concentration at \( \sigma_h = 0 \), and \( V_H \) is partial molar volume of hydrogen in steels.

As the mechanism of embrittlement by concentrated hydrogen, bond weakening by lattice expansion is not possible since elastic stress should be rather relaxed. The present study suggests interaction of nitrogen and hydrogen cause the embrittlement of grain boundaries. If segregation of nitrogen occurred first at grain boundaries, it may have reduced elastic misfit there, and enhanced embrittlement due to hydrogen is not expected so far as elastic energy is considered. It can be suggested that another mechanism, presumably due to a change of bond nature, contributes to the reduction of cohesive force.

If hydrogen reduces cohesion, it can be expressed as the reduction of Young's modulus of atomic bonding or of the surface energy when a crack is opened. The condition that a crack opens is expressed so that the tensile stress on the crack surface \( \sigma_c \) satisfies

\[
\sigma_c = \sqrt{\frac{E \gamma}{a}}
\]

where \( E \) is the Young's modulus, \( \gamma \) is the surface energy and \( a \) is the atomic distance.

Equation (2) implicitly postulates intense clustering of hydrogen for defining reduction of surface energy. The picture that is clustered of hydrogen will form an area where cohesive force is lowered, and pre-existing large stress concentration opens a crack in a discontinuous manner. Indeed, the existence of large stress concentration is necessary for satisfying equation (2), since \( \sigma_c \) in equation (2) is much higher than the flow stress in spite of the expected decrease in \( E \) or \( \gamma \). Stress con-
centration by mechanical notch is insufficient, and very sharp cracks or
pile ups of dislocations are probable stress concentrators. It is expected
that acoustic emissions are produced, then, by relaxation of dislocation
pile-ups, following the opening of the crack.

It should be noted that equation (2) is not equivalent to Griffith-Drowan
criterion which deals with the instability of an incipient crack. For
the instability criterion of a crack, the reduction of $\sigma_s$ is negligible
compared with plastic energy $\bar{\varepsilon}$, and will not contribute to the onset of
unstable fracture. Actually, weakening of cohesive force should be
effective for the initiation of a crack, since release of stress concentra-
tion and probable blunting of a crack tip will prevent the extension of
the crack bond by bond.

For the propagation of such cracks, a reasonable model is a linking by
unstable shear [12]. Fractography of hydrogen embrittlement [7,13]
characterized by rather ductile features can be understood on this basis.
Figure 4 illustrates the present model for hydrogen embrittlement.

Figure 4(a) and (b) illustrate schematically crack initiations at grain
boundaries or non-metallic inclusions promoted by hydrogen clustered to
triaxially stressed areas. Nitrogen along boundaries is expected to
enhance the embrittlement due to hydrogen. Stress concentration produced
by dislocation pile-ups can be released by the opening of a crack accom-
panying emissions of stress waves. Figure 4(c) is a representation of
linking of cracks by ductile shear.

Figure 5 is high resolution scanning electron micrographs of an area
below delayed fracture surface. Figure 5 shows micro-cracks and their
linking along grain boundary (a) and inside of a grain (b).

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Table 1 Chemical Composition of the Steel

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>Cr</th>
<th>Mo</th>
<th>Ti</th>
<th>Al</th>
<th>B</th>
<th>N</th>
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<td>0.20</td>
<td>0.75</td>
<td>0.70</td>
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<td>0.06</td>
<td>0.06</td>
<td>0.002</td>
<td>0.004</td>
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Figure 1 Monitor of Crack Growth by Electric Resistance Measurement and
Acoustic Emissions at Delayed Fracture Test of the Steel in
0.1 N HCl Under Nominal Bonding Stress of 1650 MPa
Figure 2  Acoustic Emission Wave Forms (a) At Incubation Period and (b) Crack Growth Stage. The Scales of Abscissa and Coordinate are 15.1 μs/div and 1.56V/div, Respectively.

Figure 3  Effects of Soluble Nitrogen on the Critical Stress of Delayed Fracture

Figure 4  A Model of Hydrogen Embrittlement:
(a) Pile-Up of Dislocations at Grain Boundaries and Nonmetallic Inclusions and Hydrogen Clustering at Stressed Area
(b) Crack Opening At Stressed Area
(c) Linking of Cracks by Shear
Figure 5 High Resolution Scanning Electron Micrographs of an Area Below Delayed Fracture Surface
(a) Micro-Voids and Their Linking Along Grain Boundaries
(b) A Microcrack Inside of a Grain and Its Linking with the Main Crack