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INITIATION OF CRACKS AT DELAYED FRACTURE OF A HIGH STRENGTH STEEL

M. Nagumo*

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 microstructure is martensite tempered at 573°K 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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*Fundamental Research Laboratories, Nippon Steel Corporation, 1618 Ida, Nakahara-ku, Kawasaki-City, JAPAN.

The acoustic emissions generated in the incubation period were mostly transverse waves and those generated in the crack growth stage were longitudinal ones. The signals of the former were characterized by packet-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 ordinal tensile testing were compared. Then, packet-like signals could be observed at rather early stage of deformation and just after the start of stress relaxation or unloading. It is natural to conjecture that small scale, discontinuous 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 cracks were often observed along non-metallic inclusions or grain boundaries at the notch root where hydrostatic stress is maximum. Thus it remains possible that packet-like signals are due to incipient crack formation. Actually, packet-like signals following random amplitude ones were sometimes observed. Then, even if a crack opening is a source of packet-like signals, release of elastic energy may result in the motion of dislocation groups.

Under a concept that hydrogen reduces the frictional stress of dislocation, Beachem has proposed a "Hydrogen Assisted Cracking" model [7]. In this respect, it should be discussed whether small plastic deformation depicted 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

Another feature of delayed fracture was obtained by investigating the effects of minor alloying elements. The amount of soluble nitrogen was changed from 5 to 65 ppm by varying the contents of titanium, boron and aluminum in steels having main compositions of Table 1. Delayed fracture tests in 0.1 N HCl or under cathodic charging with current density of 0.2 mA/cm^2 in 3% NaCl solution were conducted. Figure 3 shows that critical stress of steels was remarkably reduced with the increase in the amount of soluble nitrogen. The amount was experimentally determined by a hot-extraction method [8] which detects nitrogen converted into NH_3. Actually, extracted nitrogen shows definite distribution with increasing temperature according to the states of nitrogen in the steel. Soluble nitrogen was defined in the present case as nitrogen except as TiN, BN and AlN. Nitrogen which could not be attributed to such definite nitrides was extracted not always below 673°K, but also in higher temperature range. Figure 3 is a result obtained at cathodic charging tests and the same 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 tensile testing of the same steels at room temperature, ductile fracture prevailed. It has been revealed that high contents of nitrogen produce intergranular fracture in impact testing of a steel when quenched from a high austenitizing temperature [9]. In the present case, however, neither nitrogen nor hydrogen solely produced intergranular fracture in tensile or impact testing. It then suggests that hydrogen 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 energy or in more general terms to make uniform chemical potential of hydrogen [11]. Hydrogen concentration C under hydrostatic stress σ_h is given as

$$C = C_o \exp\left(\sigma_h V_H / RT\right) \tag{1}$$

where C_o 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 reasonable since elastic stress should be rather relaxed. 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 then 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 σ_c satisfies

$$\sigma_c = \sqrt{E\gamma/a} \tag{2}$$

where E is the Young's modulus, γ 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 is that cluster 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 σ_c in equation (2) is much higher than the flow stress in spite of the expected decrease in E or γ. Stress concentration 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-Orowan criterion which deals with the instability of an incipient crack. For the instability criterion of a crack, the reduction of γ_s is negligible compared with plastic energy γ_p, 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 concentration 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 accompanying 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

C	Si	Mn	Cr	Mo	Ti	Al	B	N
0.20	0.75	0.70	1.30	0.50	0.06	0.06	0.002	0.004

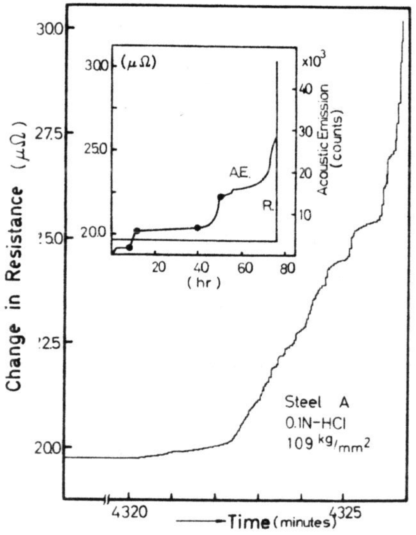
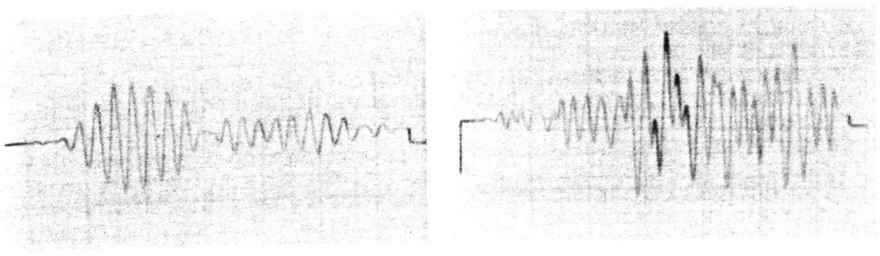


Figure 1 Monitor of Crack Growth by Electric Resistance Measurement and Acoustic Emissions at Delayed Fracture Test of the Steel in 0.1 N HCl Under Nominal Bending Stress of 1650 MPa



(a)

(b)

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

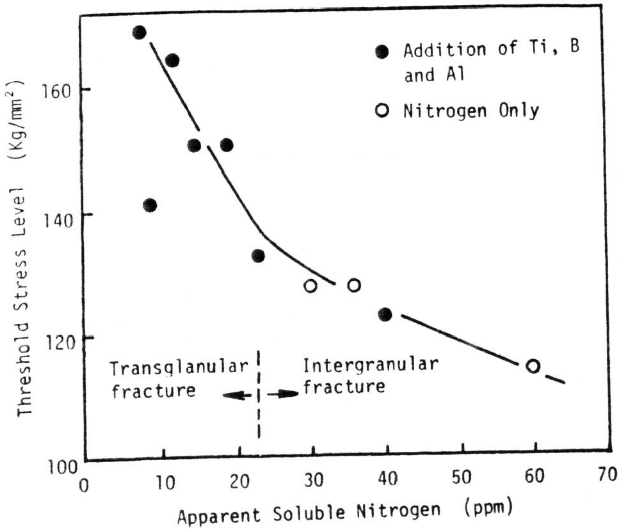


Figure 3 Effects of Soluble Nitrogen on the Critical Stress of Delayed Fracture

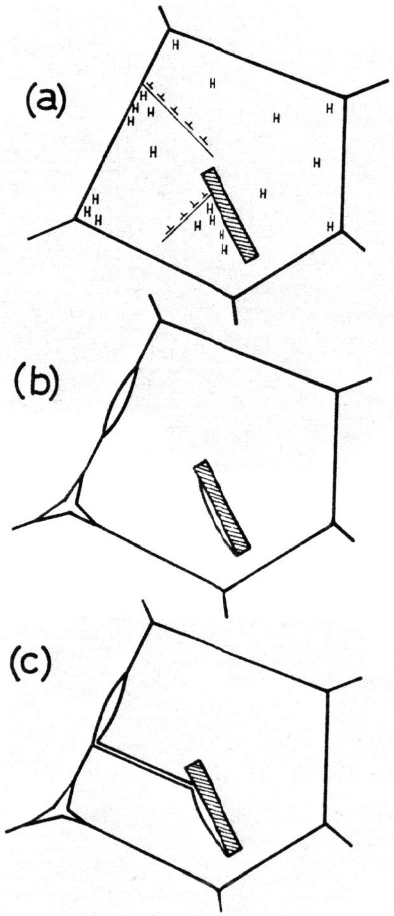
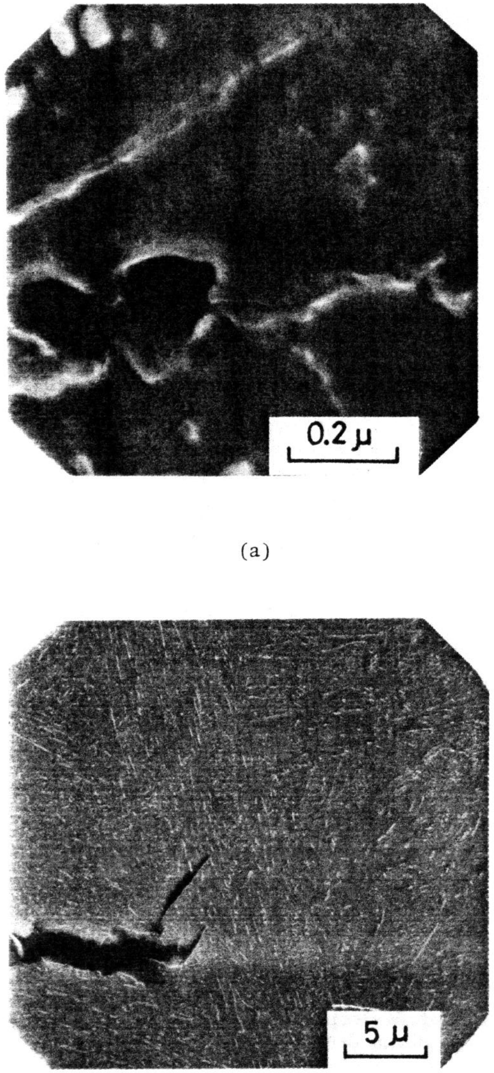


Figure 4 A Model of Hydrogen Embrittlement:

(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



(a)

(b)

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