SUBCRITICAL CRACKING BEHAVIOUR UNDER ENVIRONMENTS IN A LOW STRENGTH-HIGH TOUGHNESS STEEL

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ABSTRACT

The kinetics and mechanism of subcritical crack growth in a Ni-0.45Mo steel quenched and tempered at 600°C, have been investigated in 3.5 per cent NaCl and 5.5 per cent NaCl impregnated acetic acid solutions. The J-integral parameter is used to describe the cracking behaviour. The sulphide environment is found to be more aggressive than the chloride solution. The cracking process requires considerable energy dissipation, and the mechanism of crack growth is different in both the environments.

KEYWORDS

Environmental cracking, critical J values, energy dissipation, hydrogen embrittlement.

INTRODUCTION

High strength metals and alloys are known to be susceptible to subcritical cracking and premature failure in presence of aggressive environments (Thompson and Bernstein, 1979; Johnson and Willner, 1965; Landes and Wei, 1972; Carter, 1971; Stollwag and Kaechele, 1979). Hydrogen has been recognised to be the most damaging species in environment induced cracking (EIC) of high strength alloys (Thompson and Bernstein, 1979; Beachem, 1972; Hirth and Johnson, 1976). The kinetic behaviour of subcritical crack growth in these alloys has been successfully described using the LEFM approach (Johnson and Willner, 1965; Landes and Wei, 1972; Carter, 1971; Stollwag and Kaechele, 1979). However, subcritical crack growth behaviour study in low strength high toughness materials has received little attention.

The present communication is concerned with the studies of kinetics and mechanism of EIC in a low alloy steel tempered at 600°C. The alloy in this condition possesses relatively low strength and high fracture toughness and the K-approach is
invalid because of crack tip plasticity. An attempt has been made in the present work to apply the post-yield fracture parameters like the J-integral and COD to characterize the subcritical cracking behaviour in this alloy. The study has been confined to two hostile environments; namely, 3.5 percent aqueous sodium chloride solution and the hydrogen sulfide (H₂S) saturated acetic acid solution. These environments are frequently encountered in practice as in pipe lines for transportation of crude oil, components of marine vehicles, oil refineries, petrochemical industries.

EXPERIMENTAL PROCEDURE

Material and Heat Treatment

The chemical composition of the steel investigated is given in Table I. Sound tensile specimens of dia. 4.1 mm, gauge length 27.0 mm and SEN fracture toughness specimens of dimensions 12 mm x 25 mm x 200 mm were machined with their lengths parallel to the rolling direction. The specimens were austenitized at 850°C ± 5°C for 0.5 hr, followed by oil-quenching and liquid nitrogen treatment. The hardened specimens were tempered at 600°C for an hour and subsequently air cooled.

Table I: Chemical Composition

<table>
<thead>
<tr>
<th>C</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Si</th>
<th>Ni</th>
<th>Cr</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.36</td>
<td>0.58</td>
<td>0.018</td>
<td>0.020</td>
<td>0.24</td>
<td>1.46</td>
<td>1.10</td>
<td>0.25</td>
</tr>
</tbody>
</table>

Experiments

Tensile properties were determined by testing the specimens in an Instron m/e. Fracture toughness values were evaluated by using the precracked SEN specimens in three point bend using a Tensometer. Load (P) vs. load point displacement (LPD) and load vs. electrical potential (EP) plots (Fig. 1) were obtained and analyzed to get the Jc values corresponding to the point of crack initiation. Pre-cracked SEN specimens were subjected to cantilever loading for environmental study in a specially designed test rig. Fresh aqueous solutions of 3.5 percent NaCl (pH~5.5) and the H₂S impregnated acetic acid (pH~2.0 and H₂S concentration 1400-1500 ppm) were supplied continuously to the cracked part of the loaded specimen. The test set-up facilitated monitoring of the following during testing: (a) the onset of subcritical crack growth, (b) failure time using electrical clock system, (c) subcritical crack growth using a traveling microscope, (d) load point displacement (LPD) by LVDT, (e) crack mouth opening displacement (CMOD) with the help of a clip gauge.

J-Integral Measurement

The J-integral approach was used to describe the initiation of EAC in the steel investigated. A specimen identical to that used for environmental study was loaded in air to obtain a P - LPD plot. The J-values corresponding to various points on the plot were calculated considering the initial crack length by using Begley-Landles approach (Begley and Landles, 1972). In presence of environments the specimens were loaded to a particular J value, J_c, and the test was continued for a minimum period of 100 hrs. If the test failed to cause appreciable change in LPD or crack extension at the J_c level, the applied J value was increased slightly and the test was continued. The process was repeated till the specimen reached a critical J level where appreciable change in Δa or LPD was noticed. Once the specimen reached the critical J level, the changes in LPD and Δa as a function of time were recorded, maintaining the load constant.

RESULTS AND DISCUSSION

Tensile and Fracture Toughness Data

The tensile properties and the fracture toughness values of the alloy in 600°F tempering condition are reported in Table II. The reported (J_c) values are derived from the measured Jc value using the relation:

\[ (K_{IC})^2 = (J_{IC} \cdot b \cdot (1 - \nu^2))^{1/2} \]

Table II: Tensile and Fracture Toughness Data

<table>
<thead>
<tr>
<th>Tensile Strength</th>
<th>J_c (kJ/m²)</th>
<th>COD</th>
<th>Δa (mm)</th>
<th>N (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield</td>
<td>1117</td>
<td>11</td>
<td>52</td>
<td>45.4</td>
</tr>
<tr>
<td>Strength (GPa)</td>
<td>(σ_y)</td>
<td>(kPa)</td>
<td>(kPa)</td>
<td>(MPa)</td>
</tr>
<tr>
<td>1023</td>
<td>10.18</td>
<td>10</td>
<td>142</td>
<td>57.5</td>
</tr>
</tbody>
</table>

Subcritical Crack Growth

An appreciable crack extension was found to occur preceding unstable fracture in both the environments. (Figs. 2 and 3). The crack extension continued without exhibiting any constant crack growth rate region unlike many high strength materials (Johnson and Willner, 1965; Landes and Wei, 1973; Carter, 1974; Stellwag and Kessche, 1979; Kumar, 1982). The ratio of subcritical crack extension to original crack length i.e., Δa/ao values were found to be 0.75 and 0.62 in NaCl and H₂S environment respectively. It is also worth mentioning here that these crack extensions in NaCl and H₂S media took place over a range of applied J level equal to 77-162 kJ/m² and 10-114kJ/m² respectively. Thus, a greater reduction of the load bearing capacity of the alloy occurs in the H₂S medium than in NaCl solution is evident from the present work.

Threshold J Value, J_c

The P - LPD plots as obtained during loading and subcritical crack extension in the environments (Fig. 4) are analyzed to compute the J-integral values corresponding to various points on the plots using the instantaneous crack length. The threshold J values, J_c corresponding to the onset of subcritical cracking are reported in Table III. A range of Δa values are given as a precise determination of J_c was not possible. The J_c value under H₂S environment is found to be around 71 percent of J_c in presence of H₂S.
### TABLE III: Threshold and Terminal J Values

<table>
<thead>
<tr>
<th>Environments</th>
<th>$J_{Isc}$</th>
<th>$J_{Iscc}/J_{IC}$</th>
<th>$J_{IF}$</th>
<th>$J_{IF}/J_{IC}$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Chloride</td>
<td>17.6-20.3</td>
<td>0.42</td>
<td>162.6</td>
<td>3.6</td>
</tr>
<tr>
<td>Sulphide</td>
<td>10.4-11.5</td>
<td>0.24</td>
<td>114.0</td>
<td>2.5</td>
</tr>
</tbody>
</table>

**Energy Dissipation During SCC**

The terminal J values, $J_{IF}$, were determined from the P-LPD plots (Fig. 4) using the final crack length. The $J_{IF}$ as well as the $J_{IF}/J_{IC}$ values are reported in Table III. Large values of $J_{IF}$ compared to $J_{ISCC}$ indicate that substantial energy dissipation is needed during SCC. Also, higher $J_{IF}/J_{IC}$ ratio for NaCl suggested greater energy dissipation during subcritical cracking than that for H$_2$S medium. The energy dissipation per unit crack extension, $\Delta\gamma/\Delta\alpha (\Delta\gamma_{IF}/J_{Iscc})$ is also calculated to be 35.1 and 18.2 MJ m$^{-3}$ for NaCl and the H$_2$S mediums, respectively. The requirement of larger energy for the cracking process under NaCl solution is also evident from Fig. 4 as the load had to be increased for the continuation of crack growth unlike the H$_2$S medium. The higher load and consequently large plastic enclaves rendered the cracking process more difficult in the chloride medium. The plastic deformation was found to be much restricted under H$_2$S medium resulting in lesser energy dissipation.

**Critical COD during SCC**

The measured COD values during subcritical cracking in NaCl solution were converted to instantaneous crack tip COD values using the standard bend relationship (Veerman and Müller, 1972). The COD values are found to increase continuously with $t$ and $\Delta\alpha$ the increase being more during the final stages of crack growth (Fig. 5). The critical COD value at unstable fracture, COD$_{IF}$ is found to be 250 $\mu$m.

In addition to COD$_{IF}$, the variation in COD values at the original crack tip (i.e., COD$_{IC}$) were also computed considering the original crack length, $a_0$ (Fig. 5). The terminal value, COD$_{IF}$ is measured to be 400 $\mu$m. The large difference between the COD$_{IF}$ and COD$_{IC}$ values may be attributed to the (a) change in crack length and (b) large plastic deformation associated with subcritical cracking. On the other hand, the substantial difference between COD$_{IF}$ and COD$_{IC}$ appears only due to crack tip rounding and blunting (Kumar, 1982).

**Mechanism of Ambient vs. Environmental Fracture**

The microfractographic investigation under SEM revealed that a microvoid coalescence mode of fracture is predominant in this condition of the alloy when tested in ambient condition. The fracture surfaces obtained from testing in chloride solution, however, exhibited large number of secondary cracks, deep pits, dimples and substantial plastically deformed zones (Fig. 6). The mode of cracking is predominantly due to pitting and secondary crack formation in this environment. This may indicate that the mechanism of failure under NaCl environment is stress corrosion cracking coupled with hydrogen embrittlement. The presence of considerable amount of corrosion product (assumed to be due to anodic dissolution) on the fracture surfaces is an evidence for the contribution of SCC in inducing cracking.

The fracture surfaces formed under sulphide environment, however, were found to be characterized by the complete absence of dimples and presence of deep secondary cracks revealing an intergranular type of cracking (Fig. 7). Some scattered pits and plastically deformed regions could also be noticed. The in situ study on fracture specimens under SEM helped to reveal very fine cracks originating from the inclusions/carbides particles and their joining in the direction of stress (Kumar, 1982). This indicates that the hydrogen trapping at inclusion/carbide-matrix interfaces gives rise to initiation of cracking. The recombination of atomic hydrogen is inhibited by the formation of iron sulphides and facilitates the penetration of hydrogen into the metal (Karpenko, A.M., and I.M. Vasilevskiy, 1979). Some of the hydrogen may precipitate as hydrogen gas molecules in the microvoids around inclusions/carbides resulting in high internal pressure within the microvoids. The H$_2$S is then released when the built up internal pressure exceeds certain limiting value. From the present work the susceptibility to SCC of the alloy in sulphide environment appears to be mainly due to hydrogen embrittlement.

**Conclusions**

1. J-integral approach is useful to describe the subcritical cracking behaviour in the presence of aggressive environment in low strength-high toughness steels where large plastic deformation accompanies the crack growth. The alloy is susceptible to subcritical cracking in both the NaCl and H$_2$S media, the H$_2$S being more hostile.

2. The $J_{IF}/J_{IC}$ ratios of about 3.6 and 2.5 for the NaCl and H$_2$S environments, respectively, reveal that considerable energy dissipation occurs during the subcritical cracking process. The energy dissipation per unit crack extension for the NaCl medium is found to be twice than that for the H$_2$S medium.

3. It is believed that the hydrogen induced cracking is responsible for the crack growth under the sulphide medium whereas the stress corrosion cracking coupled with hydrogen embrittlement causes fracture under the NaCl environment.

**Acknowledgement**

The authors express their gratitude to Prof. V. Raghavan for his interest in the present study.
REFERENCES


FIG. 3 VARIATION OF Δα AND LPD WITH TIME, t

FIG. 4 P-LPD PLOT FOR Ni-Cr-Mo STEEL (600 T)

Δa = 9.07 mm
ΔT = 14.747 mm
H₂S ENVIRONMENT

J₀ = 65.4 kJ/m²
Δa = 5.65 mm

Ni-Cr-Mo STEEL (600T)

J₀ [kJ/m²] 1 2 3
NaCl ENVIRONMENT 17.6 20.6 26
H₂S ENVIRONMENT 10.4 11.6 14.0

FIG. 1 TYPICAL P-LPD AND P-EP PLOTS

FIG. 2 VARIATION OF Δα AND LPD WITH TIME, t
**FIG. 5** VARIATION OF $\text{(COD)}_i$ AND $\text{(COD)}_r$
WITH $t$ AND $\Delta a$

**FIG. 6** SEM MICROFRACTOGRAPH (Sodium Chloride environment, 500 X)

**FIG. 7** SEM MICROFRACTOGRAPH (Hydrogen sulphide environment, 500 X)