QUANTITATIVE ANALYSIS OF FATIGUE FRACTURE SURFACE IN THE DUPLEX STEEL

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ABSTRACT

We present a quantitative and qualitative analysis of the fatigue surface of Z2CND2205 duplex steel. Both the ferritic and austenitic phases of this duplex steel are investigated. The observed brittleness of the steel is here due to the presence of hydrogen. The hydrogen influence is complex in that the two phases are altered by hydrogen in different ways. Quantitative parameters describing striations spacing in each phase are measured.

Keywords: corrosion, duplex steel, fatigue, hydrogen, striation.

INTRODUCTION

The austenitic-ferritic Z2CND2205 steel is widely used in the chemical, petrochemical and power industry. The steel has a good resistance to corrosion, particularly to stress corrosion whose net effect is damaging. The advantages of both austenitic- and ferritic-steel are combined in this duplex steel. The austenitic phase ensures ductility, and resistance to the corrosive effects of electro-chemical corrosion; the ferritic phase ensures resistance to the stress corrosion. A great deal of work has shown that austenitic-ferritic duplex steels are altered by the presence of hydrogen yielding a decrease of their plastic properties (Nicodemi and Zoja, 1975; Davidson, 1990; Desestret and Charles, 1990). The hydrogen influence is complex because each of the constituting phases - ferrite and austenite- is altered its own way by hydrogen. The ferrite, due to its body-centered cubic lattice, is a priori more sensitive than the austenite exhibiting a face-centered cubic lattice. However, the sensitivity of austenite to hydrogen may actually increase in response to structural changes after tempering and α’ (martensite) and ε (carbide) formation. The brittleness of the austenitic-ferritic steels under hydrogen influence is actually four-fold:

- penetration of the hydrogen into the metal,
- the diffusion of hydrogen in the metal,
- the hydrogen trapping,
- and, the material brittleness.

Herein, one relies on quantitative fractography to evaluate the sensitivity of the Z2CND2205 steel to the degrading influence of hydrogen.

MATERIAL

The chemical composition of the Z2CND2205 steel and its mechanical properties are listed in table 1 (Iacoviello, 1997). Tensile specimens were taken in two basic directions with respect to the rolling direction (L: longitudinal; T: transversal).

Table 1. Chemical composition (in wt %) and mechanical properties of Z2CND2205 steel.

<table>
<thead>
<tr>
<th>C</th>
<th>Si</th>
<th>Mn</th>
<th>P</th>
<th>S</th>
<th>Cr</th>
<th>Ni</th>
<th>Mo</th>
<th>N</th>
</tr>
</thead>
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<td>0.019</td>
<td>0.39</td>
<td>1.51</td>
<td>0.022</td>
<td>0.002</td>
<td>22.45</td>
<td>5.50</td>
<td>3.12</td>
<td>0.169</td>
</tr>
</tbody>
</table>

Direction: Y.S. [MPa], U.T.S. [MPa], A [%], JIC [kJ/m²]

T: 565 827 35 140 (TL)
L: 513 790 39 200 (LT)

Y.S. (Yield Stress), U.T.S. (Ultimate Tensile Stress), A (Strain of 0.5%), JIC (critical J value).
The Z2CND2205, also referred to as the “Uranus 45N” steel (trade name), was quenched in water at 1050°C. Its fine-grain microstructure is characterised by streaked bands, with ferritic islands embedded in an austenitic matrix. X-ray diffraction revealed a ferritic phase content of about 55.4%. The specimens for the strength tests were tempered during three hours at 475°C in an argon atmosphere, and then cooled in water. The amount of hydrogen was determined by degassing under vacuum (10⁻³ Pa) at 600°C during 20 min. The measured amount of hydrogen was 110 ppm (Boniardi et al., 1996).

**FATIGUE TESTS**

During the fatigue tests of the Z2CND2205 steel, the da/dN cracking rate was measured as a function of ΔK, the stress intensity range, under the following conditions:

- Compact type (CT) specimens ASTM E674, type LT and TL (the first letter denotes the direction of the applied load, the second letter denotes the direction of crack growth),
- in air, temperature 20°C, frequency: 1, 10, 20 Hz,
- in a corrosive medium, temperature 20°C, 1N H₂SO₄ aqueous solution, frequency: 1, 10, 20 Hz,
- the tests were performed using a sinusoidal force with $R = K_{min}/K_{max} = 0.1$ constant.

Generally the cracking evolution rate as a function of the stress intensity range, is given by the Paris’ relation:

$$\frac{da}{dN} = C \Delta K^m$$

where: da/dN - cracking rate [mm/cycle], a – crack length, N-number of cycle,

$C$ - constant depending on the material,

$m$ - constant depending on the material and cyclic stresses,

$\Delta K$ - stress intensity range [MPa m⁻¹/²].

Fig. 1 shows examples of da/dN cracking rates as a function of $\Delta K$ (stress intensity range) measured in air, and in a corrosive environment (Iacoviello et al., 1997). In Fig. 1, the cracking rate is not given by the single Paris’ relation but consists of several segments with different coefficient $m$. For the tests in air, the cracking curve shows two segments: a first segment for $\Delta K \leq 22$ MPa m⁻¹/² and, a second for $\Delta K > 22$ MPa m⁻¹/². An increase in the cracking rate is observed in the H₂SO₄ solution. The cracking curve in the corrosive medium shows three segments: a first one for $\Delta K \leq 25$ MPa m⁻¹/², a second one for 25 MPa m⁻¹/² $\leq \Delta K \leq 55$ MPa m⁻¹/² and, a third one for $\Delta K > 55$ MPa m⁻¹/².

![Diagram](image-url)

Fig. 1. Cracking rate da/dN as a function of the $\Delta K$ stress intensity range in air and in H₂SO₄ solution (region I: non-continuum mechanisms, region II: continuum mechanisms (striation growth), region III: final failure).
FRACTOGRAPHIC ANALYSIS

Fractures in air

In the case of cracking in air, the fractographic examination shows the formation of ductile striations both in the ferrite (α) and austenite (γ) phases (Fig. 2).

Fig. 2. Fatigue striations after the tests in air.

Fractures in a corrosive medium

In the case of cracking in a corrosive environment (H₂SO₄), the formation of striations happens both in the ferrite and austenite (Fig. 3) and a higher cracking rate is obtained. The detailed examination of the fatigue fracture surfaces reveals that the ferritic phase brittleness is due to hydrogen (Shiquiong et al., 1989; Dickson et al., 1990). In the first range of the log(da/dN) = f(log ΔK) cracking curve, fatigue striations are brittle in the ferrite (Fig. 3, point 1). The river patterns are parallel to the macroscopic cracking direction (Fig. 3, point 2). Numerous secondary cracks are present at the austenite-ferrite boundary and they are not perpendicular to the cracking direction (Fig. 3 and Fig. 4, point 3). In the second range of the cracking curve, the fracture is brittle in the ferrite as revealed by the presence of the “leaf structure” (Fig. 4, point 4) and characteristic “triangles” (Fig. 5, point 5). The river patterns are not parallel to the cracking direction. Based on the corrosion figures, the cracking plane in the ferrite is of type [100] (Dickson et al., 1990). For higher cracking rates the fatigue striations are of the ductile type both in the ferrite and austenite and they are perpendicular to the cracking direction.

Fig. 3. Fatigue striations after the tests in H₂SO₄.

Fig. 4. “Leaf structure” after the tests in H₂SO₄.

Fig. 5. “Triangles” after the tests in H₂SO₄.
STRIATION SPACING

A correlation exists between the progress of fatigue cracking and fatigue striation spacing. The measurement of the striation spacing is accordingly important and can be a basic parameter used in the further studies. The above assumption is valid in cases when the macroscopic speed of cracking is controlled by propagation mode (Kocańska, 1985). We can find the projected striation spacing by counting striations number per unit of projected length (Chermant and Coster, 1979):

\[ N_L = N_S / L' \]  

(2)

\(N_S\) – number of striations,

\(L'\) – projected length.

Mean projected distance between 2 striations is then:

\[ \bar{d}' = 1 / N'_L. \]  

(3)

The measurement can be quickly made on a set of parallels (Underwood and Stark, 1979). Yet, because the local orientation of the striations is neglected, the resulting striation spacing would be overestimated (Coster and Chermant, 1983). If the locally parallel striations are oriented randomly with respect to the macroscopic cracking direction (Coster and Chermant, 1983) then, the correct projected spacing is expressed by:

\[ \bar{d}' \text{corrected} = 2 / \pi \bar{d}'. \]  

(4)

Finally \(N'_L\) (Eq. 2) being measured on a projected plane, it must be corrected according to Eq. 5:

\[ N_L = N'_L / R_L \]  

(5)

where \(R_L\) is the linear roughness index of the profile line.

Here, as in Nieh and Nix (1980), it is assumed that the fracture surface is perpendicular to the beam of electrons so that no roughness correction of type Eq. 5 is needed. The striation spacing can be obtained from the screen of the scanning microscope. A sample was carefully positioned to ensure that the beam of electrons was locally perpendicular to the fracture surface carrying the striations. Differences of grey level obtained in microscopic image were analysed by the VISILOG software. A striation spacing was measured for each group of locally parallel striations. An example of measurement is showed in Fig. 6.

Fig. 6. Measurement of \(\bar{d}'\text{corrected}\) striation spacing on histogram.

Fig. 7. Striation spacing \(\bar{d}'\text{corrected}\) \(\text{(\(\bar{d}'\text{corrected}(A)\) in austenite, \(\bar{d}'\text{corrected}(F)\) in ferrite) and \(\text{da/dN}\) cracking rate as function of the \(\Delta K\) stress intensity range in H₂SO₄ solution.}

Our results show that the striation spacing depends on the size of cracking \(a\), as well as on stress intensity factor value \(\Delta K\) (Fig. 7). Such a results allow to describe the influence of hydrogen on the cracking process in the duplex steel by fractography methods. The striation spacing in the ferrite and in the austenite are described separately. In air, the striation spacing in the ferrite is equal to the striation spacing in the austenite. In the presence of hydrogen, the striation spacing in the ferrite is smaller than in the austenite.
This result can be explained by the different speeds of transport of hydrogen in the ferrite and in the austenite. The crystallography of the ferrite fracture in the H₂SO₄ solution is the first elementary argument favouring the hydrogen embrittlement theory (Kikuta et al., 1975). The presence of twins, enhancing initially local plasticity and local plastic deformation, which are formed on the cracking front and allow a local change of the cracking direction, shows in accordance with the Bastien’s theory, that hydrogen is the reason for cracking. The difference of appearance of the fracture at the middle and the edges of the specimen in the ferrite, is the second argument: at the middle of the specimen, a brittle fracture zone occurs but at the edges there are ductile fatigue striations. This result is consistent with the tendency of the atomic hydrogen to diffuse towards areas with strong hydrostatic stress, characterising the state of the plane deformation, which is achieved in the centre of the specimen. The width of this zone on the corrosive medium increases if ∆K increases. It is consistent quantitatively with an increase in the width of the edge zone subjected to the plane stress state which corresponds to an increase in ∆K.

CONCLUSION

Our analysis of fatigue surfaces shows that the fatigue fracture of the austenite phase forms in a ductile way in air and in the H₂SO₄ solution. The austenite presence causes a decrease in the macroscopic cracking rate of the duplex steel. The hydrogen embrittlement theory is the basic theory that explains the effect of cracking rate increase. The results of the striation spacing study together with fractographic analyses give valuable information about hydrogen interaction in ferrite and in austenite in duplex steels.

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