Hydrogen-induced cracking by nanovoids in 310 stainless steel

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Abstract Hydrogen-induced cracking was investigated by TEM in-situ tension in hydrogenated stainless steel of type 310. It was found experimentally that hydrogen-induced cracking happens via nanovoid nucleation followed by quasi-cleavage along \{111\} planes when \( C_H \) is higher. Otherwise, in the case of lower \( C_H \), hydrogen enhances ductile fracture via hydrogen-enhanced microvoid nucleation, growth and connection. A new model was proposed based on the present experiments. Dislocations break away from defect atmospheres and move away from the DFZ, leaving vacancy and hydrogen clusters along \{111\} planes. Hydrogen tends to combine with vacancy clusters and initiate nanovoids along \{111\} planes. Dense nanovoids connect each other, resulting in brittle cracking. Scattered nanovoids grow into microvoids or even macrovoids, leading to ductile fracture.

Keywords: stainless steel, TEM, hydrogen-induced cracking, nanovoid, dislocation.

Many experiments\(^{[1-5]}\) have shown that hydrogen-induced cracking (HIC) in intermediate and low strength steels involved the mechanism of hydrogen-enhanced voids, which, however, is not clear yet. There are two different viewpoints about hydrogen’s promoting voids. Thompson et al.\(^{[6-8]}\) suggested that hydrogen promoted void growth and interlinkage but it had hardly any effect on void nucleation. On the other hand, Kwon and Asaro\(^{[9]}\) concluded that hydrogen enhanced void nucleation at average-sized carbide particles by reducing the critical interfacial strength. However, some experiments indicate that second phases may not be essential for initiation of voids\(^{[10]}\). Lee and Bernstein\(^{[3]}\) found that hydrogen enhanced void nucleation along the characteristic slip bands. The present investigation is aimed at finding out how hydrogen promotes voids that finally induce HIC in 310 stainless steel.

HIC could occur in hydrogenated stable austenitic stainless steel\(^{[5,12,13]}\), which had no relation with martensitic transformation\(^{[11,12]}\). Macro experiments on stable stainless steels showed that hydrogen enhanced ductile failure by promoting voids when hydrogen concentration \( C_H \) was low and the applied stress strength factor \( K_{sp} \) was high, resulting in dimple fracture\(^{[11]}\). Otherwise hydrogen induced brittle failure when \( C_H \) was high and \( K_{sp} \) was low, resulting in quasi-cleavage fracture\(^{[11]}\). The microvoids (or microcracks) that were observed in the above-mentioned macro-investigations and even some TEM observations\(^{[5]}\) were actually developed far away from the early initiation stage and might not reflect their initiation mechanism exactly. One objective of the present investigation is to observe, \textit{in-situ} by TEM, the effect of hydrogen on nanovoid

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nucleation during tensile tests of thin foils of hydrogen-precharged 310 stainless steel, as well as the different mechanisms of HIC when hydrogen concentrations are different.

Obviously, the mechanism of intrinsic failure of 310 stainless steel is fundamental for the present study. Chen et al.\textsuperscript{[14]} found by \textit{in-situ} TEM that dislocation free zones (DFZs) played an important role in failure. DFZs were beneficial to nanocrack nucleation. The local highly concentrated stresses induced supersaturated vacancies in the DFZ of which the diffusion and enrichment initiated nanovoids in the DFZs. It is certain that this high stress concentration will also enrich hydrogen in the DFZ of a precharged specimen. The other objective of this paper is to propose a model of void initiation by cooperation of hydrogen and vacancies in highly concentrated stress fields.

1 Experimental procedures

Stable austenitic stainless steel of type 310 was used on account that not only could HIC happen without involving \( \varepsilon \)-martensite in it\textsuperscript{[11,12]}, but also the diffusion coefficient of hydrogen \( D_H \) is quite small (about \( 10^{-12} \) cm\(^2\)/s\textsuperscript{[15]}). Therefore, hydrogen can stay in this kind of thin foil for a longer time than, for example, ferrite, copper, and aluminum. The testing time must be short enough so that there is sufficient hydrogen to initiate cracking in thin foils during \textit{in-situ} elongation. The variation of hydrogen concentration with testing time is estimated as follows. Suppose the thickness of the thin foil is \( l \) and the original \( C_H = C_0 \). The zero point of the coordinate is fixed at the half depth of specimens. \( x \)-axis is parallel to the normal of foils, and \( y \)-axis to the foil surface. The solution of Fick’s second law that satisfies the initial condition

\[
C_H(x, 0) = \begin{cases} 
C_0 & \left( |x| \leq \frac{L}{2} \right), \\
0 & \left( |x| > \frac{L}{2} \right),
\end{cases}
\]

is

\[
C(x, t) = \frac{C_0}{2} \left[ \text{erf} \left( \frac{x + \frac{L}{2}}{2 \sqrt{Dt}} \right) - \text{erf} \left( \frac{x - \frac{L}{2}}{2 \sqrt{Dt}} \right) \right].
\]

Figure 1 shows \( C_H(x, t)/C_0 \) after 5 and 60 min, where \( l \) equals 80, 100, 150, 200, 250 and 300 nm. Suppose \( C_0 = 200 \) ppm. It can be seen that \( C_H \) is about 25 ppm in the specimen with thickness of 200—250 nm after 1 h. Experimentally, the hydrogen concentration, which was measured by degassing at 1 000°C, was more than 200 ppm after cathodically charging for 50 h. It reduced to around 50 ppm after observation in TEM for about 60 min. The difference between experimental measure and theoretic estimation may be mainly due to many hydrogen traps in the specimen.

The composition of the present 310 stainless steel, the heat treatments and the polishing solutions are the same as the earlier work\textsuperscript{[14]}. TEM foils were prepared from a strip of 0.2 mm. First, the sheets were thinned to about 50 \( \mu \)m by chemical polishing. Then, the specimens were cathodically charged at room temperature for at least 50 h in a 0.5 mol/L \( \text{H}_2\text{SO}_4 \) solution containing 250 mg \( \text{As}_2\text{O}_3 \) per liter. A platinum counter electrode and a current density of 0.5 A/cm\(^2\) were used. Finally, the precharged specimens were electropolished. \textit{In-situ} elongation was performed in JEM 100-CX.