Conf-730605--3

HYDROGEN BRITTLENESS IN HYDROGEN
AND HYDROGEN-OXYGEN GAS MIXTURES

H. H. Johnson

Department of Materials Science and Engineering
Cornell University
Ithaca, New York

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(Paper to be presented at the International Conference on Stress-Corrosion Cracking and Hydrogen Embrittlement of Iron-Base Alloys, Unieux, Firminy, France, June, 1973, and to appear in the Proceedings)

Report #1993

Issued by

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ABSTRACT

Crack initiation and propagation characteristics are presented for high strength steels in hydrogen and hydrogen-oxygen mixtures under both static and cyclic stress. Under static stress and in hydrogen the threshold stress intensity for crack initiation was nearly independent of temperature in the range around 300°K; however, the crack growth rate was a maximum near 300°K and decreased at both higher and lower temperatures. Oxygen in the amount of 0.4% by volume eliminated hydrogen-induced slow crack growth; at lower concentrations the threshold stress intensity was approximately linear with oxygen concentration.

Under cyclic stress hydrogen-accelerated fatigue crack growth was observed for 4340 steel with yield strengths from 150 to 215 KSI. Oxygen additions eliminated the hydrogen-accelerated growth at the lower strength levels, but was only partially inhibitory at a yield strength of 215 KSI.

Possible hydrogen concentration profiles by diffusion ahead of the crack tip were calculated for two models. In the first the crack tip is modelled as a moving line source for hydrogen, and in the second as a moving semi-infinite slit source. In both models the penetration distance depends upon the ratio of the diffusivity to the crack growth velocity.

INTRODUCTION

Although hydrogen embrittlement has been known for decades, it has only recently been recognized^(1,2) that some structural alloys, particularly high strength steels, are susceptible to sub-critical crack growth and delayed failure in low pressure hydrogen gas. Perhaps the generally satisfactory performance of portable steel cylinders for pressurized hydrogen had focussed attention upon hydrogen brittleness resulting from electrochemical charging, pickling, or prior exposure to hydrogen at high temperature and perhaps pressure. These cases may be categorized as internal hydrogen brittleness, where the hydrogen is distributed throughout the microstructure prior to application of stress. The hydrogen concentration was usually supersaturated with respect to hydrogen gas at low pressure, say one atmosphere.

The sub-critical crack growth observed with low pressure hydrogen gas is best categorized as external hydrogen brittleness, for the crack is propagating in a bulk environment of molecular hydrogen gas. Crack extension occurs at a very low stress intensity, in some instances at a stress corresponding to approximately ten per cent of the yield strength.

It is natural to inquire if internal and external hydrogen brittleness display the same phenomenological characteristics, which would suggest identical mechanisms. Williams and Nelson⁽²⁾ examined the influence of hydrogen gas pressure, temperature, and displacement rate upon the sub-critical crack propagation characteristics observed in a high strength 4130 steel. Although some

uncertainty over details may remain, in broad terms the phenomenological characteristics of hydrogen gas brittleness are similar⁽²⁾ to those determined earlier^(3,4) for internal hydrogen brittleness. In pre-cracked specimens, and with a fixed hydrogen pressure, the brittleness⁽²⁾ decreased with increasing displacement rate, was a maximum in rate near 300°K and decreased at both higher and lower temperatures; similar behavior patterns are observed with internal hydrogen brittleness^(3,4). In a later paper⁽⁵⁾, partial dissociation of low pressure hydrogen gas, with a consequent increase in atomic hydrogen concentration in the gas phase, was shown to markedly increase the growth rate of hydrogen-induced cracks.

The detailed interpretation of the pressure-temperature dependence of the crack growth kinetics is controversial⁽⁶⁻⁸⁾. Williams and Nelson⁽²⁾ suggested a surface-reaction model, with the Porter and Tompkins⁽⁹⁾ adsorption equation modified by the effective area concept introduced by Smithells and Ransley⁽¹⁰⁾. The resulting equation provided a good empirical fit to the data, but a sound theoretical basis for the model is not yet apparent⁽⁶⁻⁸⁾. In addition, the effective area concept suggests a temperature regime in which the permeation rate should decrease with increasing temperature⁽⁸⁾, and this has not yet been observed.

External hydrogen gas brittleness is markedly influenced by gas purity; in particular, small amounts of oxygen completely eliminate hydrogen-induced crack growth⁽¹⁾. This is best explained in terms of preferential adsorption of oxygen on the crack tip surface, with the consequence that hydrogen is excluded

from the surface. In view of this experimental result, it is difficult to accept the implication⁽¹¹⁾ that brittleness is related to the strength of interaction between a metal and an adsorbed species, for oxygen is surely more tightly bound to the surface than hydrogen, yet oxygen does not cause crack growth.

This difficulty is avoided if hydrogen penetration some distance into the bulk near the crack tip is an essential feature of the brittleness process. Hydrogen is an extraordinarily mobile solute, but there is no experimental evidence as to whether or not significant hydrogen penetration occurs in advance of the crack. Oxygen is of course relatively immobile, and no penetration would be expected.

In this paper further information on hydrogen-induced crack growth under both static and dynamic loads is presented, and models for hydrogen diffusion ahead of propagating cracks are discussed.

STATIC LOAD

H-11 steel heat treated to a yield strength of 230,000 psi was used⁽¹²⁾. Fracture mechanics specimens of the compact tension type were tested in the plane strain regime, with crack length monitored by calibrated displacement gage measurements, in an MTS machine with an appropriate environmental chamber. The threshold stress intensity for crack propagation under rising stress, designated K_{It} , and the crack growth rate were measured as a function of temperature, pressure, and hydrogen purity⁽¹²⁾. These two parameters display different aspects, static and dynamic, of

the hydrogen induced brittleness.

Threshold Stress Intensity

The temperature dependence of the threshold stress intensity in 813 torr hydrogen, Fig. 1, is virtually nil. K_{It} was determined by cycling in stress intensity back and forth through the threshold. The stress intensity to start the crack was always slightly higher than that at which it came to rest, i.e., there was some hysteresis. This may reflect crack blunting upon cessation of crack growth; the amount of hysteresis showed scatter, but appeared to increase with temperature, and this plastic blunting may explain the slight rise in K_{It} with temperature. The insensitivity of K_{It} to temperature is in stark contrast to the strong influence of temperature upon crack growth rate⁽²⁾. Crack growth rates are evidently a poor practical measure of susceptibility to failure in hydrogen gas.

The pressure dependence of the threshold stress intensity is shown in Fig. 2 for different temperatures; it is evident that the threshold stress intensity decreases as the pressure increases, as would be expected. Specifically, for both sets of data $K_{It} \sim p^{-0.4}$, which is not far from what would be expected if K_{It} were inversely proportional to the lattice concentration and the lattice concentration obeyed Sievert's law. Although this is speculative, it suggests that lattice solution may be an essential precursor to crack growth.

It is also possible that increasing pressure influences the state of the adsorbed hydrogen on the surface; however, surface studies indicate that monolayer adsorption of hydrogen on iron is

complete at temperatures and pressures well below those used in brittleness studies. It is somewhat difficult to envision crack extension as caused by a change from single layer hydrogen coverage to perhaps multilayer coverage, although the possibility cannot be ruled out on the basis of present knowledge.

Dynamic Aspects of Hydrogen Gas Brittleness

Crack growth rates were measured as a function of stress intensity, temperature, and gas pressure. With respect to stress intensity, the most interesting features are the rapid increase in crack growth rate with stress intensity at low stress intensities, and the saturation in crack growth rate at a higher stress intensity. Presumably the saturation in growth rate occurs when the crack growth rate just threatens to outpace the rate at which hydrogen is delivered to the site at which an increment of crack extension initiates.

The influence of temperature upon the relation between crack growth rate and stress intensity curves is summarized in Fig. 3. Saturation in crack growth rate is not observed at the lowest and highest temperatures. Fracture intervenes in the low temperature regime, apparently because of the significant dependence of K_{IC} on temperature for this steel, Fig. 1. At the highest temperature diffusion is so rapid that fracture again occurs before the saturation state is reached.

The temperature dependence of the measured crack growth rates are summarized in Fig. 4. The behavior pattern is strikingly similar to that presented earlier by Williams and Nelson⁽²⁾ for a

quenched and tempered 4130 steel. Although the high and low temperature slopes are slightly different from theirs, the differences could easily arise from the use of different materials or testing techniques. The present data are regarded as confirming the earlier results of Williams and Nelson⁽²⁾; the essential feature is that the crack growth rate decreases at temperatures both above and below approximately 300°K. It should be recalled, however, that the threshold stress intensity is virtually insensitive to temperature through the same range.

The present data do suggest the possibility that crack growth rates in the saturation regime may have a different temperature dependence than crack growth rates measured in the rising part of the crack growth rate versus stress intensity curves. However, the implication rests on only one or two data points, and it was not possible with this steel to extend the temperature range of the saturation crack growth rates.

The pressure dependence of the crack growth rate is recorded in Fig. 5. The results are consistent with the more extensive pressure data of Williams and Nelson⁽²⁾ in that a $3/2$ power dependence upon the pressure is observed at the higher temperature, and the exponent decreases as the temperature drops.

Oxygen

The influence of oxygen upon hydrogen-induced crack growth is summarized in Fig. 6. At room temperature sub-critical crack growth at any stress intensity level is eliminated by an oxygen concentration in excess of 0.4% by volume. At lower oxygen levels

the threshold stress intensity is proportional to the oxygen concentration and apparently not related to the hydrogen partial pressure over the range investigated. This latter result is consistent with a simple mechanical blocking of hydrogen from the surface by oxygen which is preferentially adsorbed.

CYCLIC LOAD

The influence of hydrogen-oxygen environments upon fatigue crack growth rates was studied⁽¹³⁾ for 4340 steel heat treated by quenching and tempering to three yield strength levels, 150, 180, and 215 KSI. Fracture mechanics specimens of the compact tension type were used, with crack lengths determined from calibrated displacement measurements. The fatigue crack growth rates as a function of applied stress intensity are summarized in Figs. 7-9⁽¹³⁾.

At all three strength levels fatigue crack growth rates are higher in a pure hydrogen environment, but the increase is greatest at the high yield strength, 215 KSI. The expected frequency dependence of the fatigue crack growth rate is also greatest at this highest yield strength, Fig. 10, and somewhat surprisingly is virtually absent at the lowest yield strength, 150 KSI. Nevertheless, the hydrogen fatigue crack growth rates at 150 KSI are almost an order of magnitude larger than those measured in argon. It is unlikely that an effect of hydrogen upon static load crack growth behavior would be evident at a yield strength of 150 KSI, while of course there would be a very pronounced effect at 215 KSI. The influence of the hydrogen can be conveniently

summarized by n , the slope of the $\ln \Delta K - \ln a$ plots.

The influence of oxygen in the amount of 0.4% by volume upon the fatigue crack growth rates was also investigated. With the two lower yield strengths this concentration of oxygen was completely effective in eliminating the influence of hydrogen; the fatigue crack growth rates in hydrogen-oxygen mixtures were identical to those measured in argon. There seems little doubt but that this beneficial effect of oxygen has been very important in applications. At the highest strength level more scatter was encountered, but it seems clear that the oxygen in this concentration is only partially protective. Evidently the oxygen effect is strength level sensitive, presumably because of strength level induced changes in the crack tip geometry, and the influence of crack tip geometry upon film rupture and reformation characteristics under cyclic strain.

HYDROGEN PENETRATION AHEAD OF CRACK TIP

Hydrogen is a solute of extraordinary mobility, and it is correspondingly uncertain as to whether or not brittleness is caused by hydrogen at the crack tip surface, or that perhaps hydrogen penetration ahead of the crack tip is an essential precursor. This in turn bears upon the question of whether crack propagation occurs directly at the crack tip or by advance nucleation ahead of the crack tip.

Dimensional considerations suggest that the penetration distance ahead of the crack tip will depend upon the parameter

D/V , where D is the hydrogen diffusivity and V the crack velocity, and that the penetration distance will increase as the parameter increases. The functional form of the relation between the penetration distance and the parameter D/V will depend upon the specific crack geometry and diffusion field boundary conditions assumed.

Numerical estimates of the penetration distance may be obtained from models based upon the following assumptions:

i) Quasi-steady state crack propagation: This requires a constant crack velocity V , which is experimentally realizable through fracture mechanics techniques. This enables the use of diffusion models in a reference frame moving at the same constant velocity.

ii) Local equilibrium at the crack tip: This implies an equilibrium at the crack tip between the gaseous hydrogen and the dissolved hydrogen. The alternative is interface control, which would decrease the penetration distance. The freshly produced and straining metal surface at the crack tip is expected to be clean, and so interface control seems improbable. The local equilibrium assumption leads to a constant concentration boundary condition, if the solubility is taken to be insensitive to the elastic stress state or plasticity.

It is not possible to model the crack-diffusion system exactly, but two limiting cases may be examined, which should span the range of possibilities. In the first it is assumed that hydrogen enters only at the moving crack tip, i.e., that the crack tip acts as a moving line source. This is a reasonable assumption for

crack growth in gaseous hydrogen, since pre-exposure to hydrogen does not accelerate the cracking process⁽¹⁾. The most probable interpretation is that hydrogen enters only through the freshly strained metal at the crack tip, and that the crack walls are passive, possibly because of oxygen contamination⁽¹⁾.

Upon the assumption of local equilibrium at the crack tip between the gaseous and dissolved hydrogen, the diffusion field follows directly from the analyses of 1) heat flow from a moving line source⁽¹⁴⁾, or 2) the point defect field surrounding a climbing dislocation⁽¹⁵⁾. The concentration may be expressed in polar coordinates centered upon the line source as

$$C(r, \theta) = \beta K_0 \left(\frac{Vr}{2D} \right) \exp \left(- \frac{Vr \cos \theta}{2D} \right) \quad (1)$$

V = crack velocity

D = diffusivity

K_0 = modified Bessel function of second kind of order zero.

Since the concentration is singular at $r=0$, the constant β may be determined from the boundary condition applied at the singularity. The radius of curvature at the crack tip may be set at r_0 , and the corresponding solute concentration at C_0 , where upon

$$\frac{C}{C_0} = \frac{K_0 \left(\frac{Vr}{2D} \right) \exp \left(- \frac{Vr \cos \theta}{2D} \right)}{K_0 \left(\frac{Vr_0}{2D} \right) \exp \left(- \frac{Vr_0 \cos \theta}{2D} \right)}, \quad (2)$$

which allows an assessment of the influence of crack tip radius upon the moving diffusion field.

The other extreme is to consider that hydrogen enters from every portion of the crack surface, and that the moving crack is both very thin and semi-infinite in length. This would be physically reasonable in those situations, probably primarily in aqueous media, where the crack walls are not passivated during propagation. The solution for this geometry may be taken from a recent analysis⁽¹⁶⁾ of dendrite and Widmanstätten plate growth as

$$\frac{C}{C_0} = \operatorname{erfc} \left\{ \left(\frac{V}{2D} \right)^{\frac{1}{2}} (x+r)^{\frac{1}{2}} \right\} , \quad (3)$$

x = distance ahead of moving crack, and

$$r = (x^2 + y^2)^{\frac{1}{2}}.$$

The boundary condition is that the concentration equal C_0 at all points along the crack and at the tip. The parameter V/D appears in both solutions.

Intermediate cases might also be examined, where the crack is modeled as a slit of finite length moving through an infinite medium. The necessary analysis for cracks of varying length may be obtained from the heat flow literature⁽¹⁷⁾. However, this is not worthwhile at present, since there are no experimental data against which the calculations could be compared.

Calculated concentration profiles directly ahead of the moving crack, and in the plane of the crack, are summarized in Figures 11 and 12. For present purposes it is not necessary to consider the full diffusion field. The diffusivities and crack growth velocities used in the calculations were selected to span the range of reasonable values. The 300°K hydrogen diffusivity

is a poorly characterized parameter, with values in the literature ranging from 10^{-7} cm²/s to in excess of 10^{-5} cm²/s. This range of values reflects structural trapping, and for that reason the lower D values are generally associated with the higher strength and microstructurally more complex steels. Nevertheless, the material ahead of a moving crack is plastically straining as the hydrogen moves through, and there just are no measurements of hydrogen diffusivity through a plastically straining material. The effective diffusivity may well be higher under these conditions. A crack velocity of $V = 10^{-3} - 10^{-2}$ cm/s is well within the range of measured values. (2,12)

It is evident from Figures 11 and 12 that a significant penetration of hydrogen ahead of the propagating crack can be expected. For a crack tip radius of 5×10^{-8} cm, the moving line source model, equation 2, predicts a penetration distance for a reduced concentration of 0.5 which varies from 5×10^{-7} cm for $D/V = 10^{-6}$ to 5×10^{-5} cm for $D/V = 10^{-2}$. These results are rather insensitive to the value of r_0 selected; additional calculations show that the penetration distance in the moving line source model varies little with r_0 over the range from 5×10^{-8} to 10^{-4} cm.

When the line and semi-infinite source models are compared, the penetration distance is of course greater for the semi-infinite source model, Fig. 12. The difference is most evident for larger D/V values. As D/V decreases, the calculated concentration profiles for the two models come closer together, as would be expected. At lower values of reduced concentration, C/C_0 less than 0.2 say, the penetration distances are nearly model independent.

Evidently the hydrogen in this region comes from the crack tip vicinity, and the concentration is then reasonably independent of whether or not the walls of the crack are permeable to hydrogen.

DISCUSSION

A satisfactory understanding of the brittleness induced in high strength steels by gaseous hydrogen is not available. This is perhaps not surprising, since high strength steels are of complex microstructure, and the brittleness mechanism may be equally complex. Nevertheless, the phenomenological investigations reported to date do permit some general statements.

The decrease in crack growth rate at temperatures both above and below approximately 300°K is suggestive of two rate limiting phenomena, one thermodynamic and the other kinetic. Presumably the process so controlled is one of hydrogen migration from initial sites to final sites; at the latter the brittleness event occurs. In accord with other metallurgical phenomena where a "C" curve behavior is observed, it can be inferred that the lower temperature decrease is kinetically controlled, while the higher temperature limit is caused by a reduction in thermodynamic driving force.

It is probable that the initial sites are on the surface, but the probable location of the final sites is controversial. Williams and Nelson⁽²⁾ suggested a surface location, and introduced an effective area term for initial sites, based upon the Langmuir adsorption isotherms, to account for the high temperature decrease in crack growth rate. On this basis it was possible to account

in an approximate way for the pressure and temperature dependence of the crack growth rates. However, it is difficult to construct a rational theoretical basis for the introduction of the effective area term^(6,8), since it implies also that surface control of permeation should be observed. This assumes that the crack tip surface is not different from the surface characteristics encountered in the relatively flat geometries used in conventional permeation experiments. In particular, in the high temperature regime of decreasing crack growth rate the steady state flux in a permeation experiment should also decrease with increasing temperature. There is no evidence for this in the permeation literature, although a truly definitive investigation has not been carried out in this temperature range.

There is a possibility that the final sites are distinctly sub-surface, in which case they would be more aptly described as microstructural traps. Hydrogen penetration ahead of the crack is consistent with the calculations described in the previous section. This in a quasi-steady state situation would eliminate the surface control difficulty just mentioned, since the steady state permeation flux should be independent of the bulk trapping of hydrogen. In this respect, the fractographic results^(2,12) suggest prior austenite grain boundaries as obvious candidates for final sites or traps. It may then be tentatively suggested that the rate effects are associated with the migration of hydrogen from initial sites on the surface to the prior austenite grain boundaries. Further crack growth may then depend upon the migration characteristics of hydrogen along prior austenite boundaries.

There are difficulties with this view, which may or may not be resolved by further experiment. First, it is not clear that the measured activation energies associated with crack growth are compatible with the energies associated with the process sketched above. Second, and more important, it is not clear at present that the measured pressure dependence of the crack growth rate is consistent with the postulated model.

With respect to the brittleness mechanism at the final sites, presumably austenite grain boundaries in most instances, it can only be speculated that hydrogen decreases the cohesion of the material at these locations. There is perhaps an interesting analogy with the hydrogen-promoted evaporation of metal atoms from filament tips in the very high electric fields employed in field ion microscopy. In the absence of hydrogen the applied electric field, sufficient to pull atoms off the tip surface, generates stresses in the filament of the order of magnitude of estimated theoretical fracture stresses. The material sustains these stresses without deformation because of the relative absence of defects due to the small filament size. With very small hydrogen partial pressures in the system, the field necessary to evaporate metal atoms is dramatically reduced for many metals, which corresponds to a reduction in the theoretical fracture strength. It is speculated that this results from an interaction between the hydrogen and the slightly exposed ion cores at the filament tip, which weakens the binding between the surface atoms and the bulk.

This analogy, if indeed it is apt, suggests that hydrogen

brittleness may result whenever material under very high stress is exposed to hydrogen gas. In high strength steels very high local stresses may be expected because of the high strength level and the complex microstructure. In lower strength materials plastic deformation typically intervenes to prevent attainment of very high local stresses. This line of speculation does suggest, however, that whiskers of metals normally soft in bulk form might well be brittle in the presence of hydrogen gas, since the absence of defects in whiskers allows very high stresses to be sustained.

ACKNOWLEDGEMENT

It is a pleasure to acknowledge the financial support of the U. S. Atomic Energy Commission and the provision of facilities by the Materials Science Center, which was supported by ARPA and is now supported by NSF.

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Figure 1. Threshold (K_{It}) and fracture (K_c) stress intensity versus test temperature. H-11 steel.

Figure 2. Threshold stress intensity (K_{It}) versus partial pressure of hydrogen. H-11 steel.

Figure 3. Threshold stress intensity (K_{I_t}) versus applied stress intensity (K_I).
H-11 steel.

Figure 4. Crack growth rate as a function of temperature.
H-11 steel.

Figure 5. Crack growth rate as a function of hydrogen partial pressure. H-11 steel.

Figure 6. Threshold stress intensity (K_{It}) as a function of the partial pressure of hydrogen. H-11 steel.

Figure 7: da/dN vs ΔK for the 150,000 psi Yield Strength 4340 Steel.

Figure 8: da/dN vs ΔK for the 180,000 psi Yield Strength 4340 Steel.

Figure 9: da/dN vs ΔK for the 215,000 psi Yield Strength 4340 Steel.

Figure 10: da/dN vs $\sigma_{y.s.}$ for 4340 Steel, for $K=20 \text{ ksi} \sqrt{\text{in}}$; Pure Hydrogen Environment.

Figure 11. Concentration profile ahead of moving crack; moving line source model, crack tip radius $r_0 = 5 \times 10^{-8}$ cm.

Figure 12. Concentration profiles for crack modelled as moving line source and moving semi-infinite source.