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TEMPERATURE EFFECTS ON HYDROGEN-INDUCED CRACKING IN AN IRON-BASED SUPERALLOY

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Abstract

The effects of temperature on hydrogen-induced crack growth susceptibility were studied in the iron-based superalloy IN903 using hydrogen charged samples. The measured crack growth rates increased by more than two orders of magnitude as temperature increased from 253 to 298 K. Crack growth rates then decreased at higher temperature. Fracture in all samples initiated by fracture of matrix carbides followed by microvoid formation at slip band intersections and failure of interconnecting slip band segments. Most interestingly, the fracture surfaces appeared identical for any given applied stress intensity suggesting that fracture occurs at a critical hydrogen concentration. A transient model for impurity atom interaction with a blocked edge dislocation array was then applied to the data and showed reasonably good agreement between predicted and measured crack growth rates. This agreement supports the concept that fracture occurs when hydrogen reaches a critical concentration at the slip band intersections and the observation that rates are controlled by the diffusivity of hydrogen through the lattice. Moreover, the results show that microvoid formation at slip band intersections is the dominant event in the fracture process.

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Introduction

Austenitic stainless steels and superalloys are often used in hydrogen and hydrogen-producing environments due to their good resistance to crack growth. However, they are not immune to hydrogen-induced cracking. (1,2) Therefore determination of hydrogen-induced crack growth susceptibility and the controlling processes for crack growth under all conditions of use are of great importance in defining limits for safe and reliable performance. This requires that the effects of temperature be known. Although there have been several studies of temperature effects on hydrogen-induced crack growth in ferritic (3-6) and metastable austenitic alloys (7), there have been no studies of temperature effects on stable austenitic alloys. As a result, the effects of temperature over a range of near ambient temperatures were studied in the austenitic iron-based superalloy IN903. This alloy has been well-characterized in previous studies, is known to be very susceptible to hydrogen-induced fracture, and exhibits behavior representative of other alloys in its class. (8-10) Through a combination of fractography, transmission electron microscopy, and application of a transient model for crack growth, it will be shown that microvoid formation at slip band intersections is the dominant event in the fracture process. It will also be shown that microvoid formation and subsequent slip band fracture occurs when hydrogen reaches a critical concentration with the rate of crack growth controlled by the diffusivity of hydrogen through the lattice.

Material

IN903 is a γ strengthened, fcc superalloy with the composition given in Table I. The heat of IN903 used in this study is the same as used in a study of hydrogen effects on fracture toughness and room temperature slow crack growth. (8,9) This alloy was solution-treated at 940°C, water quenched, and then double-aged at 720°C/8h plus 620°C/8h to a yield strength of 1080 MPa. The resulting microstructure consisted of equiaxed grains approximately 28 μm in diameter with a significant number of annealing twins and matrix carbides. This alloy was strengthened by 10 volume percent of 20 nm diameter γ' precipitates and contained small grain boundary carbides. (8,9)

Crack growth rates and thresholds were measured using bolt-loaded wedge opening load (WOL) samples machined from rough-cut blanks after heat treatment. These samples are similar to those used by Novak and Rolfe (11) with load-line to back surface distances (W) equal to 56.9 mm, thicknesses (B) equal to 22.2 mm, and heights (2H) equal to 55.4 mm. The samples were fatigue pre-cracked and then hydrogen charged in purified hydrogen gas at 300°C for 60 days at a pressure of 68.9 MPa (680 atm). This procedure produced a hydrogen concentration of 2900 appm as calculated from Robertson's (12) solubility data on IN903 with pressure corrected to give effective fugacity. (13) After charging, the samples were maintained at 0°C to minimize offgassing of near surface hydrogen.

The samples were tested in an environmental chamber at 253 K, 273 K, 298 K, and 333 K with chamber temperatures controlled to within $\pm 1\text{K}$ through use of a proportional temperature controller, nichrome heating elements, and liquid nitrogen. Each sample was brought to the desired test temperature and allowed to equilibrate for one hour to insure a uniform temperature through the sample thickness and an equilibrium distribution of hydrogen in the lattice and γ trap sites. The partitioning of hydrogen between lattice and trap sites was measured in a previous study at room temperature. (14) Partitioning at other temperatures was determined using the relationship given by Johnson and Lin. (15) Total sample hydrogen concentration, concentration in the lattice, and concentration in the γ trap sites for each test temperature are given in Table II. The samples were then bolt-loaded to a crack tip stress intensity of 55 $\text{MPa}\cdot\text{m}^{1/2}$ with the loads continuously monitored as a function of time using individually calibrated load cells similar to those used by Loginow and Phelps. (16) From the load and time measurements, average crack velocity and corresponding average stress intensity values were calculated from the compliance

Table I. Composition of IN903 Used in This Study. (w/o)

Ni	Co	Ti	Al	Nb	Mn	Si	Mo	C	Fe
37.8	15.3	1.33	0.07	3.07	0.15	0.7	0.1	0.04	bal.

Table II. Hydrogen concentrations in samples charged in 68.9 MPa hydrogen gas at 300°C for 60 days.

Test Temperature (K)	Lattice Concentration (appm)	Trap Concentration (appm)	Total Concentration (appm)
333	714	2176	2900
298	411	2489	2900
273	232	2668	2900
253	129	2771	2900

and stress intensity expressions for WOL samples derived by Hudak and Saxena (17) using the assumption of a rigid bolt (constant crack opening displacement). After each test, the samples were unloaded, allowed to offgas at room temperature, and then pulled to failure.

Results

At all temperatures, crack growth occurred following a short incubation time that decreased with increasing temperature. The rates exhibited typical stage I and stage IIa behavior as shown in Fig. 1. (1) In stage I, the crack growth rates exhibited a very strong dependence on applied stress intensity, decreasing rapidly to apparent threshold values as applied stress intensity decreased. The apparent threshold values decreased as temperature increased to 298K and then increased at a temperature of 333 K. In stage IIa, the crack growth rates were strongly dependent on temperature, increasing more than two orders of magnitude as temperature increased from 253 K to 298 K. There was a reversal in behavior at higher temperatures with rates decreasing slightly as temperature increased to 333K. The stage IIa rates were weakly dependent on applied stress intensity with a slightly greater dependence on stress intensity exhibited at lower temperatures. The effects of temperature on crack growth rates and apparent thresholds is more clearly shown in the crossplot of Fig. 2. The stage IIa crack growth rates correspond to an applied stress intensity of 45 MPa-m^{1/2} and are representative of behavior observed at all stage IIa applied stress intensities.

The stage IIa crack growth rates for temperatures up to 298 K exhibit an Arrhenius-type relationship from which an apparent activation energy of 0.71±0.2 eV is found for the low-

Figure 1. A plot of crack growth rate versus applied stress intensity shows that susceptibility to hydrogen-induced crack growth increased with temperature reaching a maximum at 300 K. There was a slight decrease in crack growth susceptibility as temperature further increased to 333 K.

Figure 2. A cross-plot of crack growth rates and thresholds shows that the strong detrimental effect of hydrogen on crack growth rates and thresholds persists over a wide range of temperatures with a maximum in susceptibility occurring near room temperature.

temperature rate-controlling process at an applied stress intensity of 45 MPa·m^{1/2}. This value varies slightly as applied stress intensity approaches stage I and stage III regimes. Although the apparent activation energy is higher than the 0.5 eV for diffusion of hydrogen through the matrix of IN903, it can be directly related to the effects of trapping where the apparent activation energy for trap-delayed diffusion consists of 0.5 eV for the energy of diffusion through the matrix and 0.2 eV for the energy of trapping at γ precipitates. (12,14) Further support for a diffusion controlled mechanism comes from stress relaxation and strain rate cycling experiments showing that hydrogen has no effect on dislocation motion up to the point of tensile instability in this alloy. (18) Above 298 K, stage IIa crack growth rates deviate from Arrhenius-like behavior and decrease as temperature increases. This parallels behavior observed in a number of alloy systems where a decrease in crack growth rates above room temperature corresponds to a transition between low and high temperature rate-controlling processes. (3-5,7,19-23)

In all samples, fracture occurred primarily along slip bands with some twin band fracture as shown in Fig. 3. Matrix carbides were always present on the fracture surface with void growth around the small carbides and separation of fractured segments in large carbides. The slip band facets also exhibited a significant amount of local deformation, characterized by tear ridges, dimples, and microvoids. For any given applied stress intensity in stage IIa crack growth, the fracture surfaces were identical in appearance for all test temperatures and stage IIa crack growth rates as shown in Fig. 3. In contrast, the extent of local deformation on the slip band facets varied directly with threshold values reaching a minimum at 298 K. These observations contrast sharply with fracture in uncharged samples which occurs by microvoid coalescence. (8,9) Although the fracture modes reflect the severe effect hydrogen has on crack growth susceptibility, they do not vary with temperature and therefore do not reflect the transition in crack growth behavior above room temperature.

Discussion

Understanding the effects of hydrogen on the fracture process is vital for the safe and reliable use of this and similar alloys in hydrogen and hydrogen-producing environments. It is also needed for advances in life prediction. In all stages of crack growth in IN903, hydrogen-induced failure occurs by fracture of matrix carbides followed by failure along the coplanar slip bands. The fracture of matrix carbides creates microcracks normal to the maximum local principal stress with secondary fractures normal to the microcracks. As shown schematically in Fig. 4, these microcracks trigger void formation at slip band intersections. Fracture then occurs along the slip

Figure 3. Comparison of the fracture surfaces during stage IIa crack growth at (a) 253 K, (b) 298 K, and (c) 333 K and an applied stress intensity of $45 \text{ MPa}\cdot\text{m}^{1/2}$ showed that fracture occurred by matrix carbide fracture and slip band failure at all temperatures studied with no discernable difference in appearance.

band segments between microvoids to link the microcrack with the main crack. The most important process in this sequence of fracture events is the formation of microvoids at the slip band intersections. These intersections effectively block dislocation motion which leads to very high dislocation densities within the slip bands and high stresses at the head of the dislocation pile-ups. (24,25)

The fracture of matrix carbides is unaffected by hydrogen concentration. (8,26) However, microvoid formation and subsequent slip band fracture results from the trapping of hydrogen at slip band intersections and dislocations within the slip bands. Atomistic simulations for nickel show that between dislocations and slip band intersections, trapping at slip band intersections is the controlling step in the fracture process with binding energies within the intersection near 0.4 eV. Binding energies at individual dislocation cores are less than 0.12 eV. Trapping extends several nanometers into the surrounding lattice around both types of defects due to lattice distortion. (27) Following long exposure times encountered at threshold, an equilibrium distribution of hydrogen in traps is established by the redistribution of hydrogen from γ' precipitates to slip band intersections. This produces lower thresholds with less local plastic deformation prior to failure in samples with higher hydrogen contents.

Figure 4. Hydrogen-induced failure begins by fracture of matrix carbides which creates microcracks normal to the maximum local principal stress. These microcracks trigger microvoid formation at slip band intersections followed by failure of the interconnecting slip band segments linking the microcrack with the main crack.

Based on the assumption that the slip band length between intersections defines the characteristic fracture distance, the time available during stage IIa crack growth permits only the local redistribution of hydrogen to high energy trap sites at slip band intersections. Under these conditions, fracture occurs prior to establishment of an equilibrium distribution of hydrogen.

Two observations strongly suggest that the concentration of hydrogen for fracture is constant for any given applied stress intensity. First, stage IIa fracture surfaces appear essentially the same regardless of test temperature, and second, the void-to-carbide size ratios during stage IIa crack growth from samples with different initial hydrogen contents superimpose. The difference in crack growth rates observed at different temperatures and for different initial hydrogen concentrations can therefore be attributed to the difference in time required for accumulation to a critical concentration. The small dependence of crack growth rates on applied stress intensity suggests that the critical concentration varies with equivalent plastic strain but this is a secondary effect in the fracture process.

The effect of hydrogen trapping at slip band intersections can be quantitatively described with a transient model by Lyubov and Vlasov (28) for impurity atom diffusion and accumulation at the head of a blocked array of edge dislocations. The interaction between impurity atoms and dislocations consists of volumetric, modulus, electrostatic and chemical contributions with the volumetric contributions being the dominant factor. As a consequence, Lyubov and Vlasov used the volumetric elastic analysis of Cosgrove and Liu (29) to approximate the binding energy of an impurity atom with a blocked array of edge dislocations and a transient expression for diffusion in a force field to define the time-dependence of impurity atom diffusion and accumulation. This led to the following expression for impurity atom concentration, C , at a distance r ahead of the dislocation pile-up under an applied stress, σ , at time, t ,

$$\frac{C}{C_0} = 1 - \frac{A}{kT} \sqrt{\frac{\ell}{r}} \sin\left(\frac{\theta}{2}\right) \left\{ \operatorname{erfc} \frac{\rho-1}{2\sqrt{\tau}} - \left[\frac{1}{2}(\rho-1) + \frac{\tau}{4} \right] \operatorname{erfc} \left[\frac{\rho-1}{2\sqrt{\tau}} + \frac{\sqrt{\tau}}{2} \right] \right\} \quad (1)$$

where

$$A = \frac{8\pi(1+\nu)}{3} \epsilon_H r_1^3 \sigma$$

$$\tau = \frac{D_H t}{r_0^3} \quad (2)$$

$$\rho = \frac{r}{r_0}$$

For hydrogen in IN903, ν is Poisson's ratio equal to 0.234, r_0 is the dislocation core radius set equal to 0.15 nm, ϵ_H is the hydrogen atom misfit equal to 0.16 from the data of Lewis and Farrel (30), and r_1 is the average interatomic spacing equal to 0.15 nm. D_H is the effective hydrogen diffusivity (14,15) and C_0 is the lattice hydrogen concentration. For our application, the unstressed lattice concentration is used as there is insufficient time for long range hydrogen redistribution necessary to increase concentrations in the near crack tip region. The slip band length, ℓ , is equal to 0.75 μm based on slip band spacing measurements from SEM fracture surfaces and TEM foils from samples strained to 5 percent plastic strain. The applied shear stress on the dislocation, σ , is equal to 750

MPa at 298 K as determined from the ultimate tensile stress using the von Mises yield criterion and varies little over the range of temperatures studied. The time for diffusion and accumulation, t , is determined by dividing the slip band length by the crack growth rate. $\sin(\theta/2)$ is set equal to one to determine the maximum concentration. This leaves only the extent of the hydrogen cloud at the head of the dislocation pile-up involved in microvoid formation, r , to be defined. To a zeroth order approximation, r is taken to be equal to 16 nm, one-half the mean spacing between dislocations observed in IN903 slip bands. This is the effective extent of hydrogen atmosphere formation around individual dislocations. (28) It is also similar to the void sizes at slip band intersections measured on fracture surfaces.

To test the concept that hydrogen diffusion and accumulation at the head of a dislocation pile-up controls the kinetics of crack growth in IN903, Lyubov and Vlasov's model was applied to the hydrogen concentration data from a previous study and the the temperature data of this study. In previous work (9), the effect of hydrogen on crack growth rates and thresholds was determined at 298 K in samples charged to 1876, 2900, and 5000 appm. The crack growth rates varied almost three orders of magnitude over the range of concentrations studied ordering with sample concentration as shown in Fig. 5. The 2900 appm data was used as a reference from which critical hydrogen concentrations for fracture were determined as a function of applied stress intensity bypassing the requirement to rigorously define a failure criterion. The lattice hydrogen concentrations used in these calculations are given in Table III. Times to reach these critical concentrations were then determined for samples with 1876 and 5000 appm hydrogen. These times were converted to crack growth rates using the average slip band segment length of 0.75 μm as the characteristic length for fracture. Fig. 5 shows that these calculated crack growth rates are in good agreement with observed rates. The times to reach the critical concentrations for samples charged with 2900 appm hydrogen and tested at 253 K, 273 K, and 333 K were then calculated using the 2900 appm 298 K data as a reference and converted to crack growth rates. As shown in Fig. 6, the magnitude of the calculated stage IIa crack growth rates are in reasonably good agreement with the magnitude of the observed rates up to 298 K. However, the

Figure 5. The crack growth rates calculated from the model are in reasonably good agreement with stage IIa crack growth rates at room temperature in samples charged to three different hydrogen concentrations.

Table III. Hydrogen concentrations in samples charged in 68.9 MPa hydrogen gas at 300°C for 60 days.

Charging Pressure (atm)	Effective Fugacity (atm)	Lattice Concentration (appm)	Trap Concentration (appm)	Total Concentration (appm)
340	445	210	1670	1880
680	1160	410	2490	2900
1360	3060	1290	3710	5000

model predicts the onset of stage I behavior and thresholds at much higher stress intensities than observed. Nevertheless, if sample hydrogen concentrations were 10 percent higher than calculated from the charging conditions, which is within the scatter of hydrogen concentration measurements, agreement is very good between calculated and observed crack growth rates at all stress intensities.

These results show that the hydrogen concentration distributions for fracture at temperatures up to 298 K are well-described by a quantitative model for the elastic interaction between an impurity atom and a blocked edge dislocation array. The good agreement between predicted and observed crack growth demonstrates that the crack growth rates are controlled by the concentration and diffusivity of hydrogen in the lattice where increasing either lattice concentrations or test temperatures leads to higher crack growth rates. Moreover, the model is physically consistent with observations that slip band fracture begins with microvoid formation at slip band intersections followed by failure of interconnecting slip band segments.

Above 298 K, crack growth rates deviate from low temperature behavior and decrease with increasing temperature. This behavior parallels crack growth observed in a number of alloy systems and represents a transition to high temperature behavior where crack growth rates decrease dramatically with small increases in temperature. This transition in crack growth behavior has been attributed to several different mechanisms which vary with material and environment. In 4130, Nelson and Williams (3) attributed the decrease in stage II crack growth

Figure 6. The magnitude of the calculated stage IIa crack growth rates are in reasonably good agreement with the magnitude of the observed rates up to 298 K. However, the model predicts the onset of stage I behavior and thresholds at much higher stress intensities than observed. The model does not predict the decrease in crack growth rates observed at higher temperature.

rates to a reduction in the hydrogen supply rate whereas in 4340 Gao and Wei (31) attributed the reduction in rates to hydrogen partitioning among different microstructural sites. There may also be different sets of strong traps acting as fracture nucleation sites and as sites affecting hydrogen diffusivity as observed by Gerberich and co-workers (20,32) in cathodically charged 4340. A similar situation appears to apply for IN903 where at low temperatures short-range hydrogen redistribution occurs from lattice sites to dislocations and slip band intersections. At high temperatures, the interdiffusion distance extends beyond nearby dislocations in the coplanar slip bands where nearby dislocations can reduce the effective hydrogen diffusion rate. At the same time, lower trap site occupancy at higher temperatures requires higher hydrogen concentrations to reach the critical hydrogen concentration for fracture.

Conclusions

This study shows that temperature has a very strong effect on crack growth susceptibility in the iron-based superalloy IN903 with a maximum susceptibility measured at 298 K. For temperatures up to 298 K, the crack growth rates exhibit an Arrhenius-like behavior with an activation energy of 0.71 ± 0.2 eV. This corresponds to a controlling process of trap-delayed hydrogen diffusion. The stage IIa crack growth rates decrease at higher temperatures, but the energy for the controlling process can not be defined from the data available.

Failure in all stages of crack growth begins with the fracture of matrix carbides which form microcracks. The microcracks trigger void formation at slip band intersections and failure of the interconnecting slip band segments. Observations combined with atomistic simulations indicate that microvoid formation at the slip band intersections is the process controlling hydrogen-induced fracture of this alloy. Moreover, the similarity in fracture surface appearance strongly suggests that fracture occurs when the hydrogen concentration reaches a critical level at these intersections. The conclusions from these observations are supported by the good agreement between crack growth rates predicted by a transient model based on impurity atom diffusion and accumulation at the head of a blocked edge dislocation array and crack growth rates measured at temperatures up to 298 K. Through comparison with findings from other alloy systems, the reduction in crack growth rates at higher temperatures appears to be due to a reduction in the rate of supply and trapped concentration of hydrogen at the slip band intersections

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