A COMPARISON OF INTERNAL HYDROGEN EMBRITTLEMENT AND HYDROGEN ENVIRONMENT EMBRITTLEMENT OF X-750

D. M. Symons
Bechtel Bettis Inc.
Bettis Atomic Power Laboratory
West Mifflin, PA 15122

DE-AC11-98PN38206

RECEIVED
OCT 29 1999
OSTI

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States, nor the United States Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed, or represents that its use would not infringe privately owned rights.

BETTIS ATOMIC POWER LABORATORY

WEST MIFFLIN, PENNSYLVANIA 15122-0079

Operated for the U.S. Department of Energy
by Bechtel Bettis, Inc.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

A COMPARISON OF INTERNAL HYDROGEN EMBRITTLEMENT AND HYDROGEN ENVIRONMENT EMBRITTLEMENT OF X-750

Douglas M. Symons

Bettis Atomic Power Laboratory

West Mifflin, Pennsylvania 15122

ABSTRACT

Hydrogen has been shown to degrade the mechanical properties of nickel-base alloys. This degradation occurs whether the material is in a hydrogen producing environment or if the material has dissolved hydrogen in the metal due to prior exposure to hydrogen. Materials behave differently under these two conditions. Therefore, the degradation due to hydrogen has been split into two categories, internal hydrogen embrittlement (IHE) and hydrogen environment embrittlement (HEE). IHE may be defined as the embrittlement of a material that has been charged with hydrogen prior to testing or service while HEE may be defined by the embrittlement of a material in a hydrogen environment where the hydrogen may come from gaseous hydrogen or generated from a corrosion reaction. This work will compare IHE and HEE of fracture mechanics specimens. Different fugacities of hydrogen for HEE and hydrogen concentrations for IHE were examined for Alloy X-750, a nickel-base super alloy. The test results were analyzed and the role of hydrogen in IHE and HEE was evaluated. A model based on a critical grain boundary hydrogen concentration will be proposed to describe the behavior in both HEE and IHE conditions.

I. INTRODUCTION

The embrittlement of nickel-base alloys by hydrogen is a problem of both theoretical and practical interest. Hydrogen has been shown to degrade the mechanical properties of nickel-base alloys including pure nickel (1), solid solution strengthened Ni-Cr-Fe alloys (Alloy 600 and Alloy 690), (2)(4)(3) and precipitation strengthened Ni-Cr-Fe alloys (Alloy X-750 and Alloy 718) (6)(7)(5). For example, the practical need for understanding hydrogen embrittlement is in nuclear reactor environments, hydrogen has been shown to precipitously decrease the fracture resistance of the nickel-base structural materials (3)(7). The embrittlement of a material that has hydrogen dissolved within the matrix prior to loading has been termed internal hydrogen embrittlement (IHE) while the embrittlement of a material in a hydrogen environment has been described as hydrogen environment embrittlement (HEE). The hydrogen environment may be due to corrosion generated hydrogen or a partial pressure of hydrogen in a gas phase. Materials have been shown to behave differently HEE and IHE conditions (8)(9)(5)(10)(11).

It has been proposed that for most engineering materials, the embrittlement due to hydrogen, whether HEE or IHE, is not from the uniformly distributed hydrogen in the matrix, but the hydrogen segregated to microstructural inhomogeneities (12)(1). It was shown by Lassila and Birnbaum (1) that for pure nickel the degree of embrittlement may be modeled by the diffusive segregation of hydrogen to the grain boundaries. It will be shown here that for Ni-base alloys with grain boundary carbides, the embrittlement is controlled by the segregation of hydrogen to both the grain boundary and grain boundary carbides and that the degree of embrittlement is dependent on the "source", internal vs. external, of the hydrogen.

A review of some key papers comparing IHE and HEE will be provided as a starting point to better describe the differences observed between IHE and HEE in a specific alloy. The previous work comparing IHE to HEE proposed many differences and similarities between IHE and HEE.

It was suggested that the difference between IHE and HEE may be related to a change in the characteristic fracture distance with no change in embrittlement mechanism (8), a change in both the characteristic distance and the embrittlement mechanism (5), or no change in either embrittlement mechanism or characteristic distance (11).

In order to compare IHE and HEE, different fugacities of hydrogen and hydrogen concentrations will be examined, and the role of hydrogen evaluated. A well characterized material is required to compare the IHE and HEE behavior. The material that will be used for this comparison is Alloy X-750. Alloy X-750 has been shown to be susceptible to IHE (6)(13) and HEE (7)(14)(15).

There have been extensive microstructural studies (16)(17)(18) and IHE investigations of Alloy X-750 (6)(13)(19). The results for the IHE of X-750 show that at all dissolved hydrogen levels, the fracture was predominantly intergranular. The HEE studies for this alloy have all previously been performed in an aqueous environment where the hydrogen fugacity is not well characterized (7)(14)(15). In order to quantitatively model the HEE of this alloy, a well characterized hydrogen environment is required. New results in hydrogen gas over a temperature range of 54°C to 338°C with pressures from 0.14 MPa to 34.4 MPa will be used to characterize the HEE of Alloy X-750. An evaluation of all the data will be provided and a model to describe the behavior will be developed based on an understanding of the material-environment interaction. This work will show, similar to the work by Gerberich on steels (8), that the characteristic fracture distance is different for IHE and HEE. Further, this work shows that while both hydrogen induced decohesion and hydrogen enhanced plasticity may be responsible for IHE, HEE appears to be caused by hydrogen induced decohesion.

II. PREVIOUS WORK

The hydrogen embrittlement of materials including nickel-base alloys has been studied extensively including review articles ((20)(21)(22)) and conference proceedings((23)(24)). This section is not meant to be a review of the extensive literature that is available, but will review some key papers that describe the general trends of IHE and HEE.

Walter, Jewett, and coworkers have published numerous papers on the role of hydrogen on the behavior of structural materials using uniaxial tensile and notched tensile specimens (5)(10). Using these specimens, there were many differences observed between the IHE and HEE behavior of materials. For example, results from testing steel showed that while 2 ppm was required to embrittle precharged specimens, only 0.02 ppm was required when testing was performed in a hydrogen environment. It was further shown for the uniaxial type specimen that some alloys, for example Alloy 718 or Rene' 41, are extremely susceptible to HEE but the embrittlement due to IHE was negligible. Due to the dramatic difference observed between IHE and HEE behavior, these authors proposed that the embrittlement mechanism may be different. Fritzmeier, Walter, Meisels, and Jewett also investigated Alloy 903, a FCC Fe-Ni- base precipitation strengthened superalloy (10). Again using uniaxial specimens, this alloy was embrittled when precharged with hydrogen while no sign of embrittlement was observed when tested in a high pressure hydrogen environment. This was used for further evidence of their hypothesis that there may be a different mechanism for IHE as compared to HEE.

Moody and coworkers also investigated both IHE and HEE of Alloy 903 (11). They used precracked wedge open loaded (WOL) specimens over large ranges of hydrogen pressure for

HEE and dissolved hydrogen for IHE. A single model for the threshold stress intensity factor for IHE and HEE of this alloy was developed based on the local matrix hydrogen concentration at the peak hydrostatic stress location, including the increased solubility due to the triaxial stress field ahead. The results from this testing were interpreted as suggesting that for crack mechanics specimens, both the embrittlement mechanism and the characteristic distance do not change between IHE and HEE specimens. Therefore, by comparing the work of Moody et al. and Walter et al., there appears to be not only a difference between IHE and HEE behavior, but that the difference in behavior is specimen type dependent.

Gerberich and coworkers using fracture mechanics type specimens have also performed testing and developed models to describe HEE and IHE (8)(9). In their analysis, it was shown that the same modeling approach satisfactorily described the fracture behavior for both IHE and HEE of steels. In this work, like the work of Moody and coworkers, the model was based on achieving a critical hydrogen concentration at a characteristic distance ahead of the crack tip. It was shown that for HEE the characteristic distance is much nearer to the crack tip than for IHE while the mechanism did not appear to change. Also, the trapping energy required to model the data was different for the two conditions.

III. EXPERIMENTAL PROCEDURE

A. MATERIALS

The material used in this program was from 6.35 cm diameter Alloy X-750 bars heat treated to the HTH condition with the composition provided in Table I. The HTH condition consists of a solution anneal at 1094°C for 1-2 hours with a rapid air cool followed by aging at 704°C for 20 hours. This material has previously been characterized (6). The mean intercept grain size was 125 μm with small discrete M_{23}C_6 carbide precipitates decorating the grain boundaries as shown in Figure 1. The alloy was strengthened by ~20 nm γ' precipitates with a rounded cuboidal shape and a weight fraction of 0.126. Primary carbides and carbonitrides, $\text{M}(\text{C},\text{N})$, were observed to be aligned along the rolling direction.

TABLE 1: CHEMICAL ANALYSIS (wt. %)

Ni	Cr	Nb	Ti	Al	Fe	C
71.17	15.46	1.00	2.67	0.76	8.33	0.072

B. TESTING IN AIR OF NONCHARGED SPECIMENS

The noncharged testing used compact tension specimens that were 15.2 mm thick (B) with 20% side grooves (10% per side) and a width (W) of 30.5 mm. The side grooves were essential to obtaining valid results since this material tends to have excessive tunneling. All specimens had an L-R orientation and were precracked at a frequency of 30 Hz with a final maximum K_I of 16.5 MPa/m . The noncharged specimens were tested using an elastic-plastic fracture toughness testing technique at 25°C according to ASTM E813-89 (Standard Test Method for J_{IC} , A

Measure of Fracture Toughness). The single specimen technique was used and the crack length was determined using compliance.

C. TESTING HYDROGEN PRECHARGED SPECIMENS

i) Specimens

Compact tension (CT) specimens with a width (W) of 20.3 mm and thickness (B) of 10.1mm were machined in an L-R orientation. The specimens were fatigue precracked prior to hydrogen charging to an a/W of about 0.45 at a final maximum K_I of 16.5 MPa $\sqrt{m}$.

ii) Hydrogen Charging

The procedure for charging the specimens for the IHE investigation has previously been reported (16) and will briefly be summarized herein. The compact tension specimens were charged in a high-pressure hydrogen autoclave. Two charging runs were performed, one at a hydrogen pressure of 34 MPa and 360°C, and a second at 21 MPa and 315°C. Specimens were charged in the autoclave for three weeks at 360°C or four weeks at 315°C. After hydrogen charging, the specimens were removed from the autoclave and stored below 0°C. Alloy X-750 blanks were analyzed for hydrogen concentration using a LECO hydrogen analyzer model RH404. The hydrogen concentration of the specimens charged at 360°C was 65 ppm by weight and for the specimens charged at 315°C was 40 ppm by weight. It was previously shown that sufficient time for the specimens to achieve a uniform hydrogen concentration was allowed (16).

iii) Mechanical Testing

The fracture toughness of the hydrogen-charged specimens was determined by using linear-elastic fracture toughness testing. The majority of tests were run at 25°C with limited testing at 54°C and 100°C. The testing was run according to ASTM E399-90 (Standard Test Method for

Plane Strain Fracture Toughness of Metallic Materials) to determine K_{IC} , with the exception of the loading rates. Loading rates were varied by a factor of 50 to evaluate hydrogen diffusion effects on the fracture behavior. The testing at 54°C and 100°C were run at higher rates to minimize the loss of hydrogen out of the crack tip.

D. HYDROGEN ENVIRONMENT TESTING

i) Specimens

Compact tension (CT) specimens with a width (W) of 20.3 mm and thickness (B) of 10.1 mm were used. The specimens were fatigue precracked to an a/W of about 0.45 at a final maximum K_I of 16.5 MPa $\sqrt{m}$. All specimens were machined in an L-R orientation.

ii) Mechanical Testing

The testing was performed at 54°C. This is the test temperature that was observed to maximize the embrittlement of this alloy for HEE in hydrogenated water (10). At 54°C, the fracture tests were conducted on an electrohydraulic closed-loop machine in stroke control with a loading rate of 4.4 MPa $\sqrt{m/hr}$. One specimen was tested at a loading rate of 0.44 MPa $\sqrt{m/hr}$ to confirm that the loading rate was sufficiently slow so that rate dependent effects were not affecting the results.

Displacements were determined from an internal displacement gauge attached to the front face of the specimen. During each test the displacement was recorded continuously as a function of load, as measured by a system load cell. The tests were run at pressures of 0.14 MPa, 0.34 MPa, 3.4 MPa, and 34 MPa hydrogen at 54°C. The hydrogen gas was a minimum of 99.999% pure. In order to maintain an ultrapure atmosphere, the autoclave was evacuated prior to adding the hydrogen gas. Since all specimens tested in the hydrogen gas failed in a brittle, linear-

elastic manner, K_{IC} methods were used to determine fracture toughness behavior. Values of K_{IC} were determined using the methodology described in ASTM E399-90.

An additional test was performed on two bolt-loaded specimens to investigate the interaction of internal hydrogen and the hydrogen environment. One specimen was loaded to a stress intensity factor of $22 \text{ MPa}\sqrt{\text{m}}$ and the second was loaded to $33 \text{ MPa}\sqrt{\text{m}}$. The specimens were bolt loaded using a load transfer technique. The specimens were loaded to 98% of the final load in a tensile machine, then the load was transferred to the specimen by tightening the bolt while maintaining the crack mouth opening displacement (CMOD) constant as measured by a clip gage. After the bolt loading, the specimens were inserted into the autoclave, the autoclave was evacuated and then pressurized to 3.4 MPa and heated to 315°C . Once at temperature, the pressure was then increased to 21 MPa. At this high pressure, the autoclave was held for 4 weeks. This allowed sufficient time for the specimens to achieve a uniform hydrogen concentration of 40 ppm. Also, at the high temperature and pressure, the applied stress intensity factor was well below the fracture toughness (25). Therefore, no cracking was expected during the high temperature hold. The pressure was then kept constant, and the autoclave was cooled. At 25°C and a pressure of 21 MPa, the equilibrium hydrogen concentration is 10 ppm. Therefore, at the low temperature, the specimen was supersaturated with hydrogen. When the specimens were removed from test, the crack length was measured and the remaining load on the specimens was determined using the above described loading technique performed in reverse.

Testing the noncharged specimens and the HEE and IHE specimens per the same specification was not possible. It was not possible to perform a J test on the HEE and IHE specimens since they failed in a linear-elastic manner. It was not possible to perform a linear-elastic fracture

toughness test on the noncharged specimen because the bar was not large enough to machine specimens that would meet the size requirements for valid K_{IC} test results.

III. RESULTS

A. Air Tests of Noncharged Specimens and Hydrogen Precharged Specimens

The results from the fracture-toughness testing in air are shown in Table 2. The measured fracture toughness of the hydrogen-charged specimens represented valid K_{IC} values as determined by ASTM E399-90. Valid results for J_{IC} were obtained on the noncharged specimens and converted to K_{Jc} per ASTM E813-81.

TABLE 2 FRACTURE TOUGHNESS PROPERTIES OF
HTH ALLOY X-750 IN AIR TESTS AT 25°C AND 54°C

Hydrogen conc. (ppm)	Test Temp (°C)	Loading Rate (MPa√m/hr)	K_{Ic} (MPa√m)	K_{Jc} (MPa√m)	Valid per ASTM E399 or E813
0	25	420-1980	--	147	Yes
40	25	180	53	--	Yes
65	25	1980	52	--	Yes
65	25	180	45	--	Yes
65	25	42	42	--	Yes
65	54	180	44	--	Yes
65	100	1800	49	--	Yes

The fracture toughness was dramatically decreased by hydrogen charging the specimens. The fracture toughness dropped from 147 MPa√m with no hydrogen to as low as 42 MPa√m with 65 ppm hydrogen at 25°C. There was only a small difference between the testing at 25°C and 54°C. From 54°C to 100°C the fracture toughness showed a noticeable increase.

The loading rate also affected the toughness of the hydrogen-charged specimens. The change in fracture toughness caused by a change in loading rate was small compared to the change in fracture toughness due to hydrogen charging. The testing at 54°C was in the load rate regime where loading rate did not affect fracture toughness.

The fracture morphology in noncharged specimens was a mixture of approximately 20%-transgranular ductile dimple fracture and 80%-intergranular fracture as shown in Figure 2. The fracture morphology of all the hydrogen specimens was intergranular as shown in Figure 3. It was previously reported that the critical fracture toughness determined from the slow rate testing is the same as the threshold stress intensity factor determined from crack growth rate testing of hydrogen charged specimens (19). This further supports the statement that the rising load test was slow enough to achieve a rate independent fracture toughness. The rates required for the minimum in fracture toughness are similar to the rates that Moody et al. observed no rate effect on toughness for Alloy 903 (11).

B. Results of Testing in Hydrogen Gas

The results from the fracture-toughness testing in hydrogen gas are provided in Table 3. The measured fracture toughness of these specimens represents valid K_{IC} values as determined by ASTM E399-90. The crack extension was rapid once the critical K was achieved. Though, it should be noted that the cracking was not unstable and could be stopped if the specimen was unloaded rapidly enough. It should also be noted that the 0.34 MPa test at both 4.4 MPa $\sqrt{m/hr}$ and 0.44 MPa $\sqrt{m/hr}$ resulted in the same fracture toughness. This verifies that the testing was at a slow enough rate that the toughness was rate independent for the environmental testing.

TABLE 3: FRACTURE TOUGHNESS PROPERTIES OF
HTH ALLOY X-750 TESTED IN HYDROGEN GAS AT 54°C

Hydrogen pressure (MPa)	Solubility per Sievert's Law (23) (ppm)	Loading Rate (MPa√m/hr)	K _{IC} (MPa√m)
0.14	1.0	4.4	43
0.34	1.7	4.4	36
0.34	1.7	0.44	36
3.4	5.4	4.4	29
34.4	17	4.4	22

Again, the fractography showed that all the specimens had an intergranular fracture morphology.

At the low pressure, 0.14 MPa hydrogen, while the fractography shows a predominantly intergranular fracture, there is limited (less than 10%) transgranular faceted fracture as shown in Figure 4. At a pressures of 0.34 and over, the fractography showed over 98% intergranular fracture. The typical fractography for the higher pressure specimens is shown in Figure 5. The effect of pressure on the fracture toughness is displayed in Figure 6. Included in Figure 6 is some previously reported data for HEE of Alloy X-750 at elevated temperatures (25). The higher temperature work will be required to analyze the role of trapping on the embrittlement for HEE testing. The previous work included testing at 260°C and 338°C in hydrogen gas at a pressure of 13.8 MPa. The fracture toughness values under these conditions were 68 MPa√m at 260°C and 87 MPa√m at 338°C. The fracture morphology for the elevated temperature specimens was also intergranular (25). As shown in Figure 6, the fracture toughness increases as the pressure is decreased or the temperature is increased. A comparison of the rate independent fracture toughness values for both IHE and HEE as a function of bulk hydrogen concentration is shown in Figure 7. It may be observed from this plot that much less hydrogen is required to embrittle materials in a hydrogen environment as compared to internal hydrogen.

The bolt-loaded specimens that were hydrogen charged and then tested in the hydrogen environment had a final stress intensity factor of $20 \text{ MPa}\sqrt{\text{m}}$ for both specimens. This may be considered to be a threshold since the crack was advancing in both specimens and stopped at the same stress intensity factor even though one specimen grew 1.5 mm and the other specimen grew 5.7 mm. The fractography on both of these specimens showed an intergranular morphology.

IV. DISCUSSION

It was clear from the results, Figure 7, that Alloy X-750 is susceptible to both IHE and HEE. It was further shown that much less hydrogen is required to embrittle the material in the hydrogen environment as compared to internally charged hydrogen. This is similar to steel as described earlier. In order to quantitatively understand this behavior, a model will be proposed to describe the role of grain boundary hydrogen concentration on the fracture behavior and the characteristic distance ahead of the crack where fracture initiates.

It was previously shown by Lassila and Birnbaum that the diffusive segregation of hydrogen to the grain boundaries in nickel controls the degree of embrittlement (1). It has also been proposed that the effect of hydrogen on the behavior of the nickel-base alloys with grain boundary carbides is to reduce the strength of the interface between the matrix and the grain boundary carbides (26)(4). The reduction in the interfacial strength would result in a lower macroscopic strain required for the initiation of a microcrack. In order to understand the fracture of Alloy X-750 and compare IHE and HEE, it is important to determine the trapping energy controlling the fracture, the characteristic distance, and the role of grain boundary hydrogen on the fracture toughness. The trapping energy controlling fracture may be associated with the microstructural feature controlling fracture. The trapping energies that may be related to fracture are the trapping at grain boundary carbides (36), the grain boundary region (1), dislocations (35), and voids (37).

Previous work investigating the role of microstructure on fracture suggested that grain boundary carbides increase the degree of embrittlement in these alloys. If this is to be the case, it is expected that the trapping energy controlling fracture should be close to the trapping energy of the grain boundary carbides. The previous work used plateau etching on IHE specimens of

Alloy X-750 to show that the matrix decohered from a grain boundary carbide (19). In order to further understand the role of grain boundary carbides, other work on a solid solution strengthened nickel-base alloy, Alloy 690 (Ni-30Cr-8Fe), was investigated (4). This work showed that the fracture toughness behavior was strongly affected by the precipitation of grain boundary carbides. The fracture toughness in Alloy 690 was measured in two conditions, the solution annealed condition (SA) and the solution annealed and aged condition (SAA). The aging in these specimens resulted in extensive carbide precipitation on the grain boundaries. The precipitation of grain boundary carbides resulted in a reduction of the fracture toughness from 210 kJ/m² to 90 kJ/m². It is not possible to perform this test on Alloy X-750 since the aging for the γ' strengthening precipitates is in the same range as the temperature for carbide precipitation. The same type of effect would be expected for the Alloy X-750 since both alloys are nickel-base with the same type of grain boundary carbides.

Analysis of HEE Specimens

The first step in analyzing the fracture behavior is to determine the trapping energy that controls the embrittlement. In the case of fracture mechanics specimens, the hydrogen is preferentially trapped at the grain boundary and grain boundary carbides in the hydrostatic stress field ahead of the crack. The trapped hydrogen concentration at the grain boundary may be described by Oriani's theory of trapping (27) including the trapping at the grain boundary and grain boundary carbides (28):

$$\frac{C_T}{1 - C_T} = \frac{C_{H\sigma}}{1 - C_{H\sigma}} \exp\left(\frac{H_T}{RT}\right) \quad \text{Eq. 1}$$

This may be simplified for small matrix concentrations where $1 - C_{H\sigma} \approx 1$ to:

$$C_T = \frac{C_{H\sigma} \exp\left(\frac{H_T}{RT}\right)}{1 + C_{H\sigma} \exp\left(\frac{H_T}{RT}\right)} \quad \text{Eq. 2}$$

where C_T is the trapped hydrogen concentration that is in equilibrium with the local matrix concentration. The local concentration is denoted here as $C_{H\sigma}$ because the matrix hydrogen solubility is increased ahead of a crack due to the hydrostatic stress field opening the lattice. This has been described by Li, Oriani, and Darken (29):

where $C_{H\sigma}$ is the local hydrogen concentration in the crack tip stress field, C_H is the hydrogen concentration in the unstressed region, σ_H is the hydrostatic stress, $\bar{V}$ is the partial molar volume of hydrogen, R is the universal gas constant ($8.314 \text{ J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$) and T is the absolute temperature in K. The partial molar volume was taken to be $1.72 \text{ cm}^3/\text{mol}$ (30). The matrix

$$C_{H\sigma} = C_H \exp\left(\frac{\sigma_H \bar{V}}{RT}\right) \quad \text{Eq. 3}$$

solubility as defined by Sievert's law is shown below (26):

$$\begin{aligned} C_H(wppm) &= 39.25 \sqrt{P_{H_2}} \exp\left(\frac{-6850}{RT}\right) \\ C_H(appm) &= 2226 \sqrt{P_{H_2}} \exp\left(\frac{-6850}{RT}\right) \end{aligned} \quad \text{Eq. 4}$$

where the pressure is given in MPa.

The hydrostatic stress may be approximated using the equation developed by Akhurst and Baker (31):

$$\sigma_H = (2\sigma_{II} - \sigma_y)(1 + \nu)/3 \quad \text{Eq. 5}$$

where σ_H is the hydrostatic stress, σ_y is the yield strength, σ_{II} is the maximum stress ahead of the crack, and ν is Poisson's ratio. The stress distribution ahead of a crack may be described by Rice and Johnson's analysis of cracks under large geometry change conditions from the tip to two crack tip opening displacements (CTODs)(32). From Rice and Johnson's solution, the stress at the crack tip is four σ_y and at two CTODs it is five σ_y . The stress from two CTODs to 10 CTODs may be described using the crack tip stress solution developed by Hutchinson (33) and Rice and Rosengren (34)(HRR). The HRR solutions show that after 2 CTODs, the stress is a decreasing function of distance ahead of the crack. It will be shown latter, that the characteristic distance for HEE is within 2 CTODs. Therefore, the maximum stress, σ_{II} , will be taken as $4.5\sigma_y$ to calculate the hydrostatic stress.

Since testing was performed at a slow enough rate that reducing the rate further did not affect the results, the analysis will assume that the hydrogen concentration is at equilibrium at the location of fracture. As shown in Figure 8, all of the data could be correlated using Eq. (2) to calculate the grain boundary hydrogen concentration. The trapping energy, H_T , required for this correlation was 26 kJ/mol. This trapping energy is consistent with that determined by Young and Scully for trapping on the $M_{23}C_6$ grain boundary carbides in Alloy 600 (36). The matrix composition of Alloy 600 is similar to Alloy X-750. Therefore, it is expected that the trapping will be similar. The hypothesis that the grain boundary carbides, specifically decohesion of these

carbides, controls the embrittlement is supported by the trapping energy controlling fracture being the same as the trapping energy of hydrogen at grain boundary carbides.

The characteristic fracture distance may be evaluated by comparing the hydrogen concentration profiles to K_{IC} . The hydrogen concentration profiles are time dependent so they will be calculated at the time of fracture for each test. Since the tests were conducted at different rates and temperatures, the profiles will have different shapes. The fracture toughness is inversely proportional to the hydrogen concentration on a local scale. Therefore, the characteristic distance may be evaluated by determining the distance ahead of a crack where the local hydrogen concentration is the highest for the lowest K_{IC} , and as K_{IC} increases the local hydrogen concentration decreases. This technique will provide an approximate value of the characteristic distance.

The hydrogen profile may be determined using a matrix diffusion analysis. Yao, Meguid, and Cahoon evaluated the time dependent grain boundary hydrogen concentration with an external hydrogen source (38). Yao et al. showed that the grain boundary predominantly acts as a trap rather than a fast diffusion path. Thus, the depth of hydrogen penetration may be described by lattice diffusion and the grain boundary hydrogen concentration may be considered to be in equilibrium with the local matrix concentration. In the present analysis, the trapped hydrogen concentration will be determined using Eq.(2) which is based on the local matrix hydrogen concentration. The time-dependent matrix concentration ahead of the crack will be evaluated using a diffusion analysis based on a simplified geometry that is compatible with the boundary conditions of the crack. A semi-infinite plate solution with a point source will be used to define the geometry of the system since the far field hydrogen concentration will not be affected during the time of the test and the area being evaluated is very near the crack tip. The boundary

condition is defined as a constant surface concentration, C_s , and initial concentration in the material, C_∞ . The solution to these conditions has been determined by Crank and is shown below (39):

$$C_H(x) = C_s - (C_s - C_\infty) \operatorname{erf} \left(\frac{x}{\sqrt{4Dt}} \right) \quad \text{Eq. 6}$$

For HEE, C_H is the matrix hydrogen concentration at the distance x ahead of the crack, not including the effect of hydrostatic stress on solubility. C_∞ is zero and C_s is the concentration of dissolved hydrogen in equilibrium with hydrogen pressure through Sievert's law, Eq. (4), and D is the hydrogen diffusivity. The diffusivity is given by $D_H = D_0 \exp(-Q/RT)$ where $D_0=0.016\text{cm}^2/\text{s}$, $Q=49,000\text{ J/mol}$, R is in J/mol , T is in Kelvin, and D_H is in cm^2/s (26). Once the hydrogen profile is calculated per Eq. (6), Doig and Jones (40) showed that Eq. (3) may be used to then calculate the stress modified matrix hydrogen profile. Once the matrix hydrogen concentration is calculated, the grain boundary hydrogen concentration profile for the HEE specimens may then be determined through Eq. (2).

The hydrogen profiles for some of the HEE specimens are shown in Figure 9. On this figure one may compare the slow test in the hydrogen environment to the same pressure test at a higher rate. Since the behavior was the same for the fast and slow specimen, the hydrogen levels should be the same at the characteristic distance for the two tests. This shows that the characteristic distance should be less than $1\text{ }\mu\text{m}$.

Analysis of IHE Specimens

The trapping energy related to the fracture may also be evaluated for the IHE specimens. The IHE specimens can not be tested at elevated temperatures due to the loss of hydrogen out of the crack tip, loading rates will be used to evaluate the trapping energy associated with fracture. At 100°C, Eq. (6) shows that the specimen would lose 50% of the hydrogen in the near crack tip region in 5 minutes. An analysis based on the diffusive segregation of hydrogen will be used to evaluate rate effects.

The time dependence of the hydrogen segregation to the grain boundaries was developed by McLean (28) and is shown below:

$$\frac{C_{GBt} - C_o}{C_{GB} - C_o} = 1 - \exp\left(\frac{4Dt}{\alpha^2 d^2}\right) \operatorname{erfc}\left(\frac{2\sqrt{Dt}}{\alpha d}\right) \quad \text{Eq. 7}$$

where C_{GBt} is the grain boundary concentration of hydrogen after the test has run for time t , t is the test time in seconds, C_{GB} is the equilibrium concentration of hydrogen at the grain boundary as determined by Eq. (2), C_o is the initial concentration of hydrogen at the grain boundary, D is the diffusivity, d is the grain boundary thickness and α is equal to $\exp(H_B/RT)$. The trapped hydrogen concentration is denoted as C_{GB} here instead of C_T to indicate that the trapping is occurring on the grain boundary or grain boundary carbides.

The available results are not as extensive as the HEE results. From the available data, the trapping energy could be estimated to be between 10 kJ/mol and 18 kJ/mol for the internally charged specimens. The results are shown in Figure 10 using the trapping energy of 18 kJ/mol. The trapping energy for the IHE specimens is less than for the HEE specimens. Due to the

limited data for the IHE fracture mechanics specimens, the trapping energy responsible for fracture may be further quantified by analyzing the tensile behavior of IHE specimens. There was a large range of temperatures and strain rates used for the hydrogen charged tensile specimens, as was previously reported (6), and summarized in Table 4. Using the same analysis technique as for the fracture toughness specimen analysis, Eq.(2), the trapping energy was determined to be 18 kJ/mol. The result of the diffusive segregation analysis on the tensile behavior is shown in Figure 11. This is consistent with the 10-18 kJ/mol determined from the fracture toughness specimens.

The characteristic distance may now be determined for the IHE specimens in a similar manner to the HEE specimens. It is more complex to calculate the hydrogen profile for the IHE specimens. This is because hydrogen is both diffusing out of the specimen and segregating to the grain boundary during the test and both must be incorporated into the analysis. Eq. (6) will be used to calculate the matrix hydrogen profile. Then Eq. (7) will be used to determine the degree of segregation to the grain boundary. In Eq. (6) the boundary conditions are satisfied for the IHE case by setting C_s to zero and C_∞ to the dissolved hydrogen concentration from the hydrogen charging.

The hydrogen concentration profiles at the onset of cracking are displayed in Figure 12 for the IHE specimens. The same technique to estimate the characteristic distance for the IHE specimens as was used for the HEE specimens. This analysis results in a characteristic distance between 10 μm and 50 μm . A previous analysis of these data using a critical strain criterion also resulted in a characteristic fracture distance of $\sim 10\mu\text{m}$ (19). It was also shown in the previous work that achieving a critical strain was required to model the fracture and that critical stress did not correlate the data properly.

TABLE 4: TENSILE PROPERTIES OF HTH ALLOY X-750

Hydrogen Conc. (wppm)	Yield Stress (MPa)	True Fracture Stress (MPa)	Fracture Strain (%)	Reduction in Area (%)	% El loss	Strain rate (s ⁻¹)	Temp. (EC)
0	814	1482	23	24	0	3.33x10 ⁻⁴	-25
0	819	1375	21.2	22.1	0	3.33x10 ⁻⁴	25
0	809	1424	22	24	0	3.33x10 ⁻⁵	25
0	800	1385	21.5	--	0	3.33x10 ⁻⁴	65
0	766	1359	23	26	0	3.33x10 ⁻⁴	175
0	735	1378	30	--	0	3.33x10 ⁻⁴	285
5.7	801	1088	7.3	13.9	67	3.33x10 ⁻⁴	25
18	803	1009	4.3	11.5	80	3.33x10 ⁻⁴	25
52	806	942	2.5	8.4	89	3.33x10 ⁻⁵	25
65	837	1039	5.0	12	78	3.33x10 ⁻⁴	-25
65	837	997	3.5	7.4	85	5.00x10 ⁻⁵	-25
65	870	1170	15.5	--	29	3.33	25
65	817	1022	4.7	--	79	3.33x10 ⁻³	25
65	806	983	3.5	8.6	84	3.33x10 ⁻⁴	25
65	806	930	2.6	8.9	88	3.33x10 ⁻⁵	25
65	810	902	1.6	5.2	92	3.33x10 ⁻⁶	25
65	800	890	1.7	--	92	3.33x10 ⁻⁷	25
65	793	958	3.6	--	82	3.33x10 ⁻⁴	65
65	751	948	6.5	15	72	3.33x10 ⁻⁴	175
65	725	1145	13.5	--	55	3.33x10 ⁻⁴	285

Analysis of IHE + HEE Specimens

The maximum embrittlement for this testing was observed on the specimens that were hydrogen charged and then tested in the hydrogen environment. The higher degree of embrittlement may be due to the higher hydrogen concentration near the surface of the specimen. Since the specimen was hydrogen charged at a high temperature and then cooled at a constant pressure, the specimen was supersaturated with hydrogen on cooling. At 25°C, the specimen was in 21 MPa hydrogen that results in an equilibrium concentration of 11 ppm. The specimen was

precharged to 40 ppm in the material would be in equilibrium with 300 MPa hydrogen. Based on the previous analysis of the HEE specimens, Fig. 6, the fracture toughness of this specimen is between that for 21 MPa hydrogen ($23 \text{ MPa}\sqrt{\text{m}}$) and 300 MPa hydrogen ($17 \text{ MPa}\sqrt{\text{m}}$). This is consistent with some loss of hydrogen out of the crack, but still having more than what would be in the material at 21 MPa. This suggests that the embrittlement was due to the high hydrogen concentration near the surface, the same as was observed for the HEE specimens. This does not show a synergistic effect between IHE and HEE. Using Eq. (6), the hydrogen profile may be determined. The hydrogen profile for this specimen is shown in Figure 13. The profile is consistent with the explanation just given. This suggests that the synergistic effect of IHE + HEE is not due to a change in mechanism, but an increase in hydrogen concentration at the location of fracture.

Comparison of the IHE and HEE Specimen

The results of the IHE analysis and the HEE analysis show three key features. First, the grain boundary hydrogen concentration controls the degree of embrittlement of the nickel-base alloys. Second, the trapping energy is different for the two types of testing. Third, the characteristic fracture distance is closer to the crack tip for the HEE specimens than for the IHE specimens. The critical grain boundary hydrogen concentration is dependent on temperature or bulk hydrogen concentration only in as much as these parameters affect the grain boundary hydrogen concentration. A single continuous function could describe the embrittlement behavior over a large temperature range. Thus, the embrittlement observed at high temperature in these alloys is consistent with the high-energy grain boundary trapping sites. Further, the lack of embrittlement observed in pure Ni at the elevated temperatures is consistent with boundaries that are free from traps such as precipitates. This behavior is not specific to this alloy system, a similar behavior has been observed for steels. It was shown by Gao, Lu, and Wei that as the

pressure increased, thereby increasing the local hydrogen concentration, the maximum temperature for hydrogen induced crack growth increased (41). This again suggests that there is a critical hydrogen concentration and that as temperature is increased, trapping becomes less effective so more hydrogen is required for embrittlement. This work shows that if the pressure is increased to provide increased hydrogen or if the sites for fracture initiation are strong enough traps there is not a temperature at which hydrogen per se can not embrittle the material.

The strain profile ahead of the crack may be used to understand the increased embrittlement of the HEE specimen. Figure 14 shows the strain profile, calculated from the work of Rice and Johnson (32), for the IHE specimen and the HEE specimens that failed at $\sim 44 \text{ MPa}\sqrt{\text{m}}$. It may be seen that as the distance from the crack increases, the strain drops off precipitously. The strain at the characteristic distance for the HEE specimen is over 0.70 while for the IHE specimen the strain is less than 0.04.

The combination of the hydrogen profiles and the strain profile may now be used to explain the increased embrittlement in the hydrogen environment. When a specimen is tested in air, there are two competing diffusion events that affect the fracture behavior. At the test temperature, time is required for the diffusive segregation of hydrogen to the grain boundaries. Increasing the test time (decreasing the loading rate), results in increased grain boundary hydrogen. At the same time that hydrogen is diffusing to the grain boundary, it is diffusing out of the specimen at the crack tip. It is these two events that result in the location of fracture being inside the specimen and away from the region of maximum strain. In the HEE specimens the source of hydrogen is at the crack tip. This results in the maximum hydrogen concentration being in the high strain region and results in increased embrittlement. This may also be used to explain the lack of embrittlement observed at elevated temperatures for IHE fracture mechanics specimens.

The diffusion out of the surface is extremely rapid at elevated temperatures. Thus, the site for fracture initiation may have a very low hydrogen concentration and not show embrittlement. This further shows that when investigating hydrogen environment embrittlement, precharged specimens may lead to an incorrect prediction on the behavior of materials.

The difference in characteristic distance and hydrogen profiles may also be used to explain the difference in trapping energies between IHE and HEE specimens and lend insight into the mechanism of cracking. The micromechanism of hydrogen embrittlement cannot be positively identified in this work. Though, for the HEE specimens, since the fracture behavior is continuous over a large temperature range and may be correlated with the trapping energy on the grain boundary carbides, this would support a hypothesis that the carbide-matrix interface controls the fracture behavior. If the interface controls the fracture behavior, the embrittlement mechanism would then be consistent with hydrogen enhanced decohesion. Also, since the hydrogen is only diffusing a small distance into the metal when fracture occurs, there may not be a sufficient volume of material with hydrogen to affect the deformation behavior. This further supports the mechanism of HEE as being controlled by a reduction in the strength of the carbide matrix interface and not by hydrogen enhanced plasticity. The IHE trapping energy was near 18 kJ/mol. This binding energy is between that of the pure grain boundary (1) and that of the carbide (36). The work by Lassila and Birnbaum showed that the grain boundary trapping in Ni occurred over an area around the grain boundary. For Lassila and Birnbaum the hydrogen was throughout the specimen and the behavior was consistent with the Hydrogen Enhanced Local Plasticity (HELP) mechanism, it was proposed that the fracture was due to hydrogen enhanced local plasticity near the grain boundary. For the work described herein, the trapping energy observed for the IHE in this alloy was between the grain boundary energy and the carbide-matrix interface energy, the embrittlement may be due to both hydrogen enhanced plasticity in the

matrix near the grain boundary and hydrogen induced decohesion of the carbide matrix interface. For the IHE specimen, the local strain on continuum basis is low, and both the decohesion event and the void growth are important. The void growth would be strongly affected by hydrogen enhanced local plasticity.

VII. SUMMARY

It has been shown that much less hydrogen is required on a bulk level to embrittle Alloy X-750 in a hydrogen environment as compared to internal hydrogen. The degree of embrittlement of this alloy is controlled by the grain boundary hydrogen concentration irrespective of test temperature and bulk hydrogen concentration. In order to correlate the data, the characteristic fracture distance for the internal hydrogen case was different than the external case. For internal hydrogen, the hydrogen concentration at the peak hydrostatic stress location ($\sim 10\mu\text{m}$) described the behavior while for the hydrogen environment case the location was near $1\mu\text{m}$. Because the characteristic distance is less for the HEE case, the strain is much higher. Thus, for the same hydrogen levels in both specimens, the toughness in the HEE is much less than for IHE. It was also shown for specimens tested in a hydrogen environment that had internal hydrogen, the embrittlement was greater than in the environment alone. This again can be related to the increased grain boundary hydrogen concentration at the fracture location that induces decohesion and the high hydrogen over large distances that may enhance the plasticity.

VIII. ACKNOWLEDGMENTS

This work was supported under U. S. DOE Contract DE-AC-98PN38206 with Bettis Laboratory. The hydrogen gas testing was performed at Materials Engineering Associates and at Westinghouse Science and Technology Center. I would like to thank Dr. M. M. Hall for the many fruitful discussions on developing the correlation between grain boundary hydrogen and fracture. I would like to thank Prof. Petros Sofronis for the continuing discussions on modeling the micromechanics of hydrogen embrittlement. I would also like to acknowledge Anthony W. Thompson who was my thesis advisor during much of the testing on IHE and provided many helpful discussions on that work.

REFERENCES

- (1) D. H. Lassila and H. K. Birnbaum, *Acta Metall.*, Vol. 36, No. 10, 1988, pp. 2821-2825
- (2) D. M. Symons, "The Hydrogen Embrittlement of Ni-Cr-Fe Alloys," *Metallurgical and Materials Transactions*, March 1997, pp. 655-663.
- (3) C. M. Brown and W. J. Mills, "Fracture Toughness, Tensile, and Stress Corrosion Cracking Properties of Alloy 600, Alloy 690, and Their Welds in Water," Paper #90, CORROSION 96, NACE
- (4) D. M. Symons, "The Effect of Carbide Precipitation on the Hydrogen Enhanced Fracture Behavior of Alloy 690," *Metallurgical and Materials Transactions*, April 1998, pp. 1265-1277.
- (5) R. J. Walter, R. P. Jewett, W. T. Chandler, "On the Mechanism of Hydrogen-environment Embrittlement of Iron- and Nickel-base Alloys, *Materials Science and Engineering*, 5, 1969-70, pp. 98-110
- (6) D. M. Symons and A. W. Thompson, *Met. Trans. A*, 27A, 1996, pp. 101-110.
- (7) W. J. Mills, M. R. Lebo, J. J. Kearns, "Hydrogen Embrittlement, Grain Boundary Segregation, and Stress Corrosion Cracking of Alloy X-750 in Low and High Temperature Water, *Met. and Mat. Transactions* in press, (available through OSTI WAPD-T-3149)
- (8) W. W. Gerberich, T. Livne, X. Chen, "A Transient Model for Sub-Critical Cracking in BCC Alloys," in Modeling Environmental Effects on Crack Growth Processes, TMS, Warrendale, PA. 1986, pp. 243-257
- (9) W. W. Gerberich, J. Garry, J. F. Lessar, *Effect of Hydrogen on Behavior of Materials*, ed. A. W. Thompson and I. M. Bernstein, TMS, Warrendale, PA, 1975, pp. 70-82
- (10) L. G. Fritzemeier, R. J. Walter, A. P. Meisels, and R. P. Jewett, *Hydrogen Effects on Material Behavior*, ed. N. R. Moody and A. W. Thompson, TMS, Warrendale, PA, 1990, pp. 941-954
- (11) N. R. Moody, M. W. Perra, and S. L. Robinson, "Hydrogen Pressure and Crack Tip Stress Effects on Slow Crack Growth Thresholds in an Iron-Based Superalloy, *Scripta Met.*, 22, 1988, pp. 1261-1266
- (12) H. H. Johnson, "On Hydrogen Brittleness in High Strength Steels," in *Fundamental Aspects of Stress Corrosion Cracking*, Eds. R. W. Staehle, Sept. 1967, NACE, p. 439
- (13) M. M. Hall, D. M. Symons, and J. J. Kearns, "Strain Energy Density - Distance Criterion for the Initiation of Hydrogen-Induced Cracking of Alloy X-750," in *Parkins Symposium on Fundamental Aspects of Stress Corrosion Cracking*, TMS, Warrendale, PA, 1992, pp. 231-244.

- (14) C. A. Grove and L. D. Petzold, Corrosion of Nickel-Base Alloys, (ASM, Mars, PA, 1985) p. 165-180
- (15) M. M. Hall and D. M. Symons, "Strain Energy Density-Distance Criterion for the Initiation of Stress Corrosion Cracking of Alloy X-750," *ASTM STP 1298: Effects of the Environment on the Initiation of Cracks*, eds. W. A. Van Der Sluys, R. S. Piascik, R. Zawierucha, ASTM, West Conshohocken, PA, 1997, pp. 167-181.
- (16) O. H. Kriege and J. M. Baris, *Trans. ASM*, 62, 1969, p. 195
- (17) E. L. Raymond, "Effect of Grain-Boundary Denudation of Gamma Prime on Notch-Rupture Ductility of Inconel Nickel-Chromium Alloys X-750 and 718", *Trans. Metall. Society of AIME*, Vol. 239, pp 1415-1422
- (18) M. G. Burke, T. R. Mager, I. L. W. Wilson, "The Effect of Cooling Rate on Precipitate Morphology in Alloy X-750" in *Proceedings of the Fifth International Symposium on Environmental Degradation of Materials in Nuclear Power Systems-Water Reactors*, August 25-29, 1991, pp. 287-293
- (19) D. M. Symons and A. W. Thompson, "The Effect of Hydrogen on the Fracture Toughness of Alloy X-750," *Metallurgical and Materials Transactions*, March 1997, pp.817-824.
- (20) A. W. Thompson and I. M. Bernstein, in Advances in Corrosion Science and Technology, Vol. 7, Eds. M. G. Fontana and R. W. Staehle, Plenum Publishing, NY, 1980
- (21) H. G. Nelson, in *Treatise on Materials Science and Technology*, Vol. 25, Eds. C. L. Briant and S. K. Banerji, Academic Press, New York, NY, 1983
- (22) M. R. Louthan, G. R. Caskey, J. A. Donovan, D. E. Rawl, *Mat. Sci. Eng.*, 10, 1972, 357-368
- (23) Hydrogen Effects in Materials, Eds. Anthony W. Thompson and Neville R. Moody, TMS, Warrendale, PA, 1994
- (24) Hydrogen Effects on Material Behavior, Eds. Neville R. Moody and Anthony W. Thompson, TMS, Warrendale, PA, 1989
- (25) D. M. Symons, "The Effect of Hydrogen on the Fracture Toughness of Alloy X-750 at Elevated Temperatures," *J. Nuc. Mat.*, in press
- (26) D. M. Symons, Ph.D. Dissertation, Carnegie Mellon University, Pittsburgh, PA, November 1994
- (27) R. A. Oriani, The Diffusion and Trapping of Hydrogen in Steel, *Acta Met*, 1970, 18, 147-157
- (28) D. McLean, *Grain Boundaries in Metals*, Oxford Univ. Press, 1957
- (29) J. C. M. Li, R. A. Oriani, L. S. Darken, *Z. Phys. Chem. (N.F.)*49, 271-290, 1966
- (30) B. Baranowski, S. Majchrzak, T. B. Flanagan, *J. Phys. F: Metal Physics*, 1, 1971, p. 258-261

- (31) K. N. Akhurst and T. J. Baker, *Met. Trans. A*, 12A, 1981, p. 1059-1070
- (32) J. R. Rice and M. A. Johnson, *Inelastic Behavior of Solids*, M. F. Kannin, W. G. Adler, A. R. Rosenfield, and R. I. Jaffee, eds., McGraw Hill, New York, 1970, p. 641-671
- (33) J. W. Hutchinson, *J. Mech Phys Solids*, Vol. 16, 1968, pp.13-31
- (34) J. R. Rice and G. R. Rosengren, *J. Mech Phys Solids*, Vol. 16, 1968, pp.1-12
- (35) G. J. Thomas, in Hydrogen Effects in Metals, Eds. A. W. Thompson and I. M. Bernstein, (TMS, New York, 1980), p. 77
- (36) G. A. Young and J. R. Scully, *Scripta Mat*, Vol. 36, 1997, pp. 713-719
- (37) P. Shewmon, Y. L. Shen, C. H. Shen, M. Meshii, *Acta Met*, 1989, pp. 1913-1921
- (38) J. Yao, S. A. Meguid, J. R. Cahoon, *Met. Trans.*, 24A, 1993, pp. 105-112
- (39) J. Crank, *Mathematics of Diffusion*, Oxford at the Clarendon Press, 1956.
- (40) P. Doig and G.T. Jones, *Met. Trans*, 8A, 1977, pp. 1993-1998.
- (41) M. Gao, M. Lu, and R. P. Wei, *Met. Trans*, 15A, 1984, pp. 735-746

LIST OF FIGURES

- Figure 1: a) General microstructure and b) Grain boundary $M_{23}C_6$ precipitates
- Figure 2: Fracture morphology of noncharged Alloy X-750, a-c) Intergranular fracture morphology d-f) Transgranular fracture morphology
- Figure 3: Fracture morphology of internally charged Alloy X-750, a-c) Increasing magnification of intergranular fracture
- Figure 4: Fracture morphology of Alloy X-750 tested in 0.14 MPa hydrogen gas, a-c) Increasing magnification of intergranular fracture
- Figure 5: Fracture morphology of Alloy X-750 tested in 3.4 MPa hydrogen gas, a-c) Increasing magnification of intergranular fracture
- Figure 6: The effect of hydrogen pressure on the fracture toughness of Alloy X-750
- Figure 7: The effect of hydrogen concentration on the IHE and HEE of Alloy X-750
- Figure 8: The effect of grain boundary hydrogen on the fracture toughness of Alloy X-750 tested in hydrogen gas
- Figure 9: The hydrogen concentration profiles for the HEE specimens at the onset of fracture
- Figure 10: The effect of grain boundary hydrogen on the fracture toughness of hydrogen precharged Alloy X-750
- Figure 11: The effect of grain boundary hydrogen on the tensile ductility of Alloy X-750
- Figure 12: The hydrogen concentration profiles of the IHE specimens at the onset of fracture
- Figure 13: The hydrogen concentration profile for the hydrogen precharged specimen tested in hydrogen gas at the onset of fracture
- Figure 14: The strain profile ahead of a crack for the IHE and HEE specimens that failed at 44 MPa $\sqrt{m}$

Figure 1: a) General microstructure and b) grain boundary M_{23}C_6 carbide precipitate

Figure 2: Fracture morphology of noncharged alloy X-750, a-c) Intergranular fracture morphology

Figure 2: Fracture morphology of noncharged alloy X-750, d-f) Transgranular fracture morphology

Figure 3: Fracture morphology of internally charged Alloy X-750, a-c) Increasing magnification of intergranular fracture

Figure 4: Fracture morphology of Alloy X-750 specimen tested in 0.14 MPa hydrogen gas, a-c) Increasing magnification of intergranular fracture

Figure 5: Fracture morphology of Alloy X-750 specimen tested in 3.4 MPa hydrogen gas,
a-c) Increasing magnification of intergranular fracture

Figure 6: The effect of hydrogen pressure on the HEE fracture toughness of Alloy X-750

Figure 7: The effect of bulk hydrogen concentration on the IHE and HEE of Alloy X-750 (for HEE specimens bulk concentration was calculated using Sievert's Law based on hydrogen pressure of test)

Figure 8: The effect of grain boundary hydrogen on the fracture toughness of Alloy X-750 tested in hydrogen gas (HEE)

Figure 9: The hydrogen concentration profiles for the HEE specimens calculated at the time required for the onset of fracture

Figure 10: The effect of grain boundary hydrogen on the fracture toughness of hydrogen precharged Alloy X-750 (IHE)

Figure 11: The effect of grain boundary hydrogen on the tensile ductility of Alloy X-750

Figure 12: The hydrogen concentration profiles of the IHE specimens calculated at the time required for the onset of fracture

Figure 13: The hydrogen concentration profile for the hydrogen precharged specimen tested in hydrogen gas calculated at the time required for the onset of fracture

Figure 14: The strain profile ahead of a crack for the IHE and HEE specimens that failed at $44 \text{ MPa}\sqrt{\text{m}}$