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TEMPERATURE AQUEOUS ENVIRONMENT

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ABSTRACT

Fatigue crack propagation (FCP) rates for 304 stainless steel (304SS) were determined in 24°C and 288°C air and 288°C water using double-edged notch (DEN) specimens of 304 stainless steel (304 SS). Tests performed at matched loading conditions in air and water at 288°C with 20-60 cc H₂/kg H₂O provided a direct comparison of the relative crack growth rates in air and water over a wide range of crack growth rates. The DEN crack extension ranged from short cracks (0.03-0.25 mm) to long cracks up to 4.06 mm, which are consistent with conventional deep crack tests. Crack growth rates of 304 SS in water were about 12 times the air rate. This 12X environmental enhancement persisted to crack extensions up to 4.06 mm, far outside the range associated with short crack effects. The large environmental degradation for 304 SS crack growth is consistent with the strong reduction of fatigue life in high hydrogen water. Further, very similar environmental effects were reported in fatigue crack growth tests in hydrogen water chemistry (HWC). Most literature data in high hydrogen water show only a mild environmental effect for 304 SS, of order 2.5 times air or less, but the tests were predominantly performed at high cyclic stress intensity or equivalently, high air rates. The environmental effect in low oxygen environments at low stress intensity depends strongly on both the stress ratio, R, and the load rise time, T_r, as recently reported for austenitic stainless steel in BWR water. Fractography was performed for both tests in air and water. At 288°C in water, the fracture surfaces were crisply faceted with a crystallographic appearance, and showed striations under high magnification. The cleavage-like facets on the fracture surfaces suggest that hydrogen embrittlement is the primary cause of accelerated cracking.

II. INTRODUCTION

Fatigue crack propagation data for Type 304 stainless steel (304 SS) were obtained in air and an elevated temperature aqueous environment. The data were developed from instrumented fatigue tests on double-edged notched (DEN) fatigue specimens with two different notch root radii of 0.38 and 1.52 mm (Wire, et al. 1999). The fatigue tests were primarily designed to determine the effect of notch radius on fatigue life but also provide fatigue crack growth data. A single crack condition analyzed suggested that a large environmental effect occurred (Wire, et al., 1999), and the detailed analysis below shows the trend was widely supported by all the tests.

III. EXPERIMENTAL

The DEN specimens (Figure 1) were machined from a 127 mm diameter bar forging with the specimen orientation ASTM L-C. The chemical composition of the 304 SS material is provided in Table 1 and nominal mechanical properties are listed in Table 2. The microstructure for the test material, which is shown in Figure 2, consists of non-sensitized grains with a grain size of ASTM 2. There was evidence of annealing twins and delta ferrite stringers aligned in the primary working direction; the ferrite level was 0.4 percent. Although delta ferrite stringers were present throughout this heat, they did not appear to influence the fatigue behavior because they were normal to the cracking direction as specimens had an L-C orientation.

Table 1 Chemical Composition of Test Material

C	Cr	Ni	Mo	Mn	P	Si	N	S
0.04	18.4	8.85	0.29	1.49	0.03	0.36	0.04	0.001

A. Testing

Load controlled cyclic fatigue tests were performed in air at room temperature and 288°C and in deaerated 288°C water containing 20-60 cc H₂/kg H₂O. The room temperature pH was in the range 10.1-10.3. The electric potential drop (EPD) technique with current reversal was used to monitor initiation and growth. EPD measurements were calibrated against optical crack growth measurements.

An instrumented DEN test specimen is shown in Figure 1. The double-edge notched uniaxial specimen provides an advantage over compact tension specimens in that it can be tested in both tension-tension and tension-compression loading conditions. Tests were performed under load control in fully reversed (R=-1) and tension loading (R=0). Alignment was achieved by manually adjusting the pull rod while monitoring strain gages attached to the specimen (Figure 1). Once a satisfactory alignment was achieved, the strain gages were removed and the EPD leads were attached. For the tests in water, the assembly was then enclosed in an autoclave, which was filled with water and heated to 288°C. The specimen was cycled until crack initiation was detected, based on the electrical potential drop reading corresponding to a crack growth of 0.13 mm. Following an interim visual inspection, cycling was continued to obtain crack extension data.

Table 2 Mechanical Properties of Test Material

Hardness (BHN)	Yield Stress (MPa)	Ultimate Stress (MPa)	Elongation (%)	Reduction of Area (%)
143	288	546	67.2	80.4

EPD measurements were verified by performing crack extension measurements on the specimen sides with a toolmaker's microscope. As shown in Figure 3, the EPD results were in excellent overall agreement with the visual measurements for almost all the test specimens. In a few cases where the difference was substantial, the EPD measurements were scaled to match the visual measurements. Selected specimens were examined for tunneling after separation by fatigue. The average of the two edges was only about 7 % less than the mean crack extension including the center and edges. Since the edges were about 4% less than the EPD-inferred crack extension values (Figure 3), no corrections were made for tunneling.

B. Raw Data Analysis

The raw output from the experiment is the crack depth as assessed by EPD versus the cycles. The results are shown in Figure 4 for a typical test in water, specimen 1T604. As can be seen, the EPD measurements are typically scattered locally by about ± 0.01 mm. This appears to be random short-term variation in the readings. However, a short-term random effect can be eliminated by taking a local average over a number of data points centered about a particular value of N. Eleven point averages were sufficient to overcome the local data scatter as shown in Figure 4, and 21 point averages provided

additional smoothing. Averaging over a higher number of points might mask data trends or thresholds. Therefore, averages were typically taken over just enough data points to avoid apparent random short-term variability, but retain the sensitivity to potential thresholds or rate changes.

The crack growth rate da/dN was calculated using the secant method applied to the average extension curves. A variety of alternate methods were tried, including statistically fitting curves in segments, by either linear or higher order polynomials. The da/dN results were essentially the same, so the secant method was chosen because of its simplicity. For this data set using EPD with high sensitivity and a high density of recorded points, crack growth rates were obtained at extensions as low as 0.013 mm in order to detect any short crack effects. The rates at very low crack extension were examined for possible short crack effects.

Rate data based on a large crack extension is considered to be more appropriate for comparison to conventional fatigue crack propagation data for very deep cracks. Only crack propagation rates calculated over larger increments of crack extension were used for such comparisons.

C. Stress Intensity Factor Calculations

For shallow cracks, where $L < \rho$, the stress intensity factor solution developed by Schijve (1982) for a crack emanating from an edge notch was used to compute K:

The specific expressions used based on the Schijve treatment of the edge notch are:

$$K = F_c \Delta S (\pi(D+L))^{0.5} \text{ for } L/\rho \leq 1 \quad (1a)$$

$$F_c = K_{tg} C (L / (D + L))^{0.5} \quad (1b)$$

$$C = 1.1215 - 3.21 (L / \rho) + 5.16 (L / \rho)^{1.5} - 3.73 (L / \rho)^2 + 1.14 (L / \rho)^{2.5} \quad (1c)$$

where:

L = crack depth (ie, the distance from the notch root radius to the notch tip)

S = gross section stress

K_{tg} = gross section stress concentration factor (4.8 and 8.8 for $\rho = 1.52$ and 0.38 mm)

D = notch depth

ρ = notch root radius

When the crack depth exceeded the notch root radius, the conventional stress intensity factor solution developed by Tada, et al. (2000) for DEN specimens, which is based on the total crack length ($a = D + L$), was used to calculate K. The transition between the two formulations was made at $L = \rho$. It is noted that an independent K solution for a double-edge notched plate (Yamamoto, 1974) provided results within 4% for shallow and intermediate crack lengths over the range of the present tests.

D. Fractographic Examinations

Broken specimen halves were examined on a scanning electron microscope (SEM) to characterize the fatigue fracture surface morphology. The crack length associated with each fractograph was determined so fracture surface features could be correlated with crack growth rates and applied ΔK levels. Relative amounts of α' martensite on fracture faces were estimated using a commercial ferrite measurement instrument (Feritscope® MP3C). While fracture surface roughness and the presence of only a thin layer of martensite precluded precise measurements, relative amounts of martensite were readily determined.

IV. TEST RESULTS

A. Short Crack Effects

Before examining environmental effects, it is appropriate to evaluate the cracking behavior for short versus long cracks. Crack growth rates for short cracks can be much larger than long crack data due to differences in crack closure (Newman, 1992). Crack closure can occur in the crack wake before reaching the minimum load, reducing the effective stress intensity below the calculated amount based on the full load range. However, short cracks have little or no crack wake, and closure is subsequently reduced. To examine for such closure effects, the growth rates in air for the DEN at $R=-1$ were compared directly to growth rates from conventional deep crack tests as a function of crack depth. The crack propagation rates for deep cracks were calculated from the following equation based on James and Jones, 1985:

$$da/dN = 1.40 \times 10^{-9} \Delta K^{3.57}, \text{ mm/cycle, MPa}\sqrt{\text{m}}, \text{ air, } 288^\circ\text{C}, R=0 \quad (2)$$

The ratio of the DEN rates to rates for long cracks from Eqn 2 are shown in Figure 5. The positive portion of the loading was used to calculate stress intensity for the DEN. The Figure shows that the short crack growth rates can exceed the deep crack rates by a factor of thirty, but that the ratio quickly approaches a stable value as the crack depth becomes significant compared to the notch radius. The value of the ratio at larger crack depths ranged from approximately one to four for the tests shown, implying that the tensile portion of the loading is largely responsible for the crack propagation. For the particular notch depths studied here, short crack effects are only important below a/p of order 0.2. For the values $p=0.38$ and 1.52 mm, this corresponds to only 0.08 and 0.30 mm, respectively. Therefore, shallow crack effects can produce an order of magnitude increase in crack growth rates under fully reversed loading conditions, but this acceleration is confined to very small crack extensions, on the order of 0.3 mm. For longer cracks, conventional test data for deeply cracked specimens can be used to predict cracking behavior.

The increased rates observed for short cracks near notches is consistent with increased effective stress intensity, as reviewed in depth by Lalor, Sehitoglu, and McClung (1986). They observed that the crack opening stress increased rapidly with increasing crack depth and leveled out for crack depths

above approximately 40% of the notch radius. They were able to explain the observed crack opening stresses very well on the basis of finite element analysis of crack closure effects.

B Environmental Effects by Comparison to Controls

The effect of environment on fatigue crack growth can be seen by directly comparing the data from 288°C air and water tests, as controls were run in air at the same or very similar loading conditions to the tests in water. This allows a direct assessment of environmental effects down to the smallest detectable crack extensions, while avoiding the need for an explicit treatment of short crack effects. Rather, the da/dN values are compared directly in air and water at the same crack extension and cyclic stress. This assures that crack driving forces are the same, without having to explicitly calculate them.

The results of the tests on 304 SS are shown in Figure 6. The plots show conclusively that the crack growth rates in water are much enhanced over rates in air. The ratio of crack growth rates in water over air is called the environmental ratio (ER), for convenience. At a stress amplitude of approximately 69 MPa and lowest frequency tested of 0.0033 Hz, the ER is of order 15 (Fig 6a). The large ER in water observed in Figure 6a persisted to the end of the test, where the crack extension was 4.1mm. Hence, the environmental effects continue to crack depths of engineering significance, and are not just short crack effects. Figure 6 shows that the ER was large at the smallest detectable crack extensions of about 0.025 mm. Hence, the increases in crack growth rate explain the reductions in fatigue life reported in the literature.

Several other trends are worthy of note. Higher frequency led to a smaller ER of 10X, as shown by comparing Figure 6b and Figure 6a. The apparent increase in ER with decreasing frequency is consistent with the reduction of fatigue life at lower strain rate in the literature (Chopra and Smith, 1998). The ER for a higher stress amplitude (Figure 6c) is only about a factor of 8 compared to 15X in Figure 6a at the same low frequency, indicating a possible reduction in ER at high stress amplitude with the resulting higher crack growth rates. The ER at $R=0$ is also smaller, as shown in Figure 6d. This may be further evidence of an effect of higher effective loading provided by $R=0$ compared to $R=-1$, which has a compressive half cycle. Also, the ER in Figure 6c&d decreases at the largest crack extensions, which correspond to the highest stress intensity. Such an effect is consistent with the general notion that at high loading, mechanical effects will dominate.

C Environmental Effects Using Time-based Plots and Comparison to Literature

From a fundamental point of view, the crack tip strain rate is the appropriate crack driving parameter to correlate environmentally assisted crack propagation rate data (Scott, 1988). However, a unique method of crack-tip strain-rate calculation could not be established and variability was over a factor of ten between various models. Shoji, et al (1981) suggested using the time-based $da/dt|_a = da/dN|_a / T_r$ rate in air as a practical correlating parameter representing crack tip

strain-rate for low alloy steel fatigue crack growth data. This variable was subsequently used successfully to correlate environmental effects on low alloy steels in water.

The time-based environmental crack growth rate is defined as:

$$da/dt|_e = (da/dN)|_e / T_r \text{, where } T_r \text{ is the load rise time (s).} \quad (3)$$

Eqn. 3 is appropriate for fatigue crack growth tests with continuous cycling, which produce a time-independent rate. In the event that stress corrosion cracking or other time-dependent behavior is noted, the real time rate would be more appropriate.

The strong environmental effects observed on 304 SS are correlated reasonably well within scatter by utilizing a time-based plot, as shown in Figure 7. The 304 SS data show a clear increase in crack growth rates relative to those in air at low air rates, and are consistent with the degradation of fatigue life of up to 15X assessed by Chopra and Smith (1998) and 13X assessed by Leax (1999). As noted above, the large ER did not diminish in one test up to a crack depth of 4.1 mm. This depth is greater than associated with "short crack" effects and is significant from an engineering standpoint. Subsequent tests on conventional compact tension specimens at this laboratory verified the environmental effect continues unabated to a crack growth of 15 mm as reported by Evans and Wire (2002).

Tests were also performed under similar conditions on Inconel Alloy 600 specimens in air and water. The Alloy 600 specimens were machined from a 64mm diameter forging of heat Number 63985. This material was annealed at 871°C. The tests provided very limited FCP data because initiation in water only occurred at the highest stress amplitude conditions of $S_a = 124$ MPa. The limited data in water for Alloy 600 are very close to the air rates, as shown in Figure 8. Although there is overlap in the data, it appears that crack growth rates for Alloy 600 in water are about twice those in air.

However, it must be noted that the 304SS showed reduced environmental effects at the highest stress amplitude of 93 MPa, and the Inconel 600 tests at 124MPa may not be in a range conducive to large environmental effects.

A review of fatigue crack propagation of austenitic stainless steels was performed recently by Shack and Kassner (1994). Some data from surface crack tests are available from tests performed in low oxygen "hydrogen water chemistry" (HWC) environments, as shown in Figure 9 (Prater, et al. 1986). HWC is BWR water chemistry with hydrogen added to reduce oxygen. The filled symbols from literature tests on surface crack specimens tested in low oxygen water confirm that the large environmental effects shown here have been observed in previous tests (Prater, et al, 1985). Overall, the surface crack tests from the literature average about 14 times the air rate, very similar to DEN data. The fact that the material was sensitized stainless steel does not appear to be

important, as the cracking was transgranular in fatigue. Gordon, et al. (1985) indicated that the fatigue crack growth rates in HWC water were the same for solution annealed and sensitized 304 SS.

A comparison of crack growth data trends from DEN tests and selected conventional compact tension test data in HWC is provided in Figure 10. The data by Ljunberg (1989) show much higher than 12X enhancement, but the air rate is almost 100 times lower than the DEN data in Figure 7. These results provide further support for the observation that environmental effects tend to increase in the lower stress intensity regime where crack growth rates in air are reduced.

The tests by Andresen and Campbell (1989) show evidence for a transition to reduced environmental effects at high air rates, and more limited data by Gordon et al (1985) are consistent with such an effect. It is noteworthy that the DEN data agree qualitatively with HWC data in Figure 9, including the evidence of a transition to substantially lower environmental effects at air rates above 10^{-6} mm/s.

The hydrogen level for the HWC test data in Figure 9&10 is 150 ppb or less, much less than several ppm in the current tests. HWC environments are low in oxygen, but the hydrogen level of 150 ppb is much lower than the several ppm in the present DEN tests and in PWR water. The corrosion potential in HWC can be higher by about 0.3V SHE than that in water with higher hydrogen used in the present tests, according to Gilman (1988). Therefore, the HWC results should not be assumed to be identical to results in higher hydrogen water. Evans and Wire (2002) performed a series of tests on a 1.9 CT specimen (thickness 0.95 in) of the same heat and water conditions used in the DEN tests. The CT tests showed that large environmental effects occurred in conventional, deeply cracked compact tension specimens with high hydrogen levels and the attendant lower potential.

V. FRACTURE MECHANICS ASSESSMENT

The cyclic stress intensity was calculated from Eqn 1 and the Tada (2000) expression, including the full cyclic stress range. Crack extensions increments of 0.12 mm or larger were employed. Results from the DEN tests and the compact tension tests by Evans and Wire (2002) are shown in Figure 11. The tests shown in Figure 11 indicate a strong effect of both rise time and R.

The environmental effects can be rationalized in terms of a combined mean stress effect on closure or R ratio and a rise time or frequency effect, consistent with the literature. Bamford (1979) noted larger environmental effects at higher R ratios. He incorporated an effective $\Delta K_{eff} = K_{max}(1-R)^{0.5}$, which shifted the high R data more in line with low R data. Cullen (1985) reported strongly increased FCP rates for cast stainless steel at higher R in PWR water. The data by Bernard, et al, (1979) on Z3 CND17-12, similar to 316 SS, showed a clear rise time effect. Recently, a correlation for FCP of austenitic

stainless steels in BWR water was developed by Itatani, et. al (2001). The correlation was of the form:

$$da/dN = A (\Delta K)^m T_r^n / (1-R)^p \text{ with } m=3.0, n=0.5, \text{ and } p=2.12 \quad (5)$$

This formulation indicates the strong role of rise time and R-ratio, consistent with results described above in low oxygen water.

Figure 12 shows that the crack growth rates in Figure 11 can be reasonably well normalized by the T_r and R-ratio dependence of $T_r^{0.31}$ and $1/(1-R)^{1.1}$, respectively. Hence, the form of the correlation developed by Itatani, et al. for tension-tension appears to be promising for tension-compression. While the parameter values chosen normalize this limited data set well, much more data would be required to obtain a definitive correlation. It is of interest to apply this same normalization to data from compact tension tests at high R by Evans and Wire (2002) and at low R by Bernard, et al. in PWR water (1979). Both data sets include long rise times (450-500s), and should therefore exhibit any environmental effects. As can be seen in Figure 13, the data sets appear to be consistent after normalization, suggesting that ER decreases with increasing stress intensity. This same trend was noted above for high stress amplitude DEN tests in Figure 6c and Andresen data in Figure 10. This is consistent with the overall trend for environmental effects to be mild at high air rates, as noted by Shack and Kassner (1994).

VI. CHARACTERIZATION OF FATIGUE CRACK PROPAGATION MECHANISMS

Fracture surface features for specimens tested in air and water were evaluated to correlate operative cracking mechanisms with environmental cracking behavior. The fracture surface appearance for specimens tested in room temperature air was found to be dependent on loading conditions. A faceted morphology (Figure 14a) was observed at crack growth rates less than 1×10^{-4} mm/cycle, vast fields of well-defined striations (Figure 14b) were generated at crack growth rates between 1×10^{-4} and 1×10^{-3} mm/cycle, and a combination of fatigue striations and dimples (Figure 14c) was observed above 1×10^{-3} mm/cycle. The nature of striated fracture surfaces in the intermediate and high crack growth rate regimes resembles that typically observed in fcc materials, but the facets formed at low crack growth rates are rather unique, as discussed below. Evidence of rubbing and fretting (Figure 15) due to repeated contact between mating fracture surfaces was observed in specimens tested under fully reversed cyclic loading conditions.

Facets generated at low crack growth rates had an irregular appearance that was associated with a quasi-cleavage mechanism. Because 304 SS is a metastable alloy at room temperature, the material directly ahead of an advancing crack undergoes a strain-induced transformation to α' martensite. Therefore, cracks must propagate through the martensite, which results in a quasi-cleavage morphology that resembles the quasi-cleavage fracture surface appearance in martensitic

steels. Ferritescop measurements showed that all fatigue fracture surfaces generated at room temperature contained α' martensite, with the amount of martensite increasing at higher stress intensity factor levels, due to larger plastic zone sizes.

The nature of quasi-cleavage facets located immediately adjacent to a notch was identical to facets located well away from a notch, as long as crack growth rates were less than 1×10^{-4} mm/cycle. This indicates that the same cracking mechanisms were operative for shallow and deep cracks.

The morphology of the quasi-cleavage facets was consistent with the fracture surface appearance for 304 SS (Gao, et al. 1992) and high purity Fe-18Cr-12Ni SS (Wei, et al. 1993) tested in room temperature air, 3.5% NaCl solutions and hydrogen. Strain-induced α' martensite formed ahead of fatigue cracks in both alloys, which caused a quasi-cleavage mechanism.

To better understand the nature of the faceted growth mechanism in stainless steels, fracture surfaces for 304 SS were compared to those for 316 SS tested under comparable conditions. Unlike 304 SS, 316 SS fatigue tested in room temperature air (Mills, 1988) exhibited more conventional, cleavage-like facets, as shown in Figure 16. Apparently, this is because 316 SS is a more stable alloy due to its higher nickel content, so α' martensite transformation does not occur at room temperature. As a result, 316 SS exhibits classic, cleavage-like faceted growth as cracks propagate through stable austenite, whereas 304 SS exhibits quasi-cleavage facets as cracks propagate through transformed α' martensite.

In the low crack growth rate regime, 304 SS also exhibited localized cracking along annealing twin boundaries, but no evidence of intergranular cracking. Localized separation along favorably oriented twin boundaries produced flat, featureless facets that appear as dark islands (see arrow in Figure 14a) surrounded by quasi-cleavage facets. Gao, et al. (1992) and Wei, et al. (1993) also reported evidence of twin boundary cracking in 304 SS and high purity Fe-18Cr-12Ni SS.

Facets formed in 24° and 288°C air had a different morphology, as facets generated at high temperature exhibited a cleavage-like appearance. Comparison of Figures 17a and 17b shows that the high temperature facets had more of a crystallographic nature with some evidence of river patterns, in contrast with the irregular facets generated in room temperature air. The lack of quasi-cleavage facets indicates that 288°C is above the critical temperature where cold working induces a martensite transformation (ie, MD temperature). Based on the composition of 304 SS, the MD temperature associated with 30% cold work is on the order of 100°C (Lacombe, 1993). Indeed, Ferritescop measurements showed no detectable α' -martensite on fatigue fracture surfaces generated at 288°C.

At crack growth rates slightly above 1×10^{-4} mm/cycle, facets formed in 288°C air became rather poorly defined and parallel fracture markings associated with slip offsets were often

superimposed on them. The transition to poorly defined facets is believed to be associated with a transition from heterogeneous-to-homogeneous slip. In the low crack growth rate regime, local separation along planar slip bands results in the formation of crystallographic facets, but as crack growth rates increase, the deformation mode becomes more homogeneous so facets are not as well defined. At progressively higher crack growth rates, fracture surfaces are expected to take on a striated appearance; however, testing in 288°C air was limited to crack growth rates below 1×10^{-4} mm/cycle, so cracking mechanisms at higher crack growth rates were not evaluated.

Fracture surfaces generated in 288°C water were remarkably different than those generated in air. Facets formed in water had a crystallographic appearance with well-defined river patterns, as shown in Figure 17c. Stereo fractography revealed that the facets were highly angular, with many facets steeply inclined toward the loading direction. These sharp, cleavage-like facets formed immediately adjacent to machined notches and well away from the notches, indicating that the same faceted growth mechanism was operative for shallow and long cracks. Moreover, well-defined crystallographic facets persisted over the entire range of crack growth rates generated in this program, including crack growth rates as high as 8×10^{-4} mm/cycle where fracture surfaces generated in air exhibited poorly defined facets and vast fields of fatigue striations. There was no evidence of either intergranular cracking or annealing twin boundary cracking in 288°C water.

Although fracture surfaces generated in 288°C water exhibited well-defined cleavage-like facets, high magnification of facet faces revealed the presence of fatigue striations (Figure 18). At crack growth rates between 1×10^{-4} and 3×10^{-4} mm/cycle, parallel fracture markings on the facets were very straight, but their spacing was identical to macroscopic crack growth rates indicating that they were fatigue striations. At growth rates above 3×10^{-4} mm/cycle, striations had a ductile or wavy appearance, as shown in Figure 18c.

Facet and striation orientations on fracture surfaces generated in 288°C water revealed that local cracking directions were often very different from the overall cracking direction. Although facets were usually aligned in the cracking direction, a significant number of them were aligned normal to the macroscopic cracking direction (Figures 17c and 18a). Likewise, most striations were oriented normal to overall cracking direction, but in some regions striations had different orientations and in some cases were even parallel to the macroscopic cracking direction. These observations indicate that crack advance in water involved a very uneven process, as cracking first occurred in the most susceptible regions, which left large ligaments in the wake of the advancing crack front. As the overall crack continued to extend, local stress intensities within the more resistant ligaments increased to the point where cracking reinitiated and propagated across the ligaments. As a result, local cracking directions within these ligaments were often normal to the overall cracking direction. The rapid crack advance in the more susceptible regions is

believed to be a significant contributor to the environmental acceleration observed in high temperature water. Specifically, this rapid cracking not only increased the overall crack length, it increased local stress intensities in the wake of the crack and provided alternate paths for reinitiating local cracks along the more resistant ligaments.

The role of active path dissolution versus hydrogen embrittlement in causing accelerated cracking of stainless steel in high temperature water remains an issue. It is difficult to resolve this issue due to the coupled nature of these processes as electrochemical reactions in the vicinity of the crack tip involve both anodic dissolution of the metal and a cathodic reaction that produces hydrogen. The presence of well-defined crystallographic features indicates the absence of significant metal dissolution, thereby suggesting that slip/dissolution is not the primary cause of accelerated cracking. This observation is consistent with findings by Chopra and Smith (1998) that crack growth rates for 304 SS are greater in low dissolved oxygen water than in high dissolved oxygen water. This observation cannot be reconciled with a slip/dissolution mechanism.

The presence of sharp, crystallographic facets suggests that a hydrogen embrittlement mechanism is responsible for accelerated cracking in 288°C water. This is supported by fractographic findings by Hanninen and Hakarainen (1980) where hydrogen-precharged 304 SS exhibited cleavage-like facets without any detectable α' martensite formation. The facet morphology for the hydrogen-precharged specimens is very similar to that observed in 288°C water, thereby implicating hydrogen in promoting accelerated cracking in high temperature water. Moreover, Gao, et al. (1992) and Wei, et al. (1993) demonstrated that a hydrogen embrittlement mechanism was responsible for accelerated fatigue crack growth rates in stainless steel alloys tested in room temperature aqueous environments. Although α' martensite formation occurred in these specimens, Gao and Wei determined that this transformation did not have a critical role in controlling crack growth rates and it is not a prerequisite for hydrogen embrittlement.

Although hydrogen embrittlement is believed to be the primary cause of environmental cracking in 288°C water, it is possible that oxide film formation at the crack tip also affects cracking behavior by restricting slip reversals during the unloading portion of fatigue cycles. The importance of oxide film formation in affecting fracture surface morphology can be seen in Figure 16, which compares the fracture surface appearance in room temperature air and vacuum. Fatigue fracture surfaces generated in air possessed well-defined crystallographic facets, whereas those generated in vacuum had a nondescript, nonfaceted appearance. Apparently, the thin oxide film that forms in 24°C air serves as a dislocation barrier that impedes slip reversals during unloading cycles. Hence, damage tends to be concentrated along particular slip bands, and eventually local separation along these slip bands produces well-defined crystallographic facets. In vacuum, the absence of an oxide film promotes more effective slip

reversals, which minimizes local damage along any particular slip band. As a result, crystallographic facets do not develop in vacuum. Oxide film formation in water is also expected to restrict slip reversals and promote facet formation and higher crack growth rates; however, the degree of acceleration is expected to be much less than that associated with hydrogen embrittlement.

In summary, it is unlikely that slip/dissolution is a primary cause of environmental cracking in 288°C hydrogenated water because of the presence of crisp crystallographic features on the fracture surface and the increase in crack growth rates with decreasing dissolved oxygen levels (Chopra and Smith, 1998). The cleavage-like facets on the fracture surface, which are very similar to facets found in hydrogen-precharged 304 SS (Hanninen and Hakarainen, 1980), suggest that hydrogen embrittlement is the primary cause of accelerated cracking in high temperature water. It is also likely that the formation of crack tip oxides restricted slip reversals which also contributed to increased crack growth rates, although this effect is expected to be much smaller effect than that associated with hydrogen embrittlement.

VII. SUMMARY AND CONCLUSIONS

Instrumented corrosion fatigue tests on 304 stainless steel (304 SS) double-edged-notched (DEN) specimens provided fatigue crack growth rate data in air and water over a wide range of crack growth rates. Results in air and water at the same mechanical parameters allowed direct assessment of environmental effects, avoiding any concerns for data variability due to materials, test technique, and data correlation. Crack growth rates of 304 SS in water are about 12X times the air rate at low velocities where the environmental effects are largest. The large environmental degradation for 304 SS crack growth is consistent with the strong reduction of fatigue life in commercial PWR water. Further, very similar crack growth rate results were reported in low oxygen HWC, in both surface crack and conventional deep crack tests. The large environmental enhancement in 304 SS (12X) persisted to crack extensions up to 4.1 mm, far outside the range associated with short crack effects. The same large environmental effects observed in the DEN tests were reproduced in CT specimens at a high stress ratio and low stress intensity. The overall results can be normalized successfully by incorporating the combined effects of stress ratio and rise time, qualitatively similar to the formulation developed by Itatani to describe test results in BWR water.

Much of literature data in hydrogenated water chemistry shows an apparently mild environmental effect for 304 SS, with an ER of 2.55 or less. However, based on the current test results, larger environmental effects occur in hydrogenated water in the low ΔK regime at long rise times and higher R-ratio conditions.

The high crack growth rates in 288°C deaerated water were associated with a faceted growth mechanism. The highly angular, cleavage-like appearance of the facets suggests that

a hydrogen embrittlement mechanism was the primary cause of accelerated cracking in this environment.

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Figure 1. Double-edge Notched Fatigue Specimen with EPD (Grip details not shown, all dimensions in mm)

Figure 2. Representative Microstructure of 304 SS

Figure 3. Visual and EPD Crack Extension Measurements Close agreement indicates no corrections needed

Figure 4. Data Averaging to Reduce Scatter, Determine Rates Raw data is replaced by averages of 11 or 21 points da/dN calculated by secant method

Figure 5. Crack Growth rate From DEN Specimens Rates normalized by deep crack rates-James&Jones

Figure 6. Environmental Effects in DEN Tests by Direct Comparison to Controls in Air
Figures show relative effects of loading parameters by comparison to each other-
Fig b vs a shows reduction of ER with increasing frequency
Figure c vs b shows reduced ER at larger stress amplitude
Figure d (next page) shows reduced ER at R=0

Figure 6. Continued -Environmental Effects in DEN Tests by Direct Comparison to Controls in Air
Figure d shows reduced ER at R=0

Figure 7. 304 SS DEN Crack Growth Rates in Water vs Air
Trend shows decreased ER at high air rate
Air rates are calculated directly from control tests-Fig 6

Figure 8 Alloy 600 Crack Growth Rates from DEN Tests in Water

Figure 9. Comparison of DEN Data to Surface Crack Specimens
Literature show large environmental effect similar to DEN
HWC is BWR water with 150 ppb hydrogen, 10 ppb O

Figure 10. FCP Rates from Compact Tension Tests in HWC Tests all at high R and show large environmental effect extending to low air rates

Figure 11. 304 SS FCP Data vs Stress Intensity (Water at 288°C)

Figure 12. Normalized 304 SS FCP Data vs ΔK
Normalization functions: $(1-R)^{-1.1}$ and $T_r^{0.31}$ to $R=0$, $T_r=150$ s

Figure 13. 304 SS CT FCP Data at Low Potential vs ΔK
Normalization functions: $(1-R)^{-1.1}$ and $T_r^{0.31}$, $R=0$, $T_r=150$ s
Combined data suggests ER decreases at high ΔK

Figure 14. Fractographs of 304SS tested in 24°C air. (a) Irregular, quasi-cleavage facets at $da/dN = 8 \times 10^{-5}$ mm/cycle. Arrow denotes failed twin boundary. (b) Fatigue striations at $da/dN = 8 \times 10^{-4}$ mm/cycle. (c) Striations and dimples at 1×10^{-2} mm/cycle.

Figure 15. Evidence of repeated contact between mating crack surfaces for specimens tested at $R = -1$. (a) Rub marks at low ΔK levels in 288°C air. (b) Well-defined fatigue striations surrounded by regions that were severely rubbed in 24°C air.

Figure 16. 316 SS fatigue fracture surface. Cleavage-like facets formed in 24°C air, whereas the fracture surface formed in vacuum had a nondescript, nonfaceted morphology.

Crack Growth Direction ↑

Figure 17. Fractographs of 304 SS fatigue tested in (a) 24°C air showing irregular facets, (b) 288°C air showing cleavage-like facets, & (c) 288°C water showing crystallographic facets that have a sharp, cleavage-like appearance and are highly angular.

Figure 18. Fractographs of 304 SS fatigue tested in 288°C water. (a) (b) Highly angular facets persist to 3×10^{-4} mm/cycle. (c) High magnification of region in (a) and (b) showing fatigue striations superimposed on facet faces.

APPENDIX

Appendix to Fatigue Crack Propagation from Notched Specimens of 304 SS in an Elevated Temperature Aqueous Environment -Data Tables

Table 1 304 Stainless Steel Crack Growth Rates in Air
Waveform:Triangular Frequency 1-5 Hz

1T601		1T605		2T601		2T602	
K _I -Gross 8.8		K _I -Gross 8.8		K _I -Gross 4.8		K _I -Gross 4.8	
Rho-mm 0.38		Rho-in 0.38		Rho-mm 1.52		Rho-mm 1.52	
S _{gross} -MPa 50.3		S _{gross} -MPa 66.9		S _{gross} -MPa 88.3		S _{gross} -MPa 129.7	
R -1		R -1		R -1		R -1	
Temp-C 288		Temp-C 288		Temp RT		Temp RT	
Extension	da/dN	Extension	da/dN	Extension	da/dN	Extension	da/dN
mm	mm/c	mm	mm/c	mm	mm/c	mm	mm/c
0.132	3.94E-06	0.132	8.99E-06	0.127	3.66E-05	0.147	2.72E-05
0.251	5.28E-06	0.257	1.32E-05	0.257	5.11E-05	0.262	2.35E-05
0.513	5.99E-06	0.391	1.39E-05	0.378	6.60E-05	0.389	5.51E-05
0.762	7.62E-06	0.643	1.77E-05	0.630	7.67E-05	0.119	1.12E-04
1.021	6.50E-06	0.886	1.74E-05	0.902	9.30E-05	0.272	2.35E-04
1.278	8.38E-06	1.151	2.84E-05	1.148	1.07E-04	0.404	2.05E-04
1.527	8.10E-06	1.407	2.87E-05	1.394	1.11E-04	0.625	1.50E-04
1.786	1.09E-05	1.648	3.12E-05	1.671	1.10E-04	0.879	1.19E-04
2.035	1.02E-05	1.920	2.90E-05	1.941	1.34E-04	1.146	1.09E-04
2.294	8.08E-06	0.127	5.89E-06	2.141	1.35E-04	1.389	1.73E-04
0.130	3.05E-06	0.389	7.24E-06	2.418	1.43E-04	1.669	3.66E-04
0.254	3.48E-06	0.648	8.59E-06	2.713	1.52E-04	1.979	2.90E-04
0.386	3.81E-06					2.223	2.57E-04
0.635	4.01E-06						
0.894	4.32E-06						
1.146	4.65E-06						

Table 1 304 Stainless Steel Crack Growth Rates in Air-Continued
Waveform:Triangular Frequency 1-5 Hz

2T608		1T606		2T610		2T611	
K _I -Gross 4.8		K _I -Gross 8.8		K _I -Gross 4.8		K _I -Gross 4.8	
Rho-mm 1.52		Rho-mm 0.38		Rho-mm 1.52		Rho-mm 1.52	
S _{gross} -MPa 71.0		S _{gross} -MPa 64.8		S _{gross} -MPa 93.1		S _{gross} -MPa 69.0	
R -1		R -1		R -1		R 0	
Temp-C 288		Temp RT		Temp-C 288		Temp-C 288	
Extensionm	da/dN	Extensionm	da/dN	Extensionm	da/dN	Extensionm	da/dN
m	mm/c	m	mm/c	m	mm/c	m	mm/c
0.127	2.79E-06	0.130	2.90E-05	0.130	1.79E-05	0.129	1.01E-05
0.257	5.51E-06	0.262	4.19E-05	0.246	3.12E-05	0.255	1.89E-05
0.389	7.92E-06	0.389	3.25E-05	0.130	2.72E-05	0.382	2.92E-05
0.638	8.20E-06	0.655	9.53E-05	0.259	5.23E-05	0.658	4.95E-05
0.889	8.59E-06	0.912	6.63E-05	0.500	1.00E-04		
1.148	1.07E-05	1.125	9.55E-05	0.777	1.39E-04		
1.397	1.37E-05			1.024	1.67E-04		
1.666	1.32E-05						
1.897	1.37E-05						
2.144	1.81E-05						
2.416	1.73E-05						
2.667	1.81E-05						
2.934	1.36E-05						
0.124	4.57E-06						
0.249	8.15E-06						
0.386	8.64E-06						
0.638	1.09E-05						
0.894	1.12E-05						
1.143	1.39E-05						
1.405	1.72E-05						
1.628	1.13E-05						
1.872	1.32E-05						
2.134	2.04E-05						
2.426	2.11E-05						
2.670	2.33E-05						
2.964	1.89E-05						
3.175	1.59E-05						
3.409	1.85E-05						
3.680	3.15E-05						
3.937	2.62E-05						

Table 2 304 Stainless Steel Crack Growth Rates in Water
Waveform:Triangular water 288°C

1T603		1T604		2T607		2T606	
K _I -Gross 8.8		K _I -Gross 8.8		K _I -Gross 4.8		K _I -Gross 4.8	
Rho-mm 0.38		Rho-mm 0.38		Rho-mm 1.52		Rho-mm 1.52	
S _{gross} -MPa 69.7		S _{gross} -MPa 55.2		S _{gross} -MPa 93.1		S _{gross} -MPa 69.0	
R -1		R -1		R -1		R -1	
Freq.-Hz 0.0033		Freq.-Hz 0.0333		Freq.-Hz 0.003333		Freq.-Hz 0.0333	
Extension	da/dN	Extension	da/dN	Extension	da/dN	Extension	da/dN
mm	mm/c	mm	mm/c	mm	mm/c	mm	mm/c
0.130	6.65E-05	0.135	2.08E-05	0.251	3.07E-04	0.137	7.06E-05
0.262	1.01E-04	0.254	3.05E-05	0.391	5.56E-04	0.318	1.24E-04
0.373	1.20E-04	0.508	3.83E-05	0.658	5.92E-04	0.564	1.98E-04
0.516	1.75E-04	0.759	7.18E-05	0.836	2.43E-04	0.886	2.34E-04
0.615	2.06E-04	1.021	5.97E-05	0.909	5.49E-04	1.125	2.42E-04
0.876	2.29E-04	1.278	4.71E-05	1.153	9.07E-04	1.359	2.28E-04
1.148	2.28E-04	1.354	3.51E-05	1.387	1.06E-03	1.590	2.34E-04
1.415	2.16E-04	0.135	2.87E-05	0.127	1.42E-04	0.145	3.35E-05
1.669	2.04E-04	0.249	4.88E-05	0.254	3.12E-04	0.272	5.49E-05
1.920	2.09E-04	0.384	6.25E-05	0.505	2.45E-04	0.389	6.22E-05
2.169	2.07E-04	0.627	6.65E-05	0.767	6.25E-04	0.521	6.93E-05
0.127	7.82E-05	0.881	6.20E-05	1.021	5.31E-04		
0.203	1.32E-04	1.148	6.05E-05	1.270	8.51E-04		
0.330	1.97E-04	1.387	5.74E-05	1.519	7.04E-04		
0.457	2.10E-04						
0.592	2.40E-04						
0.856	2.92E-04						
1.097	3.18E-04						
1.339	3.00E-04						
1.603	3.15E-04						
1.864	3.43E-04						
2.146	3.56E-04						
2.446	3.89E-04						
2.690	4.17E-04						
2.941	4.37E-04						
3.190	4.17E-04						
		2T605				2T609	
		K _I -Gross 4.8				K _I -Gross 4.8	
		Rho-mm 0.06				Rho-mm 1.52	
		S _{gross} -MPa 69.0				S _{gross} -MPa 69.0	
		R -1				R 0	
		Freq.-Hz 0.0333				Freq.-Hz 0.0333	
		Extension	da/dN			Extension	da/dN
		mm	mm/c			mm	mm/c
		0.127	7.62E-05			0.269	7.01E-05
		0.254	2.54E-04			0.373	9.60E-05
		0.381	1.37E-04			0.635	1.98E-04
						0.889	2.42E-04
						0.267	8.71E-05
						0.508	1.37E-04
						0.747	3.35E-04
						0.899	2.77E-04
						1.133	2.64E-04

Table 3 Alloy 600 Crack Growth Rates in Air
Waveform:Triangular Frequency 1-5 Hz

A600 4A		A600 2	
K _I -Gross 4.8		K _I -Gross 4.8	
Rho-mm 1.52		Rho-mm 1.52	
S _{gross} -MPa 133		S _{gross} -MPa 130	
R -1		R -1	
Temp-C 288		Temp RT	
Extension	da/dN	Extension	da/dN
mm	mm/c	mm	mm/c
0.130	1.14E-05	0.132	2.43E-05
0.259	2.64E-05	0.384	8.23E-05
0.472	5.31E-05	0.638	2.24E-04
0.704	8.38E-05	0.892	1.37E-04
0.993	1.11E-04		
1.240	1.69E-04		
1.415	1.85E-04		
1.618	2.14E-04		
1.882	3.15E-04		
0.130	6.38E-06		

Table 4 Alloy 600 Crack Growth Rates in Water
Waveform:Triangular 288°C water

A600 8-1		A600 503		A600 509	
K _I -Gross 4.8		K _I -Gross 4.8		K _I -Gross 8.8	
Rho-mm 1.52		Rho-mm 1.52		Rho-mm 0.38	
S _{gross} -MPa 124		S _{gross} -MPa 124		S _{gross} -MPa 124	
R -1		R -1		R -1	
Freq.-Hz 0.0333		Freq.-Hz 0.0333		Freq.-Hz 0.0033	
Extension	da/dN	Extension	da/dN	Extension	da/dN
mm	mm/c	mm	mm/c	mm	mm/c
0.130	9.93E-06	0.130	4.34E-06	0.130	1.52E-04
0.254	2.27E-05	0.376	1.73E-05	0.264	2.30E-04
0.505	9.07E-05	0.650	2.97E-05	0.513	2.74E-04
0.752	1.66E-04	0.902	4.93E-05		
1.019	1.32E-04	1.148	5.38E-05		
1.265	3.00E-04	1.392	5.97E-05		
1.516	5.99E-04				
1.773	4.42E-04				
2.057	2.35E-04				
2.286	3.68E-04				
2.537	4.80E-04				
2.802	3.30E-04				
3.038	4.62E-04				
3.297	4.65E-04				
3.561	2.57E-04				