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EXPERIENCES AND MODELING OF HYDROGEN CRACKING IN A THICK-WALLED PRESSURE VESSEL

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13. ABSTRACT *(Maximum 200 words)*

Hydrogen cracking associated with armament structures has become more prevalent in recent times. Recent work by Troiano et al. has clearly shown that the utilization of specific barrier coatings, such as nickel, can impede the absorption of hydrogen and retard hydrogen-induced cracking.

In this work, a thick-walled, autofrettaged pressure vessel was manufactured from ASTM A723 Grade 2 steel and heat treated to a yield strength of 1170 MPa. An outside diameter keyway was then machined. The keyway was exposed to concentrated sulfuric acid, leading to apparent cracking within 20 hours of exposure.

An investigation of the affected keyway in the pressure vessel indicated that localized hardened areas were present. The base material possessed hardness values of Rc 37 to 39, while the keyway possessed localized hardened zones up to Rc 44. These zones extended to a depth of approximately 4-mm. The different hardness layers suggest that the environmental cracking incubated and propagated in two separate stages. Cracking in the hardened skin layer on the surface incubated quickly and propagated to approximately the 4-mm depth and arrested itself once it encountered the more ductile base material. Previously published crack growth (*da/dt*) test data, and new data verify that this process of incubation and propagation occurred in a matter of seconds (in the hardened skin layer). The cracking then resumed in the softer base material after approximately 300 hours of incubation time. Additional *da/dt* testing of this condition has been performed over a range of yield strengths and verifies that incubation times and crack propagation rates are similar to those observed in this pressure vessel.

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BACKGROUND

Prior history with armament structures has shown a susceptibility to environmental fracture. In 1992, a pressure vessel fractured during manufacturing due to prolonged exposure to a 50% H_2SO_4/50% H_3PO_4 acid solution (ref 1). The two-meter long, through-thickness crack initiated at an outside diameter keyway. The stress that drove the crack was due to a tensile hoop residual stress on the outside diameter of the pressure vessel as a result of the balance of stresses from the autofrettage process. The autofrettage process uses an oversized mandrel to plastically deform the bore of the pressure vessel, resulting in the formation of compressive residual stresses at the bore surface and tensile residual stresses at the outside diameter. In 1997, a piston being used as a seal component cracked as a result of a compressive overload, which resulted in a tensile residual stress field (ref 2). This stress field coupled with the aggressive hydrogen-rich gases within the pressure vessel resulted in cracking in as few as two cycles.

In order to verify previous laboratory hydrogen cracking results on high-strength armament steels (ref 3), a full-scale hydrogen-cracking test on an existing pressure vessel was proposed. A keyway measuring 4.7-mm in width, 12.7-mm in depth, and approximately 450-mm long, with an included radii of 0.4-mm was machined parallel to the axis of the pressure vessel (Figure 1). The pressure vessel, made from A723 Grade 2 steel was heat treated to a nominal 1170 MPa yield strength. The tensile residual stress, at the position in the unnotched pressure vessel wall corresponding to the root of the keyway was +500 MPa, well below the yield strength of the material.

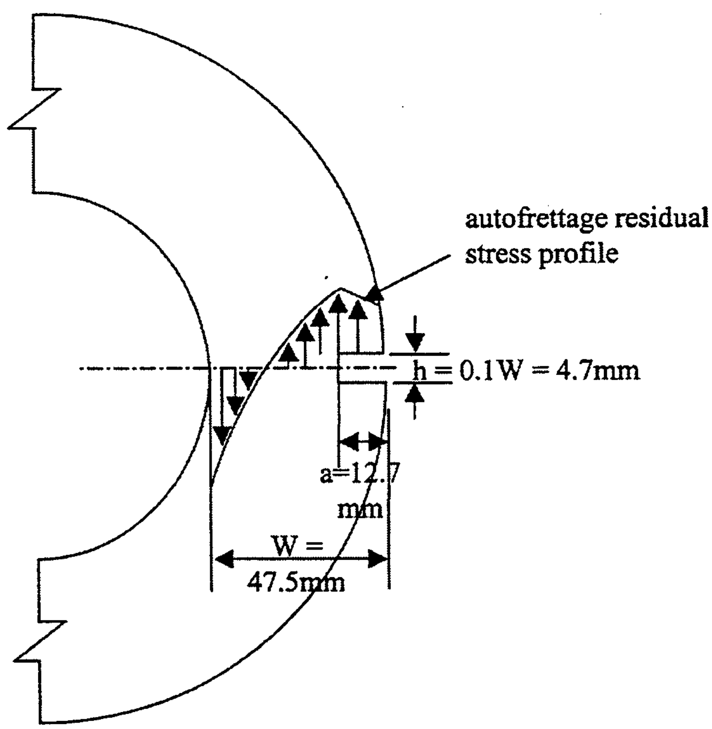

Figure 1. Configuration of pressure vessel with superimposed residual stress distribution.

Vigilante et al. (ref 4) have conducted environment testing of A723 steel over a wide range of strength levels ranging from 1145 MPa to 1380 MPa, and using a 50% H_2SO_4/50% H_3PO_4 acid solution as the source for the hydrogen (Figure 2). An applied stress intensity (K_o) of 55 MPa-m$^{1/2}$ was initially applied for all tests. Their tests were conducted with a constant displacement bolt-loaded compact specimen that had previously been fatigue cracked to induce a sharp crack initiation site. Based on their published *da/dt* versus *ΔK* data, it was estimated that the pressure vessel would not crack as a result of the sustained +500 MPa residual stress. Several conditions existed that led to this conclusion, namely, Vigilante's results were conducted with sharp fatigue cracks; the stress concentrator in this pressure vessel was much less severe; and the applied stress intensity level was believed to be near the threshold necessary to induce cracking. The prior results showed that 3000 hours of incubation time would be necessary to initiate cracking, however, this particular test was scheduled to be completed after 1500 hours.

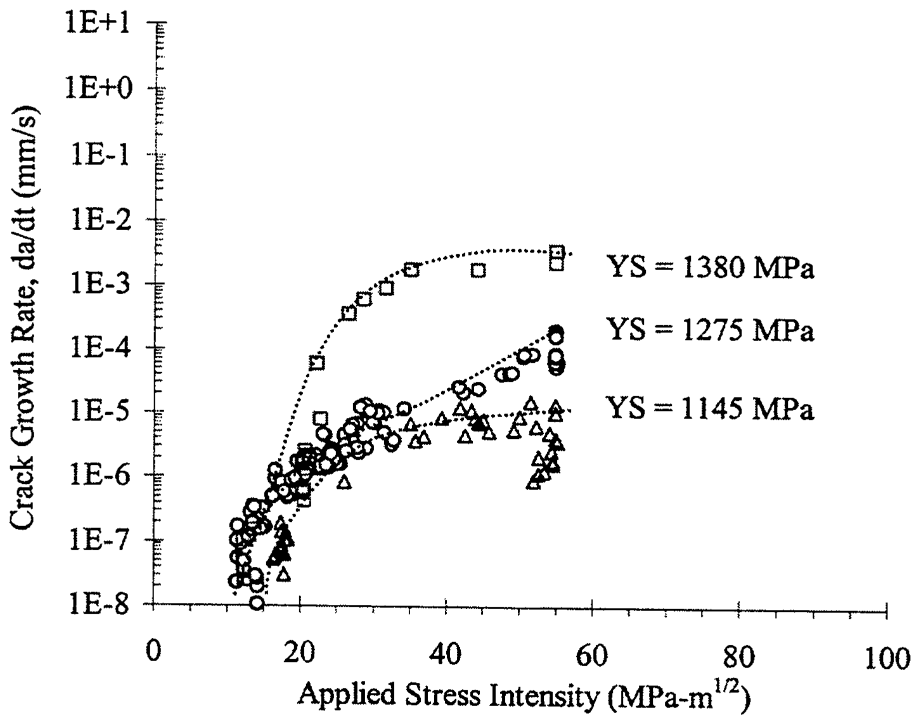

Figure 2. *da/dt* versus *ΔK* in 50% H_2SO_4/50% H_3PO_4 acid for ASTM A723 steel at three different strength levels.

ANALYSIS/TEST RESULTS

Full-Scale Keyway Tests

The keyway was inspected using magnetic particle and ultrasonic inspection methods in order to establish a baseline for future inspections. The keyway was then cleaned to remove any oils and dirt, and subjected to 13 ml of concentrated H_2SO_4. It was believed at this time that the H_2SO_4 would behave essentially the same as the 50% H_2SO_4/50% H_3PO_4 solution that Vigilante et al. (ref 4) had used in their work. The keyway was inspected visually every hour for the first 24 hours, and daily thereafter. After less than 20 hours of exposure, it was observed that some of the acid had been depleted and an additional 2 ml of acid was added to the keyway to maintain the initial level of acid in the keyway. During the course of the next 300 hours, the pressure vessel was monitored both visually and with ultrasonics to detect any cracks, yet none were observed. During this time however, the acid had become completely depleted and additional acid was added to restore the initial level of acid in the notch. A white residue (Figure 3) was observed, and is believed to be a by-product of the reaction between the acid and the steel. This residue greatly hindered the inspection process. Over the course of the remaining 1500 hours, the inspections and addition of the acid continued, but no cracking was observed. At this time, the acid was neutralized, and magnetic particle and ultrasonic inspections of the keyway were performed. It was then that a crack-like discontinuity was noticed, however the operator was still unsure as to the extent of the damage. The pressure vessel was subsequently exposed to a single-peak pressure cycle of approximately 300 MPa, and the pressure vessel split open exposing the crack surface (Figure 4).

Figure 3. Corrosion by-product on pressure vessel resulting from acid environment.

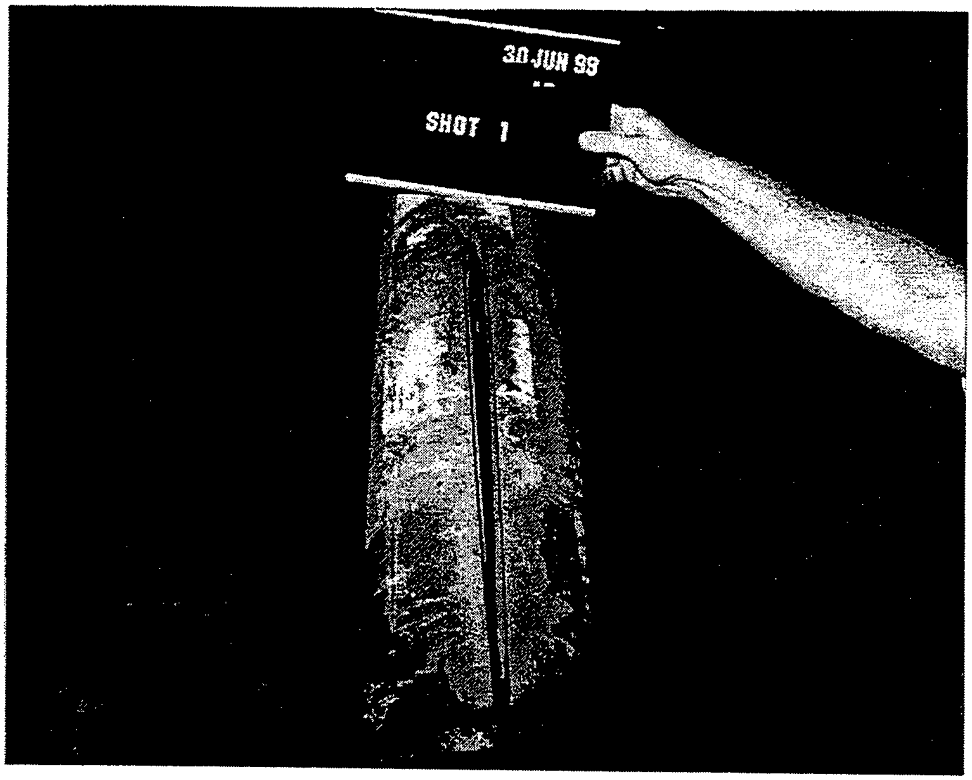

Figure 4. Pressure vessel after application of internal pressure.

Mechanical Properties/Microstructure

The material was immediately checked to verify that it was properly heat treated and that it was free of inclusions or secondary phases, which may have accelerated the failure process. The mechanical properties measured from two areas near the failure are shown in Table 1. In all instances the required mechanical properties were met.

Table 1. As-Measured Mechanical Properties

	0.2% Yield Strength (MPa)	Ultimate Tensile Strength (MPa)	Reduction-in-Area (%)	Elongation (%)	Charpy Impact Energy (-40°C, *J*)	K_Q (MPa-m$^{1/2}$)
Required	1027 min	1260 max	45 min	13 min	31 min	none
Specimen 1	1170	1227	57	15	66	138
Specimen 2	1170	1220	56	14	66	142

Bulk microstructural investigation revealed a fine-grained tempered martensitic structure with very few instances of inclusions or second phases. The ASTM grain size measurements were taken and found to be 11.5. The mechanical properties closely approximate those from recent work (ref 3), for properly processed A723 steel.

Stress/Fracture/Finite Element Analysis

A logical first step in the analysis was to determine if the pressure vessel would have burst with only the machined keyway present, i.e., by mechanical loading only and no hydrogen cracking. A conservative fracture mechanics approach was utilized, where a crack with an infinite stress concentrator was used to approximate the depth of the keyway, which possessed a finite stress concentrator. The stress intensity was the result of both the internal pressure (K_P) and the autofrettage residual stresses (K_R). Rooke and Cartwright (ref 5) have generated a stress intensity profile for the case of a thick-walled pressure vessel containing an external crack experiencing internal pressurization. The stress intensity solution is

$$K_o = {2pR_i^2\sqrt{\pi d}}\Big/{\left(R_o^2 - R_i^2\right)} \tag{1}$$

where K_0 is the stress intensity (MPa-m$^{1/2}$), p is the internal pressure (MPa), R_i is the internal radius (m), R_o is the outside radius (m), and d is the initial keyway depth (m). Then K_P, the stress intensity resulting from the applied pressure, is calculated by knowing K_o, the wall ratio R_o/R_i and the ratio of $d/(R_o^2-R_i^2)$. Using $R_i = 0.060$-m, $R_o = 0.107$-m, an initial keyway depth of $d = 0.012$-m and a pressure of 200 MPa (the reduced pressure at this axial location), K_P was calculated to be 45.6 MPa-m$^{1/2}$. The overall stress intensity is also a function of the stress intensity induced from the autofrettage process where

$$K_R = 1.12 k_t \sigma_{autofrettage}\sqrt{\pi a} \tag{2}$$

where the stress concentration factor, $k_t = 2.55$, as calculated by finite element analysis, $\sigma_{autofrettage} = 500$ MPa, and $a = 2.54$ E^{-5}-m or the size of a typical flaw that was observed within the keyway. The calculated $K_R = 12.7$ MPa-m$^{1/2}$. The resulting $K_{applied} = K_R + K_P = 58.3$ MPa-m$^{1/2}$, which is well the below the average measured K_Q of 140 MPa-m$^{1/2}$. Therefore, the pressure vessel did not burst from mechanical loading of the keyway.

A similar approach using actual measurements from the failed vessel could be used to verify whether the hydrogen had rendered any influence on the cracking of the vessel. At the center of the keyway, where the cracking was deepest, the total notch plus crack depth was measured to be $d = 0.045$-m, and K_{Ieac} as previously measured by Vigilante et al. (ref 4) was approximately 15 MPa-m$^{1/2}$. This resulted in an applied pressure of only 2.9 MPa that is necessary to induce cracking of the remaining ligament of the vessel. This is well below the applied 200 MPa pressure.

The prior analyses all suggest that the cracking was the result of the hydrogen-induced environmental cracking, to the point where final failure by mechanical overload was expected.

Scanning Electron Microscopy/Visual Examination/Metallography

Scanning electron microscopy (SEM) was performed on the fracture surface. The fracture surface was corroded and damaged as a result of the prolonged exposure to the acid environment and the single-cycle mechanical overload. The majority of the fracture surface had been so badly damaged, that the details of the event were "etched" away. As a result of this, no conclusive forensic evidence of the fracture morphology was observed. One area of speculative intergranular fracture can be observed in Figure 5.

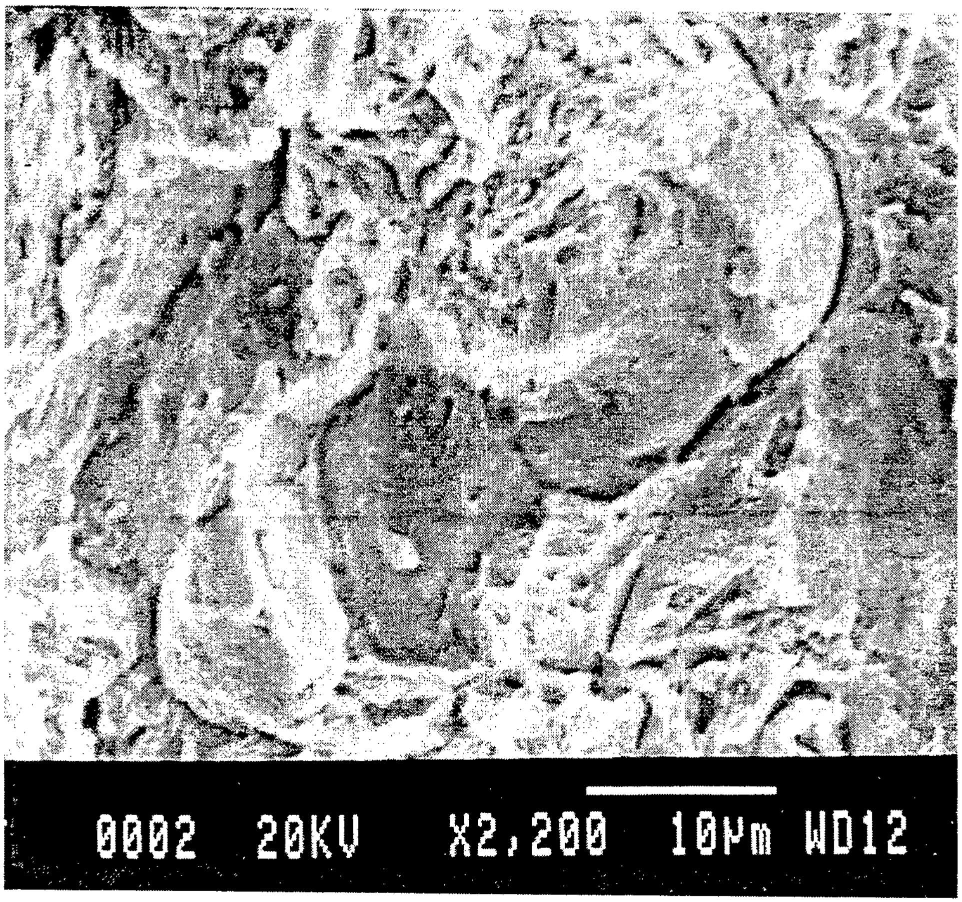

Figure 5. Speculative intergranular fracture.

Note in this photomicrograph the occurrence of what appears to be secondary cracking in the grain boundaries. An ASTM grain size calculation was performed, and a grain size of 10.7 was measured. During the SEM investigation, a sharp demarcation line at a crack depth approximately 4-mm below the base of the notch was clearly observed (Figure 6). It was obvious to the naked eye that there was a different fracture appearance in this 4-mm region than on the remainder of the fracture surface. A cross section of the fracture was obtained and a microhardness profile was taken that emanated from the notch toward the inside diameter of the pressure vessel. The microhardness reading showed a progressive gradient of hardness of R_c 44 at the notch surface and a hardness of R_c 40 at a depth 4-mm below the notch. The bulk of the material beyond the 4-mm region possessed a hardness range of R_c 37 to 39. The cause of this localized hardened region is not known.

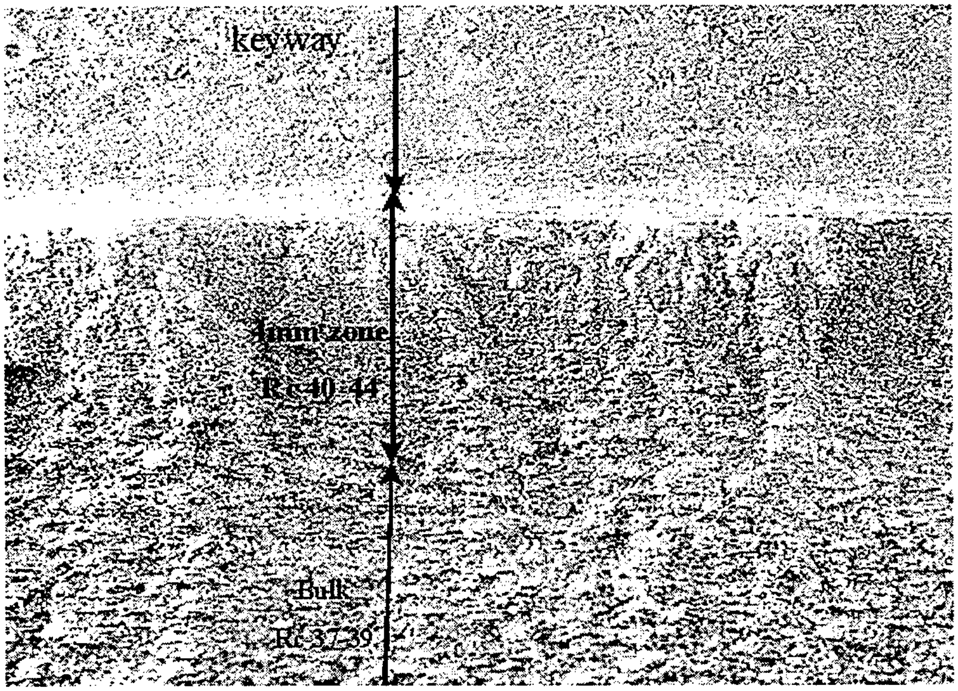

Figure 6. 4-mm fracture zone.

<u>Laboratory Hydrogen Cracking Testing</u>

Hydrogen cracking tests were conducted on modified bolt-loaded compact specimens similar to the one used in Reference 2. In these tests, a similar notch detail modeling the pressure vessel was utilized (Figure 7). An instrumented bolt, outfitted with strain gages imbedded within the body of the bolt accurately measured the applied loads. The concept was to model the stress field in the pressure vessel using finite element analysis, and simulate the same stress field in the test specimen. Many of the details in the pressure vessel were modeled, including the similar *h*, *W*, and *r* dimensions. Because of load limitations with the instrumented bolt, a longer notch depth was necessary in order to induce the same stress fields.

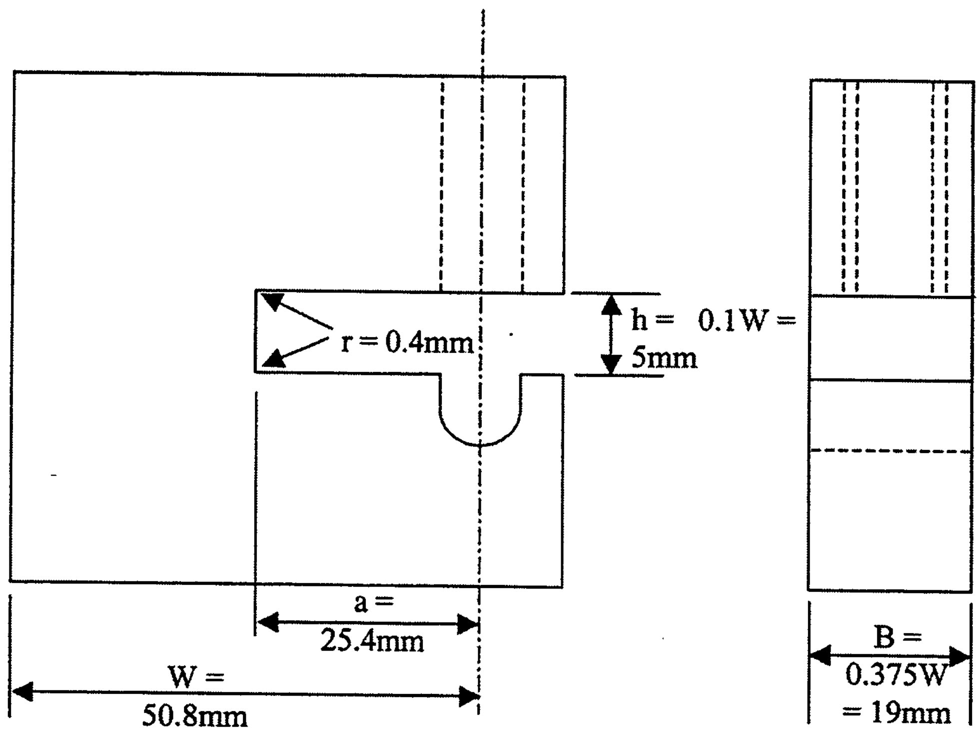

Figure 7. Configuration of modified bolt-loaded compact specimen.

After over 300 hours the specimen's exposure to concentrated H_2SO_4, no cracking was observed. It is believed that different constraint conditions were present in the pressure vessel when compared to the test specimen. The finite element analysis results matched the stress state of the specimen to that of the pressure vessel in the hoop and radial directions, and disregarded the stresses in the axial direction. However, the free surface of the 19-mm thick test specimen resulted in a predominately plane-stress condition, whereas the much larger pressure vessel was predominately plane-strain.

Environmental Testing in Concentrated H_2SO_4

Further testing of the bolt-loaded compact specimen was conducted in order to establish a baseline for hydrogen cracking in concentrated H_2SO_4. Although it was not known at this time what effect the different acids would have on the crack growth rates, it was speculated that the effects would only be minimally different than those observed with the 50% H_2SO_4/50% H_3PO_4. Further testing of 1310 MPa and 1170 MPa yield strength A723 specimens was performed. The 1310 MPa specimens were loaded to a K_o of 100 MPa-m$^{1/2}$ and 55 MPa-m$^{1/2}$, and the 1170 MPa specimens were loaded to a K_o of 80 MPa-m$^{1/2}$ and 50 MPa-m$^{1/2}$. The result of these tests can be observed in Figure 8. The results suggest a K_{Ieac} of approximately 17 MPa-m$^{1/2}$, approximately the same as that previously observed by Vigilante et al. (ref 4). Note, however, that the crack growth rates are significantly faster in concentrated H_2SO_4 than those tested in the 50% H_2SO_4/50% H_3PO_4.

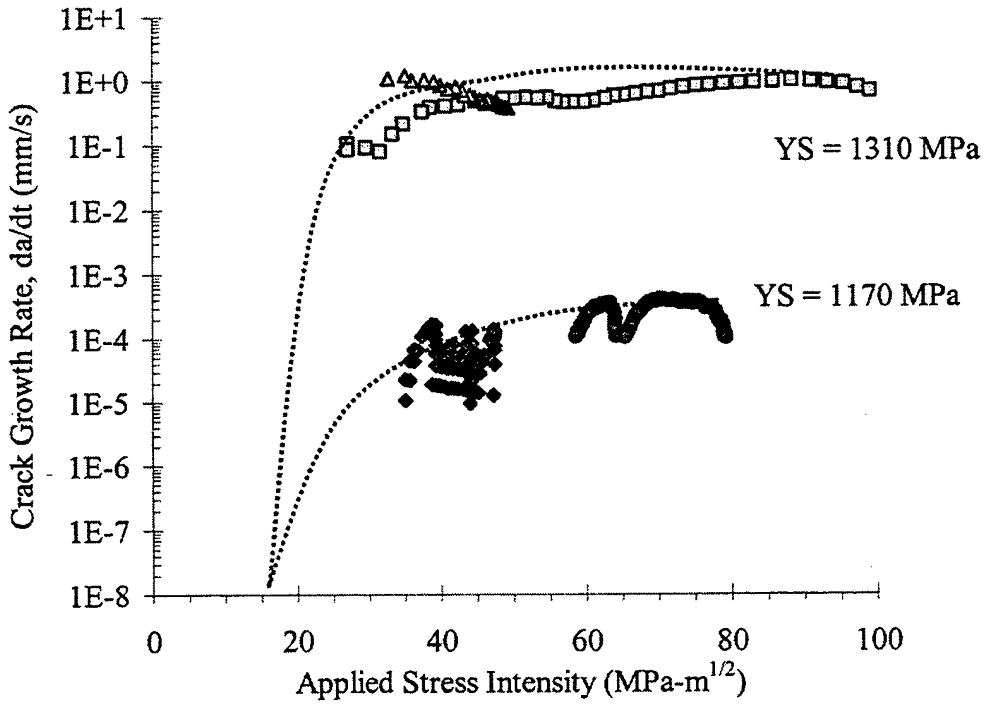

Figure 8. *da/dt* versus *ΔK* in concentrated H_2SO_4 for ASTM steel at different strength levels.

MODELING OF ENVIRONMENTAL CRACKING EVENTS

The pertinent test results and environmental cracking data in concentrated H_2SO_4 and 50% H_2SO_4/50% H_3PO_4 have been compiled in Figure 9. Several scenarios of environmental cracking will be investigated here. The model used is very simple, since we assume that all crack growth that occurs is in the Stage II region. Therefore

$$\frac{da}{dt} = C \tag{3}$$

which when integrated, results in

$$a_{final} - a_{initial} = C(t_{final} - t_{initial}) \tag{4}$$

The incubation times are simply added into the model by assuming that a dwell occurs between times of crack advancement. The data presented in Figure 9 are for applied stress intensities of 50 to 55 MPa-m$^{1/2}$. This is approximately the same as the $K_{applied}$ of 58.3 MPa-m$^{1/2}$ for the pressure vessel at the point of cracking, which justifies its use in this model.

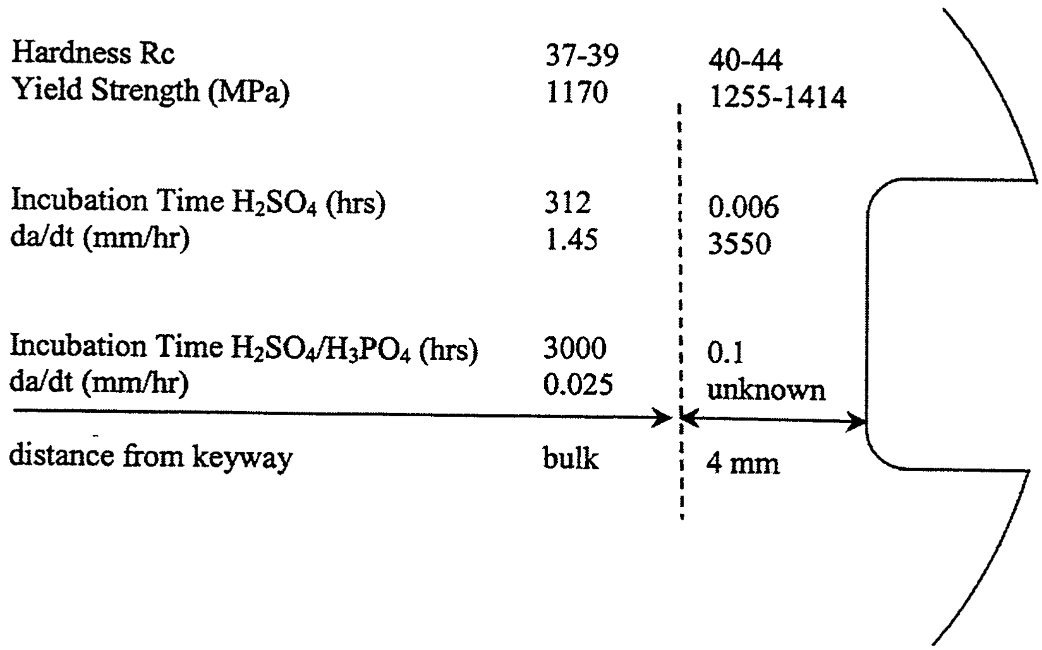

Figure 9. Schematic of keyway showing key parameters for modeling hydrogen cracking event.

Three different scenarios of environmental cracking are presented and plotted in Figure 10. They are:

- *Scenario #1* - No localized hardened layer in 50% H_2SO_4/50% H_3PO_4

Although this scenario did not exist, it does clearly explain the rationale behind our initial belief that cracking would not have occurred under these test conditions. Under this scenario an incubation time of approximately 3000 hours would have occurred, followed by crack advancement to 47-mm (the width of the pressure vessel wall). This scenario is plotted in Figure 9. Note that the predicted crack advancement is beyond the 1500 hours test duration, which validates our initial belief.

- *Scenario #2* – No localized hardened layer in concentrated H_2SO_4

Had we simply replaced the 50% H_2SO_4/50% H_3PO_4 with concentrated H_2SO_4, and not had a 4-mm deep hardened layer, it is likely that the cracking would have occurred within the duration of the 1500-hour test. An expected incubation time would be 312 hours followed by crack advancement, nearly through the wall in the next 32 hours of exposure. This scenario, however, does not explain how cracking was observed (associated with depletion of the acid in the keyway) within the first 20 hours of exposure.

- *Scenario #3* – 4-mm hardened layer in concentrated H_2SO_4

In this scenario cracking incubated in 0.006 hour and propagated to a depth of 4-mm in 0.001 hour, when it arrested. The re-incubation into the softer base material took another 312 hours, after which time it propagated nearly through the wall in approximately 30 hours. This scenario clearly suggests how cracking started within the first 20 hours, and how the crack extended nearly through the wall within the 1500 hours of exposure.

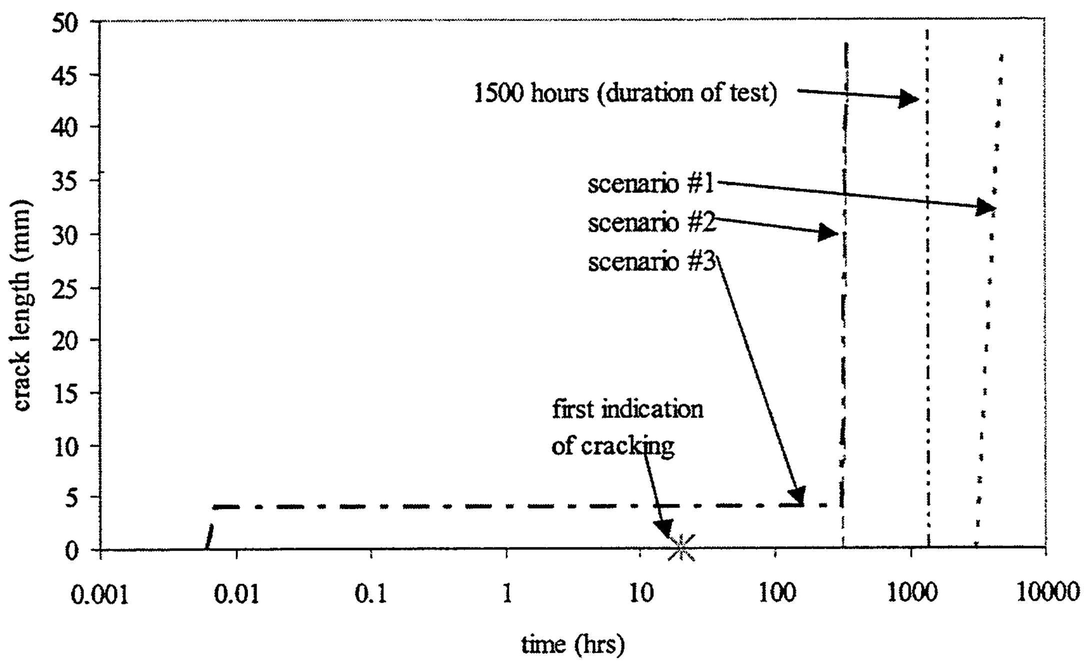

Figure 10. Modeling of several hydrogen cracking scenarios.

SUMMARY

An axial keyway was machined in a highly-stressed steel pressure vessel and exposed to concentrated sulfuric acid in order to investigate the effects of tensile residual stresses on hydrogen-induced cracking. The findings of this study lead to the following observations:

- Speculative intergranular cracking observed during the SEM evaluation, and the presence of an aggressive acid environment, suggest that environmentally-assisted cracking is the likely mechanism of failure.

- Because of the rapid cracking seen in the pressure vessel, the most probable scenario for describing the failure event is that the locally-hardened layer resulted in rapid cracking to the interface with the softer base material. A crack formed within 20 hours of acid application and extended to a crack depth of 4-mm. The crack then took time to re-incubate, and after approximately 300 hours of incubation in concentrated H_2SO_4, the crack continued nearly through the wall of the pressure vessel.

- Had the test been conducted in 50% H_2SO_4/50% H_3PO_4, it is likely that the crack would have arrested itself after encountering a depth of 4-mm. It is not likely that the crack would have re-incubated in the duration of the 1500-hour test.

- The H_2SO_4 acid environment resulted in several orders of magnitude faster crack growth than the 50% H_2SO_4/50% H_3PO_4.

- The local hardened layer at the root of the notch played a minor role in the premature cracking. The major cause of the premature failure was the substitution of concentrated H_2SO_4 for 50% H_2SO_4/50% H_3PO_4.

- Slight increases in hardness can result in drastically increased crack growth rates and much shorter incubation times.

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