Report Number: GE-GRC-Sandia-2007-3

Stress Corrosion Crack Initiation and Growth Measurements in Environments Relevant to High Level Nuclear Waste Packages

Final Report for October 1, 2006 – September 30, 2007

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GE Global Research Center

December 11, 2007

Final Report Prepared under Sandia Purchase Order 627645
by General Electric Global Research Center

Work performed in accordance with purchase order requirements and the
General Electric Global Research Center Quality Assurance
Program for the Corrosion Program, Rev. 1.2, March 12, 2003

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"Stress Corrosion Crack Initiation and Growth Measurements in Environments Relevant to High Level Nuclear Waste Packages"

Final Report on SCC Results – December 11, 2007

Peter Andresen and Young J. Kim – GE Global Research Center

This is a summary of the testing performed in FY07 on stress corrosion cracking and corrosion behavior characterization being performed at GE Global Research. The following lists the categories of tests performed:

1. SCC growth rate testing in an SCW solution at 150 °C.
2. SCC crack initiation, U-bend testing in an SCW solution at 165 °C.
3. SCC crack initiation, Keno constant load testing in a 15% BSW solution at 105 °C.
4. SCC initiation of air fatigue precracked 0.5TCT specimens at 105 °C.
5. Tensile and creep testing in air of titanium grades 7, 28 and 29.
6. Corrosion behavior of Alloy 22 and Ti alloys in salt environments.

A detailed description of the experimental techniques, solution chemistries, materials and prior results is given in References [1,2]. Copies of relevant Procedures, Electronic Data Compilations and supporting Lab Notebooks are also included in CD format.

The matrix of specimens under test in most tasks has remained unchanged since Reference [2] was written (exceptions are noted below), and the time in test is longer by the difference in reporting dates (8,760 additional hours as of October 1, 2007).

1 – SCC growth rate testing in SCW solution at 150 °C.

Three autoclave systems were used to measure growth rates in CT specimens. The test procedures represent state-of-the-art techniques as described in Reference [1,2]. The specimen pairings involved in SCC testing (they were electrically insulated but loaded in series) were:

- c287/c288 – Ti Grades 29 and 28 (as-received) tested at 27.5 MPa√m (ended March 2007)
- c263/c264 – Alloy 22, both as-welded and tested at 40 MPa√m (ended February 2007)
- c265/c266 – Alloy 22, as-welded +TCP or + LRO heat treatment at 40 MPa√m (ended November 2006)

The Alloy 22 specimens were used to study crack growth in the weld metal, and all exhibited very low growth rates despite the change to a more aggressive environment and a higher temperature (150 – 200 °C) compared to earlier tests which were run in 110 °C BSW. The crack length data on both sets of Alloy 22 specimens had become very noisy, and thus there is low statistical confidence that there was any real crack advance.

The final data on specimen c287 are shown in Figures 1 and 2, and the stress intensity factor dependency in Figure 3. Because the crack has grown quite deep, the Ti Grade 29 specimen c287 was replaced with a new Ti Grade 29 specimen. A macro photograph is shown in Figure 4.

A new specimen (c379) of titanium Grade 29 is being used to evaluate the effects of creep response in air (see Task 5).

2 – SCC crack initiation, U-bend testing in an SCW solution at 165 °C.

These tests have continued from their initial start in October 2003, and were most recently interrupted and inspected at 22,333 hours. The exposure time as of October 1, 2007 is 25,261 hours for the Alloy 22 specimens and 11,105 for the Ti Grade 29 specimens (Tables 1 – 3). At the last inspection, there was no evidence of crack initiation. However, as reported previously, there was evidence of fine pits on the outer (uncreviced) arm of a few of the Alloy 22 double U-bends, as well as crevice corrosion in a creviced section of one double U-bend. The four titanium grade 29 U-bend specimens were added to the test following the 14,769 hours shutdown; this necessitated removal of two of the alloy 22 single U-bend specimens.

3 – SCC crack initiation, Keno constant load testing in a 15% BSW solution at 105 °C.

These tests have continued, although there have been periodic forced outages (in October 2004 and February 2005) because the leak rate at the pressure-loading seals was too high. After our move in early 2006, the test was re-started in early June 2006 (with additional lower stressed Ti Grade 7 specimens added), and the system has operated without interruption for inspection since then for a total test time of about 37,000 hours as of October 1, 2007 (Figure 5) – the exact time varies because specimens were installed at different times. The Ti Grade 7 and Grade 29 introduced in June 2006 have now been exposed for 11,520 hours (Figures 6 and 7) with no new failures noted. We have machined specimens of Ti Grades 28, and will need to shut down the test to introduce these specimens.

Because the alloy 22 specimens are not electrically isolated from the stainless steel fixtures, there has been some concern for possible effects of galvanic coupling in the KENO tests. There are two areas of concern: first, there has been some localized corrosion in some modules made of Type 304 stainless steel (e.g., Figure 8). Second, some difference in corrosion potential might exist (without any localized corrosion) between stainless steel and alloy 22.

To evaluate the latter effect, the corrosion potential of coupons of stainless steel and alloy 22 were measured against a Pt coupon ("reference electrode") in autoclaves at 150 °C. Early in test, the corrosion potentials were similar, but after several hundred hours the potential of alloy 22 rose somewhat and the potential of stainless steel dropped somewhat (Figure 2-23 in reference [1]), producing a difference in corrosion potential of about 100 mV. So some effect of galvanic coupling can not be excluded, although it should be noted that elevated levels of dissolved O_2 are being used in the Keno tests, which will elevate the corrosion potential of the materials in solution. Additionally, the corrosion potential measurements in the CT systems were performed at a higher temperature (150 °C), where the difference in corrosion potential is expected to be higher than in the keno system because of the elevated corrosion rate vs. temperature.

The concern for crevice corrosion is that the anodic corrosion current occurring in the threaded regions of Type 304 stainless requires a balancing cathodic current elsewhere, producing a "cathodic protection" effect. This is why active pits do not occur immediately adjacent to each other. The balancing cathodic current occurs preferentially near the site of crevice corrosion. Crevice corrosion was observed predominantly at the location where the "specimen modules" thread into the "manifold tube" (Figure 8), and Teflon tape was used to seal the threads. The corrosion rate was relatively slow, given that a given exposure period was typically 12 – 18 months. Coupling would have occurred preferentially along all of the adjacent stainless steel surfaces (that is, the cathodic current will be supported by all stainless steel between the crevice and the Alloy 22 samples likely resulting in no polarization of the Alloy 22), and given the extremely limited crevice corrosion area associated with Teflon-taped threads, it is unlikely that measurable cathodic current would have traveled away from the threaded area, around the module, up the length of the module, then into the solution access holes and to the specimens. It is harder still to imagine that the current would travel longer distances to adjacent modules many feet away, thereby affecting most of the specimens. It is unlikely that currents were very high or sustained very long, because the extent of corrosion was limited given the very long test times. If crack initiation occurred in some or many specimens, and a correlation between uncracked specimens and areas of localized corrosion could be established, then one could argue that there was some local influence of the crevice corrosion. But with no cracks on any of the specimens over years of exposure, this is an improbable interpretation. Additionally, as time has passed, the Type 304 stainless steel modules were replaced with more resistant materials, initially Type 316 stainless steel and then later Alloy 825. So a substantial fraction of specimens are being tested in Alloy 825 modules and manifolds. Finally, in our higher temperature, more aggressive U-bend tests, the specimens are fully electrically isolated from each other and the autoclave, and there has been no SCC observed. There is no evidence to support the assertion that there is a measurable effect in any of the KENO tests of the isolated observations of crevice corrosion.

4 – SCC initiation of fatigue precracked 0.5TCT specimens in 15% BSW solution at 105°C.

Eighteen 0.5TCT specimens of Alloy 22 were fatigue precracked in air and assembled in three groups of six specimens (i.e., six were loaded in series, and all specimens were electrically isolated) in an autoclave and exposed to 15% BSW solution at 105 °C. The specimens were actively loaded at constant load using the pressure difference between the internal pressure and atmospheric pressure. The three groups of specimens were loaded using three Teflon bal seals, which were conceptually identical to those used in the keno tests. After our move in early 2006, the test was re-started in early June 2006, and the system has operated without interruption for inspection since then, for an accumulated time of 10,473 hours. The specimens were interrupted in May 2007 for inspection, and four specimens removed to make way for four new Ti Grade 28 and 29 specimens. The four Alloy 22 specimens (at 20 and 30 ksi√in) have been fatigued apart and show no sign of crack nucleation or advance from the air fatigue precrack. Table 4 shows the list of specimens, along with the specimens being removed and new Ti specimens added.

There is no evidence of crack initiation in any of these "defected" specimens, and the four alloy 22 specimens that were removed from test and fatigued apart. Macro photographs are shown in Figures 9 – 12; there is no optical evidence of crack advance. The specimens will be examined by SEM to look for more microscopic evidence of stress corrosion cracking nucleation and advance.

5 – Creep testing in air of tensile and CT specimens of titanium grades 7, 28 and 29.

This task characterizes the air creep response of Ti Grades 7, 28 and 29 using both round tensile specimens and CT geometries. The former also provide comparison data for the Keno crack initiation specimens tested in 105 °C BSW, which often failed after relatively short exposure times. The creep data is particularly important, as it shows that the failures obtained in 105 °C "ground water" solutions are likely to have been predominantly creep failures, and not SCC failures. Creep tests were performed on round tensile specimens in an autoclave in 105 °C air. The specimens were slowly loaded and then maintained at a fixed load while the elongation (displacement) is monitored. The creep tests performed in the last eight months have included a higher temperature extensometer (able to operate at 175 – 200 °C); Table 5 summarizes the data.

Tensile creep data on Ti Grade 7 + 20% cold work is shown in Figures 13 – 16. Tensile creep data on Ti Grade 29 is shown in Figures 17 – 22, and on Ti Grade 28 in Figures 23 – 27. A summary of the tensile creep data is shown in Figure 28. The 150 °C creep crack growth data on 0.5TCT specimen c379 is shown in Figures 29 – 32.

The creep crack growth rates in CT specimens tested in air are sometimes similar to the growth rates measured in water. For Ti Grade 7 (Figure 6-14 in reference [1]), the creep crack growth rate in air at 700 hours is a bit slower than the constant load growth rate observed in a 110 °C BSW environment of 1.25×10^{-8} mm/s. Similar data were obtained on a 0.5TCT of Ti Grade 29 tested in 150 °C air at 27.5 MPa√m (Figures 30 and 31), which showed very low growth rates at constant load. However, based on the tensile creep data, it's not clear that creep will explain the SCC growth rate data on Ti Grade 28; more definitive data would involve measurements of air creep crack growth in 0.5TCT specimens of Ti Grade 28. Although this apparent crack growth in air may be related to creep effects on crack advance measurements, some titanium alloys exhibit "sustained load cracking", where crack growth occurs in air at a stress intensity factor value below the critical plane strain fracture toughness value, K_{IC} but above any environmentally determined K_{ISCC} [4].

6 –Corrosion behavior of Alloy 22 and Ti alloys in salt environments

The passivity behavior of Alloy 22 and Grade 7 titanium has been studied at 95°C in a high pH salt environment characteristic of concentrated Yucca Mountain groundwater. Measurements of corrosion potential versus time, corrosion rate and hydrogen pickup were conducted to evaluate the stability of these alloys. The passive films were also analyzed by transmission electron microscopy (TEM) to obtain the chemical composition and cross-sectional view of the metal, interface, and oxide layers.

6A. Long Term Corrosion Potential Measurement

General corrosion rate and Ecorr measurements of Alloy 22 and Ti Grade 7 were obtained in argon purged, 95°C concentrated Yucca mountain (CYM) (BSW) solution after 62 months exposure. Following the initial 62 month exposure, the purge environment was changed from argon to air. The specimens, Alloy 22 and Ti-Grade 7, used for free immersion testing were cut by electrodischarging machining in the form of ½” x ½” x 1/32” and 3/8” x 3/8” x 1/32”, respectively. For the measurement of corrosion potential, the specimen was 1/8” diameter x 2” long. All specimens were wet-polished to a 600 grit paper.

The chemicals used for the test chemistry are shown in Table 6. The test solution was prepared from reagent grade chemicals dissolved in high purity water. The chemicals were mixed with water that had been heated to the boiling point in the autoclave. All testing is performed in an Alloy 22 autoclave. To prevent evaporative loss of water, a six foot long tube-in-tube heat exchanger is used, with cooling water on the outside. The solution level in the test autoclave is monitored periodically by checking for continuity between the autoclave and an insulated Alloy 600 wire feed-through bar. Ar at 100 psi was continuously purged in the test autoclave in order to maintain constant pressure above the solution. After immersion for 62 months in the solution purged with Ar, the purge gas was changed to air.

Corrosion potential behavior of Alloy 22 and Ti Grade 7 is shown in Figure 33. All data values are also shown in Table 7. In general, the corrosion potentials of both alloys are stable over the immersion time in the test solution purged with Ar or Air. There was no significant change in corrosion potential after the purge gas was changed from Ar to air. Under the long term immersion in the test solution the passive films formed on Alloy 22 and Ti Grade 7 would be expected to become so protective and stable that contributions from metal corrosion should become extremely small, and redox reactions from the species in solution should be stable. This potential response should also apply to the waste packages and drip shields, although some increase in dissolved oxygen is expected as the solution on the waste package cools from its saturated boiling point by 10 – 20 °C.

6B. Corrosion Rate and Hydrogen Pickup Measurement

After immersion for 62 months in the test solution at 95°C, a set of specimens were removed, ultrasonically cleaned in a deionized water bath, dried in air, and their weight was measured for calculating the corrosion rate. The method described in ASTM G1- Standards Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens, was used for calculating the corrosion rate of Alloy 22 and Ti-Grade 7. The total hydrogen concentration was measured by Laboratory Testing Inc.

Table 8 summarizes the corrosion rates of Alloy 22 and Ti-Grade 7, which were 0.003-0.004 μm/year and 0.2-0.3 μm/year, respectively. These values are average of three different specimens of each alloy. The high corrosion rate of Ti-Grade 7 may be due to the possible high dissolution rate of Ti oxide at high pH (12-13). It is known that the Ti oxide (TiO_2) is not thermodynamically stable in the solution at higher temperatures with high pHs (>12) [3], while the Cr enriched Ni oxide ($NiO + Cr_2O_3$ and/or $NiCr_2O_4$) formed on Alloy 22 is stable.

The hydrogen pickup by Ti-Grade 7 was also measured, as shown in Table 9, and it also includes data measured after immersion for 24 months under the same test condition. The increase in H_2 uptake may be related to a high corrosion rate of Ti-Grade 7 at high pH, described above.

6C. TEM Analysis of Oxide

The cross-sectional TEM sample was prepared using a focused ion beam (FIB) system. The bulk sample was placed into the FIB system and the region of interest is coated with a 1um thick Pt layer using the in-situ metal deposition facilities of the FIB. The Pt layer was used to protect the underlying material. Staircase shaped cuts were milled on either side of the region of interest using a Ga ion source. The ion current was reduced as the thickness of the section approached the desired dimension. The dimension of the final TEM cross-section was 10μm long, 4μm deep and 150nm thick. The sample was then removed from the FIB chamber and the TEM cross-section was picked out of the bulk sample and placed on a porous carbon grid using a micro-manipulator.

The chemical composition and structure of oxide films play very important roles in corrosion processes and passivity. The mechanisms and kinetics of the corrosion processes can be altered by the chemical and physical properties of oxide films. Figure 34 shows the TEM cross-section micrograph of the oxide film formed after 4 years on Alloy 22 in the mixed salt solution at 95^{o}C. An oxide approximately 10 nm thick, enriched with Cr, was formed. Electron diffraction patterns showed a thermodynamically stable Cr_2O_3 rich oxide film containing Ni (Figure 35). Figures 36 and 37 show the TEM cross-section micrograph and chemistry of the oxide film formed after 4 years on Ti Grade 7 in the mixed salt solution at 95^{o}C. An oxide approximately 200-400 nm thick of predominately TiO_2 containing a large amount of SiO_2 was formed. Since TiO_2 and SiO_2 are not thermodynamically stable in this test environment [3] and electron diffraction patterns do not quite agree with a typical TiO_2 or SiO_2 structure, the possible oxide structure may be $TiSiO_4$.

References

[1] – P.L. Andresen, Y.J. Kim, P.W. Emigh, G.M. Catlin and P.J. Martiniano, "Stress Corrosion Crack Initiation and Growth Measurements in Environments Relevant to High Level Nuclear Waste Packages", Report # GE-GRC-Bechtel-2005-1, Final Report to Bechtel Corp. for FY2005 under Purchase Order QA-HC4-00196, December 9, 2005.

[2] – P.L. Andresen and Y.J. Kim, "Stress Corrosion Crack Initiation and Growth Measurements in Environments Relevant to High Level Nuclear Waste Packages", Report # GE-GRC-Bechtel-2006-2, Final Report to Bechtel Corp. Purchase Order QA-HC4-00196, October 2, 2006.

[3] – M. Pourbaix, "Atlas of Electrochemical Equilibria in Aqueous Solutions", NACE, Houston, 1974.

[4] – ASM International. 1987. Corrosion. Volume 13 of Metals Handbook. 9th Edition. Metals Park, Ohio: ASM International, p. 275.

Table 1 – U-bend Inspection Results

25,261 hours of exposure on October 1, 2007

Ti Grade 29 U-bends started on May 19, 2006 and have 11,105 hours of exposure on Oct 1, 2007

Date of Inspection	Hours at Inspection	Findings
October 31, 2003	0 hours	Start of test
January 29, 2004	2166 hours	No cracks observed *1
May 5, 2004	4151 hours	No cracks observed *1
July 19, 2004	5588 hours	No cracks observed *1
January 31, 2005	9,785 hours	No cracks observed *1
March 29, 2006	14,056 hours	No cracks observed *2
October 2, 2006	17,241 hours	No cracks observed *2
May 8, 2006	22,333 hours	No cracks observed *2

*1 – AR-6 (as received) and TCP-1 (HT TCP) showed microscopic surface imperfections perhaps related to dissolution of inclusions, but no evidence of SCC.

*2 – Some double U-bends showed pitting on outer arm of outer specimen, but no crack. Some double U-bends taken apart, and crevice corrosion was observed in one case.

Table 2 – Project Supplied Double U-Bend Specimens

Identification	Heat	Condition	Arm Spread Before Test	Arm Spread After Test*
DUB1181(outer) / DUB1061(inner)	2277-9-3241	As-received	0.998”	0.975”
DUB1182(outer) / DUB1062(inner)	2277-9-3241	As-received	1.009”	0.974”
DUB1183(outer) / DUB1063(inner)	2277-9-3241	As-received	1.009”	0.977”
DUB1184(outer) / DUB1064(inner)	2277-9-3241	As-received	1.010”	0.974”
DUB1185(outer) / DUB1065(inner)	2277-9-3241	As-received	1.004”	0.973”
DUB1186(outer) / DUB1066(inner)	2277-9-3241	As-received	1.005”	0.975”
DUB1187(outer) / DUB1067(inner)	2277-9-3241	As-received	1.007”	0.970”
DUB1188(outer) / DUB1068(inner)	2277-9-3241	As-received	1.005”	0.973”
DUB1189(outer) / DUB1069(inner)	2277-9-3241	As-received	1.009”	0.975”
DUB1190(outer) / DUB1070(inner)	2277-9-3241	As-received	1.006”	0.972”

* – dimension shown for inspection after 4151hours; at that time, all U-bends were re-tightened as shown to compensate for possible stress relaxation.

Table 3 – GE GR Single U-Bend Specimens

Identification	Heat	Condition*1
C22 (227793263) TCP No. 1	2277-9-3263	TCP No. 1
C22 (227793263) LRO No. 1	2277-9-3263	LRO No. 1
Ti Grade 29 (956205) AR No. 1 *2	956205	AR No. 1
C22 (227793263) TCP No. 2	2277-9-3263	TCP No. 2
C22 (227793263) LRO No. 2	2277-9-3263	LRO No. 2
C22 (227793263) AR No. 2	2277-9-3263	AR No. 2
Ti Grade 29 (956205) AR No. 2 *2	956205	AR No. 2
C22 (227793263) LRO No. 3	2277-9-3263	LRO No. 3
C22 (227793263) AR No. 3	2277-9-3263	AR No. 3
C22 (227793263) TCP No. 4	2277-9-3263	TCP No. 4
C22 (227793263) LRO No. 4	2277-9-3263	LRO No. 4
C22 (227793263) AR No. 4	2277-9-3263	AR No. 4
C22 (227793263) TCP No. 5	2277-9-3263	TCP No. 5
C22 (227793263) LRO No. 5	2277-9-3263	LRO No. 5
C22 (227793263) AR No. 5	2277-9-3263	AR No. 5
C22 (227793263) TCP No. 6	2277-9-3263	TCP No. 6
C22 (227793263) LRO No. 6	2277-9-3263	LRO No. 6
C22 (227793263) AR No. 6	2277-9-3263	AR No. 6
Ti Grade 29 (956205) AR No. 3	956205	AR No. 3
Ti Grade 29 (956205) AR No. 4	956205	AR No. 4

*1 – machined U-bends after heat treatment below from customer supplied C-22 welded plate HT 2277-9-3263. The weld was located at middle of 6” dimension.

AR = as-received (as-welded)
TCP (topologically close packed) = 650C for 200 hrs with water quench
LRO (long range ordering) = 550C for 10 hrs with water quench

*2 – Two specimens removed to make space for the four new U-bends of Ti Grade 29: C22 (227793263) TCP No. 3 and C22 (227793263) AR No. 1.

Table 4 – Weld Defect Tolerance Specimens

Alloy 22 specimens CTS#5, #6, #13 and #15) have been removed and fatigued apart, and two specimens each of Ti Grades 28 and 29 have been added to the test.

CTS	Material and Condition	Stress Intensity Factor	Crack Length
#1	C22 As-welded (AW)	higher K (30 ksi√in)	a/W = 0.554
#2	C22 As-welded (AW)	lower K (20 ksi√in)	a/W = 0.420
#3	C22 As-welded (AW)	lower K (20 ksi√in)	a/W = 0.420
#4	C22 As-welded (AW)	higher K (30 ksi√in)	a/W = 0.554
#5	C22 AW+TCP>>Ti Grade 29	higher K (30 ksi√in)	a/W = 0.554
#6	C22 AW+TCP>>Ti Grade 29	lower K (20 ksi√in)	a/W = 0.420
#7	C22 AW+TCP	higher K (30 ksi√in)	a/W = 0.554
#8	C22 AW+TCP	lower K (20 ksi√in)	a/W = 0.420
#9	C22 AW+SHT +Water quenched	higher K (30 ksi√in)	a/W = 0.554
#10	C22 AW+SHT +Water quenched	lower K (20 ksi√in)	a/W = 0.420
#11	C22 AW+SHT +Water quenched	higher K (30 ksi√in)	a/W = 0.554
#12	C22 AW+SHT +Water quenched	lower K (20 ksi√in)	a/W = 0.420
#13	C22+SHT +Air blasted>>Ti Gr 28	lower K (20 ksi√in)	a/W = 0.420
#14	C22 AW +SHT +Air blasted	higher K (30 ksi√in)	a/W = 0.554
#15	C22+SHT +Still air cool>>Ti Gr 28	higher K (30 ksi√in)	a/W = 0.554
#16	C22 AW +SHT + Still air cool	lower K (20 ksi√in)	a/W = 0.420
#17	C22 Base	lower K (20 ksi√in)	a/W = 0.420
#18	C22 Base	higher K (30 ksi√in)	a/W = 0.554

Total load on 0.5TCT specimen is 1235 pounds to achieve indicated K
SHT = 1120C / 30 minutes / water quench unless otherwise specified
As-welded = precrack in weld metal
AW + TCP = precrack in weld metal, heat treat at 700 C / 175 hours
AW + SHT with water quench, precrack in weld metal
AW + SHT with still air cool down, precrack in weld metal
AW + SHT with air blast cool down, precrack in weld metal
As-received material, fatigue crack not in weld metal

Table 5 – Summary of Tensile Creep Data in Air on Ti Alloys

Creep Rate reported for latter part of measurement period, which might represent failure, or might represent a change or interruption in the test before failure

Specimen	Material*	Temperature	Stress	Creep Rate, s^{-1}
Ti7c1	Ti Gr. 7	105 °C	45 ksi	7.7×10^{-6} (10.1)**
Ti7c2	Ti Gr. 7	105 °C	50.7 ksi	4.0×10^{-5} (1.64)**
Ti7c3	Ti Gr. 7	105 °C	47.5 ksi	3.3×10^{-5} (6.2)**
Ti7c4	Ti Gr. 7	105 °C	40 ksi	1.5×10^{-7} (493)**
Ti7c5	Ti Gr. 7	105 °C	47.25 ksi	7.7×10^{-5} (1.17)**
Ti7c6	Ti Gr. 7	105 °C	41.3 ksi	4.6×10^{-7} (143)**
Ti7c7	Ti Gr. 7 CW	105 °C	68 ksi	6.6×10^{-7} (11.2)**
Ti7c8	Ti Gr. 7 CW	105 °C	56.7 ksi	1.7×10^{-9} (224)**
″	″	″	60.5 ksi	7.6×10^{-10} (224)**
″	″	″	64.3 ksi	1.4×10^{-8} (224)**
Ti29c1	Ti Gr. 29	150 °C	89.25 ksi	1.9×10^{-9}
Ti29c2	Ti Gr. 29	150 °C	94.5 ksi	8.9×10^{-10}
Ti29c3	Ti Gr. 29	150 °C	96 ksi	4.1×10^{-12}
″	″	″	98 ksi	2.0×10^{-10}
Ti28c2	Ti Gr. 28	150 °C	64.8 ksi	5.5×10^{-10}
″	″	″	72 ksi	1.9×10^{-6}
Ti28c3	Ti Gr. 28	150 °C	68.4 ksi	1.5×10^{-10}

* Ti Gr. 7 CW = 20% reduction in thickness by forging at 25 °C

** Specimen failed at the hours indicated in parentheses

Table 6. Chemical composition of the test solution

10.6g Na_2CO_3 (anhydrous)	9.7g KCl
8.8g NaCl	0.2g NaF
13.6g $NaNO_3$	1.4g Na_2SO_4 (anhydrous)
4.1g Na_2SiO_3*9H_2O	53.3g H_2O

Table 7. Corrosion potential values of Alloy 22 and Ti Grade 7

Date	Immersion Time	Corrosion Potential, mV(vs. SCE)		Temperature
	day	Alloy 22	Ti-Grade 7	°C
08/02/01	1	-262	-366	96
08/03/01	2	-238	-407	93
08/06/01	5	-234	-450	95
08/07/01	6.3	-241	-447	95
08/08/01	7.4	-234	-445	94
08/09/01	8.2	-241	-469	94
08/13/01	12	-241	-443	94
08/14/01	13	-242	-351	93
08/15/01	14	-239	-441	93
08/17/01	16	-237	-489	93.9
08/19/01	18	-230	-403	94
08/21/01	20	-234	-518	95
08/22/01	21	-225	-406	95.1
08/23/01	22	-231	-580	95.7
08/24/01	23	-225	-450	95.4
08/25/01	24	-214	-449	95.6
08/27/01	26	-214	-441	96.2
08/28/01	27	-213	-422	94.9
08/30/01	29	-220	-430	97.2
08/31/01	30	-218	-435	97.1
09/04/01	34	-196	-491	97.4
09/05/01	35	-194	-476	97.5
09/06/01	36	-196	-455	96.8
09/07/01	37	-186	-444	96.8
09/09/01	39	-186	-450	95.6
09/10/01	40	-184	-450	95.4
09/11/01	41	-190	-454	95.8
09/13/01	43	-180	-450	96.3
09/14/01	44	-177	-435	95
09/17/01	46	-177	-415	97.5
09/18/01	47	-177	-407	97.9
09/20/01	49	-173	-392	97.5
09/21/01	50	-170	-381	95.5
09/24/01	53	-179	-365	95.8
09/27/01	56	-179	-350	98.4
09/28/01	57	-176	-347	98.1
10/01/01	60	-180	-330	95.3
10/02/01	61	-182	-339	96.1
10/03/01	62	-187	-336	96.2
10/04/01	63	-186	-337	95.7
10/05/01	64	-180	-340	96.2
10/08/01	67	-173	-346	95.1
10/11/01	69	-174	-349	95.3
10/13/01	72	-177	-355	97.5

10/16/01	76	-181	-361	96.1
10/19/01	79	-181	-358	97.4
10/24/01	84	-186	-359	95.4
10/26/01	86	-182	-356	96.2
10/31/01	91	-188	-357	96.5
11/05/01	96	-168	-352	95.7
11/08/01	99	-199	-363	95.8
11/16/01	107	-204	-384	96.4
11/19/01	110	-184	-387	95.5
11/20/01	113	-165	-400	97.1
11/26/01	119	-189	-391	96.5
12/03/01	126	-205	-386	96.5
12/07/01	130	-210	-397	-95.5
12/17/01	140	-216	-380	96.2
12/21/01	144	-200	-375	95.7
01/02/02	156	-210	-387	96.4
01/15/02	169	-203	-385	95.4
01/22/02	176	-182	-389	97.4
02/04/02	186	-212	-387	94.5
02/13/02	195	-210	-382	96.4
02/21/02	203	-220	-378	95.7
03/06/02	216	-201	-383	96.4
03/18/02	228	-223	-363	95.2
03/26/02	234	-245	-344	97.1
04/04/02	243	-258	-324	96.5
07/08/02	339	-199	-330	95.7
07/10/02	341	-195	-327	96.3
07/15/02	346	-202	-332	95.1
07/18/02	349	-182	-304	96.8
10/10/2006*	1860	-180	-353	97.5
12/11/2006	1920	-170	-295	96.7
12/27/2006	1936	-170	-300	94.7
2/9/2007	1980	-195	-282	98
4/9/2007	2040	-171	-289	96.5
4/25/2007	2056	-174	-265	97.5
5/3/2007	2064	-176	-250	96.5
5/22/2007	2083	-180	-277	94.8
6/22/2007	2114	-175	-258	98
7/19/2007	2141	-140	-287	98
8/16/2007	2169	-203	-240	98
8/31/2007	2184	-185	-254	96
9/20/2007	2204	-123	-265	98

* Purged gas changed from argon to air

Table 8. Corrosion rate of Alloy 22 and Ti-Grade 7 after immersion for 62 month in the test solution at 95^{o}C (average value of three different specimens of each alloy)

Alloy 22	0.003- 0.004 µm/year
Ti-Grade 7	0.2 – 0.3 µm/year

Table 9. Hydrogen Pick-up by Ti Alloys

H2 Uptake by Ti-Grade 7 in CYM (No Pb)			
Immersion, mo	**H2, ppm**	**Δ H2, ppm**	**H2 Pickup Rate, ppm/mo**
0	15	0	0
24	20	5	0.208
62	29	14	0.226

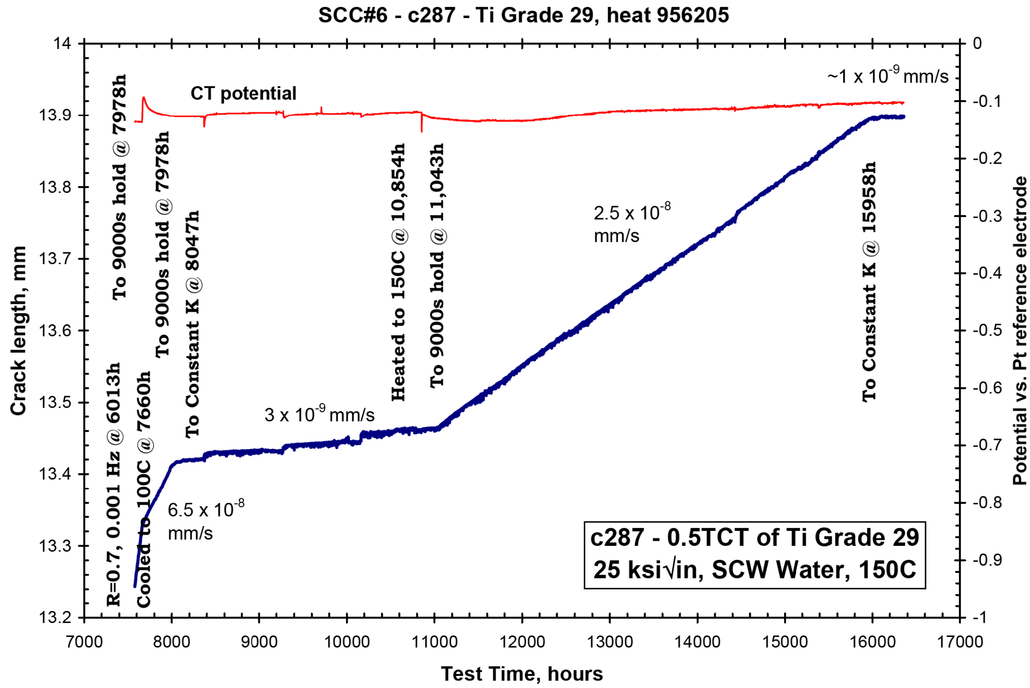

Figure 1. Crack length vs. time for specimen c287 tested in SCW at 150°C.

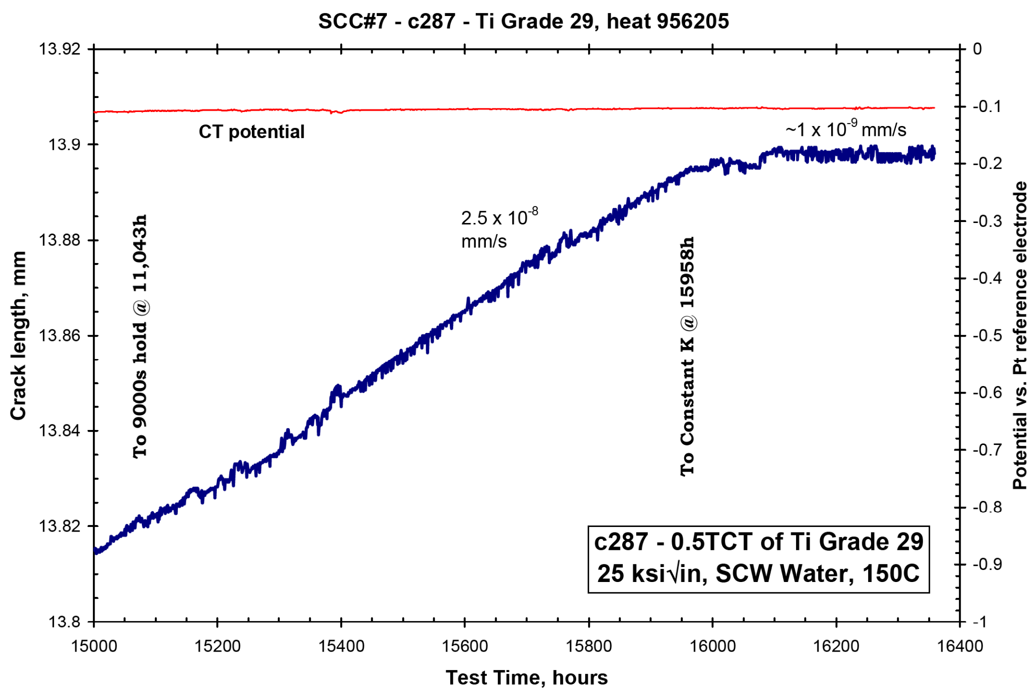

Figure 2. Crack length vs. time for specimen c287 tested in SCW at 150°C.