Investigation of Susceptibility of Titanium-Grade 2 and Titanium- Grade 12 to Environmental Cracking in a Simulated Basalt Repository Environment

**Interim Report
S. G. Pitman**

October 1981

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SUMMARY AND CONCLUSIONS

Slow strain rate tests and fatigue crack growth rate tests have been used by Pacific Northwest Laboratory (PNL) to evaluate the relative susceptibility of titanium (Ti)-grade 2 and -grade 12 to cracking in a simulated repository environment. Slow strain rate tests were performed in air and in simulated Hanford basaltic ground water at 250°C. Fatigue crack growth rate tests were done in Hanford ground water, fluoride-ion-enhanced Hanford ground water, and high-purity water. All tests were conducted at 90°C.

The following conclusions can be drawn:

- Ti-grade 2 and Ti-grade 12 exhibited strain-rate-dependent degradation of ductility in slow strain rate tests at 250°C.
- In-air tests confirmed that the loss of ductility is not limited to the simulated repository environment and may be caused by an internal strengthening or embrittlement mechanism such as dynamic strain aging.
- The ductility degradation in Ti-grade 12 was found to be highly orientation dependent. (Only one orientation of grade 2 was tested.)
- No fractographic evidence of an environmental cracking mechanism was found in the slow strain rate tests; all specimens failed by microvoid coalescence.
- The fatigue crack growth rate of Ti-grade 2 and -grade 12 was not affected relative to air or high-purity water by any of the environments used in this study; and no frequency dependence was observed. This may indicate that no environmental cracking mechanism relative to a Hanford basalt repository is operating significantly under the conditions used in this work (high oxygen fugacity and a temperature of 90°C).

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1.0 INTRODUCTION

A program directed toward the development of licensable structural engineered barrier elements^(a) for high-level nuclear waste packages has been in progress at Pacific Northwest Laboratory (PNL)^(b) since January 1979. It is currently sponsored by the Office of Nuclear Waste Isolation (ONWI). The objective of the program is to characterize and recommend materials for structural barrier elements that are capable of contributing substantially to maintaining radioisotope release rates at near-zero values for ~1000 yr.

Past work in the Engineered Barrier Development Program at PNL has involved corrosion screening of a variety of candidate alloys and ceramic materials; thermal- and radiation-degradation of polymers; and some preliminary investigations of the susceptibility of titanium (Ti)-grade 2 and Ti-grade 12 to environmentally induced fracture (Westerman 1980). These early studies revealed no unusual deterioration of the mechanical integrity of the titanium alloy due to Hanford basaltic ground water or air environments at temperatures to 250°C.

The program currently emphasizes the accelerated testing of Ti-grade 2 and -grade 12 for environmentally induced fracture in simulated Hanford basaltic ground water and the corrosion screening of cast irons and steels in the same environment.

The remainder of this report discusses the testing of Ti-grade 2 and -grade 12. Background information is provided, and the two materials studied are described. The two experimental methods that were used--slow strain rate (SSR) and fatigue crack growth rate (FCGR) tests--are detailed. The results of the work are presented and discussed, and areas for future work are recommended.

(a) The canister, overpack, and hole sleeve are all potential structural barrier elements.

(b) Operated for the U.S. Department of Energy (DOE) by Battelle Memorial Institute.

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2.0 BACKGROUND

The selection and characterization of engineered barrier materials present some unique technical challenges. The failure resistance of these materials must be estimated over a lifetime that could extend to 1000 yr based on tests of a few years maximum duration. Perhaps the most straightforward approach is to evaluate degradation rates from each failure mode individually and then design the barrier element so that it can resist the most severe (rapid) mode of degradation over the time period required. Each failure mode must eventually be investigated over the full range of enviromechanical conditions expected.^(a) The possibility of synergistic reactions between two or more aggressive environmental conditions must also be investigated by the superimposition of enviromechanical stresses in individual tests. Ultimately, reliability analyses of the degradation statistics will allow confidence limits to be set on the ability of a barrier element to perform its intended function.

There are many known failure modes that can limit the life of metallic barrier elements. Of these, stress corrosion cracking (SCC) is potentially one of the most important because it can lead to dramatic failures in relatively short times in susceptible materials. This failure mode poses a relatively difficult problem for barrier designers because it is difficult to quantify, if observed, due to its generally unpredictable nature and it is difficult to rule out completely, if not observed in the course of a (necessarily) short laboratory study. The unpredictability of SCC is largely due to the fact that there are two phases to the process--crack initiation and crack growth--and each phase has its own peculiar rate-enhancement mechanisms.

Stress corrosion cracking is often evaluated using statically loaded, pre-cracked specimens (wedge-opening load) or stressed corrosion coupons (U-bends or C-rings). These specimens are most useful for determining susceptibility

(a) For example, oxygen and hydrogen fugacities, pH, radiation dose, dose rate, mechanical stress concentration, cyclic and monotonic loading, and reactive chemical species concentration.

to SCC where service times are relatively short. Precracked specimens are used to determine values of the threshold stress intensity for SCC, K_{Isc} . These static test methods are successful only if a crack initiates and grows until the stress intensity decreases to a limiting value or if the crack growth rate is measured during the test. Tests of this type have been used on Ti-grade 2 and -grade 12 in a simulated basalt repository environment (Westerman 1980). Since cracking did not occur in the environment, K_{Isc} could not be determined. Measuring crack growth rates is further complicated by the fact that any cracking rate measurable in the laboratory would be too high a rate for 1000-yr barrier service; and extrapolation of these data to acceptable crack growth rates would entail large uncertainties. Alternatively, stress intensity threshold values determined in short-term tests may be considerably higher than threshold values determined from longer tests (Wei, Novak, and Williams 1972).

Long-term, environmentally induced cracking can be evaluated with tests that accelerate the fracture process by breaking down the surface film on the specimen and exposing fresh metallic surface to the environment. The SSR test and the environmental FCGR test are two such accelerated tests.

In the SSR test, a specimen is loaded in tension and strained to failure at low displacement rates. It has been found to be a severe test for SCC (Payer, Berry, and Boyd 1976). The straining of any material in tension increases the surface area so that unoxidized material is continuously exposed; at the same time, deformation mechanisms take place in the material. Slip planes emerging at the surface provide sites for environment-metal interaction. At high strain rates, the failure occurs so fast that reactions between the environment and the metal are limited; and at sufficiently low strain rates, the metal typically repassivates as the oxide film is broken and damage from the environment is again limited. At intermediate loading rates, interaction between the environment and the metal occurs as the film is broken and SCC occurs (Payer, Berry, and Boyd 1976). Susceptibility to SCC is usually expressed as loss of ductility at a particular strain rate. The SSR test is an accelerated test relative to statically loaded tests, but it is qualitative in nature. That is, data from these tests can show susceptibility

to a certain environment; but the data may not be useful in a quantitative sense to a designer. One factor that is missing in these tests using ordinary tension specimens is the presence of a crack. This factor may be accounted for by using precracked specimens of suitably thick material or by using FCGR tests.

In the FCGR test, a crack is forced to grow by cycling the load on a specimen and measuring the rate of crack growth. Crack growth rate is found to be a function of cyclic stress intensity^(a) and may be influenced by the environment. Data from FCGR tests can be used to compare the susceptibility of various materials to environmentally enhanced cracking in a particular environment or to provide information useful to a designer who seeks to prevent the occurrence of significant cracking during the design lifetime of the barrier element.

There are two ways of qualitatively evaluating the effects of an environment on FCGR: 1) frequency dependence and 2) accelerated crack growth rate relative to an inert environment. Environmental interactions are time-dependent; therefore, there is some high frequency at which the FCGR is independent of environment (analogous to higher strain rates in the SSR test). At lower frequencies, the crack growth rate may be advanced or retarded due to the effects of the particular environment on the crack tip. Environmental FCGR tests are usually considered conservative, rigorous measures of resistance to environmentally enhanced cracking compared with static tests (Bania and Antolovich 1976). They combine the effects of notch chemistry with slow straining and forced exposure of fresh, unoxidized materials.

Design-relevant information can be obtained from FCGR tests to determine the threshold cyclic stress intensity: the stress intensity below which a crack will not grow in the environment of interest under cyclic loading conditions. Threshold stress intensities determined under cyclic loading are

(a) Stress intensity is a function of applied and residual stresses, crack or flaw size, notch sharpness, and geometry of the specimen or structural member. The relationship between crack growth rate and cyclic stress intensity in a laboratory specimen can be related to any structural member; therefore, information obtained from FCGR tests can be applied to barrier design.

expected to be much lower than threshold stress intensities determined under static loading. Thus, the cyclic stress intensity threshold is a very conservative design parameter for repository conditions, where large numbers of cyclic loadings are not expected.

The cyclic loading tests conducted in the present study were performed in stress intensity ranges much higher than the threshold values to evaluate relative susceptibility of Ti-grade 2 and -grade 12 to various environments. Evaluation of threshold stress intensities will be the subject of future tests.

It is frequently informative to perform environmental FCGR tests in non-aggressive environments to determine the extent of damage caused by the test environment although it may be difficult to determine whether the control environment is actually inert. Seemingly innocuous environments may contain species that enhance or retard crack growth rate. For example, the humidity in laboratory air has been found to affect crack growth rate considerably (Speidel 1971). It may be most useful to think in terms of a "reference environment," rather than in terms of an "inert environment" when such comparisons are made.

3.0 MATERIALS

Two materials were used in this study: Ti-grade 2 and Ti-grade 12. Grade 2 is a common commercial purity grade; grade 12 was developed for resistance to crevice and pitting corrosion in elevated temperature high-chloride environments (Covington and Palmer 1974). Both materials were tested in the mill-annealed condition. The composition of these materials is listed in Table 1. The Ti-grade 2 sheet was 1.27 mm (0.050 in.) thick, and the Ti-grade 12 sheet was 0.79 mm (0.031 in.) thick. Room temperature mechanical properties are listed in Table 2.

The microstructure of Ti-grade 2 (see Figure 1) consists of equiaxed recrystallized alpha grains with small particles of hydride and larger particles of beta phase stabilized by impurities. This beta phase is associated with high iron content and can be expected to be absent in commercial purity titanium with low iron content.

The microstructure of Ti-grade 12 (see Figure 2) consists of a continuous matrix of alpha phase with a minor amount of beta phase. Other researchers have found, using transmission electron microscopy and diffraction analysis, that a fine precipitate of TiC existed (Abrego and Rack 1981). This precipitate is too fine to be resolved by light microscopy.

TABLE 1. Composition of Titanium-Grade 2 and Titanium-Grade 12 Used in This Study and ASTM Nominal Compositions

ASTM B-265-78 Grade	Elements, wt%						
	N	C	H	O	Fe	Mo	Ni
2 Actual	0.009	0.11	0.005	0.012	0.12		
2 ASTM Nominal(a)	0.030	0.100	0.015	0.250	0.300		
12 Actual	0.008	0.021	0.005	0.013	0.12	0.30	0.67
12 ASTM Nominal(a)	0.03	0.08	0.015	0.250	0.300	0.2 to 0.4	0.6 to 0.9

(a) Maximum compositions except as noted.

TABLE 2. Room Temperature Mechanical Properties of
Titanium-Grade 2 and Titanium-Grade 12

	Yield Strength, MPa (ksi)	Tensile Strength, MPa (ksi)	Elongation, %
Ti-Grade 2 L ^(a)	351 (51)	497 (74)	29
Ti-Grade 2 T ^(b)	393 (57)	517 (72)	28
Ti-Grade 12 L	427 (62)	600 (87)	22
Ti-Grade 12 T	600 (87)	696 (101)	18

(a) L = longitudinal.

(b) T = transverse.

- a. View of Plane
Perpendicular to
Rolling Direction
and Perpendicular
to Surface

- b. View of Plane
Parallel to
Rolling Direction
and Perpendicular
to Surface

- c. View of Surface

FIGURE 1. Microstructure of Titanium-Grade 2

- a. View of Plane
Perpendicular to
Rolling Direction
and Perpendicular
to Surface

- b. View of Plane
Parallel to
Rolling Direction
and Perpendicular
to Surface

- c. View of Surface

FIGURE 2. Microstructure of Titanium-Grade 12

4.0 EXPERIMENTAL METHODS

Long-term, environmentally induced cracking can be evaluated with tests that accelerate the fracture process by breaking down the surface film on the specimen and exposing the fresh metallic surface to the environment. The SSR test and the environmental FCGR test are two such accelerated experimental methods.

4.1 SLOW STRAIN RATE TESTS

SSR tests were conducted at 250°C using tensile specimens fabricated from sheet material (Figure 3). These specimens were inscribed with gage marks 2.54 cm apart to determine elongation after the test. The specimen width was slightly reduced in the center of the gage section to induce fracture between the gage marks.

The simulated ground-water solution was directed into the bottom of the test autoclave. It passed upwards through a layer of crushed basalt before

FIGURE 3. Tensile Specimen Used in Slow Strain Rate Tests (dimensions in cm)

reaching the specimen (Figure 4). The reservoir of solution was continuously sparged with a mixture of 85% nitrogen and 15% oxygen, which maintained a dissolved oxygen content of about 6 ppm. Solution flow rate was maintained at approximately 40 ml/h. The inlet and outlet solutions were sampled and analyzed; typical analyses are given in Table 3.

Constant extension rates were applied to the specimens using a gear-driven loading device (Figure 4). The load was measured on a load cell that was external to the autoclave, and displacement was measured on a linear variable differential transformer (LVDT) between the autoclave and the loading rod. The load, displacement, and temperature signals were recorded using a nonprogrammable datalogger,^(a) except for the last tests where the signals were recorded using a programmable datalogger.^(b) The load versus displacement data were plotted for each test so that deformation characteristics could be determined.

Reduction of area was determined by measuring the dimensions of the specimen gage length before the test and the dimensions of the fracture surface after the test. The fracture surfaces were examined optically and with a scanning electron microscope to determine the mode of failure.

4.2 FATIGUE CRACK GROWTH RATE TESTS

Ti-grade 2 and -grade 12 FCGR tests were performed in reference Hanford basaltic ground water, fluoride-ion-enhanced Hanford ground water, and high-purity water. All tests were performed at 90°C. Specimens with two orientations were tested: LT specimens, where cracking occurred perpendicular to the primary rolling direction; and TL specimens, where cracking occurred parallel to the primary rolling direction of the sheet. Center-cracked tension specimens were used (see Figure 5).

The tests were conducted at frequencies of 1.0 and 0.1 Hz. Precracking was done by cycling first at 5.0 Hz and then decreasing the frequency so that final precracking occurred at the test frequency. A sinusoidal waveform with

(a) John Fluke Mfg. Co., Seattle, Washington.

(b) Analog Devices, Inc., Norwood, Massachusetts.

FIGURE 4. Slow Strain Rate Test System

TABLE 3. Typical Inlet and Outlet Composition of Simulated Ground Water in Slow Strain Rate Tests

Ion	Concentration, ppm	
	Inlet	Outlet
B	0.02	0.03
Ca	1.2	0.4
K	3	42
Mg	0.31	<0.05
Na	285	267
Si	63	173
SN	0.1	0.1
SO ₄ ⁼	115	112
F ⁻	37	37
Cl ⁻	153	162
pH	9.84	9.46

R = 0.1 was used at all times. Ti-grade 2 was precracked and tested at cyclic loads of 907/90.7 kg (2000/200 lb); grade 12 was precracked and tested at 554/55.4 kg (1200/120 lb) to give the same stress intensity range.

The environmental chamber used for FCGR tests is shown in Figure 6. A gravity feed system provided flowing solution to the chamber, and the flow rate was controlled using an in-line metering device. A (once-through) flow rate of approximately 100 ml/h was maintained. The water chemistry of the simulated Hanford ground water was the same as given in Table 3 (inlet), except that of the fluoride-ion-enhanced ground water, which contained approximately 220-ppm F⁻.

The solution in the chamber was heated using electrical resistance heaters that were made by bending Pyrex tubing to conform to the inside chamber dimensions and inserting coiled resistance wire. A proportioning controller maintained the temperature at 90 ±2°C except for short periods when the flow rate was being adjusted. The control thermocouple was located near but not touching the center of the specimen. No metal or conducting parts were in

FIGURE 5. Center-Cracked Tension Test Specimen
(dimensions in cm)

contact with the wet portion of the specimen. The only metal parts exposed to the solution were the control thermocouple (stainless steel sheathed), readout thermocouple (type K), and a platinum wire used to assist in drainage.

The simulated ground-water solution was exposed to air before and during the tests. This experimental condition technically violates the "reference Hanford basalt ground-water" storage condition (Rockwell Hanford Operations 1980) because of absorption of CO_2 (which could cause a change in the prescribed pH) and perhaps other factors. Oxygen fugacity was not adjusted or considered to be a variable in the tests, although this factor could possibly affect repassivation during crack growth. These FCGR tests were not intended to simulate repository conditions accurately but to assess the relative susceptibility of the titanium alloys investigated to environmentally enhanced cracking.

FIGURE 6. Environmental Chamber Used in Fatigue Crack Growth Rate Tests

5.0 RESULTS AND DISCUSSION

The objectives of this study are to: 1) detect any active cracking mechanisms that could result in unsatisfactory performance of metallic barriers in repository conditions and 2) measure failure rates from cracking if any environmentally assisted cracking is observed. The test methods used in this study (SSR and FCGR tests) are intended to eliminate the initiation stage of environmentally assisted cracking and thus accelerate environmental degradation if susceptibility exists.

5.1 SLOW STRAIN RATE TEST

The dependence of elongation and reduction of area of Ti-grade 2 and -grade 12 (LT orientation) on strain rate is shown in Figures 7 and 8, respectively. It is apparent that ductility decreases with slower strain rates and that both alloys are affected. The effect of orientation can be established by comparing Figures 8 and 9. The ductility of the TL orientation (tensile axis perpendicular to the primary rolling direction) is most affected by strain rate. Although tests of the TL orientation of Ti-grade 2 are not yet complete, a similar result can be expected since both alloys exhibit the same orientation dependence in mechanical and FCGR tests and have similar microstructural textures.

The fracture surfaces of SSR specimens were examined for signs of SCC such as intergranular fracture. Specimens tested at strain rates of 10^{-4} , 10^{-5} , 10^{-6} , and 2×10^{-7} /s were examined; and no evidence of environmentally enhanced fracture was found. The fracture surfaces were examined in their entirety under a low-power optical microscope and then under a scanning electron microscope.

Faceted, roughly spherical crystals were present in some of the fractographs (see, for example, Figure 10). The composition of these crystals was evaluated using x-ray analysis, and it is evident that these particles were derived from the ground-water solution. The specimens were ultrasonically cleaned in acetone and then in demineralized water prior to fractographic examination, but the crystals did not dissolve. The crystals were present on

FIGURE 7. Elongation and Reduction of Area of Titanium-Grade 2 (LT orientation) in Hanford Ground Water at 250°C

some of the Ti-grade 12 fracture surfaces and on nonfracture surfaces as well, especially in those specimens tested at low strain rates; but none were found on any of the Ti-grade 2 specimens. The reason for this is not known.

In all cases the fracture mode appears to be microvoid coalescence, even near the edges of the specimens; no signs of cleavage or intergranular fracture were found. In a few cases, features that resemble microcracks were present, although no definite correlation between these features and loss of

FIGURE 8. Elongation and Reduction of Area of Titanium-Grade 12 (LT orientation) in Hanford Ground Water at 250°C

ductility could be established (see Figure 11). These features appeared in the grade 2 specimens that were tested at the two lowest strain rates and in the grade 12 specimens that were tested at higher strain rates. The crack-like features are probably laminations present in the material that were accentuated by deformation during fracture.

The size of dimples formed during fracture often correlates with toughness when the fracture is controlled by microvoid coalescence (Kaufman 1975).

FIGURE 9. Elongation and Reduction of Area of Titanium-Grade 12 (TL orientation) in Hanford Ground Water at 250°C

A statistical analysis of dimple size of the SSR specimens has not been performed, but some observations can be made. No difference in dimple size is apparent between the Ti-grade 2 specimens pulled at the highest ($10^{-4}/s$) and lowest ($2 \times 10^{-7}/s$) strain rates although a slight difference in depth of the fracture surface is evident (see Figures 12 and 13). The specimen strained at $2 \times 10^{-7}/s$ appears to have shallower dimples and less overall deformation.

FIGURE 10. Typical Appearance of Crystals Found on Titanium-Grade 12 Specimen

Fracture surfaces of Ti-grade 12 specimens (LT orientation) are shown in Figures 14 and 15. The dimple size of Ti-grade 12 specimens appears to be slightly larger than that of Ti-grade 2; however, the fracture surfaces appear to have less deformation (consistent with the higher strength of Ti-grade 12). The smaller dimple size of grade 2 may be attributable to the greater volume fraction of second-phase particles capable of initiating microvoids since dimple size has been found to be proportional to inclusion spacing (Broek 1973).

The LT and TL fracture surfaces from Ti-grade 12 specimens fractured at strain rates of 10^{-4} to 2×10^{-7} /s do not differ greatly (see Figures 14 through 17). Dimple spacing appears to be about the same for both orientations and for all strain rates. The amount of surface relief seems greater at higher strain rates, particularly for the LT specimens. The crack-like features do not correlate with ductility measurements; and those features that look like faceted, roughly spherical inclusions are precipitates from the ground-water solution.

Ti-Grade 12 Specimen

Ti-Grade 2 Specimen

FIGURE 11. Examples of Crack-Like Features Found on Titanium-Grade 12 Specimen Strained to Fracture at 10^{-5} /s and Titanium-Grade 2 Specimen Strained to Fracture at 2×10^{-7} /s

FIGURE 12. Fracture Surface of Titanium-Grade 2 Specimen
(LT orientation) Strained to Fracture at 10^{-4} /s

FIGURE 13. Fracture Surface of Titanium-Grade 2 Specimen
(LT orientation) Strained to Fracture at 2×10^{-7} /s

FIGURE 14. Fracture Surface of Titanium-Grade 12 Specimen
(LT orientation) Strained to Fracture at $10^{-4}/s$

FIGURE 15. Fracture Surface of Titanium-Grade 12 Specimen
(LT orientation) Strained to Fracture at $2 \times 10^{-7}/s$

FIGURE 16. Fracture Surface of Titanium-Grade 12 Specimen (TL orientation) Strained to Fracture at $10^{-4}/s$

FIGURE 17. Fracture Surface of Titanium-Grade 12 Specimen (TL orientation) Strained to Fracture at $2 \times 10^{-7}/s$

One explanation for the lower ductility of TL specimens relative to LT specimens is the degree of strain hardening. Ductility increases with strain-hardening exponent (McClintock 1968), which is related to the difference between yield strength and ultimate strength. This difference is about 130 MPa (19 ksi) for Ti-grade 2 (LT orientation) and 175 MPa (25 ksi) for Ti-grade 12 (LT orientation). The TL orientation of grade 12 exhibits a strain-hardening effect of about 69 MPa (10 ksi) or about half as much as the LT orientation. This is probably due to the large effective grain size in the direction of principal strain, the lack of suitable dislocation barriers, and the easier fracture path along the second phase. The degree of strain hardening changes slightly with strain rate (from 58 to 83 MPa), and it is possible that the loss of ductility over this range is due to a mechanism affecting dislocation mobility (such as dynamic strain aging).

The significant changes in ductility with strain rate cannot be tied microstructurally to an environmentally enhanced fracture mode. According to Payer, Berry, and Boyd (1976), "Only metallographic examination of the fractured specimen determines unequivocally the presence or absence of stress-corrosion cracking." Therefore, it is necessary to perform other tests to determine the mode of ductility degradation.

Preliminary tests have been done in air at 250°C (presumed to be an inert environment). The results of these tests (at strain rates of 10^{-4} and 10^{-6} /s) are compared to the Hanford ground-water test results in Figures 18, 19, and 20. The ductility measured in the air tests is very nearly the same as that measured in the ground-water tests. Although the air environment is not necessarily inert, it is clear that the ductility degradation mechanism is not exclusively associated with the ground-water environment. Further testing will be required to determine the mode of ductility degradation and to estimate the consequences in a repository situation. Because of the strong orientation dependence of this effect, it is likely that a different thermal or thermomechanical process could reduce the effect on the TL orientation.

Other researchers have done SSR tests on Ti-grade 12 in saturated brine, dry salt, and air at 250°C (Braithwaite, Magnani, and Munford 1979). In those studies very little change in ductility was observed at strain rates between

FIGURE 18. Comparison of Air and Hanford Ground-Water Environmental Slow Strain Rate Test Results for Titanium-Grade 2 (LT orientation)

10^{-4} and 5×10^{-7} /s. Some change in fracture mode was observed at the edges of some specimens (from dimpled rupture to quasicleavage); however, this did not cause any large changes in overall behavior. The reasons for these differences in behavior are not known.

FIGURE 19. Comparison of Air and Hanford Ground-Water Environmental Slow Strain Rate Test Results for Titanium-Grade 12 (LT orientation)

FIGURE 20. Comparison of Air and Hanford Ground-Water Environment Slow Strain Rate Test Results for Titanium-Grade 12 (TL orientation)

5.2 FATIGUE CRACK GROWTH RATE TESTS

FCGR tests of Ti-grade 2 and -grade 12 (LT orientation) were run in Hanford ground-water (see Figure 21). The data points from Ti-grade 12 lie below those from Ti-grade 2, indicating that grade 12 has a lower crack growth rate. The difference is small, and the grade 12 test was interrupted before enough points were determined to draw a definite conclusion.

FIGURE 21. Fatigue Crack Growth Rate of Titanium-Grade 2 and -Grade 12 (LT orientation) Tested in Simulated Hanford Ground Water at 90°C

The environmental FCGR of Ti-grade 2 (TL orientation) is shown in Figure 22. Two possible accelerating factors were employed in this study: lower frequency and the addition of about 220-ppm fluoride to the ground water (instead of the usual 37 ppm). The data groups shown do not cover the same stress intensity range but do indicate the same relationship between crack growth rate and stress intensity. There appears to be no crack growth rate enhancement due to either the flouride content or the lower cyclic rate.

Tests analogous to those shown in the previous figure were performed on Ti-grade 12 (LT orientation). As shown in Figure 23, neither lower frequency (0.1 Hz compared to 1 Hz) nor increased fluoride content (from 37 to 220 ppm) increased the crack growth rate at 90°C.

FIGURE 22. Fatigue Crack Growth Rate of Titanium-Grade 2 (TL orientation) Tested at 1 Hz in Simulated Hanford Ground Water and at 0.1 Hz in Fluoride-Ion-Enhanced Ground Water at 90°C

Figures 24 and 25 show the results of tests performed on Ti-grade 12 (LT and TL orientations) at 0.1 Hz. Each orientation was tested in fluoride-enhanced Hanford ground water and in high-purity water. The results show no enhancement of crack growth rate due to the environment. As in the case of the grade 2 material, there appears to be no corrosion fatigue mechanism active in grade 12 at 90°C in the frequency and stress intensity ranges studied.

Differences in FCGR in titanium can be caused by material processing and resultant grain texture. These differences can be quite pronounced, as shown in Figures 26 (grade 2) and 27 (grade 12). This crack growth rate is highest when the crack is growing perpendicular to the rolling direction (LT specimen).

FIGURE 23. Fatigue Crack Growth Rate of Titanium-Grade 12 (LT orientation) Tested at 1 Hz in Simulated Hanford Ground Water and at 0.1 Hz in Fluoride-Ion-Enhanced Ground Water at 90°C

The differences in crack growth rate due to textural effects are much greater than any differences caused by environmental effects. Each FCGR test provides multiple data points; therefore, test replication is not as important as it is in other types of tests. An example of the close agreement between two tests is shown in Figure 28 where plots of two tests of Ti-grade 12 are shown. Both tests were run in fluoride-enhanced Hanford ground water at 90°C at a frequency of 0.1 Hz, using specimens of LT orientation. The data exhibit scatter typical of well-controlled FCGR tests.

FIGURE 24. Fatigue Crack Growth Rate of Titanium-Grade 12 (LT orientation) Tested at 0.1 Hz in Fluoride-Ion-Enhanced Hanford Ground Water and at 0.1 Hz in High-Purity Water at 90°C

FIGURE 25. Fatigue Crack Growth Rate of Titanium-Grade 12 (TL orientation) Tested at 0.1 Hz in Fluoride-Ion-Enhanced Hanford Ground Water and at 0.1 Hz in High-Purity Water at 90°C

FIGURE 26. Fatigue Crack Growth Rate of Titanium-Grade 2 (LT and TL orientations) Tested at 1 Hz in Hanford Ground Water at 90°C

FIGURE 27. Fatigue Crack Growth Rate of Titanium-Grade 12 (LT and TL orientations) Tested at 1 Hz in Hanford Ground Water at 90°C

FIGURE 28. Results of Two Fatigue Crack Growth Rate Tests of Titanium-Grade 12 at 0.1 Hz in Fluoride-Ion-Enhanced Hanford Ground Water at 90°C

6.0 FUTURE WORK

The enviromechanical portion of the Engineered Barrier Development Program at PNL will continue to be directed toward understanding environmentally enhanced cracking of candidate barrier materials and ultimately toward development of data suitable for barrier design. The approach will be to discover any operative barrier failure modes through accelerated testing methods and then to quantify possible failure rates under repository conditions.

Work currently planned or underway includes:

- SSR testing of Ti-grade 2 (TL orientation) at 250°C
- SSR testing of Ti-grade 2 and Ti-grade 12 at intermediate temperatures to quantify the kinetics of the embrittlement mechanism
- SSR testing of Ti-grade 2 with low (<0.05 wt%) iron content (material does not have the impurity-stabilized beta phase and may have different enviromechanical properties)
- environmental FCGR testing of both titanium grades at 0.01 Hz at 90°C
- environmental FCGR testing of both titanium grades at lower stress intensities
- statically loaded SCC testing in Hanford ground water under γ -irradiation at 250°C; U-bend specimens will be used for testing sheet material, and wedge-opening load specimens will be used for testing plate material
- FCGR testing in a high-temperature, high-pressure system to develop design-relevant data (including threshold stress intensities and low growth rate data) under expected repository conditions.

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