STRAIN RATE SENSITIVITY OF ALLOY 718

STRESS CORROSION CRACKING

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Abstract

Stress corrosion cracking (SCC) tests were conducted in 360°C pressurized-water-reactor (PWR) primary water using Alloy 718 heat-treated to produce precipitate-free grain boundaries. Fatigue precracked, 12.5-mm-thick compact fracture specimens were loaded at displacement rates from 2.5×10^{-8} to 2.5×10^{-10} m/sec. At the fastest displacement rate no SCC is observed. At the slowest displacement rate, SCC is observed but difficulties in *in situ* crack length measurement arise due to the slow test speed. At intermediate rates, SCC occurs and crack growth rate is determined as a function of stress intensity factor.

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Introduction

Alloy 718 is a nickel-base superalloy used by the nuclear power industry for in-core components requiring high strength and resistance to corrosion and stress relaxation. These include bolts, pins, springs and beams in both pressurized-water reactors (PWRs) and boiling-water reactors (BWRs). Heat treatments developed for jet engine application of Alloy 718 are typically used for reactor components. While Alloy 718 is sufficiently corrosion resistant that the oil industry uses it in deep sour gas well applications, it is not immune to corrosion in reactor environments and research is underway toward optimizing heat treatment of the alloy for nuclear service.(1-4)

Laboratory specimens of Alloy 718 are highly resistant to initiation of stress corrosion cracking (SCC) in PWR primary-side water in the absence of a significant stress intensifier, such as a fatigue precrack.(1,4) Fatigue precracked specimens crack readily, however, and K_{Iscc} tests using precracked fracture mechanics specimens are a logical choice for characterizing the material behavior in the PWR primary-side environment.

There are numerous methods of conducting K_{Iscc} tests (1, 5-7), and none of these has been standardized. There is little K_{Iscc} data available for Alloy 718 in PWR primary water to provide guidance in choosing a test method. The experimental work reported below is part of a larger matrix involving numerous combinations of melt practice and thermomechanical processing conditions. In order to obtain results in a timely manner, a rising load type of K_{Iscc} test was selected, similar to that described by Mayville, et al(5) and Dietzel, et al(6). Recognizing that the severity of SCC depends upon applied strain rate, tests are conducted at progressively lower strain rates until the measured K_{Iscc} reaches a minimum. Crack length is monitored remotely during testing, in this case using a reversing direct current (d.c.) electrical potential drop technique.(8) Data is obtained in the form of crack growth rate as a function of applied stress intensity, K.

Experimental Procedures

The Alloy 718 test material used in this study was obtained from a dimensionally incorrect gas turbine disc provided by the Wyman Gordon Company. It was made from Teledyne Allvac Heat No. E790, which was vacuum induction melted and vacuum arc remelted. The chemistry is shown in Table I.

Table I. Chemistry of Alloy 718 Test Material

Ti	Al	Nb	Ni	Fe	Mo	Cr	C	B
1.04	0.46	5.23	bal.	19.50	3.11	18.30	0.035	0.004

Si	Co	Mn	Ta	S	P	Cu
0.059	0.24	0.16	0.051	<0.001	0.015	0.049

The material was annealed at 1093°C for 1 h, cooled at 55°C/h to 718°C and held for 4 h, cooled at 55°C/h to 621°C and held for 16 h, and air cooled to room temperature. This heat treatment was designed to develop high fracture toughness by minimizing the presence of Laves phase and grain boundary delta phase.(9) Scanning electron microscope (SEM) examination of samples electropolished in a 5% HCl/5% $HClO_4$/ethanol solution revealed no grain boundary precipitates. Yield strength is 807 MPa at 360°C, measured using a 0.64-cm-diam. round tensile bar.

Four 12.5-mm-thick compact fracture specimens were fabricated in the L-C orientation according to the ASTM Test for Plane-Strain Fracture Toughness of Metallic Materials (E399-83). The specimens were precracked by fatigue loading, with the final stress intensity range below 20 MPa√m for each specimen, as indicated in Table II.

Table II. Stress Corrosion Cracking Test Results

Specimen Number	Precracking ΔK (MPa√m)	Displacement Rate (m/sec)	Estimated K_{Iscc} (MPa√m)
1	18.2	2.5×10^{-8}	No SCC
2	16.2	2.5×10^{-9}	<30.0
3	15.7	1.3×10^{-9}	13.2
4	15.5	2.5×10^{-10}	--

Each specimen was loaded in turn into an autoclave mounted in a screw-driven loading machine. The autoclave was continuously refreshed with PWR primary-side water at 360°C.

Each specimen was loaded in tension at a constant displacement rate, as indicated in Table II. Crack length was monitored throughout each test using the reversing d.c. potential drop technique(8) and a current of 15 amps. Current was carried by leads screwed into the center top and center bottom of the specimen. Potential was measured using leads spot-welded diagonally across the crack mouth. Load was monitored with a load cell and displacement with a displacement transducer mounted on the load rod.

Results

The results are presented graphically in Figures 1-3 and in terms of estimated K_{Iscc} in Table II. Figure 1 shows the crack length versus time profile for Specimen #1, which is typical of the crack length resolution achieved with the reversing d.c. potential drop technique. Figure 1 shows a sixth-order polynomial fit to the data. The crack length value for each data record is calculated from the elapsed time using the polynomial. The calculated crack length values are used to determine stress intensity and crack growth rate. The K_{Iscc} value is estimated from a plot of log crack growth rate versus stress intensity as the value of K for a crack growth rate equal to 2.5×10^{-11} m/sec (see Figure 2). No K_{Iscc} estimate can be made for Specimen #2 because the test was erroneously begun at a high load value.

Specimen #4 was tested at the lowest strain rate, 2.5×10^{-10} m/sec, with a test duration of 2700 hrs. At this slow displacement rate, the d.c.

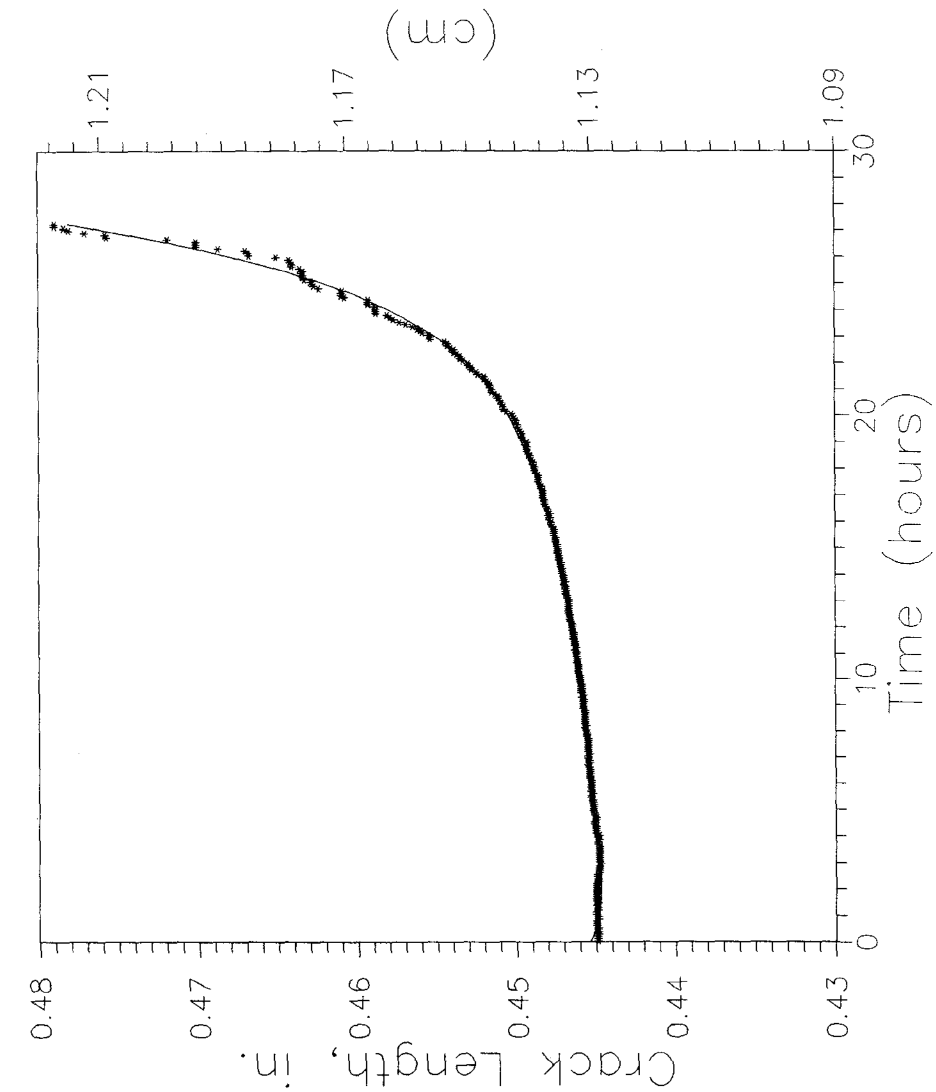

Figure 1 - Crack length versus time for Specimen #1. The solid line shows the sixth-order polynomial fit to the data.

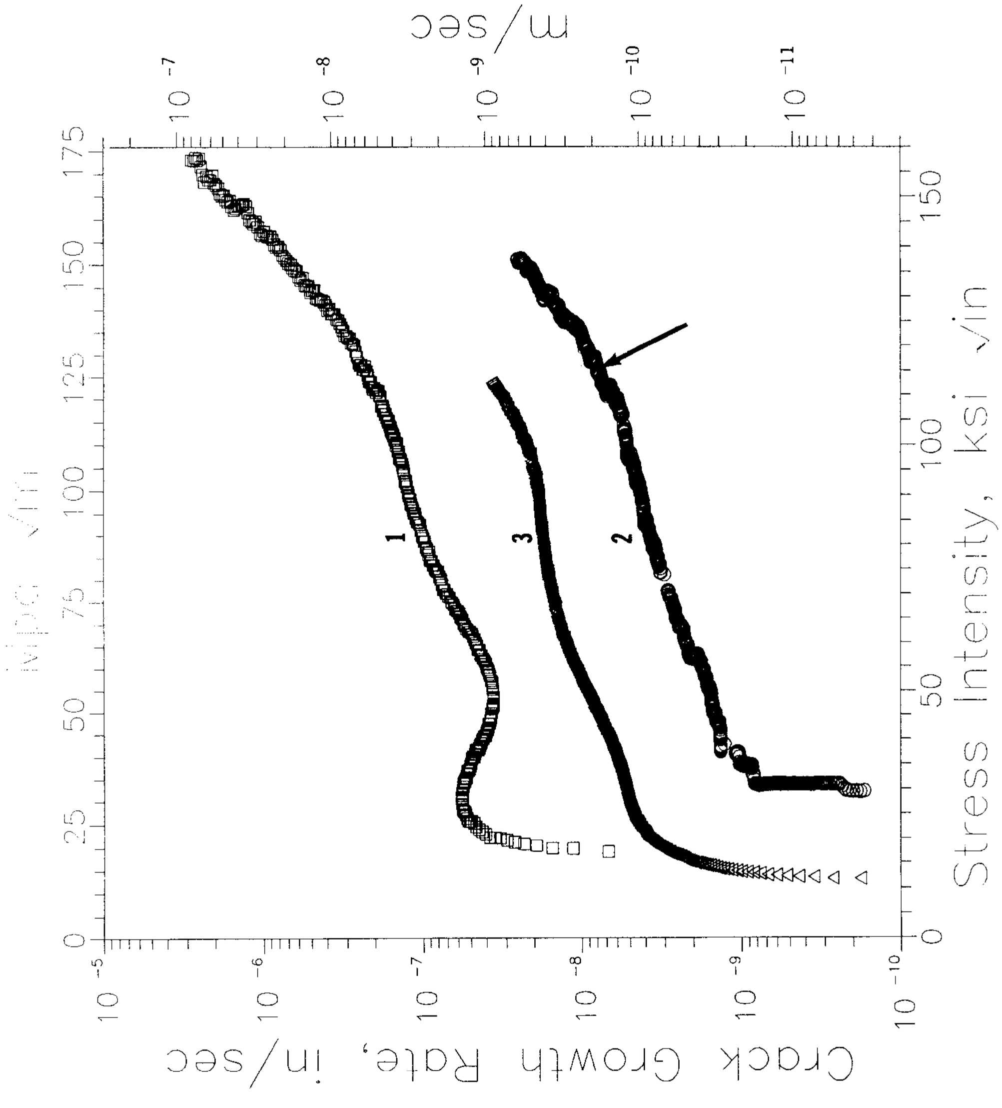

Figure 2 - Crack growth rate vs. applied stress intensity for Alloy 718 tested at: (1) 2.5 x 10^{-8} m/sec, (2) 2.5 x 10^{-9} m/sec, and (3) 1.3 x 10^{-9} m/sec. The test for Specimen #2 was inadvertently begun at 32 MPa√m. Specimen #2 cracked by SCC from 32 MPa√m to the arrow, then converted to ductile fracture.

potential drop technique did not provide accurate crack extension information, although post-test examination of Specimen #4 revealed 3.3 mm of crack extension by intergranular (IG) SCC. Figure 3 shows the crack lengths calculated from the potential drop values obtained for Specimen #4. It is possible that the crack tip was short-circuited by a process of repeated oxide formation and rupture. During oxidation the measured crack length would gradually decrease, until the oxide ruptured, abruptly increasing the crack length. Such a cyclic process of oxide formation and rupture might mask crack propagation by IGSCC.

Fractographic examination by SEM (see Figure 4) confirmed crack initiation by IGSCC for Specimens 2, 3 and 4. No SCC was observed on Specimen #1. Specimen #2 began cracking by IGSCC, and then converted to ductile tearing. Specimen #3 cracked by IGSCC up to 125 MPa√m, when loading was interrupted to allow electrochemical studies. The specimen halves were separated in air after the electrochemical studies were complete. Specimen #4 cracked by IGSCC for 3.3 mm, when cracking converted to ductile tearing.

Discussion

The results of these tests demonstrate that decreasing applied displacement rate increases susceptibility to IGSCC, in agreement with Parkins.(10) No K_{ISCC} value is available for Specimen #2; however, decreasing the displacement rate from 2.5×10^{-9} to 1.3×10^{-9} m/sec clearly increased the SCC growth rate. For a given alloy, an increase in SCC growth rate is typically associated with a decrease in K_{ISCC}.

Because of experimental difficulties with Specimen #4, the SCC growth rate is not known precisely but it can be estimated. On the fracture surface, 3.3 mm of SCC extension were measured, and from Figure 3 it appears that this crack extension occurred within the first 1600 hrs. Averaged over 1600 hrs, this results in an SCC growth rate of 5.7×10^{-10} m/sec. Following the same averaging procedure for Specimen #3 results in an average SCC growth rate of 4.8×10^{-10} m/sec. Because the applied displacement rates were different by a factor of five and similar SCC growth rates were produced, and it is likely that both specimens were tested near the critical displacement rate for maximum SCC susceptibility.(10)

The K_{ISCC} value of 13.2 MPa√m measured at a displacement rate of 1.3×10^{-9} m/sec with Specimen #3 is very low. This value can only be considered an estimate, because it is lower than the precracking ΔK and therefore may have been influenced by residual tensile stresses from fatigue precracking. If these residual tensile stresses were included in the stress intensity calculation, the actual K_{ISCC} would be somewhat greater. However, it was hypothesized that eliminating Laves and delta phase, which are deleterious to fracture toughness, from the grain boundaries of Alloy 718 would produce good SCC resistance. These initial results cast some doubt on this hypothesis.

From these results it is clear that the rising load method can be used only to estimate K_{ISCC} for this combination of alloy, heat treatment and environment. The K_{ISCC} is so low that a constant K experiment(1) is preferable. In the constant K experiment, the stress intensity is fixed at the desired value while crack growth rate is measured. Stress intensity is reduced incrementally, and crack growth rate is measured at each K level. In this way, stress intensity approaches K_{ISCC} far ahead of the precracking plastic zone.

While Specimen #1 did not undergo any SCC, it began cracking at 19 MPa√m, well below K_{IC} for this material. Using the load-displacement curve for

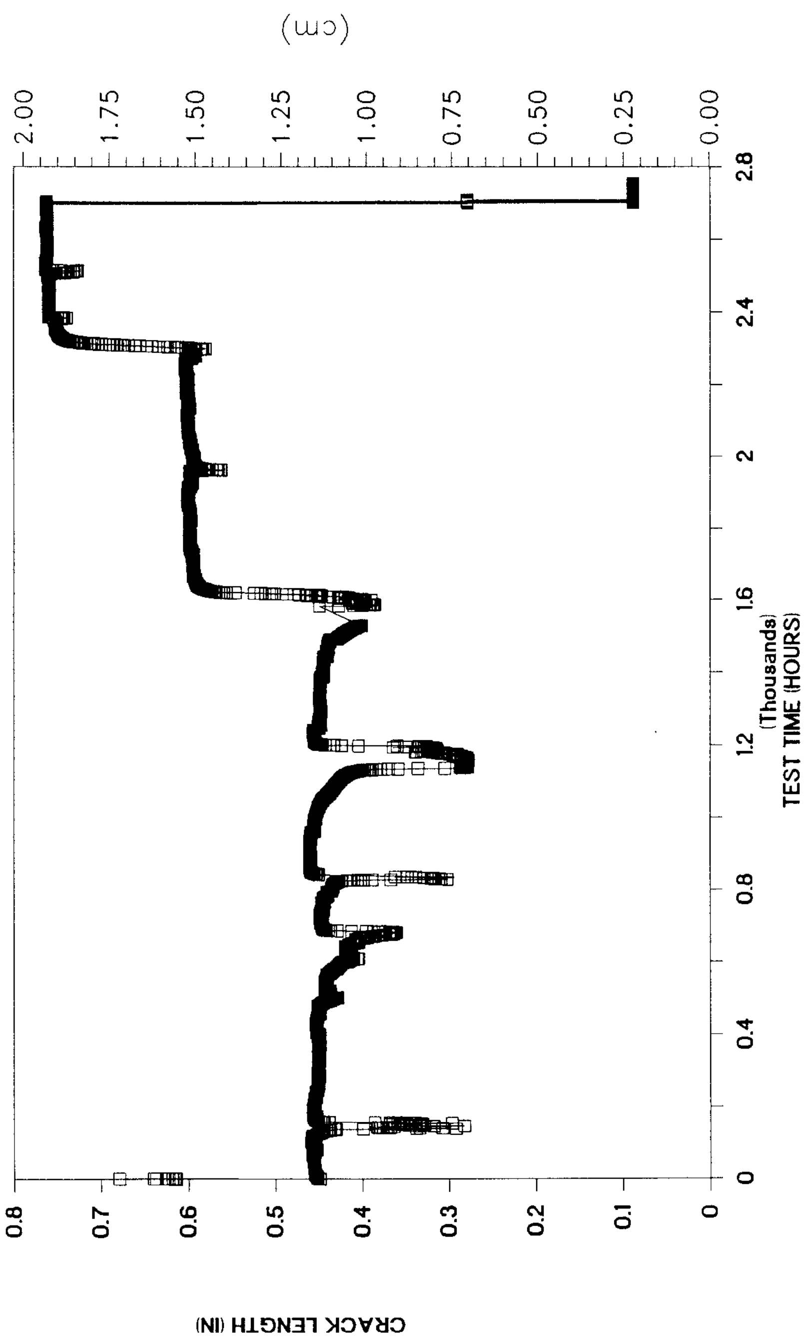

Figure 3 - Crack length vs. time record for Specimen #4. Anomalies may be caused by repeated cycles of crack tip oxidation and oxide rupture.

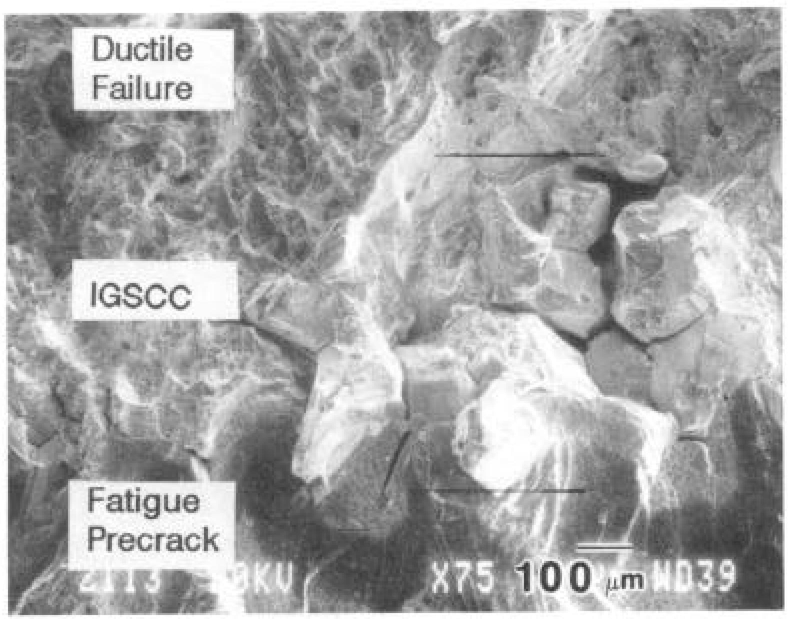

Figure 4 - SEM micrograph of Specimen #3 showing IGSCC and post-test ductile fracture.

this specimen and applying the 5% secant offset procedure of ASTM E399-83, K_{IC} is estimated at approximately 140 MPa√m. It is possible that the crack began growing at 19 MPa√m because of a corrosion-assisted tearing process. However, the observation of crack growth at such a low stress intensity level may be due to the high crack length resolution of d.c. potential drop. The 5% secant offset procedure was designed to allow approximately 2% crack extension before K_{IC}. Two percent crack extension equals 0.025 cm for this specimen, which means that the stress intensity reached K_{IC} after the specimen was in test for 23 hours. From Figure 1, due to the high crack resolution of d.c. potential drop, crack growth is observed after less than 5 hrs in test.

Conclusions

The following conclusions can be drawn regarding the behavior of precracked compact fracture specimens of Alloy 718 loaded at a constant displacement rate in 360°C PWR primary-side water:

1. An Alloy 718 heat treatment designed to produce precipitate-free grain boundaries is susceptible to IGSCC, even at low applied stress intensities.

2. Applied displacement rates of 2.5×10^{-9}, 1.3×10^{-9}, and 2.5×10^{-10} m/sec produced susceptibility to IGSCC for the heat treatment condition tested.

3. Decreasing applied displacement rate from 2.5×10^{-9} to 2.5×10^{-10} m/sec increased the IGSCC growth rate for the heat treatment condition tested.

4. Estimated K_{ISCC} for Alloy 718 in this heat treatment condition is so low as to preclude the use of the rising load test procedure for accurate K_{ISCC} determination, because of inaccuracy introduced by residual stresses from fatigue precracking.

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References

1. B. P. Miglin, M. T. Miglin, J. V. Monter, T. Sato, and K. Aoki, Proceedings of the Fourth International Symposium on Environmental Degradation of Materials in Nuclear Power Systems--Water Reactors, National Association of Corrosion Engineers, in press.

2. M. T. Miglin and H. A. Domian, J. Materials Engineering, Vol. 9, 1987, pp 113-132.

3. M. T. Miglin, EPRI Report No. NP-6392-M, June, 1989.

4. I. L. W. Wilson and T. R. Mager, Corrosion, Vol. 42, No. 6, pp. 352-361.

5. R. A. Mayville, T. W. Warren, and P. D. Hilton, J. of Testing and Evaluation, Vol. 17, No. 4, July 1989, pp. 203-211.

6. W. Dietzel, K.-H. Schwalbe, Materialpruefung, Vol. 28, No. 11, Nov. 1986, pp. 368-372.

7. R. W. Judy, Jr., W. E. King, Jr., J. A. Hauser II, and T. W. Crooker, Naval Research Laboratory Memorandum Report No. 5896, Dec. 30, 1986.

8. K.-H. Schwalbe and D. Hellmann, J. of Testing and Eval., Vol. 9, No. 3, 1981, pp. 218-220.

9. W. J. Mills, Trans. AIME, Vol. 102, Jan. 1980, pp. 118- 126.

10. R. N. Parkins, British Corrosion Journal, Vol. 14, No. 1, 1979.