Environmentally Assisted Cracking in Light Water Reactors

Semiannual Report
January 1996 - June 1996

Manuscript Completed: May 1997
Date Published: May 1997

Prepared by
O. K. Chopra, H. M. Chung, D. J. Gavenda, E. E. Gruber, T. H. Hughes,
T. F. Kassner, P. R. Luebbers, J. -H. Park, T. T. Pleune, W. E. Ruther,
W. J. Shack, W. K. Soppet, R. V. Strain

Argonne National Laboratory
9700 South Cass Avenue
Argonne, IL 60439

M. McNeil, NRC Project Manager

Prepared for
Division of Engineering Technology
Office of Nuclear Regulatory Research
U.S. Nuclear Regulatory Commission
Washington, DC 20555-0001
NRC Job Code W6610

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by

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Abstract

This report summarizes work performed by Argonne National Laboratory on fatigue and environmentally assisted cracking (EAC) in light water reactors from January 1996 to June 1996. Topics that have been investigated include (a) fatigue of carbon, low-alloy, and austenitic stainless steels (SSs) used in reactor piping and pressure vessels, (b) irradiation-assisted stress corrosion cracking of Type 304 SS, and (c) EAC of Alloys 600 and 690. Fatigue tests were conducted on ferritic and austenitic SSs in water that contained various concentrations of dissolved oxygen (DO) to determine whether a slow strain rate applied during various portions of a tensile-loading cycle are equally effective in decreasing fatigue life. Slow-strain-rate-tensile tests were conducted in simulated boiling water reactor (BWR) water at 288°C on SS specimens irradiated to a low fluence in the Halden reactor and the results were compared with similar data from a control-blade sheath and neutron-absorber tubes irradiated in BWRs to the same fluence level. Crack-growth-rate tests were conducted on compact-tension specimens from several heats of Alloys 600 and 690 in air and high-purity, low-DO water.

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Executive Summary

Environmental Effects on Fatigue Strain-versus-Life (S-N) Behavior of Primary Pressure Boundary Materials

Plain carbon steels (CSs) and low-alloy steels (LASs) are used extensively in steam supply systems of pressurized and boiling water nuclear reactors (PWRs and BWRs) as piping and pressure vessel materials. Fatigue tests are being conducted on CSs and LASs in water and in air to establish the effects of material and loading variables on fatigue life. In air, the fatigue life of CSs and LASs depends on steel type and temperature, and for some compositions, on orientation (rolling or transverse) and strain rate. Although the structure and cyclic-hardening behavior of CSs and LASs are distinctly different, they differ little or not at all in susceptibility to environmental degradation of fatigue life. The results indicate a significant decrease in fatigue life when five conditions are satisfied simultaneously, viz., when applied strain range, service temperature, dissolved-oxygen (DO) in the water, and sulfur content of the steel are above a minimum threshold level, and the loading strain rate is below a threshold value. Only a moderate decrease in fatigue life is observed in light water reactor (LWR) environments when any one threshold condition is not satisfied. This report summarizes available data on the effects of various material and loading variables, such as steel type, material orientation, DO level, strain range, strain rate, loading waveform, and surface morphology on the fatigue life of CSs and LASs in air and water environments. The existing fatigue data have been analyzed to define the threshold values of the five critical parameters. For both CSs and LASs, fatigue life in air and LWR environments can be estimated from the statistical models. Methods for evaluating the effects of LWR coolant environments on the fatigue life of CSs and LASs under actual loading histories are discussed.

Irradiation Assisted Stress Corrosion Cracking

Initial slow-strain-rate-tensile (SSRT) tests were conducted in simulated BWR water at 288°C on model stainless steel (SS) alloys that were irradiated at 288°C in helium to a fluence of $\approx 0.45 \times 10^{21}$ n-cm⁻² ($E > 1$ MeV) in the Halden reactor. Initial results indicate that the ductility of irradiated commercial-purity Type 304 SS sheet specimens is higher than that of BWR neutron-absorber tubes at a comparable fluence level. The former material appears to be more resistant to transgranular stress corrosion cracking than the latter material. High-purity (HP) Type 316 SS irradiated in the Halden reactor was somewhat more susceptible to stress corrosion cracking (SCC). This behavior is similar to that observed for HP heats of Type 304 SS.

Unusually high levels of DO in SS, either from ingot melting, component fabrication, or contamination during welding, has a deleterious effect on SCC of irradiated Type 304 SS during SSRT tests in HP oxygenated water at 288°C.

Shield-metal-arc (SMA) welds were prepared from Types 304, 304L, and 316 SS to simulate BWR core-shroud welds. Chemical composition and minor impurity contamination in the SMA and gas-tungsten-arc (GTA) welds were determined by wet-chemical analysis and secondary-ion mass spectroscopy. Significant contamination by oxygen and fluorine was observed in the fusion and heat-affected zones of the SMA welds. We attribute this contamination to the air atmosphere and CaF₂ flux in the coating on the welding electrode.

Setup and calibration of the mechanical test system for J-R fracture toughness testing was completed successfully. An extensometer with higher resolution capability was added to the system to improve control during testing. Computer hardware has been installed, and software development for computer control of the J-R tests has been initiated.

Environmentally Assisted Cracking of Alloys 600 and 690 in Simulated LWR Water

Corrosion-fatigue experiments were conducted on compact-tension specimens of annealed Alloy 600 and annealed and thermally treated Alloy 690 specimens in air and in HP deoxygenated water to investigate the effects of temperature and load ratio on crack growth rates (CGRs). Crack growth behavior of the two materials in air at 35, 130, and 289°C and in water at 240, 289, and 320°C was quite similar under the conditions in these experiments. Based on results from relatively few specimens, heat-to-heat variations and heat treatment conditions did not have a significant effect on the CGRs in air or in HP deoxygenated water.

"Best-fit" correlations for the CGR of Alloys 600 and 690 in air and water versus ΔK were obtained for each specimen and for the combined data from several specimens of the same material (different heats and/or heat treatments). The effect of HP deoxygenated water on the CGRs of each alloy relative to that in air was determined from (a) ratios of the CGRs in water and in air at 289°C, (b) from the combined data obtained at three temperatures in water and in air at 289°C, and (c) from the combined results in water and air at all of the temperatures. The results indicate that the CGRs for Alloy 600 are higher in water than in air at 289°C at ΔK values $<6 \text{ MPa}\cdot\text{m}^{1/2}$ and are lower in water than in air at higher values of ΔK . In contrast, the CGRs of Alloy 690 are only slightly higher in water than in air over the entire range of ΔK . The ratios of the CGRs from the combined data for each alloy at 240, 289, and 320°C to that in air at 289°C exhibit a similar trend. Ratios of the CGRs from the combined data for each alloy in water and air at all temperatures indicate that the CGRs of Alloy 600 in HP deoxygenated water are only slightly higher than those obtained in air over the entire range of ΔK , and that the rates for Alloy 690 are, at most, higher by a factor of 2 in water than in air at ΔK values $>10 \text{ MPa}\cdot\text{m}^{1/2}$.

The morphology of corrosion-fatigue cracks in the Alloy 600 and 690 specimens was transgranular cracking in air and HP deoxygenated water because of the strong contribution of mechanical cyclic loading in tests at load ratios of 0.1-0.8 and because of the relatively small degree of crack extension that occurred in tests at load ratios >0.8 .

Acknowledgments

The authors thank W. F. Burke, T. M. Galvin, and D. R. Perkins for their contributions to the experimental effort. This work is sponsored by the Office of Nuclear Regulatory Research, U.S. Nuclear Regulatory Commission, under Job Code W6610-6; Program Manager: Dr. M. B. McNeil. The work on artificial neural networks was performed at Argonne National Laboratory by an appointee from the Nuclear Engineering/Health Physics Fellowship program administered by the Oak Ridge Institute for Science and Education under Contract DE-AC05-76OR00033 between the U.S. Department of Energy and Oak Ridge Associated Universities; Program Manager: Sandra Johnson.

1 Introduction

Since 1967, the U.S. Nuclear Regulatory Commission (NRC) and its predecessor the U.S. Atomic Energy Commission, have conducted research programs that address aging of reactor components. The results of the research have been used to evaluate and establish regulatory guidelines to assure acceptable levels of reliability for light water reactor (LWR) components. The products of this program have been technical reports, methodologies for evaluating licensee submittals, and other inputs to the regulatory process. The results have led to the resolution of regulatory issues, as well as the development, validation, and improvement of regulations and regulatory guides. The present research on the effects of simulated reactor coolant environments on cracking of reactor components was initiated to resolve remaining critical technical issues related to cracking phenomena in LWR components. Initially, this project addressed cracking of boiling water reactor (BWR) pipes, and, subsequently, in response to the NRC Division of Nuclear Reactor Regulation (NRR) requests for assistance in dealing with developing cracking problems in aging reactors, the focus has shifted to other problems in environmentally assisted cracking (EAC) of LWR components.

The overall objective of this program is to provide data and physical models to be used by the NRC staff to assess environmentally assisted degradation of primary pressure boundary components in LWRs. The research is divided into five tasks as follows:

(1) *Environmental effects on fatigue, crack growth, and stress corrosion cracking*

Fatigue and EAC of piping, pressure vessels, and core components in LWRs are important concerns in plant operation and for extended reactor lifetimes. The degradation processes in U.S. reactors include fatigue, intergranular stress corrosion cracking (IGSCC), and propagation of fatigue or stress corrosion cracks, which initiate in the weld-sensitized heat-affected zone (HAZ) of stainless steel (SS) components. Occurrences of mechanical-vibration- and thermal-fluctuation-induced fatigue failures in LWR plants have also been documented. The objective of this task is to provide improved fatigue design curves and an assessment of the additivity of fatigue damage in piping and vessel steels under load histories typical of LWR components. The results of this work will be used to assess industry fatigue evaluations related to license renewal and advanced designs.

(2) *Component vulnerability to irradiation-assisted stress corrosion cracking*

Irradiation-assisted stress corrosion cracking (IASCC) of in-core components of both BWRs and pressurized-water reactors (PWRs) is becoming a more common problem as reactors age. The general pattern of the observed failures indicates that, as nuclear plants age and neutron fluence increases, many apparently nonsensitized austenitic materials become susceptible to intergranular failure by IASCC. Some of these failures have been reported for components subjected to relatively low or negligible stress levels, e.g., control-blade sheaths and handles and instrument dry tubes of BWRs. Although most failed components can be replaced, some safety-significant structural components, such as the BWR top guide, core plate, and shroud, would be very difficult or impractical to replace. The objective of this task is to provide data and models that are needed to assess industry analyses of the likelihood of degradation and failure of core internal components due to IASCC, and to evaluate licensee submissions concerning inspection and remediation.

(3) *Cracking of nickel alloy components of LWR primary systems*

Many reactor vessel internal components are made of nickel-base alloys, such as Alloys 600, X750, and 182, which are susceptible to IGSCC. The causes and mechanisms of this cracking are not adequately understood and lead to greater uncertainty in licensee submissions as they are related to issues such as damage accumulation and inspection intervals. The objective of this task is to provide technical data on the implications of cracks in nickel-alloy components with regard to residual life, inspection, and repair. The results will be used to support NRR staff assessments of industry crack growth models, and potential detection and mitigation measures.

(4) *Analyses of postweld heat treatment processes and validation of flaw acceptance criteria*

The objective of this task is to evaluate the effect of postweld heat treatment on the long-term resistance to environmental cracking by assessing sensitization and other microstructural changes. Emphasis is to be placed on components made of welded Alloy 600. This evaluation will provide NRC with insights for use in reviewing licensee submittals.

(5) *Assess industry crack-growth models*

This task has two objectives. The first is to perform an independent evaluation of industry models that are used to establish inspection intervals and repair criteria. The second objective is to perform more detailed analyses of flaw acceptance criteria.

Research during the past six months has focused on fatigue of carbon steels (CSs) and low-alloy ferritic steels (LASs) and SSs used in piping and pressure vessels, IASCC in slow-strain-rate tensile (SSRT) tests in simulated BWR water of SS specimens that were irradiated to low a low fluence in the Halden reactor, and EAC of Alloys 600 and 690 in high-purity (HP) deoxygenated water and in air.

2 Environmental Effects on Fatigue Strain-versus-Life (S-N) Behavior of Primary Pressure Boundary Materials

The ASME Boiler and Pressure Vessel Code Section III, Subsection NB,¹ which contains rules for the construction of Class 1 components for nuclear power plants, recognizes fatigue as a possible mode of failure in pressure vessel steels and piping materials. Cyclic loadings on a structural component occur because of changes in the mechanical and thermal loadings as the system goes from one load set (e.g., pressure, temperature, moment, and force loading) to any other load set. For each pair of load sets, an individual fatigue usage factor is determined by the ratio of the number of cycles anticipated during the lifetime of the component to the allowable cycles. Figures I-9.1 to I-9.6 of Appendix I to Section III of the Code specifies fatigue design curves that define the allowable number of cycles as a function of applied stress amplitude. The cumulative usage factor (CUF) is the sum of the individual usage factors. The ASME Code Section III requires that the CUF at each location must not exceed 1.

The Code design fatigue curves were based on strain-controlled tests of small polished specimens at room temperature in air. In most studies, the fatigue life of a test specimen is defined as the number of cycles needed for the tensile stress to drop 25% from its peak value, which corresponds to a ≈ 3 -mm-deep crack. Consequently, fatigue life N represents the

number of cycles required to initiate a crack ≈ 3 mm deep. The best-fit curves to the experimental data were expressed in terms of the Langer equation² of the form

$$\epsilon_a = B(N)^{-b} + A, \quad (1)$$

where A, B, and b are parameters of the model (Eq. 1 may be written in terms of stress amplitude S_a instead of strain amplitude ϵ_a , in which case stress amplitude is the product of strain amplitude and elastic modulus, i.e., $S_a = E \cdot \epsilon_a$). The design fatigue curves were obtained by decreasing the best-fit curves by a factor of 2 on stress or 20 on cycles, whichever was more conservative, at each point on the best-fit curve. As described in the Section III criteria document, these factors were intended to account for the differences and uncertainties in relating the fatigue lives of laboratory test specimens to those of actual reactor components. The factor of 20 on cycles is the product of three separate subfactors: 2 for scatter of data (minimum to mean), 2.5 for size effects, and 4 for surface finish, atmosphere, etc.³ "Atmosphere" was intended to reflect the effects of an industrial environment rather than the controlled environment of a laboratory. The factors of 2 and 20 are not safety margins but rather conversion factors that must be applied to the experimental data to obtain reasonable estimates of the lives of actual reactor components. They were not intended to address the effects of reactor coolant environments on fatigue life.

Subsection NB-3121, of Section III of the Code states that the data on which the fatigue design curves (Figs. I-9.1 to I-9.6) are based did not include tests in the presence of corrosive environments that might accelerate fatigue failure. Article B-2131 in Appendix B to Section III states that the owner's design specifications should provide information about any reduction to fatigue design curves necessitated by environmental conditions. Recent fatigue S-N data illustrate potentially significant effects of LWR coolant environments on the fatigue resistance of CSs, and LASs,⁴⁻¹⁰ and austenitic SSs.¹¹ Therefore, the margins in the ASME Code may be less conservative than originally intended.

Experience with operating nuclear plants worldwide reveals that many failures may be attributed to fatigue. Examples of such failures include: emergency core cooling or residual heat removal systems (USNRC Bulletin No. 88-08), pressurizer surge lines (USNRC Bulletin No. 88-11), PWR feedwater lines (USNRC Information Notice No. 79-13), BWR pressure vessels (USNRC Information Notice No. 90-29), PWR steam generator vessels (USNRC Information Notice No. 90-04), and steam generator feedwater distribution piping (USNRC Information Notice No. 91-19 and No. 93-20). These failures may be classified into three categories: thermal fatigue caused by thermal stratification, cycling, and striping loadings; mechanical fatigue due to vibratory loading; and corrosion fatigue from exposure to a corrosive environment. Significant thermal loadings due to stratification were not included in the original design basis analysis. Some of these fatigue sensitive locations are routinely monitored in nuclear power plants worldwide to better define the transients and more accurately assess CUF.

In 1991, the USNRC issued a draft Branch Technical Position (BTP) for fatigue evaluation of nuclear plant components for license renewal. The BTP raised a concern about the adequacy of the ASME Code in addressing the effects of environment on fatigue resistance of materials in operating PWRs and BWRs, whose primary-coolant pressure-boundary components are constructed as specified in Section III of the Code. Researchers at Argonne National Laboratory (ANL) have compiled and reviewed the existing fatigue S-N data to identify

key variables that influence the fatigue life of primary pressure boundary materials.^{8,9} A program was initiated at ANL to obtain fatigue data under conditions where information was lacking in the existing data base. The results from the study have been presented in previous progress reports.¹²⁻¹⁶ Based on the existing S-N data, ANL researchers have developed interim design fatigue curves that address the effects of environment on fatigue life of CSs, LASs, and austenitic SSs.¹⁷ Statistical models have also been developed for estimating the effects of various material and loading conditions on fatigue life of these materials.¹⁸ Results of the statistical analysis have been used to estimate the probability of fatigue cracking in reactor components. The Pressure Vessel Research Council has also been compiling and evaluating fatigue S-N data related to the effects of LWR coolant environments on the fatigue life of pressure boundary materials; the results have been presented by Van Der Sluys.¹⁹

The NRC, in 1993, directed its staff to treat fatigue as a potential safety issue within the existing regulatory process for operating reactors. The staff developed a Fatigue Action Plan (FAP) to resolve three principal issues: (a) adequacy of fatigue resistance of older vintage plants designed to the United States of America Standard (USAS) B31.1 Code that did not require an explicit fatigue analysis of components, (b) the effect of LWR environments on the fatigue resistance of primary pressure boundary materials, and (c) the appropriate corrective action required when the Code fatigue allowable limits have been exceeded, i.e., CUF is >1 . The Idaho National Engineering Laboratory assessed the significance of the interim fatigue design curves by performing fatigue evaluations of several components in the reactor coolant pressure boundary.²⁰ In all, six locations were evaluated from facilities designed by each of the four U.S. nuclear steam supply system vendors. Selected components from older vintage plants designed to comply to B31.1 Code were also included in the evaluation. The risk to reactor coolant pressure boundary components from failure due to fatigue was assessed under Generic Safety Issue (GSI) 78, "Monitoring of Fatigue Transient Limits for the Reactor Coolant System." On the basis of these studies, it was concluded* that no immediate action is necessary to deal with fatigue issues addressed in the FAP. The risk study indicated that a fatigue failure of piping is not a significant contributor to core-melt frequency. Although fatigue cracks may occur, they may not propagate to failure and, even if failure did occur, safety systems, such as emergency core cooling system, mitigate the consequences. On the basis of the risk assessment, a backfit of the environmental fatigue data to operating plants cannot be justified.

The objective of this task is to provide data and models for predicting environmental effects on fatigue design curves and to assess the additivity of fatigue damage in piping and vessel steels under load histories typical of LWR components. The data will be used by the NRC staff to assess industry fatigue evaluations related to license renewal and advanced designs. The information, which will be helpful in assessing applicant submissions with regard to residual lifetimes of components subjected to fatigue in reactor coolant environments, and will be provided to ASME to help improve the design curves. Current understanding of the effects of LWR coolant environments on the fatigue life of pressure vessel and piping materials are summarized in this report. Fatigue tests are being conducted on pressure boundary steels to establish the effects of various loading and environmental variables on their fatigue S-N behavior. Progress in this task during the current reporting period is presented.

* Policy Issue, SECY-95-245, Completion of the Fatigue Action Plan, Sept. 25, 1995.

2.1 Carbon and Low-Alloy Ferritic Steels (O. K. Chopra)

The sources of fatigue S-N data for CSs and LASs are the tests performed by General Electric Co. (GE) in a test loop at the Dresden 1 reactor^{21,22} and by the Electric Power Research Institute;²³ the work of Terrell at Mechanical Engineering Associates (MEA);²⁴⁻²⁶ the program at ANL on fatigue of pressure vessel and piping steels;^{8,9,21-26} and the studies in Japan,^{6,7,27-31} including the JNUFAD[†] data base for "Fatigue Strength of Nuclear Plant Components." The ASME Specifications for chemical and tensile strength requirements for CSs and LASs are listed in Table 1. The effects of various loading and environmental variables on the fatigue life of CSs and LASs are summarized below.

2.1.1 Air Environment

The fatigue life of CSs and LASs in air depends on steel type, temperature, orientation, and strain rate. The fatigue life is greater and fatigue endurance limit is higher for LASs than it is for CSs. For both steels, fatigue life decreases as temperature increases. Some steels show very poor fatigue properties in the transverse orientation. Structural factors, such as distribution and morphology of sulfides, are responsible for the poor fatigue resistance of steels in transverse orientations. The results also indicate that some heats of CS and LAS are sensitive to strain rate, e.g., the fatigue life decreases with decreasing strain rate.

The cyclic stress-strain response of CSs and LASs varies with steel type, temperature, and strain rate. In general, these steels show initial cyclic hardening followed by cyclic softening or a saturation stage at all strain rates. The CSs with a pearlite and ferrite structure and low yield stress show significant initial hardening. The LASs, with a tempered ferrite and bainitic structure and a relatively high yield stress, show little or no initial hardening and may exhibit cyclic softening during testing.

At 200–370°C, both steels exhibit dynamic strain aging that results in enhanced initial hardening and a secondary hardening stage.^{8,9,32-34} The temperature range and extent of dynamic strain aging vary with the composition and structure of the steel. Under conditions of dynamic strain aging, cyclic stress increases with decreases in strain rate. However, the fatigue life of the steel may increase or decrease.^{32,34} The decreases in fatigue life have been explained on the basis that inhomogeneous plastic deformation leads to heavy localized plastic strains, and that restricted plasticity retards blunting of propagating cracks and leads to higher crack growth rates.³² The increases in fatigue life have been attributed to retardation of crack growth rates due to crack branching and suppression of the plastic zone, although formation of cracks is easy in the presence of dynamic strain aging.³⁴

The cyclic stress-versus-strain curves for CSs, expressed in terms of the total strain range $\Delta\epsilon_t$ (%) and cyclic stress range $\Delta\sigma$ (MPa) at half life,^{8,9,16} can be represented with the equation

[†] Private communication from M. Higuchi, Ishikawajima-Harima Heavy Industries Co., Japan, to M. Prager of the Pressure Vessel Research Council, 1992. The old data base "FADAL" has been revised and renamed "JNUFAD."

Table 1. Chemical and strength specifications for carbon and low-alloy steels

Variable	SA-106 Grade B	SA-333 Grade 6	SA-516 Grade 70	SA-508 Class 1a	SA-508 Class 2	SA-508 Class 3	SA-302 Grade B	SA-533 Grade B
Steel Type	Carbon	Carbon	Carbon	Carbon	Alloy	Alloy	Alloy	Low-Alloy
Product	Seamless Pipes	Seamless & Welded Pipes	PV Plates	PV Forgings	PV Forgings	PV Forgings	PV Plates	PV Plates
C max. (%)	0.25 ^a	0.30 ^b	0.27-0.31 ^c	0.30	0.27	0.25	0.20-0.25 ^c	0.25
Cr (%)	0.40 max. ^d	-	-	0.25 max.	0.25-0.45	0.25 max.	-	-
Cu (%)	0.40 max. ^d	-	-	-	-	-	-	-
Mn (%)	0.27-0.93	0.29-1.06	0.79-1.30	0.70-1.35	0.50-1.00	1.20-1.50	1.07-1.62	1.07-1.62 ^e
Mo (%)	0.15 max. ^d	-	-	0.10 max.	0.55-0.70	0.45-0.60	0.41-0.64	0.41-0.64
Ni (%)	0.40 max. ^d	-	-	0.40 max.	0.50-1.00	0.40-1.00	-	0.37-0.73
P max. (%)	0.025	0.048	0.035	0.025	0.025	0.025	0.035	0.035
S max. (%)	0.025	0.058	0.040	0.025	0.025	0.025	0.040	0.040
Si (%)	0.10 min.	0.10 min.	0.13-0.45	0.15-0.40 ^f	0.15-0.40 ^e	0.15-0.40 ^e	0.13-0.45	0.13-0.45
V (%)	0.08 max. ^d	-	-	0.05 max.	0.05 max.	0.05 max.	-	-
Tensile Strength (MPa)	415 min.	415 min.	485-620	485-655	550-725	550-725	550-690	550-690 ^g 620-795 690-860
Yield Strength (MPa)	240 min.	240 min.	260 min.	250 min.	345 min.	345 min.	345 min.	345 min. ^g 485 min. 570 min.
Heat Treatment ^h	1	2	3	4	4	4	3	5

^a For each reduction of 0.01% below 0.30%, an increase of 0.05% Mn above 1.06% is permitted up to a maximum of 1.35% Mn.

^b For each reduction of 0.01% below the specified C maximum, an increase of 0.06% Mn above the specified maximum is permitted up to a maximum of 1.35%.

^c Maximum amount increases with increasing section thickness.

^d These five designated elements combined shall not exceed 1%.

^e The maximum Mn content may be increased to 1.65% on product analysis when Class 2 and Class 3 properties are specified and when Supplementary Requirement S3 is specified.

^f When vacuum carbon-deoxidation is required by Requirement S11, the Si content shall be 0.10% maximum.

^g The three sets of numbers correspond to Class 1, 2, and 3 strength levels.

^h Heat treatments for the various steels are as follows:

1. Hot-finished pipe need not be heat treated, and cold-drawn pipe shall be heat treated at 650°C or higher.
2. All seamless and welded pipes shall be treated to control their microstructure in accordance with one of the following: normalize and temper, quench and temper, or double-normalize and temper.
3. Plates 40 mm and under in thickness are normally supplied in the as-rolled condition. They may be ordered normalized or stress-relieved, or both. Plates over 40 mm in thickness shall be normalized.
4. The forgings shall be heated to a temperature that produces an austenite structure and then quenched in a suitable liquid medium by spraying or immersion. Quenching shall be followed by tempering at a subcritical temperature and holding for a time of 1/2 h per in. of maximum section thickness.
5. All plates shall be heat treated at 845-980°C, holding for sufficient time to obtain uniform temperature and then quenching in water. Subsequently, the plates shall be tempered at a suitable temperature not less than 595°C, with a minimum holding time of 1/2 h per in. of thickness, but not less than 1/2 h.

$$\Delta\epsilon_t = \frac{\Delta\sigma}{1965} + \left(\frac{\Delta\sigma}{C} \right)^{7.74} \quad (2a)$$

where the constant C depends on applied strain rate $\dot{\epsilon}$ (%/s) and is expressed as

$$C = 1080 - 50.9 \text{Log}(\dot{\epsilon}). \quad (2b)$$

The cyclic stress-versus-strain curve for LASs can be represented with the equation

$$\Delta \epsilon_t = \frac{\Delta \sigma}{1965} + \left(\frac{\Delta \sigma}{D} \right)^{9.09} \quad (3a)$$

where the constant D is expressed as

$$D = 962 - 30.3 \text{Log}(\dot{\epsilon}). \quad (3b)$$

For both steels, the cyclic stress response at room temperature and at temperatures outside the regime of dynamic strain aging is lower than that at 288°C, i.e., cyclic stresses are higher at 288°C than at room temperature.

The fatigue life of these steels, defined as the number of cycles to form of a 3-mm-deep crack, may be estimated from a statistical model.¹⁸ In air, the fatigue life N of CSs is expressed as

$$\ln(N) = 6.570 - 0.00133 T - 1.871 \ln(\epsilon_a - 0.11) \quad (4a)$$

and that of LASs, as

$$\ln(N) = 6.667 - 0.00133 T - 1.687 \ln(\epsilon_a - 0.15), \quad (4b)$$

where applied strain amplitude ϵ_a is in % and temperature T is in °C.

The fatigue lives of CSs and LASs in air at room temperature and 250-300°C are shown in Fig. 1 with the values estimated from Eqs. 4a and 4b. The results indicate significant heat-to-heat variation. At 250-300°C, fatigue life may vary up to a factor of 5 above or below the mean value. The large variation in life at high temperature may be due to differences in the extent of dynamic strain aging in the steel. The existing fatigue S-N data base for CSs and LASs does not include information about the size and distribution of inclusions in the steel. Consequently, the effect of orientation could not be incorporated into the statistical model. Furthermore, because of conflicting possibilities of the effect of strain rate on fatigue life, e.g., life may be either unaffected,^{8,9,16} increased for some steels,³⁴ or decreased for others,^{15,32} strain rate effects were not explicitly considered in the statistical model.

The results also indicate that the ASME mean curve for CSs is not consistent with the experimental data; at strain amplitudes <0.2%, the mean curve predicts significantly lower fatigue lives than those observed experimentally. The estimated curve for LASs is comparable to the ASME mean curve. For both steels, Eq. 4 shows good agreement with the average curves of Higuchi and Iida.⁶

2.1.2 LWR Environments

The fatigue data in LWR environments indicate significant decrease in fatigue life of CSs and LASs when five conditions are satisfied simultaneously, viz., when applied strain range,

Figure 1. Fatigue S-N behavior for CSs and LASs in air at room temperature and 250-300°C

service temperature, dissolved oxygen (DO) in the water, and sulfur content of the steel are above a minimum threshold level, and the loading strain rate is below a threshold value.^{6-9,29,31} The actual dependence of fatigue life on these variables involves complex synergistic interactions. Also, studies on the effect of high-temperature aqueous environments on fatigue crack growth behavior of LASs indicate that flow rate is an important parameter for environmental effects on fatigue life in water.³⁵⁻³⁸ However, experimental data to establish either the dependence of fatigue life on flow rate or the threshold flow rate for environmental effects on fatigue life are not available.

The environmental degradation of the fatigue life of CSs and LASs is identical, although the microstructures and cyclic-hardening behaviors of these steels differ significantly.^{8,9} For both steels, environmental effects on fatigue life are minimal if any one of the threshold conditions is not satisfied. The effects of the five critical parameters on fatigue life are discussed below in greater detail to define the threshold values.

Temperature

The results for CSs indicate a threshold temperature of 150°C, above which environment may decrease fatigue life if all other threshold conditions are satisfied.^{9,31} In the temperature range of 150–320°C, fatigue life decreases linearly with temperature. Fatigue life decreases more rapidly as sulfur content in steel and DO level in water increases, or as strain rate decreases. Available data suggest that these results may also be applicable to LASs.

Strain Rate

A slow strain rate applied during the tensile-loading cycle is primarily responsible for environmentally assisted reduction in fatigue life.^{7,8,16} Slow strain rate applied during both tensile- and compressive-loading cycles does not cause further decrease in fatigue life. However, limited data indicate that a slow strain rate during the compressive-loading cycle may also decrease fatigue life;^{7,16} however, the decrease in life is relatively small. The S-N data indicate that, for strain rates <1%/s, fatigue life decreases rapidly with decreasing strain rate. Strain rates above 1%/s have little or no effect on fatigue life of CSs and LASs in LWR environments.^{7-9,29} Limited data indicate that, for both steels, the effect of strain rate on fatigue life saturates at $\approx 0.001\%/s$ strain rate, i.e., decreases in strain rate below 0.001%/s do not cause further decrease in fatigue life.⁸ At strain rates between 1 and 0.001%/s, fatigue life decreases logarithmically with strain rate.

Dissolved Oxygen

The results indicate a minimum DO level of 0.05 ppm, above which environment decreases the fatigue life of the steel.^{28,31} The effect of DO content on fatigue life saturates at 0.5 ppm, i.e., increases in DO levels above 0.5 ppm do not cause further decrease in fatigue life.⁹ For DO levels between 0.05 and 0.5 ppm, fatigue life decreases logarithmically with DO.

Sulfur Content

The available data are not adequate to establish the dependence of fatigue life on sulfur content and either a lower threshold for sulfur content below which environmental effects are unimportant or an upper limit above which the effect of sulfur on fatigue life may saturate. Limited data suggest that environmental effects on fatigue life may saturate at sulfur contents above 0.015 wt.%. Existing fatigue S-N data suggest a threshold value of 0.003 wt.% sulfur in steel for environmental effects on fatigue life.¹⁶ The probability of environmental enhancement of fatigue crack growth rates (CGRs) in precracked specimens appears to diminish markedly for sulfur contents <0.005 wt.%,³⁸⁻⁴⁷ but enhanced growth rates have been observed in isolated heats with very low sulfur levels.*

* Fatigue Properties of Carbon Steel and Weldments in Oxygenated High Temperature Water – Evaluation of Effects of Tensile Strength and Sulfur Content, presented at the PVRC Spring Meeting, June 5–7, 1995, Milwaukee, by Mr. M. Higuchi, Ishikawajima-Hirima Heavy Industries, Japan.

Strain Amplitude

A minimum threshold strain is required to obtain an environmentally assisted decrease in fatigue life. This threshold value most likely depends both on material parameters such as amount and distribution of sulfides, and on parameters such as temperature, strain rate, and DO level in water. Limited data indicate that, for some heats of CS and LAS, the threshold strain range appears to be at $\approx 0.36\%$.^{8,9,13} This behavior is consistent with the slip-dissolution model⁴⁸ for crack propagation; the applied strain must exceed a threshold value to rupture the passive surface film for environmental effects to occur.

Low Dissolved Oxygen or Temperature

Only a moderate decrease in fatigue life is observed in water when any one of the threshold conditions is not satisfied, e.g., at temperatures below 150°C or in low-DO PWR environments (<0.05 ppm DO). For both CSs and LASs, fatigue lives in a PWR environment are lower than those in air by a factor of <2 . Furthermore, in PWR water, the effects of orientation and strain rate are similar to those in air;¹⁶ heats of CS and LAS that are sensitive to strain rate in air, also show a decrease in fatigue life in PWR water with decreasing strain rate. For some heats, fatigue life decreased by a factor of ≈ 4 when strain rate decreased from 0.4 to $0.004\%/s$.¹⁵ On the other hand, for some heats, a decrease in the strain rate by three orders of magnitude does not cause an additional decrease in fatigue life.^{8,9} The results also suggest that even the high-sulfur steels, which exhibit enhanced crack growth rates in PWR water,⁴⁹ show only modest decreases of fatigue life in low-DO PWR water.^{8,9,31} Similar behavior is expected in water at temperatures below 150°C , even when the DO levels are above 0.05 ppm and strain rates are below $1\%/s$.¹⁶

Estimation of Fatigue Life

The fatigue life of CSs and LASs may be estimated from a statistical model.¹⁸ For service conditions that satisfy all critical threshold values, e.g., temperatures $>150^\circ\text{C}$, DO levels >0.05 ppm, strain rates $<1\%/s$, and sulfur content and strain range above the critical threshold values, the fatigue life N of CSs is expressed as

$$\ln(N) = 6.186 - 1.871 \ln(\epsilon_a - 0.11) + 0.1097 S^* T^* O^* \dot{\epsilon}^* \quad (5a)$$

and that of LASs as

$$\ln(N) = 5.901 - 1.687 \ln(\epsilon_a - 0.15) + 0.1097 S^* T^* O^* \dot{\epsilon}^*, \quad (5b)$$

where

ϵ_a = the applied strain amplitude %,

T = the test temperature $^\circ\text{C}$, and

S^* , T^* , O^* , and $\dot{\epsilon}^*$ = transformed sulfur content, temperature, DO, and strain rate, respectively, defined as follows:

$$\begin{aligned} S^* &= S & (0 < S \leq 0.015 \text{ wt.}\%) \\ S^* &= 0.015 & (S > 0.015 \text{ wt.}\%) \end{aligned} \quad (6a)$$

$$\begin{aligned} T^* &= 0 & (T < 150^\circ\text{C}) \\ T^* &= T - 150 & (T = 150\text{--}350^\circ\text{C}) \end{aligned} \quad (6b)$$

$$\begin{aligned} O^* &= 0 & (\text{DO} < 0.05 \text{ ppm}) \\ O^* &= \ln(\text{DO}/0.04) & (0.05 \text{ ppm} \leq \text{DO} \leq 0.5 \text{ ppm}) \\ O^* &= \ln(12.5) & (\text{DO} > 0.5 \text{ ppm}) \end{aligned} \quad (6c)$$

$$\begin{aligned} \dot{\epsilon}^* &= 0 & (\dot{\epsilon} > 1 \text{ \%}/\text{s}) \\ \dot{\epsilon}^* &= \ln(\dot{\epsilon}) & (0.001 \leq \dot{\epsilon} \leq 1 \text{ \%}/\text{s}) \\ \dot{\epsilon}^* &= \ln(0.001) & (\dot{\epsilon} < 0.001 \text{ \%}/\text{s}) \end{aligned} \quad (6d)$$

The functional forms for S^* , T^* , O^* , and $\dot{\epsilon}^*$ were defined on the basis of the experimental data. The fatigue lives in high-DO water at 288°C are presented in Fig. 2 together with the estimated values. The predicted fatigue lives show good agreement with the experimental results.

The last term in Eqs. 5a and 5b is zero when any one of the critical threshold conditions is not satisfied, e.g., in low-DO (DO levels <0.05 ppm) PWR environments, or at temperatures <150°C, or strain rates >1%/s.

Figure 2. Fatigue S-N behavior of A106-Gr B and A533-Gr B steels, estimated from a statistical model and determined experimentally in high-DO water at 288°C

2.2 Effects of Loading Conditions on Fatigue Life (T. T. Pleune and O. K. Chopra)

As was discussed in the previous section, the fatigue life of CSs and LASs is significantly decreased in LWR environments when all threshold conditions for strain range, temperature, DO, sulfur content, and strain rate are satisfied simultaneously.^{6-9,29,31} The actual dependence of fatigue life on these variables involves complex synergistic interactions. The functional forms for the dependence of fatigue life on these variables often cannot be established from existing fatigue data because complete data sets, where one parameter is varied while others are kept constant, are required. Although the existing fatigue S-N data base covers an adequate range of material, loading, and environmental parameters, it provides incomplete data sets for most parameters. For example, fatigue tests at temperatures between room temperature and 300°C may not be conducted on the same heat of material or at the same strain rate.

Artificial neural networks (ANNs) have great potential for prediction of environmentally assisted fatigue cracking. Their main benefits are that, with ANNs, estimates of life are based purely on data and not on preconceptions, and that the network can interpolate effects by learning trends and patterns when data are not available. The factors that affect fatigue life can exert synergistic effects on one another. An ANN that can detect and utilize these effects in its predictions was used to find patterns and identify thresholds in fatigue S-N data for CSs and LASs in LWR environments. The results are compared with estimates of life based on a statistical model.¹⁸ The benefits and disadvantages of using an ANN to predict fatigue lives of materials are discussed.

2.2.1 Artificial Neural Networks

An ANN is an information-processing system whose structure is modeled on the biological neural structure. Borrowing from biology, we will call each unit or node of the network a neuron. Each neuron in an ANN receives input from several others. These inputs are multiplied by different weights and combined. This combination and an activation function determine the output, which is passed on to the next layer of neurons.

The type of ANN* that was used for this study is known as a backpropagation network. Backpropagation describes the learning algorithm not the network structure. The structure can vary as long as it is hierarchical, and highly connected. "Hierarchical" means that the ANN is created with at least two, but usually three or more, layers of neurons that consist of an input layer, an output layer, and one or two hidden layers. If the ANN contains three or more hidden layers, it is much more difficult and time-consuming to train.⁵⁰ "Highly connected" means that each neuron receives input from most of the neurons in the preceding layer and each output is used by most neurons in the following layer. Backpropagation ANNs, including the one used in this study, are usually fully connected.⁵¹

The ANN, as implemented by the Neuralyst software, can be explained in terms of Fig. 3. For this study, there were six inputs and one output. There were two hidden layers, with the first containing ten nodes and the second containing six. The output values of the neurons in the input layer are simply the input data. The output value of each neuron in the subsequent layers is determined as follows:⁵¹

- (a) For each pathway in Fig. 3, an input value O_i is multiplied by a weight w_{ij} .
- (b) The products of inputs and weights are added to give a sum U_j .
- (c) The sum U_j is added to a threshold value t_j .
- (d) An activation function $F(U_j + t_j)$ is applied; the result is the output to the next layer O_j .

This process continues at each node and layer until the output for the entire ANN is determined. Thus, the output to the next layer is expressed as

$$O_j = F [\Sigma (O_i + w_{ij}) + t_j], \quad (7)$$

* Neuralyst version 1.4 from Chesire Engineering Corporation was used with Microsoft Excel for Macintosh.

Figure 3.
Schematic representation of fully connected neural network with four input neurons, two hidden layers, and one output neuron

where the form of the function F is sigmoidal. The sigmoid function gives a number between 0 and 1 and quickly saturates when its input is a large positive or negative number.

The backpropagation process trains the network after each output from the final layer. The learning process is called backpropagation because it begins with the output layer and works backwards to the first hidden layer. For each input to the neuron, a new weight w_{ij} is expressed by the equation

$$w_{ij} = w'_{ij} + LR e_j O_i, \quad (8)$$

where w'_{ij} is the old weight, LR is the learning rate, e_j is the error of the output, and O_i is the input value that corresponds to the weight being adjusted. The learning rate is an important factor because it controls the amount by which the weight is adjusted. If the learning rate is too high (nearly unity), the ANN might overshoot the result and be difficult to train. If the learning rate is too low (less than 0.1), the ANN may train too slowly. For the present study, the learning rate was 0.85 for early training and was reduced to 0.1 as the error in the data became low.

Another factor that can improve ANN training is momentum M . To prevent the ANN from arriving at a solution that is a local, but not the true, minimum, previous weight adjustments are allowed to persist to smooth the training. To include the momentum term, Eq. 8 is modified as follows:

$$w_{ij} = w'_{ij} + (1-M) LR e_j O_i + M(w'_{ij} - w''_{ij}). \quad (9)$$

A momentum of 0.3 was chosen. The error for the output neurons is determined by comparing the output values with a given target value, and is expressed as

$$e_j = O_j (1 - O_j) (d_j - O_j), \quad (10)$$

where d_j is the target value for each output. Equation 10 can only be determined for the output layer, because there are no target values for the hidden layers. The error for the hidden layers is determined from the errors e_k and the old weights w'_{jk} , which correspond to the layer immediately succeeding the current layer, i.e.,

$$e_j = O_j (1 - O_j) \sum (e_k w'_{jk}). \quad (11)$$

This is the most important equation in backpropagation ANNs because it facilitates learning by the hidden layers.

2.2.2 Parameter Ranges in the Fatigue S-N Data Base

The data base used to train the ANN contains fatigue data obtained from smooth cylindrical specimens that were tested under fully reversed axial strain control loading. It covers an adequate range of compositional and structural parameters, loading strain rate, temperature, and water chemistry, and is composed of 490 tests in air (362 tests for LAS and 128 for CS) and 546 tests in water (239 tests for LAS and 307 for CS). Carbon steels include nine heats of A333-Grade 6, A106-Grade B, A516-Grade 70, and A508-Class 1 steels. The LASs include 14 heats of A533-Grade B and A508-Class 2 and 3 steels. The sulfur content of the steels ranges from 0.006 to 0.033 and 0.001 to 0.027 wt.% for CSs and LASs, respectively. In general, the sulfur content of LASs is lower than that of CSs. The most prevalent sulfur content for LASs in this data base is 0.003 wt.%, with some data for steels that contain 0.010 and 0.012 wt.% sulfur. Although the data for CSs range from 0.012 to 0.033 wt.% sulfur for tests in air and from 0.006 to 0.033 wt.% sulfur for the tests in water, most of the data are for steels that contain 0.012 or 0.015 wt.% sulfur.

Most of the tests in the existing data base were conducted at strain amplitudes between 0.2 and 1.0%. In this range, the ANN was able to accurately determine the effect of strain amplitude on fatigue life. However, the fatigue S-N behavior cannot be estimated beyond 10^5 cycles, because little or no high-cycle fatigue data are available, i.e., at strain amplitudes <0.2%.

Existing data cover a strain rate range of 0.004 to >1%/s, room temperature to 320°C test temperature, and 0.01 to >1 ppm DO in water. Although more than half of the tests were conducted at 0.4%/s, the ability of the neural network to recognize patterns and interpolate data resulted in good predictions in terms of strain rate. Similarly, the range of DO content is adequate to obtain good predictions between 0.01 to 1 ppm. The predictions for the temperature effects in water are meaningful because the data covered the temperature range of 100–300°C. More than half of the tests in water were conducted at 288°C, and ~50 tests each were conducted at 100, 150, 200, and 250°C. In air, however, the effect of temperature on fatigue life cannot be accurately established because most of the tests were conducted either at room temperature or at 288°C.

2.2.3 Neural Network Estimates

A data base of 1036 fatigue tests was used to train the ANN. Once the optimal ANN was designed, the ANNs were trained and used to predict fatigue life for various sets of loading and environmental conditions. The ANN was trained 12 times; six times with tests in water and six times with tests in air. Each of the six trainings was based on the same data set, but the order in which the data were presented to the ANN for training was varied and the initial ANN weights were randomized to guard against overtraining and to ensure that the network did not arrive at a solution that was a local minimum. Initially, the error in each weight is high because the weights are chosen randomly. The data set that is presented at the start of the training changes the weights more than subsequent data because of this high error. The ANN was considered properly trained when differing initial data gave the same final result.

The results provide insights into the trends and thresholds that characterize the fatigue life of CSs and LASs as a function of steel type, temperature, sulfur content, DO level in water,

and tensile strain rate. Experimentally observed values of fatigue life in air and water and values predicted by the ANN are plotted in Fig. 4. The predicted fatigue lives show good agreement with the experimental data. The R-squared values for the ANN were 0.91 and 0.82, respectively, for air and water environments. These values represent an average of all six trained ANNs.

Figure 4. Experimental values of fatigue life of CSs and LASs and those predicted by artificial neural network in air and water environments

The ANN is a powerful tool but there are limitations on its use. One problem, which was encountered as the ANNs were developed for this application, was overtraining. Overtraining occurs when the capacity of the ANN for training is too great because it is too large or is allowed too many training cycles. The ANN will try to accommodate outlying data and lose the simple patterns that are the most useful for understanding fatigue life behavior. The optimal number of cycles for training the ANN was ≈ 3000 for the data presented in this study.

Another limitation of ANNs is that they cannot be extrapolated effectively. The problem with extrapolation is especially significant for high-cycle fatigue, because existing data do not contain many tests with fatigue lives $> 10^5$ cycles. Because a functional form is not defined in an ANN and the weights are initially randomized, extrapolations beyond the range of the data base used to train the ANN are not possible. If the patterns near an extreme end of a data set indicate a certain trend that is slightly imperfect, extrapolation can magnify the error. Interpolation is much more meaningful for ANNs. By finding patterns and trends throughout the data, information learned about one heat of steel can be applied to other heats with sparse data.

Air Environment

The effects of sulfur content, strain rate, and temperature on the fatigue lives of CSs and LASs in air and those predicted from the statistical model are shown in Figs. 5–7. Artificial neural networks are especially suited for the analysis of data bases that lack complete data

sets. The ANN data indicate that, for both CSs and LASs, fatigue life decreases with increasing temperature, a finding that is in agreement with the statistical model. For CSs, one data set is humped, i.e., from room temperature, the fatigue life first increases up to $\approx 150^{\circ}\text{C}$ and then decreases at higher temperatures. It is likely that this data set is overtrained in terms of temperature. It is difficult for the ANN to learn the effect of temperature because most of the available data are at 25 or 288°C , with little in between. This is an example of why each data set was trained in six different orders with six different sets of initial weight values. Furthermore, the increased dispersion of data at high temperatures, i.e., $>300^{\circ}\text{C}$, is a good example of why an ANN cannot be used to extrapolate beyond the range of the data used to train it.

Figure 6 shows good agreement with the statistical model; however, the fatigue life is decreasing somewhat with increasing strain rate. As discussed later, this is the opposite of the trend observed in water. Although the trend is obviously significant for the CSs at high temperatures in this data base, it is caused by strain aging effects that may not be as pronounced in all heats of CSs. The data for the effect of sulfur content on fatigue life are some of the more scattered results produced by the ANN. The variation of fatigue life with sulfur content is difficult to characterize because sulfur content depends on steel type. Most of the fatigue data were obtained on steels with >0.010 wt.% sulfur for CSs and <0.005 wt.% sulfur for LASs. Also, the grain size and dispersion may affect fatigue life. Therefore, the slight decrease in fatigue life with increasing sulfur content cannot be considered significant.

LWR Environments

The effects of temperature, strain rate, DO, and sulfur content on the fatigue lives of CSs and LASs, and those predicted by the statistical model are shown in Figs. 8-11. The results indicate that at high strain rate ($0.4\%/s$), fatigue life is relatively insensitive to changes in temperature. At low strain rate ($0.004\%/s$), fatigue life decreases with increase in temperature beyond a threshold value of 150°C . These results are in complete agreement with the statistical model in magnitude as well. The precision of the data indicates that this trend is present in the data used to train the ANN.

The effect of DO on fatigue life determined by the ANN is similar to that predicted by the statistical model. Both indicate that fatigue life decreases with increasing DO content. Prediction from the ANN are more linear than those from the statistical model because the statistical model has a discontinuous slope at its threshold values and because the ANN was trained for a small number of cycles to prevent overtraining. For high strain rates, neither the ANN data nor the statistical model indicates a significant effect of DO on fatigue life.

The ANN data for the effect of strain rate on fatigue life indicate that fatigue life decreases with decreasing strain rate. The results agree well with the assumptions used to develop the statistical model. The slopes are the same for the ANN data and the statistical model, indicating that they agree on the magnitude of the effect of strain rate on fatigue life. The difference between the outputs of the ANN and the statistical model at 1 ppm DO occurs because the model predicts a larger effect of DO than the ANN.

Figure 5. Dependence of fatigue life on temperature for CSs and LASs in air at 0.4 and 0.004%/s strain rates: solid line = statistical model; open circles = experimental data; other lines = ANN estimates for the six trained sets

Figure 6. Dependence of fatigue life on tensile strain rate for CSs and LASs in air at room temperature and 288°C: solid line = statistical model; open circles = experimental data; other lines = ANN estimates for the six trained sets

Figure 7. Dependence of fatigue life on sulfur content of CSs and LASs in air at 0.4 and 0.004%/s strain rates: solid line = statistical model; open circles = experimental data; other lines = ANN estimates for the six trained sets

Figure 8. Dependence of fatigue life on temperature for CSs and LASs in water at 0.4 and 0.004%/s strain rates: solid line = statistical model; open circles = experimental data; other lines = ANN estimates for the six trained sets

Figure 9. Dependence of fatigue life on DO for CSs and LASs in water at 0.4 and 0.004%/s strain rates: solid line = statistical model; open circles = experimental data; other lines = ANN estimates for the six trained sets

Figure 10. Dependence of fatigue life on tensile strain rate for CSs and LASs in water containing 0.2 and 1.0 ppm DO: solid line = statistical model; open circles = experimental data; other lines = ANN estimates for the six trained sets

Figure 11. Dependence of fatigue life on sulfur content of CSs and LASs in water at 0.4 and 0.004%/s strain rates: solid line = statistical model; open circles = experimental data; other lines = ANN estimates for the six trained sets. Note that data above 0.02 wt.% sulfur are extrapolated.

As was discussed in the previous Section, very few data are available for steels with sulfur content >0.015 wt.%. Therefore, extrapolation beyond the existing data range, i.e., sulfur content >0.015 wt.%, is not valid and not shown in Fig. 11. Also we cannot determine from the current data base whether or not the threshold for sulfur content is at 0.015 wt.%. For sulfur content <0.015 wt.%, predictions by ANN are consistent with estimates based on the statistical model.

2.3 Crack Initiation and Crack Growth Characteristics (D. J. Gavenda, P. R. Luebbbers, and O. K. Chopra)

The fatigue S-N curve represents the process of fatigue crack initiation (or fatigue damage accumulation). It specifies that, for a given stress or strain amplitude, the number of cycles needed to form an "engineering" crack (e.g., a 3-mm-deep crack), is defined as the fatigue life of the material. Deformation and microstructural changes in the surface grains control fatigue cracking. During cyclic straining, irreversibility of dislocation glide leads to the development of surface roughness. Strain localization in persistent slip bands (PSBs) results in the formation of extrusions and intrusions. With continued cycling, microcracks ultimately form in PSBs or at the edges of slip-band extrusions. At high strain amplitudes, microcracks form in notches that develop at grain, twin, or phase boundaries (e.g., ferrite/pearlite) or by cracking of second-phase particles (e.g., sulfide or oxide inclusions).

Once a microcrack is formed, it continues to grow along its slip plane or a PSB as a Mode II (shear) crack in Stage I growth (orientation of the crack is usually at 45° to the stress axis). At low strain amplitudes, a Stage I crack may extend across several grain diameters before the increasing stress intensity of the crack promotes slip on systems other than the primary slip. A dislocation cell structure normally forms at the crack tip. Because slip is no longer confined to planes at 45° to the stress axis, the crack begins to propagate as a Mode I (tensile) crack, normal to the stress axis in Stage II growth. At high strain amplitudes, the stress intensity is quite large and the crack propagates entirely by the Stage II process. Stage II crack propagation continues until the crack reaches an engineering size (≈ 3 mm deep). In air or mildly corrosive environments, Stage II cracking is characterized by fatigue striations. The two stages of fatigue crack growth in smooth specimens is shown in Fig. 12.

Figure 12. Two stages of fatigue crack growth in smooth test specimens

Fatigue life, or number of cycles, has conventionally been represented by two stages: (a) initiation, which represents the cycles N_i for formation of microcracks on the surface; and (b) propagation, which represents cycles N_p for propagation of the surface cracks to an engineering size. Thus, fatigue life N is the sum of the two stages, $N = N_i + N_p$. The former is considered to be sensitive to the stress or strain amplitude, e.g., at low strain amplitudes, most of the life may be spent in initiating a crack whereas, at high strain amplitudes, cracks initiate easily.

Recent studies on the growth of short cracks in CSs and LASs⁵²⁻⁶¹ have prompted an alternative approach for describing fatigue life. This alternative approach considers fatigue life to be entirely composed of crack propagation;^{62,63} for polycrystalline materials, the period for the formation of surface cracks is zero, Fig. 13. Fatigue damage in a material is the current size of the fatigue crack, and damage accumulation is the rate of crack growth.⁶³ During fatigue loading of smooth test specimens, cracks form immediately at surface irregularities/discontinuities either already in existence or produced by slip bands, grain boundaries, second-phase particles, etc. Growth of these surface cracks may be divided into three regimes: (a) initial period that involves growth of microstructurally small cracks below a critical length (MSCs), characterized by decelerating crack growth, seen in region AB of Fig. 13; (b) final period of growth, characterized by accelerating crack growth, region CD; and (c) a transition period controlled, by a combination of the two regimes, region BC.

Figure 13.
Growth of cracks in smooth fatigue specimens

The growth of MSCs is very sensitive to microstructure.⁵²⁻⁵⁹ The MSCs correspond to Stage I cracks and grow along slip planes as shear cracks in the early stage of growth. For MSCs, microstructural effects are strong because of the Stage I growth, i.e., crystallographic growth. The growth rates are markedly decreased by grain boundaries, triple points, and phase boundaries. In ferritic-pearlitic steels, fatigue cracks initiate and propagate preferentially in the ferrite phase that forms as long allotriomorphs at prior austenite phase boundaries.⁵⁷⁻⁵⁹ The ferrite/pearlite phase boundaries act as strong barriers to crack propagation, and growth rates decrease significantly when small cracks grow into the pearlite from the ferrite.⁵⁷ Limited data suggest that microstructural effects are more pronounced at negative stress ratios; the compressive component of the applied load plays an important role in the formation of Stage I facets and as a driving force during the formation of cracks.⁵⁵

Fatigue cracks greater than the critical length of MSCs show little or no influence of microstructure and are termed mechanically small cracks.^{54,55} For a stress ratio of -1 , the transition from MSC to a mechanically small crack for several materials has been estimated to be ≈ 8 times the unit size of the microstructure.⁵⁵ Mechanically small cracks correspond to Stage II, or tensile, cracks characterized by striated crack growth, with a fracture surface normal to the maximum principal stress. Their growth rates tend to decrease as the cracks grow because crack closure becomes more significant for larger cracks. For ferritic-pearlitic steels, Stage II crack propagation occurs when stress intensity and mode of growth attain a critical level and break through the pearlite and join other ferrite cracks.⁵⁸

At low stress levels, e.g., $\Delta\sigma_2$ in Fig. 13, the transition from MSC growth to accelerating crack growth does not occur and the cracks are nonpropagating. This circumstance represents the fatigue limit for the smooth specimen. Although cracks can form below the fatigue limit, they can grow to engineering size only at stresses greater than the fatigue limit. Note that possible preexisting large cracks in the material, e.g., defects in welded samples, or those created by growth of microcracks at high stresses, can grow at stress levels below the fatigue limit.

Reduction in fatigue life in LWR coolant environments may arise from easy formation of surface microcracks and/or an increase in growth rates of cracks, during either the initial stage of MSC and shear crack growth or the transition and final stage of tensile crack growth. Carbon steel and LAS specimens tested in water show surface micropitting and cavities that

were produced by either corrosion or dissolution of MnS inclusions.^{12,13} These micropits can act as sites for the formation of fatigue cracks.^{64,65}

Detailed metallographic evaluation of the fatigue specimens^{12,13} and exploratory tests^{8,9} have been conducted to evaluate the contributions of environment to the formation of surface microcracks. The results indicate that the reduction in fatigue life in high-DO water is primarily due to environmental effects on crack propagation. Environment appears to have little or no effect on the formation of surface microcracks. Although all specimens tested in water show surface micropitting, there is no indication that these micropits enhance cracking. Irrespective of environment, cracks in CS and LAS nucleate either along slip bands, sulfide inclusions, carbide particles, or at the ferrite/pearlite phase boundaries. Examples of such cracks¹³ in A106-Gr B CS, tested at 288°C in water that contained 0.8 ppm DO, are shown in Fig. 14.

Figure 14. Formation of surface cracks along slip bands, carbide particles, and ferrite/pearlite phase boundaries of A106-Gr B steel. Specimen was descaled with an electrolyte of 2 g hexamethylene tetramine in 1000 cm³ of 1 N HCl.

The environmental enhancement of fatigue crack growth in pressure vessel steels in high-temperature oxygenated water and the effects of sulfur content, loading rate, and flow velocities are well known.^{36-47,49,66-69} The enhanced growth rates in LWR environments have been attributed to either slip dissolution^{48,70} or hydrogen-induced cracking⁷¹ mechanisms. The requirements for a slip dissolution model are that a protective oxide film is thermodynamically stable to ensure that a crack will propagate with a high aspect ratio without degrading into a blunt pit, and that a strain increment occurs to rupture that film and thereby expose the underlying matrix to the environment, Fig. 15. Once the passive oxide film is ruptured, crack extension is controlled by dissolution of freshly exposed surfaces and by the oxidation characteristics. The hydrogen-induced cracking of LASs is explained as follows (Fig. 16): hydrogen produced by the oxidation reaction at or near the crack tip is partly absorbed into the metal; the absorbed hydrogen diffuses ahead of the crack tip and interacts with MnS inclusions and leads to the formation of cleavage cracks at the inclusion matrix interface; and linkage of the cleavage cracks results in discontinuous crack extension in addition to extension caused by mechanical fatigue.

Figure 15.
Schematic illustration of film
rupture/slip dissolution process

Figure 16.
Schematic illustration of hydrogen-
induced cracking of low-alloy steels

Both mechanisms depend on oxide rupture rates, passivation rates, and liquid diffusion rates. For environmental effects on fatigue life, a critical level of sulfide ions is needed at the crack tip. Dissolution of MnS inclusions changes the water chemistry near the crack tip, making it more aggressive. The result is enhanced crack growth rate because either the dissolved sulfides decrease the repassivation rate, which increases the amount of metal dissolution for a given oxide rupture rate;⁷⁰ or the dissolved sulfide poisons the recombination of hydrogen atoms liberated by corrosion, which enhances hydrogen uptake by the steel at the crack tip.⁶⁹ Recent studies, based on a balance between sulfide supply rate due to exposure of sulfide inclusions from an advancing crack and mass transport of sulfur ions away from the crack tip by diffusion, indicate that a critical velocity of 5×10^{-7} mm/s and a minimum crack size of 2.5 mm are necessary to produce the sulfur concentration that is necessary for environmentally assisted enhancement of crack growth in low-DO environments.⁶⁷ For smooth cylindrical fatigue specimens, fatigue life is defined as the number of cycles necessary to produce an ≈ 3 -mm crack. Consequently, environmental effects should not decrease fatigue life in low-DO water. The existing fatigue S-N data are consistent with these results; fatigue life of CSs and LASs in low-DO water (<0.01 ppm DO) at 288°C is lower than that in air by a factor of <2 . However, it is plausible that, in high-DO water environments, MSCs or

nonpropagating cracks could grow by anodic dissolution or other processes and thereby decrease the fatigue life of the material.

During this reporting period, fatigue tests have been conducted to determine the crack initiation and crack growth characteristics of A533-Gr B LAS in air and LWR environments. Results of fatigue tests that examine the influence of reactor environment on formation and growth of short cracks in A533-Gr B LAS are presented below. Crack length as a function of fatigue cycles was determined in air by a surface replication technique, and in water, by block loading that leaves marks on the fracture surface.

2.3.1 Experimental

Experimental conditions for the various tests in air and water environments are given in Table 2. All tests were conducted at 288°C in stroke control mode with fully reversed axial loading. Details about the test facility and procedure have been described elsewhere.^{8,12} Surface replication by an acetate film was employed to measure the surface length of cracks in specimens tested in room temperature air. This technique provides $\approx 1\text{-}\mu\text{m}$ resolution of the surface and allows identification of surface features such as PSBs and cracking. The tests were interrupted periodically to allow for surface replication. To ensure reproducibility between replicas, reference markers were inscribed in the shoulder region of the specimens at three equal intervals along the circumference. A detailed examination of the gage surface of the specimen was conducted to establish the morphology of final fatigue crack. The region on surface replicas corresponding to the final crack was examined to determine the length of surface cracks as a function of fatigue cycles.

Table 2. Fatigue results for A533-Gr B steel in air and water environments

Test No.	Specimen		Temp. (°C)	Strain Range (%)	Strain Rate (%/s)		Stress Range (MPa)	Life N ₂₅ (Cycles)	Number of Blocks
	Id.	Environ.			Ten.	Comp.			
1785	44-68	Air	25	0.76	0.4	0.4	763.7	8,840	-
1779	44-67	Air	25	0.76	0.004	0.4	759.8	5,960	-
1786	44-71	Air	25	0.40	0.4	0.4	687.7	61,100	-
1800	44-75	Water	288	0.81	0.004	0.4	879.9	303	5 ^a
1803	44-77	Water	288	0.82	0.004	0.4	913.9	272	7 ^b

^aTest interrupted every 50 cycles and subjected to a block loading of 170 cycles at 0.4% strain range with a triangular waveform and 0.4%/s strain rate.

^bTest interrupted every 30 cycles and subjected to a block loading of 170 cycles at 0.4% strain range with a triangular waveform and 0.4%/s strain rate.

The crack morphology and length of the final crack for an A533-Gr B specimen tested at 0.75% strain range with a saw-tooth waveform (Test No. 1779, Specimen Id. 44-67) are shown in Fig. 17. Surface crack lengths were used as reference markers to define probable crack fronts on the fracture surface. Fatigue striations on the fracture surface were used as guidelines to determine the shape of the crack fronts. The probable crack fronts for the surface crack in Fig. 17, after 5316, 5779, 5955, and 6195 cycles, are shown in Fig. 18. The results indicate that three cracks merged to form the final fracture surface. The primary crack initiated near an inclusion and reached a surface length of $\approx 100\text{ }\mu\text{m}$ after 3062 cycles (i.e., $\approx 50\%$ of fatigue life). Two secondary cracks merged with the primary crack after ≈ 5700 and 6000 cycles. Crack depth, a , was determined from the surface crack length S by the relationship

Figure 17. Morphology and length of surface crack after varying number of cycles for A533-Gr B steel tested in air at room temperature, 0.75% strain range, and slow/fast saw-tooth waveform. Letters denote reference points used to locate the same regions on the replicas.

Figure 18. Fracture surface and probable crack front after varying numbers of fatigue cycles for surface crack shown in Fig. 17

$$\frac{a}{S} = \frac{1}{\pi}.$$

(12)

The calculated values agree closely with measurements of the probable crack depth on the fracture surface.

For the tests in high-temperature water, crack growth characteristics were determined with block loading, e.g., the slow/fast fatigue tests were interrupted each period $\approx 10\%$ of life, and the specimen was subjected to a block of fast/fast cycles at either the same or a lower strain range. The block of fast/fast cycles leaves a beach mark on the fracture surface that can be used to characterize crack size as a function of fatigue cycles for the slow/fast test. A photomicrograph of the fracture surface of a slow/fast test at 0.75% strain range interrupted every 50 cycles and subjected to 170 cycles of fast/fast loading at 0.4% strain range is shown in Fig. 19. Beach marks produced from four of the five fast/fast loading sequences at the low strain range can be seen clearly on the fracture surface.

Figure 19.

Beach marks produced by four of five block loading sequences on the fracture surface of A533-Gr B steel tested at 288°C in high-DO water

2.3.2 Results

The results of recent tests (Table 2) are presented in Fig. 20 with data obtained earlier^{8,15} on A533-Gr B LAS in air at room temperature and in high-DO water at 288°C. The recent test results are consistent with the earlier data. At room temperature, the fatigue life is $\approx 50\%$ lower in water than in air. Although the test at 0.004%/s strain rate shows slightly lower life than that at 0.4%/s, the available S-N data indicate that in both air and water environments strain rate has little or no effect on fatigue life of CS and LAS.

The fatigue lives of specimens subjected to block loading are approximately the same as fatigue lives of specimens tested at a constant strain range. The results indicate a significant reduction in fatigue life in high-DO water at 288°C and a strong dependence on strain rate. Fatigue life decreases rapidly with decreasing strain rate. The fracture behavior for block loading tests, however, differs from that that for constant strain tests. Photomicrographs of the fracture surface from tests that were interrupted every 50 and 30 cycles and subjected to block loading of 170 cycles at 0.4% strain range are shown in Fig. 21. The fracture surface consists of several cracks that initiated at various axial and circumferential locations and then do not merge into a single primary crack. The fracture surface of specimens tested at constant strain range typically consist of a single crack, or a few cracks that merge to form the final fracture surface, e.g., three cracks merged to form the final fracture surface in Fig. 18.

The depth of the largest crack as a function of fatigue cycles or life fraction for the tests in air and water environments is plotted in Fig. 22. The results indicate that in room temperature air, crack size at various life fractions is independent of strain range and strain rate; depth of the largest crack at different life fractions is approximately the same at 0.75 and 0.4% strain ranges and at 0.4 and 0.004%/s strain rates. The results also show that fatigue cracks 10 μm or longer form quite early during fatigue, e.g., at $<10\%$ of fatigue life. The largest crack is $<100 \mu\text{m}$ at half life. The data for room temperature air show good agreement with earlier studies on CSs^{57,61} and LASs,⁷² Fig. 23; the crack depth was determined from surface crack length with Eq. 12. The crack depth vs. life fraction plots are approximately the same over a wide range of strain or stress. These results indicate that, for strain or stress

Figure 20. Fatigue lives of A533-Gr B steel tested in air at room temperature and in high-DO water at 288°C

Figure 21. Photomicrographs of fractured specimen from tests that were interrupted every (a) 50 and (b) 30 cycles and subjected to block loading at lower strain range

ranges above the fatigue limit, linear damage summation (Miner's rule) is valid for CS and LAS in either air or water environments.

The results in high-DO water indicate that the decrease in fatigue life of LASs is primarily caused by the effects of environment during early stages of fatigue damage, i.e., growth of cracks $<100 \mu\text{m}$ in depth (Stage I crack growth). For example, only 30-50 cycles are needed to form a $100\text{-}\mu\text{m}$ crack in high-DO water (average growth rate of $2\text{-}3 \mu\text{m}/\text{cycle}$), whereas it takes more than 3000 cycles to form a $100\text{-}\mu\text{m}$ crack in air (average growth rate of $0.033 \mu\text{m}/\text{cycle}$). Figure 22 also indicates that linear damage summation may be non-conservative in a water environment, e.g., a fatigue usage of ≈ 0.2 in water is equivalent to a usage of ≈ 0.5 in air, e.g., both result in an $\approx 100 \mu\text{m}$ crack. The crack growth rates determined from the crack-depth-versus-cycles data of Fig. 22 are plotted as a function of crack depth in Fig. 24. For crack sizes $>100 \mu\text{m}$, crack growth rates are an order of magnitude higher in high-DO water than in air. Additional tests are being conducted to confirm these results.

Figure 22. Depth of largest crack plotted as a function of fatigue life or fractional life for A533-Gr B low-alloy steel tested in air and water environments

Figure 23. Crack depth plotted as a function of fractional life for CSs and LASs tested in room-temperature air

Figure 24. Crack growth rates plotted as a function of crack size for A533-Gr B LAS tested in air and water environments. Probable growth rates during the early stages of fatigue damage shown as dotted lines.

2.4 Austenitic Stainless Steels

(D. J. Gavenda and O. K. Chopra)

The primary sources of relevant S-N data for austenitic SSs are the JNUFAD data base for "Fatigue Strength of Nuclear Plant Component" from Japan and the data compiled by Jaske and O'Donnell⁷³ for developing fatigue design criteria for pressure vessel alloys. In addition, fatigue tests have been conducted on austenitic SSs in air by Conway et al.⁷⁴ and Keller,⁷⁵ and in water by GE in a test loop at the Dresden 1 reactor^{21,22} and by ANL.¹¹

The existing fatigue S-N data indicate that the fatigue lives of austenitic SSs in air are independent of temperature in the range from room temperature to 427°C. Fatigue lives of Types 304 and 316 SS are comparable to each other, whereas those of Type 316NG SS are superior. The effects of strain rate on fatigue life cannot be established from the existing S-N data. Limited results suggest that some heats are sensitive to strain rate; fatigue life may decrease up to 30% with decreasing strain rate. The fatigue S-N data for austenitic SSs indicate a significant decrease in fatigue life in water. The effect of environment on fatigue life increases with decrease in strain rate. Environmental effects on fatigue life are the same for Types 304, 316, and 316NG SS. However, the existing data base is inadequate to establish the effects of temperature or DO content on the fatigue lives of these steels; most of the data have been obtained at 288–320°C in water that contained ≥ 0.2 ppm DO.

Using existing S-N data, we have developed interim design fatigue curves that address the effects of environment on fatigue life of austenitic SSs.¹⁷ Statistical models have also been developed for estimating the effects of various material and loading conditions on the fatigue life of these steels.¹⁸ In air, the fatigue life N of Types 304 and 316 SS is expressed as

$$\ln(N) = 6.690 - 1.980 \ln(\epsilon_a - 0.12) \quad (13a)$$

and that of Type 316NG SS, as

$$\ln(N) = 7.072 - 1.980 \ln(\epsilon_a - 0.12), \quad (13b)$$

where applied strain amplitude ϵ_a is in percent. The fatigue lives of Types 304, 316, and 316NG SS in air at various temperatures are presented in Fig. 25 with values estimated from Eqs. 13a and 13b. At 25–450°C, the fatigue lives of Types 304 and 316 SS in air show no dependence on temperature. For Type 316NG SS, the results at room temperature show good agreement with estimates from the statistical model or the Jaske and O'Donnell curve,⁷³ but the data at 288°C are lower than the estimated values at strain amplitudes $< 0.2\%$. Also, the ASME mean curve is not consistent with the existing fatigue S-N data for austenitic SSs. At strain amplitudes $< 0.5\%$, the mean curve predicts significantly longer fatigue lives than those observed experimentally; the fatigue lives at 288°C and $\approx 0.25\%$ strain range fall very close to the ASME Code design curve. When the Code fatigue S-N curve for austenitic SSs was extended to 10^8 cycles, this discrepancy was taken into account, but no change was made to the curve for $< 10^6$ cycles. The statistical model is in good agreement with the Jaske and O'Donnell average curve.

In LWR environments, the fatigue life N of Types 304 and 316 SS is expressed as

Figure 25.
Fatigue S-N behavior for Types 304,
316, and 316NG SS in air at various
temperatures

$$\ln(N) = 6.331 - 1.980 \ln(\epsilon_a - 0.12) + 0.134 \dot{\epsilon}^* \quad (14a)$$

and that of Type 316NG, as

$$\ln(N) = 6.713 - 1.980 \ln(\epsilon_a - 0.12) + 0.134 \dot{\epsilon}^*, \quad (14b)$$

where the applied strain amplitude ϵ_a is in % and $\dot{\epsilon}^*$ is the transformed strain rate defined as

$$\begin{aligned} \dot{\epsilon}^* &= 0 & (\dot{\epsilon} > 1\%/s) \\ \dot{\epsilon}^* &= \ln(\dot{\epsilon}) & (0.001 \leq \dot{\epsilon} \leq 1\%/s) \\ \dot{\epsilon}^* &= \ln(0.001) & (\dot{\epsilon} < 0.001\%/s). \end{aligned} \quad (14c)$$

The statistical model is recommended for predicting fatigue lives $\leq 10^6$ cycles. The lower boundary of 0.001%/s on the strain rate is based on results for CSs and LASs.

The objective of this task is to conduct fatigue tests on austenitic SSs under conditions where information is lacking in the existing S-N data base. Fatigue tests are being conducted on Types 304 and 316NG SS in air and LWR environments to evaluate the effects of various

material and loading variables, e.g., steel type, strain rate, DO, strain range, and loading waveform, on fatigue life of these steels. The results of these tests and data obtained previously on Type 316NG in air and high-DO water are presented.

2.4.1 Experimental

Fatigue tests have been conducted on Types 316NG and 304 SS to establish the effects of LWR coolant environments on fatigue lives of these steels. The composition of the two steels is given in Table 3. Smooth cylindrical specimens with a 9.5-mm diameter and 19-mm gage length were used for the fatigue tests. The specimen gage was given a 1- μ m surface finish in the axial direction to prevent circumferential scratches that might act as sites for crack initiation.

Table 3. Composition (wt.%) of austenitic stainless steels used for fatigue tests

Material	Heat	Source	C	P	S	Si	Cr	Ni	Mn	Mo	Cu	N
Type 316NG ^a	D432804	Vendor	0.011	0.020	0.001	0.52	17.55	13.00	1.76	2.49	0.10	0.108
		ANL	0.013	0.020	0.002	0.49	17.54	13.69	1.69	2.45	0.10	0.105
Type 304 ^b	30956	Vendor	0.060	0.019	0.007	0.48	18.99	8.00	1.54	0.44	-	0.100

^aASME SA312 seamless stainless steel pipe (hot finished), 610 mm O.D. and 30.9 mm wall, fabricated by Sumitomo Metal Industries, Ltd. Solution annealed at 1038–1093°C for 0.5 h and water quenched.

^bSolution annealed at 1050°C for 0.5 h.

All tests were conducted at 288°C with fully reversed axial loading (i.e., $R = -1$) and a triangular or sawtooth waveform. The strain rate for the triangular wave and fast-loading half of the sawtooth wave was 0.4%/s. Tests in water were conducted in a small autoclave under stroke control, where the specimen strain between two locations was controlled from outside the autoclave. Tests in air were performed under strain control with an axial extensometer; specimen strain between the two locations used in the water tests was also recorded. Information from the air tests was used to determine the stroke required to maintain constant strain in the length of the specimen gage for tests in water; the stroke is gradually increased during the test to account for cyclic hardening of the material and to maintain constant strain in the specimen gage section. Details about the test facility and procedure have been described elsewhere.^{8,12}

2.4.2 Results

During the current reporting period, fatigue tests were conducted on Types 316NG and 304 SS in air and water environments to establish the effects of DO on fatigue life. The results from these tests and those obtained previously¹¹ on Type 316NG in air and high-DO water are listed in Tables 4 and 5. The fatigue S-N data for Types 316NG and 304 SS in air are shown in Fig. 26, together with S-N curves estimated from the statistical model,¹⁸ a best-fit curve proposed by Jaske and O'Donnell,⁷³ and the ASME mean curve. The results show good agreement with estimates from the statistical model or the Jaske and O'Donnell curve;⁷³ however, for Type 316NG SS, the data at 288°C are lower than the predicted values at strain ranges <0.4%.

The cyclic strain hardening of Type 316NG tested in air at room temperature and 288°C and strain ranges of 0.25–1.0% is shown in Fig. 27. At both temperatures, the steel exhibits

Table 4. Fatigue test results for Type 316NG austenitic stainless steel

Test No.	Environ.	Diss. Oxygen ^a (ppb)	pH at RT	Conductivity (mS/cm)	Ten. Rate (%/s)	Comp. Rate (%/s)	Stress Range (MPa)	Strain Range (%)	Life N ₂₅ (Cycles)
<u>Room Temp.</u>									
1394	Air	-	-	-	5.0E-1	5.0E-1	694.7	1.51	4,649
1391	Air	-	-	-	5.0E-1	5.0E-1	554.8	1.00	13,561
1390	Air	-	-	-	5.0E-1	5.0E-1	518.1	0.75	25,736
1396	Air	-	-	-	5.0E-1	5.0E-1	506.7	0.76	30,000
1420	Air	-	-	-	4.9E-1	4.9E-1	495.3	0.49	54,249
1392	Air	-	-	-	5.0E-1	5.0E-1	475.9	0.51	60,741
1393	Air	-	-	-	5.0E-1	5.0E-1	464.7	0.41	127,386
1395	Air	-	-	-	5.0E-1	5.0E-1	456.7	0.35	183,979
1397	Air	-	-	-	5.0E-1	5.0E-1	446.0	0.30	347,991
1398	Air	-	-	-	5.0E-1	5.0E-1	436.7	0.27	666,000
1399	Air	-	-	-	5.0E-1	5.0E-1	431.8	0.25	>1,900,000
1400	Air	-	-	-	5.0E-1	5.0E-1	427.4	0.25	1,775,000
<u>288°C</u>									
1408	Air	-	-	-	5.0E-1	5.0E-1	416.6	0.76	21,548
1790	Air	-	-	-	5.0E-3	5.0E-1	452.8	0.75	16,765
1409	Air	-	-	-	5.0E-1	5.0E-1	377.2	0.50	53,144
1410	Air	-	-	-	5.0E-1	5.0E-1	377.6	0.50	51,194
1792	Air	-	-	-	5.0E-3	5.0E-1	413.4	0.51	35,710
1407	Air	-	-	-	5.0E-1	5.0E-1	364.4	0.40	82,691
1430	Air	-	-	-	5.0E-1	5.0E-1	348.3	0.30	168,852
1435	Air	-	-	-	5.0E-1	5.0E-1	342.0	0.25	314,352
1480	Air	-	-	-	4.9E-1	4.9E-1	340.1	0.25	319,308
1485	Air	-	-	-	5.1E-1	5.1E-1	340.4	0.25	369,206
<u>320°C</u>									
1405	Air	-	-	-	5.0E-1	5.0E-1	426.0	0.75	20,425
1404	Air	-	-	-	5.0E-1	5.0E-1	387.4	0.50	47,011
1406	Air	-	-	-	5.0E-1	5.0E-1	371.6	0.40	82,691
<u>288°C</u>									
1426	Hi DO	>200	-	-	8.0E-1	8.0E-1	405.1	0.80	12,069
1427	Hi DO	>200	-	-	8.2E-2	8.2E-2	421.7	0.82	6,679
1428	Hi DO	>200	-	-	7.4E-3	7.4E-3	441.4	0.74	5,897
1797	Hi DO	750	5.9	0.076	5.0E-3	5.0E-1	437.3	0.78	4,520
1414	Hi DO	>200	-	-	5.0E-1	5.0E-1	375.3	0.50	26,230
1418	Hi DO	>200	-	-	5.0E-1	5.0E-1	375.5	0.50	25,714
1423	Hi DO	>200	-	-	5.0E-2	5.0E-2	378.8	0.50	17,812
1425	Hi DO	>200	-	-	4.9E-3	4.9E-3	393.2	0.49	13,684
1431	Hi DO	>200	-	-	2.9E-1	2.9E-1	356.5	0.29	116,754
1434	Hi DO	>200	-	-	2.9E-2	2.9E-2	350.0	0.29	40,643
1436	Hi DO	>200	-	-	2.5E-2	2.5E-2	354.0	0.25	>1,719,851
1512	Hi DO	>200	-	-	2.4E-1	2.4E-1	361.2	0.24	2,633,954
1796	PWR	5	6.4	20.20	5.0E-1	5.0E-1	403.6	0.80	12,500
1812	PWR	2	6.5	20.00	5.0E-2	5.0E-1	413.9	0.80	6,375
1791	PWR	4	6.5	19.23	5.0E-3	5.0E-1	441.9	0.77	3,040
1793	PWR	4	6.4	19.23	5.0E-3	5.0E-1	434.3	0.80	3,020
1794	PWR	4	6.4	20.00	5.0E-3	5.0E-1	390.9	0.50	7,370

^a PWR = simulated PWR water that contained 2 ppm lithium and 1000 ppm boron.

rapid hardening during the first 50-100 cycles of fatigue life. The extent of hardening increases with applied strain range and is greater at room temperature than at 288°C. The initial hardening is followed by softening and a saturation stage at 288°C, and by continuous softening at room temperature. Type 304 SS shows identical cyclic hardening behavior. The effect of strain rate on cyclic hardening of the two steels is shown in Fig. 28. Cyclic stresses

Table 5. Fatigue test results for Type 304 austenitic stainless steel

Test No.	Environ.	Diss. Oxygen ^a (ppb)	pH at RT	Conductivity (mS/cm)	Ten. Rate (%/s)	Comp. Rate (%/s)	Stress Range (MPa)	Strain Range (%)	Life N ₂₅ (Cycles)
288°C									
1801	Air	-	-	-	4.0E-1	4.0E-1	419.2	0.76	24,500
1805	Air	-	-	-	4.0E-3	4.0E-1	467.9	0.76	14,410
1804	Air	-	-	-	4.0E-1	4.0E-1	382.8	0.51	61,680
1806	PWR	4	6.0	18.87	4.0E-1	4.0E-1	428.9	0.73	11,500
1810	PWR	5	6.4	18.89	4.0E-2	4.0E-1	447.6	0.77	5,800
1808	PWR	4	6.4	18.87	4.0E-3	4.0E-1	468.3	0.77	2,850
1807	PWR	4	6.5	18.87	4.0E-1	4.0E-1	374.6	0.51	25,900

^a PWR = simulated PWR water containing 2 ppm lithium and 1000 ppm boron.

Figure 26. Total strain range-versus-fatigue-life data for Types 316NG and 304 SS in air

Figure 27. Effect of strain range on cyclic strain-hardening behavior of Type 316NG SS in air at room temperature and 288°C and 0.4%/s strain rate

increase with decreasing strain rate. For Type 316NG, the extent of softening after the initial hardening period is less at slow strain rates.

Figure 28. Effect of strain rate on cyclic strain-hardening behavior of Types 316NG and 304 SS in air at 288°C

Figure 29. Cyclic stress-strain curve for Types 304, 316, and 316NG SS in air at room temperature, 288, and 288-430°C

The cyclic stress-versus-strain curves for Types 304, 316, and 316NG SS at room temperature and 288°C are shown in Fig. 29; cyclic stress corresponds to the value at half life and at a strain rate of 0.4%/s. For the various steels, cyclic stresses increase in the following

order; Types 316NG, 304, and 316 SS. Furthermore, cyclic stresses are 20–30% lower at 288–430°C than at room temperature. At room temperature, the total strain amplitude ϵ_a (%) can be expressed in terms of the cyclic stress amplitude σ_a (MPa) for Type 316 SS by the equation

$$\epsilon_a = \frac{\sigma_a}{1950} + \left(\frac{\sigma_a}{588.5} \right)^{1.94}, \quad (15a)$$

for Type 304 SS, by the equation

$$\epsilon_a = \frac{\sigma_a}{1950} + \left(\frac{\sigma_a}{503.2} \right)^{2.19}, \quad (15b)$$

and for Type 316NG, by the equation

$$\epsilon_a = \frac{\sigma_a}{1950} + \left(\frac{\sigma_a}{447.0} \right)^{2.59}, \quad (15c)$$

At 288–430°C, the cyclic stress-versus-strain curve can be expressed for Type 316 SS by

$$\epsilon_a = \frac{\sigma_a}{1760} + \left(\frac{\sigma_a}{496.8} \right)^{2.19}, \quad (15d)$$

for Type 304 SS, by

$$\epsilon_a = \frac{\sigma_a}{1760} + \left(\frac{\sigma_a}{373.9} \right)^{2.31}, \quad (15e)$$

and for Type 316NG, by

$$\epsilon_a = \frac{\sigma_a}{1760} + \left(\frac{\sigma_a}{330.1} \right)^{3.24}, \quad (15f)$$

The cyclic stress-versus-strain curves for Type 316 SS at 400°C (from JNUFAD data base) and 0.1, 0.01, and 0.001%/s strain rate, are shown in Fig. 30. The results indicate that, at 288–430°C, cyclic stresses are increased with decreasing strain rate.

Figure 30.
Effect of strain rate on cyclic stress-strain curve for Type 316 SS in air

The fatigue S-N data for Types 316NG and 304 SS in water at 288°C are shown in Fig. 31; the ASME Code fatigue design curve is also shown in the figure. The results indicate a significant decrease in fatigue life in water and a strong dependence on strain rate. A decrease in strain rate from 0.4 to 0.004%/s, decreases fatigue life by a factor of ≈ 8 . At slow strain rates, e.g., $\approx 0.004\%/s$, the reduction in fatigue life in a simulated PWR environment (<10 ppb DO) is greater by a factor of ≈ 2 than it is in high-DO water (≥ 200 ppb DO). The water environment has no effect on the cyclic stress response of austenitic SSs, as shown in Fig. 32.

The fatigue lives of Types 316NG and 304 SS in air and simulated PWR, and high-DO water environments are plotted as a function of strain rate in Fig. 33. In all of the environments, the fatigue lives of these steels decrease with decreasing strain rate. The effect of strain rate is the smallest in air and largest in a low-DO PWR environment. Fatigue tests are in progress at lower strain rates to establish a possible saturation effect. Tests are also planned at low strain ranges to define a threshold value for strain range below which environmental effects on fatigue life of austenitic SSs do not occur.

Figure 31. Total strain range vs. fatigue-life data for Types 316 NG and 304 SS in air and water

Figure 32. Cyclic strain-hardening behavior of Type 316NG SS in air and PWR water at 288°C

Figure 33.
Effect of strain rate on fatigue lives of austenitic SSs in air, and simulated PWR and high-DO water environments

Figure 34 shows photomicrographs of the fracture surface at approximately the same crack length for Type 316NG specimens tested in air, high-DO water, and low-DO PWR water. Fatigue striations can be seen clearly on all specimens. The spacing between striations indicates that crack growth rates increase in the following sequence: air, high-DO water, and low-DO PWR water.

Figure 34. Photomicrographs of fracture surface of Type 316NG SS specimens tested at 288°C and 0.75% strain range in (a) air, (b) high-DO water, and (c) simulated low-DO PWR water

Fatigue test specimens were examined in detail to investigate the role of high-temperature oxygenated water on fatigue cracking. In general, the specimens tested in air show slight discoloration, whereas the specimens tested in oxygenated water developed a gray/black corrosion scale. Photomicrographs of the gage surface of Type 316NG specimens tested in air and simulated PWR and high-DO water environments are shown in Fig. 35. Specimens tested in water contain crystalline oxides and exhibit a thin gray corrosion scale. X-ray diffraction analyses of specimens tested in water indicate that the corrosion scale consists primarily of

(a)

(b)

(c)

Figure 35.

SEM photomicrographs of gage surface of Type 316NG SS specimens tested in (a) air, (b) simulated PWR, and (c) high-DO water environments

magnetite (Fe_3O_4) or ferroferric oxide (FeFe_2O_4), chromium oxide (CrO), and maghemite ($\gamma\text{-Fe}_2\text{O}_3$). In addition to these phases, a specimen tested in high-DO water also contained hematite (ferric oxide or $\alpha\text{-Fe}_2\text{O}_3$).

The crack frequency, i.e., number of cracks per unit length of the specimen gage surface, was measured along longitudinal sections of specimens tested in various environments. Preliminary results indicate that crack frequency is lower in high-DO water than in air or in the simulated PWR environment. For Type 316NG SS tested at 288°C , $\approx 0.75\%$ strain range, and $0.005\%/s$ strain rate, the number of cracks along a 7-mm gage length that were longer than $20\ \mu\text{m}$ was 16, 14, and 8 in air and simulated PWR and high-DO water environments, respectively. A detailed metallographic examination of the test specimens is in progress to investigate the differences in the formation and growth of fatigue cracks in air and LWR environments.

3 Irradiation-Assisted Stress Corrosion Cracking of Austenitic SS

In recent years, failures of reactor-core internal components in both BWRs and PWRs have increased after accumulation of relatively high fluence ($>5 \times 10^{20}\ \text{n}\cdot\text{cm}^{-2}$, $E > 1\ \text{MeV}$). The general pattern of the observed failures indicates that, as nuclear plants age and neutron

fluence increases, various apparently nonsensitized austenitic SSs become susceptible to intergranular failure. Some components (e.g., BWR core shroud, control-blade handle and sheath) are known to have cracked under minimal applied stress. Although most failed components can be replaced, some safety-significant structural components (e.g., BWR top guide, shroud, and core plate) would be very difficult or impractical to replace. Therefore, the structural integrity of these components after accumulation of high fluence has been a subject of concern and extensive research to provide an understanding of this type of degradation, which is commonly known as IASCC.

Primary effects of irradiation include alteration of microchemistry, microstructure, and mechanical properties of the structural material, usually fabricated from ASTM Type 304, 316, or 348 SSs. Irradiation produces defects and defect clusters in grain matrices, and alters the dislocation network, dislocation loop, and dislocation channel structures, leading to radiation-induced hardening. Irradiation also leads to changes in the stability of second-phase precipitates and the local alloy chemistry near grain boundaries, precipitates, and defect clusters. Grain-boundary microchemistry that differs significantly from that of the bulk composition can be produced in association with not only radiation-induced segregation (RIS) but also thermally driven equilibrium and nonequilibrium segregation of alloying and impurity elements. Neutron irradiation also alters the water chemistry, in particular, near the grain-boundary surfaces in a crack-tip region.

Irradiation-induced grain-boundary depletion of chromium has been considered for many years as the primary metallurgical process that causes IASCC. One of the most important factors that has been considered by many investigators to support the chromium-depletion mechanism is the observation that the dependence on water chemistry (i.e., oxidizing potential) of IGSCC of nonirradiated thermally sensitized material and of IASCC of BWR-irradiated solution-annealed material is similar. Recent attention also has focused on the effects of fabrication-related variables, i.e., grain-boundary segregation and depletion of alloying and impurity elements by thermal processes, the effect of final thermomechanical treatment, hardening by coldwork, the use of recycled scrap metals, uptake of minor impurities during iron- and steelmaking, and contamination by minor impurities during welding of field components such as BWR core shrouds.

In the present reporting period, our effort has focused on: (1) SSRT testing of model SS alloys irradiated in the Halden reactor, (2) characterization of deleterious impurities from contamination during welding of core internal components, and (3) grain-boundary segregation and depletion by a thermal process. Preliminary results obtained on base-metal components, such as neutron-absorber tubes and control-blade sheath irradiated in several BWRs up to $\approx 2.6 \times 10^{21}$ n·cm⁻², indicated deleterious effects of inadvertent contamination by impurities such as fluorine and oxygen. Consistent with this preliminary finding, significant fluorine and oxygen contamination were observed in a BWR core shroud weld specimen and in nonirradiated weld specimens of Type 304 SS prepared by a shielded-metal-arc (SMA) process.

3.1 Slow-Strain-Rate-Tensile Tests of Austenitic Stainless Steels Irradiated in the Halden Reactor (H. M. Chung, W. E. Ruther, and R. V. Strain)

SSRT tests were conducted on model SS specimens irradiated at $\approx 288^\circ\text{C}$ in a helium environment in the Halden reactor to a fluence of $\approx 0.45 \times 10^{21}$ n·cm⁻² ($E > 1$ MeV). The composition of the model alloys tested to date is given in Table 6. The tests were conducted at

Table 6. Composition (in wt.%) of model stainless steel alloys used for SSRT tests in simulated BWR water at $\approx 288^{\circ}\text{C}$

Alloy ID	Ni	Si	P	S	Mn	C	N	Cr	Mo/Nb	O (wppm)	Remarks
L23	12.04	0.68	0.030	0.047	0.96	0.043	0.092	17.30	Nb 1.06	93	CP348
L7	10.60	0.18	0.040	0.038	1.02	0.007	0.111	15.40	-	274	High O
L14	7.93	1.49	0.080	0.002	1.76	0.107	0.028	15.00	-	45	High C
L17	8.00	0.66	0.090	0.009	0.48	0.061	0.078	15.30	-	90	-
L6	10.00	1.90	0.020	0.005	1.13	0.096	0.087	17.10	-	58	High Si
L27	10.30	0.96	0.040	0.002	0.97	0.057	0.019	15.30	Mo 2.01	-	B 0.030
L26	8.09	0.79	0.004	0.002	0.91	0.070	0.089	17.20	-	80	B <0.001
C1	8.12	0.50	0.038	0.002	1.00	0.060	0.060	18.11	-	-	-
L5	9.66	0.90	0.113	0.028	0.47	0.006	0.033	21.00	-	-	High P
L22	13.30	0.24	0.015	0.004	0.40	0.003	0.001	16.10	Mo 2.04	-	HP 316
C3	8.91	0.46	0.019	0.004	1.81	0.016	0.083	18.55	-	-	B < 0.001
C16	12.90	0.38	0.014	0.002	1.66	0.020	0.011	16.92	-	-	B <0.001

Table 7. SSRT^a test and SEM fractography results for specimens of model austenitic stainless steel alloys irradiated in helium at 288°C in the Halden reactor

Alloy Ident. No.	Fast-Neutron Fluence, ($\text{n}\cdot\text{cm}^{-2}$)	SSRT No.	Feedwater Chemistry				SSRT Parameters			Fracture Morph.	
			Oxygen Conc. (ppm)	Average ECP (mV SHE)	Cond. at 25°C ($\mu\text{S}\cdot\text{cm}^{-1}$)	pH at 25°C	Failure Time (h)	Max. Stress (MPa)	Total Elong. (%)	TGSCC (%)	IGSCC (%)
L23	0	CHR-1	8.6	+228	0.07	6.65	240.3	480	18.6	-	-
L7	0	CHR-2	8.0	+217	0.07	7.37	141.2	370	8.4	20 ^b	0
L14	0	CHR-3	8.6	+208	0.07	7.37	794.4	474	47.2	-	0
L17	0	CHR-4	7.5	+262	0.06	7.09	236.1	412	14.0	-	-
L27	0	CHR-5	8.0	+247	0.08	6.96	405.0	483	24.1	-	-
L26	0	CHR-6	9.4	+223	0.07	6.65	703.9	596	41.8	-	-
C1	0.45×10^{21}	HR-1	8.3	+184	0.07	7.03	299.9	680	17.8	4	0
L5	0.45×10^{21}	HR-2	9.7	+208	0.07	6.89	552.9	539	32.8	2	2
L22	0.45×10^{21}	HR-3	8.0	+236	0.07	6.80	160.4	596	9.5	50	15
C3	0.45×10^{21}	HR-4	8.7	+161	0.07	6.68	552.0	491	32.8	5	0
C16	0.45×10^{21}	HR-5	8.3	+204	0.08	6.74	368.6	527	21.9	2	0

^aStrain rate of $1.65 \times 10^{-7} \text{ s}^{-1}$ (strain rate for Test CHR-1 was $2.15 \times 10^{-7} \text{ s}^{-1}$).

^bBrittle crack morphology on $\approx 20\%$ of the fracture surface in association with dense aggregates of internal oxides that are tetrahedral in shape and 10-20 μm in size.

$\approx 288^{\circ}\text{C}$ in simulated BWR water that contained ≈ 8 ppm DO. Similar tests are being conducted on archive (nonirradiated) materials under the same conditions. The SSRT test conditions and SCC failure behavior are summarized in Table 7.

Figure 36 shows typical stress-strain curves of Alloy C1 (commercial heat of Type 304 SS), and specimens from a BWR neutron-absorber tube and a BWR control-blade sheath that were fabricated from various commercial heats of Type 304 SS and irradiated to a similar fluence level. The results indicate that ductility (total elongation) of Heat C1, irradiated in the Halden reactor, is similar to that of the commercial-purity BWR control-blade sheath, and the ductility of both materials is considerably higher than that of Type 304 SS neutron-absorber tubes⁷⁶ at a similar fluence level of $\approx 0.5 \times 10^{21} \text{ n}\cdot\text{cm}^{-2}$ ($E > 1 \text{ MeV}$). However, susceptibility of the latter material to IGSCC was rather insignificant, as indicated by the $\approx 2\%$ intergranular crack

Figure 36.
Stress-versus-strain of commercial heat C1 of Type 304 SS irradiated in Halden reactor, and of BWR components irradiated and tested under similar conditions (fluence $\approx 0.5 \times 10^{21} \text{ n}\cdot\text{cm}^{-2}$, $E > 1 \text{ MeV}$)

morphology and $\approx 55\%$ transgranular (TG) morphology on the fracture surface. A high carbon content of irradiated Type 304 SS is known to suppress IGSCC and promote susceptibility to TGSCC.⁷⁶ Although the content of the three heats of Type 304 SS is similar, the susceptibility to TGSCC of the neutron-absorber tube was considerably higher than that of the control-blade sheath and Heat C1; we do not know why. SSRT results on specimens from actual BWR components are rather sparse,^{76,77} and, except for tests on the control-blade sheath at ANL,⁷⁶ virtually all of the data were obtained on tubular specimens, i.e., from neutron-absorber tubes at ANL⁷⁶ and dry tubes at Japan Nuclear Fuel Development Corporation (NFD).⁷⁷ Tests to determine SCC have been conducted on tubular specimens under circumferential loading produced by swelling of B_4C pellets during irradiation in a PWR and two BWRs to simulate the behavior of neutron-absorber tubes.⁷⁸⁻⁸⁰ In general, during fabrication, tubular components receive more severe thermomechanical treatment than sheet components and they are usually cleaned in HF-containing pickling solutions after fabrication. Additional data and microstructural analysis of the sheet specimens irradiated in the Halden reactor should clarify the effects of chemical composition and fabrication.

Fracture surface morphology of several specimens was characterized by scanning electron microscopy (SEM); these results are also summarized in Table 7. High-purity Type 316 SS (Heat L22), irradiated to a fluence of $\approx 0.45 \times 10^{21} \text{ n}\cdot\text{cm}^{-2}$ ($E > 1 \text{ MeV}$), showed intergranular fracture and a smooth featureless TGSCC fracture morphology on ≈ 15 and $\approx 50\%$ of the fracture surface, respectively. This is consistent with the relatively low elongation ($\approx 9.5\%$) observed in this specimen. Except for this heat, susceptibility to IGSCC of the other four alloys was insignificant. The relatively higher susceptibility to IGSCC observed for the HP heat of Type 316 SS appears to be consistent with that of the HP Type 304 SS neutron-absorber tubes reported previously (Fig. 37).⁷⁶

A nonirradiated specimen of Type 304 SS, Heat L7, which had an unusually high oxygen content (i.e., 274 wppm versus a typical value of < 90 wppm), exhibited brittle fracture in simulated BWR water. We attribute this finding to the dense aggregation of internal oxides or spinels that cover $\approx 20\%$ of the fracture surface. Consistent with the fracture surface morphology, the specimen exhibited relatively low ductility (total elongation of $\approx 8.4\%$) and the percent of the surface with an intergranular morphology was negligible. A similar specimen tested in air at the same temperature and strain rate did not fail even at an elongation of $\approx 32\%$.

Figure 37.
Percent IGSCC of SS heats irradiated in helium in the Halden reactor to a fluence $\approx 0.45 \times 10^{21} \text{ n}\cdot\text{cm}^{-2}$ ($E > 1 \text{ MeV}$). Similar results from BWR neutron-absorber tubes and control-blade sheath irradiated to $\approx 0.6 \times 10^{21} \text{ n}\cdot\text{cm}^{-2}$ ($E > 1 \text{ MeV}$) are shown for comparison. HP = high purity; CP = commercial purity.

This observation seems to indicate that unusually high oxygen dissolved in the material is deleterious to SSRT behavior in water. In previous studies on BWR neutron-absorber tubes and a control blade sheath, we observed that susceptibility to IGSCC increased significantly for higher oxygen content in the irradiated materials (Fig. 38). Also, results of a preliminary examination of a specimen from the shroud of a Type 304 SS BWR core by Auger electron spectroscopy (AES) indicated a similar trend.⁷⁶ That is, an unusually high oxygen level and oxides or spinels in high density in the HAZ of the core shroud specimen increase susceptibility to IGSCC. Thus, it appears that high oxygen dissolved in SSs, either from ingot melting, component fabrication, or contamination during welding, can have a deleterious effect on SCC, even at low fluence.

Figure 38.
Susceptibility to IGSCC versus bulk oxygen content of specimens from Type 304 SS BWR components irradiated to a fluence of $\approx 2 \times 10^{21} \text{ n}\cdot\text{cm}^{-2}$ ($E > 1 \text{ MeV}$)

3.2 Properties of Stainless Steel Welds (J.-H. Park and H. M. Chung)

Many large components, such as BWR core shrouds, are field-welded by either SMA or submerged-arc (SA) procedures. These welding procedures use welding electrodes that are coated with a welding flux. Consequently, contamination by impurities can occur in the fusion and HAZs, and the microchemistry and microstructure of these regions differ from the

base metal; factors that can influence IGSCC and IASCC behavior. Impurity contamination and grain-boundary depletion of chromium via thermal sensitization promote susceptibility of HAZ to SCC during irradiation in a BWR environment.

3.2.1 Submerged-Metal Arc, Gas-Tungsten Arc, and Electron-Beam Welds

To investigate the structure and properties of SS welds, weldments of 12.7-mm-thick plates of three commercial-grade heats of austenitic SS were prepared by an SMA procedure. Similar studies on SA welds are also planned. In addition, gas-tungsten-arc (GTA), and electron-beam (EB) welds were prepared from the same heats of SS for comparison with SMA welds. The composition of the commercial heats of Type 304, 304L, and 316 SS is given in Table 8. Specimens for SSRT testing are being machined to determine IGSCC behavior of the materials before and after irradiation in helium-filled capsules in a Phase-II irradiation experiment in the Halden reactor. The microchemistry and microstructure of the SMA, GTA, and EB welds are being determined. Irradiation of the base metal of the same heats to a fluence of $\approx 3 \times 10^{21}$ n-cm⁻² ($E > 1$ MeV) in a Phase-I experiment in the Halden reactor is near completion. SSRT behavior of specimens from Heats C1 and C3 irradiated to $\approx 0.45 \times 10^{21}$ n-cm⁻² ($E > 1$ MeV) is summarized in Table 7 and Fig. 37.

Table 8. Composition (in wt.%) of commercial heats of Types 304, 304L, and 316 SS that were used to investigate SCC and IASCC behavior of welds relative to BWR core shroud cracking

ANL ID	Type	Heat ID	Ni	Si	P	S	Mn	C	N	Cr	Mo
C1	304	DAN-70378	8.12	0.50	0.038	0.002	1.00	0.060	0.060	18.11	-
C3	304L	PNL-C1	8.91	0.46	0.019	0.004	1.81	0.016	0.083	18.55	-
C21	316	DAN-12455	10.24	0.51	0.034	0.001	1.19	0.060	0.020	16.28	2.08

3.2.2 Weld Electrode Analysis

Welding electrodes that were used to prepare the SMA welds are described in the ASME Specification Section II SFA 5.4 (identical to AWS Specification A5.4-92), "Specification for Stainless Steel Electrodes for Shielded Metal Arc Welding." However, the chemical composition of the weld electrode coating is not described in the specification. The chemical composition of the coating is important because weld flux in the coating is the primary source of contamination of the weld fusion zone and HAZ. The composition of the electrodes obtained from the supplier is given in Table 9.

Table 9. Approximate composition^a (in wt.%) of E308-16 welding electrode for shielded metal arc welding

Fe	Cr	Ni	Mn	Si	Mo	C	Cu	Nb	V	CaCO ₃	CaF ₂	SiO ₂	TiO ₂	Al ₂ O ₃	Silicate
67.5	20.5	9.7	1.17	0.52	0.39	0.052	0.1	0.02	0.07	2-8	1-10	1-8	1-12	0-3	<5

^aObtained from electrode supplier.

The welding electrode consists of $\approx 40\%$ coating and $\approx 60\%$ Type 308 SS (Fig. 39). The composition of the coating, which is the major source of fluorine and oxygen contamination, was characterized independently by energy-dispersive spectroscopy (EDS) in a scanning electron microscope. Most of the fluorine was contained in granules of fluorspar (CaF₂) that

were dispersed in a silicate binder. FeS was also identified in the composite material of the coating. Similar analysis by AES is being conducted.

A typical EDS spectrum obtained from several local regions of the specimen in Fig. 39 is shown in Fig. 40. The spectrum shows strong signals of calcium and fluorine, which indicates that the local regions were CaF_2 ; the gold signal is from a thin film coating on the specimen. Fluorine content in the coating, determined by wet chemical analysis and ion chromatography, was 2.86 wt.%, which is consistent with the results in Table 9.

Figure 39.
SEM image of cross section of
welding electrode used for
shielded metal arc welding

Figure 40.
EDS spectrum obtained from
cross section of welding
electrode used for shielded
metal arc welding, showing
 CaF_2

3.2.3 Chemical Analysis of SMA and GTA Welds

The composition of SMA and GTA welds of Type 304, 304L, and 316 SS heats was determined by wet chemistry. Specimens were taken from the heat-affected zone (denoted by the letter "R" after the Heat ID designation, e.g., SMA C3R) and the fusion zone (denoted by letter "M" after Heat ID, e.g., TIG C21M) of the SMA and TIG welds. The results for chromium, nickel, manganese, molybdenum, copper, carbon, oxygen, silicon, phosphorus, and sulfur are shown in Fig. 41.

Figure 41. Composition of SMA and GTA welds of Type 304 (Heat C1), 304L (Heat C3), and 316 (Heat C21) SS. "R" and "M" denote specimens sectioned from heat-affected and fusion zones, respectively. Only oxygen and carbon were measured in Specimen TIG C3R.

The size of Sample TIG 3CR allowed only carbon and oxygen to be determined. Concentrations of aluminum, niobium, titanium, and boron are below the detection limits of 100, 100, 100, and 10 wppm, respectively, and are not plotted in the figure.

The following trends are observed:

Figure 41. (Continued)

- (1) Oxygen contamination is significant in the HAZ and fusion zones in SMA welds but not in GTA welds,
- (2) Manganese, molybdenum, and copper contents are higher in the GTA weld in Type 316 SS than in the SMA weld,
- (3) Sulfur contents in SMA welds in Types 304 and 316 SS are somewhat higher than in GTA welds, and
- (4) Silicon in is somewhat higher in the SMA weld of Type 304 SS than in the GTA weld.

3.2.4 Analysis of Fluorine by Secondary-Ion Mass Spectroscopy

The detection limit of fluorine by the wet-chemistry method is $< \approx 30$ wppm when several grams of material are available for analysis, but ≈ 120 wppm (≈ 360 appm) when the sample mass is < 1 mg. Therefore, we decided to analyze fluorine in an SMA weld specimen of Type 304 SS (Heat C1) by secondary-ion mass spectroscopy (SIMS). The weld specimen that contained the fusion and HAZ was polished, etched in a dilute oxalic acid solution, and ultrasonically cleaned in alcohol before SIMS analysis. Secondary ions of fluorine that were ejected from the weld-fusion zone and the HAZ were analyzed by using cesium primary ions in one case and oxygen ions in the other. Two fluorine line scans determined in this way are shown in Fig. 42. Fluorine contamination in the fusion zone and HAZ in the SMA weld of Type

Figure 42.
Fluorine concentration profiles in fusion and heat-affected zones of SMA weld of Type 304 SS, Heat C1, determined by SIMS technique. Quantitative measurements are sensitive to procedure used, but the results clearly indicate presence of fluorine.

304 SS is $\approx 7\text{--}20$ wppm ($\approx 20\text{--}60$ appm) and the fluorine atoms were distributed more or less equally in the fusion zone and in the HAZ. Calcium was also detected in the weld. Fluorine contamination is caused by CaF_2 in the coating of the welding electrode.

3.3 Development of Hot-Cell J-R Testing Facility (E. E. Gruber and T. H. Hughes)

The DC potential-drop method is being used to determine changes in crack length in J-R fracture toughness tests of irradiated SS compact-tension (CT) specimens. The resistance depends on both crack length and specimen temperature. The furnace system was modified to accurately control the specimen temperature at 288°C . Successively deeper cuts in cold-worked SS specimens were made by electrical discharge machining, and the depths, determined with an optical comparator, were correlated with electrical resistance measured at room temperature and 288°C .

The components of the load train for the CT specimens have been fabricated and assembled. The hydraulic system was installed, and the Instron mechanical-test system has been set up and calibrated. Preliminary tests of the facility indicated that the resolution of the linear-variable-differential transformer (LVDT) in the actuator limits its usefulness as the primary control mode during testing. A small-displacement (± 2.5 mm) Instron LVDT was purchased and installed to improve the resolution of the load-line displacement; this LVDT has been calibrated and is being used in current tests of the system and software.

A computer was set up to serve as the interface to the digitally controlled test system and work was begun to develop software for use in the J-R tests. We plan to use a displacement-controlled ramp to induce each increment of crack extension. To determine crack length, the specimen resistance will be measured during a hold period following each ramp. A computer program in Microsoft Visual Basic for DOS has been modified to control the Instron actuator and acquire data during a ramp. Options include the capability to hold in either position or load control, or to unload to a predetermined load or position at the end of the ramp. Load and displacement data can be acquired both by writing directly to the computer hard drive during the test and by using the Instron data buffers, which accommodate very rapid acquisition rates. Preliminary tests of the system and software were carried out successfully with a surrogate aluminum specimen in the test train.

4 Environmentally Assisted Cracking of Alloys 600 and 690 in Simulated LWR Water (W. E. Ruther, W. K. Soppet, and T. F. Kassner)

The objective of this work is to evaluate the resistance of Alloys 600 and 690 to EAC in simulated LWR coolant environments. High-nickel alloys have experienced general corrosion (tube wall thinning), localized intergranular attack (IGA), and SCC in LWRs. Secondary-side IGA* and axial and circumferential SCC** have occurred in Alloy 600 tubes at tube support plates in many steam generators. Primary-water SCC of Alloy 600 steam generator tubes in PWRs at roll transitions and U-bends and in tube plugs*** is a widespread problem that has been studied intensively. Cracking has also occurred in Alloy 600 and other high-nickel alloys (e.g., Inconel-82 and -182 and Alloy X750) that are used in applications such as instrument nozzles and heater thermal sleeves in the pressurizer† and the penetrations for control-rod drive mechanisms in reactor vessel closure heads in the primary system of PWRs;†† in dissimilar-metal welds between SS piping and LAS nozzles, in jet pump hold-down beams,††† and in shroud-support-access-hole covers§ in BWRs. Alloy 600, in general, undergoes different thermomechanical processing for applications other than those used for steam generator tubes. Because environmental degradation of the alloys in many cases is very sensitive to processing, further evaluation of even SCC is needed. In addition, experience strongly suggests that materials that are susceptible to SCC are also susceptible to environmental degradation of fatigue life and fatigue-crack growth properties. In this investigation, we have obtained preliminary information on the effect of temperature, load ratio R, and stress intensity (K) on EAC of Alloys 600 and 690 in simulated BWR and PWR water.

4.1 Experimental Methods for CGR Measurements on Alloys 600 and 690

Compositions, microstructures, and tensile properties of heats of Alloys 600 and 690 that are being used in the fatigue-crack-growth investigation were reported previously.^{81,82} During this reporting period, crack growth rate (CGR) tests were conducted on 1T compact-tension (1TCT) specimens from several heats of material in air and water environments in

* USNRC Information Notice No. 91-67, "Problems with the Reliable Detection of Intergranular Attack (IGA) of Steam Generator Tubing," Oct. 1991.

** USNRC Information Notice No. 90-49, "Stress Corrosion Cracking in PWR Steam Generator Tubes," Aug. 1990; Notice No. 91-43, "Recent Incidents Involving Rapid Increases in Primary-to-Secondary Leak Rate," July 1991; Notice No. 92-80, "Operation with Steam Generator Tubes Seriously Degraded," Dec. 1992; Notice No. 94-05, "Potential Failure of Steam Generator Tubes with Kinetically Welded Sleeves," Jan. 1994.

***USNRC Information Notice No. 89-33, "Potential Failure of Westinghouse Steam Generator Tube Mechanical Plugs," March 1989; Notice No. 89-65, "Potential for Stress Corrosion Cracking in Steam Generator Tube Plugs Supplied by Babcock and Wilcox," Sept. 1989; Notice No. 94-87, "Unanticipated Crack in a Particular Heat of Alloy 600 Used for Westinghouse Mechanical Plugs for Steam Generator Tubes," Dec. 1994.

†USNRC Information Notice No. 90-10, "Primary Water Stress Corrosion Cracking (PWSCC) of Inconel 600," Feb. 1990.

††USNRC Generic Letter 97-01: "Degradation of Control Rod Drive Mechanism and Other Vessel Closure Head Penetrations," Apr. 1, 1997; USNRC Information Notice No. 96-11, "Ingress of Demineralizer Resins Increases Potential for Stress Corrosion Cracking of Control Rod Drive Mechanism Penetrations," Feb. 1996; INPO Document SER 20-93 "Intergranular Stress Corrosion Cracking of Control Rod Drive Mechanism Penetrations," Sept. 1993.

†††USNRC Information Notice No. 93-101, "Jet Pump Hold-Down Beam Failure," Dec. 1993.

§USNRC Information Notice No. 92-57, "Radial Cracking of Shroud Support Access Hole Cover Welds," Aug. 1992.

which the orientation of crack plane in the specimens corresponds to the identification code L-T for plates in ASTM Specification E399.

The experimental procedures relevant to our mechanical loading systems are similar to those in ASTM E647. The tests are performed under controlled loading conditions with closed-loop servo-controlled machines (MTS™ systems). The contribution to the load that arises from the pressure difference between the inside and the outside of the autoclave is taken into consideration. Additional mechanical contributions that arise from friction between the pull rod in the load train and the autoclave pressure seal are typically <0.4% of the mechanical loading force under autoclave operating pressure and temperature.

The tests in air are performed in a system that is also used for tests in simulated reactor coolant environments. Air is circulated at a slow rate through the autoclave at atmospheric pressure to help maintain a uniform temperature within the autoclave. For tests in air, there is no mechanical contribution due to pressure differences between the inside and the outside of the autoclave or from friction in the load train because there is no seal between the pull rod and the autoclave.

The DC potential-drop measurement system is used for both the tests in air and those in an HP water environment. The specimens in the load train are electrically insulated from the autoclave and each other by using oxidized Zircaloy 705 pins (oxidized by heating at 550°C for 4 - 8 h) to attach the specimens to the load train. The resistance across the crack is measured by the potential drop for each specimen as a computer automatically controls a programmable DC power supply to direct the electrical current first in one direction and then in the reverse direction. The DC and drops in potential are measured by high-accuracy, stable scanning multimeters that are controlled by the computer over an IEEE-488 interface. Hundreds of measurements are averaged for one reading, which is transformed to a crack length by a software algorithm that is integral to the control computer. A corrosion-fatigue test is held at operating conditions without mechanical loading until the resistance of the specimens stabilizes, typically one week, before the initial fatigue loading at $R = 0.4$ is applied. Resolution of the crack length varies somewhat from one specimen to another and depends on the alloy composition and uniform crack length; a typical value is ± 0.05 mm for Alloy 600 or 690.

When tests are conducted in a simulated PWR environment (boric acid, lithium hydroxide and a hydrogen overpressure), the DC potential-drop system, which indicates negative CGRs, fails shortly after the cracks begin to grow. Our best explanation at present is that hydrogen enters the metal matrix and changes the intrinsic resistance of the specimen. For CGR tests in simulated PWR water, a compliance technique that was used previously was upgraded to perform measurements without interrupting the corrosion-fatigue test.

The compliance measurement system is comprised of MTS™ Model 632.10 hermetically sealed crack-opening-displacement (COD) gages that are interfaced to a computer data acquisition system along with a conditioned load cell signal. The COD gages are calibrated to an accuracy of $\pm 1\%$ at the desired test temperature prior to each test with a micrometer fixture traceable to a National Institute of Standard. The resulting typical COD gage output transfer function is 0.075 mm/V. Experience with compliance measurements with MTS™ clip gages indicates that the resolution is ± 0.025 mm (0.001 in.). The computer data acquisition system accepts the multiplexed load and COD voltage signals with a resolution of $\pm 0.05\%$ of the

applied voltage range. Compliance data from the load cell and COD gages are acquired during the unloading phase of the ramp waveform. Initially, the load-versus-COD data are graphically analyzed to select the region of the waveform with the steepest linear slope to optimize the data acquisition period. The compliance value for a test cycle is computed by a least-squares linear regression analysis of 250 data samples. The computer software monitors the linear regression curve fit for a minimum correlation coefficient of 0.999. During a corrosion-fatigue test, ten successive load-line compliance values are recorded at 1-h time intervals, averaged, and applied to a subroutine that calculates crack length by means of a polynomial elastic compliance expression derived for CT and wedge-opening-load specimens.⁸³ However, for conditions of accelerated crack growth, the measurement time interval can be reduced to 10 min. The resolution of the compliance technique crack-length measurement system is ± 0.075 mm (± 0.003 in.).

Both the initial crack length and the crack length and shape at the end of the test are determined from the fracture surface for comparison with measurements inferred from COD or DC potential-drop measurements. The specimen is cut into two pieces along the midplane of the thickness and the crack in one piece is extended by fracturing at a cryogenic temperature. One of these segments is used to examine the fracture surface by SEM. In the other piece, excess metal in the noncracked region is removed with a cut off wheel and a transverse section is prepared and polished for metallographic evaluation of the region that contains the corrosion-fatigue crack to confirm the nature of the crack path and whether crack branching had occurred.

Water for the CGR test systems is obtained from a reverse osmosis purification system and is passed through a demineralizer with mixed-bed ion-exchange resins; the conductivity is $< 0.1 \mu\text{S}\cdot\text{cm}^{-1}$. To avoid contamination by nonionic organic contaminants, the water flows through a final water purification system (e.g., a Millipore Super-Q Ultrapure Water SystemTM coupled with an Ultra Dynamics UV PurifierTM) that contains a bed of activated charcoal, an Organex bed, a resin bed, and a $0.2\text{-}\mu\text{m}$ filter. The purified water is stored in 135-liter SS feedwater tanks that are refilled weekly. The feedwater tanks are connected to the autoclave systems with SS tubing. The tanks are spray sterilized/passivated with a 10% hydrogen peroxide solution and thoroughly rinsed with deionized water prior to refilling, whenever the conductivity of the stored feedwater solution increases to $\geq 0.1 \mu\text{S}\cdot\text{cm}^{-1}$ during the storage period. The DO level is adjusted by vacuum sparging of the filled tanks; nitrogen or hydrogen is used for low-DO tests and oxygen-nitrogen gas mixtures are used to established predetermined DO levels. Final adjustment of the effluent DO in the latter tests is achieved by slightly varying the storage tank pressure and/or the flow rate of water in the autoclave. The DO concentration in the effluent stream from the autoclaves is monitored by Chemetrics ampules and/or by an OrbisphereTM DO monitor. The electrochemical potential (ECP) of platinum and SS electrodes is measured versus a room-temperature 0.1N KCl/Ag/AgCl reference electrode located in the high-temperature effluent stream (289°C) from the autoclave. The platinum potential, in particular, responds rapidly to changes in DO concentrations in simulated LWR coolant water.

4.2 CGRs of Mill-Annealed Alloy 600 and Thermally Treated Alloy 690 in Air at 35, 130, and 289°C

Crack growth rate tests have been conducted on specimens of Alloys 600 and 690 in air at 35, 130, and 289°C. Crack length was determined by the DC potential-drop technique. The data at 35 and 130°C in Tables 10 and 11 were obtained from solution-annealed specimens of both materials (Heat Nos. NX8844J-26 and NX8244HK-1B, respectively) and the results at 289°C in Table 12 were obtained from specimens of Alloys 600 and 690 (Heat Nos. NX8197 and NX8662HG-33) in the mill-annealed and mill-annealed plus thermally treated (715°C for 5 h) conditions, respectively. The chemical composition, grain size, carbide morphology, hardness, and tensile properties of these materials at 25, 289, and 320°C have been reported previously.^{81,82}

Table 10. Crack growth results for Alloy 600 and 690 specimens^a in air at 35°C

Test No.	Test Time (h)	Load Ratio ^b	Alloy 600			Alloy 690		
			Specimen No. J26-03			Specimen No. K1B-03		
			K _{max} ^c (MPa · m ^{1/2})	ΔK ^d	Rate (10 ⁻¹⁰ m · s ⁻¹)	K _{max} ^c (MPa · m ^{1/2})	ΔK ^d	Rate (10 ⁻¹⁰ m · s ⁻¹)
1	715-820	0.50	30.6	15.30	9.34	30.5	15.25	10.5
2	820-870	0.45	30.9	17.00	11.4	30.9	17.00	13.4
3	872-908	0.40	31.3	18.78	16.4	31.3	18.78	15.5
4	981-1004	0.35	31.6	20.54	21.9	31.6	20.54	21.8
5	1013-1052	0.30	32.3	22.61	28.4	32.5	22.75	38.1
6	1054-1076	0.25	32.8	24.60	36.5	33.3	24.98	56.7
7	1078-1084	0.20	33.0	26.40	40.8	33.7	26.96	83.0
8	1086-1148	0.55	33.4	15.03	9.97	34.5	15.53	15.3
9	1320-1485	0.70	34.1	10.23	3.58	35.4	10.62	4.90
10	1485-1655	0.80	34.3	6.85	1.38	35.6	7.12	2.05
11	1655-1752	0.65	34.6	12.11	5.68	36.1	12.63	8.04
12	1752-2230	0.90	34.7	3.47	0.16	36.1	3.61	0.12
13	2330-2570	0.85	34.8	5.22	0.56	36.9	5.53	0.69

^a Compact tension specimens (1TCT) of Alloys 600 and 690, Heat Nos. NX8844J-26 and NX8244HK-1B, respectively. The specimens (Nos. J26-03 and K1B-03) were tested in the solution-annealed condition (1038°C for 1h and 1093°C for 1h, respectively). Yield stresses of the materials at 25°C for these heat-treatment conditions are 246 and 216 MPa, respectively. Crack length determined by the DC potential-drop technique.

^b Frequency and rise time of the positive sawtooth waveform were 8×10^{-2} Hz and 12 s, respectively.

^c Stress intensity K_{max} values at the end of the time period.

^d $\Delta K = K_{\max}(1 - R)$, where load ratio $R = K_{\min}/K_{\max}$.

Table 11. Crack growth results for Alloy 600 and 690 specimens^a in air at 130°C

Test No.	Test Time (h)	Load Ratio ^b	Alloy 600			Alloy 690		
			Specimen No. J26-03			Specimen No. K1B-03		
			K _{max} ^c (MPa · m ^{1/2})	ΔK ^d (10 ⁻¹⁰ m · s ⁻¹)	Rate (10 ⁻¹⁰ m · s ⁻¹)	K _{max} ^c (MPa · m ^{1/2})	ΔK ^d (10 ⁻¹⁰ m · s ⁻¹)	Rate (10 ⁻¹⁰ m · s ⁻¹)
1	2694-2711	0.60	35.3	14.13	10.6	37.8	15.14	15.80
2	2711-2757	0.70	35.5	10.64	4.60	38.1	11.42	7.06
3	2757-2835	0.80	35.5	7.11	1.29	38.2	7.65	2.39
4	2835-3338	0.90	35.6	3.56	0.11	38.3	3.83	0.17
5	3338-3365	0.50	35.9	17.96	17.6	38.7	19.37	19.9
6	3366-3384	0.40	36.3	21.79	29.6	39.4	23.63	43.2
7	3390-3405	0.30	36.8	25.79	48.8	40.4	28.26	82.9
8	3406-3413	0.20	37.2	29.78	68.3	41.2	32.97	134.0
9	3413-3673	0.85	37.5	5.62	0.99	41.8	6.27	1.72

^a Compact tension specimens (1TCT) of Alloys 600 and 690, Heat Nos. NX8844J-26 and NX8244HK-1B, respectively. The specimens (Nos. J26-03 and K1B-03) were tested in the solution-annealed condition (1038°C for 1h and 1093°C for 1h, respectively). Yield stresses of the materials at 25°C for these heat treatment conditions are 246 and 216 MPa, respectively. Crack length determined by the DC potential-drop technique.

^b Frequency and rise time of the positive sawtooth waveform were 8×10^{-2} Hz and 12 s, respectively.

^c Stress intensity K_{max} values at the end of the time period.

^d $\Delta K = K_{\max}(1 - R)$, where load ratio $R = K_{\min}/K_{\max}$.

Figures 43 and 44 show the dependence of the CGRs on ΔK (i.e., K_{max}-K_{min}) for experiments at the three temperatures and the combined data for each alloy, respectively. The results indicate that Alloys 600 and 690 exhibit the same dependence on ΔK over a wide range of load ratios (R = 0.2-0.95). The coefficient-of-fit of the data to a simple power law is quite good. The results indicate that the CGRs are independent of temperature in the range of 35-289°C.

The dependence on R of calculated CGRs from the power law expressions in Figs. 43 and 44 divided by ΔK raised to the power of ≈3, where $\Delta K = K_{\max}(1 - R)$, indicated an additional dependence of the CGRs on R, i.e., S(R), whereby S(R) increases as R increases to ≈0.8 and then decreases as R increases from ≈0.8 to 0.95. To account for this observation, the CGRs in air for both materials can be expressed by the equations

$$\text{CGR} = 3.556 \times 10^{-13} (1/T_p) \cdot S(R) \cdot \Delta K^{3.029} \quad (16)$$

$$S(R) = 10.25 + 3.92 \cdot R \quad (R < 0.8) \quad (17)$$

$$S(R) = 14.390 - 1.256 \cdot R \quad (R \geq 0.8) \quad (18)$$

Table 12. Crack growth results for Alloy 600 and 690 specimens^a in air at 289°C

Test No.	Test Time h	Load Ratio ^b	Alloy 600			Alloy 690		
			Specimen No. 197-10			Specimen No. HG-10		
			$K_{\max}^c$ (MPa·m ^{1/2})	ΔK^d (10 ⁻¹⁰ m·s ⁻¹)	Rate (10 ⁻¹⁰ m·s ⁻¹)	$K_{\max}^c$ (MPa·m ^{1/2})	ΔK^d (10 ⁻¹⁰ m·s ⁻¹)	Rate (10 ⁻¹⁰ m·s ⁻¹)
1	160-220	0.20	31.4	25.12	58.8	31.7	25.36	83.9
2	230-360	0.80	31.5	6.30	1.99	31.9	6.38	2.74
3	360-395	0.40	32.2	19.32	33.0	32.9	19.74	46.8
4	405-670	0.90	32.3	3.23	0.05	32.9	3.29	<0.02
5	672-724	0.60	40.1	16.04	18.6	41.3	16.52	28.5
6	725-747	0.40	41.4	24.84	76.8	43.1	25.86	107.0
7	765-774	0.20	42.6	34.08	188.0	45.3	36.24	281.0
8	775-1005	0.90	42.7	4.27	0.34	45.4	4.54	0.55
9	1035-1054	0.40	46.8	28.08	115.0	53.0	31.80	194.0
10	1056-1064	0.60	47.7	19.08	32.1	55.0	22.00	61.6
11	1110-1245	0.90	47.8	4.78	0.52	55.2	5.52	1.21
12	1270-1275	0.30	55.6	38.92	137.0	67.9	47.53	533.0
13	1277-1343	0.90	55.8	5.58	1.12	68.6	6.86	2.02
14	1343-1850	0.95	55.9	2.80	0.07	68.6	3.43	0.08
15	1850-2370	1.00	55.9	0	<0.01	68.6	0	<0.01

^a Compact tension specimens (1TCT) of Alloys 600 and 690, Heat Nos. NX8197 and NX8662HG-33, respectively. The specimens (Nos. 197-10 and HG-10) were tested in the mill-annealed and mill-annealed and thermally treated (715°C for 5h) conditions, respectively. Yield stresses of the materials at 290°C for these heat-treatment conditions are 317 and 237 MPa, respectively. Crack length determined by the DC potential-drop technique.

^b Frequency and rise time of the positive sawtooth waveform were 8×10^{-2} Hz and 12 s, respectively.

^c Stress intensity $K_{\max}$ values at the end of the time period.

^d $\Delta K = K_{\max}(1 - R)$, where load ratio $R = K_{\min}/K_{\max}$.

where, as mentioned previously, the $S(R)$ factors account for the additional dependence of the rates on R at values <0.8 and ≥ 0.8 . The rise time T_r of the loading waveform is in seconds, the stress intensity factor ΔK is in MPa·m^{1/2}, and CGR is in m·s⁻¹.

Figure 45 shows calculated CGRs from Eqs. 16-18 versus the experimental values for the two alloys. The above equations yield a good representation of the experimental data over the range of $\approx 5 \times 10^{-11}$ – 5×10^{-8} m·s⁻¹. Figure 46a shows the dependence of the CGRs on ΔK from Eqs. 16-18 for a wide range of $K_{\max}$ (33–100 MPa·m^{1/2}) and R (0.1–0.98) at rise time of 12 s. In Fig. 46b, the calculated CGRs divided by $\Delta K^{3.029}$ are plotted versus R ; this quotient is independent of R over the entire range and indicates that Eqs. 17 and 18 adequately account

Figure 43. Dependence of the CGR of Alloys 600 and 690 on ΔK in air at 35, 130, and 289°C and data at three temperatures

Figure 44. Combined data at 35, 130, and 289°C for the dependence on ΔK of CGRs of Alloys 600 and 690 in air

for the dependence of the CGRs on the S(R) factors at $R < 0.8$ and $R \geq 0.8$, respectively. The results in air can be used to assess the degree of enhancement in CGRs of these materials in simulated reactor coolant environments at temperatures between 35 and 320°C.

4.3 CGRs of Alloy 690 in HP Deoxygenated Water at 289 and 320°C

Corrosion-fatigue experiments were conducted on Alloy 690 specimens in HP deoxygenated water at 289 and 320°C to determine the effects of heat treatment conditions, load ratio and stress intensity on CGRs. Two specimens from Heat No. NX8244HK (Nos. K1A-01 and K1B-01) were solution-annealed at 982°C for 1 h and 1093°C for 1 h, which produced ASTM grain sizes of 5 and 2, respectively.^{81,82} The other specimen (No. G21-02) from Heat No. NX8625HG-21 was in the solution-annealed plus thermally treated (715°C for 5h) condition and had an ASTM grain size of 5. These heat treatment conditions produced continuous intergranular carbides in all of the specimens; however there were fewer intragranular carbides in the solution-annealed specimens from Heat No. NX8244HK. The ultimate tensile σ_u and yield σ_y strengths for the materials at 320°C are: Heat No. NX8625HG-21, $\sigma_u = 568$ and $\sigma_y = 220$ MPa; and Heat No. NX8244HK, $\sigma_u = 572$ and $\sigma_y = 196$ MPa, and $\sigma_u = 499$ and $\sigma_y = 151$ MPa for solution anneals at 982 and 1093°C for 1 h, respectively.^{81,82}

Figure 45. Calculated versus experimental CGRs for Alloys 600 and 690 in air

Figure 46. Calculated CGRs for Alloys 600 and 690 in air versus ΔK for stress intensity factors between 33 and 100 MPa·m^{1/2} and load ratios of 0.1–0.98 from Eqs. 16-18, and calculated CGRs/ $\Delta K^{3.029}$ versus R , which illustrates that $S(R)$ relationships in Eqs. 17 and 18 adequately account for R dependence in Eq. 16

The experimental conditions and results from 15 CGR tests are given in Table 13. Crack growth was determined by the DC potential-drop method. Initial CGRs were obtained at 320°C in water that contained <1 ppb DO at several load ratios between 0.2 and 0.9, and $K_{\max}$ values of 31–44 MPa·m^{1/2} (Tests 1–7 in Table 13). The temperature was decreased to 289°C in Tests 8–11 at load ratios of 0.9, 0.8, and 0.4. The temperature was increased from 289 to 320°C in Test 12 and the load ratio was decreased from 0.4 to 0.9. The maximum stress intensity for these specimens at the end of this test (≈ 60 MPa·m^{1/2}) was considerably higher than in Test Nos. 2 and 7 at this load ratio. Tests 13–15 were conducted at 289°C at load ratios of 0.87, 1.0 (constant load), and 0.1, respectively, which produced a wide range of CGRs between $\approx 1 \times 10^{-11}$ (at $R = 1.0$) and $\approx 1 \times 10^{-8}$ m·s⁻¹ (at $R = 0.1$) at a maximum stress intensity of >60 MPa·m^{1/2}.

The dependence of the CGRs of the three specimens on stress intensity factor ΔK is shown in Fig. 47. Crack growth behavior of the materials is quite similar under the conditions in all experiments.

Figure 47.

Dependence of CGRs on ΔK of Alloy 690 specimens from two heats with differing heat treatment conditions in deoxygenated HP water at 289 and 320°C

4.4 CGRs of Alloys 600 and 690 in HP Deoxygenated Water at 240 and 320°C

Crack growth tests have been conducted on a set of Alloy 600 and 690 specimens in HP deoxygenated water at 240 and 320°C to determine the effect of alloy composition and temperature on corrosion-fatigue behavior. One specimen each of Alloy 600 and 690 (Nos. J26-01 and G21-01) from Heat Nos. NX8844J-26 and NX8625HG-21 were tested in the solution annealed (1038°C for 1h) and solution-annealed and thermally treated (715°C for 5h) conditions, respectively. The specimens have ASTM grain sizes of 4 and 5, respectively, and contain continuous intergranular carbides plus intragranular carbides.^{81,82} The yield and ultimate strengths of the materials at 320°C are also similar; Heat No. NX8844J-26, $\sigma_u = 639$ and $\sigma_y = 247$ MPa; and Heat No. NX8625HG-21, $\sigma_u = 568$ and $\sigma_y = 220$ MPa.^{81,82}

The experimental conditions and results from 11 CGR tests are given in Table 14. Crack growth was determined by the DC potential-drop method. Initial CGRs were obtained at 320°C in water that contained <1 ppb DO at several load ratios of 0.2 and 0.9, and $K_{\max}$ values of 31–45 MPa·m^{1/2} (Tests 1–3 in Table 14). The temperature was decreased to 240°C in Tests 4–9 at several load ratios between 0.2 and 0.9. In Test 10, the temperature was

Table 13. Crack growth results for Alloy 690 specimens^a in high-purity deoxygenated water at 289 and 320°C

Test No.	Test Time (h)	Test Temp. (°C)	Water Chemistry			Electrode Potential 304 SS mV(SHE) @ 289°C	Potential Pt @ 289°C	Load Ratio ^c	Alloy 690 Specimen No. G21-02			Alloy 690 Specimen No. K1A-01			Alloy 690 Specimen No. K1B-01		
			O ₂ ^b Conc.	Cond. at 25°C	pH at 25°C				K _{max} ^d	ΔK ^e	Rate	K _{max} ^d	ΔK ^e	Rate	K _{max} ^d	ΔK ^e	Rate
			(ppb)	(μS·cm ⁻¹)					(MPa·m ^{1/2})		(10 ⁻¹⁰ m·s ⁻¹)	(MPa·m ^{1/2})		(10 ⁻¹⁰ m·s ⁻¹)	(MPa·m ^{1/2})		(10 ⁻¹⁰ m·s ⁻¹)
1	381-392	320	<1	0.07	6.80	-573	-594	0.20	30.8	24.64	58.9	31.2	24.96	95.4	31.0	24.80	84.1
2	900-1490	320	<1	0.07	6.85	-569	-585	0.90	31.0	3.10	0.02	31.3	3.13	0.07	31.7	3.17	0.04
3	1490-1540	320	<1	0.07	6.58	-542	-576	0.20	38.4	30.72	191.0	38.7	30.96	216.0	39.4	31.52	210.0
4	1565-1590	320	<1	0.07	6.79	-575	-593	0.30	40.8	28.56	219.0	40.5	28.35	184.0	41.0	28.70	173.0
5	1590-1660	320	<1	0.07	6.79	-587	-605	0.50	42.0	21.00	105.0	42.9	21.45	95.3	43.7	21.85	95.3
6	1850-1881	320	<1	0.07	6.59	-587	-605	0.70	43.0	12.90	29.4	43.8	13.14	30.0	44.5	13.35	26.2
7	1950-2340	320	<1	0.08	6.39	-577	-595	0.90	43.1	4.31	0.06	43.9	4.39	0.20	44.6	4.46	0.03
8	2360-2740	289	<1	0.08	6.55	-571	-591	0.90	43.1	4.31	<0.01	43.9	4.39	<0.01	44.6	4.46	<0.01
9	2760-2766	289	<1	0.08	6.69	-571	-593	0.40	53.5	32.10	271.0	53.4	32.04	346.0	54.0	32.20	255.0
10	2770-2830	289	<1	0.08	6.69	-571	-593	0.80	54.8	10.96	10.2	54.2	10.84	12.2	54.7	10.94	11.1
11	2830-2842	289	<1	0.07	6.59	-577	-601	0.40	59.4	35.64	391.3	58.6	35.16	326.0	58.8	35.28	312.0
12	2860-2995	320	<1	0.08	6.58	-578	-595	0.90	60.4	6.04	1.64	59.1	5.91	1.88	59.2	5.92	1.80
13	3580-3715	289	<1	0.07	6.85	-542	-562	0.87	62.0	8.06	1.53	61.2	7.96	4.17	60.3	7.84	3.52
14	3800-4200	289	<1	0.07	6.78	-548	-574	1.00	62.1	0	0.06	61.4	0	0.10	60.5	0	0.11
15	4221-4224	289	<1	0.07	6.76	-549	-575	0.10	68.9	62.01	1545.0	65.0	58.50	1228.0	62.9	56.61	899.0

^a Compact tension specimens (1TCT) of Alloy 690 (Heat Nos. NX8625HG-21 and NX8244HK). The specimens (Nos. G21-02, K1A-01, and K1B-01) were tested in the solution-annealed and thermally treated (715°C for 5h) and solution-annealed (982°C for 1h and 1093°C for 1h) conditions, respectively. Yield stresses of the materials at 320°C for these heat-treatment conditions are 220, 196, and 151 MPa, respectively. Crack length determined by the DC potential-drop technique.

^b Effluent dissolved-oxygen concentration was determined with high-sensitivity Chemetrics ampoules.

^c Frequency and rise time of the positive sawtooth waveform were 8×10^{-2} Hz and 12 s, respectively.

^d Stress intensity, K_{max} , values at the end of the time period.

^e $\Delta K = K_{max}(1 - R)$, where K_{max} where load ratio $R = K_{min}/K_{max}$.

Table 14. Crack growth results for Alloy 600 and 690 specimens^a in high-purity deoxygenated water at 240 and 320°C

Test No.	Test Time (h)	Test Temp. (°C)	Water Chemistry			Electrode Potential		Load Ratio ^c	Alloy 600 Specimen No. J26-01			Alloy 690 Specimen No. G21-01		
			O ₂ ^b Conc. (ppb)	Cond. at 25°C (μS·cm ⁻¹)	pH at 25°C	Alloy 600 mV(SHE) @ 289°C	Pt		K _{max} ^d (MPa·m ^{1/2})	ΔK ^e (10 ⁻¹⁰ m·s ⁻¹)	Rate (10 ⁻¹⁰ m·s ⁻¹)	K _{max} ^d (MPa·m ^{1/2})	ΔK ^e (10 ⁻¹⁰ m·s ⁻¹)	Rate (10 ⁻¹⁰ m·s ⁻¹)
1	115-125	320	<1	0.09	6.99	-443	-434	0.20	30.8	24.64	94.8	30.7	24.56	69.6
2	600-1400	320	<1	0.07	6.73	-501	-500	0.90	31.1	3.11	0.14	30.9	3.09	0.19
3	1400-1750	320	<1	0.09	6.35	-546	-536	0.90	45.1	4.51	0.55	45.2	4.52	0.53
4	1750-2320	240	<1	0.09	6.72	-551	-531	0.90	44.5	4.45	0.22	44.6	4.46	0.27
5	2330-2355	240	<1	0.07	6.93	-560	-531	0.40	48.4	29.04	172.0	47.7	28.62	134.0
6	2355-2415	240	<1	0.07	6.93	-560	-531	0.80	48.8	9.76	4.22	48.1	9.62	6.80
7	2425-2444	240	<1	0.07	6.93	-559	-538	0.60	49.7	19.88	61.1	48.9	19.56	51.1
8	2468-2494	240	<1	0.07	6.93	-559	-538	0.20	52.2	41.76	572.0	55.6	44.48	444.0
9	2500-2760	240	<1	0.07	6.75	-568	-537	0.90	56.2	5.62	2.99	60.7	6.07	0.70
10	3790-3930	320	<1	0.07	6.91	-614	-600	0.90	59.9	5.99	0.82	61.2	6.12	2.67
11	4008-4115	320	<1	0.07	6.64	-	-	0.85	60.7	9.11	5.17	63.1	9.47	11.6

^a Compact tension specimens (1TCT) of Alloys 600 and 690, Heat Nos. NX8844J-26 and NX8625HG-21, respectively. The specimens (Nos. J26-01 and G21-01) were tested in the solution-annealed (1038°C for 1h) and annealed plus thermally treated (715°C for 5h) conditions, respectively. Yield stresses of the materials at 320°C for these heat-treatment conditions are 247 and 220 MPa, respectively. Crack length determined by the DC potential-drop technique.

^b Effluent dissolved-oxwgen concentration was determined with high-sensitivity Chemetrics ampules.

^c Frequency and rise time of the positive sawtooth waveform were 8×10^{-2} Hz and 12 s, respectively.

^d Stress intensity, K_{max}, values at the end of the time period.

^e $\Delta K = K_{max}(1 - R)$, where K_{max} where load ratio $R = K_{min}/K_{max}$.

increased from 240 to 320°C while the load ratio was maintained at 0.9; the load ratio was decreased slightly to 0.85 in Test 11. The maximum stress intensity for the specimens at the end of the last test was $\approx 60\text{--}63 \text{ MPa}\cdot\text{m}^{1/2}$.

The dependence of the CGRs of the specimens on stress intensity factor ΔK is shown in Fig. 48 and the combined data for both specimens at the two temperatures are shown in Fig. 49. Crack growth behavior of the materials is quite similar under the conditions in all of the experiments. Crack growth rates versus ΔK for Alloy 690 at 240, 289, and 320°C and the combined results from Tables 13 and 14 at the three temperatures are shown in Fig. 50. A power-law fit to the data is indicated in each figure, and, in all cases, the coefficient of the fit to the data was quite good.

Figure 48. Crack growth rate of Alloys 600 and 690 in HP deoxygenated water at 240 and 320°C versus ΔK

4.5 Comparison of CGRs of Alloys 600 and 690 in HP Deoxygenated Water and Air

Several approaches can be devised to compare CGRs of Alloys 600 and 690 with each other as well as CGRs of these alloys in water and in air under identical temperature and loading conditions. Material heat-treatment condition could also be considered, namely, solution annealing at various temperatures versus material that was annealed and thermally

Figure 49.
Combined data for dependence on ΔK of crack growth rate of Alloys 600 and 690 in HP deoxygenated water at 240 and 320°C

Figure 50. Dependence of CGRs of Alloy 690 specimens in HP deoxygenated water on ΔK at 240, 289, and 320°C and combined results at the three temperatures

treated. This latter treatment produces mechanical properties (e.g., yield and ultimate strengths), grain sizes, and carbide distributions in the materials that differ from those produced by the former treatment. Limited individual comparisons of CGRs under nominally the same loading and temperature conditions can be made for the two alloys in the same test (in water or in air), or among specimens of the same alloy but from different heats and with different heat-treatment conditions, e.g., from the results in Tables 10-14. Fewer direct

comparisons are possible between CGRs in water and air, and normal variability in the measurements may either obscure or enhance any differences that may exist in the rates.

In general, the present results do not reveal large differences in CGR behavior of Alloys 600 and 690 in HP deoxygenated water or in air under the conditions in these experiments. Similarly, temperatures in the range of 240-320°C in water and 35-289°C in air do not appear to have a large effect on the rates. This observation is based on relatively few specimens and it is possible that other heats of material with different heat-treatment conditions could produce a wider variation in the results. Nevertheless, comparison of the present results in water and air based on "best-fit" correlations for the CGR versus ΔK data presented in each of the figures. For example, a ratio of the CGRs in water to those in air can easily be obtained from the correlations for each specimen, or from the combined data from several specimens of the same material (different heats and/or heat treatments). The most useful comparison would be for rates determined at the same temperature on specimens from the same heat of material with the same heat treatment; however, the present results preclude such a comparison. In view of the relatively small effect of differing material conditions (heat and heat treatment) and temperature on the CGRs, several comparisons have been made based on the results in water and air in Figs. 47-49 and 43 and 44, respectively.

Figure 51 shows the dependence on ΔK of the ratio of the CGRs of Alloys 690 and 600 at 289°C in HP deoxygenated water to that in air at 289°C, the ratio of the CGRs from the combined data at the three temperatures in water to that in air at 289°C, and the ratio based on the combined results in water and air at all temperatures. The ratios of the CGRs of Alloys 600 and 690 in water and air at 289°C (Fig. 51a) indicate that the rates for Alloy 600 are higher in water than in air (ratio >1) at ΔK values <6 MPa·m^{1/2} and are lower in water than in air (ratio <1) at higher values of ΔK . In contrast, the CGRs of Alloy 690 are only slightly higher in water than in air over the entire range of ΔK . The ratios of the CGRs from the combined data for each alloy at 240, 289, and 320°C to that in air at 289°C (Fig. 51b) are similar to those in Fig. 51a.

The ratios of the CGRs from the combined data for each alloy in water and air (Fig. 51c) indicate that the CGRs of Alloy 600 are only slightly higher in HP deoxygenated water than in air over the entire range of ΔK , and that the rates for Alloy 690 are, at most, higher by a factor of 2 in water than in air at ΔK values >10 MPa·m^{1/2}. Figures 51b and 51c, which are based on all data obtained in water at 240, 289, and 320°C, suggest that CGRs of Alloy 600 are somewhat higher in water than in air (factor of $\approx 1.2 - 2$) at low ΔK and that the CGRs of Alloy 690 are slightly lower in HP deoxygenated water than in air at low ΔK . These results suggest that larger differences in the CGRs of the two materials in water and of the materials in water versus air may occur under constant load, i.e., at $R = 1.0$ and $\Delta K = 0$. Oxygenated water (e.g., 300 ppb DO), in contrast to water with <5 ppb DO, may lead to additional differences in the rates for the two materials and for the materials in water versus air under constant load conditions. These factors are being investigated.

Figure 51.

Dependence on ΔK of (a) ratio of CGRs of Alloys 600 and 690 at 289°C, (b) combined data at 240–320°C in HP deoxygenated water to CGRs in air at 289°C, and (c) ratio of CGRs from combined data at 240–320°C in HP deoxygenated water to CGRs from combined data in air at 35–289°C

4.6 Morphology of Crack Path and Surface of Alloy 600 and 690 Specimens

Morphology of corrosion-fatigue cracks in the Alloy 600 and 690 specimens tested in air and HP deoxygenated water listed in Tables 10-14 has been determined. The 1TCT specimens were sectioned vertically, and one-half of each specimen was split in the plane of the crack in liquid nitrogen. Corrosion-product films were removed from the fracture surface by a chemical process to reveal the morphology of the underlying material. The intact portion of the specimen that encompassed the crack was polished and etched to corroborate the mode of crack propagation and also to determine if crack branching had occurred during the test. The total crack lengths at the end of the test were consistent with values obtained by the DC potential-drop technique and no branching of the cracks had occurred. Figures 52-60 show the fracture surface, fracture morphology, and crack path in the crack-tip region of the specimens. The Alloy 600 and 690 specimens from the CGR experiments in air and HP deoxygenated water exhibited a transgranular mode of crack propagation. This mode of crack propagation can be attributed to the strong contribution of mechanical cyclic loading in tests at R of 0.1-0.8 and the relatively small degree of crack extension that occurred in tests at $R > 0.8$.

ALLOY 600	HEAT TREATMENT	LOAD CONDITIONS	ENVIRONMENT
Spec. No. J26-03 Heat No. NX8844J-26	Solution Annealed 1038°C/1 h	$K_{max} = 31-38 \text{ MPa} \cdot \text{m}^{1/2}$ $R = 0.2-0.9$ Freq. = 0.077 Hz	Air 35 and 130°C

Figure 52. Crack path, fracture surface, and fracture morphology of 1TCT specimen of Alloy 600 (No. J26-03) after crack growth experiment in air at 35 and 130°C

ALLOY 690	HEAT TREATMENT	LOAD CONDITIONS	ENVIRONMENT
Spec. No. K1B-03 Heat No. NX8244HK	Solution Annealed 1093°C/1 h	$K_{max} = 31-42 \text{ MPa} \cdot \text{m}^{1/2}$ $R = 0.2-0.9$ Freq. = 0.077 Hz	Air 35 and 130°C

Figure 53. Crack path, fracture surface, and fracture morphology of 1TCT specimen of Alloy 690 (No. H1B-03) after crack growth experiment in air at 35 and 130°C

ALLOY 600	HEAT TREATMENT	LOAD CONDITIONS	ENVIRONMENT
Spec. No. 197-10 Heat No. NX8844J-26	Mill Annealed	$K_{max} = 31-56 \text{ MPa} \cdot \text{m}^{1/2}$ $R = 0.2-1.0$ Freq. = 0.077 Hz at $R < 1.0$	Air 289°C

Figure 54. Crack path, fracture surface, and fracture morphology of 1TCT specimen of Alloy 600 (No. 197-10) after crack growth experiment in air at 289°C

ALLOY 690	HEAT TREATMENT	LOAD CONDITIONS	ENVIRONMENT
Spec. No. HG-10 Heat No. NX8662HG-33	Mill Annealed and Thermally Treated 715°C/5 h	$K_{max} = 32-69 \text{ MPa} \cdot \text{m}^{1/2}$ $R = 0.2-1.0$ Freq. = 0.077 Hz at $R < 1.0$	Air 289°C

Figure 55. Crack path, fracture surface, and fracture morphology of 1TCT specimen of Alloy 690 (No. HG-10) after crack growth experiment in air at 289°C

ALLOY 600	HEAT TREATMENT	LOAD CONDITIONS	ENVIRONMENT
Spec. No. J26-01 Heat No. NX8844J-26	Solution Annealed 1038°C/1 h	$K_{max} = 31-61 \text{ MPa}\cdot\text{m}^{1/2}$ $R = 0.20-0.90$ Freq. = 0.077 Hz	<1 ppb DO $0.07-0.09 \mu\text{S}\cdot\text{cm}^{-1}$

Figure 56. Crack path, fracture surface, and fracture morphology of 1TCT specimen of Alloy 600 (No. J26-01) after crack growth experiment HP water at 240 and 320°C

ALLOY 690	HEAT TREATMENT	LOAD CONDITIONS	ENVIRONMENT
Spec. No. G21-01 Heat No. NX8625HG-21	Solution Annealed and Thermally Treated 715°C/5 h	$K_{max} = 31-63 \text{ MPa}\cdot\text{m}^{1/2}$ $R = 0.20-0.90$ Freq. = 0.077 Hz	<1 ppb DO $0.07-0.09 \mu\text{S}\cdot\text{cm}^{-1}$

Figure 57. Crack path, fracture surface, and fracture morphology of 1TCT specimen of Alloy 690 (No. G21-01) after crack growth experiment in HP water at 240 and 320°C

ALLOY 690	HEAT TREATMENT	LOAD CONDITIONS	ENVIRONMENT
Spec. No. G21-02 Heat No. NX8625HG-21	Solution Annealed and Thermally Treated 715°C/5 h	$K_{max} = 31-69 \text{ MPa}\cdot\text{m}^{1/2}$ $R = 0.1-1.0$ Freq. = 0.077 Hz at $R < 1.0$	<1 ppb DO $0.07-0.08 \mu\text{S}\cdot\text{cm}^{-1}$

Figure 58. Crack path, fracture surface, and fracture morphology of 1TCT specimen of Alloy 690 (No. G21-02) after crack growth experiment in HP water at 289 and 320°C

ALLOY 690	HEAT TREATMENT	LOAD CONDITIONS	ENVIRONMENT
Spec. No. K1A-01 Heat No. NX8244HK	Solution Annealed 982°C/1 h	$K_{max} = 31-65 \text{ MPa}\cdot\text{m}^{1/2}$ $R = 0.1-1.0$ Freq. = 0.077 Hz at $R < 1.0$	<1 ppb DO $0.07-0.08 \mu\text{S}\cdot\text{cm}^{-1}$

Figure 59. Crack path, fracture surface, and fracture morphology of 1TCT specimen of Alloy 690 (No. K1A-01) after crack growth experiment in HP water at 289 and 320°C

ALLOY 690	HEAT TREATMENT	LOAD CONDITIONS	ENVIRONMENT
Spec. No. K1B-01 Heat No. NX8244HK	Solution Annealed 1093°C/1 h	$K_{max} = 31-63 \text{ MPa}\cdot\text{m}^{1/2}$ $R = 0.1-1.0$ Freq. = 0.077 Hz at $R < 1.0$	<1 ppb DO $0.07-0.08 \mu\text{S}\cdot\text{cm}^{-1}$

Figure 60. Crack path, fracture surface, and fracture morphology of 1TCT specimen of Alloy 690 (No. K1B-01) after crack growth experiment in HP water at 289 and 320°C

5 Summary of Results

5.1 Environmental Effects on Fatigue S-N Behavior of Primary Pressure Boundary Materials

Available data on the effects of various material and loading variables such as steel type, material orientation, dissolved-oxygen (DO) level, strain rate, temperature, and strain range on the fatigue life of low-alloy and carbon steels (LASs and CSs) in air and water environments have been summarized. In an air environment, the fatigue S-N curves for CSs and LASs indicate significant heat-to-heat variation. At 288°C, fatigue life may vary up to a factor of 5 above or below the mean value. The results also indicate that the ASME mean curve for LASs is in good agreement with the experimental data and that for CSs it is somewhat conservative.

In air, the fatigue life of CSs and LASs depends on steel type, temperature, orientation (rolling or transverse), and strain rate. The fatigue life of CSs is a factor of ≈ 1.5 lower than that of LASs. For both steels, fatigue life decreases with increase in temperature up to 320°C. Some heats of CSs and LASs exhibit effects of strain rate and orientation. For these heats, fatigue life decreases with decreasing strain rate. Also, based on the distribution and morphology of sulfides, fatigue properties in the transverse orientation may be inferior to those in the rolling orientation. Both CSs and LASs exhibit dynamic strain aging at temperatures between 200 and 370°C, which leads to enhanced cyclic hardening, a secondary hardening stage, and negative strain rate sensitivity, i.e., cyclic stress increases with decreases in strain rate. The temperature range and extent of dynamic strain aging vary with compositional and

structural factors. However, at the temperatures of dynamic strain aging, fatigue life of the steel may increase or decrease.

The fatigue S-N data indicate that environmental effects on fatigue life are significant when five conditions are satisfied simultaneously, viz., applied strain range, temperature, DO in water, and sulfur content in steel are above a minimum threshold level, and strain rate is below a critical value. There is little or no difference in susceptibility to environmental degradation of fatigue life of CSs and LASs. The fatigue life of these steels in air and LWR environments can be estimated from a statistical model.

For both steels, the fatigue data indicate threshold values of 150°C for temperature, 0.05 ppm for DO, and 0.003 wt.% for sulfur, above which fatigue life may be decreased in LWR environments. The effect of DO content on life saturates at 0.5 ppm. The data also indicate a threshold strain rate of 1%/s below which fatigue life is decreased in LWR environments; the effect saturates at $\approx 0.001\%/s$. Limited data suggest that the threshold strain is either equal to or slightly greater than the fatigue limit of the material. In the temperature range of 150–320°C, fatigue life decreases linearly with temperature. When the threshold conditions for all five parameters are satisfied, fatigue life decreases logarithmically with decreasing strain rate and DO level.

Only a moderate decrease in fatigue life is observed in LWR environments when any one of the threshold condition is not satisfied, e.g., at temperatures $\leq 150^\circ\text{C}$, in low-DO PWR environments (≤ 0.05 ppm DO), or for low-sulfur steels (≤ 0.003 wt.% sulfur). Under these conditions, life in water is 30–50% lower than that in air.

An artificial neural network (ANN) was used to find patterns and identify trends in the existing fatigue S-N data for CSs and LASs in air and LWR environments. Once the optimal ANN was designed, ANNs were trained and used to predict fatigue life for specified sets of loading and environmental conditions. By finding patterns in the data, the ANN can determine the fatigue life of these steels as a function of steel type, temperature, sulfur content, DO level in water, and tensile strain rate. The results from ANNs showed good agreement with estimates based on a statistical model. Their main benefits are that the fit of the data is based purely on the data and not on preconceptions, and that the ANN can interpolate effects by learning trends and patterns when complete data sets are not available. However, the benefit of the ANN is lost when a model is needed to extrapolate beyond available experimental data. Because a functional form is not defined in an ANN and the weights are initially randomized, extrapolations beyond the range of the data base used to train the ANN cannot be made. Because the body of experimental fatigue data does not contain many tests in which numerous stress cycles were reached, the S-N behavior in the regime of high-cycle fatigue cannot be estimated with an ANN.

Fatigue tests have been conducted to determine the crack initiation and crack growth characteristics of A533-Gr B LAS in air and LWR environments. Results of fatigue tests that examine the influence of reactor environment on the formation and growth of short cracks in A533-Gr B LAS are presented. Crack lengths as a function of fatigue cycles were determined in air and water environments. In air, fatigue cracks 10 μm or longer form quite early during fatigue ($<10\%$ life). The largest crack is <100 μm at half life. The results indicate that, for CSs and LASs, linear damage summation (Miner's rule) should be valid for strain or stress ranges

above the fatigue limit, e.g., the crack depth-versus-life fraction plots are approximately the same over a wide range of strains or stresses.

The results in high-DO water indicate that the decrease in fatigue life of LASs is primarily caused by the effects of environment during early stages of fatigue damage, i.e., growth of cracks <100 μm in depth (Stage I crack growth). Only 30–50 cycles are needed to form a 100- μm crack in high-DO water (average growth rate of 2–3 $\mu\text{m}/\text{cycle}$), whereas it takes more than 3000 cycles to form a 100- μm crack in air (average growth rate of 0.033 $\mu\text{m}/\text{cycle}$). For crack sizes >100 μm , crack growth rates are an order of magnitude higher in high-DO water than in air.

The existing fatigue S–N data for austenitic SSs in air and water environments have been summarized. The results indicate that the fatigue lives of Types 304 and 316 SS are comparable and those of Type 316NG SS are superior. In air, the fatigue lives of austenitic SSs are independent of temperature from room temperature to 427°C. Limited results suggest that some heats are sensitive to strain rate; fatigue life may decrease up to 30% with decreasing strain rate. The results indicate that the current ASME mean curve is not consistent with the existing fatigue S–N data for austenitic SSs. At strain amplitudes <0.5%, the mean curve predicts significantly longer fatigue lives than those observed experimentally; the fatigue lives at 288°C and $\approx 0.25\%$ strain range fall very close to the ASME Code design curve.

Fatigue tests have been conducted on Types 316NG and 304 SS to establish the effects of LWR coolant environments on fatigue lives of these steels. The results confirm the significant decrease in fatigue life in water; the decrease in life depends both on strain rate and DO content of the water. Environmental effects on fatigue life are the same for Types 304 and 316NG SS. In a simulated PWR environment, a decrease in strain rate from 0.4 to 0.004%/s decreases fatigue life by a factor of ≈ 8 . However, unlike CSs and LASs, environmental effects are more pronounced in low-DO than in high-DO water. At a strain rate of $\approx 0.004\%/s$, reduction in fatigue life is greater by a factor of ≈ 2 in water that contains <10 ppb DO than in water that contains ≥ 200 ppb DO. Experimental results have been compared with estimates of fatigue life that are based on a statistical model.

5.2 Irradiation Assisted Stress Corrosion Cracking

Initial slow-strain-rate-tensile (SSRT) tests were conducted in simulated BWR water at 288°C on model SS alloys that were irradiated at 288°C in helium to a fluence of $\approx 0.45 \times 10^{21}$ n-cm⁻² ($E > 1$ MeV) in the Halden reactor. Although not conclusive, initial results indicate that ductility of irradiated sheet specimens in simulated BWR water is higher than that of BWR neutron-absorber tubes at a comparable level of fluence. The former material appears to be more resistant to transgranular stress corrosion cracking than the latter material. High-purity (HP) Type 316 SS irradiated in the Halden reactor was somewhat more susceptible to SCC. This behavior is similar to that observed for HP heats of Type 304 SS.

Unusually high levels of oxygen dissolved in SS, either from ingot melting, component fabrication, or contamination during welding, can have a deleterious effect on SCC of the steel in HP oxygenated water, even if the material is irradiated to a low fluence.

Shield-metal-arc (SMA) welds were prepared from Types 304, 304L, and 316 SS to simulate BWR core shroud welds. Chemical composition and minor impurity contamination in the SMA and gas-tungsten-arc welds were determined by wet chemical analysis and secondary-ion mass spectroscopy. Significant contamination by oxygen and fluorine was observed in the fusion and heat-affected zones of the SMA welds. We attribute this contamination to the air atmosphere and the CaF_2 flux in the coating on the welding electrode.

Setup and calibration of the mechanical test system for J-R fracture toughness testing was completed successfully. An extensometer with higher resolution capability was added to the system to improve control during testing. Computer hardware has been installed, and software development for computer control of the J-R tests has been initiated.

5.3 EAC of Alloys 600 and 690 in Simulated LWR Water

Corrosion-fatigue experiments were conducted on compact-tension specimens of annealed Alloy 600 and annealed and thermally treated Alloy 690 specimens in air and in HP deoxygenated water to investigate the effects of temperature and load ratio on crack growth rates (CGRs). Crack growth behavior of the two materials in air at 35, 130, and 289°C and in water at 240, 289, and 320°C was quite similar under the conditions of these experiments. Based on results from relatively few specimens, heat-to-heat variations and heat treatment conditions (solution-annealing temperature and heat treatment at 715°C for 1h after solution annealing) did not have a significant effect on the CGRs in air or in HP deoxygenated water. Other heats of material with differing heat-treatment conditions could produce a wider variation in the results.

"Best-fit" correlations for the CGR of Alloys 600 and 690 in air and water versus ΔK were obtained for each specimen and for the combined data from several specimens of the same material (different heats and/or heat treatments). The effect of HP deoxygenated water on the CGRs of each alloy relative to that in air was determined from (a) a ratio of the CGRs at 289°C in water to the CGRs in air at 289°C, (b) a ratio of the CGRs from the combined data at three temperatures in water to that in air at 289°C, and (c) a ratio based on the combined results in water and air at all temperatures. The results indicate that the rates for Alloy 600 are higher in water than in air at 289°C at ΔK values $<6 \text{ MPa}\cdot\text{m}^{1/2}$ and are lower in water than in air at higher values of ΔK . In contrast, the CGRs of Alloy 690 are only slightly higher in water than in air over the entire range of ΔK . The ratios of the CGRs from the combined data for each alloy at 240, 289, and 320°C to that in air at 289°C exhibit a similar trend.

Ratios of the CGRs from the combined data for each alloy in water and air at all temperatures indicate that the CGRs of Alloy 600 are only slightly higher in HP deoxygenated water than in air over the entire range of ΔK , and that the rates for Alloy 690 are, at most, higher by a factor of 2 in water than in air at ΔK values $>10 \text{ MPa}\cdot\text{m}^{1/2}$. Larger differences in the CGRs of the two materials in water and of the materials in water versus air may occur under constant load, i.e., at $R = 1.0$ and $\Delta K = 0$. Oxygenated water (e.g., 300 ppb DO), in contrast to water with <5 ppb DO, may lead to additional differences in the rates for the two materials and for the materials in water versus air under constant load conditions. These factors are being investigated.

The morphology of corrosion-fatigue cracks in the Alloy 600 and 690 specimens was determined. Transgranular cracking occurred in air and HP deoxygenated water because of the strong contribution of mechanical cyclic loading in tests at R values of 0.1-0.8 and the relatively small degree of crack extension that occurred in tests at $R > 0.8$.

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BIBLIOGRAPHIC DATA SHEET

(See instructions on the reverse)

1. REPORT NUMBER
(Assigned by NRC. Add Vol., Supp., Rev.,
and Addendum Numbers, if any.)
NUREG/CR-4667, Vol. 22
ANL-97/9

2. TITLE AND SUBTITLE

Environmentally Assisted Cracking in Light Water Reactors.
Semiannual Report January 1996—June 1996

3. DATE REPORT PUBLISHED

MONTH

YEAR

May

1997

4. FIN OR GRANT NUMBER

W6610

5. AUTHOR(S)

O. K. Chopra, H. M. Chung, D. J. Gavenda, E. E. Gruber, T. H. Hughes,
T. F. Kassner, P. R. Luebbers, J.-H. Park, T. T. Pleune, W. E. Ruther,
W. J. Shack, W. K. Soppet, and R. V. Strain

6. TYPE OF REPORT

Technical; Semiannual

7. PERIOD COVERED (Inclusive Dates)

January 1996—June 1996

8. PERFORMING ORGANIZATION - NAME AND ADDRESS (If NRC, provide Division, Office or Region, U.S. Nuclear Regulatory Commission, and mailing address; if contractor, provide name and mailing address.)

Argonne National Laboratory
9700 South Cass Avenue
Argonne, IL 60439

9. SPONSORING ORGANIZATION - NAME AND ADDRESS (If NRC, type "Same as above"; if contractor, provide NRC Division, Office or Region, U.S. Nuclear Regulatory Commission, and mailing address.)

Division of Engineering Technology
Office of Nuclear Regulatory Research
U.S. Nuclear Regulatory Commission
Washington, DC 20555-0001

10. SUPPLEMENTARY NOTES

M. McNeil, NRC Project Manager

11. ABSTRACT (200 words or less)

This report summarizes work performed by Argonne National Laboratory on fatigue and environmentally assisted cracking (EAC) in light water reactors from January 1996 to June 1996. Topics that have been investigated include (a) fatigue of carbon, low-alloy, and austenitic stainless steels (SSs) used in reactor piping and pressure vessels, (b) irradiation-assisted stress corrosion cracking of Type 304 SS, and (c) EAC of Alloys 600 and 690. Fatigue tests were conducted on ferritic and austenitic SSs in water that contained various concentrations of dissolved oxygen (DO) to determine whether a slow strain rate applied during various portions of a tensile-loading cycle are equally effective in decreasing fatigue life. Slow-strain-rate-tensile tests were conducted in simulated boiling water reactor (BWR) water at 288°C on SS specimens irradiated to a low fluence in the Halden reactor and the results were compared with similar data from a control-blade sheath and neutron-absorber tubes irradiated in BWRs to the same fluence level. Crack-growth-rate tests were conducted on compact-tension specimens from several heats of Alloys 600 and 690 in air and high-purity, low-DO water.

12. KEY WORDS/DESCRIPTORS (List words or phrases that will assist researchers in locating this report.)

Corrosion Fatigue
Crack Growth
Irradiation-Assisted Stress Corrosion Cracking
Radiation-Induced Segregation
Stress Corrosion Cracking
A106-Gr B Steel
A533-Gr B Steel
Type s 304, 316, and 316NG Stainless Steel
Alloys 600 and 690

13. AVAILABILITY STATEMENT

Unlimited

14. SECURITY CLASSIFICATION

(This Page)

Unclassified

(This Report)

Unclassified

15. NUMBER OF PAGES

16. PRICE