Fuel Performance Improvement Program

Research on Mechanisms of Stress Corrosion Cracking in Zircaloy

June 1981

Consumers
Power
Company

EXXON NUCLEAR
COMPANY, Inc.

Battelle

Pacific Northwest Laboratory

NOTICE

This report was prepared as an account of work sponsored by the United States Government. Neither the United States nor the Department of Energy, nor any of their employees, nor any of their contractors, subcontractors, or their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness or usefulness of any information, apparatus, product or process disclosed or represents that its use would not infringe privately owned rights.

The views, opinions and conclusions contained in this report are those of the contractor and do not necessarily represent those of the United States Government or the United States Department of Energy.

Work on this program is performed for the
UNITED STATES DEPARTMENT OF ENERGY

by

CONSUMERS POWER COMPANY and EXXON NUCLEAR COMPANY, INC.

Under Contract DE-AC02-76ET34215

and by

PACIFIC NORTHWEST LABORATORY, *Operated by* BATTELLE MEMORIAL INSTITUTE;

Under Contract DE-AC06-76RLO 1830

Printed in the United States of America

Available from

National Technical Information Service

United States Department of Commerce

5282 Port Royal Road

Springfield, Virginia 22151

Price: Printed Copy \$____*; Microfiche \$3.00

*Pages	NTIS Selling Price
001-025	\$4.00
026-050	\$4.50
051-075	\$5.25
076-100	\$6.00
101-125	\$6.50
126-150	\$7.25
151-175	\$8.00
176-200	\$9.00
201-255	\$9.25
226-250	\$9.50
251-275	\$10.75
276-300	\$11.00

FUEL PERFORMANCE IMPROVEMENT PROGRAM

**Research on Mechanisms of Stress
Corrosion Cracking in Zircaloy**

June 1981

**D. B. Knorr
R. M. Pelloux
Massachusetts Institute of Technology
Cambridge, Massachusetts 02139**

This is a report of work performed by the Department of Materials Science and Engineering, Massachusetts Institute of Technology during the period July 1, 1980 to March 31, 1981 under Contract DE-AC02-76ET34215.

Research on Mechanisms of Stress Corrosion Cracking in Zircaloy

Abstract

The results of internal gas pressurization tests, primarily at 320°C, on cladding tubes from two suppliers, Supplier A and Supplier B, are presented. The two lots show a substantial difference in iodine SCC susceptibility so a test matrix is used to resolve the relative contributions of surface condition, residual stress, and texture. Additional tests with constant deflection split-ring specimens and with unstressed cladding segments are used to understand crack initiation and the early crack growth stages of SCC. The difference in SCC susceptibility is due to crystallographic texture. Other variables such as surface finish, stress relief temperature, and residual stress have little or no effect. Mechanical properties, crack initiation, and crack propagation all depend on texture. Both initiation and propagation features are analyzed by scanning electron microscopy. A mechanism for crack initiation consistent with most observations in this study and with the work of other investigators is proposed. At 320°C, lifetime is crack initiation limited while several tests at 390°C indicate that lifetime is less initiation limited at higher temperature.

Table of Contents

<u>Section Number</u>	<u>Page Number</u>
I. Introduction	1
II. Experimental Procedures	1
IIA. Materials Characteristics	1
IIB. Test Equipment and Procedures	4
II.B.1 Internal gas pressurization	4
II.B.2 Split ring tests	5
II.B.3 Iodine exposure of unstressed cladding segments	6
IIC. Test Matrix for Internal Gas Pressurization Tests	6
III. Results	7
III.A. Tube Pressurization Tests	7
III.A.1 Burst and creep rupture	7
III.A.2 Iodine stress rupture	7
III.A.3 Examination of failed specimens	8
a. Sample plan	8
III.B Fractography	9
III.C. Metallography	10
III.D Split-ring Tests	11
IV. Discussion	11
IV.A SCC Behavior of Suppliers A and B	11
IV.A.1. Effect of heat treatment	11
IV.A.2 Effect of surface condition	12
a. Pickling	12
b. Honing	12
IV.A.3 Effect of Texture	14
IV.A.4 Threshold Behavior of Iodine SCC	16
IV.B. Crack Initiation	17
IV.B.1 Analysis of experimental results	17
a. Iodine attack	17
b. Crack formation	17
c. Texture effect	18

IV.B.2	Analysis of sites and mechanism	19
a.	Evaluation of initiation sites	19
b.	Microstructural mechanism for crack initiation	20
IV.C	Propagation	21
IV.C.1	Microstructure observations	21
IV.C.2	Relative contribution of initiation and propagation time	23
IV.D	Split Ring Tests	25
V.	Summary and Conclusions	25
	References	27

I. Introduction

Failure of some fuel pins in light water nuclear reactors is attributed to stress corrosion cracking in the Zircaloy fuel cladding. This occurs following a local increase in fuel rod power when pellet clad interaction (PCI) generates a stress on the cladding and volatile fission products, presumably iodine, released through fuel cracks, provide the aggressive environment. The purpose of this research program is to study several critical aspects of SCC processes in Zircaloy. The effects of both materials variables and environmental variables on crack initiation and propagation are being considered.

Previous reports [1,2] presented the results of SCC crack arrest tests on fracture mechanics specimens. Double cantilever beam and wedge opening load specimens were tested in various orientations relative to the principal direction of two rolled plates. The effects of crystallographic texture, heat treatment, and microstructure on K_{ISCC} and the microscopic fracture appearance were studied.

This progress report details the results of internal gas pressurization tests on cladding tubes produced by two different suppliers. These lots, termed Supplier A and Supplier B, have been characterized in the EPRI program on stress corrosion cracking of Zircaloy [3-5]. However, many questions remain unresolved regarding the substantial difference in SCC behavior between the two lots. The present experiments are designed to more clearly understand the origin of the difference in SCC susceptibility between these materials. In particular, the relative contributions of surface condition, residual stress, and texture to SCC behavior are defined by the choice of a test matrix which considers the above materials variables. Additional tests with constant deflection split-ring specimens and with unstressed cladding segments are used to understand crack initiation and early crack growth in these materials.

II. Experimental Procedures

A. Materials Characterization

Two lots of reactor grade Zircaloy-2 tubing were selected for study. The nominal dimensions of the as-received material are 10.9, 12.8, and 0.95 mm (0.429, 0.504, and 0.037 inch) for the inside diameter (ID), outside

diameter (OD), and wall thickness, respectively. Chemical compositions for both lots are given in Table 1.

Crystallographic texture is determined by the x-ray diffraction techniques of inverse pole figure and direct pole figure [6]. Texture measurements are made at four locations in the tube wall: ID surface, 0.10 mm (4 mils) from the ID Surface, 0.47 mm (18.5 mils) from the ID surface, and 0.10 mm from the OD surface. The following basal pole figures are presented: ID surface of Supplier A, Figure 1; 0.10 mm from ID surface of Supplier A, Figure 2; ID surface of Supplier B, Figure 3; 0.10 mm from ID surface of Supplier B, Figure 4. Since these pole figures are representative of the two tubing lots, the remaining pole figures, radial-tangential (R-T) scans, and inverse pole figures are omitted.

Crystallographic texture is quantified by the calculation of texture numbers [6]. The Kearns texture parameter f_i [7,8] represents the resolved fraction of basal poles in a reference direction, i . In integral form, f_i is defined as

$$f_i = \int_0^{\pi/2} I_\phi \sin\phi \cos^2\phi d\phi \quad (1)$$

where I_ϕ is the pole density at an angle ϕ from the reference direction, i , averaged over all rotations about that reference direction. This texture number has the properties that f_i values of 0.0 and 1.0 indicate perfect basal pole alignment perpendicular and parallel to the i^{th} direction, respectively. For a random texture $f = 1/3$ in each of three orthogonal directions in a sample. The sum of f_i 's in three orthogonal directions in any sample, textured or random, equals unity. The f_i values are calculated from inverse pole figure data by the method of Kearns [7].

The texture numbers F_T [6,8] and $(SD)_T$ [6] are used to characterize the basal pole texture in the R-T plane. F_T is scaled to give limiting values of 1.0 for a radial basal pole texture and -1.0 for a tangential basal pole texture. The standard deviation $(SD)_T$, is a measure of the dispersion of basal poles in the R-T plane. $(SD)_T$ reaches a limiting value of 0.303 for a uniform basal pole distribution in the R-T plane. The mathematical definition of these quantities is:

$$F_T = \frac{\int_{-\pi/2}^{\pi/2} I(\phi) \cos 2\phi d\phi}{\int_{-\pi/2}^{\pi/2} I(\phi) d\phi} \quad (2)$$

$$(SD)_T = [\text{second moment} - (\text{first moment})^2]^{1/2} \quad (3)$$

$$\text{where first moment} = \frac{\int_{-\pi/2}^{\pi/2} I(\phi) \sin \phi d\phi}{\int_{-\pi/2}^{\pi/2} I(\phi) d\phi} \quad (3a)$$

$$\text{second moment} = \frac{\int_{-\pi/2}^{\pi/2} I(\phi) \sin^2 \phi d\phi}{\int_{-\pi/2}^{\pi/2} I(\phi) d\phi} \quad (3b)$$

Values of the texture numbers f_r , F_T , and $(SD)_T$ are given in Table 2 for four through-wall-thickness locations in each lot. The f_r values are plotted as a function of wall thickness location in Figure 5.

Two steps in the final tubing making operation have a potentially large affect on SCC behavior: heat treatment and pickling. Zircaloy tubing is normally stress-relieved following the final cold reduction pass. However, it is standard practice for tubing vendors to straighten the tubing following the stress-relief anneal which reintroduces some residual stress. To measure the hoop residual stress, 31.3 cm lengths of tubing are slit axially and the resulting dimensional changes converted into residual stress values. The formula [9] for the maximum hoop stress is:

$$\sigma_{\max} = \frac{Et}{1-\nu} \frac{D_m^1 - D_m^0}{D_m^1 D_m^0} \quad (4)$$

where E is Young's modulus, t is the wall thickness, ν is Poisson's ratio, and D_m^0 and D_m^1 are the mean tubing diameter ($\frac{OD - ID}{2}$) before (superscript) and after (superscript) slitting. It is important to note that these residual stress values assume a linear stress gradient across the wall of the cladding. Both linear [10] and non-linear [11] gradients are reported depending on the processing history of the material. Residual stress values are given in Table 3 for the as-received (AR condition) tubing where

straightening stresses are present and for the same tubing following a 500°C stress-relief anneal for two hours (SR condition). The AR residual stress values are much higher in Supplier A than in Supplier B but fall to approximately the same low value following the SR treatment.

The final pickling operation for AR material results in a substantial difference in ID surface finish between Supplier A and Supplier B. The surface of Supplier A has a comparatively smooth surface with particles protruding from the surface as shown in Figure 6(a). The particles are identified by energy dispersive x-ray analysis on the scanning electron microscope (SEM) to be primarily second phase particles (Zr/Fe/Ni/Cr) with occasional inclusions (Zr/Ca/Al/K). The AR surface of Supplier B, Figure 6(b) contains two pit populations. The large pits probably result from either gouging of the ID surface during the final tube making pass or selective local attack during pickling. The second pit population is composed of small pits (~0.5µm diameter) resulting from selective removal of second phase particles. A pickling (45% H₂O, 45% HNO₃, 10% HF) operation, (P), of the AR materials removed 37-50µm of metal giving the surfaces shown in Figure 6(c) and 6(d) for Suppliers A and B, respectively. Both surfaces are now indistinguishable with a population of only small pits. Because no large pits formed during pickling, it is likely that Supplier B large pit population of Figure 6(b) is a consequence of the tube making operation. A final surface treatment is honing (H) with a hone made of a bonded coarse grit. Tube ID surface receives multiple passes over the hone which is cooled and lubricated by a light oil. Approximately 0.5µm is removed leaving an ID surface with circumferential scratches. A stress relief anneal and pickling follow the honing operation. The H + SR + P surface is a somewhat rougher surface than a pickled surface and is between supplier A and B as-received material in terms of roughness. Table 4 summarizes the important features of ID surface condition.

B. Test Equipment and Procedures

I. Internal gas pressurization

Internal gas pressurization SCC tests are performed with the test system shown schematically in Figure 7. High purity (>99.999%) argon is pumped into a reservoir using a compressed air drive gas booster pump. System capability is approximately 76 MPa (11,000 psi). The closed end loading gives a hoop to axial stress ratio of two to one. Results are reported in terms of the applied hoop stress.

Specimens are ~15 cm long with a ~5 cm gage section where ~0.25 mm is removed from the tube OD. Prior to testing, all specimens are cleaned with methanol and dried. Circumferential scribe lines are placed at three OD locations on the reduced section and carefully measured by a micrometer accurate to ± 0.0025 mm. The ID and OD away from the gage section are also measured. Specimen assembly involves a series of steps. The rear end plug is inserted and the end is sealed by a Swagelok end cap. For SCC tests, 10 ± 1 mg of reagent grade iodine crystals (>99.8%) are weighed on an analytical balance and placed inside the specimen with a two segment volume displacing mandrel of Zircaloy rod. The front end plug is inserted and the front end sealed to a Swagelok fitting. A schematic of the specimen assembly is shown in Figure 8. The assembly is then connected to the pressurization system and purged of residual air and water vapor by repeated low pressure pressurization and venting (8-10 times). A thick walled pipe encloses the specimen assembly for safety reasons. An argon cover gas (>99.7% purity) is flowed through the chamber to minimize oxidation of the specimen OD. Temperature is monitored and controlled by a Chromel/Alumel thermocouple which touches the specimen at approximately the center of the gage section. Heatup time is ordinarily 180 min. When the test temperature is reached, the specimen is pressurized from the reservoir by bleeding the argon gas through a micro-metering valve while pressure is monitored by an in-line pressure transducer. Stress is calculated from the formula:

$$\sigma = \left(\frac{D_{ID}^2 + D_{OD}^2}{D_{OD}^2 - D_{ID}^2} \right) p \quad (5)$$

where D_{ID} and D_{OD} are the ID and OD diameters, respectively, and p is the pressure inside the specimen. Time is monitored by a timer which is shut off by specimen failure. Post test analyses include ID and OD measurements at the fracture location and at the scribe line locations.

2. Split ring tests

A limited number of split ring tests are done on Suppliers A and B cladding. Cladding segments ~1.3 cm long are slit axially with a slow speed cut off wheel. A Zircaloy wedge is pressed into the slot to give the desired opening, δ . The maximum tensile stress occurs at the clad ID

opposite the wedge. The hoop stress is calculated by

$$\sigma_{\max} = \frac{4Et\delta}{3\pi(1-\nu^2)D_m^0 D_m^1} \quad (6)$$

where the terms are defined for Eqn. (4). Specimens are exposed to 4 P_a (0.03 torr) iodine vapor carried by >99.999% purity Argon. Test temperatures of 300°C and 320°C are used.

3. Iodine exposure of unstressed cladding segments

Four cladding segments ~1.3 cm in length are loaded into an internal gas pressurization test specimen with iodine. The samples are from Supplier A and B tubes in both AR and AR + P surface conditions. The ID surface of each specimen is marked with a small punch indentation and examined near that point by SEM prior to exposure. The specimen reaches 320°C in 115 min. and is held at this temperature and low argon pressure for 180 min. The clad segments are then removed, cleaned, and examined by SEM near the mark location. The purpose is to find the surface sites most susceptible to iodine attack by analyzing the same surface location before and after iodine attack under chemical conditions equivalent to those seen by the cladding in the internal gas pressurization tests.

C. Test Matrix for Internal Gas Pressurization Tests

Figure 9 shows the test matrix for the internal gas pressurization tests. The test matrix is designed to isolate the effects of the following materials variables on SCC behavior: surface condition, heat treatment which influences the strength and residual stress, and texture.

Figure 10 gives a test matrix where tubing wall thickness is the key variable. Tests at a given condition (nominal wall thickness/nominal stress and supplier) are done in duplicate. The nominal stresses are chosen to give short times to failure (<2 hr) and long times to failure (>12 hr) for the "high" and "low" stresses, respectively.

Finally, several tests are run at 390°C rather than 320°C. The material condition depends on what specimens are available. Stresses are chosen to give "short" and "long" times to failure.

III. Results

A. Tube Pressurization Tests

1. Burst and creep rupture

Ramp to failure and creep rupture results are listed in Table 5. Ramp test takes 3-5 minutes to complete. Burst strength represents the ultimate tensile strength of the material and is calculated by Eqn. (5) from diameter values measured adjacent to the failure location to minimize the influence of local strain in the region of tube rupture. Creep rupture tests (stress versus time to failure) are done prior to any iodine tests in the test system. The data are plotted on Figures 11 and 12 for comparison with the iodine SCC results of suppliers A and B, respectively. The error bars have two sources: slight pressure fluctuation due to leaks at fittings and dimensional changes due to cladding creep. Uniform strain represents the maximum strain in the gage section away from the failure location which has a large amount of local strain associated with the penetration of the tubing wall.

2. Iodine stress corrosion cracking

The stress versus time to failure results for the test matrix in Figure 9 are presented in Figures 11-13. The data are tabulated in Table 6. Data for all material conditions of Supplier A and B cladding are presented in Figures 11 and 12, respectively, with burst and creep rupture results included for comparison. Figure 13 compares the stress/time to failure data for the AR condition of both suppliers. Curves for the tests at SRI International [3-5] performed on the same tubing at the same temperature and with the same type of test, but with a larger quantity of iodine (250 mg I_2 versus 10 mg I_2 in the present tests) are also shown. Strain at failure ϵ_f , and average strain rate, $\dot{\epsilon}_{avg}$, in the tube pressurization tests can be plotted against time to failure, strain in percent is defined as:

$$\epsilon = \frac{D_{OD}^f - D_{OD}^i}{D_{OD}^i} \times 100 \quad (7)$$

where D_{OD}^f and D_{OD}^i are the final and initial outside diameters, respectively. They are measured at the same location on the specimen corresponding to the location of failure if no local ductility increase occurs at a location near

the failure if any ballooning is present. Accuracy is judged to be $\pm 0.03\%$ except when tube ovality is present which lowers accuracy to $\pm 0.05\%$. The average strain rate is calculated as:

$$\dot{\epsilon}_{avg} = \frac{\epsilon}{t_f} \quad (8)$$

Figures 14 and 15 present ϵ and $\dot{\epsilon}_{avg}$ versus t_f , respectively. Data from all material conditions of both suppliers are included.

The results for the test matrix with the variable wall thickness specimens plotted in Figure 10 are tabulated in Table 7. Four tests are done at 390°C, and the results are presented in Table 8.

3. Examination of failed specimens

a. Sample Plan

Several techniques are used to examine a representative sample of failed SCC specimens. An 1.0 - 1.5 cm long annular section of tubing which contains the crack is cut from the remainder of the specimen. This ring is sectioned axially to give two half circular arcs, one containing the through wall crack at the bottom of the arc. An SEM photomontage of the ID surface of the crack is made, typically at 100x magnification. The specimen is then broken open at liquid nitrogen temperature to reveal the fracture surface. Macrophotographs of the through wall crack are taken at 40x. SEM examination of the crack surface identifies the fracture features. For selected specimens a 1000x photomontage is taken across the fracture face from the ID to the end of the SCC region near the OD. During all SEM evaluations features of interest are frequently examined at higher magnification. The half circular ring opposite from the main crack is sectioned in the tangential plane, mounted, and polished metallographically to expose the tube wall directly opposite from the crack. The circumference along the tube ID is carefully analyzed at 500x and 1000x for incipient cracks which are counted and measured for depth of penetration into the tube wall. In several cases, an entire tube cross-section away from the crack but still in the reduced section, is evaluated metallographically.

The sampling scheme for the iodine SCC tests is shown in Figure 16. The tests, arranged by supplier and surface condition, are listed by stress. Any examinations are indicated in a shorthand code next to the specimen. Due to the large number of specimens, it is not feasible to examine every specimen. The alternate approach is to design a sampling matrix emphasizing the following comparisons:

- specimens from the same supplier with the same material condition at high stress (short time to failure) and low stress (long time to failure);
- specimens from the same supplier with different material conditions at approximately the same stress; emphasis is placed on AR and SR + P conditions since σ versus t_f behavior differs the most within one supplier (see Figures 11 and 12);
- a stress level of $\sim 340 \text{ MP}_a$ ($\sim 50 \text{ k}_\text{Si}$) which is common to both suppliers and all material conditions;
- analysis of anomalous samples such as fractures outside the specimen reduced section (A-6, A-24, B-13, and B-19) and duplicate specimens with different times and strains at failure (A-22, A-24).

The presentation of fractographic results utilizes the matrix in Figure 16. Emphasis is given to fracture features common to most samples.

B. Fractography

The ID surface of a through wall crack for specimen A-27 is shown in Figure 17(a). Several features should be noted. Below the main crack, a large secondary crack (A) and smaller secondary cracks (B) away from the primary cracks are observed. The region of different contrast at the opposite end of the main crack (labeled C) is shown at higher magnification in Figure 17(b) to be composed of many small cracks.

The crack opening at the tube ID varies with stress and plastic strain. This effect is quantified by defining a crack opening angle (COA) as:

$$\text{COA} = \tan^{-1} \left(\frac{\text{ID surface opening}}{\text{wall thickness}} \right) \quad (9)$$

Since COA is calculated for through wall cracks, its usefulness as a fracture mechanics parameter is limited. It is intended to be used to compare the plastic contribution to crack opening for many tests on the same basis.

The formula is applicable to specimens where at least 75% of the through wall propagation is SCC, and where the crack penetrated the tube OD by either a pinhole or tight slit, i.e. the ID opening is much larger than the OD opening. Tests at the highest stress (>440 MPa) do not meet these criteria. COA is plotted against stress in Figure 18. An attempt to plot COA versus plastic strain at failure gives significantly greater data scatter.

At stresses above 350 MPa, many ID surface secondary cracks beyond the ends of the main crack are observed. The main crack has a thumbnail shape as shown in Figure 19(a). Below 350 MPa secondary cracking is much less prevalent, and the crack shape as seen in Figure 19(b) is best represented by a truncated ellipse with the maximum crack length, $2c$, located at a depth one-quarter to one-third of the wall thickness. Although supplier A specimens are used as examples in Figure 19, the observations apply equally to supplier B cladding.

Fracture surfaces are presented in Figures 20-22 for samples A-27, B-12, and B-28, respectively. The photomontages are taken near the center of the crack and extend from the ID to approximately the end of the SCC region near the OD. The fracture appearance depends more on the stress level than on the time to failure. Samples A-27 and B-12 are typical of the high stress (~ 345 MPa) failures while sample B-28 is representative of low stress (221 MPa) failures.

C. Metallography

The specimens examined by metallographic cross-section for tests at 320°C are indicated in the sample plan of Figure 16. Specimens tested at 390°C , Table 8, are also examined. The cracks in each cross-section are counted and their depths are measured. The results are presented as histograms in Figures 23 and 24 for the 320°C and 390°C specimens, respectively. Figure 25 shows several cracks from 390°C test specimens. The short cracks ($<5\mu\text{m}$) in Figure 25(a) and (c) are straight and propagate normal to the hoop stress. The direction of crack propagation is radial on the macroscopic scale for deeper cracks ($>10\mu\text{m}$), but a crack changes direction during propagation on the microscopic (i.e. grain to grain) scale. This behavior is more pronounced in supplier A, Figure 25(b), than in supplier B, Figure 25(d). The long cracks ($>40\mu\text{m}$) in supplier A tubing are branched. The supplier A cracks exhibit more crack opening than cracks in supplier B cladding.

D. Split-ring Tests

Several constant deflection split ring tests are performed. The purposes are: 1) to determine if previous split ring specimens did not fail due to inherent SCC resistance of the material, or due to experimental factors such as stress relaxation in the specimens, and 2) to initiate cracks for subsequent SEM characterization. The test conditions and results are listed in Table 9. No specimens failed by through wall cracking, however, microcracks are observed in supplier B specimens, Figure 26, but not in supplier A specimens.

IV. Discussion

A. SCC Behavior of Suppliers A and B

The results in Figures 11-13 reveal a substantial effect of some materials variable(s) on SCC susceptibility. The test matrix in Figure 9 is designed to separate the difference in iodine SCC susceptibility using the following comparisons:

1. Heat Treatment: AR + P versus SR + P
2. Surface condition: AR versus AR + P
SR + P versus H + SR + P
3. Texture: Supplier A versus supplier B, the same material condition

Synergistic interactions among any of the above materials variables are also possible. Each factor will be considered separately in the following discussion.

1. Effect of Heat Treatment

The 500°C/2 hr stress-relief treatment has two effects: 1) reduction of residual stresses from the tube straightening operation as indicated in Table 3, and 2) change in mechanical properties manifested by a minor reduction in burst strength and probable increase in creep rate. The reduction in burst strength is greater in supplier A than the negligible difference noted in supplier B, Table 5. For comparable stress and time to failure in SCC, the strain at failure is greater for the SR + P than for the AR + P in both suppliers, Figure 14. The difference is much more pronounced in supplier A reflecting a change in the creep properties where the higher creep rate produces more creep strain over a given period of test time. The behavior is consistent with the heat treatment of the materials following

the final tube forming operation. Supplier B tubing is known to receive a higher temperature stress relief treatment than the supplier A tubing so its mechanical properties are less affected by the 500°C/2 hr anneal. A similar trend in mechanical properties with heat treatment is demonstrated by Videm and Lunde [13].

The stress versus time to failure behavior for the AR + P and SR + P is essentially the same for supplier A, Figure 11, and is only slightly different for supplier B, Figure 12, indicating a negligible effect of residual stress on SCC behavior. The creep strain accumulated by the specimen relieves the residual stress for strain greater than the maximum residual strain (residual stress/Young's modulus) [16]. This corresponds to .09% for supplier A/AR condition and .02% for supplier B/AR. The implication is that residual stress is eliminated early in the lifetime of any specimen.

2. Effect of surface condition

a. Pickling

Little effect of a pickling operation on SCC behavior of as-received material is evident. The supplier A specimens for the AR and AR + P conditions have very similar times to failure at a given stress indicating that the different surface conditions shown in Figures 6(a) and 6(c) do not affect SCC behavior. A modest difference in stress versus time to failure between the AR and AR + P conditions in supplier B is evident in Figure 12. In the AR condition the surface with the large pits, Figure 6(b), has a lower time to failure at a given stress than the AR + P condition, Figure 6(d). SEM observations of incipient cracks reveal that many initiate from the large pits, e.g. the crack shown in Figure 26. These conclusions disagree with the work of other investigators who observe a substantial increase in time to failure with pickling in split ring tests [13] and in tube pressurization tests [15].

b. Honing

The effects of mechanical surface preparation (honing, sandblasting, and shot peening) on SCC susceptibility as reported in the literature are varied. Wood [17] reports that shot peening greatly increases time to failure in split ring SCC tests at 300°C. Peehs et al. [18] found no effect of sandblasting or honing on microcrack density in tube pressurization tests at 400°C, but times to failure are not given. Some grit blasted surfaces show a slight increase in SCC susceptibility in tube pressurization

tests at 320°C [19] and 343°C [20], while shot peening substantially reduces SCC resistance at 320°C [19]. It should be noted that no additional surface treatments are performed on these mechanically altered surfaces.

The most substantial effect of material condition on SCC behavior within each supplier is observed in the honed, stress-relieved and pickled specimens. The difference between the results for the SR + P and H + SR + P clearly indicates an effect of the mechanical removal of material by honing which carries through subsequent surface preparation steps. The differences are most prevalent in the high stress specimen A-5 and B-6. The honed surface is the least well characterized of any condition considered in this study since the depth and extent of "damage" is not known. Most likely, a thin layer of heavily worked, gouged metal is present with less deformation as depth from the ID surface increases.

Several mechanisms are postulated [20] to explain the effects of surface finish:

1. oxide film breakdown at surface flaws;
2. local stress concentrations at surface damage locations;
3. initiation through local changes in chemistry;
4. removal of surface layer with a favorable texture;
5. introduction of a less favorable surface texture;
6. introduction of high ID surface tensile residual stresses;
7. introduction of a "damaged", i.e. cold-worked layer without a texture change.

The final pickling operation removes most of the damaged layer and thereby eliminates many of the six possible explanations listed above. Anomalous surface texture [mechanisms 4) and 5)] and local chemistry differences introduced by honing [mechanism 3)] become unlikely. Both the SR + P and H + SR + P conditions have a thin, air formed oxide film easily penetrated by iodine [12] so mechanism 1) is not relevant. The SR treatment makes mechanism 6), high tensile residual stress, unlikely. The pickling operation removes the deep honing gouges so the H + SR + P surface is less rough than supplier B/AR whose large pits have only a marginal impact on SCC performance making mechanism 2) unlikely. The surface pickling might not remove enough metal to completely eliminate the cold worked surface and/or possible small honing cracks. Thus mechanism 7) is the most feasible, albeit unverified, explanation for the increased SCC susceptibility of the H + SR + P condition.

3. Effect of texture

Residual stress and surface finish have a negligible effect on SCC susceptibility so another explanation for the large difference in behavior between supplier A and supplier B cladding must be found. The remaining factor to be considered is texture. The potential effect of an anomalous ID surface texture is precluded since none is found in the texture evaluation of Figures 1-5 and pickling does not significantly alter the behavior between the two suppliers.

Supplier A ($f_T = 0.63$ at ID surface) has a significantly more radial basal pole texture than supplier B ($f_T = 0.54$ at ID surface). Figure 27 shows the radial tangential (R-T) scan for ID surface samples of both suppliers. The peak intensity is at $\phi \approx 35^\circ$ for both suppliers but is sharper and more intense for supplier A. The basal pole intensity at high tilt angles ($\phi > 70^\circ$), where planes are well oriented for transgranular cleavage, is greater for supplier B. Figure 28 compares the textures in another way. Inverse pole figure data for radial samples are used to construct a plot of cumulative basal pole volume fraction versus α . The angle $\alpha = 90^\circ - \phi$ is measured from the edge of the inverse pole figure, i.e. $\alpha = 0^\circ$ corresponds to $\phi = 90^\circ$ from the radial direction, and $\alpha = 90^\circ$ corresponds to $\phi = 0^\circ$. Since the basal poles are most strongly concentrated near the R-T plane, α is a good (but not exact) approximation of the angle measured from the tangential direction toward the radial direction, i.e. the complementary angle to ϕ in the R-T scan. Figure 28 shows that a much larger volume fraction of grains have basal poles near the tangential direction for supplier B tubing than for supplier A tubing. For example, approximately twice the volume fraction of grains in supplier B tubing have basal poles oriented within 35° of the tangential direction than in supplier A tubing (0.36 versus 0.18).

Support for a texture effect comes from several sources. The tests by Peehs et al. [18] on tubing machined from heavily textured plate indicates that initiation is texture dependent. Tube pressurization tests on tubing with various textures [19,20] prove that the more radial basal pole texture has both a higher threshold stress and a longer time to fracture at any stress above threshold than tubing with more tangentially oriented basal poles. This is the exact trend observed for suppliers A and B. Later discussion considers the texture effect on the initiation and propagation stages of iodine SCC.

The largest variations in stress versus time to failure within a supplier are with groups of samples with the same material/surface condition. Figure 13 includes the SRI, International test results [3-5] on the same A and B tube lots in the AR condition. Slightly longer times to failure are observed for supplier B, but the behavior is much different for supplier A, particularly the longer failure times near the stress threshold. Results on SR + P specimens are reported in Tables 6 and 7 and are plotted in Figure 29. The specimens for the tests in Table 6 were received as a group at the same time, Group A, so they likely came from the same tube or similar tubes. The specimen for the tests in Table 7 were received much later so they likely came from different archive tubes and are termed Group B. The other processing steps and material characteristics discussed previously (residual stress, surface condition, deformation characteristics) could not cause the magnitude of the observed differences since they have a marginal effect on SCC susceptibility. This leaves within lot texture variation as the most likely explanation.

The substantial difference in SCC susceptibility between supplier A and B is reflected in the plots of strain and average strain rate versus time-to-failure, Figures 14 and 15, respectively. To understand this behavior, information on the creep behavior of these materials is required. Figure 30 shows creep curves at 138 MPa (20 ksi) and 300°C [24]. Supplier B is much more creep resistant than supplier A because the more transverse texture has a lower transverse creep strain. The same trend in creep behavior with texture is observed over the temperature range 300°C [22] to 400°C [24,28]. For a comparable time-to-failure, texture causes supplier B specimens to accumulate less strain. This also translates to a lower average creep rate calculated with Eqn. (8). Figure 30 shows that both lots experience a decreasing creep rate with time in the transition from primary to secondary creep behavior, therefore, Eqn. (8) is only an approximation of the actual creep behavior. Within one supplier an effect of the SR heat treatment is evident where higher strains (and therefore average strain rates) are generally observed in the heat treated material. This trend is opposite to the trend observed by Kallstrom et al. [8] and could be due to differences in prior material processing steps such as degree of cold work and stress relief anneals.

4. Threshold behavior for iodine SCC

Much controversy exists regarding the threshold(s) controlling the SCC process. Arguments have been made for stress, stress intensity, strain, and strain rate. A detailed review and analysis of literature data is not attempted here, however, several relevant observations are made using the present data and the results of other investigators. The concept of a single valued stress or strain threshold is not valid. Texture must be considered explicitly in discussion of a stress threshold at a constant temperature. A temperature effect is indicated by the limited results at 390°C in Table 8 and by the more extensive results of others [14, 23]. The effect of strain and strain rate must be analyzed in terms of the deformation characteristics of the material. Reports of strain versus time-to-failure [15,23] resemble the behavior of supplier A, however, the texture in the case of Jones et al. [23] is very similar to the texture of supplier A. A minimum in strain versus time-to-failure is observed in some cases [15,23], but is not seen either in the supplier B or in the data scatter of the supplier A tests.

The role of stress, strain, and strain rate must be evaluated in terms of the critical steps in the SCC process:

1. crack initiation (film rupture and crack formation), and
2. crack propagation (environmentally assisted growth of the flow to a critical size and final ductile rupture).

Previous attempts to correlate SCC behavior with mechanical variables have not separated stress, strain, and strain rate effects on these separate steps. This is one explanation for the poor correlation with strain and strain rate, i.e. values for total strain at fracture and for "average" strain rate do not provide any insight to the potentially different processes occurring during initiation and propagation. It is probable that no single mechanical parameter controls SCC behavior for all stresses and during all stages of cracking. For example, it is conceivable that one quantity such as strain controls the crack initiation process while another quantity such as stress intensity controls the propagation. This is not proposed as the only explanation but as an example of one of many possible SCC scenarios. It represents the type of transition that might be applicable. The following sections consider the initiation and propagation portions of SCC separately with consideration of the chemical, materials, and mechanical effects on each step.

B. Crack Initiation

1. Analysis of experimental results

a. Iodine attack

The crack initiation process has two stages: 1) penetration of iodine through the surface oxide film to the underlying metal, and 2) formation of a microcrack or "crack nucleus". The former process occurs readily in the tube pressurization tests. The very thin air-formed film on the surface is penetrated without the application of strain [12]. The ID surface of the samples and unstressed cladding segments reveal slight pitting. Hydrolysis of residual iodides occurs at the end of the test when specimens are opened to the air indicating complete reaction of the initial iodine charge.

The initial vaporization of the iodine crystals results in a relatively high iodine partial pressure which decreases quite rapidly due to attack of the ID surface with zirconium iodide formation. In some cases lower iodides or oxyiodides are detected on the ID and crack surfaces after the test by energy dispersive analysis in the SEM.

The iodine exposure of unstressed cladding segments indicates that iodine attack occurs preferentially at particles. Figure 31 shows the series of steps starting with general attack of some regions of the surface. If particles are present in that region of general attack, localized attack occurs along the matrix/particle interface resulting in final removal of the particles. Analysis shows Zr, Cr and Fe to be present. Very few particles are found to contain any Ni which is also known to be present in certain types of intermetallic particles [34]. No localized attack is evident at pre-existing pits (0.5 - 1 μ m in size). The behavior described here is observed in both suppliers in both the as-received and pickled surface conditions. The behavior of slotted ring specimens in a constant flowing iodine partial pressure is somewhat different. At 40 Pa (0.3 torr) extensive pitting and attack at particles is seen. At 4 Pa (0.03 torr) little attack is observed but cracking still occurs as seen in Figure 26.

b. Crack formation

Before further discussion of crack initiation can proceed, it must be determined when a crack actually exists. Examination of failed specimens by metallography identifies cracks two microns in depth. SEM observation of incipient secondary cracks on the ID surface detects cracks

on the order of four microns in length as seen in Figure 17(b). An "initiated crack", therefore, is established by the time a depth of one grain is reached and probably for cracks smaller than that size.

Two sources of information on crack initiation are available: the slotted ring test in Table 9 and the analysis of incipient cracks reported in Figures 23 and 24. For pressurized tube tests the number of incipient cracks is comparable for both suppliers at 320°C although a few deeper cracks are observed in supplier A. At 390°C more cracking is evident in terms of both number and depth for supplier A compared to supplier B. This behavior is explained by considering the complementary effects of plastic strain, texture, and time at stress.

The effect of plastic strain is most clearly demonstrated in Figure 24 for the 390°C tests. When supplier B samples accumulate plastic strain levels comparable to supplier A samples, e.g. specimen B-38, the number and distribution of microcracks becomes nearly equal. Sample B-34 exhibits fewer microcracks than any other sample which correlates with its low plastic strain ($\epsilon_p \sim 0.26\%$) compared to other samples ($\epsilon_p > 1.2\%$). Low strain values in long time tests of supplier B specimens at 320°, e.g. B-16, are associated with few microcracks. The presence of numerous microcracks in the plastic zone on the ID surface of the major cracks as in Figure 17 is further indication that plastic strain promotes microcracking.

c. Texture effect

Texture also affects crack initiation. An experiment by Peehs et al. [18] is particularly relevant here. A tube is machined from heavily textured plate giving a strong circumferential texture gradient. It is tested by tube pressurization at 400°C with some iodine. Microcracks formed preferentially where basal poles are located 50° to 75° from the radial direction. Very few microcracks form where basal poles are normal to the tube surface. This microcrack distribution indicates that crack initiation depends on texture. The slotted ring test results agree with this hypothesis since microcracks as seen in Figure 26 form in the more tangentially oriented supplier B material as opposed to no microcracks in supplier A material for equivalent test conditions (temperature, time, environment, material condition, and initial stress). If a strong tangential texture is more susceptible to cracking, then more cracks should be observed in the metallographic

cross-sections of supplier B than supplier A. This is not the case since the trend reveals a slightly higher microcrack density in supplier A than in supplier B. The possible role of strain in this apparent anomaly has already been discussed. It should also be noted that at higher stresses, the relatively rapid times to failure in supplier B might preclude the development of significant numbers of microcracks, i.e. rapid propagation occurs before sufficient strain/time are able to nucleate additional cracks. The role of texture in crack propagation is discussed in the section dealing with crack propagation.

2. Analysis of sites and mechanism

No self-consistent picture of the crack initiation process that accounts for most experimental observations has yet emerged. An extensive search for microstructural features contributing to or causing initiation is performed, but no microstructural feature is conspicuous in the initiation region for any crack in either supplier. Several initiation sites are reported in previous studies:

1. Intermetallic and inclusion particles [26,27]
2. Intergranular sites [12,14,15,25-28,30]
3. Pits and flaws [12,14,20,26,31,35]
4. Hydrides [32]
5. Transgranular sites [14,25,29]

Evidence for each site will be reviewed incorporating observation from this study. An initiation mechanism that considers all of the above sites will then be proposed.

a. Evaluation of initiation sites

Iodine attack prefers two types of sites in material with a thin oxide coating: particles [present work, 26,27] and intergranular sites [26,27]. Intergranular sites are most evident in recrystallized material, but inconsistencies exist regarding the stress level for attack. Cubicciotti et al. [27] found intergranular attack below the threshold stress and initiation at intergranular particles above the threshold stress while Videm and Lunde [26] report the opposite result, i.e. attack at particles below the threshold stress and intergranular attack with no particles present above the threshold stress. The G.E. group [28,30] reports intergranular initiation in both irradiated and unirradiated recrystallized material.

Although intergranular initiation is clearly preferred in recrystallized material, any role of intergranular initiation in cold worked material is difficult to define because of the heavily deformed and twinned microstructure. Areas of indeterminate features are observed which are judged to be intergranular initiation sites [12]. Similar features are sometimes observed at suspected initiation sites of through-wall cracks in this study. Although these features are clearly not transgranular, they are not sufficiently distinct to be confirmed as intergranular features. The preferential attack at particles in unstressed material is not observed to result in crack initiation in stressed specimens based on examination of small incipient cracks and regions associated with initiation of through-wall cracks. Initiation is associated with either the indistinct features (possibly intergranular) or transgranular propagation. Propagation from the crack initiation sites become clearly transgranular at depths no more than a few grains diameters from the tube ID as seen near the tube ID in Figures 20-22.

Two sources of surface defects must be considered. Crack initiation occurs from pre-existing flaws and from the large pits of supplier B/AR condition and from surface treatment defects reported from other work [4,20,35]. An alternate source of surface flaws is iodine pitting attack on the Zircaloy surface. Stress is known to enhance this pitting attack [21]. When an impervious oxide film or oxidizing environment is present, attack occurs at oxide cracks which are not necessarily associated with particles or grain boundaries [12,26,31].

b. Microstructural mechanism for crack initiation

An evaluation of the initiation process must include considerations of mechanism to resolve the fact that many initiation sites are observed. Plastic deformation is known to be a prerequisite for initiation, therefore, slip might be the key factor leading to initiation at any number of potential sites. Peehs et al. [33] noted extensive microcracking at 400°C where creep rates are expected to be rapid. At high strain levels, increased microcracking is promoted as seen in Figures 23 and 24. Gangloff and Coffin [29] report a strain threshold and more numerous cracking with increased strain levels beyond the threshold. An additional requirement is strain localization which is necessary to build up local stress levels at potential initiation sites. Slip band formation due to channeling is observed to cause

intergranular crack initiation in irradiated material [20]. In another case intergranular microcrack formation is inferred from difference in slip behavior in grains adjacent to microcracks [27].

The dual requirements of strain accumulation and strain localization are capable of explaining texture, heat treatment, and irradiation effects on initiation. The role of texture is two fold: it promotes larger plastic strain at a given stress for radial textures, and it provides tangential basal poles properly oriented for transgranular initiation in the more tangential textures. The cleavage event leading to initiation could result from local stress buildup similar to the case of intergranular initiation. As annealing temperature increases, larger initiation strains are necessary to cause strain localization. For recrystallized material, several percent strain is required to cause cracking since a dislocation structure capable of supporting local stress concentration must be established. This is supported by observations on recrystallized material [27,30]. Intense slip bands, or channels, are known to develop when recrystallized and irradiated Zircaloy is strained a small amount which accounts for the reduced strain to failure in SCC.

The site that is activated depends on several factors: texture, surface condition, flaws, pits, second phase particles, and intermetallic particles, microstructure (cold worked versus recrystallized), residual cold work in cold-worked and stress-relieved material, and irradiation. With sufficient strain and strain localization, the easiest site is activated followed by secondary sites. This accounts for the observation of several types of sites on the same specimen and for changes in the type of site with stress level [26,27].

C. Propagation

1. Microstructure observations

Both macroscopic and microscopic fracture features depend on stress and texture for supplier A and B materials. At high stress (>345 MPa) aligned or nearly aligned cracks tend to link to become a single crack. This large crack propagates to failure and is flanked by several small secondary cracks as shown in Figures 17(a) and 19(a). In specimen B-24 two cracks of equal size impinged and propagated to failure. The problem of aligned cracks is modeled by Peehs et al. [33]. At lower stress (<345 MPa),

one large crack is observed as in Figure 17(b). An interesting aspect of the low stress cracks is the final shape of the crack front where the maximum crack length, $2c$, is located at one quarter to one third of the wall from the ID rather than at the ID. This indicates that the strain field at the end of the main crack, is not sufficiently large to produce smaller secondary cracks.

Texture has an effect on crack propagation in the tubing. Examination of incipient cracks in supplier B samples, Figure 25(b), reveals propagation on a microscopic (grain to grain) scale that is more normal to the applied hoop stress than supplier A, Figure 25(d). More crack branching is found in supplier A samples. This behavior is related to the larger volume fraction of basal planes with proper orientation for cleavage, i.e. near radial, as shown in Figure 28.

An alternate explanation for the texture effect is provided by the study of crack arrest in fracture mechanics specimens [1,2]. The lower K_{ISCC} for a texture like supplier B compared to supplier A affects the propagation behavior in two ways. For a given stress above threshold and crack length, the crack velocity is greater in a supplier B texture than in a supplier A texture. For a given crack length, a crack propagates at a lower stress in supplier B texture compared to a supplier A texture. Without considering the initiation stage, the result is both a shorter time to failure at equivalent stress levels and a lower threshold stress for a texture like supplier B.

Examination of the fracture surfaces in Figures 20-22 provides further evidence of a stress and texture dependent on crack propagation. Figures 20 and 21 are micrographs of specimens from suppliers A and B, respectively, at approximately 345 MPa (50 ksi). Neither surface is macroscopically flat and both show cleavage features with small facets and tearing between facets. Figures 21 and 22 compare supplier B at two stress levels, 345 MPa and 221 MPa (32 ksi) respectively. The specimen at lower stress shows propagation with larger cleavage facets, with less tearing between facets, and with a more macroscopically flat surface than the higher stress specimen. The stress intensity and crack velocity is lower for any given crack length at 221 MPa than at 345 MPa. The difference in failure appearance is exactly analogous to the behavior in the fracture mechanics specimen. High K /high velocity produces small cleavage facets and tearing between facets while

low K near K_{ISCC} /low velocity produces large cleavage facets and substantially less tearing between facets. In conclusion, the microscopic fracture features in the tubing SCC tests depend primarily on stress level with a secondary texture (supplier) dependence and are in qualitative agreement with the results from fracture mechanics specimens. The stress level controls the stress intensity, or other relevant fracture mechanics parameter, and the crack velocity. The texture controls the microscopic propagation where crack advance is more difficult, i.e. requires higher stress, when few basal planes are well oriented for cleavage.

The texture effect is expected to be most critical for short cracks. Once nucleated, a short crack can arrest if it encounters grain(s) not well oriented for cleavage. This is one explanation for the somewhat larger number of incipient cracks in supplier A at 320°C and the tendency to have cracks in the 5-20 μ m range. Alternatively, a crack nucleated in material well oriented for cleavage tends to propagate readily. If this situation is to apply to supplier B material, initiation must be somewhat difficult, particularly at low stress/long time to failure to account for the small number of incipient cracks observed at 320°C.

2. Relative contribution of initiation and propagation time

Consideration is now given to the separation of the time to failure into an initiation time and propagation time. Other investigators have attempted such a separation by direct or indirect means. Peehs et al. [18] find that approximately 55% of a ≤ 3 hr split ring test at 400°C is consumed by incubation followed by an enhanced deformation period which is correlated with crack propagation. A tube pressurization test by Peehs et al. [33] at 400°C and 225 MPa showed a strong acoustic emission signal within the first 20 minutes of the 600 minute test which is interpreted as rapid crack initiation. Videm and Lunde [26] test smooth and precracked cladding segments of recrystallized material at 340°C. They conclude that roughly three-quarters of the lifetime is consumed by crack initiation for times to failure in the approximate range 30-1200 minutes and that the initiation fraction of life increases as the temperature decreases. The large number of microcracks observed at 390°C compared to 320°C indicates that cracks initiate more readily at high temperature, consistent with the results of other investigators [26,33].

At 320°C analysis of the limiting cases must be used to define the relative initiation propagation fraction of lifetime. If initiation occurs readily and most of life is spent in propagation, many microcracks would be evident. This is clearly not the case. If initiation requires most of specimen life, very few microcracks would be present and crack opening angle would be small since little creep strain would be accumulated during propagation to open the crack. Supplier A and supplier B must be compared separately regarding this point. Few microcracks are found in supplier A but several have propagated beyond a depth of one or two grains. A relatively large crack opening angle is shown in Figure 18. A best guess is that crack initiation occurs in the middle third of specimen lifetime. Few microcracks are also found in supplier B. Crack opening angles decrease with stress. However, the total creep strain also decreases, so this trend is not unexpected. At high stresses (>345 MPa), the COA is comparable to supplier A despite lower strain at failure and shorter life time so initiation occurs quite rapidly, probably at less than half the lifetime. At low stresses (<300 MPa) both strain and COA are small so a judgement is not clearcut. More time is spent in propagation at lower stress than at high stress on an absolute scale based on a comparison of fractography in Figures 21 and 22. Considering the limited number of microcracks formed over the length of time for the low stress tests, initiation is possibly delayed to later in life, possibly until the order of two thirds of lifetime is accumulated.

In conclusion, initiation requires most of the reported times to failure with the possible exception of the high stress levels in susceptible (supplier B) material. This conclusion is supported by the tests on tubing of different wall thicknesses, Table 7, where no consistent difference in time to failure emerges between the two wall thicknesses. An important point is that the absolute times for initiation must be quite different for the two suppliers to account for the appreciable differences in behavior. Some, but not all, the difference can be justified on the basis of variation in the rates of crack propagation between supplier A and supplier B.

D. Split Ring Tests

The two goals of these tests are to explain the relative iodine SCC susceptibilities in the split ring test for different material conditions and to produce SCC features for microscopic examination when cracking occurs. Failure in the split ring test is known to depend on both the stress relaxation behavior and the inherent susceptibility of the material [20,36]. The requirement for failure is that the stress must remain high enough for a length of time sufficient for the failure curve to be reached. The stress relaxation behavior controls the stress level. The material condition ("susceptibility") determines the location of the failure curve. A comparison of the initial and final stress values for suppliers A and B, Table 9, shows that the amount of stress relaxation is only slightly greater for supplier B than supplier A. The failure curves from the tube pressurization tests are very different as seen in Figures 11-13. For the times and stresses attained in split ring tests of supplier A, it is clear that these specimens remained far from the failure curve, therefore, the absence of SCC cracks is expected. For the supplier B split ring tests, the time and stress levels bring these specimens very close to the failure curves of this more susceptible material. Therefore, the detection of microcracks is not surprising.

V. Summary and Conclusions

Tube pressurization tests are used to determine differences in stress versus time to failure for two different lots of Zircaloy-2 tubing. Macroscopic examination and scanning electron microscopy are utilized to evaluate the fracture features. The conclusions are:

1. Substantial differences are observed in stress versus time to failure behavior of cladding from different suppliers;
2. The difference between suppliers is due to crystallographic texture differences where a more tangential texture results in greater susceptibility;
3. Other materials variables such as surface finish and heat treatment temperature have a much smaller effect on SCC susceptibility than the texture effect;

4. Residual stress has no effect on SCC susceptibility in tube pressurization tests;
5. A consistent interpretation of many observations of crack initiation is possible if strain accumulation and strain localization are necessary steps in the initiation process;
6. Crack propagation is dependent on both stress and texture as determined by fractography;
7. At 320°C lifetime is crack initiation limited while at 390°C crack initiation is more readily accomplished.

References

1. D.B. Knorr and R.M. Pelloux, Progress Report 5 covering January 1, 1980 to June 30, 1989, dated July 28, 1980.
2. D.B. Knorr and R.M. Pelloux, "Effects of Texture and Microstructure on the Propagation of Iodine Stress Corrosion Cracks in Zircaloy", submitted to Met. Trans.
3. D. Cubicciotti, R.L. Jones, and B.C. Syrett, EPRI Report NP-1329, March 1980.
4. B.C. Syrett, D. Cubicciotti, and R.L. Jones, J. Nucl. Mater., 92, 89 (1980).
5. B.C. Syrett, D. Cubicciotti, and R.L. Jones, ANS Topical Meeting on Light Water Reactor Fuel Performance, Portland, p. 113, 1979.
6. D.B. Knorr and R.M. Pelloux, J. Nucl. Mater., 71, 1 (1977).
7. J.J. Kearns, WAPD-TM-472 (1965).
8. K. Kallstrom, T. Andersson, and A. Hofvenstom, ASTM STP 551, p. 160 (1974).
9. G. Sacks and G. Espey, Trans. AIME, 147, 348 (1942).
10. K. Une, J. Nucl. Sci. Tech., 14, 610 (1977).
11. J.C. Wood, J. Nucl. Mater., 45, 105 (1972/73).
12. B. Cox and J.C. Wood, Corrosion Problems in Energy Conversion and Generation, C.S. Tedman, ed., p. 275, The Electrochemical Society, New York (1974).
13. J.C. Wood, J. Nucl. Mater., 45, 105 (1972/73).
14. C.C. Busby, R.P. Tucker, and J.E. McCauley, WAPD-TM-1149 (1974).
15. K. Videm and L. Lunde, Ann. Nucl. Energy, 3, 305 (1976).
16. F.A. McClintok and A.S. Argon, Mechanical Behavior of Materials, Addison-Wesley, Reading, MA, p. 433 (1966).
17. J.C. Wood, Nucl. Tech., 23, 63 (1975).
18. M. Peehs, H. Stehle, and E. Steinberg, ASTM STP 681, p. 244 (1979).
19. B.C. Syrett, D. Cubicciotti, and R.L. Jones, "The Effect of Texture and Surface Condition on the Iodine Stress Corrosion Cracking Susceptibility of Unirradiated Zircaloy-2", submitted to J. Nucl. Mater.

References (continued)

20. H.W. Wilson, K.K. Yoon, and D.L. Baty, ANS Topical Meeting on Light Water Reactor Fuel Performance, Portland, p. 246 (1979).
21. R.L. Jones, D. Cubicciotti, and B.C. Syrett, J. Nucl. Mater., 91, 277 (1980).
22. P.A. Ross-Ross, V. Fidleris, and D.E. Fraser, Can. Met. Quart., 11, 101 (1972).
23. R.L. Jones, D. Cubicciotti, and B.C. Syrett, J. Nucl. Mater., 91, 277 (1980).
24. Exxon Nuclear Company Report XN-NF-77-21, EPRI Contract RP 895-1-4, October 1977.
25. K. Videm and L. Lunde, ANS Topical Meeting on Water Reactor Fuel Performance, St. Charles, p. 274 (1977).
26. K. Videm and L. Lunde, ASTM STP 681, p. 229 (1979).
27. D. Cubicciotti, S.M. Howard, and R.L. Jones, J. Nucl. Mater., 78, 2 (1978).
28. D.S. Tomalin, R.B. Adamson, and R.P. Gangloff, ASTM STP 681, p. 122 (1979).
29. R.P. Gangloff and L.F. Coffin, G.E. Report 79 CRD016, February 1979.
30. D. Lee and R.B. Adamson, ASTM STP 633, p. 385 (1977).
31. R.P. Gangloff, D.E. Graham, and A.W. Funkenbusch, Corrosion, 35, 316 (1979).
32. B. Cox, ASTM STP 681, p. 306 (1979).
33. M. Peehs, H. Stehle, and E. Steinberg, IAEA Specialists Meeting on Pellet Clad Interaction in LWR's, Paper 11, Riso, September 1980.
34. P. Chemelle, SM Thesis, MIT, September 1980.
35. D. Cubicciotti, B.C. Syrett, and R.L. Jones, "Influence of Surface Condition on Crack Initiation in Iodine Induced Stress Corrosion Cracking of Zircalloys", submitted to Nucl. Tech.
36. R. Sejoha and J.C. Wood, ASTM STP 681, p. 261 (1979).

Table 1

Chemical Composition of Tubing from Suppliers A and B

<u>Element (a)</u>	<u>Supplier A (b)</u>	<u>Supplier B (b)</u>
Sn (%)	1.47 (0.01)	1.51 (0.02)
Fe (%)	0.15 (0.02)	0.14 (0.08)
Cr (%)	0.11 (0.01)	0.07 (0.01)
Ni (%)	0.07 (0.004)	0.05 (0.004)
O	1170	1395
H	12	7
N	32	30
C	176 (12)	200 (35)
Al	44 (2)	40 (5)
Si	73 (5)	56 (1.0)

(a) Quantities in ppm unless otherwise noted

(b) Average of three analyses; standard deviation is shown in brackets where available.

Table 2

Texture Numbers for Zircaloy Tubing

<u>Supplier</u>	<u>Location</u>	Texture Numbers		
		<u>f_r</u>	<u>F_T</u>	<u>$(SD)_T$</u>
A ↓	ID surface	0.628	0.264	0.243
	0.10 mm from ID surface	0.647	0.329	0.241
	0.47 mm from ID surface	0.625	0.263	0.247
	0.10 mm from OD surface	0.594	0.167	0.258
B ↓	ID surface	0.538	0.161	0.247
	0.10 mm from ID surface	0.550	0.152	0.269
	0.47 mm from ID surface	0.543	0.119	0.274
	0.10 mm from OD surface	0.498	0.125	0.278

Table 3
Residual Stress Data

<u>Supplier</u>	<u>Condition</u>	<u>Residual Stress at Tube ID (ksi) (MPa)</u>	
A	AR	-15.9	-110
	SR	- 3.0	- 21
B	AR	- 2.7	- 19
	SR	- 1.5	- 10

*Assume linear stress gradient across tube wall.

Table 4
Surface Condition

As-Received (AR)

Supplier A: Smooth (4 μ m peak-to-peak)*
 Second phase and inclusion particles
 imbedded in surface

Supplier B: Rough (9 μ m peak-to-peak)
 Pitted surface
 A) Large pits 10-20 μ m
 B) Small pits 0.5-1 μ m

Pickled (P)

Both Suppliers: Smooth (approaching Supplier A
 As-Received
 Small pit population 0.5-1 μ m

Honed + Pickled (H+P)

Both Suppliers: Rougher than pickled only surfaces
 (intermediate between suppliers
 A and B As-Received)
 Small pit population 0.5-1 μ m

*After Syrett et al., J. Nucl. Mater., 92 (1980) 102

Table 5
Tube Burst and Creep Rupture Data

<u>Supplier</u>	<u>Heat Treatment</u>	<u>Rupture Strength</u> (MPa) (ksi)		<u>Time to Failure (min)</u> ¹	<u>Uniform Strain(%)</u>
A ↓	AR ↓	527	76.4	ramp	1.8
		540	78.3	ramp	2.2
		465 $\pm$ 8	67.5 $\pm$ 1.1	384.5	2.9
		441 $\pm$ 3	64.0 $\pm$ 0.5	3689.5	4.3
	↓	421 $\pm$ 14	61.1 $\pm$ 2.0	100014.1(nf)	-
	SR ↓	445	64.5	ramp	4.5
		447	64.9	ramp	1.8
B ↓	AR ↓	474	68.7	ramp	0.81
		465	67.5	ramp	1.0
		463	67.2	ramp	-
		432 $\pm$ 5	62.7 $\pm$ 0.7	138.7	1.4
	↓	416 $\pm$ 19	60.4 $\pm$ 2.7	10000.0(nf)	-
	SR ↓	443	64.3	ramp	2.3
		465	67.4	ramp	2.5

(1) ramp indicates constant loading (pressurization) rate to failure, duration 3-5 minutes.

(nf) means no failure for specimen.

Table 6

IODINE SCC DATA FOR INTERNAL GAS PRESSURIZATION TESTS AT 320°C

Condition ¹	Sample ²	Stress		Time to Failure (min)	Strain at Failure (%)	Comment
		(MPa)	(Ksi)			
AR	A-12	476	69.1	143.1	1.9	
	A-11	421	61.1	340.7	0.78	
	A-15	345	50.0	634.2	0.53	
	A-17	343	49.8	809.0	0.37	
	A-18	314	45.5	1788.1	0.35	
	A-16	272±5	39.5±0.7	8600	0.37	No failure
AR+P	A-23	474	68.7	108.9	1.14	
	A-19	421	61.0	243.7	0.74	
	A-21	390	56.5	413.1	0.39	
	A-20	349	50.6	2157.4	0.51	
	A-24	333	48.3	1906.6	0.35	Fail ¼" outside reduced section
	A-22	325	47.1	2280.4	0.33±0.03	Fail outside reduced section
SR+P	A-28	423	61.3	287.5	1.9	
	A-26	403	58.5	405.7	1.68	Several cracks penetrate wall
	A-27	345	50.0	1390.4	0.60	
H+SR+P	A-5	366	53.1	140.5	1.0	
	A-6	310	45.0	1141.9	<0.53	Fail outside reduced section
	A-9	292	42.3	2088.0	0.47	
	A-10	276	40.0	5680.0	0.66	
AR	B-10	435	63.1	48.3	0.59	
	B-12	347	50.3	99.7	0.20	
	B-13	283	41.1	276.9	--	Fail 11/16" outside reduced section
	B-14	277	40.2	261.7	0.14	
	B-15	242	35.1	1098.9	0.09	
	B-16	225	32.6	2067.8	0.07	
	B-18	208	30.2	2715.0	0.10	
	B-19	165	24.0	1463.9	--	Fail near end fitting

Table 6 (continued)

<u>Condition</u> ¹	<u>Sample</u> ²	<u>(MPa)</u>	<u>(ksi)</u>	<u>Time to Failure (min)</u>	<u>Strain at Failure (%)</u>	<u>Comment</u>
AR+P	B-20	413	59.9	75.7	0.43	
	B-21	345	50.1	179.2	0.16	
	B-22	275	39.9	540.6	0.10	
	B-26	220	31.9	2430.0	0.09	
SR+P	B-25	418	60.6	53.1	0.92	
	B-24	345	50.0	217.6	0.32	
	B-27	276	40.0	892.9	0.14	
	B-28	221	32.0	5334.5	0.10	
H+SR+P	B-6	376	54.5	50.1	0.49	
	B-7	310	44.9	426.1	0.20	

Notes¹AR = As received

P = Pickled, 1.5 - 2.0 mils removed

SR = Stress-received at 500°C for 2 hours

H = Honed, coarse grit, ~0.5 mils removed

²Samples listed as "A" are Supplier A

Samples listed as "B" are Supplier B

Table 7

Iodine SCC Tests for Tubing Specimens with Different Wall Thicknesses

Nominal Stress			Nominal 0.74 mm (29 mil) Wall				Nominal 0.36 mm (14 mil) Wall			
Supplier	(MPa)	(ksi)	Specimen	t_f (min)	ϵ (%)	σ (MPa)	Specimen	t_f (min)	ϵ (%)	σ (MPa)
A	414	60	A-30	108.0	>0.96	412	A-34	23.2	>0.33	421
			A-31	64.3	>1.33	424	A-35	2.3	>3.2	419
	331	48	A-32	788.3	0.61	334	A-36	891.4	0.50	331
			A-39	1015.5	0.45	338	A-37	928.7	0.74	338
	379	55	B-29	32.0	0.57	377	B-31	75.4	0.30	383
			B-30	27.6	0.27	379	B-32	105.1	0.28	383
B	241	35	B-33	1304.8	<0.08	243	B-36	2132.8	0.11	250
			B-35	1301.1	0.08	240	B-37	892.1	0.20	347

Material Condition: SR+P

Test Temperature: 320°C

Table 8

Iodine SCC Tests on Tubing at 390°C

<u>Supplier</u>	<u>Condition</u>	<u>Sample</u>	<u>Stress</u>		<u>Time to Failure (min)</u>	<u>Strain at Failure (%)</u>
			<u>(MPa)</u>	<u>(ksi)</u>		
A	AR	A-33	283	41.0	320.0	1.31
	SR+P	A-38	252	36.6	377.1	1.26
	AR	B-34	223	32.3	163.0	0.26
B	SR+P	B-38	138	20.0	6900(nf)	
			169	24.5	2700(nf)	
			210	30.4	3300(nf)	
			238	34.5	209.8	1.24

Table 9
Constant Deflection Split Ring Tests in 4 Pa (0.03 torr)
Flowing Iodine Environment

<u>Supplier</u>	<u>Condition</u>	<u>Temperature</u> <u>(°C)</u>	<u>Initial Stress</u>		<u>Time of</u> <u>Exposure</u> <u>(min)</u>	<u>Final Stress</u>		<u>Microcracks</u> <u>Observed</u>
			<u>(MPa)</u>	<u>(ksi)</u>		<u>(MPa)</u>	<u>(ksi)</u>	
A	AR	320	374	54.3	300	265	38.5	No
	AR+P	300	379	54.9	360	295	42.8	No
B	AR	320	367	53.3	300	242	35.1	Yes
	AR+P	300	387	56.1	360	284	41.2	Yes

Figure 1
Basal Pole Figure for Supplier A Tubing, ID Surface

Figure 2
Basal Pole for Supplier A Tubing, 0.1 mm from Tube ID

Figure 3
Basal Pole Figure for Supplier B Tubing, ID Surface

Figure 4
Basal Pole Figure for Supplier B Tubing, 0.1 mm from Tube ID

Figure 5

Through-Thickness Texture Numbers for Tubing

Figure 6 SEM micrographs of ID surface for supplier A and supplier B cladding in the as-received and pickled conditions

Figure 7
Schematic Drawing of Tube Pressurization Facility

Figure 8

Schematic cutaway of the specimen sealing system and internal components

AR = AS-RECEIVED

P = PICKLED, 37-50 MICRONS REMOVED
FROM TUBE ID

SR = STRESS-RELIEVED AT 500°C/2 HR

H = HONED WITH COARSE GRIT, ~13 MICRONS
REMOVED FROM TUBE ID

TEST TYPE : INTERNAL GAS PRESSURIZATION

TEST TEMPERATURE : 320°C

Figure 9

Test Matrix for Zircaloy-2 Tubing

Material Condition: SR + P

Test Temperature: 320°C

Figure 10
Test Matrix for Variable Wall Thickness Specimens

Figure 11
Hoop Stress versus Time to Failure for Supplier A Tubing at 320°C

Figure 12

Hoop Stress versus Time to Failure for Supplier B Tubing at 320°C

Figure 13

Comparison of Supplier A and Supplier B in the AR Condition at 320°C

Figure 14

Strain versus Time to Failure for Iodine SCC Tests at 320°C

Figure 15

Average Strain Rate versus Time to Failure for Tests at 320°C

Figure 16
Sampling Plan Summary for Iodine SCC Tests at 320°C

Figure 17

SEM micrographs of failure site from tube pressurization specimen.

a) A through-wall iodine SCC crack is seen from the ID surface of specimen A-27. Large (A) and small (B) secondary cracks are evident.

b) The region of different contrast (C) in a) is shown at high magnification to be composed of numerous small cracks.

Figure 18

Correlation Between Hoop Stress and Crack Opening Angle for 320°C Tests

a) Specimen A-11 $\sigma = 421$ MPa $t_f = 340.7$ min.

b) Specimen A-15 $\sigma = 345$ MPa $t_f = 634.2$ min.

Figure 19

Macrophotographs of supplier A specimens showing well-defined regions of stress corrosion crack propagation.

Figure 21

59

SEM composite of the fracture
surface for specimen B-12

AR condition

$\sigma = 347 \text{ MPa}$

$t_f = 99.7 \text{ min.}$

$\epsilon = 0.20\%$

3mm —

2mm —

1mm —

ID

10 μm

6mm —

5mm —

4mm —

Figure 22

SEM composite of the fracture
surface for specimen B-28

SR+P condition

$\sigma = 221 \text{ MPa}$

$t_f = 5334.5 \text{ min.}$

$\epsilon = 0.10\%$

Figure 23
Distribution of Incipient Microcracks for Failed Iodine SCC
Specimens Tested at 320°C

Figure 24
Distribution of Incipient Microcracks for Failed Iodine SCC
Specimens Tested at 390°C

Figure 25

Incipient cracks revealed in metallographic cross-sections of tubing specimens
from tests at 390°C

SUPPLIER

A

20 μ m

SUPPLIER

B

Figure 26

SEM micrograph of the ID surface of a split ring test specimen is shown. The supplier B cladding in the as-received condition is exposed to a flowing argon + 4Pa (.03 torr) iodine gas stream at 320°C for 300 minutes. A microcrack is evident in the center of the micrograph.

+ = Supplier A ID surface
= Supplier B ID surface

Figure 27
Radial-Tangential Scan for Supplier A and Supplier B ID Surface

Figure 28

Variation of Basal Pole Volume Fraction with Tilt Angle

Figure 29

Comparison of Stress versus Time to Failure for Different Specimen Groups

Figure 30
Biaxial Creep Test Results Plotted as Circumferential Strain
Versus Time for Tests at 300°C and 138 MPa (20 ksi)

Figure 31

Unstressed supplier B cladding material in the pickled condition is exposed to the iodine environment present in a tube pressurization test at 320°C. Features of interest are regions of general attack (A), local attack at particles (C) where the particles are finally removed from the surface (B), and pre-existing pits that exhibit no localized attack (D). Energy dispersive analysis of the particles reveals Zr, Cr, and Fe to be present.

Figure 31

Unstained samples of stained material in the plastic container exposed to the latter treatment were placed in a tube pressurized tank at 100°C. Figure 31 shows the results of general tests (A), local attack of particles (C) where the particles are clearly visible from the surface (B), and pre-existing and local attack in localized attack (B). Energy dispersive analysis of the particles reveals Fe, Cr, and Fe to be present.

DISTRIBUTION

No. of
Copies

No. of
Copies

OFFSITE

2 A. A. Churm
DOE Patent Division
9800 S. Cass Avenue
Argonne, IL 60439

6 Martin Langsam
DOE Contracting Officer
9800 S. Cass Avenue
Argonne, IL 60439

197 U.S. DOE Distribution
Category UC-78

D. L. Ziemann
(Docket No. 50-55,
License DPR-6,
Big Rock Point Plant)
U.S. Nuclear Regulatory
Commission
Division, Operating Reactors
Mail Stop 542
Washington, DC 20555

Dr. W. V. Johnston
Chief, Fuel Behavior
Research Branch
U.S. Nuclear Regulatory
Commission
Division Reactor Safety Research
Washington, DC 20555

Ralph Meyer
ATTN: M. Tokar
Reactor Fuels Section
Division of Systems Safety
U.S. Nuclear Regulatory
Commission
Washington, DC 20555

H. Sobel
American Electric Power Service
Corporation
2 Broadway
New York, NY 10004

Tony Mansell
Arkansas Power and Light Company
P. O. Box 551
Little Rock, AR 72203

J. S. Tulenko
Babcock and Wilcox Company
Nuclear Power Generation Division
P. O. Box 120
Lynchburg, VA 24505

W. A. Weinreich
Bettis Atomic Power Laboratory
Box 79
West Mifflin, PA 12512

B. A. Pasternak
Booz-Allen Applied Research
4330 East-West Highway
Bethesda, MD 20014

R. N. Duncan
Combustion Engineering, Inc.
1000 Prospect Hill Road
Windsor, CT 06095

W. M. Kiefer
Commonwealth Edison Company
P. O. Box 767
Chicago, IL 60690

D. R. D'Boyle
Commonwealth Edison Company
P. O. Box 767
Chicago, IL 60690

No. of
Copies

M. L. Lee
Consolidated Edison Company
of New York
4 Irving Place
New York, NY 10003

D. B. Wehmeyer
Detroit Edison Company
2000 Second Avenue
Detroit, MI 48226

J. Korthever
Duke Power Company
P. O. Box 2178
Charlotte, NC 28342

T. Snead
Duke Power Company
P. O. Box 2178
Charlotte, NC 28342

F. E. Gelhaus
Electric Power Research
Institute
P. O. Box 10412
Palo Alto, CA 94303

J. T. A. Roberts
Electric Power Research
Institute
P. O. Box 10412
Palo Alto, CA 94303

J. R. Tomonto
Florida Power and Light Company
P. O. Box 013100
Miami, FL 33101

S. Armijo
General Electric Company
175 Curtner Avenue
San Jose, CA 95125

H. W. Schadler
Manager, Metallurgy Laboratory
General Electric Company
P. O. Box 8
Schenectady, NY 12301

No. of
Copies

G. Bond
General Public Utilities
Service Corporation (GPU)
260 Cherry Hill Road
Parsippany, NJ 07054

D. J. Grotch
Knolls Atomic Power Laboratory
Box 1072
Schenectady, NY 12301

W. J. Tunney
Long Island Lighting Company
175 E. Old Country Road
Hicksville, NY 11801

S. W. Wilczek, Jr.
Niagara Mohawk Power Corporation
300 Erie Blvd. West
Syracuse, NY 13202

J. P. Cagnetta
Northeast Utilities Service
P. O. Box 270
Hartford, CT 06101

J. Hallam
Quadrex
1700 Dell Avenue
Campbell, CA 95008

T. Row
Oak Ridge National Laboratory
P. O. Box X
Oak Ridge, TN 37830

I. Spiewak
Oak Ridge National Laboratory
P. O. Box X
Oak Ridge, TN 37830

No. of
Copies

G. F. Deabeler
Philadelphia Electric Company
2301 Market Street
P. O. Box 8699
Philadelphia, PA 19101

K. Mahna
Public Service Electric and Gas
Company
80 Park Place
Newark, NJ 07101

R. R. O'Laughlin
Public Service Indiana
1000 East Main Street
Plainfield, IN 46168

W. R. Harris
Rand Corporation
1799 Main Street
Santa Monica, CA 90406

E. Straker
Science Applications, Inc.
8400 Westpark Drive
McLean, VA 22101

R. J. Mullin
Tennessee Valley Authority
1300 Commerce Union Bank Bldg.
Chattanooga, TN 37401

R. S. Miller
Westinghouse Electric Corp.
P. O. Box 355
Pittsburgh, PA 15230

R. M. Grube
Yankee Atomic Electric Company
20 Turnpike Road
Westboro, MA 01581

No. of
Copies

ONSITE

DOE Richland Operations Office

H. E. Ransom

Hanford Engineering Development
Laboratory

R. Omberg

Washington Public Power Supply
System

D. L. Larkin

24 Pacific Northwest Laboratory

W. J. Bailey
J. O. Barner
C. E. Beyer
L. R. Bunnell
M. E. Cunningham
M. D. Freshley (2)
R. J. Guenther
C. R. Hann
P. E. Hart
D. D. Lanning
C. L. Mohr
F. E. Panisko
R. E. Schreiber
M. S. Smith
R. E. Williford
R. D. Widrig
Publishing Coordination File (2)
Technical Information Files (5)

INTERNAL

8 Consumers Power Company

W. J. Beckius
D. P. Blanchard
F. W. Buckman
R. B. Dewitt
G. F. Pratt (3)
Library

No. of
Copies

34 Exxon Nuclear Company, Inc.

M. J. Ades
E. R. Astley
S. J. Beard
G. J. Busseman
M. H. Campbell
C. E. Crouthamel (10)
P. L. Farnsworth
J. W. Fredericks
W. L. Hampson
O. L. Kruger
A. J. Martenson
K. R. Merckx

No. of
Copies

J. N. Morgan
W. S. Nechodom
R. Nilson
J. F. Patterson
N. Kjaer-Pedersen
G. A. Sofer
L. Van Swam
R. K. Welty
H. E. Williamson
K. E. Willis
K. N. Woods
Records File
Library