In-Reactor Creep Behavior of Zircaloy-2

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Abstract - A BWR pre-irradiated re-crystallized (RXA) Zircaloy-2 tube was creep tested in the Halden Reactor in a dedicated creep rig connected to a light water supply and connected to a variable pressure gas supply allowing the magnitude and direction of the applied stress to be changed during the test. A three-point contact diameter gauge was used for making in-reactor outside diameter measurements on the sample. The sample was tested for 14,410 hours. A total of eight hoop stress changes were applied in series to the sample during different irradiation periods.

The data and two different evaluations of the data were previously reported. This paper updates the data and evaluation of the data taking into account new oxide thickness measurements and observations of the morphology of the oxide on the sample and on the diameter gauge calibration steps. The sample creep strains were re-calculated, correcting for the time dependent oxide thickness increases over the region of the sample that exhibited intact oxide. The oxide thickness correction decreased the irradiation creep strains. That is, for positive strains, the correction decreased the strain to lower values. In the case of negative strains, the correction resulted in larger negative numbers. The oxide thickness correction changed the creep rates. The oxide thickness did not affect the saturated transient strains. The transient irradiation creep component was dependent on the magnitude of the stress change. The re- occurrence was associated with a threshold stress change of about 41 MPa. The irradiation creep rates in tension are higher than those in compression. Comparison of the irradiation creep rates with other investigations suggests that the creep rate data are in transient irradiation creep and not in steady state irradiation creep. Alternatively, the differences may be due to differences between RXA Zircaloy-2 and RXA Zircaloy-4, which was used in the other investigations.

I. INTRODUCTION

The Halden Reactor Project, as part of its international Joint Programme, conducted an in-reactor study during 1992 – 1995 on the creep behaviour of a pre-irradiated Zircaloy-2 tube under conditions of stress reversal. The Zircaloy-2 tube sample was fitted with end-caps connected to a high-pressure gas supply and the test rod was housed in a flask connected to a light water coolant loop at 165 bar. By varying the internal gas pressure to the rod and maintaining system pressure, eight hoop stress changes of different magnitude and direction were applied to the sample over a total operating period of 14,410 full power hours. A 3-point contact, scanning diameter gauge was used to measure the outer diameter (OD) of the test rod as a function of axial position. Two different evaluations of the creep strain data generated from the test sample have been reported previously.1,2 One of the main differences in the two evaluations came from the approach that was considered appropriate when determining creep strain from the OD measurements with respect to
the effects of sample outer surface oxidation on the in-pile measurements.

Following these different evaluations of the data, the test rod was subjected to post irradiation examination (PIE) in order to ascertain the actual oxide thickness on its outer surfaces. There were two consequences from this examination: a re-analysis of the axial diameter-traces, generating a new set of values for specimen OD change ($\Delta D$) as a function of time, plus the production of a new set of specimen creep strain data determined using an oxide correction.

The purpose of this paper is to present the updated creep strain data for the Zircaloy-2 tube sample together with a re-evaluation of the corrected data in terms of the aims of the original study, i.e., assessing the in-reactor creep behaviour of Zircaloy-2 under variable loading conditions.

II. EXPERIMENTAL

Details of the test rod material, the in-pile test rig design and operating conditions have previously been reported, but the relevant details are given below.

The test rod was made from a 220 mm section of RXA Zircaloy-2 BWR water rod material, with nominal ID/OD of 14.85/15.0 mm. The chemical composition of the water rod was 1.55 wt% Sn, 0.35 wt% Fe+Cr+Ni with 0.11 wt% oxygen and the final manufacturing step was 70% cold work followed by a 3 hour 576°C anneal which resulted in a recrystallized microstructure with a grain size of 12 µm. Prior to manufacturing into a Halden test rod, the water rod was irradiated in a commercial reactor at 290°C to a neutron fluence of $6 \times 10^{21}$ n cm$^{-2}$ (>1 MeV), which was estimated to have resulted in an outer surface oxide layer of 10 µm and a hydrogen content of 60 ppm.

The test rod was placed in a pressure flask test rig (IFA-585) connected to a PWR loop in the Halden Reactor. Pre-heated light water was circulated through this loop at a pressure of 165 bar and the coolant temperature local to the Zircaloy-2 test rod was 300-323°C over the life of the experiment (given that the test rod was un-fuelled and gamma heating in the Halden Reactor is relatively low, the mid-wall temperature of the Zircaloy-2 tube was assumed to be the same as the local coolant temperature). Twelve high enrichment fuel rods surrounding the pressure flask produced a local neutron fast flux at the sample of $2.7 \times 10^{13}$ n cm$^{-2}$s$^{-1}$ (>1 MeV). The tube section was filled with Zircaloy pellets (no fissile fuel) with a 300 µm diametral gap to avoid pellet-clad interaction during creep testing and was sealed with end-plugs fabricated from un-irradiated Zircaloy-2. The upper end plug also contained a gas inlet/outlet for connection to a high-pressure gas system with on-line control for the purpose of varying the rod internal pressure.

The test rod was operated for a total of eight Halden Reactor cycles or 14,410 full power hours over which time nine stresses of different magnitude, direction and duration were applied to the sample by varying the rod internal gas pressure ($P_i$) while maintaining the system pressure ($P_o$). $P_i$ and $P_o$ were monitored throughout the experiment and used to calculate hoop stress in the test segment using the expression for a thick-walled tube. A 3-point contact, scanning diameter gauge was used to make approximately 200 in-pile measurements of the outer diameter of the test rod as a function of axial position at appropriate time intervals throughout the irradiation. Each axial diameter trace was analyzed by comparing it to a reference trace taken near the beginning of the experiment by a pattern recognition procedure. Figure 1 shows a visual example of such a comparison: first the trace calibration steps (machined into the test rod end-plugs) are aligned axially and diametrically, then the diametral difference between the two traces over the axial extent of the tube section is determined and a mean value of total diameter change, $\Delta D$, for the tube is thus obtained. This is converted from arbitrary units to µm with reference to the known magnitude of the calibration steps (measured precisely after machining). The advantage of using a calibration step as opposed to a single calibration diameter is that no matter what the dimensional changes in-pile, the step should remain the same size.

III. RESULTS

III.A. Oxide Correction

The oxide layer on the tube section of the test rod after the Halden in-pile creep testing was assumed to be compact, showing little axial
variation in thickness, and that the increase in oxide layer thickness during the test would be comparable to that having occurred on the end-caps of the test rod (i.e. the site of the diameter gauge calibration steps). This was the justification for the approach in [1] of making no oxide correction to the \( \Delta D \) values obtained from the diameter trace comparisons before converting them to creep strain. In order to ascertain the actual oxide thickness on the outer surfaces of the test rod after the Halden in-pile creep testing, the tube section and end-caps of the test rod were subjected to PIE in 2002.

Transverse cross-sections were cut from seven different axial locations on the test rod and prepared for optical metallography. Oxide thickness measurements were performed on each metallography sample at eight equally spaced circumferential locations. The average oxide thickness values for each axial location, determined from all the measurements taken at each of the eight circumferential locations, are presented in Figure 2. The oxide thicknesses determined for the two end-caps exhibit a small increase from the bottom to the top of the test rod. This behaviour can be attributed to the slight coolant temperature increase over the test rod - the test rod was oriented bottom-to-top in the test rig, and the coolant flow direction was also bottom-to-top, such that the gamma heating generated in the test rod (which contained dummy Zircaloy pellets and not fissile pellets) created a small axial temperature gradient in the coolant. In the case of the tube section of the test rod, the oxide thickness values exhibit an approximately linear decrease from the bottom to the top of the section. This axial variation in oxide thickness is opposite to that exhibited by the end-caps and cannot be explained by the coolant temperature gradient in the test rig. The oxide gradient was assumed to be due to a pre-existing axial gradient in oxide thickness attributed to the pre-Halden BWR base-irradiation. In addition, the thickest oxide layer near the bottom of the tube section exhibited cracking in all eight circumferential locations studied – see Figure 3. On the other hand, significantly less cracking was observed near the tube section axial mid-point and upper end. Only one out of eight circumferential locations exhibited oxide cracking at the axial mid-point and three out of eight at the top.

The first consequence of these new observations was a re-analysis of the axial diameter-traces. Since the diameter gauge traces will include the cumulative effect of any oxide crack opening widths on the measured test rod diameter, the regions of the diameter gauge traces where extensive oxide cracking had been observed were excluded. This applied to the lower end of the tube section (but not the middle or upper end) and the lower third of each trace was excluded in a re-run of the diameter trace comparison analysis using a revised pattern recognition procedure. This generated a slightly different, new set of values for specimen total diameter change (\( \Delta D \)) as a function of time.

The second consequence of these new observations was the decision to use an oxide correction approach in determining the specimen creep strain values from the \( \Delta D \) values. Any difference between the oxide thickness increase on the tube sample and on the end-caps (containing the calibration steps) must be subtracted from the \( \Delta D \) measurements in order to calculate the diameter irradiation creep strain. Based on the oxide thickness measurements made on both end-caps and at three axial locations on the tube sample, it could not be concluded that the same oxide layer growth had occurred during the test on the tube section as had occurred on the end-caps. One of the factors adding to this uncertainty is the fact that the thickness of the oxide layer measured on the tube section after the Halden irradiation (from 13.4 \( \mu \)m near the bottom of the tube to 6.2 \( \mu \)m near the top) could not be reconciled with the mean axial oxide thickness estimated to have been present before the Halden irradiation started, i.e., 10 \( \mu \)m after the BWR base-irradiation. A separate oxide thickness equation was therefore developed for the tube sample and the end-caps, respectively, as a function of irradiation time (or fluence).

The oxide thickness irradiation time dependence for both tube section and end-caps was assumed to be consistent with that of RXA Zircaloy-2 coupon corrosion data from a Halden test supplied by Peter Bennett. In support of this, the irradiation parameters for the coupon corrosion testing were similar to those for the creep rod testing in that the coupons were irradiated under through-wall isothermal conditions in coolant with a temperature range of
300-318°C while the creep tube sample was irradiated with a very small temperature gradient across the wall and at temperatures of 300-323°C.

The oxide thickness versus time behaviour for the RXAZircaloy-2 coupons, shown in Figure 4, exhibits three stages. The initial stage is a rapid primary phase oxidation rate that decreases to a steady state rate after about 4 μm. The steady state oxidation rate is followed by a post-transition accelerated oxide growth rate. These oxide thickness data were fitted to an equation of the form:

\[ \text{Oxide thickness} = E[1 - e^{-Ft}] + Gt + H(t - t_0) \]  

where \( E, F, G \) and \( H \) are material coefficients, \( t \) is the irradiation time and \( t_0 \) is the threshold time associated with the post-transition accelerated corrosion/oxidation. The first term is the pre-transition corrosion primary component. The second term is the pre-transition corrosion rate. The third term is the post-transition accelerated rate.

The measured end-cap oxide thickness values from the creep test rod are in good agreement with Equation 1. The calculated oxide thickness from the mean operating conditions is 4.8 μm at the end of testing compared to the measured values of 3.7 μm and 4.8 μm for the bottom and top end-caps, respectively. The data in Figure 4 suggest that the tube section would have oxidized at differing rates compared to the end-caps. The red lines in Figure 4 denote the Halden irradiation periods for the end-caps (oxidized from fresh) and the tube section (assuming a nominal starting oxide layer thickness of 3.5 μm). Initially, the end-caps do not have any oxide and exhibit a rapid increase in oxide thickness. On the other hand, the tube section would initially be in the steady state region of Figure 4, associated with a relatively low oxidation rate. The relative oxide growth rates of the end-caps and tube section reverse with increasing time. At the end of creep-testing, the oxidation rate of the end-caps would be decreasing to the relatively low steady state value while the oxidation rate of the tube sample would be increasing to the post-transition accelerated rate, which is much larger than the steady state rate. Using Equation 1, the calculated oxide thickness increase on the tube section from the duration of the creep testing and assuming a nominal starting oxide layer thickness of 3.5 μm is 4.7 μm for a total thickness of 8.2 μm. The average oxide thickness measured on the tube section at the end of creep testing, over the axial range of the diameter traces used for the new \( \Delta D \) determination, is 8.2 μm. The implication of this is that the mean oxide layer thickness present on the tube section prior to creep testing was 3.5 μm. This is not in good agreement with the 10 μm given as an axial mean estimate for the water rod section as-supplied. In order to deal with this apparent uncertainty in the value of the oxide thickness produced by the BWR base-irradiation, three cases were used to envelope the oxidation behaviour of the tube sample. The three cases are listed in Table 1. Each case takes the final oxide layer thickness on the tube section to be the actual mean value as measured by PIE. For the first case, no oxidation correction is applied to the \( \Delta D \) values obtained from the re-analysis of the diameter traces. For the nominal case, the oxide growth predicted by Equation 1 during creep testing is assumed. And for the third case, the oxide growth during creep testing is taken to be 35% greater than in the nominal case.

**III.B. Corrected Creep Strain Data**

Each of the measured creep sample diameter change values (\( \Delta D \)) were corrected for oxide thickness, according to the three cases as a function of irradiation time, consistent with the Pilling-Bedworth oxide to metal ratio. The creep strain results for one of the constant stress periods in the Halden creep test, period BWR3 (\( \sigma_0 = -217.7 \text{ MPa} \)), has been selected as an illustration of the effect of the corrections. Figures 5 to 7 present the creep strain results versus test time (in full power hours) for increasing oxide thickness correction. That is, the cases are in the order of no correction (Figure 5), nominal correction (Figure 6) and +35% of the nominal correction (Figure 7).

The oxide thickness correction decreases the irradiation creep strain values. That is, for positive strains, the correction decreased the strain to lower values. In the case of negative strains, the correction resulted in larger negative numbers. The effect of the oxide thickness correction may be evaluated by comparing the diametral creep strains at 5000 fph. In the case of no oxide correction, \( \Delta D/D_0 \) is about 0.016%.
The strain decreased to about 0.004% with the nominal oxide correction and with the maximum correction, the strain decreased further to about 0.005%.

A second effect of the oxide thickness correction was to change the irradiation creep rates. In Figures 5 and 6, the solid lines denote the data used to calculate the creep rate. The solid line was calculated by linear regression. The dashed lines denote the data in transient creep. The creep data in Figure 7 are shown fitted to an equation of the form:

\[ \frac{\Delta D}{D_o} = A[1 - e^{-Bt}] + Ct \]  

(2)

where the first term is the transient component and the second term is the creep rate component. The material coefficients A, B and C are the saturated transient creep strain, the transient decay coefficient and the creep rate, respectively. Such an analysis was carried out for each of the five constant hoop stress periods of the creep test and Figure 8 presents the effect of the oxide thickness correction for each of the five constant tensile hoop stress periods. The ratio of the oxide thickness corrected irradiation creep rate (i.e. from cases 2 and 3) to the uncorrected rate (i.e. from case 1) is plotted versus the mid time point in the given constant hoop stress period. Early in the creep test, at short times, the oxide thickness corrected irradiation creep rates are larger than the uncorrected values. The oxide thickness correction increased the initial creep rates by about 4-5%. With increasing time, the oxide thickness corrected creep rates are smaller than the uncorrected rates. The oxide thickness correction decreased the creep rates by a maximum of about 9-14%.

The oxide thickness correction did not affect the transient strain component. Table 2 lists the saturated transient component values (i.e. the A coefficient in Equation 2) for the case of no oxide correction, nominal oxide correction and +35% of nominal oxide correction. The results show that the saturated transient component is unchanged by the oxide corrections. Table 2 also lists the duration of the transient component. Note that the transient component duration is directly related to the magnitude of the saturated transient strain (the A coefficient in Equation 2) and the hoop stress change.

IV. DISCUSSION OF CREEP STRAIN DATA

Re-occurrence of the irradiation creep transient depends on the magnitude of the stress change. Figures 9 to 11 show the irradiation creep strain versus time behavior with the nominal oxide correction for samples with positive hoop stress changes. Figures 9 to 11 are presented in the order of increasing hoop stress change. Figure 9 shows that no transient component is associated with \( \Delta \sigma_0 = 40.7 \) MPa. Figure 10 shows when \( \Delta \sigma_0 \) is increased to 51.3 MPa, that a small transient component is observed. Figure 11 shows when \( \Delta \sigma_0 \) is increased to 287.0 MPa, that a large transient component is observed. Figure 12 presents the saturated transient component data for all of the stress periods. The results show for positive \( \Delta \sigma_0 \) values, that the transient component exhibits a \( \Delta \sigma_0 \) threshold of about 41 MPa (based on linear regression), and that the magnitude increases approximately linearly with increasing \( \Delta \sigma_0 \). In the case of negative \( \Delta \sigma_0 \) values, the data are not sufficient to confirm either a threshold or a linear dependence on \( \Delta \sigma_0 \). The trend lines in Figure 12 were plotted assuming a threshold stress of -41 MPa and a linear dependence of the saturated transient strain similar to that exhibited for positive \( \Delta \sigma_0 \) values. Confirmation of this behavior for negative \( \Delta \sigma_0 \) values is necessary.

Evaluation of the irradiation creep rates in tension and compression suggest that the tension creep rates are higher than those in compression. However, comparison of the irradiation creep rates with other investigations suggests that the creep rate data are not in steady state creep. Figure 13 presents the calculated creep rates using the nominal oxide thickness correction. In addition, Figure 13 presents the steady state creep rates for RXA Zircaloy-4 based on data reported by Baty et al.,4 and Soniak et al.5 Note that the creep rates are higher than the values associated with Baty et al., and Soniak et al. In the case of compression stresses, the creep rates, although larger, are in reasonable agreement with Baty et al., given the test-to-test scatter. On the other hand, in the case of tension stresses, the creep rates are significantly higher than the value associated with Soniak et al. This increase may be due to the low fluences associated with the present study. According to Soniak et al., the transition from transient to steady state...
irradiation creep occurs at a fluence level of about $3.0 \times 10^{21}$ n/cm$^2$ E>1 MeV. The fluence increases under stress in this study (during the fixed stress periods) are in the range of 0.10 to $0.28 \times 10^{21}$ n/cm$^2$ E>1 MeV, which are factors of 10 to 30 less than the transition from transient to steady state creep reported by Soniak et al. Hence, the creep rate data associated with this study most likely are in transient irradiation creep and not steady state irradiation creep. On the other hand, Baty et al., and Soniak et al., used RXA Zircaloy-4 whereas this study used RXA Zircaloy-2. An alternate explanation of the higher creep rates of this study relative to Baty et al., and Soniak et al., is that the creep differences are due to the material differences between Zircaloy-4 and Zircaloy-2.

The differences in temperature between this study and Soniak et al., will not explain the observed differences of the creep rates in tension. In fact, the temperature differences of the tests suggest that higher creep rates would be expected at the test conditions used by Soniak et al. Soniak et al., performed the creep measurements at 350°C, which is significantly higher than the temperature range of 300-323°C of the present study. According to Soniak et al., RXA Zircaloy-4 has an activation energy between 4184 and 7173 K. Based on an activation energy of 5679 K (the average value of 4184 and 7173 K), the increase in temperature from 310 to 350°C is associated with an increase of 1.9. This suggests that the creep rate at the test conditions used by Soniak et al., should be a factor of 1.9 higher than those of the present study.

V. CONCLUSIONS

The following conclusions may be drawn based on the above results and discussion:

1. An oxide thickness correction was made to the measured change in diameter ($\Delta D$) data. The oxide thickness correction decreased the irradiation creep strains. That is, for positive strains, the correction decreased the strain to lower values. In the case of negative strains, the correction resulted in larger negative numbers. The oxide thickness correction and changed the creep rates. The oxide thickness correction did not affect the saturated transient component strains.

2. The re-occurrence of the irradiation creep transient depends on the magnitude of the stress change. The re-occurrence of the transient is associated with a threshold stress change of about 41 MPa.

3. The irradiation creep rates in tension are higher than those in compression. Comparison of the irradiation creep rates with other investigations suggests that the creep rate data are in transient creep and not in steady state creep. Alternatively, the differences may be due to differences between RXA Zircaloy-2 and RXA Zircaloy-4 material, which was used by the other investigators.

ACKNOWLEDGEMENTS

The authors would like to acknowledge B. C. Oberländer, H. K. Jenssen and M. Espeland at IFE-Kjeller hot labs for the PIE investigation of the test sample after the Halden creep testing.

REFERENCES


Table 1. Oxide thickness cases.

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<tr>
<th>Oxide Thickness Case</th>
<th>Total Oxide Thickness (μm)</th>
<th>Halden Oxide Thickness (μm)</th>
<th>BWR Oxide Thickness (μm)</th>
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<tr>
<td>No correction</td>
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<td>0.0</td>
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<tr>
<td>Nominal</td>
<td>8.2</td>
<td>4.7</td>
<td>3.5</td>
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<td>+35%</td>
<td>8.2</td>
<td>6.3</td>
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Table 2. Saturated transient irradiation creep component strains.

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<th>Period</th>
<th>Hoop Stress (MPa)</th>
<th>Hoop Stress Change (MPa)</th>
<th>A (%)</th>
<th>A (%)</th>
<th>A (%)</th>
<th>Transient Duration (h)</th>
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<tr>
<td></td>
<td>Oxide Correction: None</td>
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<td>2</td>
<td>58.3</td>
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<td>0.0144</td>
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<tr>
<td>3</td>
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<td>-217.7</td>
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<td>4</td>
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<tr>
<td>5</td>
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<td>-0.0265</td>
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<tr>
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<tr>
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Figure 1. Visual example of the diameter trace comparison method used to determine total change in creep sample diameter (ΔD) accumulated in the test time interval 1602 to 5162 fph.
Figure 2. Creep sample and end-cap oxide thickness at the end-of-testing.
Figure 3. Micrograph from the transverse cross-section cut from a lower axial position of the tube creep sample showing typical oxide morphology with cracking.
Figure 4. RXA Zr-2 corrosion in Halden.
Figure 5. Diameter strain data versus time for irradiation period BWR3 uncorrected for oxide corrosion.
Figure 6. Diameter strain data versus time for irradiation period BWR3 corrected for nominal oxide corrosion.
IFA-585 Period BWR3 with +35% Oxide Correction
-159.4 MPa Hoop Stress, -217.7 MPa Hop Stress Change

Figure 7. Diameter strain data versus time for irradiation period BWR3 corrected for 35% of nominal oxide corrosion.
Figure 8. Effect of the oxide correction on the irradiation creep rates for tension hoop stresses.
Figure 9. Diameter strain versus time for irradiation period BWR6 with the nominal oxide correction showing no transient irradiation creep for a 40.7 MPa hoop stress change.
IFA-585 Period BWR7 with Nominal Oxide Correction
27.6 Mpa Hoop Stress, 51.3 Mpa Hoop Stress Change

Figure 10. Diameter strain versus time for irradiation period BWR7 with the nominal oxide correction showing a moderate transient irradiation creep component for a 51.3 MPa hoop stress change.
Figure 11. Diameter strain versus time for irradiation period BWR4 with the nominal oxide correction showing a large transient irradiation creep component for a 287.0 MPa hoop stress change.
Figure 12. Saturated diameter strain irradiation creep versus hoop stress for the nominal oxide correction.
RXA Zr-2 and RXA Zr-4 Steady State Irradiation Creep Rates
(IFA-585 data has the nominal oxide correction)

Figure 13. Diameter irradiation creep rates.