Post-Quench Ductility Results for Zry-4 and M5
Oxidized at 1200˚C, Slow Cooled to 800˚C and Quenched

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March 23, 2004

Summary

Ring compression tests have been completed for Zry-4 and M5 samples oxidized to nominal ECR values of 5, 10, 15, 17 and 20% at 1200˚C. The 25-mm-long samples were exposed individually to two-sided steam oxidation in the same test apparatus for about the same test times, slow cooled to 800˚C and water-quenched. Based on sample weight increase (normalized to the surface area), weight gain was determined and compared to CP predictions. Very good agreement was achieved among both alloys and the predictions for the 1200˚C samples. For the highest oxidation time (≈400 s), the M5 and Zry-4 weight gain values were within 5% of the CP-predicted values. These measured weight gains, along with the cladding thickness (0.57 mm for Zry-4 and 0.61 mm for M5), were used to determine measured ECR.

Ring-compression samples (8-mm-long) were cut from the oxidized samples and tested in a Model 5566 Instron at room temperature and 2 mm/min. cross-head displacement rate. This rate corresponds to a diametral strain rate of 0.35%/s for the 9.50-mm-OD cladding. Load-displacement curves were analyzed by the offset-displacement method to determine plastic ductility. This method assumes that the unloading stiffness is the same as the loading stiffness. The results of both loading-unloading-reloading tests and post-test diameter measurements confirm that the stiffness decreases with deformation. Thus, the standard offset method results in an over-prediction of permanent displacement and strain. For non-oxidized and 5.5%-oxidized Zry-4, the difference between offset and measured permanent displacement is ≤0.2 mm (≤2% nominal hoop strain). Based on the criterion that >2% offset strain implies ductile behavior, Zry-4 and M5 would be classified as ductile at ≤8% and <13% ECR, respectively. At ≥10% ECR, the offset strain decreases from 1.0% to 0.5% for Zry-4 and 3.4% to 1.4% for M5. The permanent strains measured after the test decrease from 0.5% to 0.4% for Zry-4 and from 1.6% to 0.6% for M5. With such low ductility values, it is a matter of interpretation regarding the classification of "ductile" or "brittle" for Zry-4 at ≥11% ECR and M5 at ≥13% ECR

The hydrogen concentration (<20 wppm) and pickup (<14 wppm) values are very low for both alloys oxidized at 1200˚C. Thus, ductility decrease is due solely to the oxygen pickup and distribution of oxygen concentration in the prior-beta layer. Continuous networks of alpha incursions (oxygen-stabilized alpha) across the thickness of the prior-beta layer are particularly embrittling. Metallography and microhardness have been completed for samples oxidized to 13% ECR (17% Baker-Just) and 20% ECR. Although not conclusive, the results suggest brittle behavior of Zry-4 and M5 at ≥13% ECR. Ring-compression tests will be repeated at 135˚C and 3.5%/s to determine the ECR embrittlement threshold at post-LOCA relevant temperatures. Samples for these tests will be prepared by oxidation at 1200˚C to 10-17% ECR.

1. Introduction

The purpose of this program is to determine the post-quench ductility of advanced cladding alloys M5 and ZIRLO, as compared to the post-quench ductility of Zry-4. While extensive literature data are available for traditional Zircaloy claddings (Zry-4 and Zry-2), relatively little data have been published for M5 and ZIRLO. Also, the published data for advanced alloys were generated in different laboratories by very different methods. In this program, all samples are oxidized in the same apparatus at the same ramp rates, hold times, and cooling rates (slow-cooled to 800˚C and water-quenched). The 25-mm-long samples are exposed to two-sided steam oxidation prior to cooling. Also, the samples are compressed in the same Instron machine, and the load-displacement data are analyzed by a common method to determine ductility.

The Zry-4 and ZIRLO tubing (17×17 PWR) provided by Westinghouse has an outer diameter of 9.50 mm and a wall thickness of 0.57 mm. The M5 tubing (17×17 PWR) provided by Framatome has an outer diameter of 9.50 mm and a wall thickness of 0.61 mm. Table 1 summarizes the test matrix for oxidizing the samples prior to ring-compression, post-quench ductility testing. The times listed are the equivalent isothermal times at the test temperature to give Cathcart-Pawel (CP) calculated ECR values of 5, 10, 15, 17, and 20%, for an assumed wall thickness of 0.57 mm. Actual ECR values vary depending on the weight gain for each sample and the pre-oxidized thickness of the sample.

Following oxidation and quench, 8-mm rings are cut from near the middle of the 25-mm-long samples. Ring compression tests are performed at room temperature at a displacement rate of 2 mm/min. The load-displacement curves are analyzed by the traditional offset-displacement method used in analyzing tensile-test data. The offset displacement, which is a measure of permanent displacement, is normalized to the outer diameter (9.50 mm) to give a nominal plastic hoop strain. Samples that exhibit offset strains >2% are considered to be ductile. However, for samples with ≤2% offset strain, another method is used to better determine permanent deformation and ductility. For this second method, the sample is unloaded after the first significant load drop indicating through-wall failure along the length of the sample. The post-test diameters along and normal to the loading direction are measured directly and compared to the pre-test diameter to give a direct measure of permanent strain. For these low-offset-strain samples, the permanent diameter change in the loading direction is used as a direct measure of ductility.

2. Steam Oxidation and Quench

Table 2 lists the measured weight gain and ECR values vs. predicted ECR values for the M5 and Zry-4 samples oxidized at 1200˚C. Figure 1 shows the weight gain results for M5 and Zry-4 vs. the Cathcart-Pawel (CP) predicted values. For the longest test times, the measured values are only <1% (M5) and 5% (Zry-4) higher than the CP-predicted values. These variations are within experimental uncertainties.

Table 1 Test Matrix for Oxidation of Samples for Post-Quench Ductility Tests. The times and ECR values listed correspond to those calculated using the Cathcart-Pawel weight gain correlation, a nominal wall thickness of 0.57 mm, and two-sided isothermal oxidation in steam. The relationship between ECR (%) and normalized weight gain (Δw in mg/cm^2) is ECR = 1.538 Δw for 0.57-mm-thick cladding samples.

Temperature ˚C	**ECR %**	**Equivalent Oxidation Time s**
1000	5	210
	10	841
	15	1892
	17	2430
	20	3364
1100	5	67
	10	266
	15	599
	17	769
	20	1065
1200	5	25
	10	99
	15	222
	17	285
	20	394
1260	5	14
	10	58
	15	130
	17	167
	20	231

Table 2 Weight Gain (Δw in mg/cm^2) and Measured ECR (%) Values for Zry-4 and M5 Oxidized in Steam at 1200°C. ECR = 1.538 Δw for Zry-4 (0.57-mm wall) and ECR = 1.437 Δw for M5 (0.61-mm wall). Alloys were oxidized for the same ramp rate, approximate hold time and cooldown rate for each target ECR value.

Material	Cathcart-Pawel ECR, %	Measured Weight Gain (Δw), mg/cm^2	Measured ECR, %
Zry-4 M5	4.9 4.6	3.6 2.8	5.5 4.0
Zry-4[a]	7.8	5.30	8.2
Zry-4 M5	10.0 9.1	7.1 6.3	10.9 9.1
Zry-4[b] M5[b]	13.0 12.2	8.35 9.11	12.8 13.1
Zry-4 M5	15.7 14.1	11.1 9.6	17.1 13.8
Zry-4 M5	16.8 16.0	11.7 10.9	18.0 15.7
Zry-4 M5	20.1 18.7	13.5 13.1	20.8 18.8

[a]Additional test conducted to better determine ductility decrease in the range of 5 to 10% ECR.

[b]Additional tests run to determine post-quench ductility at 13% CP-predicted ECR and 17% Baker-Just-predicted ECR.

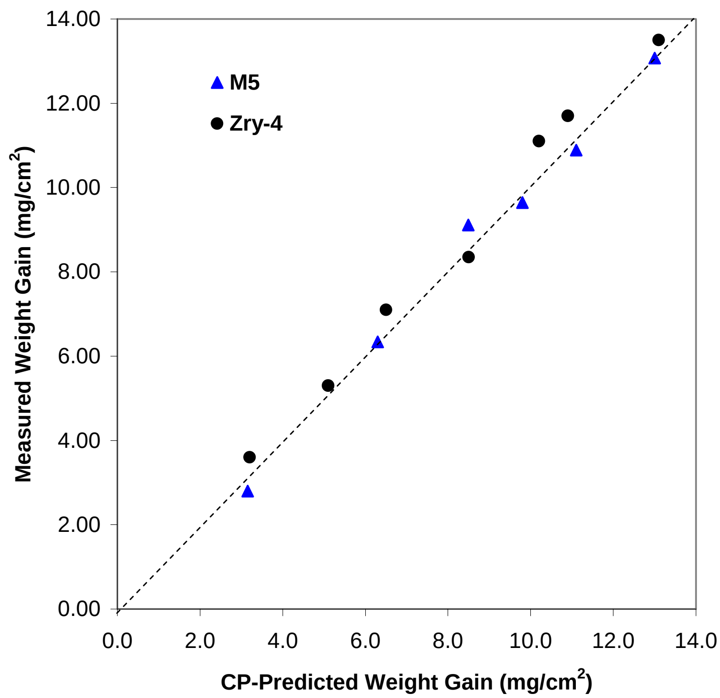


Fig. 1. Comparison between weight gain data for M5 and Zry-4 and weight gain predicted by the Cathcart-Pawel (CP) correlation for samples oxidized (two-sided) in steam at 1200˚C.

3. Ring Compression Tests

3.1 Validation of the Offset Strain Method

The methodology of using offset strain to determine permanent plastic strain works very well in uniaxial tensile tests. In these tests, at least in the uniform deformation regime, the loading remains uniaxial, the sample retains its initial shape, and the effective stiffness for loading and unloading remains reasonably constant. For the ring-compression tests, the sample geometry changes (circular-to-oval ring with some flattening at load-support locations) as the material deforms, the loading becomes more distributed at the load-support locations, and residual stresses are present during unloading. Based on test results at 1000˚C and 1100˚C [1], it was found that the offset strain tends to be larger than the permanent displacement determined from post-test measurements of diameter change along, and normal to, the loading direction. This would certainly be the case if the effective stiffness decreased with deformation.

Several loading-unloading-reloading tests were conducted to confirm that the effective stiffness ($K = F/\delta_e$, where F is the force measured by the load cell and δ_e is the cross-head displacement in the linear deformation range) decreases with deformation. For non-oxidized, as received Zry-4 tubing, the effective stiffness decreased from 0.87 kN/mm to 0.81 kN/mm to 0.75 kN/mm for sequential loading displacements of 1.1 mm, 1.3 mm, and 1.6 mm. At the end of the 3rd loading cycle the offset strain based on the initial stiffness was 0.94 mm, as compared to 0.74 mm determined from the decreased stiffness. Thus, depending on the maximum load and the extent of displacement, the standard offset method may give permanent strain values that are high by ≤2% (≤0.2 mm displacement). The loading-unloading test was conducted again using a Zry-4 sample oxidized at 1200˚C to 5.5% ECR (see Fig. A.1b in Appendix A). The effective stiffness of the 5.1-mm-long sample, normalized to the standard 8-mm length, decreased from 0.87 kN/mm to 0.60 kN/mm to 0.60 kN/mm to 0.47 kN/mm after sequential loading-cycle displacements of 1.3 mm, 2.4 mm and 1.3 mm, and 1.5 mm. For each loading cycle, the decreasing stiffness resulted in offset strains ≈2% lower than determined by mathematically unloading the sample at the initial stiffness value. The results of both tests are particular to flat-plate loading of samples with 9.50-mm outer diameter and 0.57-mm wall thickness.

These test results, along with previous comparisons between the standard offset strains and post-test measurements of diameter change, demonstrate that samples exhibiting standard offset strains of ≤2% should be analyzed by a more accurate method. The procedure adopted in this study is to stop the test after the first significant load drop – indicating through wall failure – and measure the diameter decrease along the loading direction and the diameter increase 90˚ from the loading direction. This works well for the ring-compression samples because the unloaded sample has a very tight through-wall crack – so tight that magnification must be used to view it. Thus, the more accurate measure of ductility at low strains is based on diameter decrease in the loading direction normalized to the outer diameter of the pre-oxidized cladding.

3.2 Post-Quench, Ring-Compression Data

From the samples listed in Table 2, 8-mm-long rings were cut from the central region for ring-compression testing. These tests were conducted at room temperature and a cross-head displacement rate of 2 mm/min. The load-displacement curves for these tests are given in Appendix A for Zry-4 and in Appendix C for M5. These curves were analyzed by the standard offset-displacement method described in 3.1. Analyses of the load-displacement curves in Appendices A and C and post-test physical examination of the samples were used to determine the offset and permanent displacement values, respectively, prior to through-wall failure. These values are listed in Table 3 for samples oxidized at 1200˚C. Graphical representation of these results is shown in Fig. 2. Figure 2a shows offset strain vs. measured ECR, while Fig. 2b shows measured permanent strain vs. measured ECR. The permanent strain values were derived from diameter decrease measured after the test along the loading direction.

Based on the criterion that standard offset strains > 2% imply ductility, Zry-4 is ductile for ECR < ≈10% and M5 is ductile for ECR < 12%. For ECR > 10%, measured permanent strains are quite low for Zry-4 (0.4-0.7%) and M5 (0.6-0.9%).

3.3 Post-Test Characterization

Based on the permanent-displacement data presented in Table 3, it is not clear if the >10%-ECR samples should be classified as ductile because the measured permanent strains are very low (<1%). Also, the measurements were performed on samples with a single, tight through-wall crack. Given that the ring compression test is really a structural test, it is important to characterize the material to verify if it should behave in a ductile or brittle manner in such a test. From this perspective, a ductile material should have a relatively low hydrogen content (<≈100 wppm), a sufficient amount (>0.2 mm) of low-oxygen-containing metal (prior-beta), and a room-temperature microhardness of <350 DHP for this prior-beta layer.

20% ECR Samples

Table 4 summarizes the results of the post-test characterization for Zry-4 and M5 samples at the highest ECR values. The hydrogen concentration and pickup values are too low (<20 wppm) to cause embrittlement. Metallography of etched Zry-4 and M5 samples oxidized to the highest ECR values is shown in Fig. 3. Based on Table 4 and Fig. 3, it is clear that the ratio of total oxide layer thickness to weight gain is constant for these two alloys. Figures 3a (Zry-4) and 3b (M5) show oxygen-stabilized alpha incursions locally extending through the entire prior-beta-layer thickness. These likely formed during slow cooling from 1200˚C to 800˚C. Because of the higher solubility (≈0.6 wppm) of oxygen in the beta layer at 1200˚C, more oxygen is available for precipitation of alpha incursions during cooling. Microhardness values for Zry-4 >350 DPH across the prior-beta layer would suggest that the material is essentially brittle [2, 3]. If the continuous alpha-incursion network aligns either with the maximum bending stress locations, the material would behave in a brittle manner in the ring-compression test. Other alignments may result in small values of plastic deformation prior to through-wall cracking. Layer-thickness and microhardness results are listed in Table 4 for the oxide, oxygen-stabilized-alpha and prior-beta layers. Figures 4 and 5 show examples for Zry-4 and M5, respectively, of photographs with microhardness indents.

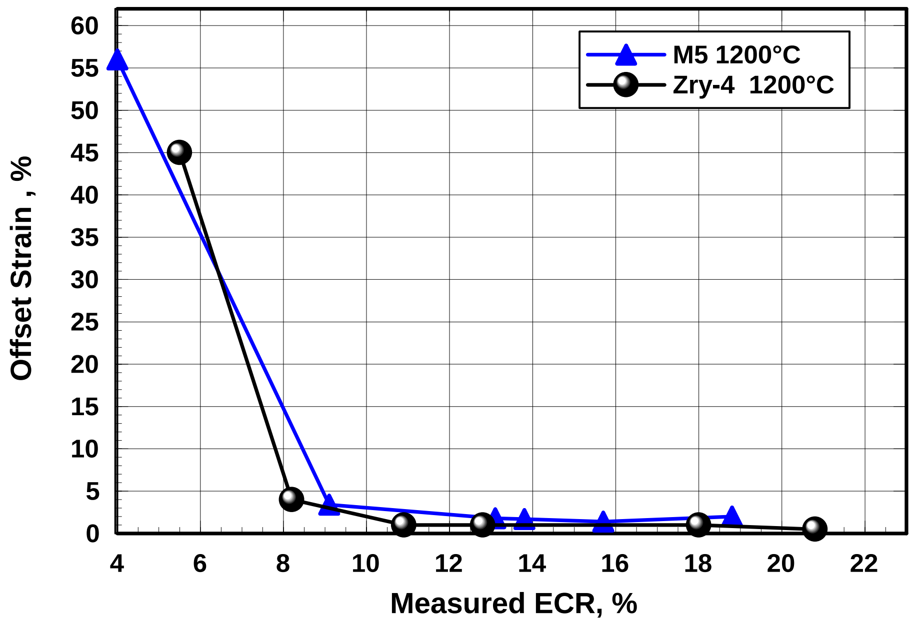
Table 3 Ring Compression Test Results for Samples Oxidized at 1200˚C and Quenched (see Table 2). Tests were performed with a Model 5566 Instron on 8-mm-long samples at room temperature and at 2 mm/min. cross-head displacement. For tests that were stopped after the first significant load drop, diameters were measured directly after the test along the loading direction and perpendicular to the loading direction. The decrease in diameter along the loading direction, normalized to 9.50 mm, is shown in the last column ("Post-Test Hoop Strain").

Material	ECR, %		Displacement, mm		Offset Hoop Strain %	Post-Test Hoop Strain %
	CP	Meas.	Total	Offset		
Zry-4 Zry-4 M5	4.9 4.9 4.5	5.5 5.5 4.0	>3.76 --- >5.5	>3.5 4.28 >5.35	>37 45[a] >56	>34 --- >53
Zry-4[b]	7.8	8.2	0.85	0.384	4.0	2.2
Zry-4 M5	10.0 9.1	10.9 9.1	0.44 0.79	0.097 0.324	1.0 3.4	0.5 1.6
Zry-4[c] M5[c]	13.0 12.1	12.8 13.1	0.44 0.48	0.090 0.170	1.0 1.8	0.7 0.9
Zry-4 M5	15.7 14.1	17.1 13.8	--- 0.46	0.04-0.12 0.160	≈0.8 1.7	--- 0.7
Zry-4 M5	16.8 16.0	18.0 15.7	0.35 0.38	0.097 0.130	1.0 1.4	0.6 0.6
Zry-4 M5	20.1 18.7	20.8 18.8	0.27 0.42	0.049 0.195	0.5 2.0	0.4 0.6

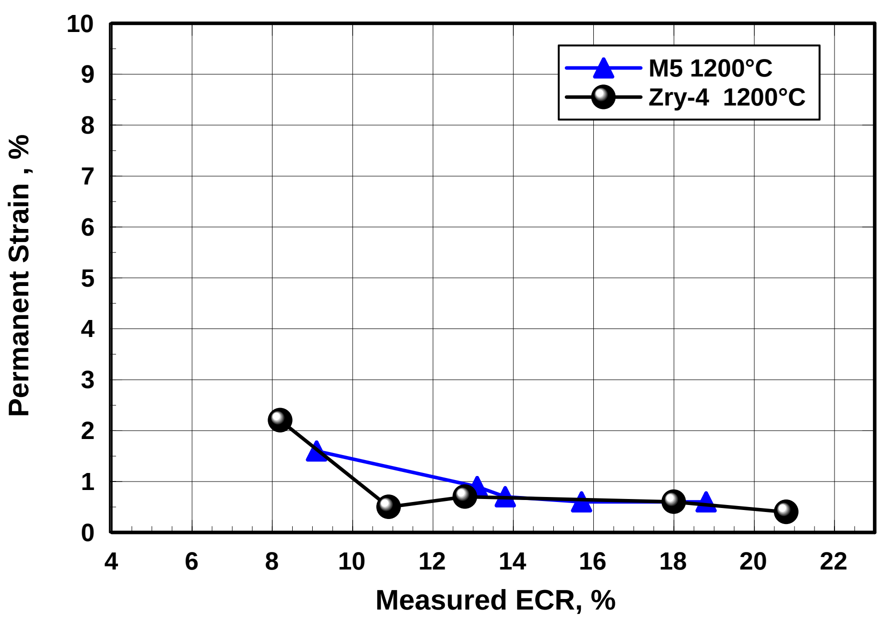
[a]Based on decreasing stiffness from 4-cycle loading-unloading test.

[b]Additional test to determine ductility decrease of Zry-4 in the range of 5-10% ECR

[c]Additional tests run to determine post-quench ductility at 13% CP-predicted ECR and 17% Baker-Just-predicted ECR.



(a)



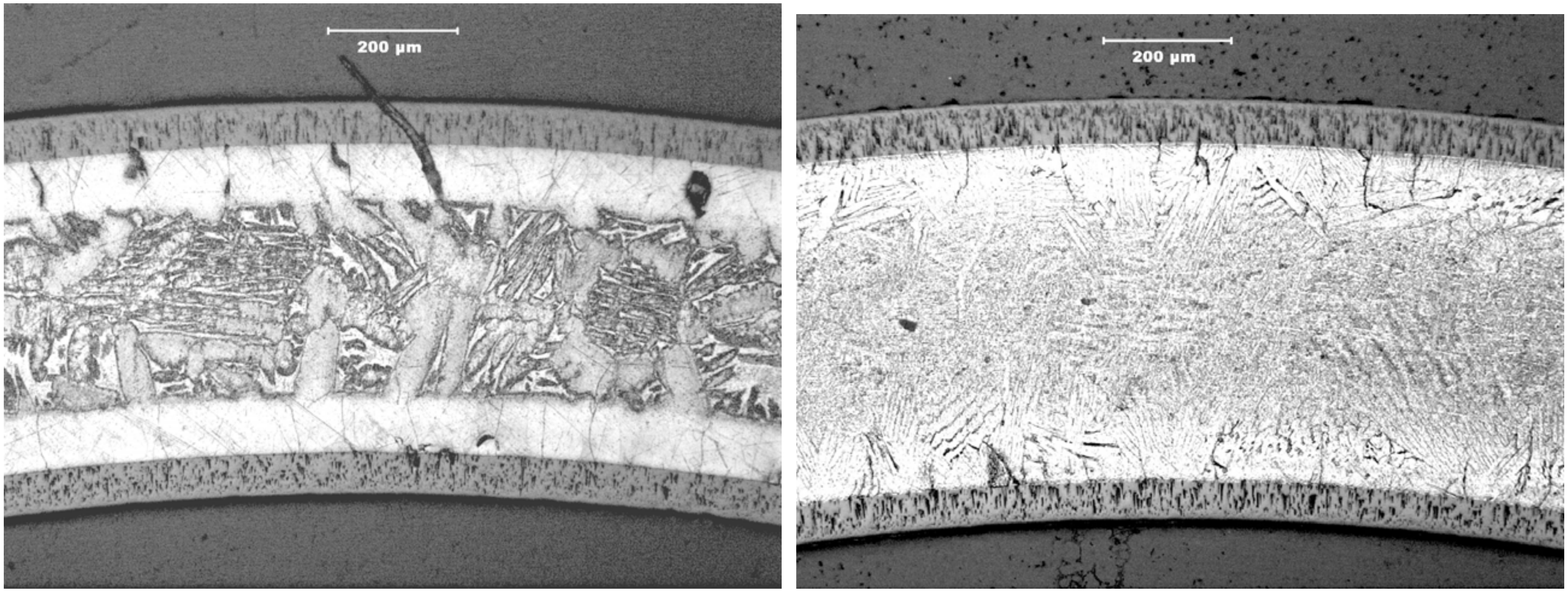
(b)

Fig. 2. Post-quench ductility vs. measured ECR for M5 and Zry-4 oxidized in steam at 1200˚C, quenched and ring-compressed at room temperature and a cross-head displacement rate of 2 mm/min. In Fig. 2a, ductility is based on the offset strains determined from ring-compression-test load-displacement curves. In Fig. 2b, ductility is based on the directly measured change in diameter along the loading direction for each sample.

Table 4 Summary of Characterization of Zry-4 and M5 Samples after Exposure to Steam at 1200˚C to 13% and 20% CP-ECR, Slow-cooling to 800˚C and Water Quench

Parameter	Zry-4		M5	
	20% ECR	13% ECR	20% ECR	13% ECR
Effective Time, s	400	166	400	166
Weight Gain, mg/cm^2	13.5	8.35	13.1	9.11
Measured ECR, %	20.8	12.8	18.8	13.1
Offset Displacement, mm	0.049	0.090	0.195	0.114
Measured Permanent Displacement, mm	0.04	0.07	0.06	0.09
Ductility, %	≤0.4	≤0.7	≤0.6	≤0.9
H Content, wppm	17	low	19	low
Hydrogen Pickup, wppm	13	low	14	low
OD/ID Oxide Thickness, µm	68/66	42/41	68/61	46/40
Prior-Beta-Layer Thickness, µm	266	419	≤360	≈442
Microhardness, DPH Oxide Layers Alpha Layers Prior-Beta Layer*	 570-960 530-730 280-600	 600-770 600-700 260-360	 650-780 580-850 280-450	 580-680 680-880 300-410

*Range includes microhardness values of oxygen-rich alpha incursions in this layer



(a) Zry-4 (b) M5

Fig. 3. Metallography of etched Zry-4 (a) and M5 (b) oxidized in steam at 1200˚C for ≈400 s and, slow cooled to 800˚C and water quenched. Measured ECR values are 20.8% for Zry-4 and 18.8% for M5.

Fig. 4. Microhardness indents across the radius of Zry-4 oxidized at 1200˚C to 20.8% ECR.

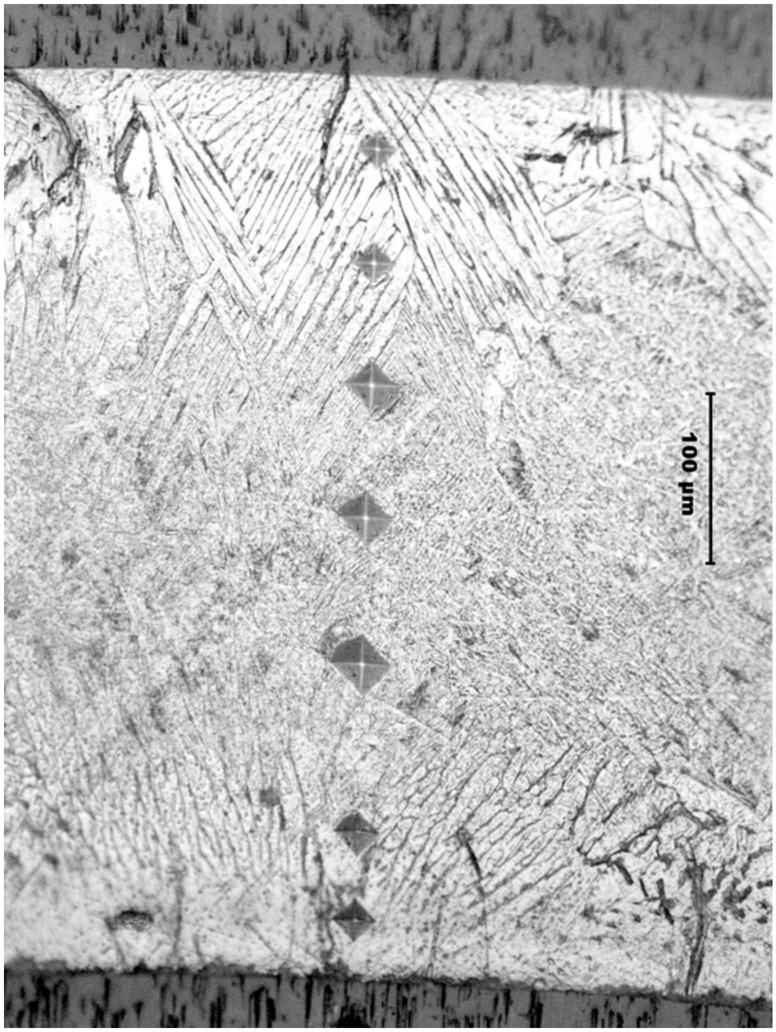


Fig. 5. Microhardness indents across the radius of M5 oxidized at 1200˚C to 18.8% ECR.

13% ECR Samples

Also listed in Table 4 are the results of the metallographic, microhardness and hydrogen analyses of the 13%-ECR samples, along with the weight gain and ductility results. Although the prior-beta layer is considerably thicker for Zry-4 (419 μm vs. 266 μm) and M5 (≈ 442 μm vs. ≤360 μm) at 13% ECR vs. 20% ECR and the average prior-beta-region microhardness is smaller for both Zry-4 and M5, the post-quench ductility results are essentially the sample (≤0.9% diametral strain). Examples of the microstructure and microhardness indents for 13%-ECR Zry-4 and M5 are shown, respectively in Figs. 6 and 7. In comparing these to Figs. 4 and 5, one would expect more ductile behavior from the 13% ECR samples. However, "more ductile" may not translate into sufficient ductility to cause the ring sample to behave in a ductile manner in the ring-compression test. At 1200˚C, it is certainly possible the both materials are brittle in the 13-20% ECR range.

4. Discussion

The ring-compression test proved to be useful for ductility screening of Zry-4 and M5 oxidized at 1000˚C and 1100˚C [1], as well as E110 oxidized at 1000˚C [4]. The post-quench ductility of Zry-4 and M5 decreases with increasing oxygen pickup and ECR and levels off at 2-5% offset strain in the 15-20% CP-ECR range. For E110, there is the expected precipitous drop in post-quench ductility with calculated ECR and measured hydrogen pickup, such that the offset strain essentially drops to zero at ≥7% ECR. However, the ring-compression results for Zry-4 and M5, oxidized at 1200˚C, are more difficult to interpret because the offset strain decrease abruptly with ECR to ≤2% strain and measured permanent strains level off at low values of 0.4-0.9% at >10% ECR. It is difficult to interpret these low permanent strain values in terms of the "ductile" vs. "brittle" classifications.

Materials parameters that should influence ductility were characterized at 13% and 20% ECR. These parameters include hydrogen pickup, prior-beta layer thickness, and microhardness. Post-quench ductility is governed by the thickness of the prior-beta layer, the distribution of oxygen within this layer (e.g., higher concentrations within alpha incursions), the range of microhardness values within this layer, and the hydrogen content within local regions of prior beta. In order to correlate ring-compression results with material parameters, comparisons can be made within one alloy type (e.g., vs. ECR) and between alloys (e.g., Zry-4 and M5 at the same ECR). For both alloys, the hydrogen pickup is too low to be of concern. For Zry-4, the oxygen solubility limit in the beta phase at 1200˚C is ≈0.6 wt.%. Although the oxygen concentration within the beta phase at the alpha/beta interface should be ≈0.6 wt.% at 1200˚C, the average oxygen concentration in the beta phase approaches 0.6 wt.% with time, as it is diffusion limited. Also, diffusion-limited oxidation causes growth of the oxide and alpha layers and decrease in beta-layer thickness with time. The 13%-ECR Zry-4 sample should have more ductility than the 20%-ECR sample, unless the alloy is already brittle at 13% ECR. Given this logic, the alloy might be brittle at 11% measured ECR, based on the measured offset and permanent strains. Similarly, in comparing M5 to Zry-4, the metallography and microhardness measurements suggest that M5 should have more ductility than Zry-4 at ≥13% ECR, even though the offset and permanent strains are essentially the same. The implication is that neither alloy may have any room-temperature post-quench ductility after oxidation at 1200˚C to ≥13% ECR.

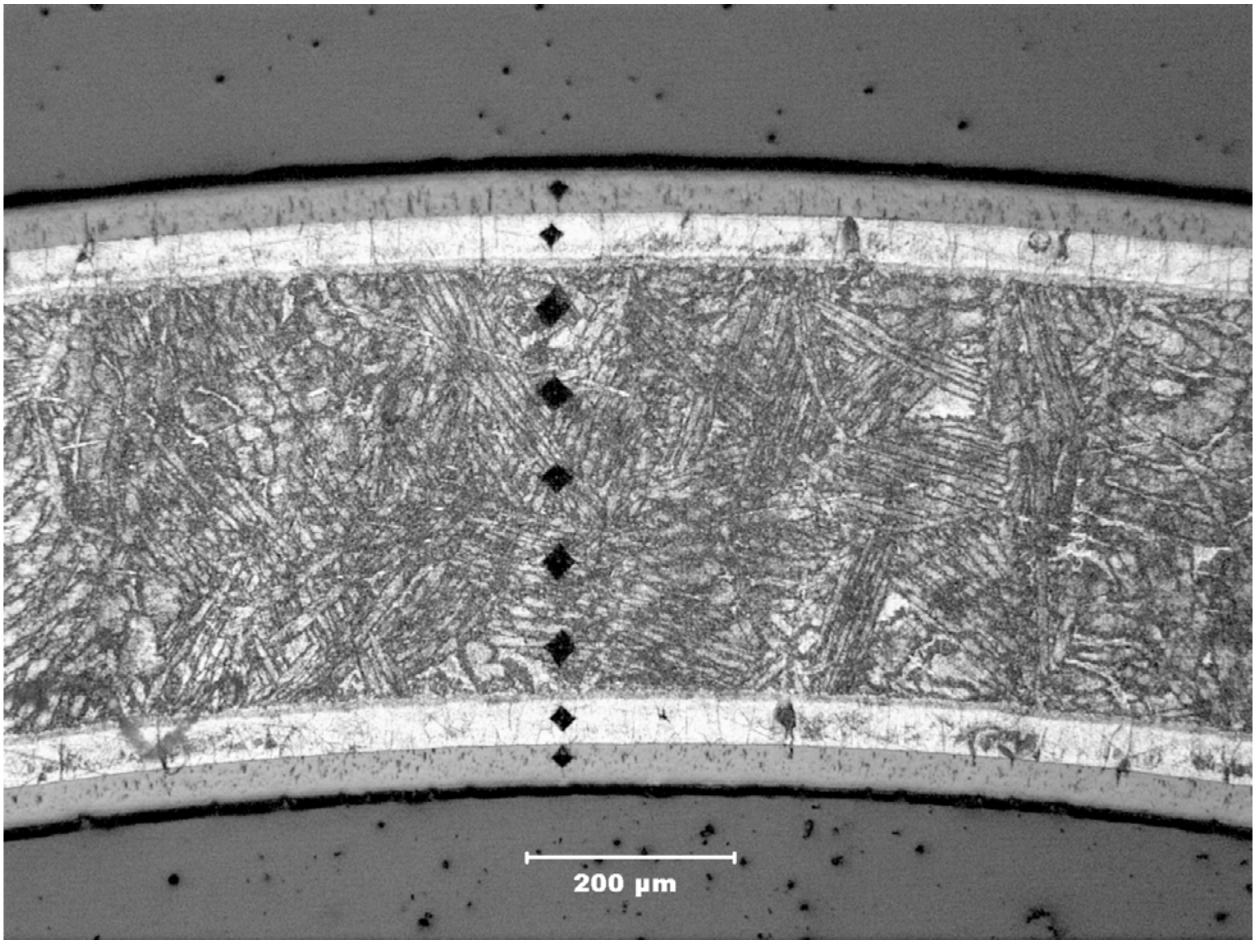


Fig. 6. Metallography and microhardness indents across the radius of Zry-4 oxidized at 1200˚C to 12.8% measured ECR.

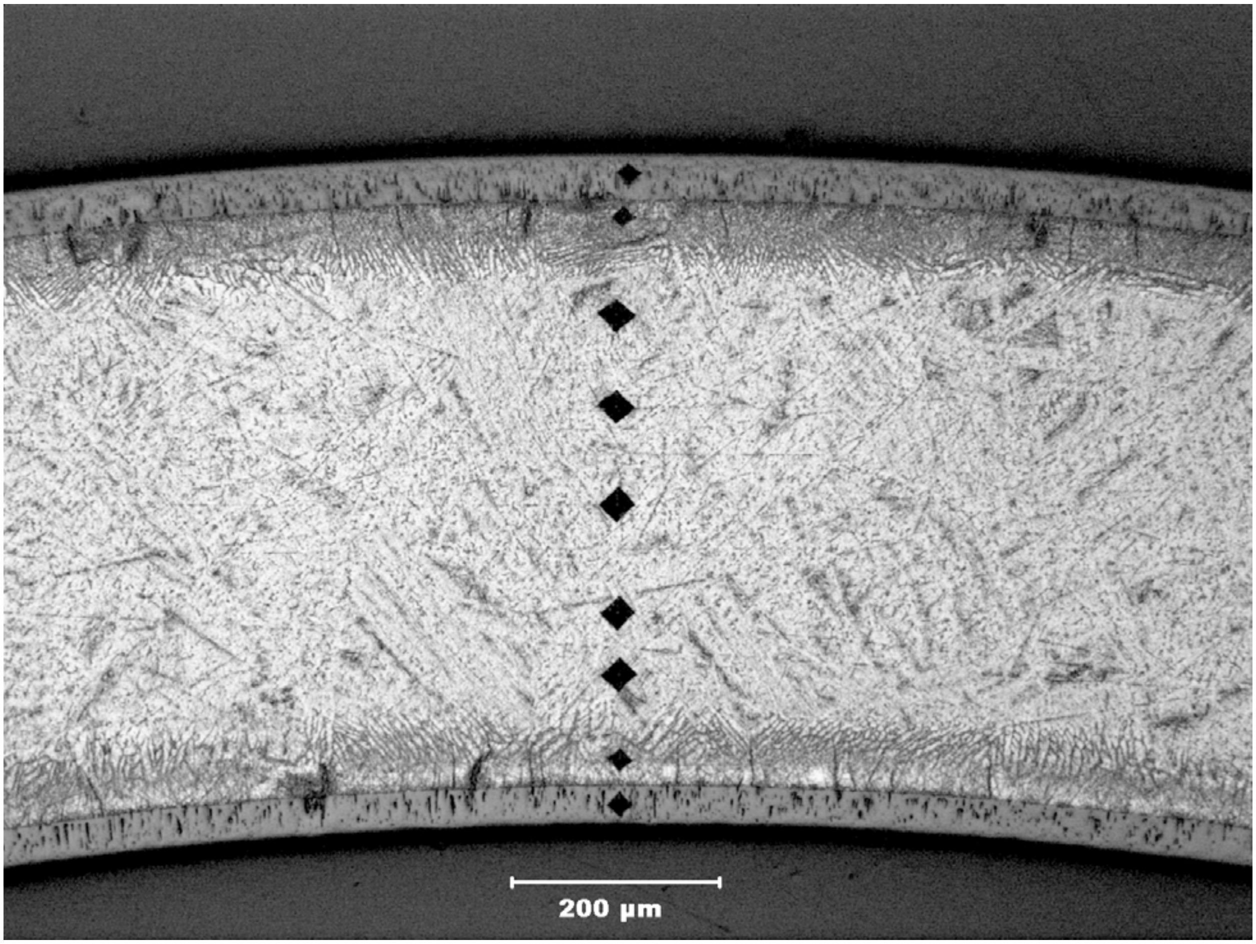


Fig. 7. Metallography and microhardness indents across the radius of M5 oxidized at 1200˚C to 13.1% measured ECR.

4. Summary of Results and Future Work

Weight gain measurements and ring-compression tests have been completed for Zry-4 and M5 cladding-alloy samples, which were oxidized in steam at 1200˚C, slow-cooled to 800˚C and water quenched. 25-mm-long samples were exposed to two-sided oxidation for test times up to ≈400 s for Zry-4 and M5. Both alloys exhibited about the same weight gain for each of the five test times and were in agreement with CP model predictions. Hydrogen pickup for the 13-20% ECR samples was low (<20 wppm).

Ring-compression tests were performed at room temperature and a displacement rate of 2 mm/minute to determine post-quench ductility. Tests were generally conducted through the first significant load drop indicative of a through-wall crack extending along the length of the sample. Maximum offset displacement for these cases was determined by mathematically unloading the sample at the same slope as the loading stiffness. The offset displacements determined from the load-displacement curves, normalized to the 9.50-mm outer diameter, were used as one measure of ductility. Samples with offset strains >2% (>0.2-mm displacement) were classified as ductile. This 2% limit is based on uncertainties in the unloading slope, which has been demonstrated to decrease with increasing displacement. Based on this offset-strain criterion, Zry-4 and M5 oxidized at 1200˚C exhibit room-temperature ductility for ECR ≤ 8% and ≤9%, respectively.

For ring samples with small offset strains (≤2%), a more direct method was used to determine permanent displacement and strain. The post-test diameter along the loading direction was measured and compared to the pre-test diameter. The diameter changes are 0.01-to-0.17 mm lower than the ones determined by the offset method. Additional loading-unloading-reloading tests were conducted to confirm that the unloading stiffness is less than the initial loading stiffness used in the offset-strain method. Thus, the magnitude of the difference in ductility determined by the two methods depends on the maximum load and the stiffness just prior to through-wall-crack initiation. Based on this approach, permanent strains dropped abruptly from ≈40-60% at ≈5% ECR to 0.4-2.0% at ≥10% CP-ECR for both alloys. For such low values of permanent strain, microstructural and microhardness characterization was performed on 13%- and 20%-ECR samples to aid in the "ductile" vs. "brittle" assessment. Both metallography and microhardness suggest that the 13% ECR samples should be more ductile than the 20% ECR samples. Given that there is no observable change in ductility in the ECR range of 13-20%, the implication is that both Zry-4 and M5 are brittle at ≥13% ECR and Zry-4 is brittle at ≥11%. Although there is very little overlap in test conditions and data interpretation between the ANL study and Hobson's study [2, 3], Table 5 shows that the ANL results for Zry-4 are consistent with Hobson's results. Table 5 also indicates that the cladding alloys may be ductile at ≥13% ECR when they are tested at a higher temperature. Consequently, ring compression tests will be repeated at the post-LOCA relevant temperature of 135˚C. To partially compensate for the loss in temperature conservatism, the 135˚C-tests will be conducted at a higher strain rate (3.5%/s).

Future work consists of performing ring compression tests at 135˚C on 1200˚C-oxidized-and-quenched rings. M5 and Zry-4 LOCA integral tests will also be performed at 1200˚C and 13-17% calculated ECR. The ANL-proposed method of determining post-quench ductility is to subject the LOCA post-quench samples to a four-point-bend test, followed by ring compression tests using 8-mm-long rings cut from the beyond-balloon-neck regions.

Table 5 Comparison of Hobson's Results for 0.675-mm-thick Zry-4 with ANL Results for 0.57-mm-thick Zry-4 for Samples Oxidized at 1204˚C (Hobson) and 1200˚C (ANL). Hobson compressed his samples to 3.81 mm, which is 36% for his 10.72-mm OD samples. Hobson's nominal test times of 4, 6, 8, and 10 minutes were converted to Cathcart-Pawel ECR values for this comparison.

Strain Rate (slow = 0.4%/s) (fast = impact)	Ring-Compression Test Temperature ˚C	ECR	Hobson Results	ANL Results Permanent Strain %
Slow	RT	13	4 fractures	0.7
	RT	19	brittle	0.5
	RT	21	brittle	0.4
	93	13	ductile	---
	93	21	brittle	---
	149	19	1-3 fractures	---
Fast	93	13	4 fractures	---
	93	16	Brittle	---
	149	13	1 fracture	---
	204	16	Brittle	

References

1. Y. Yan, T. Burtseva and M. C. Billone, "Post-Quench Ductility Results for Zry-4 and M5 Oxidized at 1000˚C and 1100˚C," letter report to NRC and Framatome, January 31, 2004.

2. D. O. Hobson and P. L. Rittenhouse, "Embrittlement of Zircaloy-Clad Fuel Rods by Steam during LOCA Transients," ORNL 4758, January 1972.

3. D. O. Hobson, "Ductile-Brittle Behavior of Zircaloy Fuel Cladding," Proc. ANS Topical Mtg. on Water Reactor Safety, Salt Lake City, March 26, 1973, pp. 274-288.

4. M. C. Billone, T. Burtseva and Y. Yan, "Hydrogen Pickup and Ring-Compression Ductility for E110 Oxidized in Steam at 1000˚C," letter report to NRC and RRC KI, January 31, 2004.

APPENDIX A

Load-Displacement Curves for Oxidized-and-Quenched Zry-4 Samples Subjected to Ring-Compression Tests at Room Temperature and a Cross-head Displacement Rate of 2 mm/minute

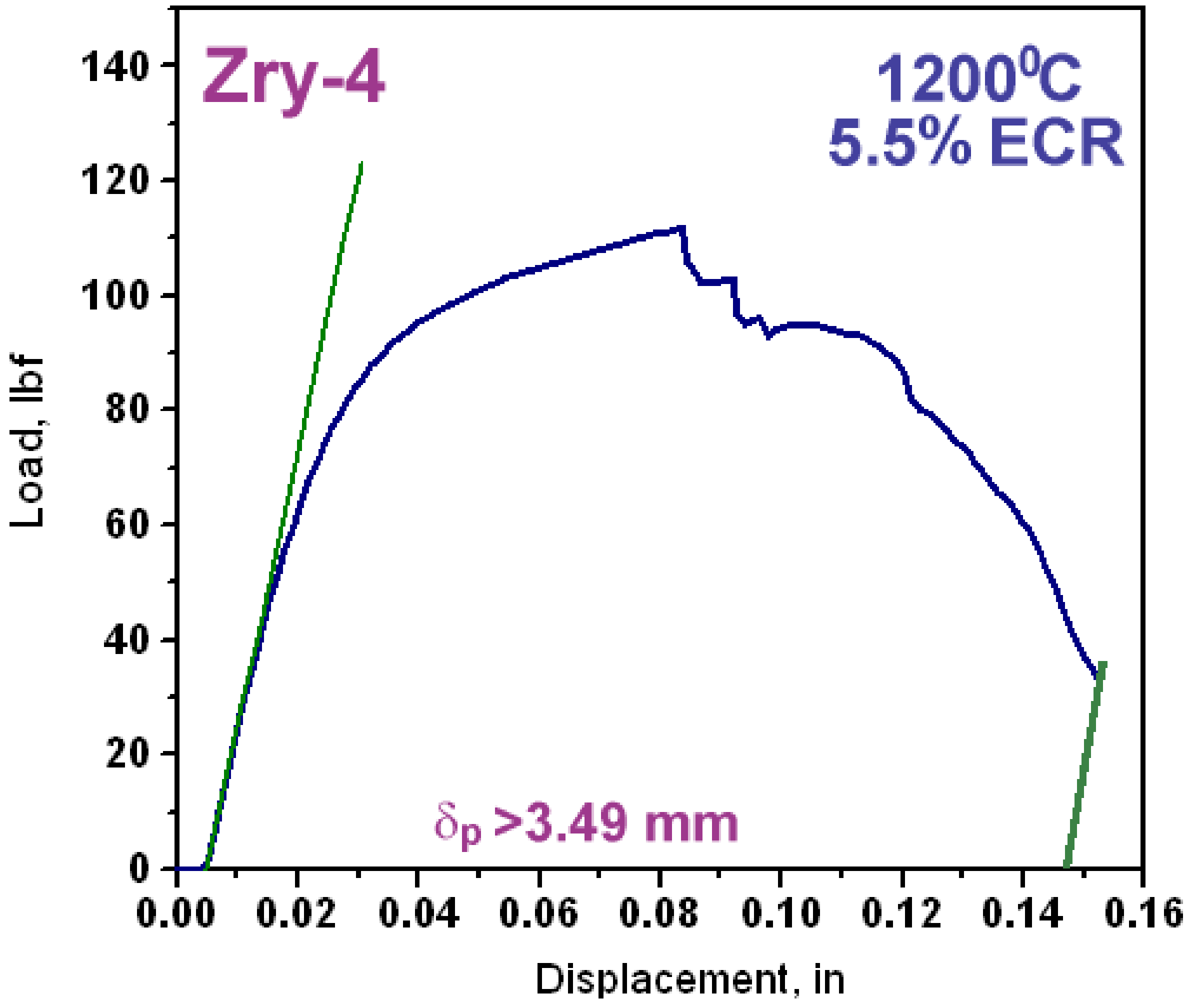


Fig. A.1a Ring-compression load-displacement data for Zry-4 oxidized to 5.5% ECR at 1200˚C. Sample was intact; no through-wall crack observed. Offset displacement prior to failure is >3.5 mm based on unloading from the end point of the curve. Permanent displacement based on pre-test minus post-test measurement along loading line is 3.23 mm.

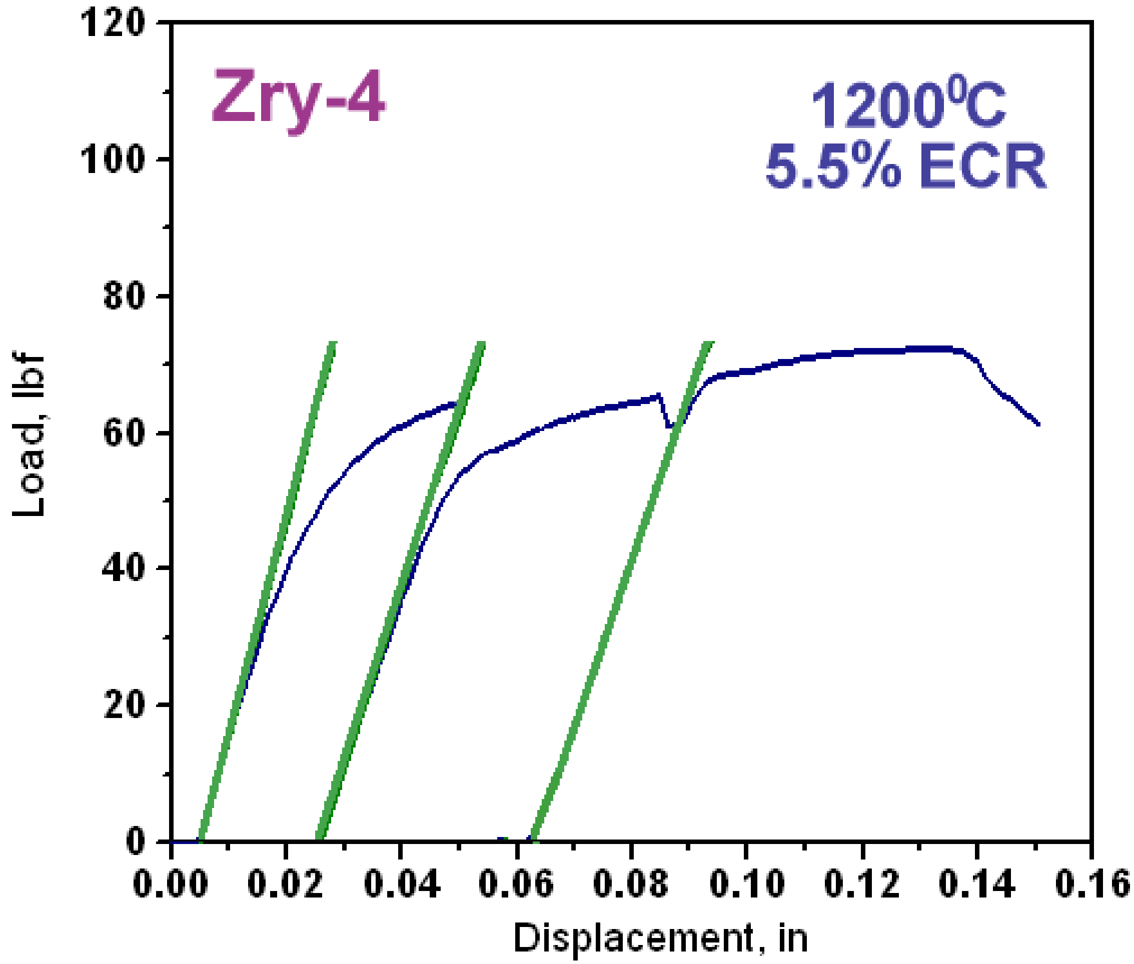


Fig. A.1b Ring-compression load-displacement data for Zry-4 oxidized to 5.5% ECR at 1200˚C (5.1-mm-long sample from same oxidized sample used to cut 8-mm ring for results shown in Fig. A.1a). The sample was loaded and unloaded four times during the test to study the change in stiffness with displacement (see Fig. A.1c for the 4th loading history). The stiffness (slope of loading line) decreased from 0.56 to 0.38 to 0.38 kN/mm for the 3 loading cycles.

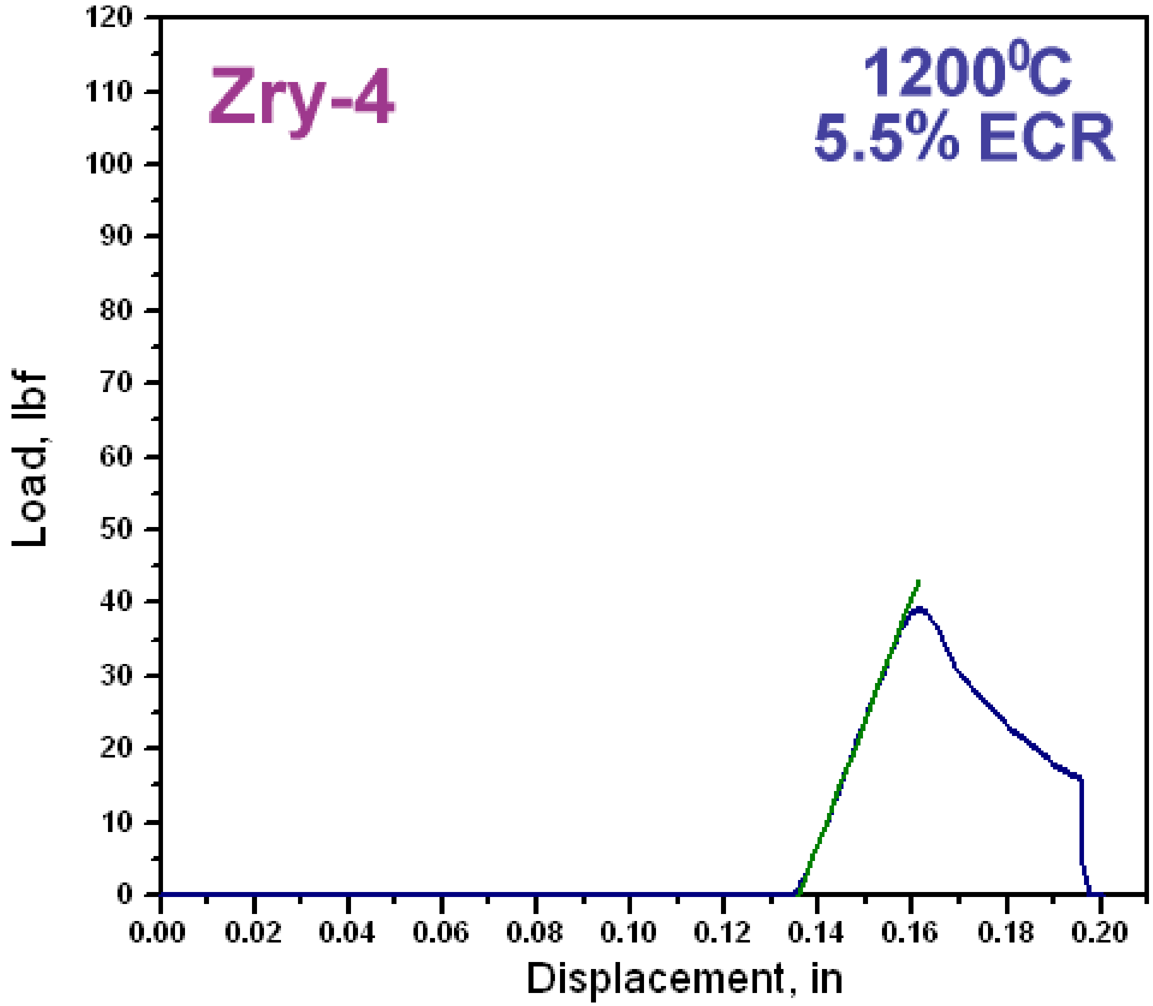


Fig. A.1c 4th-cylce ring-compression load-displacement data for Zry-4 oxidized to 5.5% ECR at 1200˚C (5.1-mm-long sample from same oxidized sample used to cut 8-mm ring for results shown in Fig. A.1a). The stiffness (slope of the loading line) decreased from 0.38 kN/mm (3rd loading cycle) to 0.30 kN/mm (4th loading cycle). The cumulative permanent displacement prior to failure, based on decreasing stiffness values, is 4.28 mm.

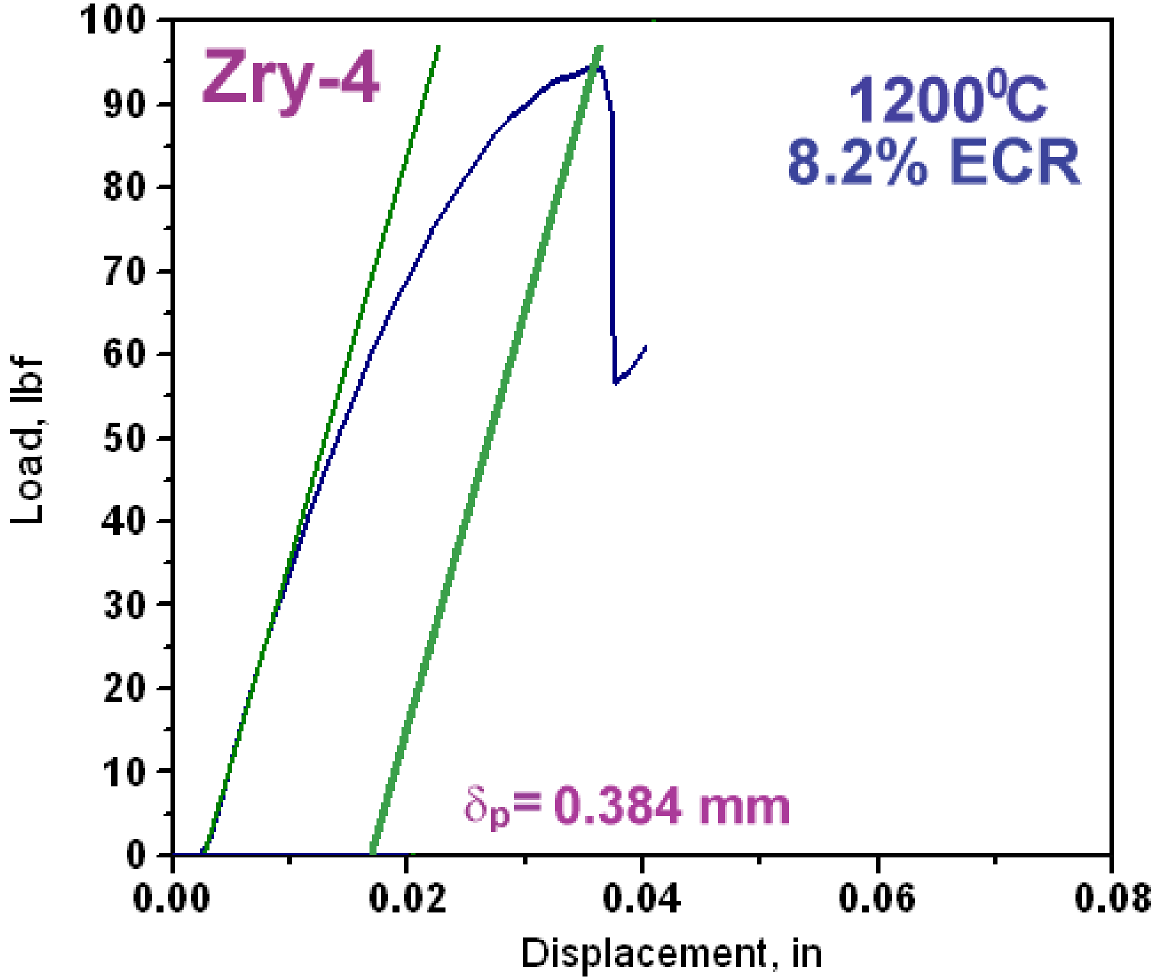


Fig. A.2 Ring-compression load-displacement data for Zry-4 sample (6.91-mm-long) oxidized to 8.2% ECR at 1200˚C. Offset displacement is 0.384 mm. Diameter decrease in the loading direction is 0.21 mm. Diameter increase 90˚ from loading direction is 0.18 mm.

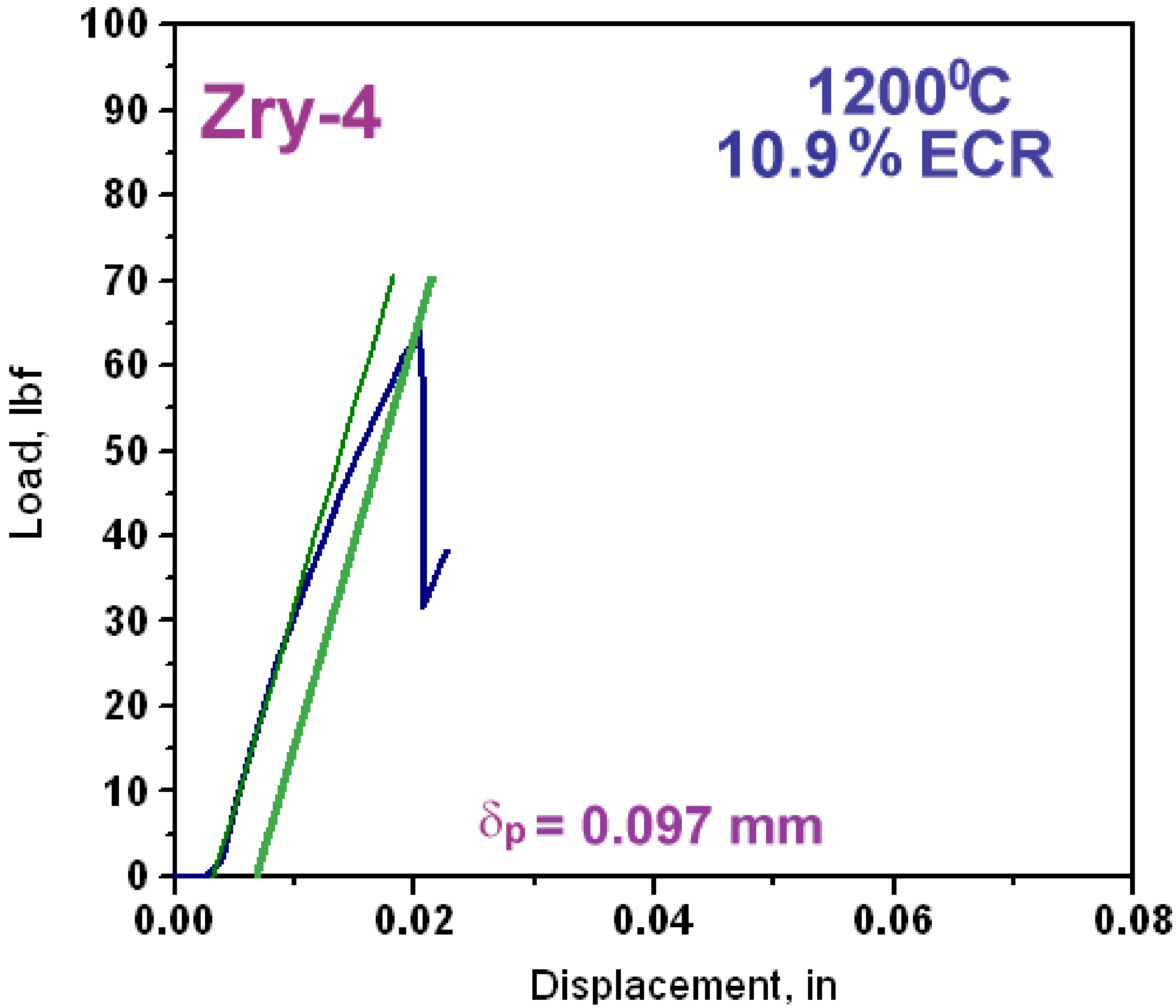


Fig. A.3 Ring-compression load-displacement data for Zry-4 oxidized to 10.9% ECR at 1200˚C. One very tight through-wall crack was observed at the load application point. Offset displacement is 0.097 mm. Permanent displacements based on post-test measurements are -0.05 mm along the loading direction and +0.04 mm normal to the loading direction.

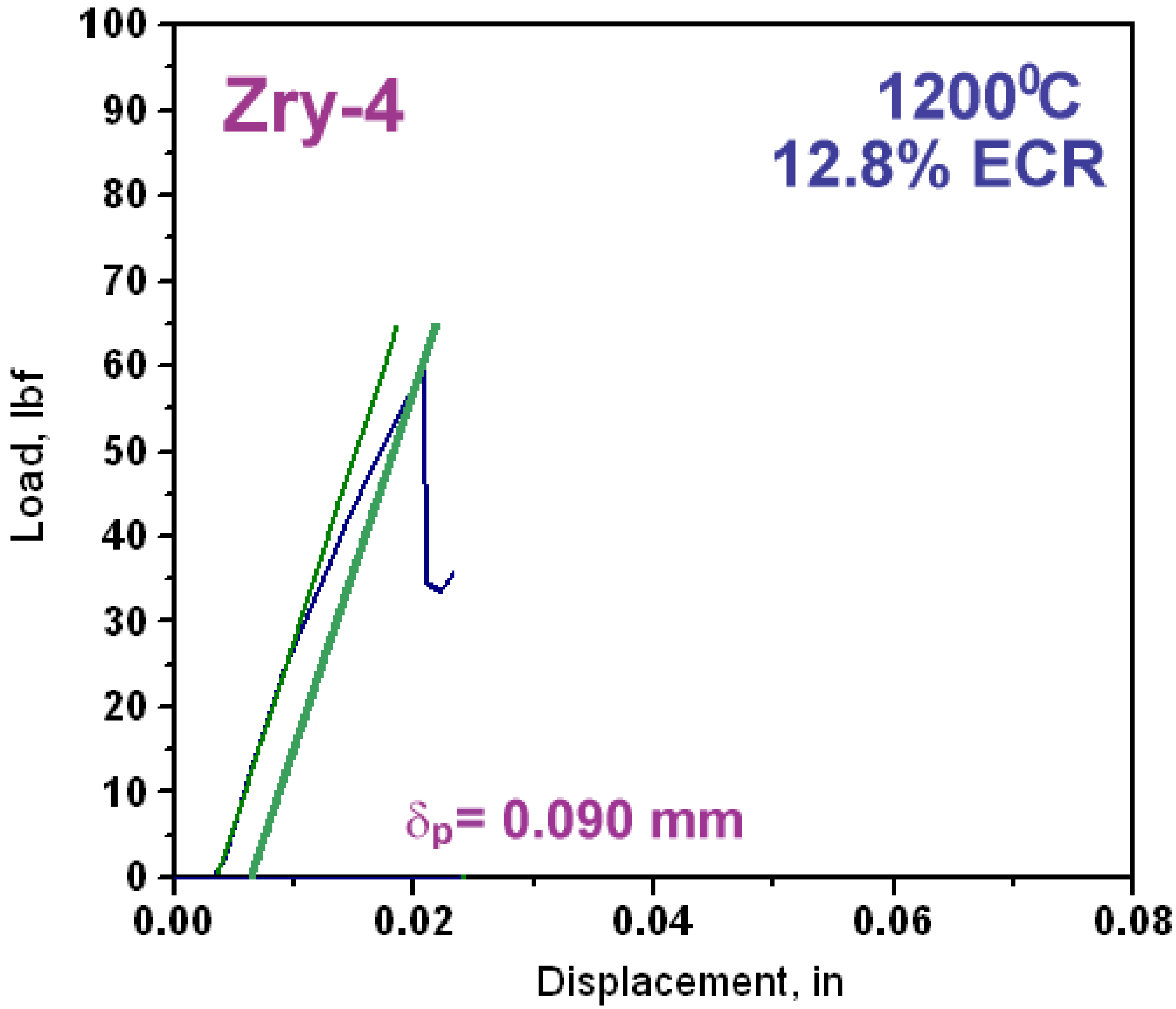


Fig. A.4 Ring-compression load-displacement data for Zry-4 oxidized to 12.8% ECR at 1200˚C. One very tight through-wall crack was observed at the load application. Offset displacement is 0.090 mm. Permanent displacements based on post-test measurements are -0.07 mm along the loading direction and +0.06 mm normal to the loading direction.

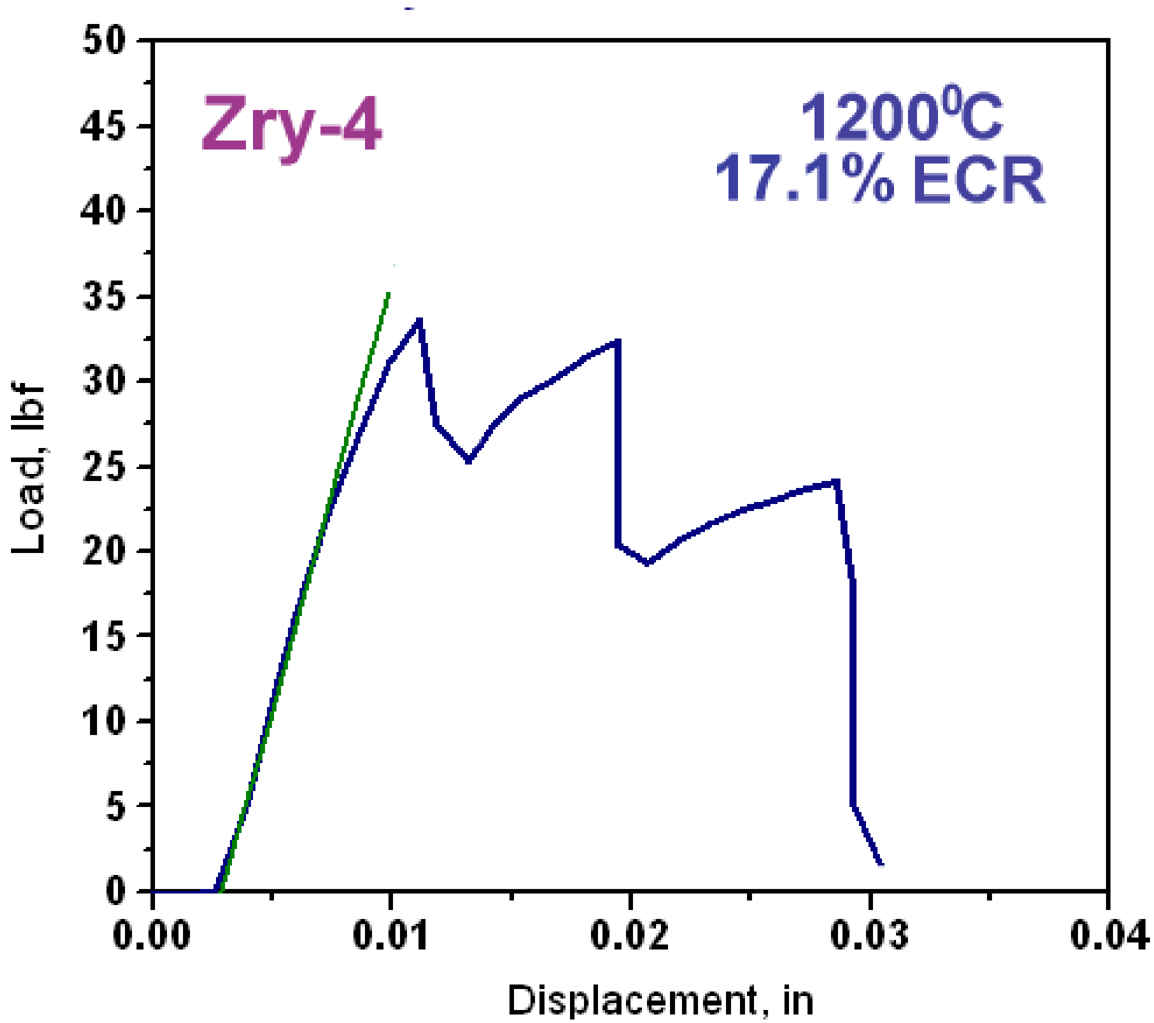


Fig. A.5a Composite ring-compression load-displacement data for Zry-4 oxidized to 17.1% ECR at 1200˚C. The test was stopped after the first load drop (0.04-mm offset based on Fig. A.5b), which may or may not have represented a through wall failure, and then the sample was reloaded. The offset strain associated with the second peak is 0.12 mm (see Fig. A.5c).

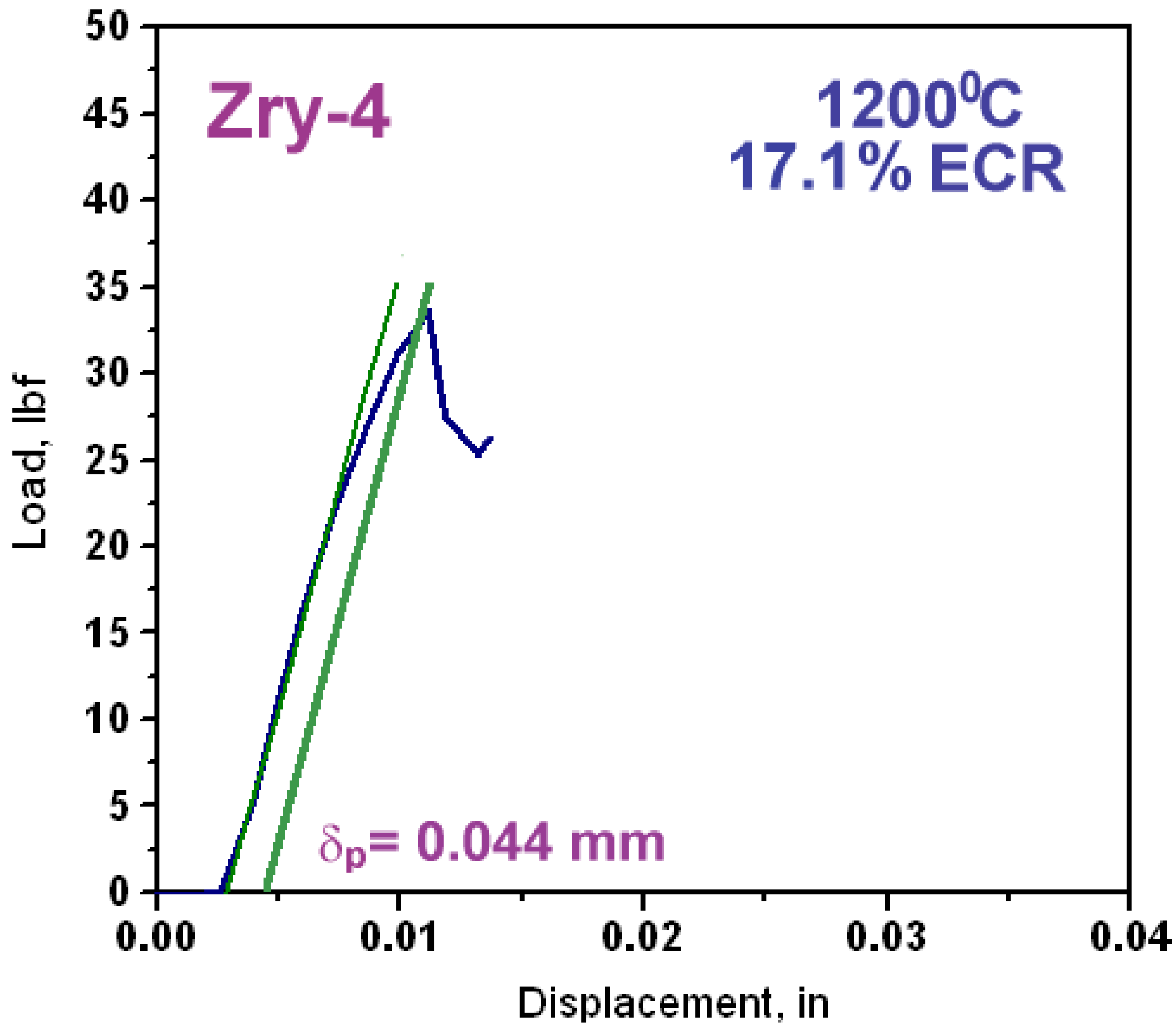


Fig. A.5b Initial ring-compression load-displacement data for Zry-4 oxidized to 17.1% ECR at 1200˚C. The test was stopped after the first load drop, which may or may not have represented a through wall failure. The offset strain associated with this first load drop is 0.044 mm.

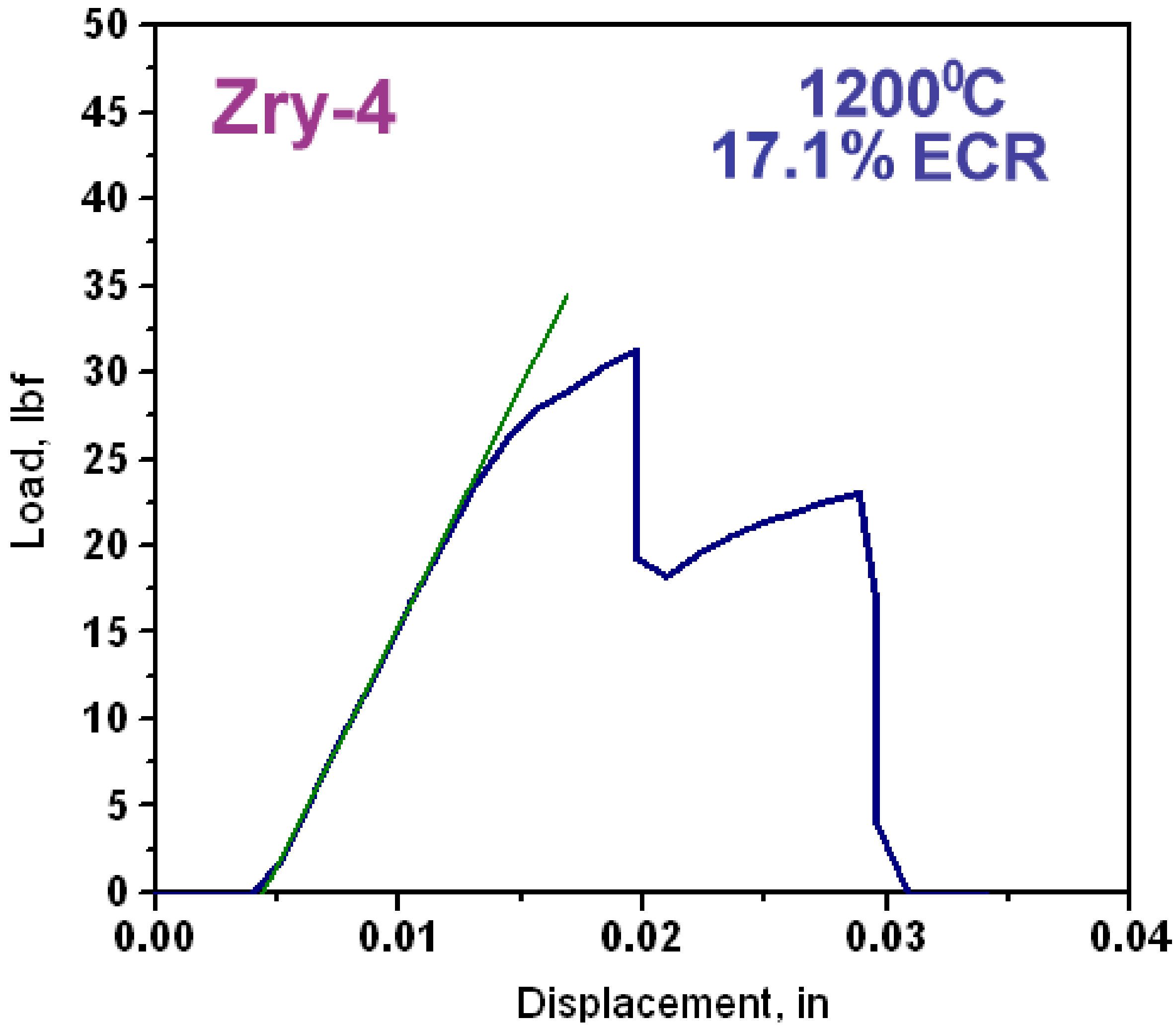


Fig. A.5c Continuation of ring-compression load-displacement data for Zry-4 oxidized to 17.1% ECR at 1200˚C. The sample fractured into 3 pieces, with through-wall cracks at the support location and ±90˚ from this location. Based on unloading from the first peak in this curve at the reduced slope and including the displacement from Fig. A.5b gives an offset strain of 0.12 mm.

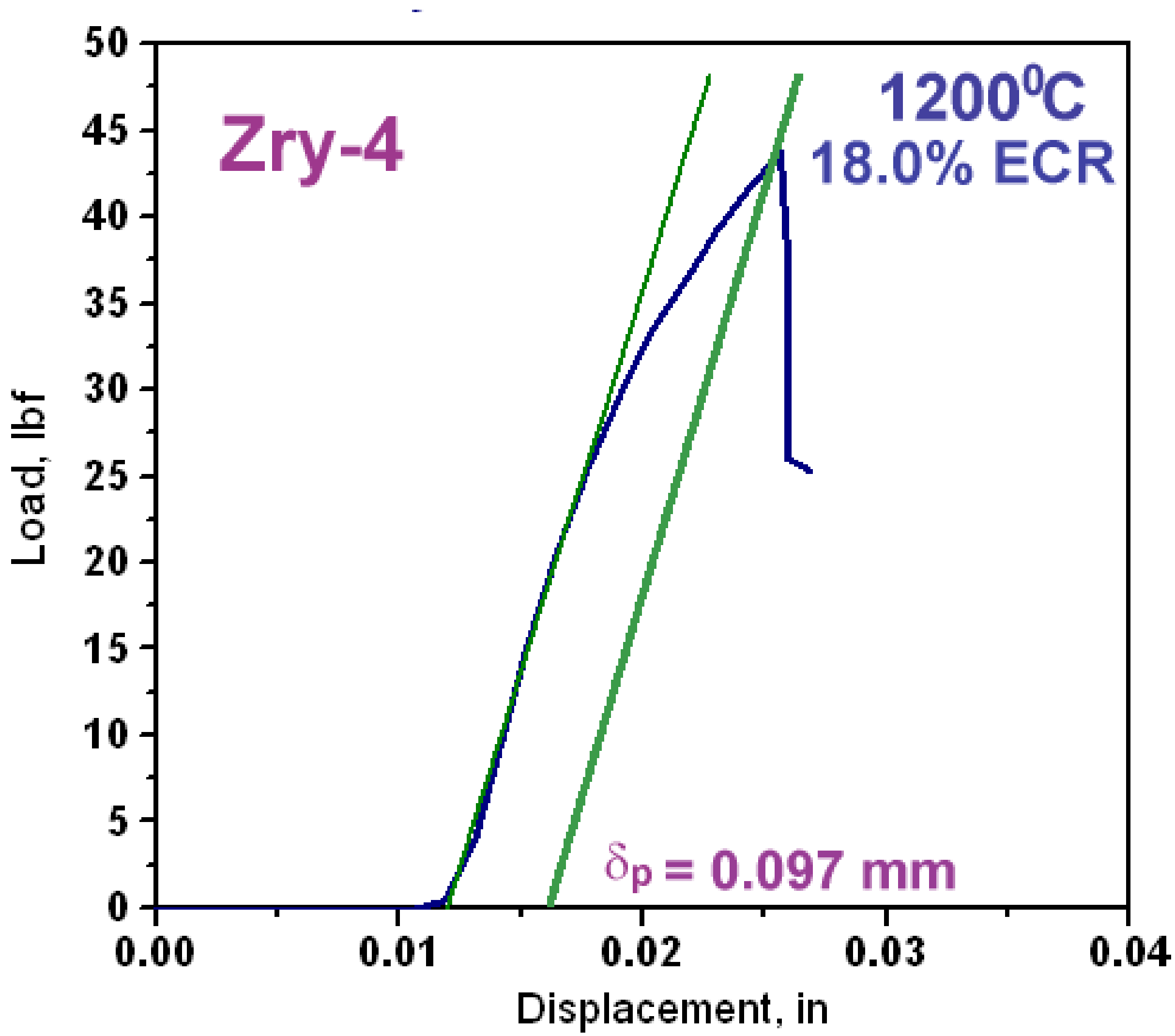


Fig. A.6 Ring-compression load-displacement data for Zry-4 oxidized to 18.0% ECR at 1200˚C. One through-wall crack was observed at the load application point. The offset displacement is 0.097 mm. Permanent displacements based on post-test measurements are -0.04 mm along the loading direction and +0.06 mm normal to the loading direction.

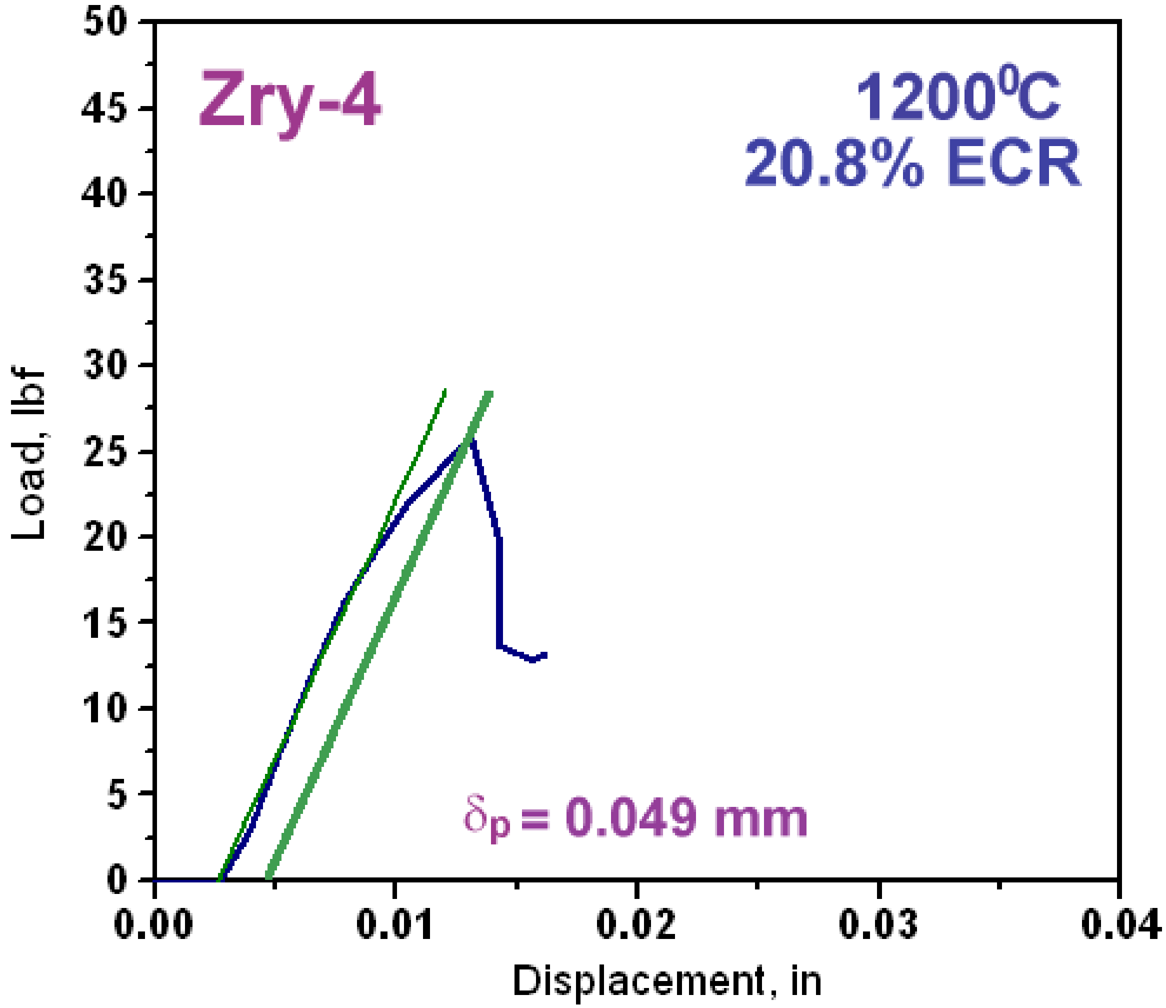


Fig. A.7 Ring-compression load-displacement data for Zry-4 oxidized to 20.8% ECR at 1200˚C. One through-wall crack was observed at the support location. The offset displacement is 0.049 mm. Permanent displacements based on post-test measurements are -0.04 mm along the loading direction and +0.03 mm normal to the loading direction.

APPENDIX C

Load-Displacement Curves for Oxidized-and-Quenched M5 Samples Subjected to Ring-Compression Tests at Room Temperature and a Cross-head Displacement Rate of 2 mm/minute

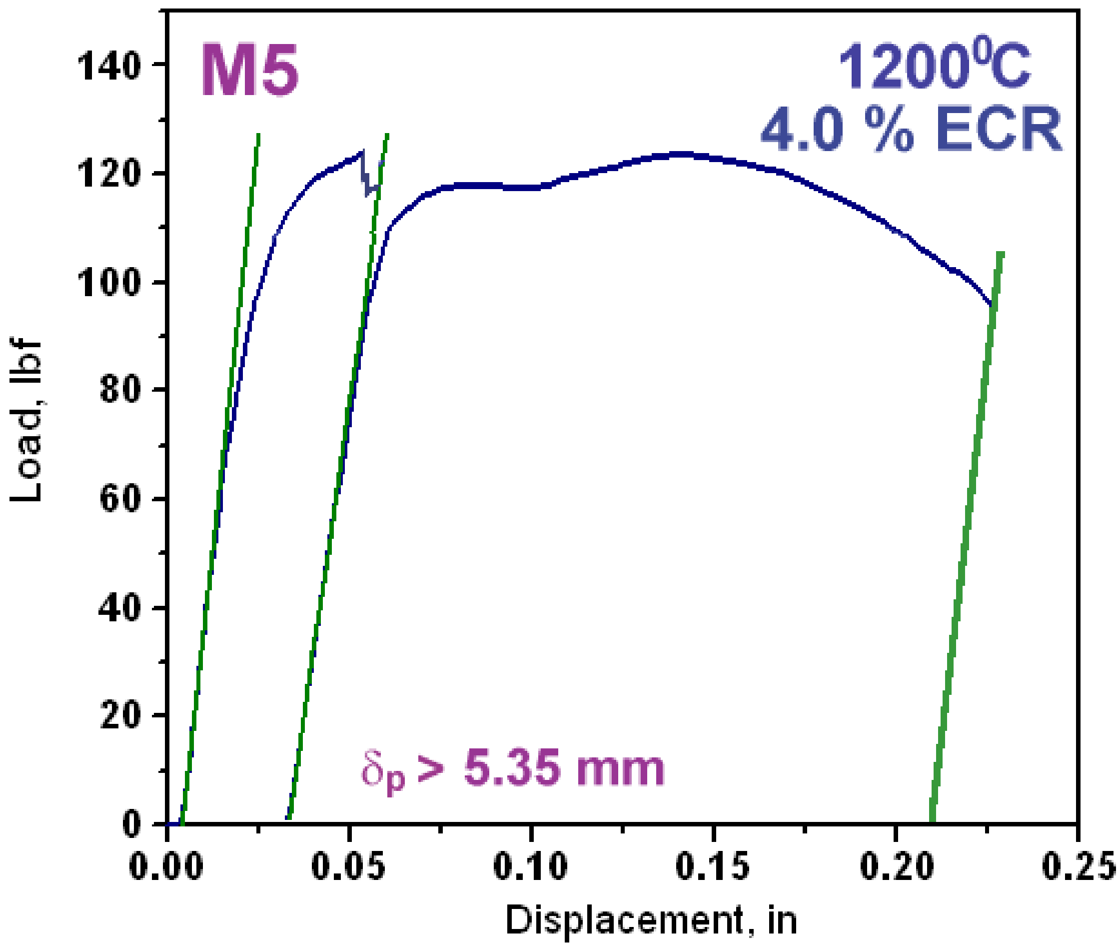


Fig. C.1 Ring-compression load-displacement data for M5 oxidized to 4% ECR at 1200˚C. The sample was loaded and unloaded twice during the test to study the change in stiffness with displacement. The loading stiffness decreased from 1.07 to 0.79 kN/m from the initial loading to the second loading. The sample was intact. Based on unloading at the initial stiffness, the offset displacement to failure is >5.35 mm. Based on direct post-test diameter decrease measurement, the permanent displacement is 5.0 mm.

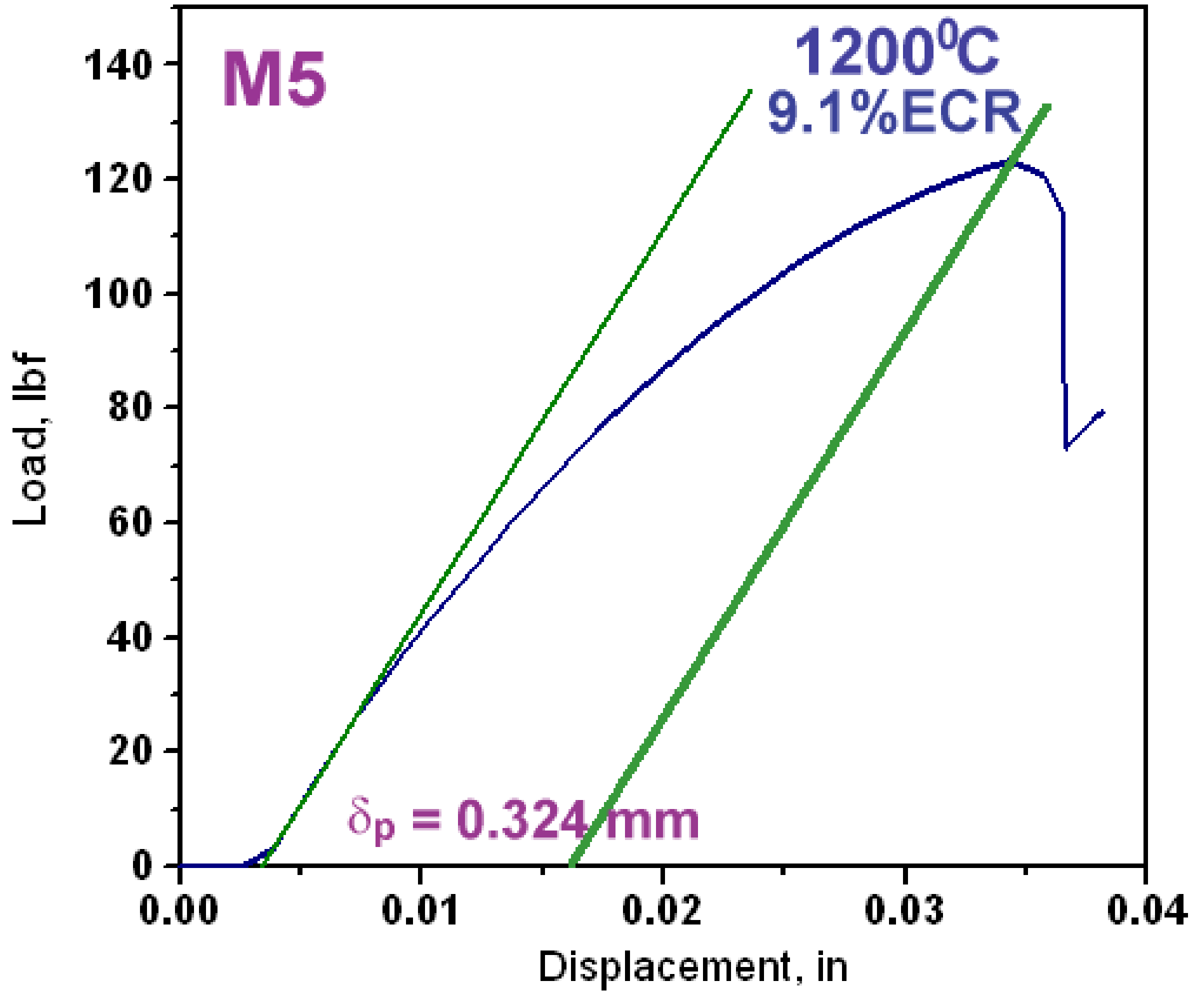


Fig. C.2 Ring-compression load-displacement data for M5 oxidized to 9.1% ECR at 1200˚C. One very tight through-wall crack was observed at support point. Offset displacement is 0.324 mm. Permanent displacements based on post-test measurements are -0.15 mm along the loading direction and +0.16 mm normal to the loading direction.

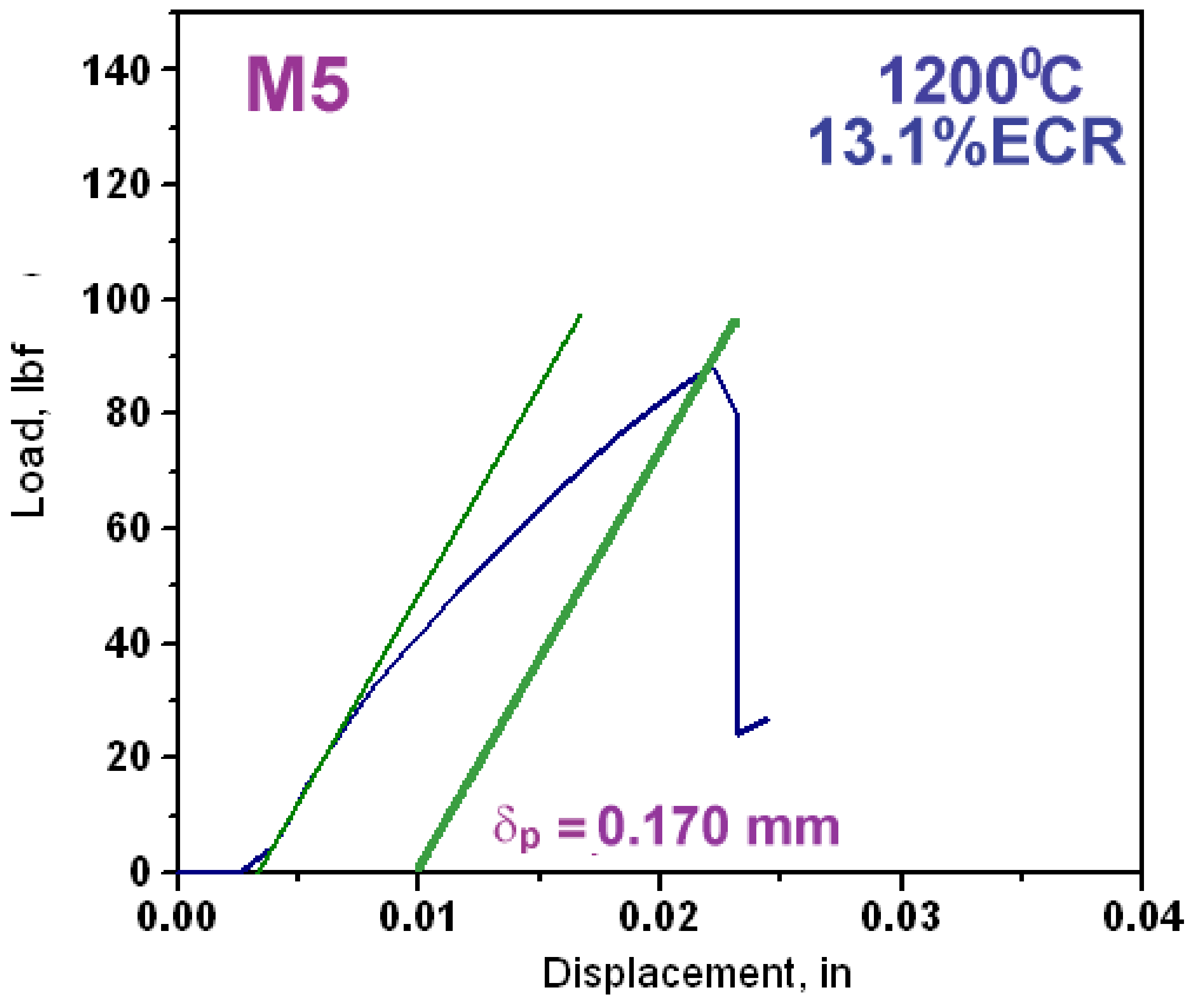


Fig. C.3 Ring-compression load-displacement data for M5 oxidized to 13.1% ECR at 1200˚C. Through-wall cracks were observed at loading (open) and the support (tightly closed) points. Offset displacement is 0.170 mm. Permanent displacements based on post-test measurements are -0.09 mm along the loading direction and +0.10 mm normal to the loading direction.

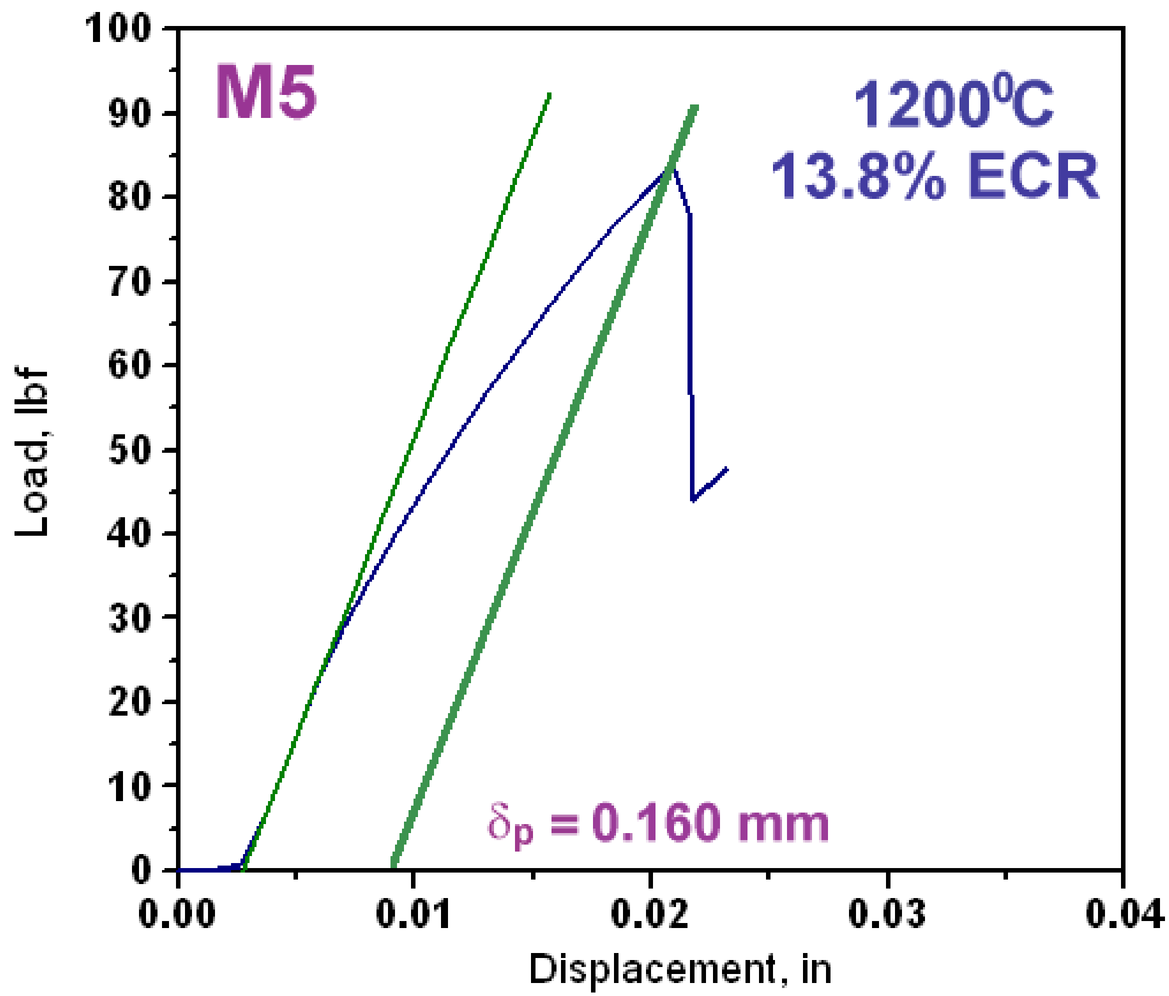


Fig. C.4 Ring-compression load-displacement data for M5 oxidized to 13.8% ECR at 1200˚C. One very tight through-wall crack was observed at the load point. Offset displacement is 0.160 mm. Permanent displacements based on post-test measurements are -0.07 mm along the loading direction and +0.06 mm normal to the loading direction.

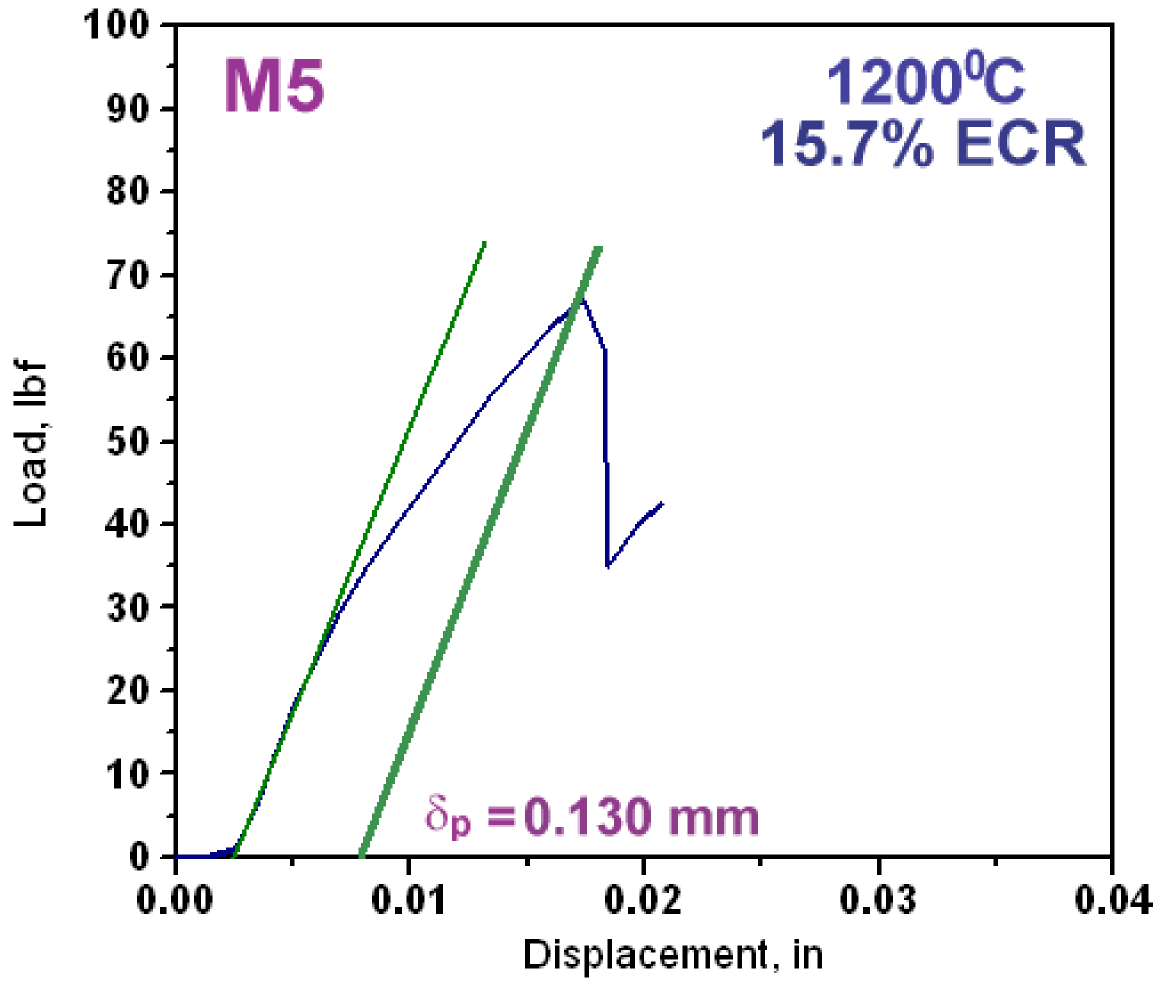


Fig. C.5 Ring-compression load-displacement data for M5 oxidized to 15.7% ECR at 1200˚C. One very tight through-wall crack was observed at the support point. Offset displacement is 0.130 mm. Permanent displacements based on post-test measurements are -0.06 mm along the loading direction and +0.05 mm normal to the loading direction.

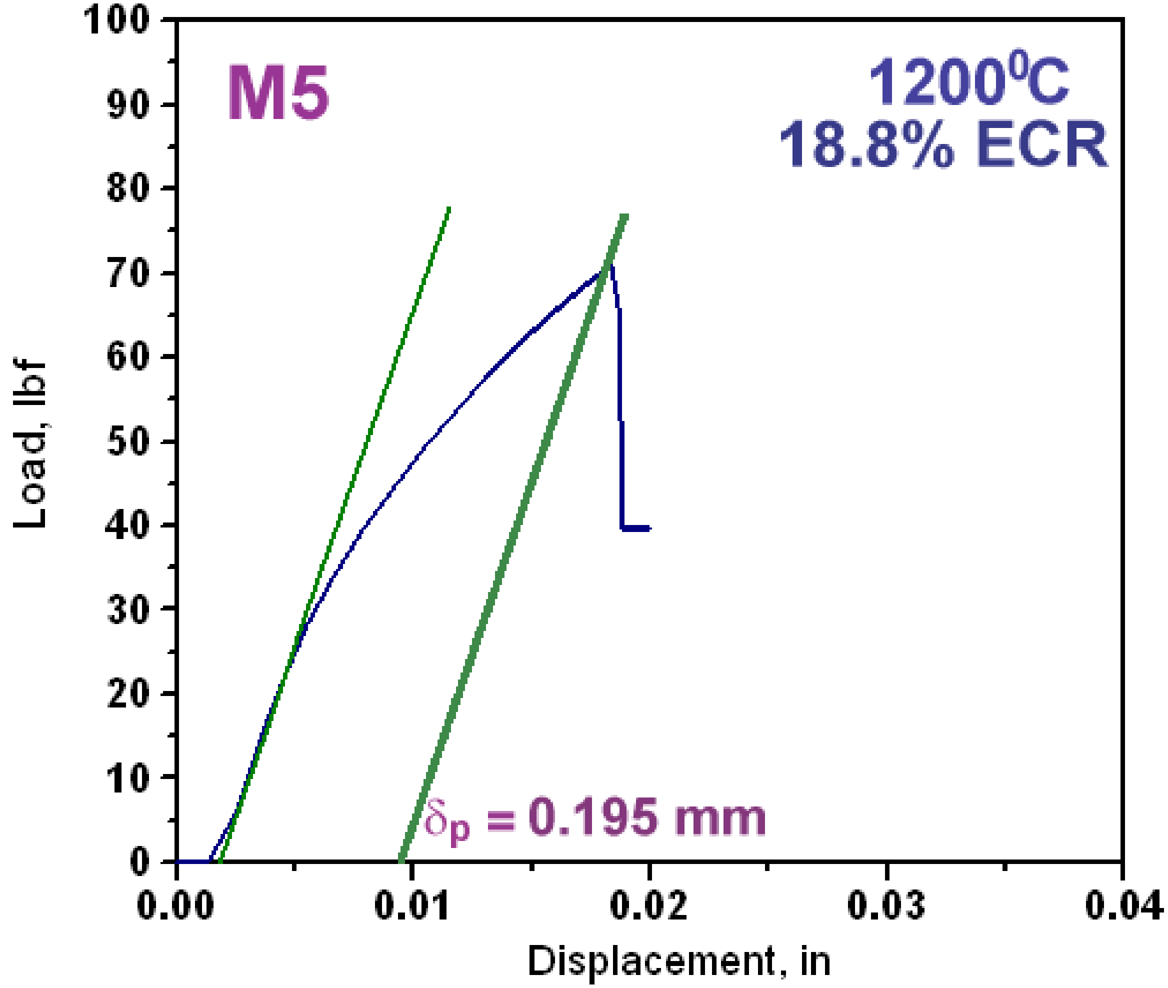


Fig. C.6 Ring-compression load-displacement data for M5 oxidized to 18.8% ECR at 1200˚C. One very tight through-wall crack was observed at the load point. Offset displacement is 0.195 mm. Permanent displacements based on post-test measurements are -0.06 mm along the loading direction and +0.06 mm normal to the loading direction.