U.S. NUCLEAR REGULATORY COMMISSION
OFFICE OF NUCLEAR REGULATORY RESEARCH

March 2014
Division 1

DRAFT REGULATORY GUIDE

Contact: M. Flanagan
(301) 251-7547

DRAFT REGULATORY GUIDE DG-1262

(Proposed New Regulatory Guide)

TESTING FOR POSTQUENCH DUCTILITY

A. INTRODUCTION

This guide describes an experimental technique that is acceptable to the U.S. Nuclear Regulatory Commission (NRC) for measuring the ductile-to-brittle transition for a zirconium (Zr)-based cladding alloy, as called for in Title 10, Section 50.46c, of the *Code of Federal Regulations* (Ref. 1). The experimental technique utilizes ring-compression testing (RCT) of Zr-based cladding alloys following exposure to oxidation and quench conditions related to a loss-of-coolant accident (LOCA).

The experimental technique described in this guide is acceptable for generating data for new Zr-based cladding alloys. These data can be used to demonstrate comparable performance with the database established in the NRC's LOCA research program. In some instances, a Zr-based cladding alloy may experience the transition from ductile-to-brittle behavior at a higher level of oxidation than indicated in the database established in the NRC's LOCA research program. The experimental technique provided in this guide is acceptable for generating data to support the development of specific analytical limits on peak cladding temperature and integrated time at temperature for Zr-based cladding alloys. Further, some emergency core cooling systems (ECCSs) may perform such that the maximum oxidation temperature is significantly below the maximum peak cladding temperature criteria in 10 CFR 50.46 of 2,200 degrees Fahrenheit (°F) (1,204 degrees Celsius (°C)). The database in the NRC's LOCA research program is intended to bound ECCS performance by testing materials at the maximum oxidation temperature permitted by 10 CFR 50.46. Oxidation at lower temperatures has been shown to increase the allowable calculated oxidation before embrittlement. Therefore, conducting tests at lower peak temperatures may provide additional margin for some Zr-based cladding alloys. The experimental technique provided in this guide is acceptable for generating data to support the development of specific analytical limits for Zr-based cladding alloys at peak oxidation temperatures below 2,200 °F (1,204 °C).

The NRC issues regulatory guides to describe to the public methods that the staff considers acceptable for use in implementing specific parts of the agency's regulations, to explain techniques that the staff uses in evaluating specific problems or postulated accidents, and to provide guidance to

applicants. Regulatory guides are not substitutes for regulations and compliance with them is not required.

This regulatory guide contains information collection requirements covered by 10 CFR Part 50 that the Office of Management and Budget (OMB) approved under OMB control number 3150-0011. The NRC may neither conduct nor sponsor, and a person is not required to respond to, an information collection request or requirement unless the requesting document displays a currently valid OMB control number. This regulatory guide is a rule as designated in the Congressional Review Act (5 U.S.C. 801–808). However, OMB has not found it to be a major rule as designated in the Congressional Review Act.

B. DISCUSSION

Background

In 1996, the NRC initiated a fuel-cladding research program intended to investigate the behavior of high-exposure fuel cladding under accident conditions. This program included an extensive LOCA research and testing program at Argonne National Laboratory (ANL), as well as jointly funded programs at the Kurchatov Institute (Ref. 2) and the Halden Reactor project (Ref. 3), to develop the body of technical information needed to evaluate LOCA regulations for high-exposure fuel. The research findings have been summarized in Research Information Letter 0801, "Technical Basis for Revision of Embrittlement Criteria in 10 CFR 50.46," dated May 30, 2008 (Ref. 4). The detailed experimental results from the program at ANL are contained in NUREG/CR-6967, "Cladding Embrittlement During Postulated Loss-of-Coolant Accidents," issued July 2008 (Ref. 5).

The research program identified new cladding embrittlement mechanisms and expanded the NRC's knowledge of previously identified mechanisms. The research results revealed that alloy composition has a minor effect on embrittlement, but cladding corrosion, which occurs as fuel burnup increases, has a substantial effect on embrittlement. One of the major findings of the NRC's research program was that hydrogen, which is absorbed in the cladding as a result of waterside corrosion under normal operation, has a significant influence on embrittlement during a hypothetical accident.

Hydrogen-Enhanced Beta-Layer Embrittlement

As explained in Section 1.4 of NUREG/CR-6967 (Ref. 5), oxygen diffusion into the base metal under LOCA conditions promotes a reduction in the size (referred to as beta-layer thinning) and ductility (referred to as beta-layer embrittlement) of the metallurgical structure within the cladding which provides its overall ductility. The presence of hydrogen within the cladding enhances this embrittlement process.

During normal operation, some hydrogen from the corrosion process is absorbed in the cladding metal. When that cladding is exposed to high-temperature LOCA conditions, the elevated hydrogen levels increase the solubility of oxygen in the beta phase and the rate of diffusion of oxygen into the beta phase. Thus, even for LOCA temperatures below 2,200 °F (1,204 °C), embrittlement can occur for times corresponding to less than 17% oxidation in corroded cladding with significant hydrogen pickup.

Figure 1 illustrates the effect of hydrogen on RCT ductility measurements. The figure shows that ductility is lost in high-burnup Zircaloy-4 at a lower calculated equivalent cladding reacted (ECR) than as-fabricated (fresh) 15×15 Zircaloy-4. Significantly, Figure 1 indicates that ductility is lost in this high-burnup Zircaloy-4 cladding at a level of oxidation well below 17%.

As shown in Figure 1, ductility measurements were made on segments oxidized to a number of Cathcart-Pawel (CP) calculated oxidation levels. In characterizing the ductile-to-brittle transition behavior of the alloys tested in the NRC's LOCA research program, multiple postquench ductility (PQD) measurements were made at the same oxidation level to characterize the variability in test results. This is reflected in the test procedure described in the Regulatory Position section of this guide.

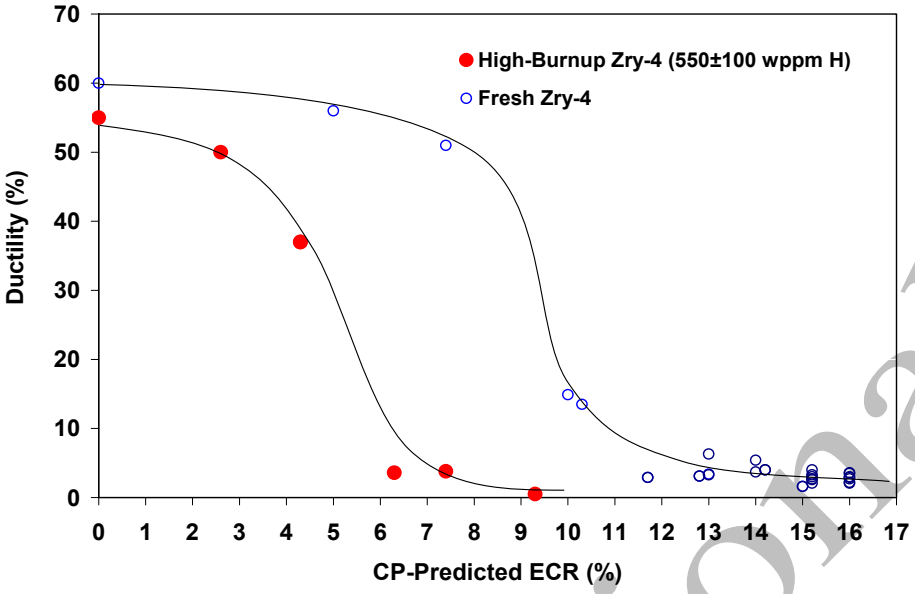

Figure 1. Measured offset strains[1]
(Source: NUREG/CR-6967 (Ref. 5))

Addressing Hydrogen-Enhanced Beta-Layer Embrittlement with Performance-Based Requirements

In the 1973 version of the ECCS performance rule, the preservation of cladding ductility, through compliance with regulatory criteria for peak cladding temperature and local cladding oxidation, provided a level of assurance that fuel cladding would not experience gross failure. Hence, the fuel rods would remain within their coolable lattice arrays. The recent LOCA research program identified new cladding embrittlement mechanisms, which demonstrate that the current combination of peak cladding temperature (2,200 °F (1,204 °C)) and local cladding oxidation (17% ECR) criteria may not always ensure PQD.

The rule in 10 CFR 50.46c considers the findings of the NRC's LOCA research program and calls for the establishment of analytical limits on peak cladding temperature and time that correspond to the measured ductile-to-brittle transition for the Zr-alloy cladding material. This guide describes an experimental technique that is acceptable for measuring the ductile-to-brittle transition for a Zr-alloy cladding by RCT, which can be used to (1) justify the use of the analytical limit defined in Draft Regulatory Guide (DG)-1263, "Regulatory Guidance on Establishing Analytical Limits for Zirconium-Based Alloy Cladding" (Ref. 6), for alloys not tested in the NRC's LOCA research program, (2) support the development of a specific limit for a Zr-based cladding alloy, or (3) support the development of analytical limits at peak oxidation temperatures less than 2,200 °F (1,204 °C).

[1] Test specimens included high-burnup Zircaloy (Zry)-4 (corrosion-layer thickness of 71–74 micrometers) and as-fabricated (fresh) HBR-type 15×15 Zry-4. Cladding samples were two-side oxidized at ≈1,200 ºC and cooled at ≈11 ºC/second (s) to 800 ºC. As-fabricated samples were quenched at 800 ºC, while the high-burnup samples were slow-cooled from 800 ºC to room temperature. The ductile-to-brittle transition oxidation level for high-burnup cladding is 8% for cladding cooled without quench and 5% for cladding quenched at 800 °C.

C. REGULATORY POSITION

The following procedure describes an acceptable experimental technique that can be used to measure the ductile-to-brittle transition for a Zr-based cladding alloy to establish specified and acceptable analytical limits on peak cladding temperature and integrated time at temperature, as called for by the proposed language of 10 CFR 50.46c.

Procedure for Conducting Oxidation and Postquench Ductility Tests with Zirconium-Based Cladding Alloys

1. Purpose and Scope of the Tests

Performance-based tests are needed to ensure that fuel rod cladding retains ductility following oxidation in steam at ≤1,200 ºC and quench at ≤800 ºC.[2] This procedure describes the tests to be conducted with fresh and prehydrided cladding samples for determination of the ductile-to-brittle transition oxidation level as a function of hydrogen content and either the hold temperature or the maximum oxidation temperature for samples that embrittle during the heating ramp. The procedure for conducting in-cell oxidation-quench tests with high-burnup cladding samples is similar to the one described for out-of-cell tests with unirradiated cladding samples. The critical differences in a procedure for conducting testing on irradiated, high-burnup material are in the area of thermal and weight-gain benchmarks. The sections addressing thermal and weight-gain benchmarks highlight the differences in test procedure for irradiated material.

The oxidation level is defined as the ECR calculated using the CP weight gain correlation. Retention of PQD is defined as the accumulation of ≥1.0% permanent strain prior to failure during ring-compression loading at a temperature of 135 ºC[3] and a displacement rate of 0.033 millimeters (mm)/s. The ductile-to-brittle transition oxidation level is defined as the maximum CP-ECR (rounded to the nearest percent) for which ductility is retained.

2. Background

During a LOCA, the cladding outer surface will be exposed to steam at elevated temperatures. This results in the growth of an outer surface oxide layer, an oxygen-stabilized metal alpha layer, and a metal beta layer with low oxygen content. The oxide and alpha layers are brittle, but the beta layer will retain ductility as long as its oxygen content is low (e.g., <0.6 weight parts per million (wppm)). This is close to the oxygen solubility limit at 1,200 ºC for the beta layer of as-fabricated cladding. As such, modern cladding alloys used in U.S. reactors oxidized at 1,200 ºC will retain ductility up to a time-at-temperature corresponding to a calculated oxidation level of 17–20% CP-ECR (Experimental Procedures (EP)-Ref. 1), where CP refers to the use of the Cathcart-Pawel (EP-Ref. 2) weight-gain correlation for the ECR calculation. During a LOCA, cladding embrittlement is the result of oxygen diffusion into the beta layer of the base metal and is not directly related to the growth of the Zr dioxide layer on the outside cladding diameter. The calculated maximum local oxidation limit is used as a surrogate to limit

---

[2] These test conditions were selected with the objective of bounding the performance of ECCSs. They are considered relevant and bounding for current light-water reactor ECCSs. However, it may be necessary to evaluate and possibly modify the conditions accordingly for the ECCSs of advanced reactor designs.

[3] During the 1973 hearing, investigators suggested that a test temperature no higher than the saturation temperature during reflood (i.e., ≈135 °C) be considered. This test condition is considered relevant for current light-water reactor ECCSs. However, it may be necessary to evaluate and possibly modify the conditions accordingly for the ECCSs of advanced reactor designs.

integrated time at temperature, associated oxygen diffusion into the beta layer, and ductility decrease caused by the increased oxygen content in the beta layer. This surrogate approach works because both oxidation rate and diffusion rate share similar temperature dependences. In particular, for as-fabricated alloys tested at 1,200 °C, there is a linear correlation for CP-ECR ≤17% between calculated oxidation level (CP-ECR) and increase in average oxygen content in the beta layer, which is the cause of embrittlement. However, for some alloys tested at 1,000 °C, the measured oxide-layer thickness growth rate and weight-gain rate decrease significantly with oxidation time, while the ductility continues to decrease with oxidation time. This demonstrates that diffusion of oxygen into the beta layer and oxygen solubility in the beta layer control embrittlement. In general, the decrease in ductility with time correlates quite well with the increase in CP-predicted ECR.

However, hydrogen pickup during reactor operation can cause a significant decrease in the ductile-to-brittle transition oxidation level (e.g., from 19% to 5% for 550 wppm hydrogen (EP-Ref. 1)). Hydrogen increases the oxygen solubility limit in the beta layer, as well as the rate of diffusion of oxygen into that layer. In addition, for oxidized cladding that undergoes very rapid cooling during quench, hydrogen is intrinsically embrittling. As hydrogen does not influence measured or calculated weight gain and ECR, the combinations of hydrogen content and calculated oxidation level (CP-ECR) that lead to embrittlement need to be determined.

In addition to oxygen pickup from the cladding outer surface, the cladding inner surface can pick up oxygen from the fuel-cladding bond and from fuel that may be adherent to this bond. This burnup-dependent phenomenon results in an oxygen-stabilized alpha layer on the inner surface and additional oxygen pickup by the beta layer.

For cladding that balloons and ruptures during the LOCA heating ramp, a third source of oxygen is available from the steam that enters through the rupture opening. Thus, within the localized balloon region (≈75–100 mm), an oxide layer will form on the cladding inner surface, and hydrogen pickup (secondary hydriding) will occur through this surface, especially near the neck regions of the balloon which experience delayed inner-surface oxide formation. The procedure outlined in this document provides a method to characterize the PQD of cladding as a function of initial hydrogen, which is present before the LOCA begins. This test procedure does not measure other effects, such as secondary hydriding, on the behavior of the ballooned region.

To characterize the PQD of cladding outside the balloon region, it is sufficient to perform tests using nonpressurized and nondeformed cladding samples. Such tests have been conducted using two-sided oxidation (EP-Ref. 1) and outer-surface-only oxidation (EP-Ref. 3) with cladding samples sectioned from as-fabricated Zircaloy (Zry-2, Zry-4), ZIRLO™, and M5, prehydrided Zry-4 (EP-Ref. 1, 3) and M5 (EP-Ref. 3), and high-burnup Zry-4, ZIRLO™, and M5 (EP-Ref. 1). The procedures used by ANL for conducting oxidation-quench testing and PQD testing are documented in EP-Ref. 1. The purposes of the procedures that follow are both to generalize the ANL methods to include a range of acceptable methods and to describe the methods in finer detail.

3. Sample Selection and Testing Frequency

3.1 Sample Selection

Although it is desirable to use samples representative of the fueled cladding that is loaded into the reactor, generally it is sufficient to select samples from finished cladding after polishing and cleaning processes. The one exception would be if postpolishing cleaning at the fuel fabrication facility includes etching with a hydrofluoric (HF)-containing acid mixture. If this is the case, then this step should also be

used on cladding before sample selection, as such treatment can result in early embrittlement even at temperatures as high as 1,200 ºC.

3.2 Frequency of Testing

Unlike breakaway-oxidation embrittlement, high-temperature embrittlement is relatively insensitive to trace elements and minor variations in alloy composition (within established ranges) and surface finishing. For cladding materials fabricated within specifications, PQD testing should be performed once for a particular cladding material and does not have to be repeated at a specified frequency. The effects of hydrogen on PQD are far more detrimental to cladding ductility than are minor changes to fabrication processes.

4. Sample Preparation and Characterization

4.1 Hydrogen Content Determination for As-Fabricated and Prehydrided Samples

The hydrogen content of as-fabricated cladding is expected to be low (5–15 wppm) and to be available from the tubing vendor. If it is not available, it should be measured. For prehydrided cladding used to simulate high-burnup effects, measurement of hydrogen content from locations close to sample locations is essential. Most techniques for prehydriding cladding are company-proprietary. However, based on reported results, the methods used result in samples with relatively uniform (<10% variation) hydrogen concentration along the axis and circumference of the sample. As shown in EP-Ref. 4, the pretest radial distribution of hydrogen is relatively unimportant as hydrogen homogenizes across the cladding wall very quickly for temperatures ≥900 ºC.

There are several ways to measure hydrogen content in metals. Vacuum fusion is one method. American Society for Testing and Materials (ASTM) E1447, "Standard Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity Method" (EP-Ref. 5), documents the recommended method. This method has been used successfully to determine the hydrogen content in other metals such as Zr alloys. EP-Ref. 6 documents the detailed procedure used to generate the results in EP-Ref. 1.

Along with the instrumentation needed (e.g., LECO RH-404 hydrogen determinator), calibration coupons are available from the vendor. These titanium coupons have hydrogen contents traceable to National Institute of Standards and Technology (NIST) standards. Titanium coupons with 218 wppm are recommended for calibration. As these machines are very sensitive, it is important to perform the calibration at least once in any given day before data generation. For measurements of hydrogen content that appear to be inconsistent, postmeasurement calibration verification should be performed using standard coupons as unknown samples. If low hydrogen content (e.g., <140 wppm) is measured for a cladding sample, then vendor standard coupons with 24 to 71 wppm may be tested as unknown samples to determine the error in extrapolating to lower hydrogen contents. Also, standard titanium coupons are available directly from NIST. In addition to the NIST titanium standard with about 215-wppm hydrogen, NIST also provides a standard with 114-wppm hydrogen.

4.2 Minimum Sample Lengths for One- and Two-Sided Oxidation Tests

The minimum sample length for two-sided oxidation samples should be 25 mm. This length was used for the oxidation-quench phase of the testing reported in EP-Ref. 1. Part of the reason for using this relatively short sample length was the limited supply of high-burnup cladding available for use in the ANL program. A length of 30 mm would have been more convenient, as it would have allowed three 8-mm-long ring-compression samples and a few postoxidation hydrogen samples to be sectioned from a

single oxidation-quench sample. Although there is no maximum limit prescribed, the two-sided oxidation sample should be no longer than the length of the uniform temperature region of the furnace. "Uniform" is defined as less than or equal to ±10 ºC variation at the target temperature.

In preparing samples for one-sided oxidation tests, welded end-caps have been used to prevent steam from coming into contact with the cladding inner surface. To minimize larger end effects resulting from the heat-affected zones and possible hydrogen diffusion from the sample to the end-caps, the minimum sample length for one-sided oxidation tests should be 75 mm. For prehydrided samples, axial distribution of hydrogen before end-cap welding and after oxidation testing should be measured to determine how much of the 75 mm is available for PQD testing. Also, samples should be evacuated before end-cap welding. An alternative approach is to introduce the flow of high-purity inert gas inside the cladding. It is important to minimize impurities (e.g., nitrogen) and gas pressure (less than or equal to steam pressure) acting on the inner surface of the cladding.

4.3 End-Cap Mass and Welding Procedure for One-Sided Oxidation Samples

Standard procedures are available for circumferential welding of end-caps to cladding samples. Because the welds and end-caps are not subjected to pressure, the end-caps should be small and the masses should be minimized as they serve as sinks for hydrogen.

4.4 Length, Outer Diameter, and Wall Thickness Measurements

Outer diameter and wall thickness vary somewhat along the length of fuel rod cladding. They should be measured and recorded for each sample. For cladding with a nominal diameter of 9.50 mm, the actual diameter of the sample can vary from 9.46 to 9.50 mm. The outer diameter should be determined to two decimal places (in mm) based on the average of the maximum and minimum diameters. For cladding with a nominal wall thickness of 0.57 mm, the actual wall thickness can vary from about 0.56 to 0.60 mm. Wall thickness should be determined for each sample to two decimal places (in mm) based on four readings at locations ≈90º apart. The sample length should be measured and recorded to one decimal place accuracy (e.g., 25.1 mm). Also, the ends of the sample should be polished to remove burrs before the sample length measurement. The ends of the sample should be relatively flush (90±5º relative to longitudinal axis). Outer diameter, wall thickness, and length are used to normalize sample weight gain to exposed surface area. The average wall thickness is used to calculate the CP-ECR.

4.5 Pretest Cleaning with Chemical Detergent or Organic Solvent and Rinsing

Appendix X1 ("Guide to Specimen Preparation") to ASTM G2/G2M–06, "Standard Test Method for Corrosion Testing of Products of Zirconium, Hafnium, and Their Alloys in Water at 680 °F [360 °C] or in Steam at 750 °F [400 °C]," describes sample cleaning procedures in Section X1.2 (EP-Ref. 7). These procedures should be followed for oxidation-quench test sample preparation. Specifications and requirements in Sections X1.1 ("Tubes with a Second Material on Inner Diameter") and X1.3 ("Etching") should be ignored. Based on EP-Ref. 1 and subsequent work at ANL, samples should not be etched with an HF-containing acid mixture as part of the test cleaning process. However, ANL has demonstrated that polishing of about 1 micron from the etched surface is sufficient to negate the possible negative effects of HF. Following cleaning, direct contact with the sample should be avoided by using surgical gloves for handling.

4.6 Pretest Sample Weight Measurement (after Drying)

Pretest sample weight should be measured to the nearest 0.1 milligram (mg) as specified in 7.1.3 of EP-Ref. 7. As drying after cleaning may take several hours, it is also permissible to measure pretest

sample weight after cleaning with an organic solvent such as ethanol that vaporizes rather quickly. The pretest weight is used in the determination of sample weight gain. Although measured weight gain is not used to determine oxidation level (i.e., ECR) for these tests, it is used as a partial validation of the reported isothermal oxidation temperatures and a check on steamflow conditions.

5. Temperature Heatup and Cooldown Rates and Heating Methods

5.1 Temperature Heatup and Cooldown Rates

For a given oxidation level and hydrogen content, the heating rate to 1,200 ºC is a critical parameter for samples that embrittle after short test times; weight gain accumulated at lower temperatures results in lower beta-layer oxygen content at the end of the heating phase and higher ductility. For samples with as-fabricated levels of hydrogen, test times at 1,200 ºC leading to embrittlement are in the range of 300–500 seconds for cladding with a thickness 0.57–0.67 mm that is exposed to two-sided oxidation. Embrittlement times at 1,200 ºC for one-sided oxidation tests with as-fabricated cladding samples are considerably longer than 500 seconds. As such, the embrittlement oxidation level is less sensitive to heating rate for as-fabricated cladding than it is for prehydrided cladding. The heating rate from 300 ºC to 1,000 ºC should be relatively fast (>20 ºC/s or <35 seconds to reach 1,000 ºC), and the heating rate from 1,000 ºC to 1,200 ºC should be >2ºC/s (<100-second duration). Use of slower heating rates that lead to higher embrittlement oxidation levels should be justified. The cooling rate from 1,200 ºC to the quench temperature (i.e., the wetting temperature at which very rapid cooling occurs) may be important, but it is less critical than the heating rate. The cooling rate to the quench temperature should be >2 ºC/s (e.g., <200 seconds from 1,200 ºC to 800 ºC). Use of slower cooling rates should be justified. The recommended quench temperature is 800 ºC. Use of lower quench temperatures should be justified. Based on results presented in EP-Ref. 1, no difference in ductile-to-brittle transition was observed for as-fabricated cladding materials cooled with or without quench. For prehydrided Zry-4, no significant difference was found in ductility for samples quenched at 800 ºC, 700 ºC, and 600 ºC, as all samples were brittle. However, prehydrided samples at the same hydrogen content and CP-ECR were ductile following cooling without quench. Temperature overshoot during the heating phase can have a significant effect on embrittlement oxidation level for prehydrided samples. As such, temperature overshoot should be limited to ≤20 ºC for ≤20 seconds.

As the target oxidation temperature is decreased from 1,200 ºC to 1,100 ºC to 1,000 ºC, the embrittlement oxidation level becomes less sensitive to heating rate. However, to standardize heating rates for these lower temperatures, the heating rate to within 100 ºC of the target hold temperature should be >20 ºC/s, and the heating rate from that temperature (e.g., 1,000 ºC or 900 ºC) to the hold temperature should be >2 ºC/s. Similarly, the cooling rate to the quench temperature (800 ºC) should be >2 ºC/s. The use of lower heating rates, cooling rates, and quench temperatures should be justified. However, the use of higher cooling rates to the quench temperature and higher quench temperatures does not have to be justified as these conditions will lead to embrittlement at lower oxidation levels.

For "uncontrolled" cooling rates (e.g., those resulting from furnace and sample cooling alone), the cooling rate will decrease with cooling time. The rates listed above refer to average values determined from $\Delta T/\Delta t$. For controlled cooling rates (e.g., by means of a thermocouple (TC) welded to the sample with feedback to furnace power), constant cooling rates may be achieved. For a postulated LOCA transient temperature history, the cooling rate increases from the maximum temperature to the wetting temperature. Although this cooling history is difficult to simulate experimentally, a constant cooling rate comes closer than one that decreases with time.

5.2 Radiant Heating

Radiant heating in a quad-elliptic furnace has been used to generate the PQD data reported in EP-Ref. 1. This heating method, along with furnace power controlled by feedback from a TC on or near the sample, allows for controlled heating rates and relatively fast cooling rates (>10 ºC/s or <40 seconds for cooling from 1,200 ºC to 800 ºC). For 25-mm-long samples, axial temperature variations are negligible, but circumferential temperature variations are in the range of 10–20 ºC for cladding outer diameters ranging from 9.50 to 11.0 mm. These variations can be reduced by using radiant-heating furnaces with more than four lamps. With proper thermal benchmarking, radiant-heating furnaces are acceptable for generating PQD specimens for ductility determination.

5.3 Resistance Heating

Resistance heating has been used to generate the results reported in EP-Ref. 3. As compared to radiant-heating furnaces, these furnaces are characterized as having a larger uniform temperature zone and as having very slow heating and cooling rates. Controlled movement of the sample into and out of the furnace achieves faster heating and cooling rates. Benchmark tests should be performed to determine the heating and cooling rates. With proper thermal benchmarking, resistance-heating furnaces are acceptable for generating PQD specimens for ductility determination.

5.4 Induction Heating

Induction heating has the advantage of rapid sample heating and cooling rates. The CINOG program in France (EP-Ref. 8) has used it to generate weight-gain kinetics data for Zry-4, M5, and developmental alloys. Although the data from these tests appear reliable, reported weight gains for Zry-4 are about 10–12% lower than those predicted using the CP correlation and are in better agreement with the weight-gain correlation (LS) derived by Leistikow and Schanz (EP-Ref. 9) using data from resistance heating. However, this should not be an important factor in determining the embrittlement oxidation level as calculated with the CP weight-gain correlation as long as the oxidation temperatures are accurate. These temperatures are determined using optical pyrometry. With proper thermal benchmarking, induction-heating furnaces might be acceptable for generating PQD samples. However, as it is not clear how to do the benchmarking, the use of induction heating is not recommended for preparing PQD samples.

5.5 Direct Electrical Heating

Direct electrical heating of cladding has been used in the past for studies relevant to LOCAs. Because resistance and heating rate change with temperature, direct electrical heating of cladding is not recommended for preparing PQD samples. However, "indirect" electrical heating may be an acceptable method for internal heating of another material inside the cladding to generate a heat flux simulating heating of the cladding by means of decay heat from the fuel.

6. Temperature Control and Monitoring

6.1 Thermocouples

For oxidation temperatures ≤1,200 °C, Type S (Pt/10%Rh-Pt) TCs should be used to record temperature and control furnace power. The TCs should be calibrated using instrumentation and standards that are traceable to NIST. Generally, this service is provided by the TC vendor, who, for an extra fee, provides a certificate of calibration. Every TC used to measure sample temperature either directly or indirectly should have a certificate of calibration showing the results of the calibration at three

temperatures: 1,200 ºC, 1,100 ºC, and 1,000 ºC. Copies of these certificates should accompany the data report. Verification should be provided demonstrating that the vendor actually did the calibration according to the standards in internationally recognized standards organizations, such as the International Organization for Standardization (ISO), and American National Standards Institute/National Conference of Standards Laboratories (ANSI/NCSL).

6.2 Thermal Benchmarks

For short (e.g., 25–30 mm) two-sided oxidation samples, direct welding of TCs onto the sample outer surface is not recommended for data-generating tests. The interaction between the TC and the cladding metal causes a local flaw. Also, it is difficult to get an accurate posttest weight measurement after removing the welded TCs. Although measured weight gain is not used to determine the oxidation level (CP-ECR), it is used to check that the target temperature and hold time at that temperature are achieved.

For longer two-sided and one-sided oxidation samples, TCs may be welded near the sample ends for data-generating tests. For one-sided samples, in particular, the TC may be welded to the cladding outer surface in the heat-affected zone.

In most cases, the control TC will be welded onto the sample holder or as close to the sample as possible without contacting the sample. This requires thermal benchmarks to be performed to establish the relationship between the control TC that will be used during data-generating tests and the temperature of the sample outer surface. Generally, the control TC will experience slower heating and cooling rates than the sample. The thermal benchmarking should be performed at three sample temperatures: 1,200 ºC, 1,100 ºC, and 1,000 ºC. An important phase of the benchmarking is to determine the control TC temperature at which quench water should be introduced to rapidly cool the sample at the prescribed temperature. For the work reported in EP-Ref. 1, two to three TCs (120º apart) were welded directly onto the benchmark sample outer surface. These readings were compared to the readings of three TCs welded onto the sample holder at a location just above the sample. For radiant heating and large-diameter (≈11-mm) cladding, three TCs were welded directly to the cladding outer surface to better define the average and one-standard-deviation cladding temperature. For smaller diameter cladding (9.50 mm), only two TCs welded directly to the cladding surface were needed. It is important that the thermal benchmark tests be conducted under the same flowing steam conditions as used in the data-generating tests.

For resistance-heating furnaces, thermal-benchmarking methods similar to the ones described for radiant-heating furnaces can be used. However, other methods commonly used (e.g., suspended and movable TC) may not be adequate for characterizing the heating rate of the sample. Samples with low thermal mass and high initial heats of oxidation, exposed to low steamflow rates, may heat up much faster than more massive sample holders. The results of the thermal benchmark tests should be documented and included in the data report.

For irradiated high-burnup material which has developed a corrosion layer, it is expected that pretest cladding corrosion would slow the initial oxidation rate and the heating rate associated with the exothermic oxidation reaction. The presence of the corrosion layer could affect the peak temperature reached during the very rapid heating ramp. The work reported in EP-Ref. 1 with out-of-cell tests confirmed this effect. The following procedure was used in the work reported in EP-Ref. 1 and could be used to validate a thermal benchmark for irradiated material which has developed a corrosion layer:

a. With TCs welded onto bare as-fabricated cladding, conduct the thermal benchmark test for a hold time selected to grow an oxide layer relevant to the irradiated material to be tested.

b. Cool to 300 ºC and repeat the thermal benchmark test using the same controller parameters as were used in (a); compare the two sets of results with emphasis on the maximum temperature at the end of the rapid temperature rise (first peak), the time to reach the hold temperature, and the hold temperature.

c. If necessary, increase the holder control temperature to achieve the desired hold temperature for cladding with pretransient oxide layers.

6.3 Weight-Gain Benchmarks

Following thermal benchmarking, samples should be tested without TCs welded onto the sample to determine the weight gain. These tests should be conducted at 1,200 ºC, 1,100 ºC, and 1,000 ºC for a test time corresponding to 10% CP-ECR. For all cladding materials tested in the ANL program (EP-Ref. 1), weight gains were comparable to each other and to the CP-correlation predictions at oxidation temperatures of 1,200 ºC and 1,100 ºC. If the measured weight gain for these oxidation temperatures differed from the CP-predicted weight gain by ≥10%, then data-generating tests were not initiated until the root cause of the problem was found and corrected (EP-Ref. 1). Generally, this occurred only when the TCs used for the thermal benchmarking read 15 to 20 ºC higher or lower than the actual sample temperature, even though the vendor had certified the TCs. As weight gain may depend somewhat on heating method, the weight gain for a particular cladding material should deviate by less than 10% from the vendor-established database for that material before the start of data-generating tests. For Zr-lined Zry-2 and alloys of Zr-1 and niobium, the measured weight gain at 1,000 ºC is considerably lower than the CP-predicted weight gain.[4] For these materials, the results of the weight-gain benchmark should be compared to the published or vendor-proprietary, material-specific databases.

The weight-gain benchmarks are designed as a supplement to the thermal benchmarks to ensure adequate TC readings and adequate steamflow. The results of the weight-gain benchmark tests should be documented and included in the data report.

For irradiated high-burn material that has developed a corrosion layer, the corrosion layer may affect the oxidation kinetics, and it should be confirmed that the weight gain of the irradiated material is comparable to the CP correlation. For the testing reported in EP-Ref. 1, which included oxidation tests with high-burnup cladding, the measured oxide layer thickness and the weight gain determined from layer thicknesses agreed quite well with CP-predicted values for as-fabricated and high-burnup cladding. To validate that, for irradiated material which has developed a corrosion layer, the CP correlation accurately predicts weight gain, mass and metallographic examination can be used to determine that the measured weight gain and the measured oxide layer thicknesses are in good agreement with CP-predicted values. A test with the hold time chosen to give a weight gain equivalent to 10% CP-ECR is recommended. If the out-of-cell and in-cell weight gains and converted ECR values are in good agreement, then the use of the test train in the in-cell furnace can be considered validated, and the weight gain can be used as a supplement to the thermal benchmarks to ensure adequate TC readings and adequate steamflow.

[4] Even when the measured weight gain at 1,000 ºC was considerably lower than the CP-predicted weight gain for a particular alloy, the CP correlation accurately predicted the embrittlement of cladding alloys tested. It is important to clarify that the loss of cladding ductility is the result of oxygen diffusion into the base metal and is not directly related to the growth of a Zr dioxide layer on the outside cladding diameter (i.e., weight gain). A limit correlated with peak local oxidation, as calculated by the CP correlation, is used as a surrogate to limit integrated time at temperature and associated oxygen diffusion. This surrogate approach is possible because both CP-calculated oxidation and diffusion share similar temperature dependences.

7. Water Quality, Steamflow Rate, and Steam Pressure

7.1 Water Quality

Purified water should be used for generating steam. Grade A water with ≤45 parts per billion (ppb) oxygen should be used for corrosion tests in pressurized water and steam (EP-Ref. 7). Laboratory-grade Type I (distilled and/or deionized) water is also of sufficient purity for oxidation tests at ≥1,000 ºC. ASTM, the National Committee for Clinical Laboratory Standards (now Clinical and Laboratory Standards Institute), and ISO 3696, "Water for Analytical Laboratory Use—Specification and Test Methods," have similar definitions for Type I purified water.

7.2 Steamflow Rate

The average steamflow rate used to oxidize PQD samples should be determined (and reported) from the mass of condensed water collected during the test, or by the mass of water that is input to the steam chamber, divided by the test time, and normalized to the net cross-sectional area of the steam chamber. The average steamflow rate should be in the range of 0.8 to 30 mg/square centimeter (cm^2) s). Justification for this range is provided in the following.

Leistikow and Schanz (EP-Ref. 9) and Uetsuka (EP-Ref. 10) studied the effects of low steamflow rates on the oxidation kinetics of Zry-4 at 1,000 ºC. Figure 9 of EP-Ref. 9 summarizes their results. In terms of flow rate normalized to the cross-sectional area of the steam chamber, the oxidation kinetics began to decrease due to steam starvation for flow rates <0.05 mg/(cm^2 s). For the EP-Ref. 9 work, the sample length was 30 mm and oxidation was two-sided. Aomi, et al. (EP-Ref. 11) studied the relationship between weight gain and steamflow rate for oxidation temperatures up to 1,200 ºC. They found that the weight gain for fixed test times and temperatures was independent of steamflow rates in the range of 0.8 to 7.8 mg/(cm^2 s). Kawasaki, et al. (EP-Ref. 12) also performed high-temperature oxidation tests to determine the range of steamflow rates for which the weight gain for a given test time was independent of steamflow rate. They report this range as 3 to 28 mg/(cm^2 s).

Although EP-Ref. 11 and EP-Ref. 12 give maximum steamflow rates of 7.8 mg/(cm^2 s)and 28 mg/(cm^2 s), it is not clear why higher steamflow rates would have an effect on weight gain and oxidation kinetics. It is desirable to have a steamflow rate higher than 0.8 mg/(cm^2 s) to reduce temperature overshoot during the heating phase for bare cladding. Although the maximum steamflow rate may not be as critical as the minimum steamflow rate, it should be limited to ≤30 mg/(cm^2 s). The use of steamflow rates >30 mg/(cm^2 s) should be justified.

7.3 Steam Pressure

Oxidation tests for preparation of PQD samples should be conducted at a steam pressure at or slightly above atmospheric pressure. This is consistent with steam pressures used in previous studies (e.g., EP-Refs. 1–3).

8. Procedure for Oxidation and Quench Tests

The specific details of the test procedure depend on the heating furnace used. Listed below are the steps used in EP-Ref. 1, along with some generalizations that would apply to other heating and cooling methods (e.g., those used in EP-Ref. 3).

8.1 Test Train and Steam Chamber

The test train or sample holder and the steam chamber form a unit that should be designed to contain the steamflow and to prevent impurities, especially nitrogen, from entering the chamber. By using steam that has a pressure slightly greater than the surrounding atmosphere, the test train/steam chamber does not have to be highly "leaktight" to provide a pathway for steamflow and protect the sample from gas-phase impurities.

In choosing the material for the test train or sample holder, it is desirable to have a nonoxidizing or limited-oxidizing material such as stainless steels or nickel (Ni) alloys (e.g., Inconel 600). Particular attention should be given to direct contact of the sample with materials such as iron (Fe) and Ni alloys, because of the low-temperature eutectics for Zr and these elements. Eutectic reactions between Zr-based alloys and test train materials must be prevented. Hofmann and Markiewicz (EP-Ref. 13) studied the reaction rates and eutectics of Zry-4 and Inconel-718. They also present binary phase diagrams for Zr-Fe and Zr-Ni, which have eutectic temperatures as low as ≈930 ºC and 980 ºC, respectively. In EP-Ref. 1, alumina inserts and zirconia washers were used between the Inconel holder and the sample to prevent such reactions from occurring. Testing laboratories may institute controls other than those used in EP-Ref. 1 to prevent eutectic reactions between Zr-based alloys and the test train materials.

8.2 Purging Steam Chamber and Stabilizing Steamflow

Before heating and steamflow initiation, the steam chamber is filled with gas representative of the environment of the test facility (usually air). The test chamber may be purged with a high-purity inert gas (e.g., argon) before the start of steamflow, or it may be purged with low-temperature steam before the temperature ramp. If steam is used to purge the steam chamber, then steamflow should be maintained for 500 seconds before the temperature ramp.

Steamflow should be initiated at a test chamber temperature of ≈30 ºC. Following introduction of steam into the chamber, furnace heating should commence for a pretest hold temperature of 300 ºC. Stabilization of steamflow and 300 ºC sample temperature should occur within 500 seconds.

Deviations from this procedure may be pursued but should be justified. Deviations that may have a significant effect on test results include heating the sample to the target temperature in an inert gas before the introduction of steamflow. Impurities in the inert gas will result in an oxide or oxide-nitride film on the cladding that is not relevant to the LOCA. Also, the heat of oxidation would be very high for such a scenario, leading to significant temperature overshoot.

8.3 Ramping Temperature and Holding Temperature at Target Value

The target test temperature is predetermined. It should be based on the average sample temperature. Depending on the heating method used, axial and circumferential variations could be significant. For a single sample, the axial temperature variation should be ≤10 ºC, and the circumferential temperature variation should be ≤20 ºC.

For resistance furnaces, the sample heating rate is controlled by the rate of movement of the sample into the furnace heating zone. For radiant-heating furnaces, the heating rate is controlled through feedback from a TC welded onto the holder to the furnace power. For the radiant heating used in EP-Ref. 1, the temperature ramp rate for as-fabricated cladding materials was programmed to be very fast (>50 ºC/s) from 300 ºC to within 50–100ºC of the target temperature and slow (2 to 3 ºC/s) from that temperature to the target temperature. This programmed ramp was designed to eliminate temperature

overshoot. In later studies with prehydrided cladding and high-burnup cladding, the 1,200 ºC tests were conducted with rapid heating to 1,000 ºC followed by slower heating (2 to 3 ºC/s) to 1,200 ºC.

8.4 End of Heating Phase and Cooldown

After the target test time has been reached, furnace power should be turned off or decreased in a controlled manner while steamflow is maintained. The rate of temperature decrease will depend on the heating method used and the method of removing the sample from the furnace. For in situ cooling, the steamflow should be maintained until the sample temperature reaches 800 ºC. For the EP-Ref. 1 work, this corresponded to a holder temperature of 700–720 ºC. Following this step, there was ample moisture in the steam chamber during the very brief period between steamflow and quench-water flow.

8.5 Determination of Equivalent Cladding Reacted

The CP-ECR is calculated to determine test time. It should be calculated by integration of the CP weight-gain rate correlation with respect to test time. Equations 5 and 6 from EP-Ref. 1 are repeated below for conversion of CP weight gain (Wg in grams (g)/cm^2) to oxidation level (ECR in percent):

One-sided oxidation $\quad ECR = 43.9\ [(Wg/h)/(1 - h/Do)], \quad (1)$

Two-sided oxidation $\quad ECR = 87.8\ Wg/h, \quad (2)$

where h is cladding thickness in cm, and Do is the cladding outer diameter in cm.

9. Post-Oxidation-Quench Measurements and Characterization

9.1 Sample Drying Time

To determine an accurate posttest sample weight, it is important that the sample be free of moisture. For drying in stagnant air, the drying time should be 2 hours or more. This time can be reduced significantly by the use of forced-air drying. The sample weight will continue to decrease during the drying process until it reaches a minimum and holds at that minimum. Whatever drying method is used, the drying time should be verified by weight measurements.

9.2 Weight Measurement and Use of Weight Gain To Verify Oxidation Temperature

The posttest sample weight should be measured to the nearest 0.1 mg as specified in 7.1.3 of EP-Ref. 7. The weight gain (in mg) is determined by subtracting the pretest weight from the posttest weight and normalizing this value to the steam-exposed surface area of the sample. Although this normalized weight gain is not used to determine the oxidation level, it is used to validate temperature control and monitoring, as well as adequacy of steamflow and test procedures throughout the data-generating phase of testing.

9.3 Hydrogen Content Measurement

If it has been demonstrated and documented that prehydrided samples have very little axial variation in hydrogen content, then posttest hydrogen analysis would not be needed. Significant axial variation is defined as >30 wppm along the test sample length. For such samples, posttest hydrogen analyses could be performed using rings 2–3 mm in length, sectioned from both sides of the 8-mm-long ring-compression sample. Alternatively, posttest hydrogen analysis could be performed using the

8-mm-long rings after RCT. In either case, posttest hydrogen values should be corrected for weight gain so that the reference weight for hydrogen content is the pretest weight. Hydrogen pickup during the oxidation-quench phase is expected to be small (<20 wppm), based on the results presented in EP-Ref. 1, as long as breakaway oxidation does not occur.

10. Matrix for Oxidation and Quench Tests

10.1 As-Fabricated Cladding

Based on the results presented in EP-Ref. 1, embrittlement of as-fabricated cladding with very low hydrogen content (e.g., 5–15 wppm) is not expected to occur at oxidation temperatures of 1,100 ºC and 1,000 ºC for oxidation levels up to 20% CP-ECR. The reason for this is the relatively low oxygen solubility limit in Zr-based cladding alloys at these temperatures. Even after the beta layer is saturated with oxygen, it remains ductile. Further oxidation simply increases the oxide and oxygen-stabilized alpha layer thickness values and reduces the beta layer thickness. Strength (i.e., maximum load at failure) continues to decrease, but ductility remains essentially constant until significant beta-layer thinning occurs at >20% CP-ECR.

At an oxidation temperature of 1,200 ºC, the oxygen solubility limit (e.g., 0.6 wt. % for Zry-4) in Zr-based cladding alloys is close to the embrittlement limit at a ring-compression test temperature of 135 ºC. Cladding materials experience a significant decrease in ductility (from >40% to <10%) in the oxidation range of 10% to 17% CP-ECR, following oxidation at 1,200 ºC. Thus, it is recommended that scoping tests be performed at oxidation levels of 10%, 13%, 17%, and 20% CP-ECR. For each oxidation sample ≥30 mm long, at least three ring-compression samples can be sectioned. Based on these results, additional tests can be performed in a narrow CP-ECR range. If the cladding is ductile at 17% and brittle at 20%, then multiple tests should be performed at 18% and 19% CP-ECR to determine the ductile-to-brittle transition CP-ECR. Three oxidation-quench tests are recommended at each intermediate CP-ECR values to give nine PQD data points at each oxidation level. This would be sufficient to determine the ductile-to-brittle transition to the nearest percent CP-ECR.

10.2 Prehydrided Cladding

For tests with prehydrided cladding, the hydrogen contents selected should be in a range relevant to the cladding material. It has been a common practice to rely on data concerning the thickness of the corrosion layer, for which there is much data as a function of axial position and burnup, and a hydrogen pickup fraction to determine hydrogen content in the cladding. However, this approach is not reliable because cladding hydrogen absorption and distribution vary with alloy composition, cladding heat treatment, cladding temperature distribution, proximity of cladding to dissimilar metal (shadow corrosion under non-Zr grid cage components), corrosion layer thickness, axial location, burnup, and plant chemistry. Also, the hydrogen measured in hot cells for defueled cladding samples includes the hydrogen in the corrosion layer and the hydrogen in the cladding metal. In expressing it in units of wppm, the total weight of the sample (corrosion layer, metal, fuel-cladding bond, and both fission products and actinides within and adherent to the bond) is used. This practice may be relatively accurate for low-burnup cladding with thin corrosion layers and no fuel-cladding bond. However, the hydrogen in the cladding metal may be lower or higher than what is reported for intermediate- and high-burnup cladding. Only the hydrogen in the metal contributes to embrittlement. Hydrogen levels used in PQD testing with prehydrided cladding materials should cover the anticipated range of hydrogen in the metal of irradiated cladding.

For samples to be oxidized at ≤1,200 ºC (i.e., >2 ºC/s heating rate from 1,000 ºC to the 1,200 ºC hold temperature), the ductile-to-brittle transition oxidation level is highly dependent on the hydrogen

content. For this oxidation temperature, the embrittlement threshold provided in DG-1263 (Ref. 6) as a function of hydrogen content may be used as a guide in selecting the range of oxidation levels to be included in the test matrix. Table 1 below provides the embrittlement threshold in DG-1263 in tabular form for clarity. For low hydrogen contents (<150 wppm) typical of those measured for high-burnup M5, the results presented in EP-Ref. 3 may be used as a guide. EP-Ref. 3 also presents PQD data for prehydrided M5 and Zry-4 oxidized at lower test temperatures. For a specific hydrogen content (e.g., 300 wppm), the first test should be conducted at the CP-ECR determined from EP-Ref. 1 embrittlement data (e.g., 9% CP-ECR for 300 wppm hydrogen). Depending on the results, the second test should be conducted at a CP-ECR 2% higher (if ductile at 9%) or lower (if brittle at 9%). Assuming that ductile and brittle oxidation levels have been found, then three tests should be conducted at the intermediate CP-ECR to confirm the embrittlement threshold.

Table 1. Embrittlement Threshold

Hydrogen Content (wppm)	Embrittlement ECR
10	18%
100	15%
200	12%
300	9%
400	6%
500	5%
600	4%

Unlike as-fabricated cladding, prehydrided cladding oxidized at 1,100 °C and 1,000 °C will embrittle at ECR values significantly below 17%. This is because of the hydrogen-induced increase in oxygen diffusion rate and in oxygen solubility in the beta phase, as well as some intrinsic hydrogen embrittlement. Testing prehydrided cladding at temperatures lower than 1,204 °C is important if an applicant can demonstrate that calculated LOCA temperatures are significantly lower than 1,204 °C.

11. Procedure for Conducting Ring-Compression Postquench Ductility Tests

11.1 Pretest Activities

The materials test system (MTS) used to conduct ring-compression tests should be subjected to an annual verification of calibration with regard to measurement of compressive loads by the load cell, the determination of crosshead displacement, and the determination of crosshead speed. The calibration should be performed to NIST-traceable standards. This service is offered by the vendor (e.g., Instron), who provides documentation of calibration verification.

The TC or TCs used to control furnace or oven power corresponding to a ring test temperature of 135 ºC should be calibrated to an NIST-traceable standard. The TC vendor provides this service for a fee and supplies a certificate of calibration along with the TC. The calibration should be performed at 135 ºC. A variety of TCs could be used at this low temperature. Type K (chromel-alumel) TCs are recommended. The standard deviation between the TC reading and the NIST-traceable standard is quite low (e.g., ±0.3 °C for room temperature (RT) to 200 ºC).

In addition to the annual verification of calibration, it is recommended that six ring-compression tests be performed using as-fabricated cladding to determine the relationship between offset and permanent displacements: three tests at RT and three tests at 135 ºC. Appendix A gives the results of six

tests conducted with as-fabricated ZIRLO™. This specific verification of calibration is also used to determine if the measured loads are reasonable.

Rings sectioned from LOCA oxidation-quench samples should be in the range of 7–10 mm long and should not include oxidized ends (two-sided samples) or weld-heat-affected zones (one-sided samples). The reference length for ANL tests was 8 mm. For two-sided oxidation samples, it is sufficient to cut off 1–2 mm from the ends of the oxidation samples. The ends of the sectioned rings should be deburred, and the samples should be cleaned in a chemical detergent or organic solvent following deburring.

Following sectioning, the length of the rings should be measured to one decimal place (e.g., 7.9 mm), and the minimum and maximum diameter of the oxidized rings should be measured to two decimal places (e.g., 9.51 mm). As the ring should be positioned such that the minimum diameter aligns with the loading direction, only the minimum diameter is used in the calculation of permanent displacement and strain. Micrometers used to measure length and diameter should be calibrated to an NIST-traceable standard.

11.2 Test Temperature and Crosshead Displacement Rate

It is recommended that an oven, rather than a furnace, be used to heat the test ring to 135 ºC. For such uniform heating, it is sufficient to use a single TC in contact with the inner surface of the sample at the bottom support position. The spring-loading of the TC also serves to fix the location of the ring relative to the top loading rod. Tests in such a heating device should be conducted at a test temperature of 135 ±1 ºC. The PQD test results in EP-Ref. 1 for as-fabricated and prehydrided LOCA samples used oven heating for the tabletop Instron Model 5566 MTS, along with a single TC strapped to the bottom inner surface of the ring.

It is more common that the MTS would be equipped with a clamshell radiant-heating furnace provided by the vendor. Such furnaces are known to result in circumferential temperature gradients for rings because of the relationship between the ring location and the focal point of the furnace. For such furnaces, the bottom TC, which is in intimate contact with the sample, should be used to control furnace power to achieve a steady temperature of 135 ºC. Additional TCs at the 3 and 9 o'clock positions, which initially contact the sample through spring loading, should be used to determine the circumferential variation in temperature. These TCs, which contact the sample outer surface with mild spring loading, are less accurate than the bottom TC. Tests should be initiated when the average deviation of the side TC readings is ≤5 ºC relative to the 135 ºC control TC reading. The use of test temperatures higher than 135 ºC requires justification, while test temperatures lower than 135 ºC do not require justification.

This heating and temperature monitoring method, along with an Instron Model 8511 servohydraulic MTS, was used to generate the EP-Ref. 1 results for high-burnup cladding LOCA samples. The results presented in Appendix A were generated with the Instron 8511.

The crosshead displacement rate for ring-compression samples should be in the range of 0.083 to 0.033 mm/s (0.5 to 2 mm/minute). These rates are consistent with those used in past research (EP-Ref. 1, 3), and they are slow enough to allow test termination after the first significant load drop.

11.3 Test Conduct

The test should be conducted in the "displacement-controlled mode" rather than the "force-controlled mode." Software inputs include the constant displacement rate and the maximum displacement. The maximum displacement (i.e., crosshead travel) is important to protect the control TC

and the MTS itself. Because of the "bow-tie" shape of a highly deformed ring, the maximum displacement should be less than the inner diameter of the cladding minus the TC diameter. For standard 17×17 cladding with an inner diameter of about 8.3 mm, the maximum displacement should be <6 mm.

Test conduct is standard with regard to setup and operation. EP-Ref. 14 gives details for ring-compression tests conducted with the screw-type Instron Model 5566 used to generate data reported in EP-Ref. 1 for as-fabricated and prehydrided cladding.

11.4 Test Termination

The preferred method for ending the test is to release the compressive load as soon as there is a sharp load drop >30%. This is achieved by simply pushing the reset button. Given the slow displacement rate, there is ample time to terminate the test very shortly after the load drop is observed. Based on the experience reported in EP-Ref. 1, load drops in the range of 30–50% indicate a single through-wall crack, which may be very tight or loose because of recoil following test termination. For tight cracks, an accurate posttest diameter can be measured in the loading direction. For a single loose crack, the posttest diameter reading is not very accurate. For load drops of about 70–80%, the sample should have two cracks. For load drops of 80–100%, it is likely that the sample cracked into three or four pieces. Examples of load-displacement curves, offset strains, and permanent strains for oxidized and quenched cladding samples are provided in Appendix B.

The more common method used in RCT (e.g., EP-Ref. 3) is to run the test for a fixed displacement. As multiple cracks are likely to occur, no useful posttest diameter can be measured. Although this method is acceptable, it is not recommended, as the only data that can be obtained are the offset displacement and strain.

11.5 Posttest Measurements

After removing the compressed ring from the oven or furnace, cooling to RT occurs rather quickly. The sample should be examined visually to determine if cracking has occurred, the number of cracks, and the location of the cracks. For samples that are likely to have a single tight crack, the visual examination should include one at about 4X magnification to verify that the crack is through-wall (from examination of ring ends) and extends along the whole length of the sample (from examination of outer and inner surfaces).

If the test was terminated following a steep 30–40% load drop and visual examination indicates a single tight crack, then the outer diameter in the loading direction should be measured.

The offset displacement should be determined from the load-displacement curve using methods illustrated in Appendices B and C. In general, this means mathematically unloading the sample at the load just before the steep load drop. The linearized slope (i.e., ring stiffness in kilo Newton/mm) of the initial loading curve is used to do the mathematical unloading. For ductile rings that exhibit a gradual load drop with increasing displacement, the offset strain determination is dependent on the visual examination of the posttest ring. If the posttest sample has a through-wall crack, then the offset strain should be determined based on the location on the curve where the load has decreased by 50%. For samples that have no posttest cracks, the full load-displacement curve may be used to determine the offset displacement. These cases are not important in the determination of the ductile-to-brittle transition oxidation level, as they represent samples with very high ductility.

To convert offset and permanent displacement to strain, it is recommended that the outer diameter of the as-fabricated cladding be used to normalize these displacements. Prehydriding samples will result

in a small increase in the outer diameter and the wall thickness. Oxidation will result in additional increases in diameter and wall thickness. However, these increases have only a small effect on the calculated normalized displacements. It is recommended that strain be reported in percent as displacement divided by the diameter of the as-fabricated cladding used for oxidation or for prehydriding and oxidation. Based on measurement error and data scatter, these strains should be reported to one significant decimal place. If the postoxidation diameter in the loading direction is used to calculate permanent strain, it should be reported along with the displacements and the converted strains.

After determination of the offset and permanent strains, the compressed ring should be used to measure the postoxidation hydrogen content in the ring. This hydrogen content should be corrected for weight gain. The measurement should be performed if oxidation samples are expected to have >10% axial and circumferential variation in hydrogen content relative to the average hydrogen content.

12. Data Reporting and Assessment

12.1 Hydrogen Level, Test Temperature, Test Time, CP-ECR, Offset Strain, and Permanent Strain

Tabular results should include hydrogen level, test temperature, test time (from 300 °C to the quench time), CP-ECR, offset strain, and permanent strain. A footnote should clarify which diameter was used to determine strain from displacement.

Graphical results should include the load-displacement curves (including determination of offset strain) and summary graphs of offset strain versus CP-ECR and permanent strain versus CP-ECR.

12.2 Determination of Ductile-to-Brittle Transition CP-ECR

Rings that exhibit ≥1.0% permanent strain are classified as ductile. The 1.0% is based on uncertainties in diameter readings, in recoil (or spring-back) of cracked rings versus intact rings, and in diameter reduction due to flaking off of oxide. It is also based on trend curves of permanent strain versus CP-ECR. For samples that are clearly brittle, measured permanent strains are generally in the range of 0.2–0.8%, which for cladding with an outer diameter of 9.50 mm corresponds to permanent displacement of 0.2–0.8 mm. These displacements and strains are considered to be in the "noise" of uncertainty. The ductile-to-brittle transition CP-ECR is defined as the CP-ECR corresponding to 1.0% permanent strain (i.e., the maximum CP-ECR for which ductility is retained). For multiple data points at the same sample and test conditions, the average permanent strain should be calculated. The ductile-to-brittle CP-ECR should be based on average permanent strain ≥1.0%. As it is unlikely to measure exactly 1.0% permanent strain, the CP-ECR may be determined from interpolation between an oxidation level for which the permanent strain is >1.0% (ductile) and an oxidation level for which the permanent strain is <1.0%. These CP-ECR values should differ by no more than 2%. In this case, the transition should be identified to occur at the highest CP-ECR at which the permanent strain is ≥1.0%. The ductile-to-brittle transition oxidation level should be reported to the nearest percent. For example, if the sample is ductile at 8% CP-ECR and brittle at 10% CP-ECR and no further testing is conducted, the transition CP-ECR would be reported as 8%. However, it is recommended that three confirmation tests be conducted at 9% CP-ECR. Figure 2 shows an example from the work reported in EP-Ref. 1 for as-fabricated HBR-type 15×15 Zry-4. Based on multiple oxidation tests in a narrow range and multiple ring-compression samples, the permanent strains were 1.5±0.4% at 15.2% CP-ECR and 1.1±0.3% at 16% CP-ECR, where the ± values represent one standard deviation caused by data scatter from repeat tests. Based on linear extrapolation, the transition CP-ECR is calculated to be 16%.

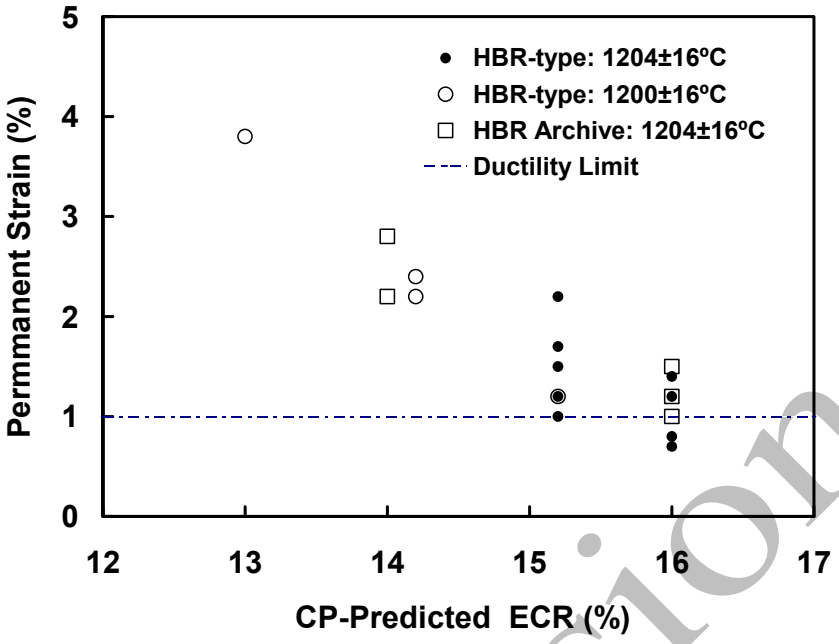

Figure 2. Determination of ductile-to-brittle transition CP-ECR based on the permanent strain criterion (≥1.0% implies ductility) for as-fabricated HBR-type 15×15 Zry-4 oxidized at ≈1,200 °C and quenched at 800 °C. Ring-compression tests were conducted at 2 mm/minute displacement rate and 135 °C ring temperature. The ductile-to-brittle transition oxidation level is 16% CP-ECR based on average permanent strain ≥1.0%.

If ring-compression tests are not interrupted at the first significant load drop, then the ring will crack into pieces, which renders measurement of posttest diameter impractical and unreliable. One must rely on offset displacement and strain to assess whether a ring is ductile or brittle. The method for determining the offset displacement has an inherent error because the unknown unloading slope will always be less than the loading slope. Appendix C summarizes the data reported in EP-Refs. 1 and 15, along with the HBR data presented in Figure 2, for rings sectioned from cladding alloys oxidized at 1,200 ºC. The trend curve shown in Figure C1 indicates that the error associated with offset strain displacement increases with calculated oxidation level (CP-ECR). This leads to the following ductility criterion based on offset strain:

$$\text{Average Measured Offset Strain} \geq 1.41 + 0.1082\ \text{CP-ECR} \qquad (3)$$

Equation 3 gives ≥2.0% at 5% CP-ECR and ≥3.6% at 20% CP-ECR. However, because of the large data scatter in Figure C1, the offset strain criterion given on the right side of Equation 3 represents the one-sigma upper bound of the data. This method is illustrated in the following example.

The offset strains corresponding to the permanent strain data shown in Figure 2 are plotted in Figure 3.

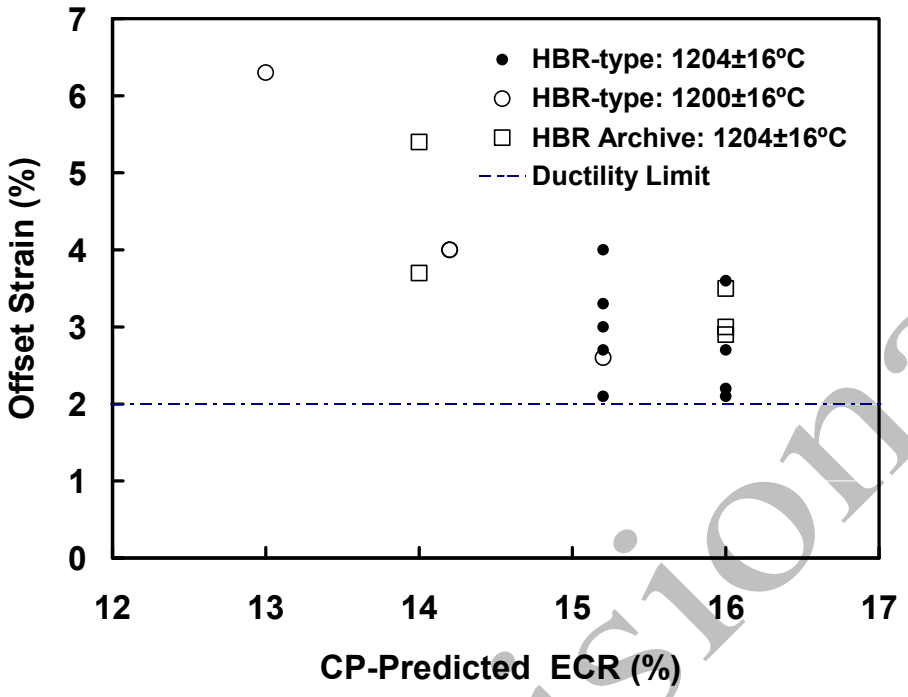

Figure 3. Offset strains determined for as-fabricated HBR-type 15×15 Zry-4 oxidized at ≈1,200 ºC and quenched at 800 ºC (see Figure 2 for corresponding permanent strains).

Unlike permanent strains, offset strains level off to about 3% at 15.2 and 16.0 CP-ECR (3.0±0.7% at 15.2% CP-ECR and 2.9±0.5% at 16.0% CP-ECR). Based on Equation 3, offset strains ≥3.1% imply ductility at 15–16% CP-ECR. The average measured offset strains (3.0%) at 15.2% and 16% CP-ECR are slightly less than the ductility limit (3.1%). By interpolation between the data at 14.2% and 15.2% CP-ECR, the ductile-brittle-transition CP-ECR would be 15%. Thus, there is a penalty of 1% CP-ECR in using the less precise offset strain criterion as compared to the permanent strain criteria.

13. References for the Experimental Procedures

EP-1. M. Billone, Y. Yan, T. Burtseva, and R. Daum, "Cladding Embrittlement during Postulated Loss-of-Coolant Accidents," NUREG/CR-6967, July 2008 (ADAMS Accession No. ML082130389).

EP-2. J. Cathcart, R. Pawel, R. McKee, R. Druschel, G. Yurek, J. Campbell, and S. Jury, "Zirconium Metal-Water Oxidation Kinetics IV. Reaction Rate Studies," ORNL/NUREG-17, August 1977.

EP-3. J. Mardon, J. Brachet, L. Portier, V. Maillot, T. Forgeron, A. Lesbros, and N. Waeckel, "Influence of Hydrogen Simulating Burn-Up Effects on the Metallurgical and Thermal-Mechanical Behavior of M5™ and Zircaloy-4 Alloys under LOCA Conditions," *13th International Conference on Nuclear Engineering*, ICONE13-50457, pp. 1–9, Beijing, China, May 16–20, 2005.

EP-4. F. Nagase and H. Uetsuka, "Hydrogen Morphology and Hydrogen Embrittlement of Zircaloy Fuel Cladding used in NSRR/HBO Experiment," *Proceedings of the International Topical Meeting on Light Water Reactor Fuel Performance*, pp. 677–684, Portland, OR,

March 2–6, 1997, American Nuclear Society, La Grange Park, IL, 1997.

EP-5. American Society for Testing and Materials, ASTM E1447-05, "Standard Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Thermal Conductivity Method," Book of Standards, Vol. 03.05, ASTM International, West Conshohocken, PA, 2005; also available online at http://www.astm.org/standards/E1447.html.

EP-6. T. Burtseva, "Procedure for Hydrogen Analysis of Refractory Metals," ANL Intra-Laboratory Memo, IPS-467-00-00, Argonne National Laboratory, Argonne, IL, November 25, 2005.

EP-7. American Society for Testing and Materials, Designation G2/G2M-06, "Standard Test Method for Corrosion Testing of Products of Zirconium, Hafnium, and Their Alloys in Water at 680ºF [360ºC] or in Steam at 750ºF [400ºC]," pp. 1186–1193, ASTM International, West Conshohocken, PA, 2006.

EP-8. Claude Grandjean and Georges Hache, "A State of the Art Review of Past Programmes Devoted to Fuel Behaviour under Loss-of-Coolant Conditions. Part 3. Cladding Oxidation. Resistance to Quench and Post-Quench Loads," Institut de Radioprotection et de Sûreté (IRSN) Technical Report DPAM/SEMCA 2008-093, Saint Paul-lez-Durance Cedex, France, 2008.

EP-9. S. Leistikow and G. Schanz, "Oxidation Kinetics and Related Phenomena of Zircaloy-4 Fuel Cladding Exposed to High Temperature Steam and Hydrogen-Steam Mixtures under PWR Accident Conditions," *Nuclear Engineering and Design* 103, pp. 65–84, 1987.

EP-10. H. Uetsuka, "Oxidation of Zircaloy-4 under Limited Steam Supply at 1000 and 1300ºC," Kernforschungszentrum Karlsruhe (KfK) 3848, Kernforschungszentrum Karlsruhe GmbH, Postfach 360, 76021 Karlsruhe, 1984.

EP-11. M. Aomi, et al., "Behavior of BWR Fuel Cladding Tubes under Simulated LOCA Conditions," *7th International Conference on Nuclear Engineering*, ICONE-7435, 1999.

EP-12. S. Kawasaki, T. Furuta, and M. Suzuki, "Oxidation of Zircaloy-4 under High Temperature Steam Atmosphere and Its Effect on Ductility of Cladding," *Journal of Nuclear Science and Technology*, Vol. 15, No. 8, pp. 589–596, 1978.

EP-13. P. Hofmann and M. Markiewicz, "Chemical Interaction between As-Received and Pre-Oxidized Zircaloy-4 and Inconel-718 at High Temperatures," Kernforschungszentrum Karlsruhe (KfK) 4729, Kernforschungszentrum Karlsruhe GmbH, Postfach 360, 76021 Karlsruhe, June 1994.

EP-14. T.A. Burtseva, "Procedure for Conducting Ring-Compression Tests in Laboratory DL-102A," ANL-IPS Memo, IPS-495-00-00, November 26, 2007.

EP-15. Y. Yan, T.A. Burtseva, and M.C. Billone, "Post-quench Ductility Results for North Anna High-burnup 17×17 ZIRLO Cladding with Intermediate Hydrogen Content," ANL letter report to NRC, April 17, 2009 (ADAMS Accession No. ML091200702).

D. IMPLEMENTATION

The purpose of this section is to provide information on how applicants and licensees[5] may use this guide and information regarding the NRC's plans for using this regulatory guide. In addition, it describes how the NRC staff complies with the Backfit Rule (10 CFR 50.109) and any applicable finality provisions in 10 CFR Part 52.

Use by Licensees

Licensees may voluntarily[6] use the guidance in this document to demonstrate compliance with the underlying NRC regulations. Methods or solutions that differ from those described in this regulatory guide may be deemed acceptable if they provide sufficient basis and information for the NRC staff to verify that the proposed alternative demonstrates compliance with the appropriate NRC regulations.

Licensees may use the information in this regulatory guide for actions which do not require NRC review and approval such as changes to a facility design under 10 CFR 50.59 that do not require prior NRC review and approval. Licensees may use the information in this regulatory guide or applicable parts to resolve regulatory or inspection issues.

Use by NRC Staff

During regulatory discussions on plant specific operational issues, the staff may discuss with licensees various actions consistent with staff positions in this regulatory guide, as one acceptable means of meeting the underlying NRC regulatory requirement. Such discussions would not ordinarily be considered backfitting even if prior versions of this regulatory guide are part of the licensing basis of the facility. However, unless this regulatory guide is part of the licensing basis for a facility, the staff may not represent to the licensee that the licensee's failure to comply with the positions in this regulatory guide constitutes a violation.

If an existing licensee voluntarily seeks a license amendment or change and (1) the NRC staff's consideration of the request involves a regulatory issue directly relevant to this new or revised regulatory guide and (2) the specific subject matter of this regulatory guide is an essential consideration in the staff's determination of the acceptability of the licensee's request, then the staff may request that the licensee either follow the guidance in this regulatory guide or provide an equivalent alternative process that demonstrates compliance with the underlying NRC regulatory requirements. This is not considered backfitting as defined in 10 CFR 50.109(a)(1) or a violation of any of the issue finality provisions in 10 CFR Part 52.

The NRC staff does not intend or approve any imposition or backfitting of the guidance in this regulatory guide. The NRC staff does not expect any existing licensee to use or commit to using the guidance in this regulatory guide, unless the licensee makes a change to its licensing basis. The NRC staff does not expect or plan to request licensees to voluntarily adopt this regulatory guide to resolve a generic regulatory issue. The NRC staff does not expect or plan to initiate NRC regulatory action which would require the use of this regulatory guide. Examples of such unplanned NRC regulatory actions include issuance of an order requiring the use of the regulatory guide, requests for information under

[5] In this section, "licensees" refers to licensees of nuclear power plants under 10 CFR Parts 50 and 52; and the term "applicants," refers to applicants for licenses and permits for (or relating to) nuclear power plants under 10 CFR Parts 50 and 52, and applicants for standard design approvals and standard design certifications under 10 CFR Part 52.

[6] In this section, "voluntary" and "voluntarily" means that the licensee is seeking the action of its own accord, without the force of a legally binding requirement or an NRC representation of further licensing or enforcement action.

10 CFR 50.54(f) as to whether a licensee intends to commit to use of this regulatory guide, generic communication, or promulgation of a rule requiring the use of this regulatory guide without further backfit consideration.

If a licensee believes that the NRC is either using this regulatory guide or requesting or requiring the licensee to implement the methods or processes in this regulatory guide in a manner inconsistent with the discussion in this Implementation section, then the licensee may file a backfit appeal with the NRC in accordance with the guidance in NUREG-1409 and NRC Management Directive 8.4.

GLOSSARY

alpha layer—For the purposes of this regulatory guide, refers to the zirconium phase which is characterized by a hexagonally close-packed crystal structure and is stable at room temperature. At high temperatures, the beta phase is stable; however, dissolved oxygen can stabilize the alpha phase at high temperature.

beta layer—For the purposes of this regulatory guide, refers to the zirconium phase which is characterized by a cubic crystal structure and is stable at elevated temperatures of ≈1,000 °C.

breakaway oxidation—For the purposes of this regulatory guide, the fuel-cladding oxidation phenomenon in which weight-gain rate deviates from normal kinetics. This change occurs with a rapid increase of hydrogen pickup during prolonged exposure to a high-temperature steam environment, which promotes loss of cladding ductility

corrosion—For the purposes of this regulatory guide, the formation of a zirconium oxide layer resulting from the reaction of zirconium with coolant water during normal operation.

loss-of-coolant accident (LOCA)—A hypothetical accident that would result from the loss of reactor coolant, at a rate in excess of the capability of the reactor coolant makeup system, from breaks in pipes in the reactor coolant pressure boundary up to and including a break equivalent in size to the double-ended rupture of the largest pipe in the reactor coolant system.

offset strain—For the purposes of this regulatory guide, the value determined from a load-displacement curve by the following procedure: (1) linearize the initial loading curve, (2) use the slope of the initial loading curve to mathematically unload the sample at the peak load before a significant load drop (≈30 to 50%) indicating a through-wall crack along the length of the sample, and (3) determine the offset displacement (distance along the displacement axis between loading and unloading lines). This offset displacement is normalized to the outer diameter of the preoxidized cladding to determine a relative plastic strain.

oxidation—For the purposes of this regulatory guide, the formation of a zirconium oxide layer resulting from the reaction of zirconium with high-temperature steam during LOCA conditions.

permanent strain—For the purposes of this regulatory guide, the difference between the posttest outer diameter (after the sample is unloaded) and the pretest outer diameter of a cladding ring, normalized to the initial diameter of the cladding ring.

monoclinic oxide—For the purposes of this regulatory guide, the oxide phase that develops during normal operation and is neither fully dense nor protective. Although the oxide phase that typically develops under LOCA conditions is the tetragonal oxide phase, there are conditions that might occur during a small-break LOCA (such as extended time-at-temperature around 1,000 °C) which promote a transformation to the monoclinic phase.

tetragonal oxide—For the purposes of this regulatory guide, the oxide phase that develops under LOCA conditions which is dense, adherent, and observed to be protective with respect to hydrogen pickup.

REFERENCES[7]

1. "Proposed Rule FRN," ADAMS Accession No. ML12283A174

2. NUREG/IA-0211, "Experimental Study of Embrittlement of Zr-1%Nb VVER Cladding under LOCA-Relevant Conditions," L. Yegorova, et al., March 2005 (ADAMS Accession No. ML051100343).

3. IFE/KR/E-2008/004, "LOCA Testing of High Burnup PWR Fuel in the HBWR. Additional PIE [Post Irradiation Examination] on the Cladding of the Segment 650-5," B.C. Oberlander, M. Espeland, H.K. Jenssen, April 11, 2008 (ADAMS Accession No. ML081750715).

4. NRC Research Information Letter 0801, "Technical Basis for Revision of Embrittlement Criteria in 10 CFR 50.46," May 30, 2008 (ADAMS Accession No. ML081350225).

5. NUREG/CR-6967, "Cladding Embrittlement during Postulated Loss-of-Coolant Accidents," M. Billone, et al., July 2008 (ADAMS Accession No. ML082130389).

6. Draft Regulatory Guide 1263, "Regulatory Guidance on Establishing Analytical Limits for Zirconium-Based Alloy Cladding," U.S. Nuclear Regulatory Commission, Washington, DC.

[7] Publicly available NRC published documents are available electronically through the NRC Library at: http://www.nrc.gov/reading-rm/doc-collections/. The documents can also be viewed on-line or printed for a fee in the NRC's Public Document Room (PDR) at 11555 Rockville Pike, Rockville, MD; the mailing address is USNRC PDR, Washington, DC 20555; telephone (301) 415-4737 or (800) 397-4209; fax (301) 415-3548; and e-mail pdr.resource@nrc.gov.

APPENDIX A

RING-COMPRESSION RESULTS FOR ROOM TEMPERATURE AND 135 ºC TESTS WITH AS-FABRICATED CLADDING SAMPLES

Document IPS-495-00-00 (Ref. 1) describes the procedure for verification of calibration, for test conduct, and for data interpretation for ring-compression tests (RCTs) conducted with the Instron Model 5566 Material Test System (MTS) used by Argonne National Laboratory to conduct RCTs with as-fabricated and prehydrided oxidized cladding samples. That document describes Phase 3 pre-data-generation tests to ensure that the Instron, as well as associated control and data acquisition systems, is performing within the expected range. It is recommended that these tests be conducted with any MTS before the generation of postquench ductility (PQD) data, particularly if the MTS has been used to conduct other tests (e.g., axial tensile tests) requiring modification to the load train, if the machine has been idle for more than a couple of months, or if testing is conducted beyond the due date for calibration verification. Three tests at room temperature (RT) and three tests at 135 degrees Celsius (ºC) are to be conducted with as-fabricated cladding rings at 2 millimeters (mm)/minute (0.033 mm/second) to a maximum crosshead displacement of 2 mm. The data output (load displacement curves) is to be analyzed in terms of the measured loading stiffness K_m (linearized slope of the load versus displacement curve in kilo Newton/mm) and the measured offset displacement (δ_d, in mm). The measured loading stiffness K_m is compared to the calculated ring stiffness K_c according to the textbook formula:

$$K_c = (1/1.8)\, E\, L\, (h/R)^3, \qquad \text{(A1)}$$

where E is Young's modulus in kN/mm^2, L is the actual length of the ring in mm, h is the wall thickness in mm, and R is the ring midwall radius in mm. Eq. A1 is applicable to the elastic behavior of a thin-wall ring of uniform length, outer diameter, and wall thickness.

The reference length for the test rings is 8 mm. However, as offset displacement should be independent of ring length and stiffness varies linearly with ring length, actual values of sectioned rings may be within the range 8.0±1.0 mm. The wall thickness (h) and the outer diameter (D_o) will vary somewhat along the length of cladding tubes. The pretest wall thickness for each sample should be measured at four circumferential orientations (0º, 90º, 180º, and 270º). The value of h used in Equation B1 is the average of the four readings. The pretest minimum [$(D_{oi})_{min}$] and maximum [$(D_{oi})_{max}$] outer diameters should be determined for each sample and averaged to give D_{oi}, where the "i" refers to initial or pretest value. The value of R used in Equation A1 is calculated from the relationship $R = (D_{oi} - h)/2$. Young's modulus, E, for cladding alloys is assumed to be the same as the isotropic modulus reported for Zircaloy (Zry)-4: E = 92.4 kN/mm^2 (92,400 megapascals (MPa)) at RT, and E = 86.5 kN/mm^2 (86,500 MPa) at 135 ºC. For rings of uniform length, thickness, and outer diameter, the measured stiffness should be within about 10% of the calculated stiffness for machines with relatively high load-train stiffness. Because machine compliance tends to reduce the measured stiffness, the expectation is that the measured stiffness will be less than or equal to the calculated stiffness. Measured stiffness values ≥10% higher than the values predicted by Equation A1 indicate that the load cell and/or crosshead displacement indicator may be out of calibration.

The offset displacement (δ_p) is to be compared to the permanent displacement (d_p), which is determined from the difference in the pretest and posttest diameters in the loading direction. The ring is to be positioned in the Instron such that the loading direction is along the minimum pretest diameter. Based on the error introduced by assuming that the unloading stiffness is equal to the loading stiffness, the expectation is that $\delta_p < d_p$ and that the difference is $\delta_p - d_p \leq 0.2$ mm, which is based on an extensive dataset for as-fabricated cladding displaced to 1.5–2.0 mm at RT and 2 mm/minute in the Instron 5566.

Two assessments are made using the measured stiffness and offset displacement values: one is precision (repeatability), and the other is adequacy of load and displacement measurements. The most important determination of the adequacy of the Instron MTS is the repeatability of offset and permanent displacements, as well as the difference of these two numbers. In making this assessment, the measured stiffness should be normalized to 8 mm by multiplying the measured stiffness by (8 mm/L): $K_{mn} = (8 \text{ mm/L}) K_m$. The calculated stiffness should also be normalized to 8 mm to give K_{cn}. Also, as the stiffness is highly dependent on the wall thickness, this factor should be considered in the data assessment.

The procedure described in IPS-495-00-00 Phase 3 should be used for any MTS, including the Argonne National Laboratory Instron 8511, which was used to perform RCTs with oxidized high-burnup cladding samples. This appendix presents the ring-compression verification test results for the Instron 8511 as an example of the procedure to follow and the methodology for interpreting the results.

The RT tests check the physical components, the data control software, and the data acquisition software. The elevated temperature tests check the physical and software components of the furnace system, as well as the performance of the Instron at 135 ºC. Before conducting the elevated temperature tests with control and monitoring thermocouples, it should be demonstrated that the thermocouples have been calibrated to a National Institute of Standards and Technology (NIST)-traceable standard. The option is available to have the vendor do this calibration and supply a certificate for each thermocouple in a batch of thermocouples or one thermocouple in the batch. If only one thermocouple in a batch has a calibration certificate, then the thermocouples to be used to control and monitor ring temperature should be calibrated by comparison to the certified thermocouple. Generally, Type K thermocouples are used to monitor low temperatures such as 135 ºC. The expected error for this type of thermocouple is ±0.3 ºC relative to an NIST-traceable standard.

As the Instron 8511 is a servo-hydraulic machine, some checks were made to ensure that all moving parts, all auxiliary equipment, and all data recorders functioned properly. Three RCTs were then conducted at RT. Based on the RT results (see Table A-1 and Figures A-1 through A-3), the average difference between the offset and permanent displacement was 0.19 mm, which is consistent with previous experience. Therefore, the Instron 8511 crosshead displacement indicator was determined to be accurate enough for ring-compression testing. The measured loading stiffness values were about 15% lower than the predicted values. The loading stiffness is expected to be less than or equal to the calculated stiffness because of the influence of machine compliance. Based on experience with the Instron Model 5566, the measured stiffness has been within 10% of the calculated stiffness. The Instron Model 8511 has a much longer load train, higher machine compliance, and lower machine stiffness. This may account for the difference between measured and calculated stiffness values. Although load is not an important parameter in ring-compression ductility tests, the stiffness results indicate that the load-cell output values are adequate for RCTs.

The results at 135 ºC (see Table A-1 and Figures A-4 through A-6) proved to be more consistent than the RT results. For ductility determination, the most important parameter is the difference between offset displacement and permanent displacement, which was 0.20 mm on average. The measured stiffness values were about 8% lower than the calculated values, which suggests adequate load-cell performance at 135 ºC.

The results of the six tests support the use of the Instron Model 8511 for performing RCT ductility tests using oxidized high-burnup cladding samples.

Table A-1. Results of Instron 8511 Checkout Tests to Verify Calibration for Conducting Ring- Compression Tests at a Displacement Rate of 0.0333 mm/s

K_c is calculated ring stiffness (i.e., spring constant), K_m is stiffness determined from linearized slope of load displacement curve, subscript "n" refers to stiffness normalized to 8 mm, δ_p is the offset displacement determined from the load-displacement curve, and d_p is the permanent displacement in the loading direction determined from pretest diameter minus posttest diameter.

Sample ID	Test T, ºC	D_{oi}, mm	L, mm	h, mm	K_{cn}, kN/mm	K_m, kN/mm	K_{mn}, kN/mm	δ_p, mm	d_p, mm	$\delta_p - d_p$, mm
109B1	RT	9.49	7.67±0.06	0.587±0.003	0.94	0.77	0.80	1.24	1.07	0.17
109B2	RT	9.49±0.01	7.75±0.06	0.589±0.003	0.95	0.77	0.79	1.18	1.00	0.18
109B3	RT	9.49±0.01	8.03±0.02	0.589±0.003	0.95	0.85	0.85	1.31	1.08	0.23
RT Summary	RT	9.49	7.82	0.59	0.95		0.81	1.24	1.05	0.19
109B4	132±5	9.495±0.005	7.81±0.02	0.589±0.003	0.89	0.81	0.83	1.40	1.21	0.19
109B5	135±5	9.495±0.005	8.01±0.07	0.590±0.003	0.89	0.83	0.83	1.40	1.21	0.19
109B9	135±5	9.475±0.005	8.21±0.06	0.588±0.003	0.91	0.85	0.83	1.40	1.17	0.23
135 ºC Summary		9.49	8.00	0.59	0.90		0.83	1.40	1.20	0.20

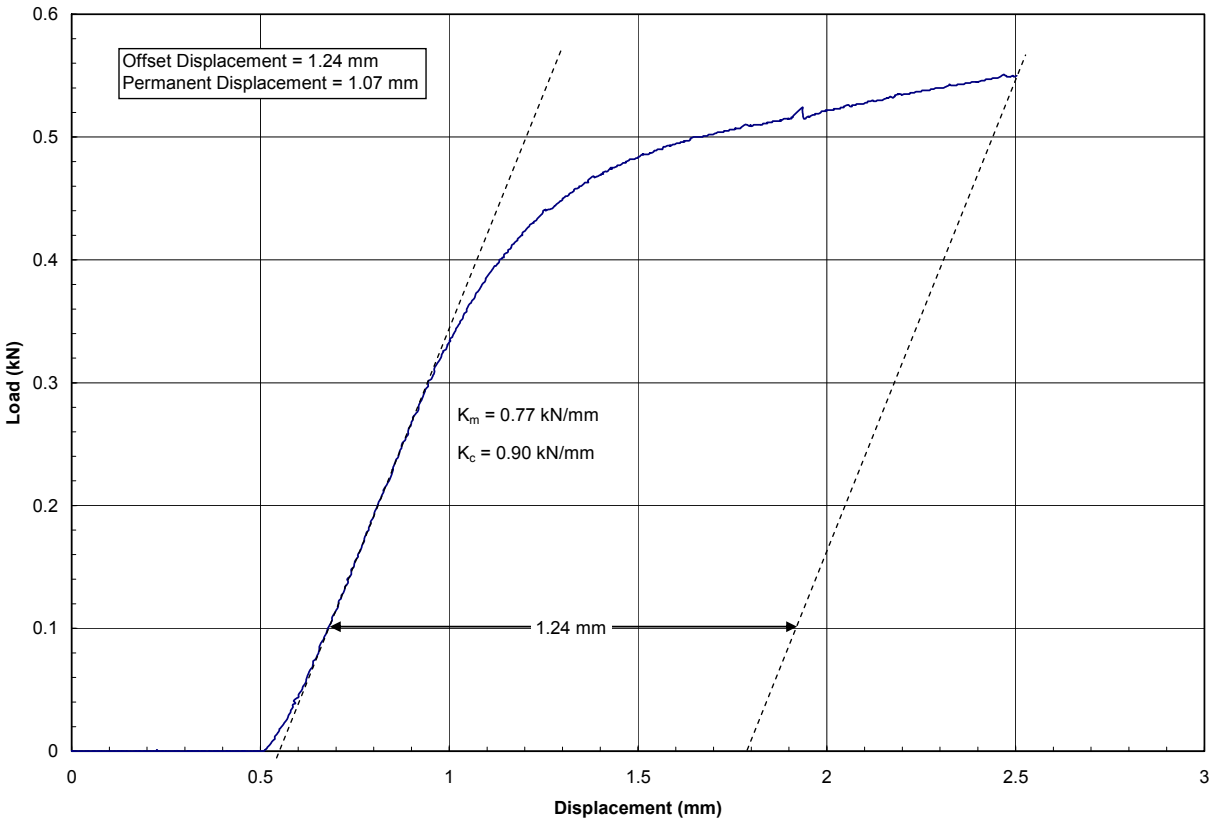

Figure A-1. Load-displacement curve for ZIRLO™ sample 109B1 compressed at RT and 2 mm/minute to 2-mm total displacement.

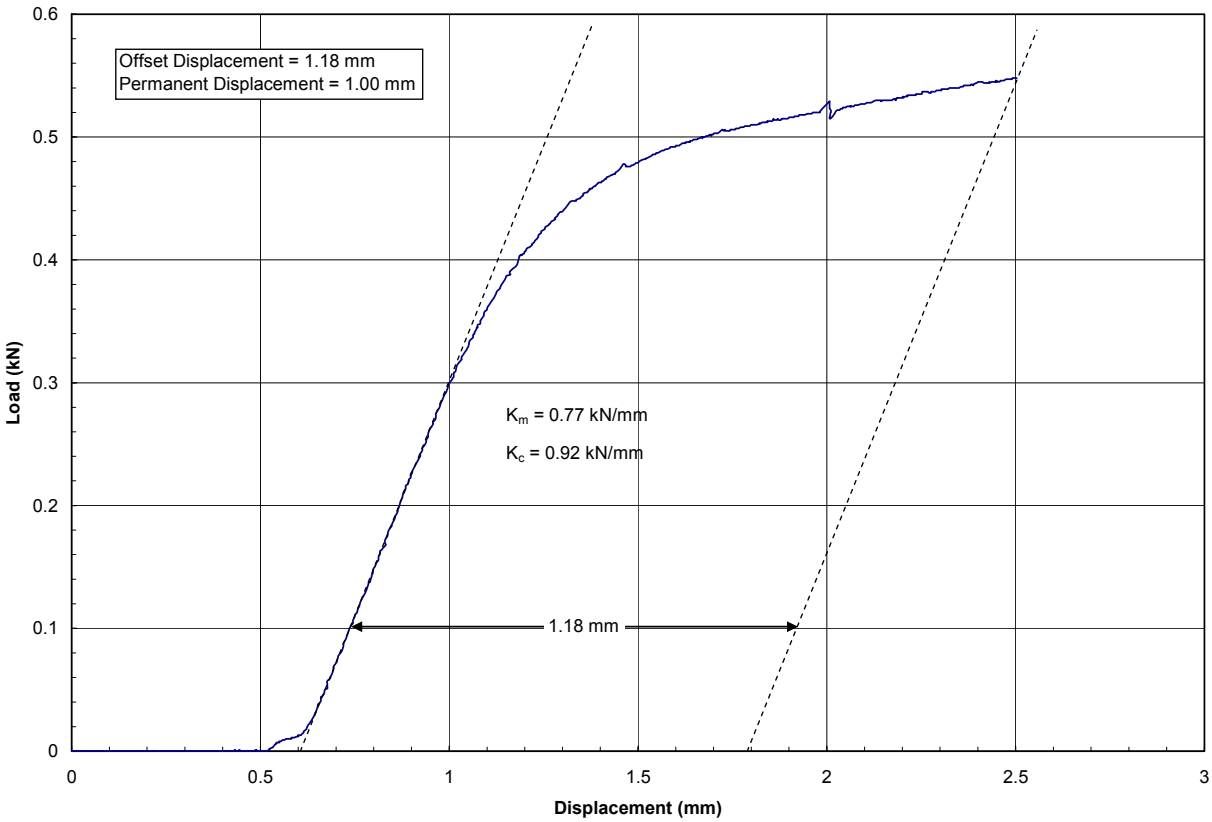

Figure A-2. Load-displacement curve for ZIRLO™ sample 109B2 compressed at RT and 2 mm/minute to 2-mm total displacement.

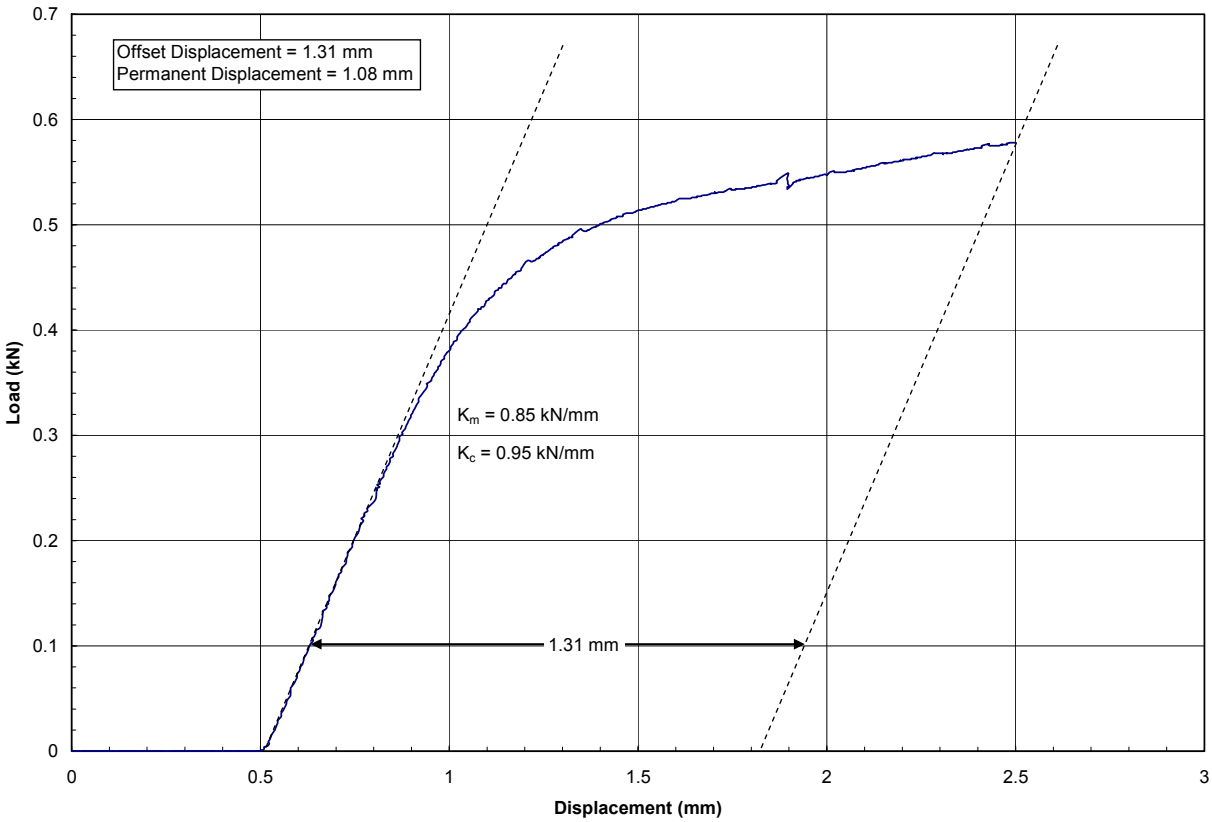

Figure A-3. Load-displacement curve for ZIRLO™ sample 109B3 compressed at RT and 2 mm/minute to 2-mm total displacement.

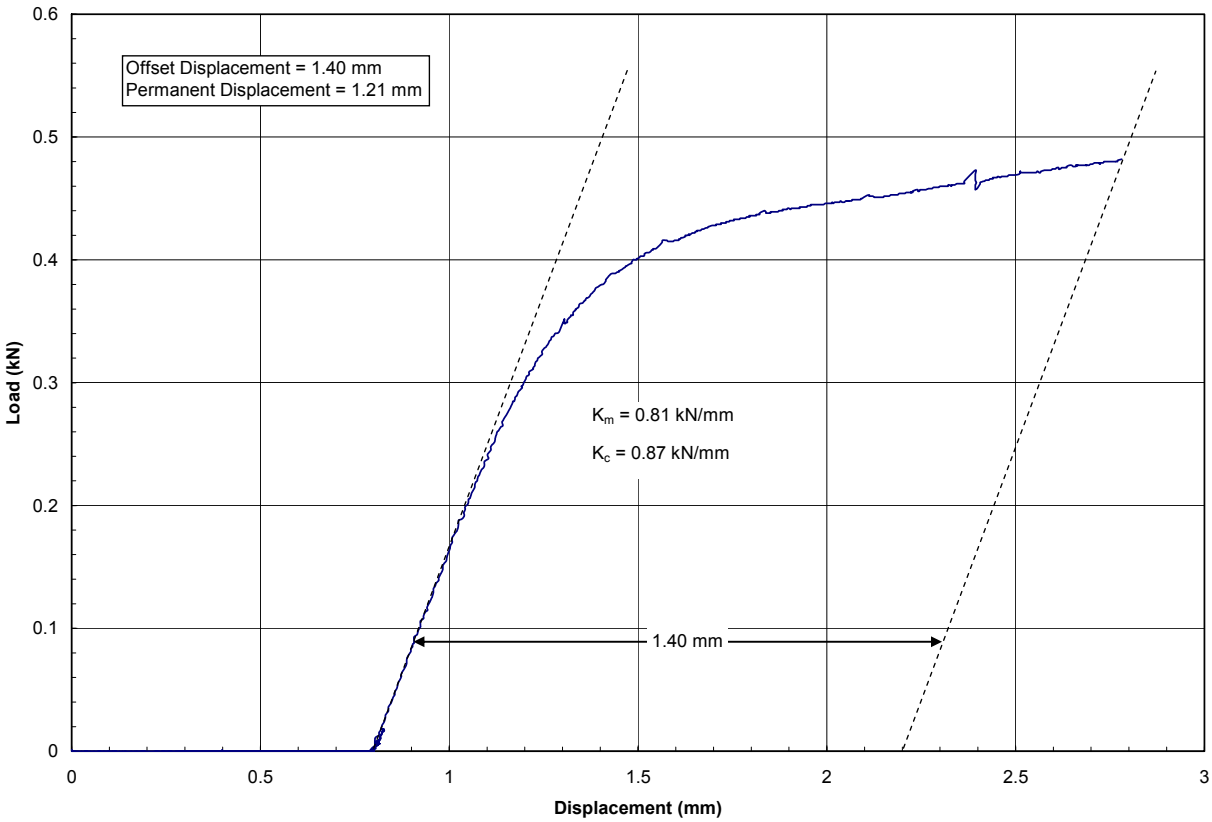

Figure A-4. Load-displacement curve for ZIRLO™ sample 109B4 compressed at 135 ºC and 2 mm/minute to 2-mm total displacement.

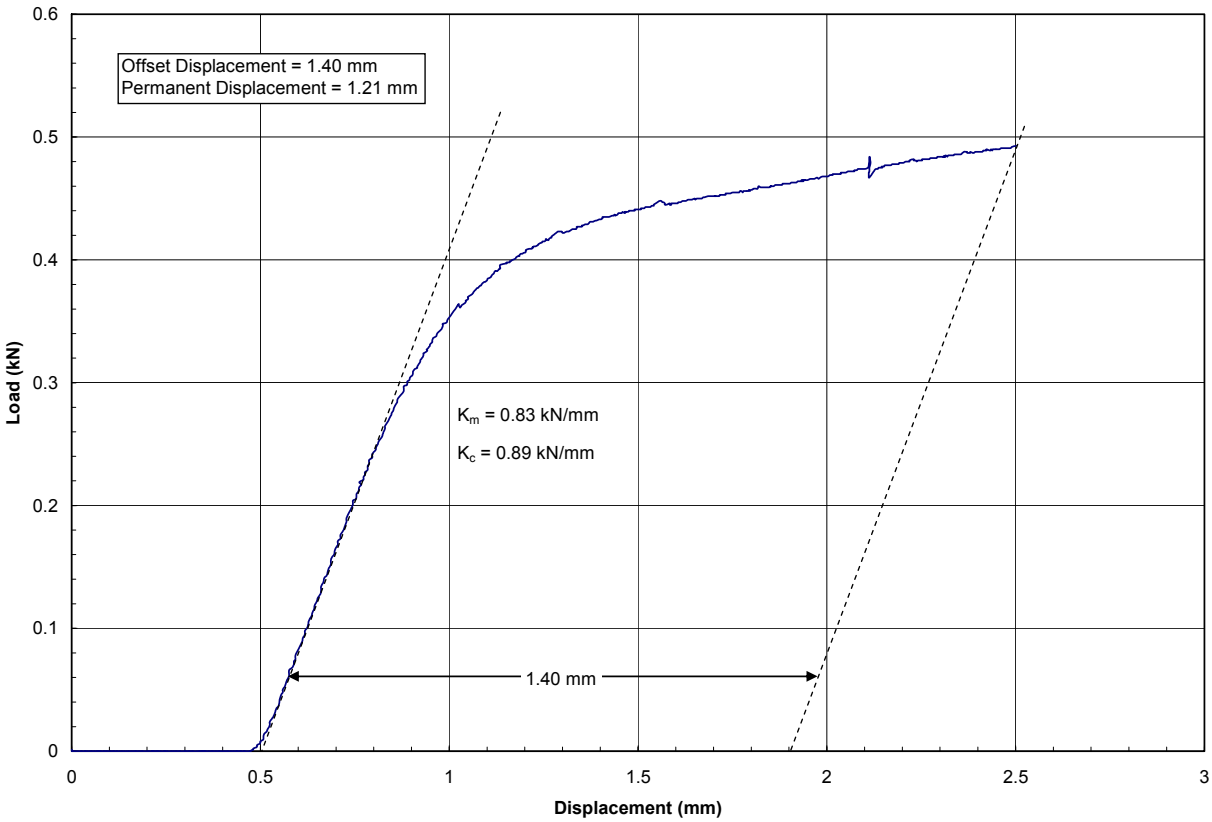

Figure A-5. Load-displacement curve for ZIRLO™ sample 109B5 compressed at 135 ºC and 2 mm/minute to 2-mm total displacement.

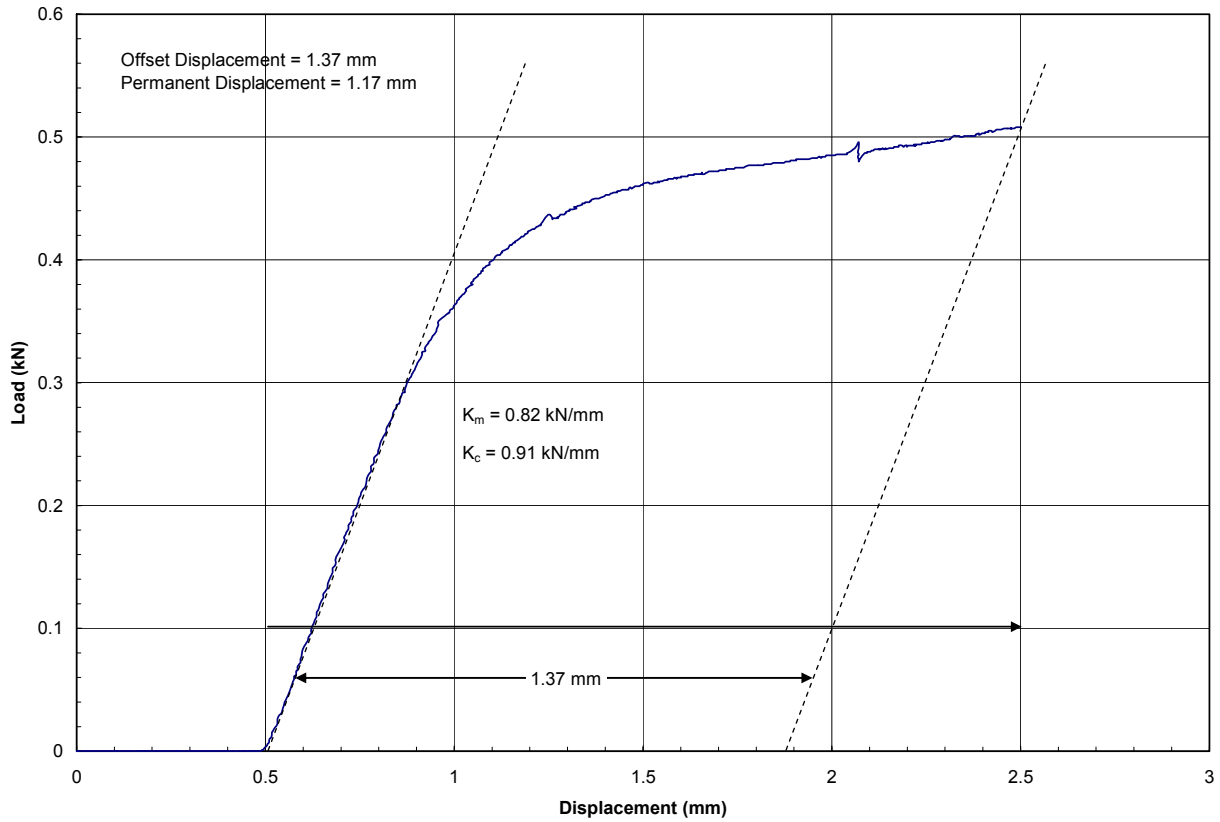

Figure A-6. Load-displacement curve for ZIRLO™ sample 109B9 compressed at 135 ºC and 2 mm/minute to 2-mm total displacement.

References[1]

1. T.A. Burtseva, "Procedure for Conducting Ring-Compression Tests in Laboratory DL-102A," ANL-IPS Memo, IPS-495-00-00, November 26, 2007.

[1] Publicly available NRC published documents are available electronically through the NRC Library at: http://www.nrc.gov/reading-rm/doc-collections/. The documents can also be viewed on-line or printed for a fee in the NRC's Public Document Room (PDR) at 11555 Rockville Pike, Rockville, MD; the mailing address is USNRC PDR, Washington, DC 20555; telephone 301-415-4737 or (800) 397-4209; fax (301) 415-3548; and e-mail pdr.resource@nrc.gov.

APPENDIX B

EXAMPLES OF LOAD-DISPLACEMENT CURVES, OFFSET STRAINS, AND PERMANENT STRAINS FOR OXIDIZED AND QUENCHED CLADDING SAMPLES

Figures B-1 through B-8 show examples of load-displacement curves and offset displacements determined from these curves. For this series of tests, prehydrided vintage 15×15 Zircaloy (Zry)-4 cladding samples, comparable to H.B. Robinson vintage cladding, were oxidized to 6% Cathcart-Pawel (CP) equivalent cladding reacted (ECR) at a maximum temperature of 1,200ºdegrees Celsius (°C). As shown in Table B-1 (Table 52 in NUREG/CR 6967, "Cladding Embrittlement during Postulated Loss-of-Coolant Accidents," issued July 2008, Agencywide Documents Access and Management System Accession No. ML082130389), the quench temperature was varied from 800 ºC to 700 ºC to 600 ºC to slow-cooling without quench. The load-displacement curves (see Figures B-1 through B-6) for the quenched samples indicate that all of these samples were brittle. Based on both offset strains and permanent strains, only the slow-cooled samples retained ductility (see Figures B-7 through B-8). For the samples that retained ductility, the difference between the offset strains and the permanent strains was only 0.9%. For prehydrided Zry-4, the ductility criteria used in EP-Ref. 1 is that ≥1.0% permanent strain implies ductility.

Table B-1. Postquench Ductility of Prehydrided HBR-type 15×15 Zry-4 Cladding Oxidized to 6% CP-ECR at 1,200 °C, Cooled at 13 °C/s to 800 °C, and Quenched at 800 °C, Cooled from 800 °C to 700 °C at 3 °C/s and Quenched at 700 °C, Cooled from 700 °C to 600 °C at 2 °C/s and Quenched at 600 °C, or Slow-cooled from 600 °C to RT at <2 °C/s

CP-ECR calculated from beginning of ramp to end of hold time; ring-compression tests performed on ≈8-mm-long samples at 135 °C and 0.0333 mm/s crosshead displacement rate

Sample and Test Conditions			ECR %		Plastic Displacement, mm		Plastic Strain, %	
Q-T, ºC or SC	Test Time[a] s	H wppm	CP	Meas.	Offset	Permanent	Offset	Permanent
800	106	450	6.0	6.5	0.10	0.08	0.9	0.7
800	106	450	6.0	6.5	0.09	0.07	0.8	0.7
700	106	450	6.0	6.6	0.07	0.05	0.6	---
700	106	450	6.0	6.6	0.10	0.05	0.9	0.5
600	106	460	6.0	6.5	0.08	0.05	0.7	0.5
600	106	460	6.0	6.5	0.13	0.08	1.2	0.7
SC	106	470	6.0	6.4	0.22	0.18	2.1	1.7
SC	106	470	6.0	6.4	0.53	0.39	4.9	3.6

[a] From beginning of ramp at 300 ºC to end of hold time at ≈1,200 ºC

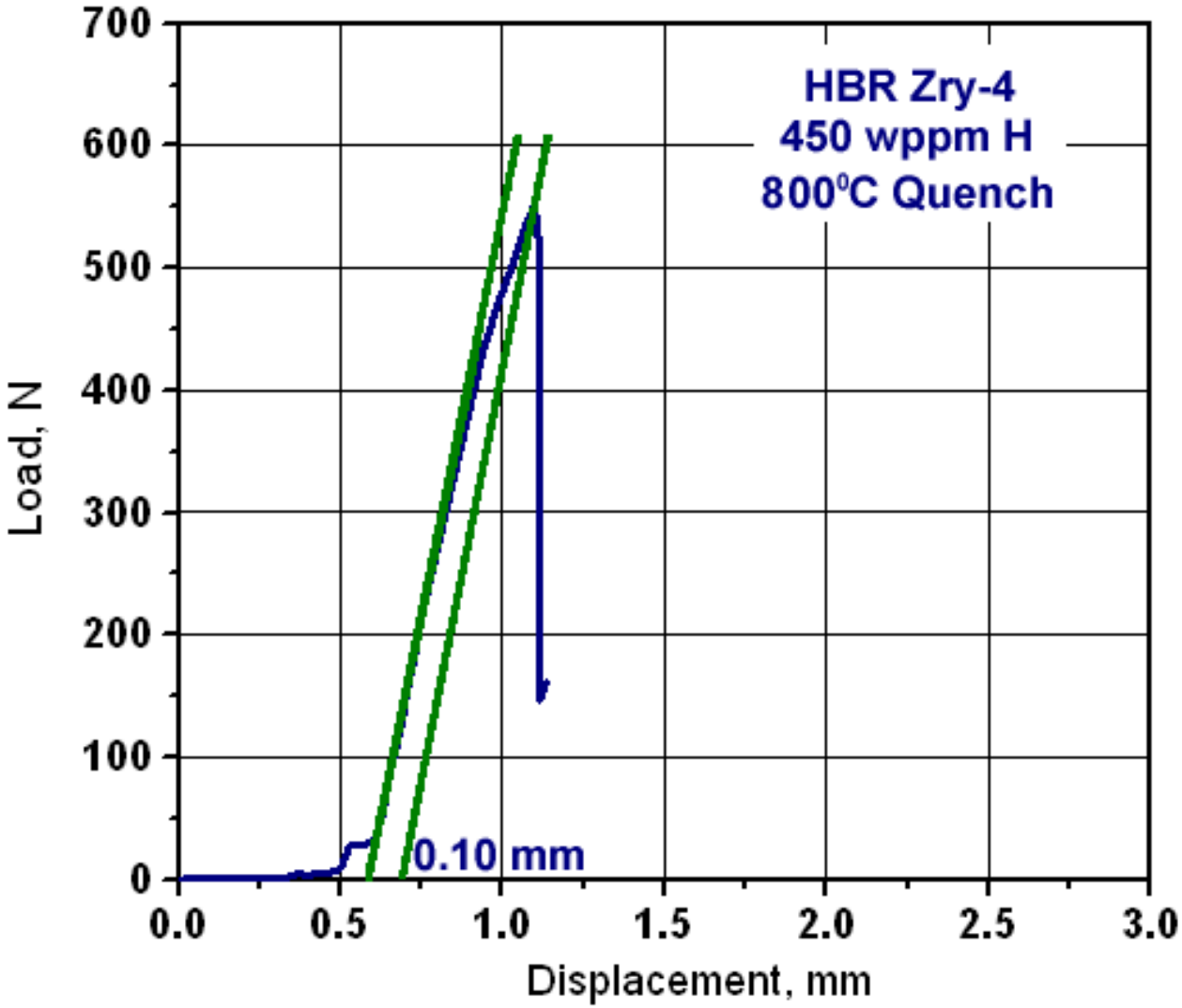

Figure B-1. Load-displacement results for ring #1 (8.12 mm long) of a prehydrided (450 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 °C and quenched at 800 °C. The posttest sample had tight through-wall cracks at load and support locations. Offset and permanent displacements were 0.10 mm and 0.08 mm, respectively.

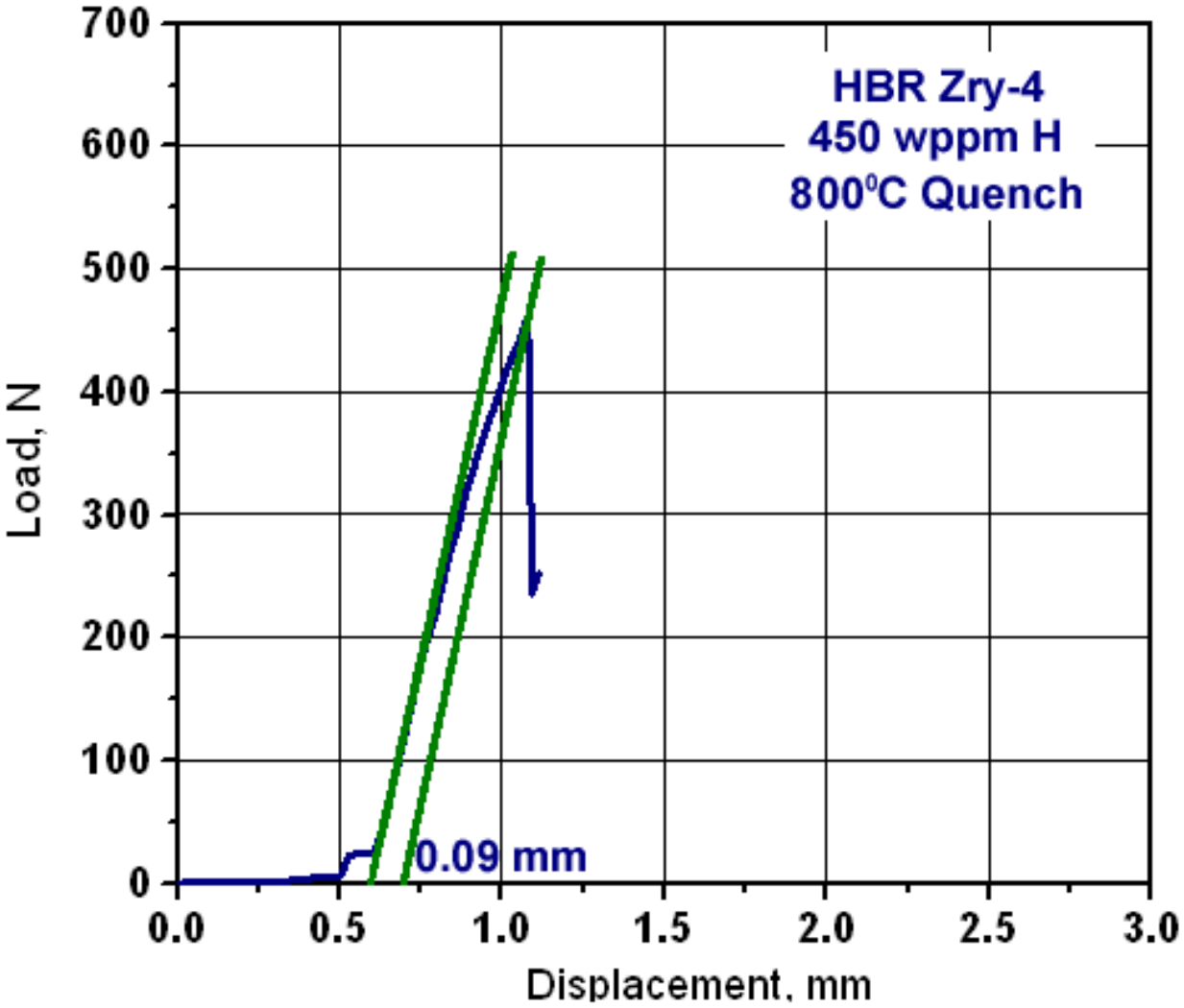

Figure B-2. Load-displacement results for ring #2 (6.84 mm long) of a prehydrided (450 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 °C and quenched at 800 °C. The posttest sample had a tight through-wall crack at the support locations. Offset and permanent displacements were 0.09 mm and 0.07 mm, respectively.

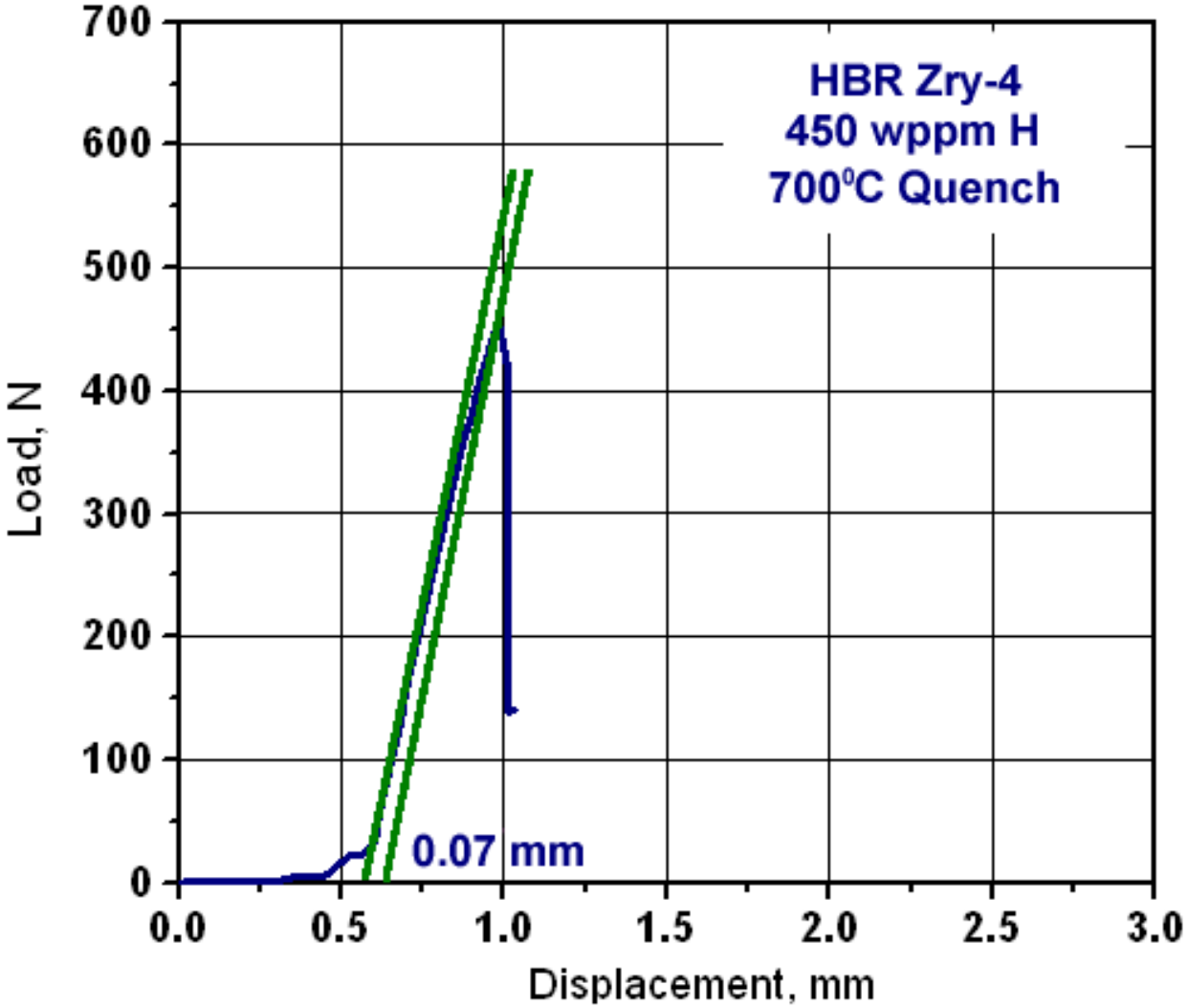

Figure B-3. Load-displacement results for ring #1 (8.08 mm long) of a prehydrided (450 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 °C and quenched at 700 °C. The posttest sample had through-wall cracks at load and support locations. Offset displacement was 0.07 mm.

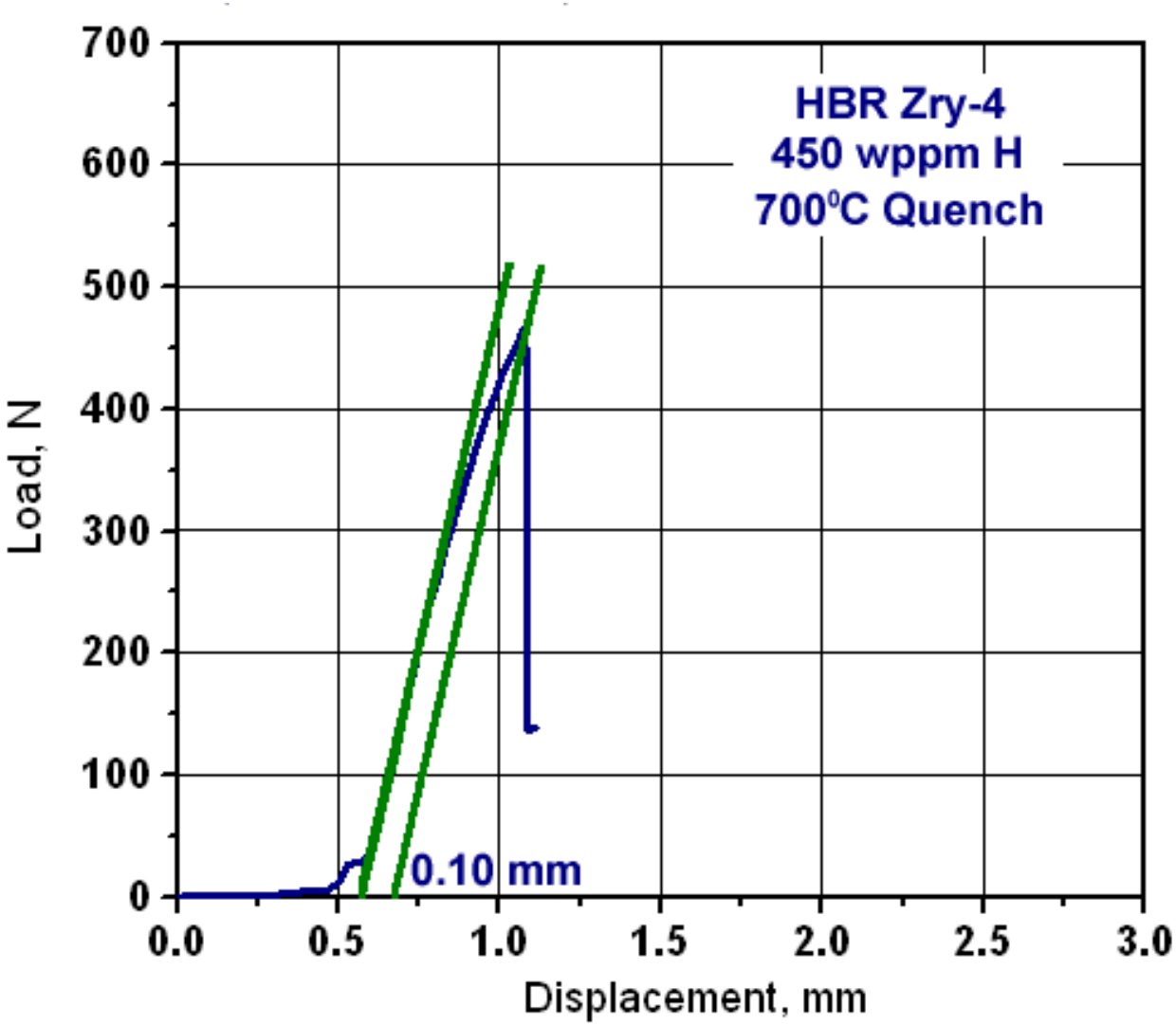

Figure B-4. Load-displacement results for ring #2 (7.30 mm long) of a prehydrided (450 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 °C and quenched at 700 °C. The posttest sample had tight through-wall cracks at load and support locations. Offset and permanent displacements were 0.10 mm and 0.05 mm, respectively.

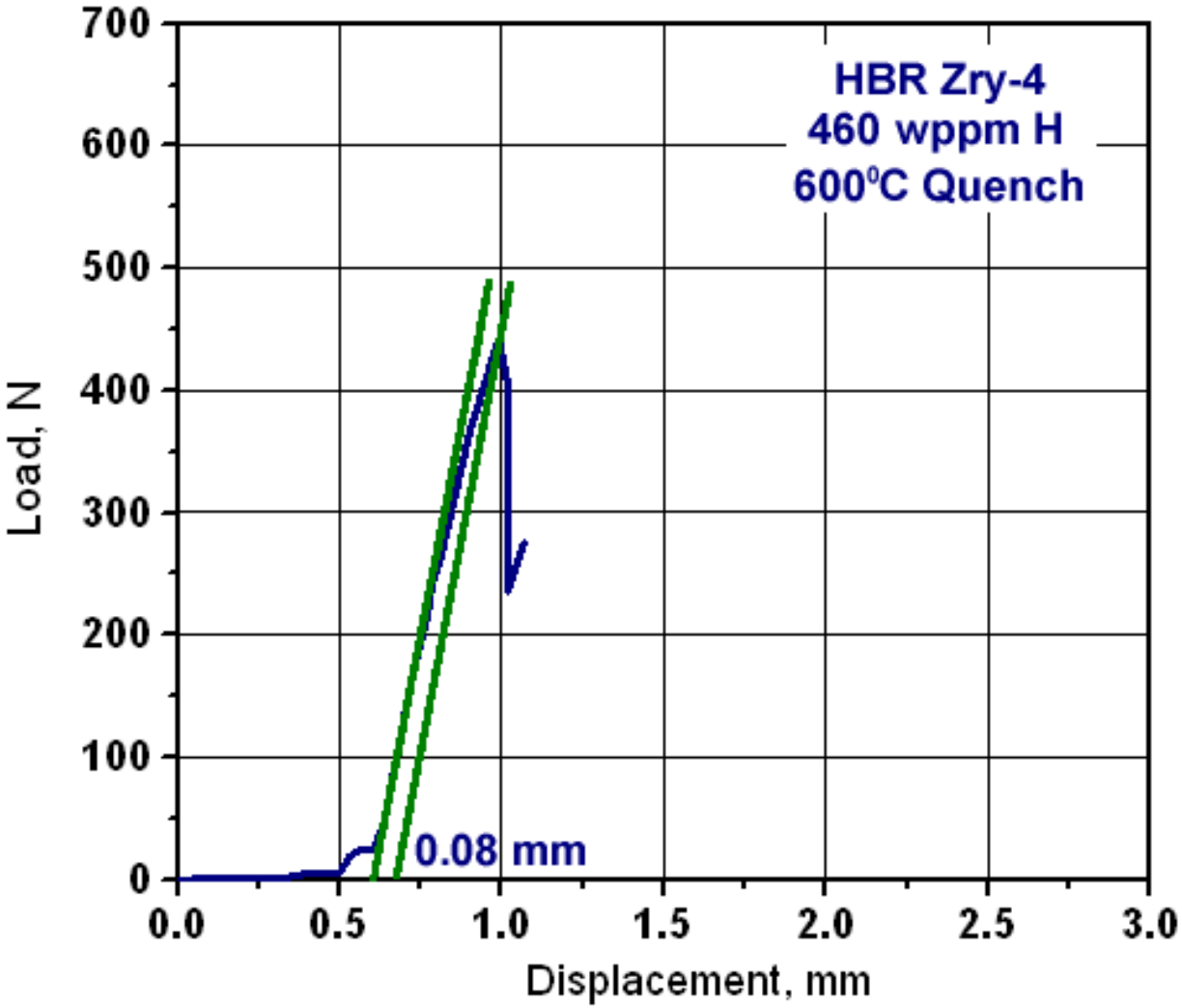

Figure B-5. Load-displacement results for ring #1 (7.93 mm long) of a prehydrided (460±30 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 °C and quenched at 600 °C. The posttest sample had a tight through-wall crack at the support location. Offset and permanent displacements were 0.08 mm and 0.05 mm, respectively.

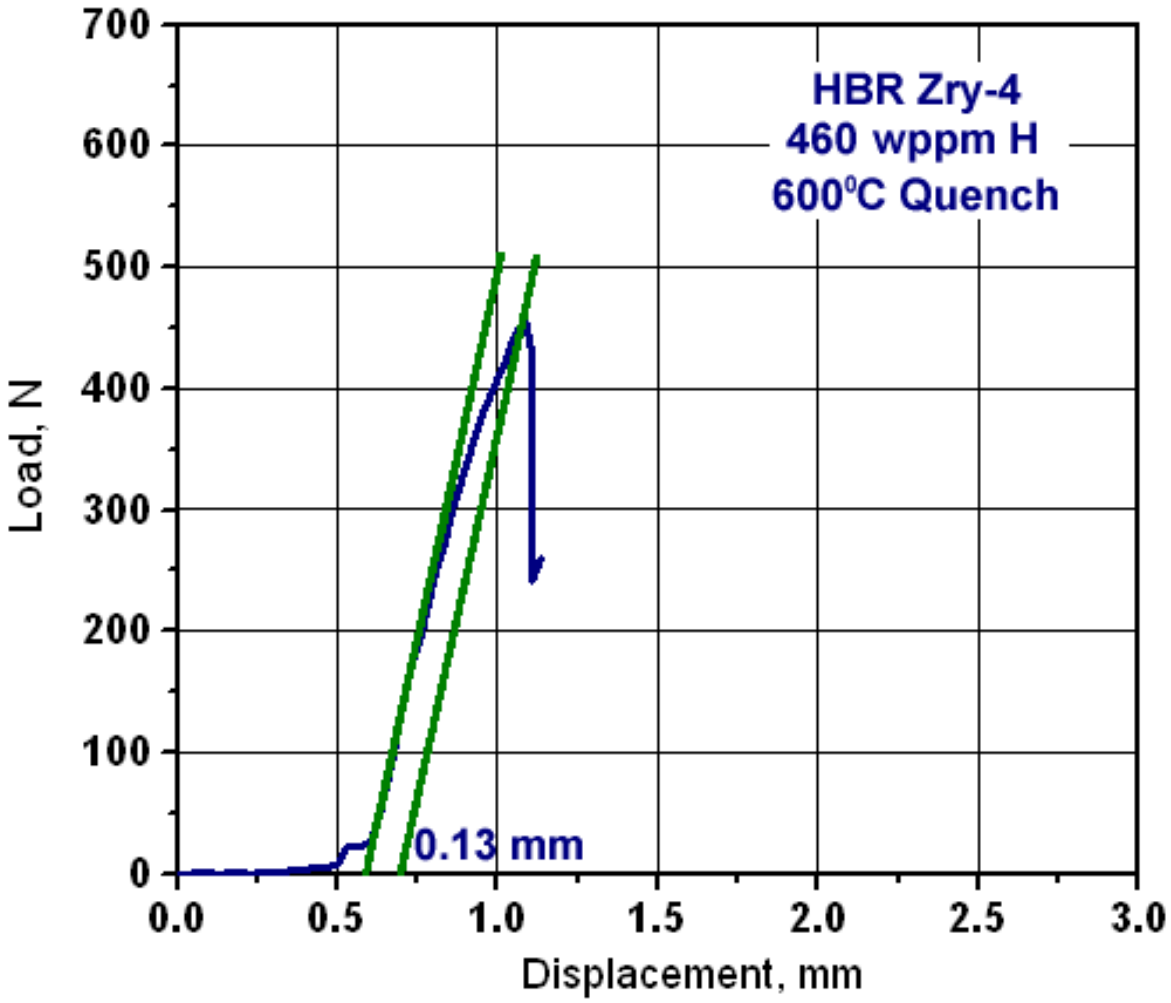

Figure B-6. Load-displacement results for ring #2 (7.05 mm long) of a prehydrided (460±30 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 °C and quenched at 600 °C. The posttest sample had a through-wall crack at the loading location. Offset and permanent displacements were 0.13 mm and 0.08 mm, respectively.

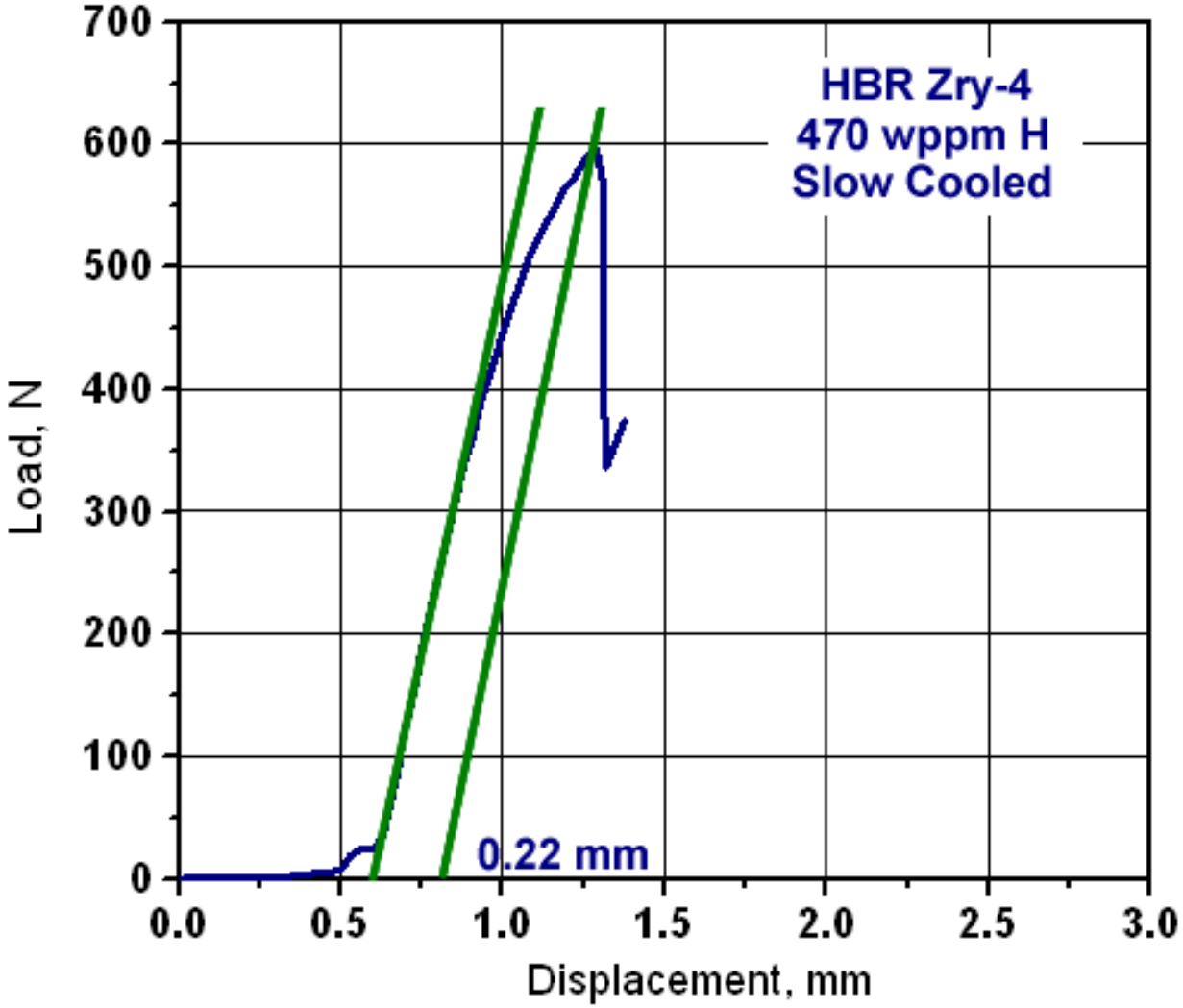

Figure B-7. Load-displacement results for ring #1 (8.04 mm long) of a prehydrided (470±30 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and cooled to room temperature without quench. The posttest sample had a tight through-wall crack at the support location. Offset and permanent displacements were 0.22 mm and 0.18 mm, respectively.

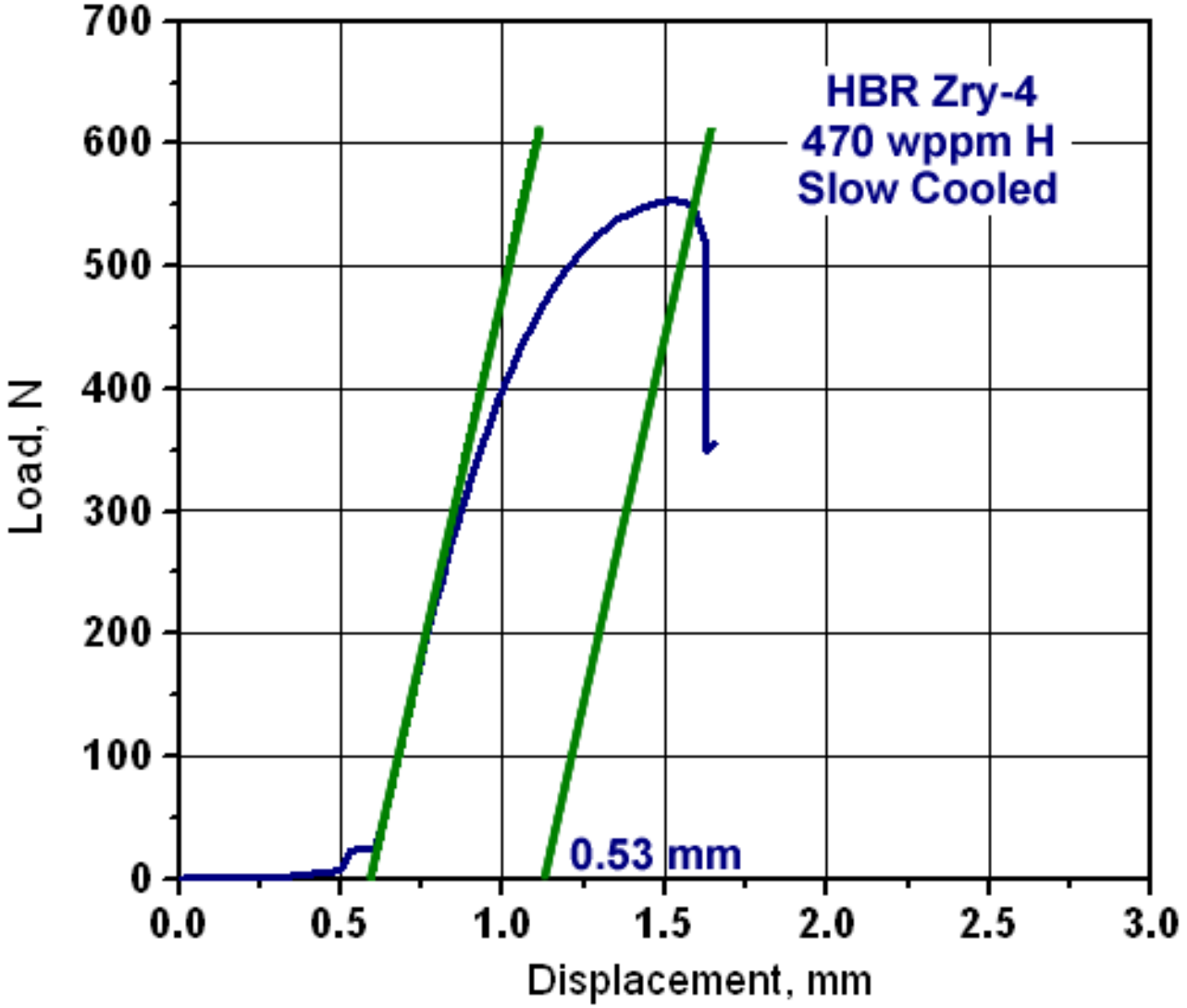

Figure B-8. Load-displacement results for ring #2 (7.05 mm long) of a prehydrided (470±30 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and cooled to room temperature without quench. The posttest sample had a tight through-wall crack at the loading location. Offset and permanent displacements were 0.53 mm and 0.39 mm, respectively.

APPENDIX C

RELATIONSHIP BETWEEN OFFSET STRAIN AND PERMANENT STRAIN

For as-fabricated cladding compressed at room temperature (RT) or at 135 degrees Celsius (ºC) and at 0.033 millimeters/second (mm/s) to a total displacement of 2 mm, the difference between offset displacement and permanent displacement is ≤0.2 mm, which corresponds to a strain difference of ≈2%. As the applied displacement is decreased, the plastic deformation decreases, and the deviation between offset and permanent strain also decreases. This was demonstrated by conducting a set of ring-compression tests designed to result in low permanent strains of 1.0 to 2.3%. Table C-1 shows the results of these tests.

Table C-1. Results of Ring-Compression Tests Conducted with As-Fabricated Cladding Samples at RT and 2 mm/minute Displacement Rate

Total applied displacements were chosen to give low permanent strains (d_d/D_o) in the range of 1.0 to 2.3% and corresponding low offset strains.

Material (D_o, mm)	Sample ID IPS or AG No.	Offset Displacement δ_d, mm	Permanent Displacement d_d, mm	Permanent Strain d_d/D_o, %	Strain Difference $(\delta_d - d_d)/D_o$, %
15×15 Zry-4 (10.91 mm)	101B7	0.24	0.21	1.9	0.3
	101B8	0.20	0.17	1.6	0.3
	101B9	0.20	0.18	1.6	0.2
	101B10	0.16	0.14	1.3	0.2
17×17 ZIRLO™ (9.48 mm)	109D7	0.25	0.22	2.3	0.3
	109D8	0.17	0.16	1.7	0.1
	109D9	0.14	0.12	1.3	0.2
	109D10	0.14	0.12	1.3	0.2
17×17 M5 (9.48 mm)	636B2	0.18	0.19	2.0	0.0
	636B3	0.14	0.14	1.5	0.0
	636B4	0.15	0.15	1.6	0.0

For as-fabricated and prehydrided cladding oxidized at ≤1,200 ºC, the difference between offset and permanent displacement depends on both the oxidation level and the magnitude of the permanent displacement. For material with high ductility, the difference in displacements can be as high as 0.5 mm. For material with essentially no ductility, both the offset and permanent displacement values are in the "noise of uncertainty," and their difference can be as low as 0.01 mm.

However, of relevance to the determination of the ductile-to-brittle transition oxidation level is the error in offset strain as determined by the difference between offset (δ_p/D_o in %) and permanent (d_p/D_o in %) strains for permanent strains in the range of 1.0 to 2.3%. Figure C-1 summarizes the data reported in EP-Refs. 1 and 2, in Figures 2 and 3 of DG-1262, and in Table C-1. The data are plotted as a function of Cathcart-Pawel equivalent cladding reacted (CP-ECR). Low values of permanent strain at low CP-ECR levels (e.g., 5–10%) are from prehydrided Zircaloy (Zry)-4 and high-burnup Zry-4 and ZIRLO™ samples. Low values of permanent strain at intermediate CP-ECR levels (10–18%) are from high-burnup ZIRLO™ and M5 samples. Low values of permanent strain at high ECR values (15–20%) are from as-fabricated cladding materials. The following equation gives the best linear fit to the data:

$$\delta_p/D_o - d_p/D_o = 0.25 + 0.0863 \text{ CP-ECR} \qquad \text{(C1)}$$

The one-sigma upper bound to the data is given by:

$$\delta_p/D_o - d_p/D_o = 0.41 + 0.1082 \text{ CP-ECR} \quad \text{(C2)}$$

Because of the large data scatter in Figure C-1, the one-sigma upper bound is used to establish the offset-strain ductility criterion. It is derived by setting the permanent strain (d_p/D_o) in Equation C2 to 1.0%:

$$\delta_p/D_o \geq 1.41 + 0.1082 \text{ CP-ECR} \quad \text{(C3)}$$

For multiple offset-strain data points at the same CP-ECR level, the average value for the dataset, rounded to the nearest tenth of a percent, should be used for δ_p/D_o in Equation C3. Similarly, the limit calculated from the right-hand side of Equation C3 should also be rounded to the nearest tenth of a percent.

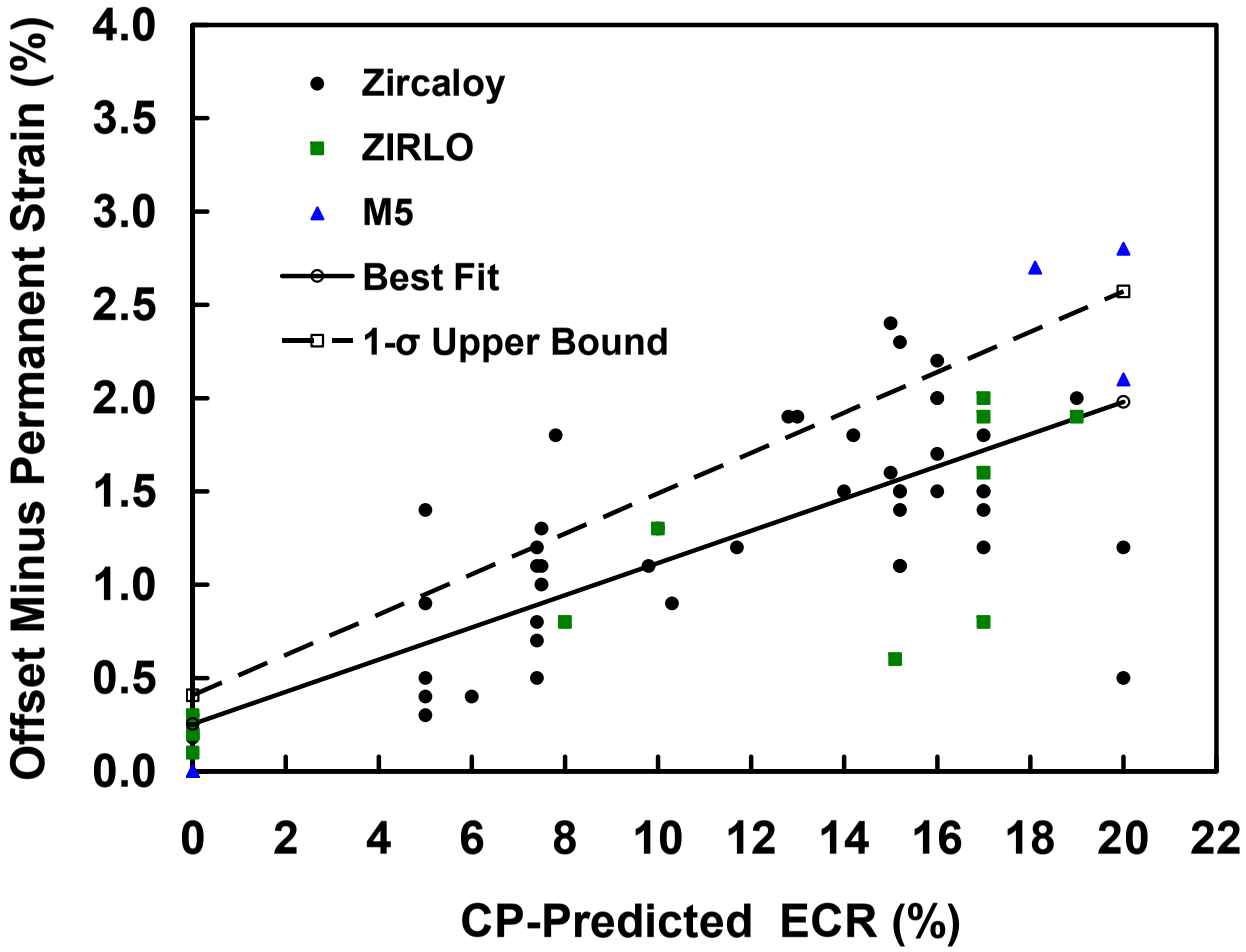

Figure C-1. Difference in offset strain and permanent strain as a function of calculated oxidation level (CP-ECR) for permanent strains near the embrittlement threshold (1.0 to 2.3%) for as-fabricated, prehydrided, and high-burnup cladding alloys oxidized at 1,200 ºC and ring-compressed at room temperature and 135 ºC and at 0.033 mm/s.

References[1]

1. M. Billone, Y. Yan, T. Burtseva, and R. Daum, "Cladding Embrittlement during Postulated Loss-of-Coolant Accidents," NUREG/CR-6967, July 2008 (ADAMS Accession No. ML082130389).

2. Y. Yan, T.A. Burtseva, and M.C. Billone, "Post-quench Ductility Results for North Anna High-burnup 17×17 ZIRLO Cladding with Intermediate Hydrogen Content," ANL letter report to NRC, April 17, 2009 (ADAMS Accession No. ML091200702).

[1] Publicly available NRC published documents are available electronically through the NRC Library at: http://www.nrc.gov/reading-rm/doc-collections/. The documents can also be viewed on-line or printed for a fee in the NRC's Public Document Room (PDR) at 11555 Rockville Pike, Rockville, MD; the mailing address is USNRC PDR, Washington, DC 20555; telephone 301-415-4737 or (800) 397-4209; fax (301) 415-3548; and e-mail pdr.resource@nrc.gov.