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DETERMINING POST QUENCH DUCTILITY

Note: Public availability of this draft document is intended to inform stakeholders of the current status of the NRC staff's preliminary draft final rule package and associated documents for § 50.46c of Title 10 of the Code of Federal Regulations (10 CFR). These preliminary draft documents are in support of a February 4, 2016, Advisory Committee on Reactor Safeguards (ACRS) subcommittee meeting. This draft document has not been subject to all levels of NRC management review. Accordingly, the document may be subject to further revision before the staff provides the final draft rule language package to the Commission (currently scheduled to be provided to the Commission in February 2016).

A. INTRODUCTION

Purpose

This regulatory guide (RG) describes an experimental technique that the U.S. Nuclear Regulatory Commission (NRC) accepts in the implementation of the requirements of Title 10 of the *Code of Federal Regulations* (10 CFR) 50.46c, "Emergency Core Cooling System Performance during Loss of Coolant Accidents (LOCA)" (Ref. 1), subsection (g), "Fuel system designs: uranium oxide or mixed uranium-plutonium oxide pellets within cylindrical zirconium-alloy cladding." This RG offers an acceptable experimental technique for measuring the ductile-to-brittle transition for a zirconium-based cladding alloy. The experimental technique uses ring compression testing of zirconium-based cladding alloys following exposure to oxidation and quench conditions related to a LOCA.

Applicable Rules and Regulations

- Regulations contained in 10 CFR 50.46c require analytical limits on peak cladding temperature and integral time at temperature be established that correspond to the measured ductile-to-brittle transition for the zirconium-alloy cladding material based on an NRC-approved experimental technique.

Written suggestions regarding this guide or development of new guides may be submitted through the NRC's public Web site under the Regulatory Guides document collection of the NRC Library at http://www.nrc.gov/reading-rm/doc-collections/reg-guides/contactus.html.

Electronic copies of this regulatory guide, previous versions of this guide, and other recently issued guides are available through the NRC's public Web site under the Regulatory Guides document collection of the NRC Library at http://www.nrc.gov/reading-rm/doc-collections/. The regulatory guide is also available through the NRC's Agencywide Documents Access and Management System (ADAMS) at http://www.nrc.gov/reading-rm/adams.html, under ADAMS Accession No. ML15238B079. The regulatory analysis may be found in ADAMS under Accession No. ML15323A122 and the staff responses to the public comments on DG-1262 may be found under ADAMS Accession No. ML15238B193.

Related Guidance

- RG 1.222, "Measuring for Breakaway Oxidation Behavior" (Ref. 2), describes a method that NRC staff considers acceptable to measure and periodically confirm the breakaway oxidation behavior of a zirconium-alloy cladding material.

- RG 1.224, "Establishing Analytical Limits for Zirconium-Alloy Cladding Material" (Ref. 3), describes acceptable methods to establish analytical limits on peak cladding temperature, integral time-at-temperature and limits related to breakaway oxidation behavior for zirconium-alloy cladding material.

Purpose of Regulatory Guides

The NRC issues RGs 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. Methods and solutions that differ from those set forth in RGs will be deemed acceptable if they provide a basis for the findings required for the issuance or continuance of a permit or license by the Commission.

Paperwork Reduction Act

This regulatory guide contains and references information collections covered by 10 CFR Part 50 that are subject to the Paperwork Reduction Act of 1995 (44 U.S.C. 3501 et seq.). These information collections were approved by the Office of Management and Budget (OMB), control number 3150-0011.

Public Protection Notification

The NRC may not conduct or sponsor, and a person is not required to respond to, a request for information or an information collection requirement unless the requesting document displays a currently valid OMB control number.

B. DISCUSSION

Reason for Issuance

This regulatory guide was developed to support the performance-based rule language in 10 CFR 50.46c. The rule requires that analytical limits on peak cladding temperature and integral time at temperature be established that correspond to the measured ductile-to-brittle transition for the zirconium-alloy cladding material based on an experimental technique approved by the NRC. This guide provides an experimental technique that is acceptable for generating data to support the analytical limits on peak cladding temperature and integral time at temperature for zirconium based cladding alloys.

Background

In the 1973 version of the emergency core cooling system (ECCS) performance rule, the preservation of cladding ductility, through compliance with regulatory criteria for peak cladding temperature and local cladding oxidation, offered a level of assurance that fuel cladding would not experience gross failure. Hence, the fuel rods would remain within their coolable lattice arrays.

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) (See NUREG/CR-6967, "Cladding Embrittlement during Postulated Loss-of-Coolant Accidents" (Ref. 4)), as well as jointly funded programs at the Kurchatov Institute (Ref. 5) and the Halden Reactor project (Ref. 6), 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" (Ref. 7).

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 associated with high-temperature steam oxidation (in contrast to the finding that alloy composition can have a significant effect on breakaway oxidation behavior), but cladding corrosion, which occurs as fuel burnup increases, has a substantial effect. One of the major findings of the NRC's research program was that hydrogen, which is absorbed in the cladding because of cladding corrosion, has a significant influence on embrittlement during a postulated accident. An implication of this finding is that the current combination of peak cladding temperature (1,204 degrees Celsius (C), or 2,200 degrees Fahrenheit (F)) and local cladding oxidation (17 percent equivalent cladding reacted (ECR)) criteria established in 10 CFR 50.46 may not always ensure post quench ductility (PQD).

As explained in Section 1.4 of NUREG/CR-6967, 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 1,204 degrees C (2,200 degrees F), embrittlement can occur for times corresponding to less than 17 percent oxidation in corroded cladding with significant hydrogen pickup.

Figure 1 illustrates the effect of hydrogen on ring compression test (RCT) ductility measurements. Test specimens included high-burnup Zircaloy (Zry)-4 (corrosion-layer thickness of 71–74 micrometers) and as-fabricated H.B. Robinson (HBR)-type 15×15 Zry-4. 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 percent.

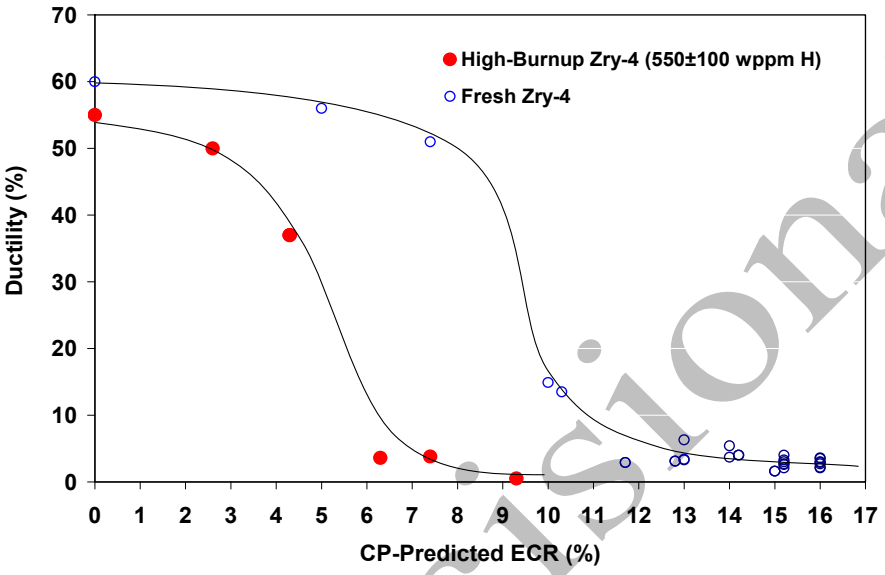

Figure 1. Measured offset strains

The NRC's LOCA research program included tests with as-fabricated, pre-hydrided and irradiated material of a number of different zirconium cladding alloys. Zirconium cladding samples were subjected to oxidation in steam at less than or equal to 1,200 degrees C (2,192 degrees F) and quench at less than or equal to 800 degrees C (1,472 degrees F). The test conditions established in the guidance were selected with the objective of bounding the performance of ECCS designs. The peak cladding oxidation temperature of 1,200 degrees C (2,192 degrees F) was selected because 10 CFR 50.46c (g)(1)(i) "Peak cladding temperature," establishes a requirement that fuel element cladding temperature shall not exceed 1204 degrees C (2,200 degrees Fahrenheit). The oxidation level was defined as the ECR calculated using the Cathcart-Pawel weight gain correlation (this correlation is documented in Ref. 8). Ring compression tests were conducted at a temperature of 135 degrees C (275 degrees F) to measure ductility following oxidation. Retention of ductility was defined as the accumulation of greater than or equal to 1.0 percent permanent strain before failure during ring-compression loading. During the 1973 hearing, investigators suggested that a test temperature no higher than the saturation temperature during reflood (i.e., about 135 degrees C or 275 degrees F) be considered. This test condition is considered relevant for current light-water reactor ECCSs. It may be necessary to evaluate and possibly modify the oxidation conditions and ring compression test temperature accordingly for the ECCSs of new reactor designs. Section C of this RG offers guidance on justifying the proposed methods for advanced designs.

The results were combined to define 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 embrittled during the heating ramp. RG 1.224 includes an analytical limit on peak cladding temperature and integral time at temperature for the post-quench ductility requirements in (g)(1)(ii) developed from the ANL results.

Some ECCSs may perform such that the maximum oxidation temperature is significantly below the peak oxidation temperature of the ANL tests. Oxidation at lower temperatures has been shown to increase the allowable calculated oxidation before embrittlement. Therefore, conducting tests at lower peak temperatures may supply added margin for some zirconium-based cladding alloys.

The rule in 10 CFR 50.46c considers the findings of the NRC's LOCA research program and requires that analytical limits on peak cladding temperature and integral time at temperature are established, which correspond to the measured ductile-to-brittle transition for the Zr-alloy cladding material.

Harmonization with International Standards

The International Atomic Energy Agency (IAEA) has established a series of safety guides and standards constituting a high level of safety for protecting people and the environment. IAEA safety guides present international good practices and increasingly reflects best practices to help users striving to achieve high levels of safety. Pertinent to this RG, IAEA Safety Guide NS-G-1.9, "Design of the Reactor Coolant System and Associated Systems in Nuclear Power Plants" (Ref. 9), issued September 2004, provides recommendations and guidance to regulatory bodies, nuclear power plant designers and licensees on the design of the Reactor Coolant Systems and associated systems, including the ECCS in order to maintain the integrity of the fuel cladding. This RG describes testing related to the development of analytical limits for ECCS performance evaluation and is consistent with the basics safety principles provided in IAEA Safety Guide NS-G-1.9.

Documents Discussed in Staff Regulatory Guidance

This regulatory guide endorses, in part, using one or more codes or standards developed by external organizations, and other third party guidance documents. These codes, standards, and third-party guidance documents may contain references to other codes, standards, or third-party guidance documents ("secondary references"). If a secondary reference has itself been incorporated by reference into NRC regulations as a requirement, then licensees and applicants must comply with that standard as set forth in the regulation. If the secondary reference has been endorsed in a regulatory guide as an acceptable approach for meeting an NRC requirement, then the standard constitutes a method acceptable to the NRC staff for meeting that regulatory requirement as described in the specific regulatory guide. If the secondary reference has neither been incorporated by reference into NRC regulations nor endorsed in a regulatory guide, then the secondary reference is neither a legally binding requirement nor a "generic" NRC-approved acceptable approach for meeting an NRC requirement. However, licensees and applicants may consider and use the information in the secondary reference, if appropriately justified, consistent with current regulatory practice, and consistent with applicable NRC requirements.

C. STAFF REGULATORY GUIDANCE

This RG defines an acceptable experimental technique that can be used to measure the ductile-to-brittle transition for a zirconium-based alloy cladding to establish specified and acceptable analytical limits on peak cladding temperature and integrated time at temperature, as required by 10 CFR 50.46c. This RG is applicable for plants that are equipped with emergency core cooling systems bounded by the oxidation conditions of a peak cladding temperature of 1,204 degrees C (2,200 degrees F) and quench temperature of 800 degrees C (1,472 degrees F). The experimental technique is acceptable for measuring the ductile-to-brittle transition for a zirconium-based cladding alloy by RCT, which can be used to (1) justify using the analytical limit on peak cladding temperature and integral time at temperature (associated with the post-quench ductility requirements in (g)(1)(ii)) defined in RG 1.224 for alloys not tested in the NRC's LOCA research program, (2) support the development of an alternative limit that is unique to a specific zirconium-based cladding alloy, or (3) support the development of analytical limits at peak oxidation temperatures less than 1,204 degrees C (2,200 degrees F).

The method of documentation of a new fuel design is in a license amendment request or vendor topical report that is submitted to the NRC for review and approval.

(1) This amendment or report should include all technical information, justifications, etc., related to testing to measure the ductile-to-brittle transition for a zirconium cladding alloy.

   (a) The ductile-to-brittle transition oxidation level should be defined as a function of hydrogen content and either the hold temperature or the maximum oxidation temperature. This is applicable for samples that embrittle during the heating ramp following the experimental procedure supplied in Appendix A, "Procedure for Conducting Oxidation and Post-Quench Ductility Tests with Zirconium-Based Cladding Alloys."

   (b) The results of testing supporting establishment of an analytical limit on peak cladding temperature and integral time at temperature should be provided.

D. IMPLEMENTATION

The purpose of this section is to provide information on how applicants and licensees[1] may use this guide and information regarding the NRC's plans for using this RG. In addition, it describes how the NRC staff complies with 10 CFR 50.109, "Backfitting" and any applicable finality provisions in 10 CFR Part 52, "Licenses, Certifications, and Approvals for Nuclear Power Plants."

Use by Applicants and Licensees

Applicants and licensees may voluntarily[2] use the guidance in this document to demonstrate compliance with the underlying NRC regulations. Methods or solutions that differ from those described in this RG 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. Current licensees may continue to use guidance the NRC found acceptable for complying with the identified regulations as long as their current licensing basis remains unchanged.

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

Use by NRC Staff

The NRC staff does not intend or approve any imposition or backfitting of the guidance in this RG. The NRC staff does not expect any existing licensee to use or commit to using the guidance in this RG, 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 RG. Examples of such unplanned NRC regulatory actions include issuance of an order requiring the use of the RG, requests for information under 10 CFR 50.54(f) as to whether a licensee intends to commit to use of this RG, generic communication, or promulgation of a rule requiring the use of this RG without further backfit consideration.

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 RG and (2) the specific subject matter of this RG 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

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1 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.

2 In this section, "voluntary" and "voluntarily" mean 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.

guidance in this RG 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.

Additionally, an existing applicant may be required to comply with new rules, orders, or guidance if 10 CFR 50.109(a)(3) applies.

If a licensee believes that the NRC is either using this RG 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 NRC Management Directive 8.4, "Management of Facility-Specific Backfitting and Information Collection" (Ref. 10), and NUREG-1409, "Backfitting Guidelines" (Ref. 11).

GLOSSARY

alpha layer The zirconium phase that is characterized by a hexagonally close-packed crystal structure and is stable at room temperature.

beta layer The zirconium phase that is characterized by a cubic crystal structure.

breakaway oxidation The fuel-cladding oxidation phenomenon in which the oxygen weight gain rate deviates from normal oxidation 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 The formation of a zirconium oxide layer resulting from the reaction of zirconium with coolant water during normal operation.

loss-of-coolant accident A postulated accident that would result from in 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 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 (about 30 to 50 percent) 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.

permanent strain The difference between the post-test 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 The oxide phase that develops during normal operation and is neither fully dense nor protective with respect to hydrogen pickup. Although the oxide phase that typically develops under LOCA conditions is the tetragonal oxide phase, conditions might occur during a small-break LOCA (such as extended time-at-temperature around 1,000 degrees C, or 1,832 degrees F) that promote a transformation to the monoclinic phase.

tetragonal oxide The oxide phase that develops under LOCA conditions that is dense and adherent and that is observed to be protective with respect to hydrogen pickup.

REFERENCES[3]

1. U.S. Nuclear Regulatory Commission (NRC), Proposed Rule, "Performance-Based Emergency Core Cooling Systems Cladding Acceptance Criteria," Washington, DC, March 2014. (Agencywide Documents Access and Management System (ADAMS) Accession No. ML12283A174).

2. NRC, Regulatory Guide 1.222, "Conducting Testing for Breakaway Oxidation Behavior," Washington, DC, DATE.

3. NRC, Regulatory Guide 1.224, "Establishing Analytical Limits for Zirconium-Alloy Cladding Material," Washington, DC, DATE.

4. NRC, NUREG/CR-6967, "Cladding Embrittlement during Postulated Loss-of-Coolant Accidents," Washington, DC, July 2008.

5. NRC, NUREG/IA-0211, "Experimental Study of Embrittlement of Zr-1%Nb VVER Cladding under LOCA-Relevant Conditions," Washington, DC, March 2005.

6. Institute for Energy Technology, 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," Institute for Energy Technology, Kjeller, Norway, April 2008 (ADAMS Accession No. ML081750715).

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

8. NRC, ORNL/NUREG-17, "Zirconium Metal-Water Oxidation Kinetics IV. Reaction Rate Studies," Washington, DC, August 1977 (ADAMS Accession No. ML052230079).

9. IAEA Safety Guide NS-G-1.9, "Design of the Reactor Coolant System and Associated Systems in Nuclear Power Plants," issued September 2004.[4, 5]

10. NRC Management Directive 8.4, "Management of Facility-Specific Backfitting and Information Collection."

11. NRC, NUREG-1409, "Backfitting Guidelines," Washington, DC, July 1990.

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[3] All NRC documents that are publicly available may be accessed through the Electronic Reading Room on the NRC's public Website at http://www.nrc.gov/reading-rm/doc-collections/ and through the NRC's Agencywide Documents Access and Management System (ADAMS) at http://www.nrc.gov/reading-rm/adams.html The documents can also be viewed online or printed for a fee in the NRC's Public Document Room (PDR) at 11555 Rockville Pike, Rockville, MD. For problems with ADAMS, contact the PDR staff at 301-415-4737 or 800-397-4209; fax 301-415-3548; or e-mail pdr.resource@nrc.gov.

[4] Copies of International Atomic Energy Agency (IAEA) documents may be obtained through its Web site: www.iaea.Org/ or by writing the International Atomic Energy Agency P.O. Box 100 Wagramer Strasse 5, A-1400 Vienna, Austria. Telephone +431-2600-0, fax +431-2600-7, or e-Mail at Official.Mail@IAEA.Org

[5] A copy of this document is available for review by the public at the NRC's Technical Library, by appointment, which is located at Two White Flint North, 11545 Rockville Pike, Rockville, Maryland 20852; telephone: 301-415-7000; e-mail: Library.Resource@nrc.gov.

APPENDIX A

PROCEDURE FOR CONDUCTING OXIDATION AND POST-QUENCH DUCTILITY TESTS WITH ZIRCONIUM-BASED CLADDING ALLOYS

A-1. Purpose and Scope of the Tests

Performance-based tests are needed to ensure that fuel rod cladding retains ductility after oxidation in steam at less than or equal to 1,200 degrees Celsius (C), or 2,192 degrees Fahrenheit (F) and quench at less than or equal to 800 degrees C (1,472 degrees F). 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 in this procedure 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 equivalent cladding reacted (ECR) calculated using the Cathcart-Pawel (CP) weight gain correlation. Retention of post-quench ductility (PQD) is defined as the accumulation of greater than or equal to1.0 percent permanent strain before failure during ring-compression loading at a temperature of 135 degrees C (275 degrees F) and a displacement rate of 0.033 millimeters per second (mm)/s (0.001 inches per second (in.)/s. The ductile-to-brittle transition oxidation level is defined as the maximum CP-ECR (rounded to the nearest tenth of a percent) for which ductility is retained.

A-2. Background

During a loss-of-coolant accident (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., less than 0.6 wt. percent). This is close to the oxygen solubility limit at 1,200 degrees C (2,192 degrees F) for the beta layer of as-fabricated cladding. As such, unirradiated, as-fabricated modern cladding alloys used in U.S. reactors and oxidized at 1,200 degrees C (2,192 degrees F) will retain ductility up to a time-at-temperature corresponding to a calculated oxidation level of 17-20 percent CP-ECR (for more information, see NUREG/CR-6967, “Cladding Embrittlement during Postulated Loss of Coolant Accidents” (Ref. A-1)), where CP refers to using the Cathcart-Pawel weight-gain correlation (ORNL/NUREG-17, “Zirconium Metal-Water Oxidation Kinetics IV. Reaction Rate Studies” [Ref. A-2]) 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 zirconium dioxide layer on the outside cladding diameter. The calculated maximum local oxidation limit is used as a surrogate to limit 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 dependencies. In particular, for as-fabricated alloys tested at 1,200 degrees C (2,192 degrees F), there is a linear correlation for CP-ECR less than or equal to 17 percent 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 degrees C (1,832 degrees F), 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 well with the increase in CP-predicted ECR.

Hydrogen pickup during reactor operation can cause a significant decrease in the ductile-to-brittle transition oxidation level (e.g., from 19 percent to 5 percent for 550 weight parts per million (wppm) hydrogen, NUREG/CR-6967). 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 could be adhering to this bond. This burnup-dependent phenomenon results in an oxygen-stabilized alpha layer on the inner surface and extra 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 (about 75-100 mm, or about 3-4 in.), 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 that experiences delayed inner-surface oxide formation. The procedure outlined in this Appendix offers 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 test using non-pressurized and non-deformed cladding samples.

A-3. Sample Selection and Testing Frequency

A-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 unirradiated cladding after polishing and cleaning processes. The one exception would be if post-polishing cleaning at the fuel fabrication facility includes etching with a hydrofluoric (HF)-containing acid mixture. If an HF mixture is used, 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 degrees C (2,192 degrees F).

A-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.

A-4. Sample Preparation and Characterization

A-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 proprietary. However, based on reported results, the methods used result in samples with relatively uniform (less than 10 percent variation) hydrogen concentration along the axis and circumference of the sample. As shown by F. Nagase et. al. in, "Hydrogen Morphology and Hydrogen Embrittlement of Zircaloy Fuel Cladding Used in NSRR/HBO Experiment" (Ref. A-3), the pretest radial distribution of hydrogen is relatively unimportant as hydrogen homogenizes across the cladding wall very quickly for temperatures less than or equal to 900 degrees C (1,652 degrees F).

There are several ways to measure hydrogen content in metals. Vacuum fusion is one method. An acceptable method is documented in ASTM E1447-09, "Standard Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity/Infrared Detection Method" (Ref. A-4). If a method other than ASTM E1447-09 is used, the method should be documented and available for inspection.

ASTM E1447-09 was used to determine the hydrogen content in other metals, such as zirconium alloys. For additional information, the detailed procedure used to generate the results in NUREG/CR 6967 is documented by T. Burtseva in "Procedure for Hydrogen Analysis of Refractory Metals" (Ref. A-5).

The necessary instrumentation (e.g., LECO RH 404 hydrogen determinator) and calibration standards (titanium calibration coupons with known hydrogen content) should be traceable to the National Institute of Standards and Technology (NIST), or another internationally recognized standards organization and proof of traceability should be available for inspection. Titanium coupons with hydrogen contents in the range of the zirconium cladding alloy's anticipated hydrogen content should be used for calibration. Standard titanium coupons are available directly from NIST. Following data-generation during the course of a day, it is required that a calibration standard coupon be tested as an unknown to verify that the machine maintained its calibration (i.e., no significant change to calibration constant K) during the data-generation tests. Verification consists of ensuring that the post-test measurement is within the mean ± two-sigma (e.g., 218 ± 9.5 wppm or within the range of 209 to 227 wppm). If this is not the case, then data generated during the course of the day is considered to be invalid.

A-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 (0.98 in.). If material availability is not a significant issue, a length of 30 mm (1.18 in.) is beneficial, because it will allow 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 plus or minus10 degrees C (18 degrees F) 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 (2.95 in.). 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 (2.95 in.) 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.

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

The procedures from the manufacturer's manual of a welding device should be followed 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. For one-sided oxidation samples with welded end-caps, it will be difficult to accurately determine the weight gain of the test sample. Section A-6.3 of this regulatory guide (RG) specifies that only two-side oxidation samples should be used for weight gain benchmarking.

A-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 (0.372-0.374 in.). The outer diameter should be measured in millimeters to two decimal places (e.g., 9.51mm, or 0.374 in.) based on the average of the maximum and minimum diameters. For cladding with a nominal wall thickness of 0.57 mm (0.0224), the actual wall thickness can vary from about 0.56 to 0.60 mm (0.0220-0.0236 in). Wall thickness should be determined for each sample to two decimal places (in mm) based on four readings at locations about 90 degrees apart. The sample length should be measured and recorded to one decimal place accuracy (e.g., 25.1 mm, or 0.988 in.). 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 plus or minus 5 degrees 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.

A-4.5 Pretest Cleaning with Chemical Detergent or Organic Solvent and Rinsing

The cleaning procedures described in Section X1.2 of 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 degrees F [360 degrees C] or in Steam at 750 degrees F [400 degrees C]," should be followed for oxidation-quench test sample preparation (Ref. A-6). Specifications and requirements in Sections X1.1 ("Tubes with a Second Material on Inner Diameter") and X1.3 ("Etching") should be ignored. Caution should be exercised when using a hydrofluoric-containing acid mixture as part of the test cleaning process. Light etching with hydrofluoric-containing acid can be used to facilitate pre-hydriding, as long as samples are polished before the oxidation tests. 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.

A-4.6 Pretest Sample Weight Measurement (after Drying)

Pretest sample weight should be measured to the nearest 0.1 milligram (mg). 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 laboratory grade 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 steam flow conditions.

A-5. Temperature Heat up and Cool down Rates and Heating Methods

A-5.1 Temperature Heat up and Cool down Rates

An initial sample hold temperature should be established between 100 and 300 degrees C (212 and 572 degrees F). The heating rate from the initial sample hold temperature to 1,000 degrees C (1,832 degrees F) should be relatively fast (greater than 20 degrees C /s (36 degrees F/s) or less than 35 seconds to reach 1,000 degrees C (1,832 degrees F)), and the heating rate from 1,000 degrees C (1,832 degrees F) to 1,200 degrees C should be greater than 2 degrees C /s (3.6 degrees F/s) (less than 100-second duration). Temperature overshoot during the heat up should be limited to less than or equal to 20 degrees C (36 degrees F/s) for less than or equal to 20 seconds. The cooling rate to the quench temperature should be greater than 2 degrees C /s (e.g., less than 200 seconds from 1,200 degrees C to 800 degrees C (2,192 degrees F to 1,472 degrees F)). The recommended quench temperature is 800 degrees C (1,472 degrees F). As the target oxidation temperature is decreased from 1,200 degrees C (1,472 degrees F) to 1,100 degrees C (2,012 degrees F) to 1,000 degrees C (1,832 degrees F), the embrittlement oxidation level becomes less sensitive to heating rate. However, to standardize heating rates for these lower temperatures, the heat up and cool down rates specified above for 1,200 degrees C 2,192 degrees F) tests should also be used. The heating rate to within 100 degrees C (180 degrees F) of the target hold temperature should be greater than 20 degrees C /s (36 degrees F/s), and the heating rate from that temperature (e.g., 1,000 degrees C (1,832 degrees F) or 900 degrees C(1,652 degrees F)) to the hold temperature should be greater than 2 degrees C /s (3.6 degrees F/s). Similarly, the cooling rate to the quench temperature (800 degrees C) should be greater than 2 degrees C /s (3.6 degrees F/s). Using slower heating rates, slower cooling rates, and lower quench temperatures should have a documented justification. However, using higher cooling rates to the quench temperature and higher quench temperatures do not have to be justified as these conditions will lead to embrittlement at lower oxidation levels. More discussion of these parameters is provided below.

For a given oxidation level and hydrogen content, the heating rate to 1,200 degrees C (2,192 degrees F) is a critical parameter. This is particularly true for samples that embrittle after short test times because the 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 degrees C (2,192 degrees F) leading to embrittlement are in the range of 300-500 seconds for cladding with a thickness 0.57-0.67 mm (0.022-0.026 in.) that is exposed to two-sided oxidation. Embrittlement times at 1,200 degrees C (2,192 degrees F) 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. Work documented in NUREG-6769 showed that heating rates slower than greater than 2 degrees C/s (3.6 degrees F) lead to higher embrittlement oxidation levels and that temperature overshoot during the heating phase can have a significant effect on embrittlement oxidation level for prehydrided samples. The cooling rate from 1,200 degrees C (2,192 degrees F) to the quench temperature (i.e., the wetting temperature at which very rapid cooling occurs) may also be important, but it is less critical than the heating rate. The recommended quench temperature is 800 degrees C (1,472 degrees F). Based on results presented in NUREG/CR-6967, 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 degrees C (1,472 degrees F), 700 degrees C (1,292 degrees F), and 600 degrees C (1,112 degrees F), as all samples were brittle. However, prehydrided samples at the same hydrogen content and CP-ECR were ductile following cooling without quench.

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 the change in temperature divided by the change in time. 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.

A-5.2 Radiant Heating

Radiant heating, 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 (greater than 10 degrees C/s (18 degrees F/s) or less than 40 seconds for cooling from 1,200 degrees C (2,192 degrees F) to 800 degrees C (1,472 degrees F)). For 25-mm-long samples, axial temperature variations are negligible, but circumferential temperature variations are in the range of 10–20 degrees C for cladding outer diameters ranging from 9.50 to 11.0 mm (0.374 to 0.433 in.). 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.

A-5.3 Resistance Heating

Resistance heating furnaces are characterized as having a larger uniform temperature zone and as having very slow heating and cooling rates, as compared to radiant heating furnaces. Controlled movement of the sample into and out of the furnace achieves faster heating and cooling rates. Benchmark tests should determine the heating and cooling rates. With proper thermal benchmarking, resistance-heating furnaces are acceptable for generating PQD specimens for ductility determination.

A-5.4 Induction Heating

Induction heating has the advantage of rapid sample heating and cooling rates Although the data from these tests appear reliable, reported weight gains for Zry-4 are about 10–12 percent lower than those predicted using the CP correlation and are in better agreement with the weight-gain correlation 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. Induction-heating furnaces may be acceptable for generating PQD samples, provided there has been a demonstration of accurate temperature control through benchmarking.

A-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.

A-6. Temperature Control and Monitoring

A-6.1 Thermocouples

For oxidation temperatures less than or equal to 1,200 degrees C (2,192 degrees F), Type S (Pt/10%Rh-Pt) or Type R TCs should be used to record temperature and control furnace power. Type K thermocouples have been shown to have much less accuracy than Type S and Type R thermocouples at high temperatures and should not be used to record and control furnace power for high-temperature steam oxidation testing. At 1200 degrees C, a typical Tolerance Value for Type K TC is plus or minus 0.75 percent (plus or minus 9 degrees C) and the Tolerance Value for Type S TC is ±0.25 percent (plus or minus 3 degrees C). Type R thermocouples have the same accuracy as Type S thermocouples. The TCs should be calibrated using instrumentation and standards that are traceable to NIST, or another internationally recognized standards organization and proof of traceability should be available for inspection. Typically, this service is supplied by the TC vendor, who, for an extra fee, supplies 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 degrees C (2,192 degrees F), 1,100 degrees C (2,012 degrees F), and 1,000 degrees C (1,832 degrees F). Copies of these certificates should accompany the data report.

A-6.2 Thermal Benchmarks

For short (e.g., 25-30 mm or 1-1.2 in.), two-sided oxidation samples, direct welding of TCs onto the sample outer surface should not be used 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 post-test 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 degrees C (2,192 degrees F), 1,100 degrees C (2,012 degrees F), and 1,000 degrees C (1,832 degrees F). 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 NUREG/CR-6967, two to three TCs (120 degrees 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 (about 11 mm or 0.43 in.) 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 or 0.37 in.), 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) might not be adequate for characterizing the heating rate of the sample. Strapped thermocouples might be acceptable for thermal benchmarking applications in resistance-heating furnaces; however, data would be needed to directly compare welded and strapped thermocouples in thermal benchmarking applications. One aspect that will be important to demonstrate is the ability of different

thermocouple attachment methods to capture the zirconium-water reaction heat, which will be more significant in low steam flow environments such as resistance furnace set ups. Samples with low thermal mass and high initial heats of oxidation, exposed to low steam flow 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 that 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 following procedure should be used to validate a thermal benchmark for irradiated material which has developed a corrosion layer:

(1) 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.

(2) Cool to 300 degrees C (572 degrees F) and repeat the thermal benchmark test using the same controller parameters as were used in (1); 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.

(3) If necessary, adjust the holder control temperature to achieve the desired hold temperature for cladding with pre-transient oxide layers.

A-6.3 Weight-Gain Benchmarks

After thermal benchmarking, two-sided samples should be tested without TCs welded onto the sample to determine the weight gain. These tests should be conducted at 1,200 degrees C (2,192 degrees F) and 1,100 degrees C (2,012 degrees F) for a test time corresponding to 10 percent CP-ECR. 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 steam flow. The results of the weight-gain benchmark tests should be documented and included in the data report.

For irradiated high-burnup material that has developed a corrosion layer, the corrosion layer will affect the oxidation kinetics early in the transient by slowing down the growth of the tetragonal oxide layer. Also, the sample may gain or lose weight depending on how well it was defueled. As such, the weight-gain benchmark is not reliable. For the testing reported in NUREG/CR-6967, which included oxidation tests with high-burnup cladding, the measured oxide layer thickness and the weight gain determined from layer thicknesses for as fabricated cladding was used to perform the weight-gain benchmark. Validation of the temperature for corroded cladding was performed during the thermal benchmarking using as-fabricated cladding and oxidized as-fabricated cladding.

If oxide spallation is seen when completing the weight gain benchmark activities, extra testing and calibration checks should be completed to identify the root cause of the issue.

A-7. Water Quality, Steam Flow Rate, and Steam Pressure

A-7.1 Water Quality

Purified water should be used for generating steam. Laboratory-grade Type I or Type II water is of sufficient purity for oxidation tests at greater than or equal to 1,000 degrees C (1,832 degrees F).

A-7.2 Steam Flow Rate

The average steam flow 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 test chamber, divided by the test time, and normalized to the net cross-sectional area of the test chamber. The average steam flow rate should be in the range of 0.8 to 30 mg/square centimeter (cm^2) s. Justification for this range is supplied in the following.

Leistikow and Schanz ("Oxidation Kinetics and Related Phenomena of Zircaloy-4 Fuel Cladding Exposed to High Temperature Steam and Hydrogen-Steam Mixtures under PWR Accident Conditions" (Ref. A-7)) and Uetsuka ("Oxidation of Zircaloy-4 under Limited Steam Supply at 1,000 and 1,300ºC" (Ref. A-8)) studied the effects of low steam flow rates on the oxidation kinetics of Zircaloy-4 at 1,000 degrees C (1,832 degrees F). Their results are summarized in Leistikow and Schanz, Figure 9 (Ref. A-7). In terms of flow rate normalized to the cross-sectional area of the test chamber, the oxidation kinetics began to decrease because of steam starvation for flow rates less than 0.05 mg/(cm^2 s). For the Leistikow and Schanz work, the sample length was 30 mm (1.18 in.) and oxidation was two-sided. Aomi, et al. (M. Aomi, M. Nakatuka, et al., "Behavior of BWR Fuel Cladding Tubes under Simulated LOCA Conditions" (Ref. A-9)) studied the relationship between weight gain and steam flow rate for oxidation temperatures up to 1,200 degrees C (2,192 degrees F). They found that the weight gain for fixed test times and temperatures was independent of steam flow rates in the range of 0.8 to 7.8 mg/(cm^2 s). Kawasaki, et al. ("Oxidation of Zircaloy-4 under High Temperature Steam Atmosphere and Its Effect on Ductility of Cladding" (Ref. A-10)) also performed high-temperature oxidation tests to determine the range of steam flow rates for which the weight gain for a given test time was independent of steam flow rate. They report this range as 3 to 28 mg/(cm^2 s).

It is desirable to have a steam flow rate higher than 0.8 mg/(cm^2 s) to reduce temperature overshoot during the heating phase for bare cladding. Although the maximum steam flow rate may not be as critical as the minimum steam flow rate, using steam flow rates greater than 30 mg/(cm^2 s) should be justified by data that indicate local temperature can be accurately recorded. It is possible that very high steam flow rates may cause TCs to record local temperatures lower than the average sample temperature because of the "fin-cooling" effect.

A-7.3 Steam Pressure

Oxidation tests for preparation of PQD samples should be conducted at a steam pressure at or slightly above atmospheric pressure.

A-8. Procedure for Oxidation and Quench Tests

The specific details of the test procedure depend on the heating furnace used. The steps below were developed for a radiant heating furnace. In some cases, generalizations that would apply to other heating and cooling methods are also offered.

A-8.1 Test Train and Test Chamber

The test train or sample holder and the test chamber form a unit that should be designed to contain the steam flow 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/test chamber does not have to be highly "leak tight" to provide a pathway for steam flow 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 non-oxidizing or limited-oxidizing material such as stainless steels or nickel alloys (e.g., Inconel 600). Particular attention should be given to direct contact of the sample with materials such as iron and nickel alloys, because of the low-temperature eutectics for zirconium and these elements. Eutectic reactions between zirconium-based alloys and test train materials must be prevented. The following documents supply insights and experience to aid in material selection.

In NUREG/CR-6967, 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 NUREG/CR-6967 to prevent eutectic reactions between zirconium-based alloys and the test train materials. Hofmann and Markiewicz ("Chemical Interactions between As-Received and Pre-Oxidized Zircaloy 4 and Inconel 718 at High Temperatures" (Ref. A-11)) studied the reaction rates and eutectics of Zry-4 and Inconel-718. They also present binary phase diagrams for zirconium-iron and zirconium-nickel, which have eutectic temperatures as low as about 930 degrees C (1,705 degrees F) and 980 degrees C (1,796 degrees F), respectively. This reference can be used to evaluate the potential for eutectic reactions.

A-8.2 Purging Test chamber and Stabilizing Steam Flow

Before heating and steam flow initiation, the test 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 steam flow, or it may be purged with low-temperature steam before the temperature ramp. If steam is used to purge the test chamber, then steam flow should be maintained for 500 seconds before the temperature ramp.

Steam flow should be initiated at a test chamber temperature of about 30 degrees C (86 degrees F). After introduction of steam into the chamber, furnace heating should commence for a pretest initial sample hold temperature between 100 and 300 degrees C (212 and 572 degrees F). When selecting the initial sample hold temperature, any known phase transition temperatures should be avoided. Stabilization of steam flow and the initial sample hold temperature should occur within 500 seconds.

Deviations from this procedure may be pursued but should have a documented justification. Deviations that could have a significant effect on test results include heating the sample to the target temperature in an inert gas before the introduction of steam flow. 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.

A-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 less than or equal to 10 degrees

C (18 degrees F), and the circumferential temperature variation should be less than or equal to 20 degrees C (36 degrees F). Other information pertinent to radiant and resistance heating methods is supplied below.

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 Ref. A-1, the temperature ramp rate for as-fabricated cladding materials was programmed to be very fast (greater than 50 ºC/s (90 degrees F/s)) from the initial sample hold temperature to within 50–100 degrees C (90 180 degrees F) of the target temperature and slow (2 to 3 degrees C/s (3.6 to 5.4 degrees F/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 degrees C (2,192 degrees F) tests were conducted with rapid heating to 1,000 degrees C (1,832 degrees F) followed by slower heating (2 to 3 degrees C/s (3.6 to 5.4 degrees F/s))to 1,200 degrees C (2,192 degrees F).

A-8.4 End of Heating Phase and Cool Down

After the target test time has been reached, furnace power should be turned off or decreased in a controlled manner while steam flow 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 steam flow should be maintained until the sample temperature reaches 800 degrees C (1,472 degrees F) plus or minus 20 ºC (36 degrees F), followed by quench water flow.

Direct quench from the target test temperature can also be used, however it is noted that this rapid cooling method has been shown to have a detrimental effect on cladding post-quench ductility. Direct quench methods are acceptable because they will produce conservative results. For direct quench cooling from the target value hold temperature, it is not necessary to maintain a prescribed cool down rate or steam cooling to a particular temperature.

A-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. The following equations should be used for conversion of CP weight gain (w_g in grams (g)/cm^2) to oxidation level (ECR in percent):

One-sided oxidation $\quad ECR = 43.9\ [(w_g/h)/(1 - h/Do)],$ (A1)

Two-sided oxidation $\quad ECR = 87.8\ w_g/h,$ (A2)

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

A-9. Post-Oxidation-Quench Measurements and Characterization

A-9.1 Sample Drying Time

To determine an accurate post-test 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 using 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.

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

The post-test sample weight should be measured to the nearest 0.1 milligram (mg). The weight gain (in mg) is determined by subtracting the pretest weight from the post-test 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 steam flow and test procedures throughout the data-generating phase of testing.

A-9.3 Hydrogen Content Measurement

If it has been demonstrated and documented that prehydrided samples have very little axial variation in hydrogen content, then post-test hydrogen analysis would not be needed. Significant axial variation is defined as greater than 30 wppm along the test sample length. For such samples, post-test hydrogen analyses could be performed using rings 2–3 mm (0.079-0.12 in.) in length, sectioned from both sides of the 8-mm-long ring-compression sample. Alternatively, post-test hydrogen analysis could be performed using the 8 mm (0.31 in.) long rings after RCT. In either case, post-test 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 (less than 20 wppm), based on the results presented in NUREG/CR-6967, as long as breakaway oxidation does not occur.

In general, post-test hydrogen analyses using LECO measurements provides the total sample hydrogen content (C_H) = (H_S/M_S) × 10^6, where H_S is the mass of hydrogen evolved from the sample and M_S is the mass of the sample. Therefore for pre-hydrided cladding samples before oxidation, the sample is all metal (note that the hydrogen-content contribution to the sample mass and density is negligible) such that the hydrogen content of the sample is the hydrogen content of the metal and the mass of the sample is the mass of the metal ($H_S = H_M$, $M_S = M_M$). In this case, the concentration of the hydrogen measured is the concentration of hydrogen in the metal ($C_H = C_{HM}$). But for samples after oxidation, the sample mass is $M_S = M_M + W_G$, where W_G is the oxidation weight gain in mass units, or $M_S = M_M (1+W_g/100)$, where W_g is the oxidation weight gain in percent (see Table A-1). Hence, the LECO-measured hydrogen content for oxidized pre-hydrided samples expressed in wppm is $(C_H)_{pox} = (H_M/[M_M+W_G]) \times 10^6 = ([H_M/M_M]/[1 + W_g/100]) \times 10^6$. This value can be multiplied by a mass-correction factor to determine C_{HM}:

$$C_{HM} = (1 + W_G/M_M)\,(C_H)_{pox} = (1 + W_g/100)\,(C_H)_{pox} \qquad \text{(A3)}$$

Table A-1. Oxidation conditions and results for pre-hydrided ZIRLO samples.

Oxidation Test ID	Sample ID	Pre-test Estimated C_{HM}, wppm	Test Time, s	CP-ECR, %	Meas. ECR, %	W_g, %	w_g, mg/cm^2
ZLUPH#3	A15C3	231±8	166	11.0	11.8	4.23	7.56
ZLUPH#4	A15C1	245±23	145	10.0	10.4	3.73	6.69
ZLUPH#5	A13C3	256±46	166	11.0	11.5	4.11	7.41
ZLUPH#6	A13C1	226±16	166	11.0	11.5	4.12	7.40
ZLUPH#7	A13C5	263±36	110	8.0	8.3	2.95	5.34
ZLUPH#8	A15C5	314±91	127	9.0	9.5	3.47	6.13

Note: W_G is the measured change in sample weight (in %) and w_g is weight gain normalized to surface area.

A-10. Matrix for Oxidation and Quench Tests

A-10.1 As-Fabricated Cladding

Based on the results presented in NUREG/CR-6967, 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 degrees C (2,012 degrees F) and 1,000 degrees C (1,832 degrees F) for oxidation levels up to 20 percent CP-ECR. The reason for this is the relatively low oxygen solubility limit in zirconium-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 greater than 20 percent CP-ECR.

At an oxidation temperature of 1,200 degrees C (2,192 degrees F), the oxygen solubility limit (e.g., 0.6 wt. % for Zry-4) in zirconium-based cladding alloys is close to the embrittlement limit at a ring-compression test temperature of 135 degrees C (275 degrees F). Cladding materials experience a significant decrease in ductility (from greater than 40 percent to less than 10 percent) in the oxidation range of 10 percent to 17 percent CP-ECR, after oxidation at 1,200 degrees C (2,192 degrees F). Thus, it is recommended that scoping tests be performed at oxidation levels of 13 percent, 17 percent, and 20 percent CP-ECR. For each oxidation sample more than 30 mm (1.18 in.) long, at least three ring-compression samples can be sectioned. Based on these results, more tests can be performed in a narrow CP-ECR range between the ECR levels where ductile and brittle results are measured. For example, if the cladding is ductile at 17 percent and brittle at 20 percent, then multiple tests should be performed at 18 percent and 19 percent CP-ECR to determine the ductile-to-brittle transition CP-ECR. The number of tests needed to characterize the transition from ductile-to-brittle behavior will vary depending on the observed data scatter. More discussion will be supplied in Section 12, “Data reporting and assessment.”

A-10.2 Pre-hydrided Cladding

For samples to be oxidized at less than or equal to 1,200 degrees C (2,192 degrees F) (i.e., greater than 2 degrees C/s (3.6 degrees F/s) heating rate from 1,000 degrees C (1,832 degrees F) to the 1,200 degrees C (2,192 degrees F) hold temperature), the ductile-to-brittle transition oxidation level is highly dependent on the hydrogen content. Therefore, it is necessary to examine the ductile-to-brittle oxidation level for cladding materials for a range of hydrogen content. The number of different hydrogen content levels that should be examined to establish an analytical limit on peak cladding temperature and integral time at temperature will vary depending on the zirconium cladding alloy. Guidance on this subject is documented in RG 1.224, “Regulatory Guidance on Establishing Analytical Limits for Zirconium-Alloy Cladding Material” (Ref. A-12). For tests with prehydrided cladding, the hydrogen contents selected should extend to the best-estimate hydrogen concentration at maximum burnup.

Pre-hydriding zirconium cladding alloys can be difficult. In particular, it can be difficult to achieve specific levels of hydrogen absorption repeatedly. However, to establish the ductile-to-brittle oxidation level, testing must be conducted at multiple oxidation levels on samples with very similar hydrogen content for comparison. Acknowledging the difficultly in pre-hydriding zirconium cladding alloys to specific hydrogen levels repeatedly, a “binning” approach can be used. Samples can be “binned” together for evaluation if the average hydrogen content of the samples is within plus or minus 10 percent or plus or minus 30 wppm, whichever is less, of the bin level.

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-zirconium 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 extend to the best-estimate hydrogen concentration at maximum burnup.

For tests conducted at the 1,200 degrees C (2,192 degrees F) hold temperature, the embrittlement threshold offered in RG-1.224 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 A-2 below gives the embrittlement threshold in RG-1.224 in tabular form for clarity. For low hydrogen contents (less than150 wppm) typical of those measured for some high-burnup zirconium alloys, the results presented in J. Mardon, et.al., "Influence of Hydrogen Simulating Burn-Up Effects on the Metallurgical and Thermal-Mechanical Behavior of M5® and Zircaloy-4 Alloys under LOCA Conditions" (Ref. A-13) may be used as a guide. J. Mardon, et.al. 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 NUREG/CR-6967 embrittlement data (e.g., 9 percent CP-ECR for 300 wppm hydrogen). Depending on the results, the second test should be conducted at a CP-ECR 2 percent higher (if ductile at 9 percent) or lower (if brittle at 9 percent). 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 A-2. 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 degrees C (2,012 degrees F) and 1,000 degrees C (1,832 degrees F) will embrittle at ECR values significantly below 17 percent. 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 1,200 degrees C (2,192 degrees F) is important if an applicant can demonstrate that calculated LOCA temperatures are significantly lower than 1,204 degrees C (2,200 degrees F).

A-11. Procedure for Conducting Ring-Compression Post-Quench Ductility Tests

A-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 use instrumentation and standards that are traceable to NIST, or another internationally recognized standards organization and proof of traceability should be available for inspection. For testing activities conducted outside of the United States, relevant recognized standards are acceptable. This service is offered by the MTS vendor (e.g., Instron), who supplies documentation of calibration verification.

The temperature sensor used to control furnace or oven power corresponding to a ring test temperature of 135 degrees C (275 degrees F) should be calibrated using instrumentation and standards that are traceable to NIST, or another internationally recognized standards organization and proof of traceability should be available for inspection. The temperature sensor vendor supplies this service for a fee and supplies a certificate of calibration along with the temperature sensor. The calibration should be performed at 135 degrees C (275 degrees F). A variety of temperature sensors could be used at this low temperature. The standard deviation between the TC reading and NIST-traceable standard is quite low (e.g., plus or minus 0.3 degrees C for room temperature (RT) to 200 degrees C (392 degrees F)). Resistance thermometers are also acceptable to control furnace or oven power at 135 degrees C (275 degrees F) for ring compression tests, provided they are calibrated to recognized standards.

It is required that two benchmark tests (one at RT and one at 135 degrees C) be conducted using as-fabricated cladding samples before PQD samples are tested if any of the following apply: (a) the MTS has been idle for a period of time greater than a month; (b) the MTS has been used to conduct other tests (e.g., axial tensile tests, bend tests, etc.) that require modification of the loading train and/or support fixture during the interval of time between conducting ring-compression tests; or (c) testing of PQD samples is conducted beyond the due date for calibration recommended by the MTS manufacturer. If condition (c) applies, the PQD data can only be reported after calibration verification indicates no significant changes in results. The procedure provided in Appendix B of this RG can be used for benchmark tests. Appendix B contains rationale, results and assessment of results for six benchmark tests (three at RT and three at 135 degrees C) routinely conducted by Argonne before data-generation tests are conducted.

Rings sectioned from LOCA oxidation-quench samples should be in the range of 7-10 mm (0.27-0.39 in.) 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 (0.3 in.). For two-sided oxidation samples, it is sufficient to cut off 1-2 mm (0.039-0.079 in.) 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.

After sectioning, the length of the rings should be measured to one decimal place in millimeters (e.g., 7.9 mm, or 0.31 in.), and the minimum and maximum diameter of the oxidized rings should be measured to two decimal places (e.g., 9.51 mm, or 0.374 in.). 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. For testing activities conducted outside of the United States, relevant recognized standards are acceptable.

A-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 degrees C (275 degrees F). For such uniform heating, it is sufficient to use a single temperature sensor in contact with the inner surface of the sample at the bottom support position. The spring-loading of the temperature

sensor 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 degrees C (275 degrees F) plus or minus 1 degrees C (1.8 degrees F). For reference, the PQD test results in NUREG/CR-6967 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 supplied 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 temperature sensor, which is in intimate contact with the sample, should be used to control furnace power to achieve a steady temperature of 135 degrees C (275 degrees F). Additional temperature sensors in positions perpendicular (90 degrees away from) the loading axis, which initially contact the sample through spring loading, should be used to determine the circumferential variation in temperature. These temperature sensors, which contact the sample outer surface with mild spring loading, are less accurate than the bottom temperature sensor. Tests should be initiated when the average deviation of the side temperature sensor readings is less than or equal to 5 degrees C (9 degrees F) relative to the 135 degrees C (275 degrees F) control temperature sensor reading. Using test temperatures higher than 135 degrees C (275 degrees F) should have a documented justification, while test temperatures lower than 135 degrees C (275 degrees F) do not require justification.

This heating and temperature monitoring method, along with an Instron Model 8511 servo-hydraulic MTS, was used to generate the NUREG/CR-6967 results for high-burnup cladding LOCA samples.

The crosshead displacement rate for ring-compression samples should be in the range of 0.0083 to 0.033 mm/s (0.5 to 2 mm/minute). These rates are slow enough to allow test termination after the first significant load drop.

A-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. 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 reference, standard 17×17 cladding with an inner diameter of about 8.3 mm (0.327 in.), the maximum displacement should be less than 6 mm (0.24 in.).

Test conduct is standard with regard to setup and operation. Reference A-14 supplies details for ring-compression tests conducted with the screw-type Instron Model 5566 used to generate data reported in NUREG/CR-6967 for as-fabricated and prehydrided cladding.

A-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 greater than 30 percent. 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 laboratory data, load drops in the range of 30-50 percent indicate a single through-wall crack, which may be very tight or loose because of recoil after test termination. For tight cracks, an accurate post-test diameter can be measured in the loading direction (see Section 12.2 for additional discussion). For a single loose crack, the post-test diameter reading is not very accurate. For

load drops of about 70-80 percent, the sample should have two cracks. For load drops of 80-100 percent, 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 supplied in Appendix B of this RG.

The more common method used in RCT is to run the test for a fixed displacement. As multiple cracks are likely to occur, no useful post-test 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.

A-11.5 Post-test 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 after a steep 30-40 percent 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 post-test ring. If the post-test 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 percent. For samples that have no post-test 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, the outer diameter of the as-fabricated cladding should be used to normalize these displacements. Pre-hydriding 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. Strain should be reported in percent as displacement divided by the diameter of the as-fabricated cladding used for oxidation or for pre-hydriding and oxidation. Based on measurement error and data scatter, these strains should be reported to one significant decimal place in millimeters. If the post-oxidation 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 post-oxidation 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 greater than 10 percent or 30 wppm, whichever is less, axial and circumferential variation in hydrogen content relative to the average hydrogen content.

A-12. Data Reporting and Assessment

A-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 the initial rapid temperature ramp 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.

A-12.2 Measuring Permanent Strain and "tight" cracks

Permanent strain can only be measured on samples characterized by "tight" cracks and cannot be measured on samples characterized by "loose" cracks. Section 11.4 describes test termination protocol that can limit cracks during ring compression testing such that an accurate post-test diameter can be measured in the loading direction. A "loose" crack can be seen by the naked eye. If a crack is "tight," it will not be possible to see the crack through the cladding wall with the naked eye. A "tight" crack feature may be observed on both the OD and ID, however the crack width is so tight that one cannot observe light pass through the wall thickness. The figures below are offered as examples of "tight" and "loose" cracks in ring compression test samples.

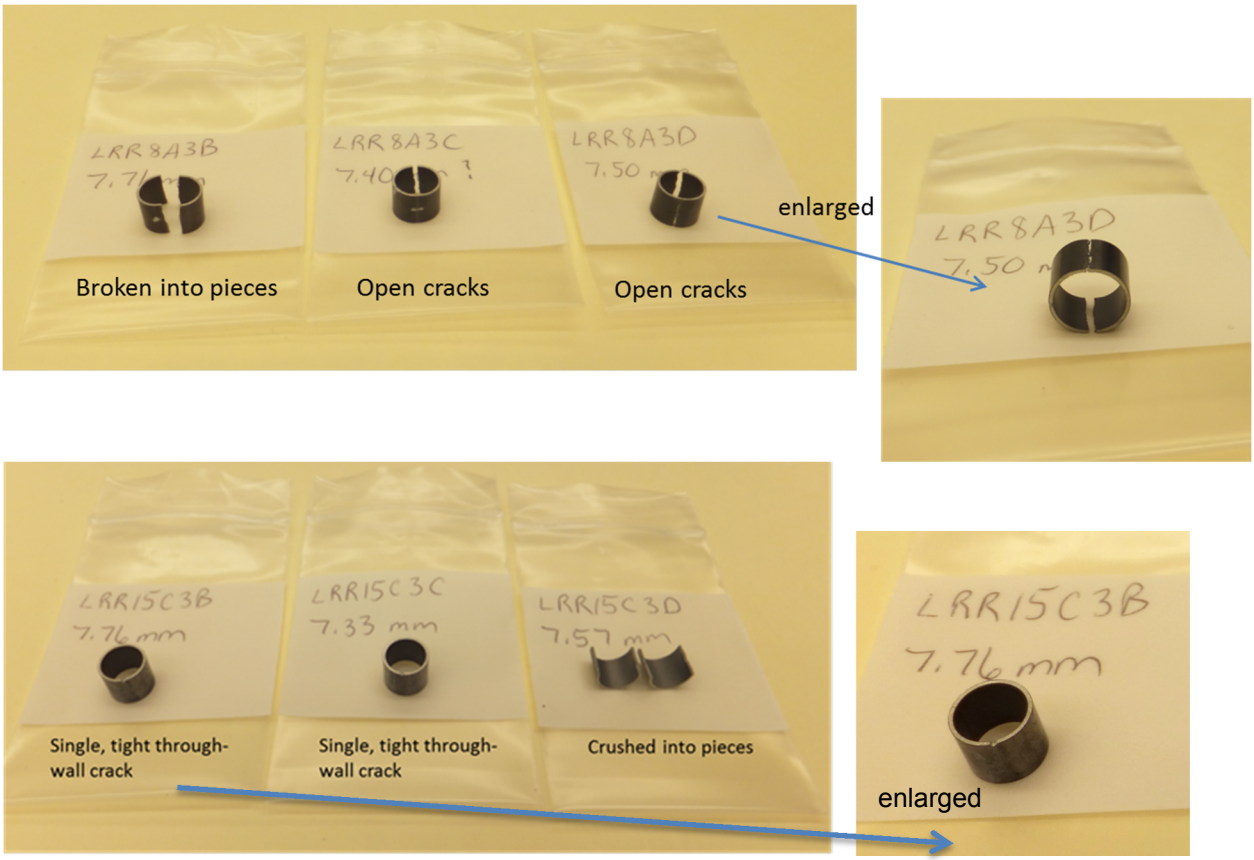

Figure A-1. Examples of "tight" and "loose" or open cracks.

A-12.3 Ductility Criteria and Basis

Rings that exhibit greater than or equal to 1.0 percent permanent strain are classified as ductile. The 1.0 percent is based on uncertainties in diameter readings, in recoil (or spring-back) of cracked rings versus intact rings, and in diameter reduction because of 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 percent, which for cladding with an outer diameter of 9.50 mm (0.374 in.) corresponds to permanent displacement of 0.2-0.8 mm (0.008-0.03 in.). 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 percent permanent strain (i.e., the maximum CP-ECR for which ductility is retained).

If ring-compression tests are not interrupted at the first significant load drop, then the ring will crack into pieces, which renders measurement of post-test 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 D of this RG presents an empirical trend curve relating offset strain measurements to permanent strain measurements. The trend curve shown in Figure D-1 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:

$$\text{Average Measured Offset Strain} \geq 1.41 + 0.1082\ \text{CP-ECR} \qquad \text{(A4)}$$

where the offset strain and CP-ECR are in units of percent.

Equation A4 gives greater than or equal to 2.0 percent at 5 percent CP-ECR and ≥3.6 percent at 20 percent CP-ECR. However, because of the large data scatter in Figure D-1 of this RG, the offset strain criterion given on the right side of Equation A4 represents the one-sigma upper bound of the data.

A-12.4 Determination of Ductile-to-Brittle Transition CP-ECR

This guide offers two approaches to determine the ductile-to-brittle transition CP-ECR from ring compression test data for as-received cladding material and for cladding material in a specific hydrogen "bin," as discussed in section 10.2.

One approach is to evaluate the ring compression test results from a single oxidation sample as a set, to define a single outcome (ductile, brittle or "transitional") at each oxidation level. With this approach, the follow guidelines should be followed:

(1) If the average of three RCT results from a single oxidation sample is ductile (average measured offset or permanent strain is above the ductility criteria relevant for that oxidation level), declare that oxidation level ductile.

   (a) Offset strain values greater than or equal to 2 percent can be averaged with other ductile results. Permanent strain values greater than or equal to 0.8 percent can be averaged with other ductile results.

   (b) If the RCT results from a single oxidation sample include both ductile and brittle (less than 2 percent offset strain or 0.8 percent permanent strain) results declare that oxidation level transitional.

(2) If the average of three RCT results from a single oxidation sample is brittle (average measured offset or permanent strain is below the ductility criteria relevant for that oxidation level), declare that oxidation level brittle.

(3) Values of hydrogen content for each ring should be measured following RCT and noted in the table.

(4) The ductile-to-brittle transition (DBT) could be determined as the CP-ECR, rounded to the nearest 0.1 percent, from interpolation between an oxidation level for which the RCT results were ductile and an oxidation level for which the RCT results were brittle and the CP-ECR values differ by no more than 2.0 percent. This method is illustrated in Figure A-2 below.

(5) Alternatively, the DBT could be determined as the CP-ECR, rounded to the nearest 0.1 percent, from interpolation between an oxidation level for which the RCT results were ductile and an oxidation level for which the RCT results were transitional and the CP-ECR values differ by no more than 1.0 percent.

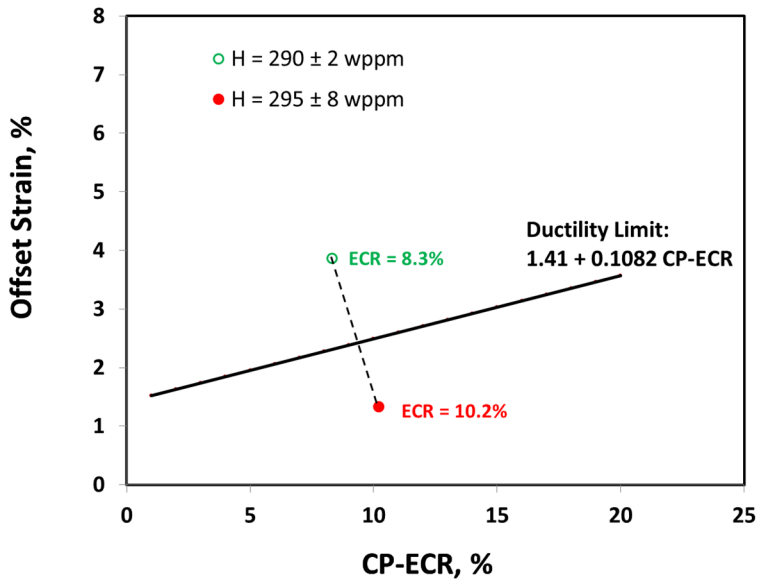

Figure A-2. Determination of ductile-to-brittle transition CP-ECR based on interpolation between ductile and brittle results measured at CP-ECR values within 2.0%.

Another approach is to use a curve fit through the full set of ring compression test data and interpolate the DBT at the intersection of the curve fit line with the appropriate ductility criteria (either the permanent strain or offset strain criteria). This approach can be used provided:

- Each data point is plotted relative to the hydrogen content measured for that ring
- RCT data are found to be both above and below the appropriate ductility criteria
- RCT data below and above the ductility criteria resulted from tests no more than 1.0 percent ECR apart

- The line is drawn through only RCT data with less than 7 percent offset or 5 percent permanent strain

Both linear and exponential fits could be used in this range to determine the intersection of the data trend with the ductility criterion, as they will produce very similar results in this range. The intersection can be rounded to the nearest 0.1 percent ECR.

This method is illustrated in the following example. Figure A-3 shows an example for as-fabricated HBR-type 15×15 Zircaloy-4. Based on multiple oxidation tests in a narrow range and multiple ring-compression samples, the permanent strains were 1.5 plus or minus 0.4 percent at 15.2 percent CP-ECR and 1.1 plus or minus 0.3 percent at 16 percent CP-ECR, where the plus or minus 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 percent. Figure A-3 includes both a linear and an exponential fit for the full set of data and it can be seen that both fits would result in a very similar intersection with the ductility criteria.

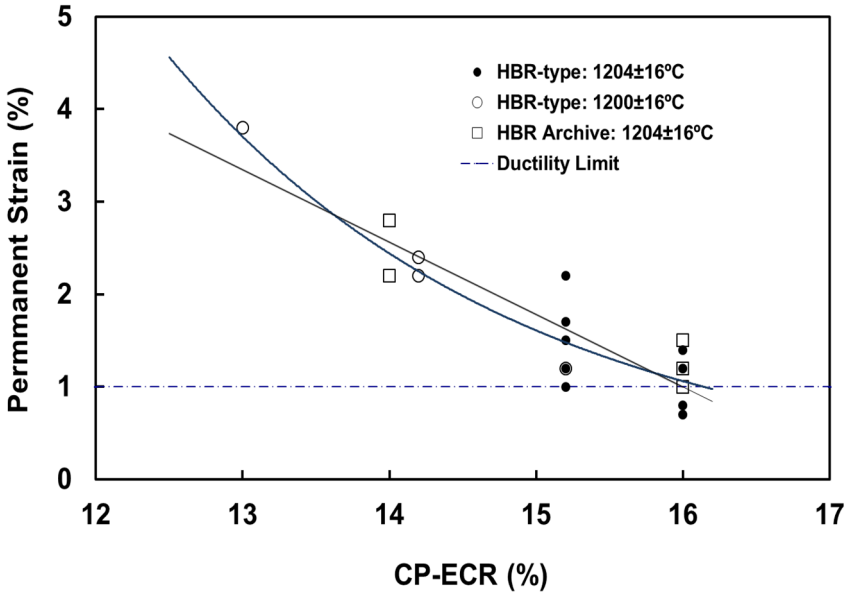

Figure A-3. Determination of ductile-to-brittle transition CP-ECR based on the permanent strain criterion for as-fabricated HBR-type 15×15 Zry-4 oxidized at ≈1,200 °C and quenched at 800 °C. The ductile-to-brittle transition oxidation level is 16% CP-ECR based on average permanent strain ≥1.0%.

The offset strains corresponding to the permanent strain data shown in Figure A-3 are plotted in Figure A-4.

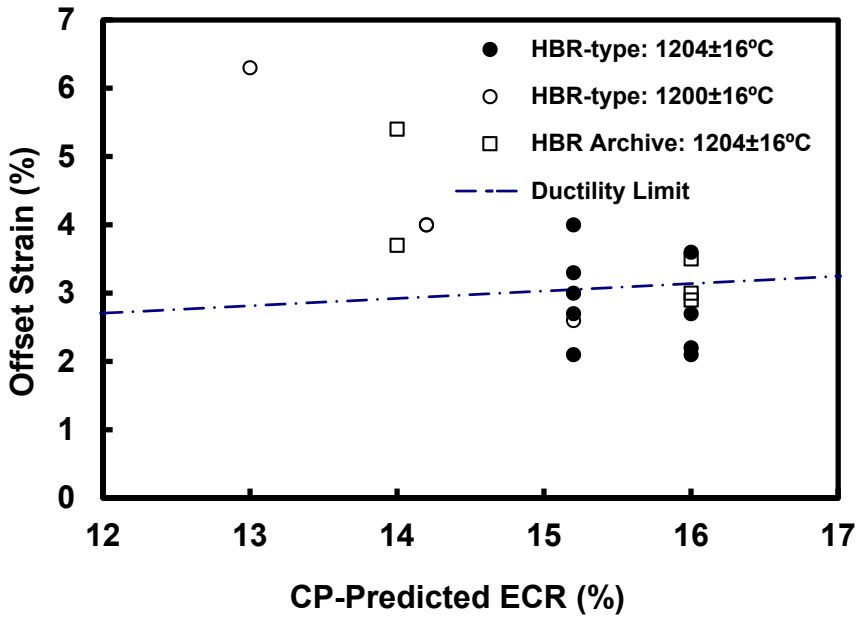

Figure A-4. Offset strains determined for as-fabricated HBR-type 15×15 Zry-4 oxidized at ≈1,200 ºC and quenched at 800 ºC (see Figure A-3 for corresponding permanent strains).

Unlike permanent strains, offset strains level off to about 3 percent at 15.2 and 16.0 CP-ECR (3.0 plus or minus 0.7 percent at 15.2 percent CP-ECR and 2.9 plus or minus 0.5 percent at 16.0 percent CP-ECR). Based on Equation A4 offset strains greater than or equal to 3.1 percent imply ductility at 15–16 percent CP-ECR. The average measured offset strains (3.0 percent) at 15.2 percent and 16 percent CP-ECR are slightly less than the ductility limit (3.1 percent). By interpolation between the data at 14.2 percent and 15.2 percent CP-ECR, the ductile-brittle-transition CP-ECR would be 15 percent. Thus, there is a penalty of 1 percent CP-ECR in using the less precise offset strain criterion as compared to the permanent strain criteria.

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1 All U.S. Nuclear Regulatory Commission (NRC) documents that are publicly available may be accessed through the Electronic Reading Room on NRC's public Web site at http://www.nrc.gov/reading-rm/doc-collections/ and through the NRC's Agencywide Documents Access and Management System (ADAMS) at http://www.nrc.gov/reading-rm/adams.html The documents can also be viewed online or printed for a fee in the NRC's Public Document Room (PDR) at 11555 Rockville Pike, Rockville, MD. For problems with ADAMS, contact the PDR staff at 301-415-4737 or 800-397-4209; fax 301 415-3548; or e-mail pdr.resource@nrc.gov.

2 A copy of this document is available for review by the public at the NRC's Technical Library, by appointment, which is located at Two White Flint North, 11545 Rockville Pike, Rockville, Maryland 20852; telephone: 301-415-7000; e-mail: Library.Resource@nrc.gov.

3 Copies of American Society for Testing and Materials (ASTM) standards may be purchased from ASTM, 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, Pennsylvania 19428-2959; telephone (610) 832-9585. Purchase information is available through the ASTM Web site at http://www.astm.org.

4 Copies of this paper may be purchased through ScienceDirect Website: http://www.sciencedirect.com

A-10. Kawasaki, S., 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*, 15(8): 589–596.[2, 5]

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A-12. Regulatory Guide 1.224, "Establishing Analytical Limits for Zirconium-Alloy Cladding Material," Washington, DC, DATE.

A-13. Mardon, J., et al., "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. [2]

A-14. Burtseva, T.A., "Procedure for Conducting Ring-Compression Tests in Laboratory DL-102A," ANL-IPS Memo, IPS-495-00-00, November 26, 2007 (ADAMS Accession No. ML15083A522).

A-15. Yan, Y., 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 U.S. Nuclear Regulatory Commission, April 17, 2009 (ADAMS Accession No. ML091200702).

[5] Copies of this paper may be purchased through Taylor & Francis Online. Website: http://www.tandfonline.com

APPENDIX B

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

T.A. Burtseva, "Procedure for Conducting Ring-Compression Tests in Laboratory DL-102A" (Ref. B-1), Document IPS-495-00-00, 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 pre-data-generation tests to ensure that the MTS, 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 post-quench 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. Conducting such benchmark tests is a recommendation and is not a requirement. The following describes the approach to interim verification/validation.

Three tests at room temperature (RT) and three tests at 135 degrees Celsius (C), or 275 degrees Fahrenheit (F), should be conducted with as-fabricated cladding rings at 2 millimeters (mm)/minute (0.033 mm/second) to a maximum crosshead displacement of 2 mm (0.08 in.). 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:

$$K_c = E\,(1.79\,[1 - \nu^2])^{-1}\,L\,(h/R)^3, \qquad \text{(B1)}$$

where E is Young's modulus in kN/mm^2, ν is Poisson's ratio, L is the length of the ring in mm, h is the wall thickness in mm, and R is the mid-wall radius in mm. Eq. B1 is applicable to the elastic behavior of long ($L/h >> 1$) thin-wall rings of uniform length, outer diameter, and wall thickness.

The reference length for the test rings is 8 mm (0.3 in.). However, offset displacement should be independent of ring length and stiffness varies linearly with ring length, and therefore actual values of sectioned rings may be within the range 8.0 plus or minus 1.0 mm (0.3 plus or minus 0.04 in.). 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 (0 degrees, 90 degrees, 180 degrees, and 270 degrees). The value of h used in Equation A1 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 B1 is calculated from the relationship: $R = (D_{oi} - h)/2$. Young's modulus, E, for cladding alloys for this evaluation was assumed to be the same as the isotropic modulus reported in MATPRO for recrystallized-annealed (RXA) Zircaloy (Zry)-4: E = 92.5 kN/mm^2 (92,500 megapascals (MPa)) at 25 degrees C (77 degrees F), and E = 86.5 kN/mm^2 (86,500 MPa) at 135 degrees C (275 degrees F). Poisson's ratio (ν) can also be derived from correlations presented in MATPRO for Young's modulus (referred to as Y in MATPRO) and shear modulus (G) according to $\nu = Y/(2\,G) - 1$. Poisson's ratio varies slowly with temperature: 0.362 at 25degrees C (77 degrees F) and 0.370 at 135degrees C (275 degrees F). (Note: use of the cold work fraction (C) greater than 0 for Y and G in MATPRO will result in spurious values for Poisson's ratio.) 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: $K_m/K_c \leq 1$. Measured stiffness values higher than values predicted by Equation B1 indicate that the load cell and/or crosshead displacement indicator may be out of calibration. Changes in K_m/K_c values over time also indicate that the MTS 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 post-test diameters in the loading direction. The ring is to be positioned in the MTS 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 δ_p greater than d_p and that the difference will be $\delta_p - d_p$ less than or equal to 0.2 mm, which is based on an extensive dataset for as-fabricated cladding displaced to 1.5–2.0 mm (0.06-0.08 in.) at RT and 135degrees C (275 degrees F) and at 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 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 (0.31 in.) by multiplying the measured stiffness by (8 mm/L): $K_{mn} = (8\ mm/L)\ K_m$. The calculated stiffness should also be normalized to 8 mm (0.31 in.) to give K_{cn}. 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 method for interpreting the results.

RT tests are conducted to check the physical components, data control software, and data acquisition software. Elevated temperature tests are conducted to check the physical and software components of the furnace system, as well as the performance of the MTS at 135 degrees C (275 degrees F). Before conducting the elevated temperature tests with control and monitoring thermocouples, thermocouples should be calibrated using instrumentation and standards that are traceable to the National Institute for Standards and Technology (NIST), or another internationally recognized standards organization and proof of traceability should be available for inspection. The option is available to have the thermocouple 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 degrees C (275 degrees F). The expected error for this type of thermocouple is plus or minus 0.3 degrees C relative to the standard.

As the Instron 8511 is a servo-hydraulic machine, some checks were made to ensure that all moving parts, auxiliary equipment, and data recorders functioned properly. Three RCTs were then conducted at RT. Based on the RT results (see Table B-1 and Figures B-1 through B-3), the average difference between the offset and permanent displacement was 0.19 mm, which is consistent with previous experience. Therefore, the 8511 crosshead displacement indicator was determined to be accurate enough for ring-compression testing. The measured loading stiffness values were 26 percent (on average) lower than the predicted values. The Instron Model 8511 has a long load train, higher machine compliance, and lower machine stiffness (K_{mach}), which accounts for the difference between measured and calculated stiffness values. Although load is not an important parameter in ring-compression ductility

tests, the stiffness results shown in Table A-1 are consistent with previous benchmark test results and indicate that the load-cell output values are adequate for RCTs.

The results at 135 degrees C (275 degrees F) (see Table B-1 and Figures B-4 through B-6) were assessed to be acceptable. The difference between offset and permanent displacements was 0.20 mm (0.0079 in.) (on average). The measured stiffness values were about 21 percent lower than the calculated values and are consistent with values previously obtained in benchmark tests using the same cladding material. The stiffness results indicate adequate load-cell performance at 135 degrees C (275 degrees F). The results of the six tests support using the Instron Model 8511 for performing RCT ductility tests using oxidized high-burnup cladding samples.

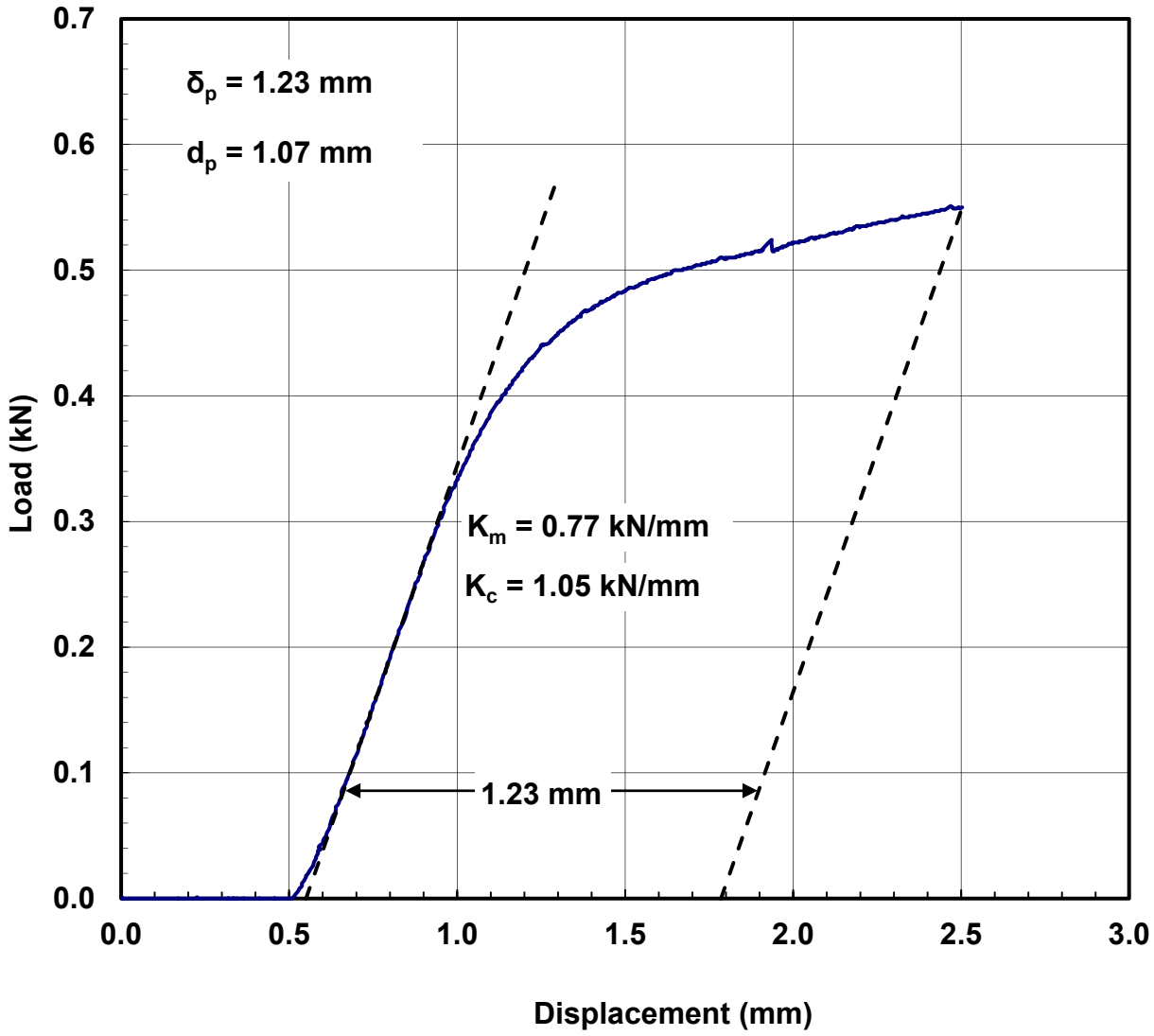

Figure B-1. Load-displacement curve for ZIRLO™ sample 109B1 compressed at RT and 2 mm/minute to 2-mm total displacement.

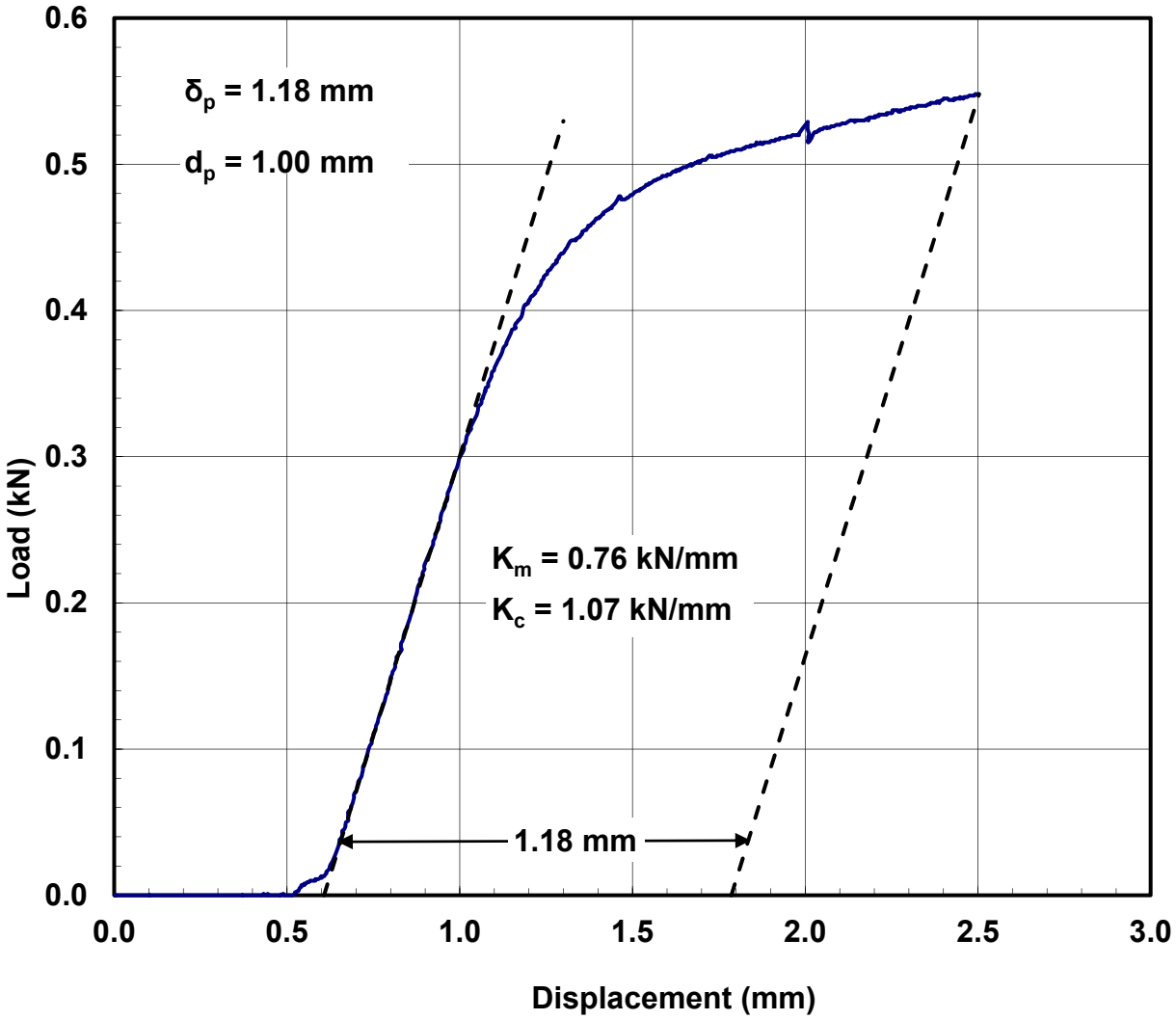

Figure B-2. Load-displacement curve for ZIRLO™ sample 109B2 compressed at RT and 2 mm/minute to 2-mm total displacement.

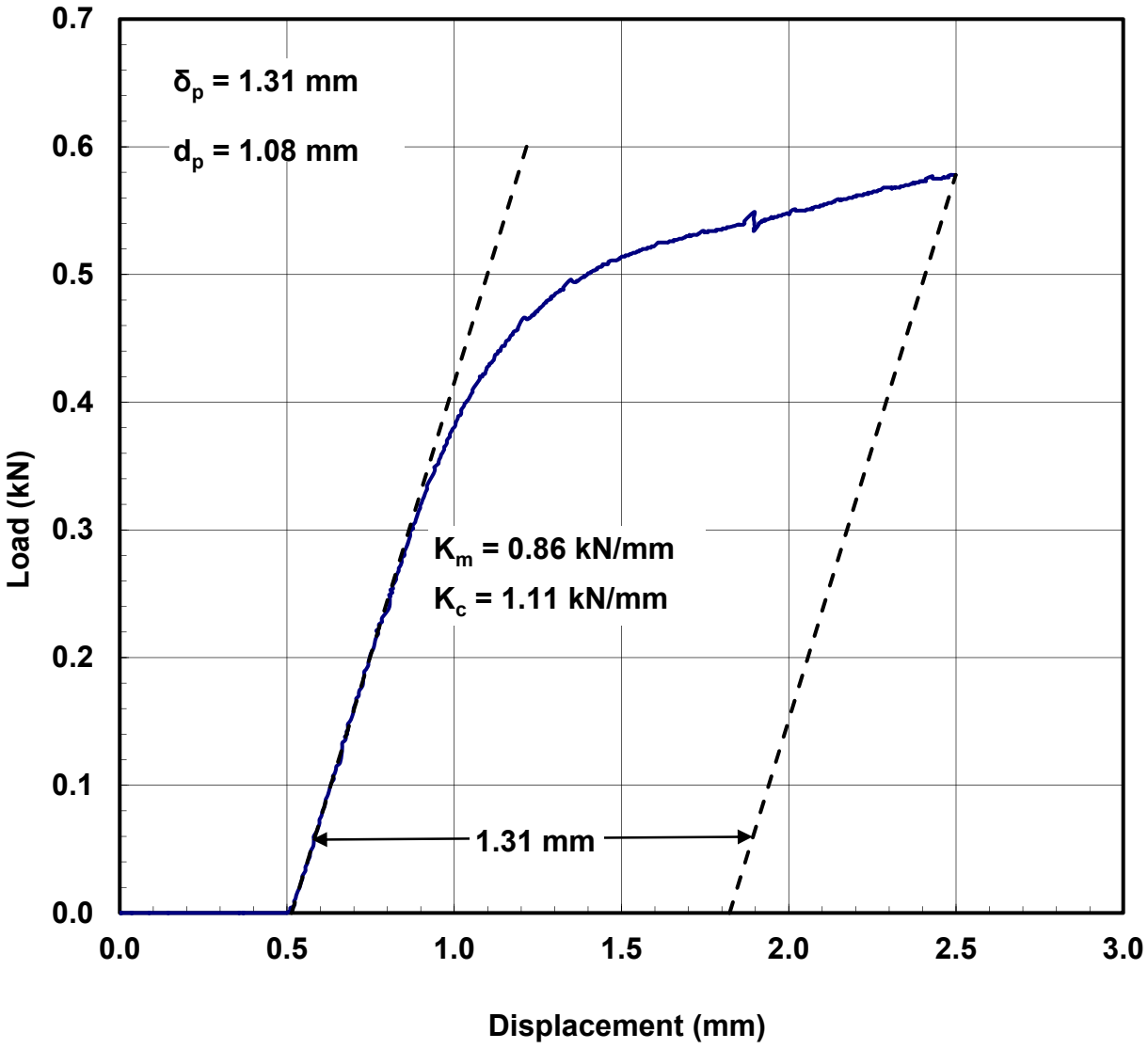

Figure B-3. Load-displacement curve for ZIRLO™ sample 109B3 compressed at RT and 2 mm/minute to 2-mm total displacement.

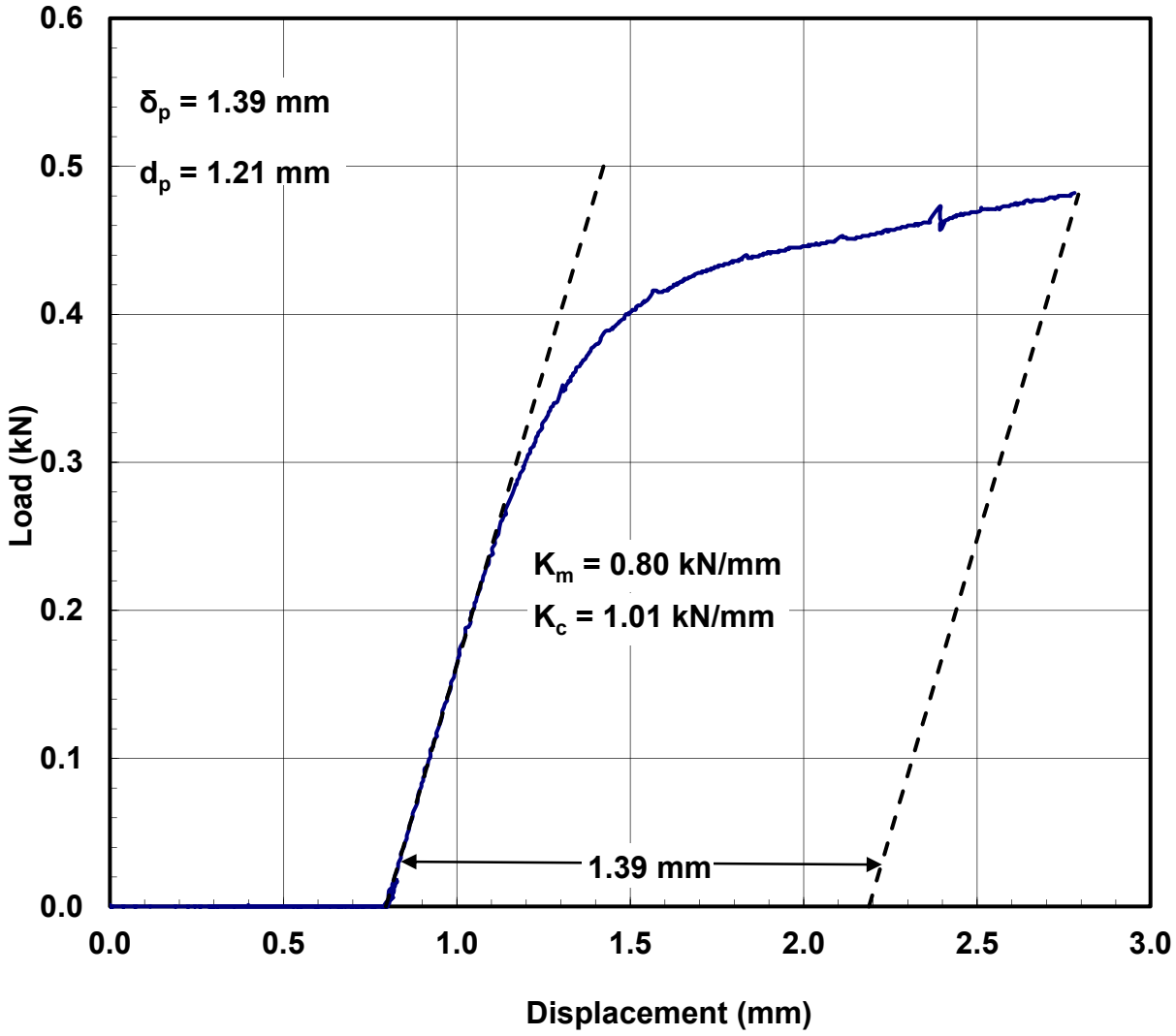

Figure B-4. Load-displacement curve for ZIRLO™ sample 109B4 compressed at 135 ºC and 2 mm/minute to 2-mm total displacement.

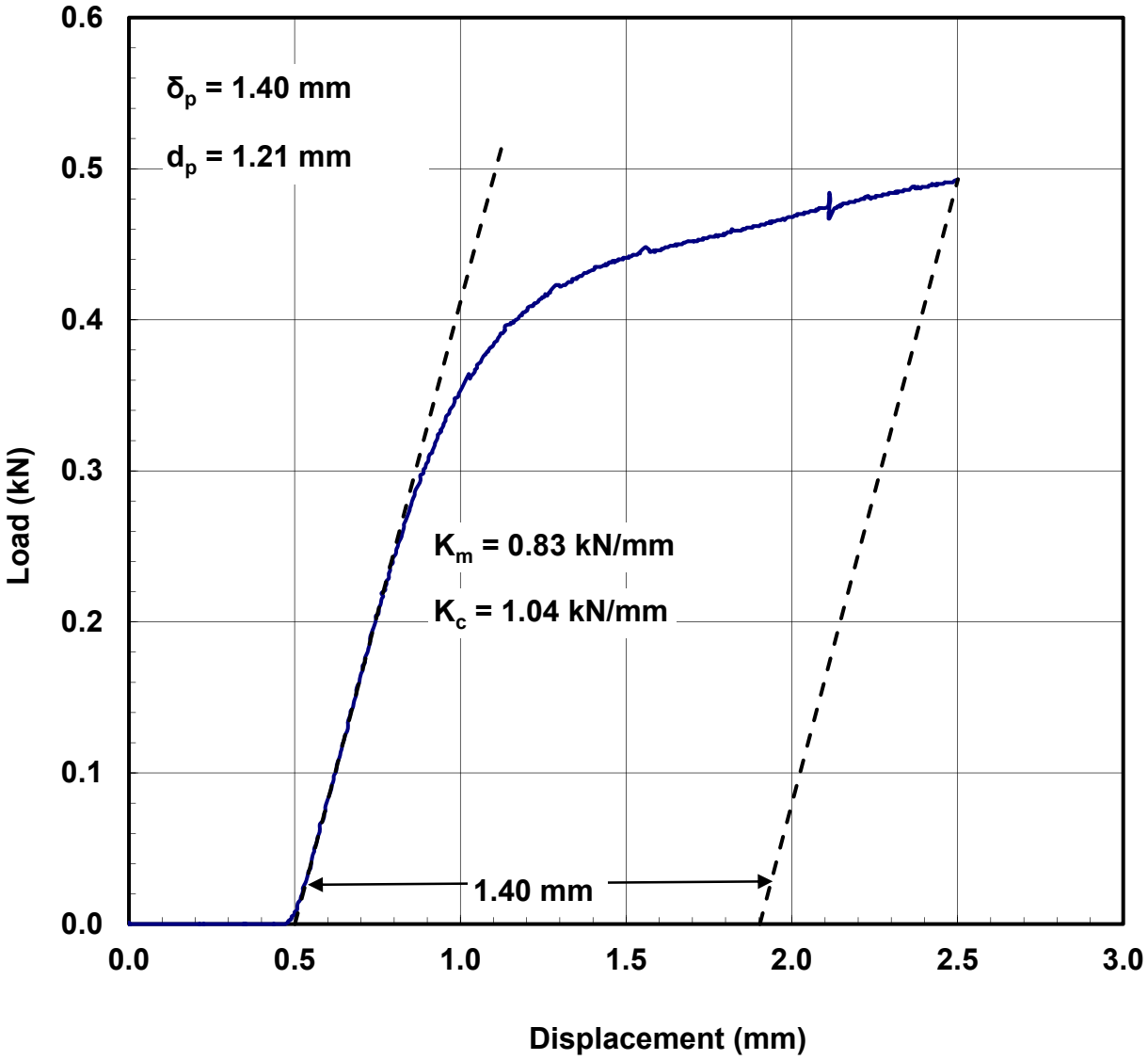

Figure B-5. Load-displacement curve for ZIRLO™ sample 109B5 compressed at 135 ºC and 2 mm/minute to 2-mm total displacement.

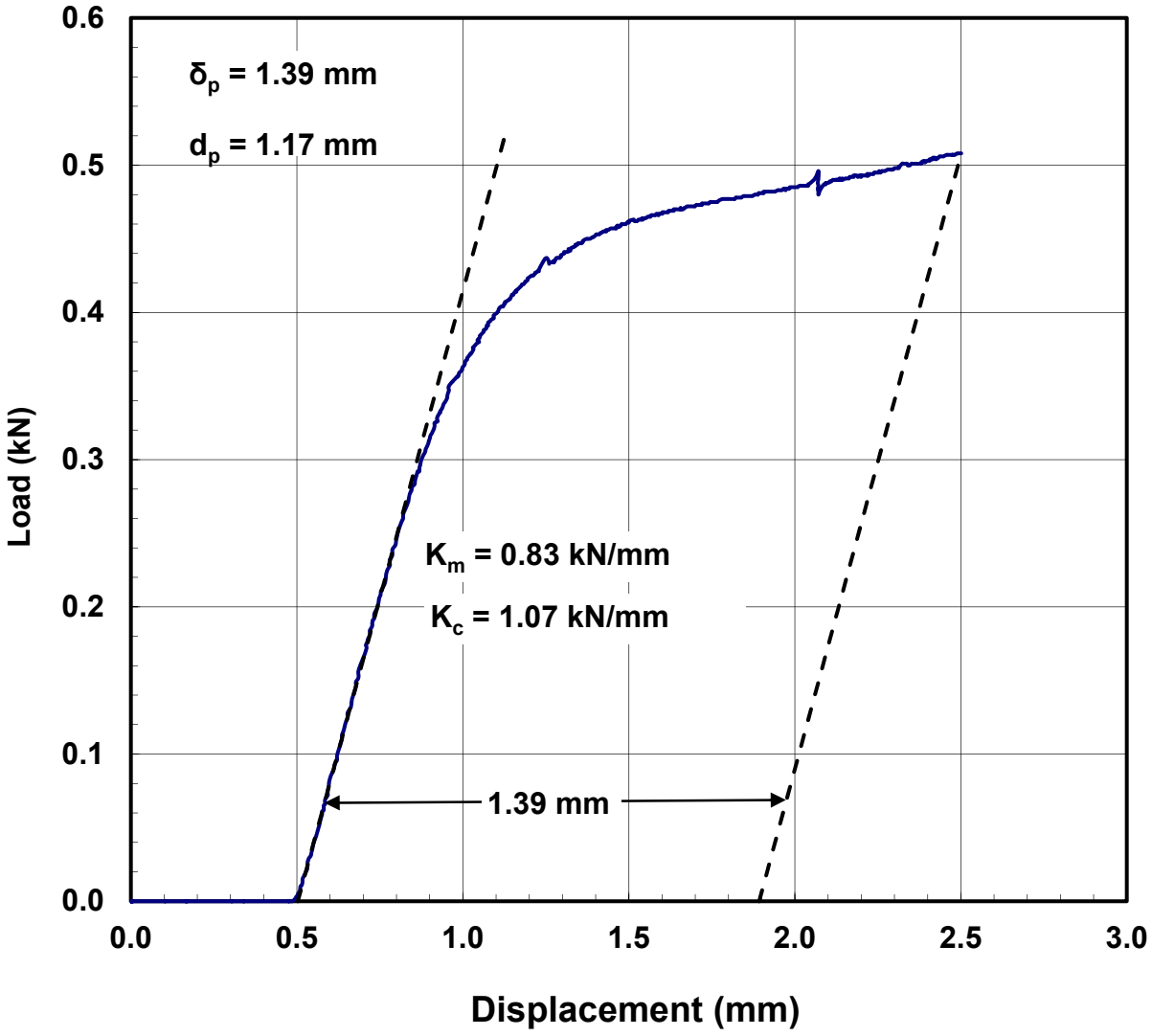

Figure B-6. Load-displacement curve for ZIRLO™ sample 109B9 compressed at 135 ºC and 2 mm/minute to 2-mm total displacement.

Table B-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 post-test 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	1.09	0.77	0.80	1.23	1.07	0.16
109B2	RT	9.49±0.01	7.75±0.06	0.589±0.003	1.10	0.76	0.79	1.18	1.00	0.18
109B3	RT	9.49±0.01	8.03±0.02	0.589±0.003	1.10	0.86	0.86	1.31	1.08	0.23
RT Summary	RT	9.49	7.82	0.59	1.10	0.80	0.82	1.24	1.05	0.19
109B4	132±5	9.495±0.005	7.81±0.02	0.589±0.003	1.04	0.80	0.82	1.40	1.21	0.18
109B5	135±5	9.495±0.005	8.01±0.07	0.590±0.003	1.04	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	1.04	0.83	0.81	1.40	1.17	0.22
135 ºC Summary		9.49	8.00	0.59	1.04	0.82	0.82	1.40	1.20	0.20

APPENDIX B REFERENCES[1]

B-1. Burtseva, T.A., "Procedure for Conducting Ring-Compression Tests in Laboratory DL-102A," ANL-IPS Memo, IPS-495-00-00, November 26, 2007 (ADAMS Accession No. ML15083A522).

[1] All U.S. Nuclear Regulatory Commission (NRC) documents that are publicly available may be accessed through the Electronic Reading Room on NRC's public Web site at http://www.nrc.gov/reading-rm/doc-collections/ and through the NRC's Agencywide Documents Access and Management System (ADAMS) at http://www.nrc.gov/reading-rm/adams.html The documents can also be viewed online or printed for a fee in the NRC's Public Document Room (PDR) at 11555 Rockville Pike, Rockville, MD. For problems with ADAMS, contact the PDR staff at 301-415-4737 or 800-397-4209; fax 301 415-3548; or e-mail pdr.resource@nrc.gov.

APPENDIX C

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

Figures C-1 through C-8 show examples of load-displacement curves and offset displacements determined from these curves. For this series of tests, prehydrided vintage 15x15 Zircaloy (Zry)-4 cladding samples, comparable to H.B. Robinson vintage cladding, were oxidized to 6 percent Cathcart-Pawel (CP) equivalent cladding reacted (ECR) at a maximum temperature of 1,200 degrees Celsius (C) (2,192 degrees Fahrenheit (F)). As shown in Table C-1 (Table 52 in NUREG/CR 6967, "Cladding Embrittlement during Postulated Loss-of-Coolant Accidents" (Ref. C-1)), the quench temperature was varied from 800 degrees C (1,472 degrees F) to 700 degrees C (1,292 degrees F) to 600 degrees C (1,112 degrees F) to slow-cooling without quench. The load-displacement curves (see Figures C-1 through C-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 C-7 through C-8). For the samples that retained ductility, the difference between the offset strains and the permanent strains was only 0.9 percent. For prehydrided Zry-4, the ductility criteria used in NUREG/CR-6967 is that greater than or equal to 1.0 percent permanent strain implies ductility.

Table C-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

Sample and Test Conditions			ECR[a] %		Plastic Displacement[b], mm		Plastic Strain[b], %	
Quench Temperature, ºC or SC (Slow Cooled)	Test Time[c] 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] CP-ECR calculated from beginning of ramp to end of hold time

[b] ring-compression tests performed on ≈8-mm-long samples at 135 degrees C and 0.0333 mm/s crosshead displacement rate

[c] From beginning of ramp at 300 degrees C (572 degrees F) to end of hold time at about 1,200 degrees C (2,192 degrees F).

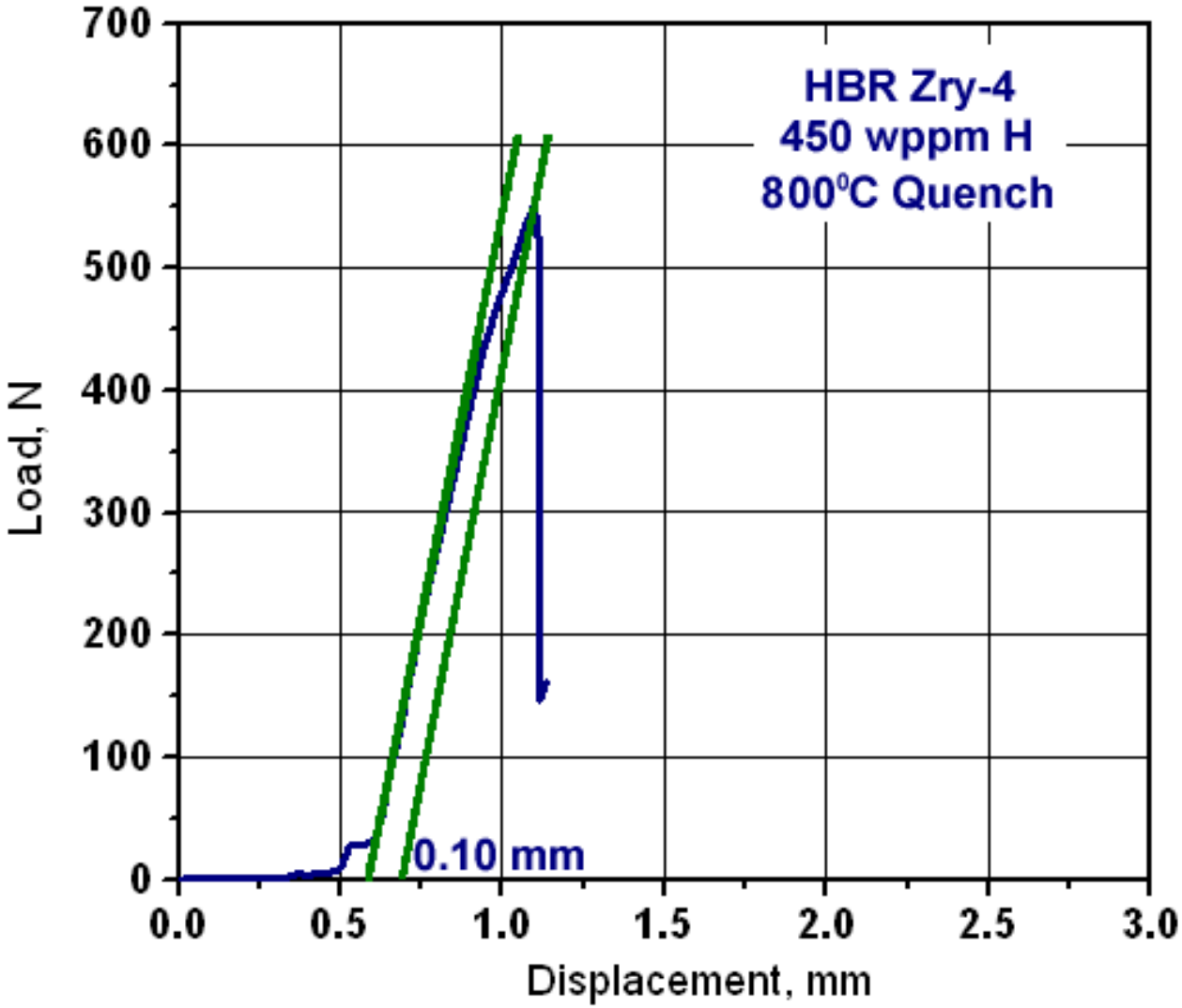

Figure C-1. Load-displacement results for ring #1 (8.12 mm long) of a prehydrided (450 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and quenched at 800 ºC. The post-test sample had tight through-wall cracks at load and support locations. Offset and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

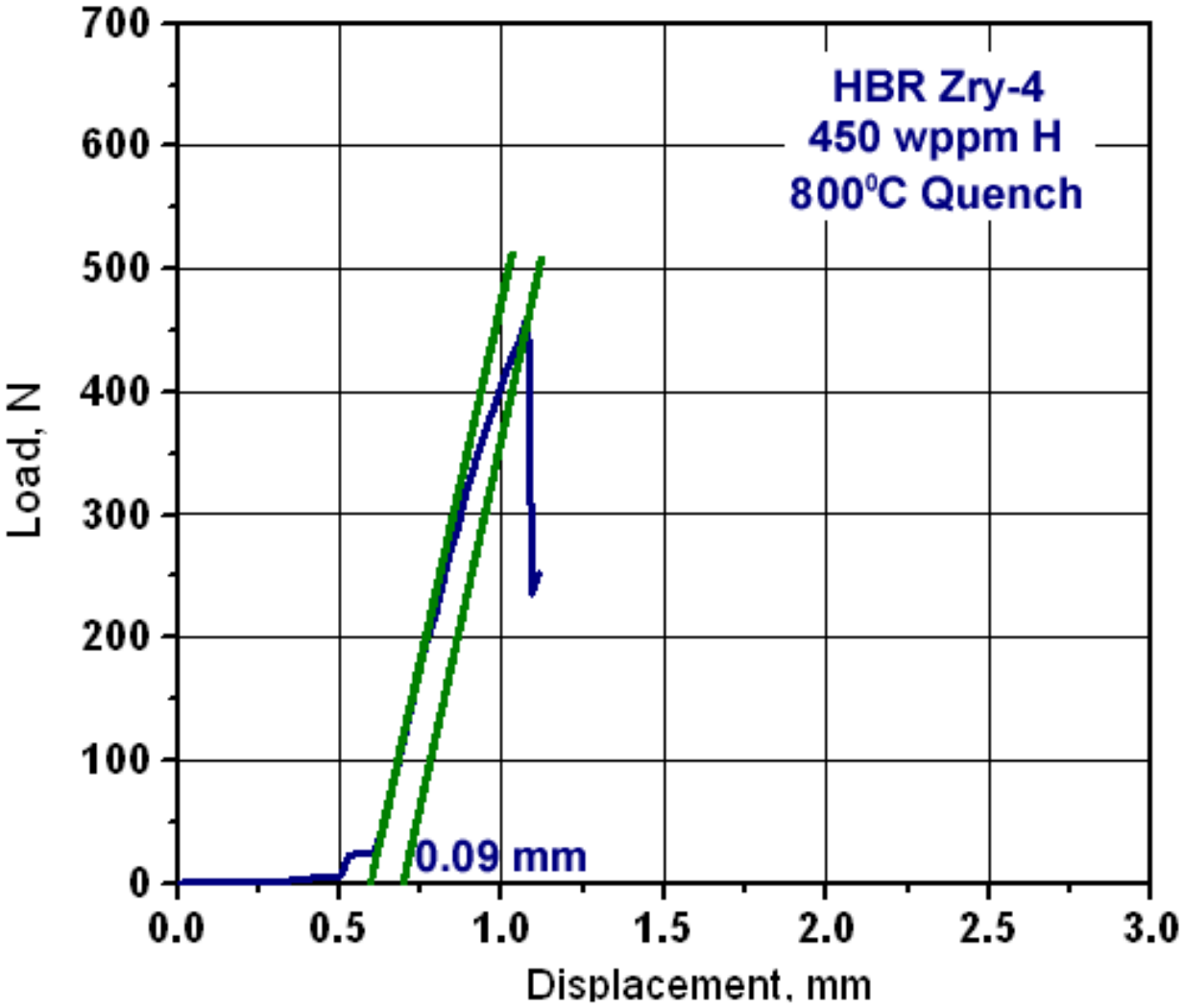

Figure C-2. Load-displacement results for ring #2 (6.84 mm long) of a prehydrided (450 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and quenched at 800 ºC. The post-test sample had a tight through-wall crack at the support location. Offset and permanent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

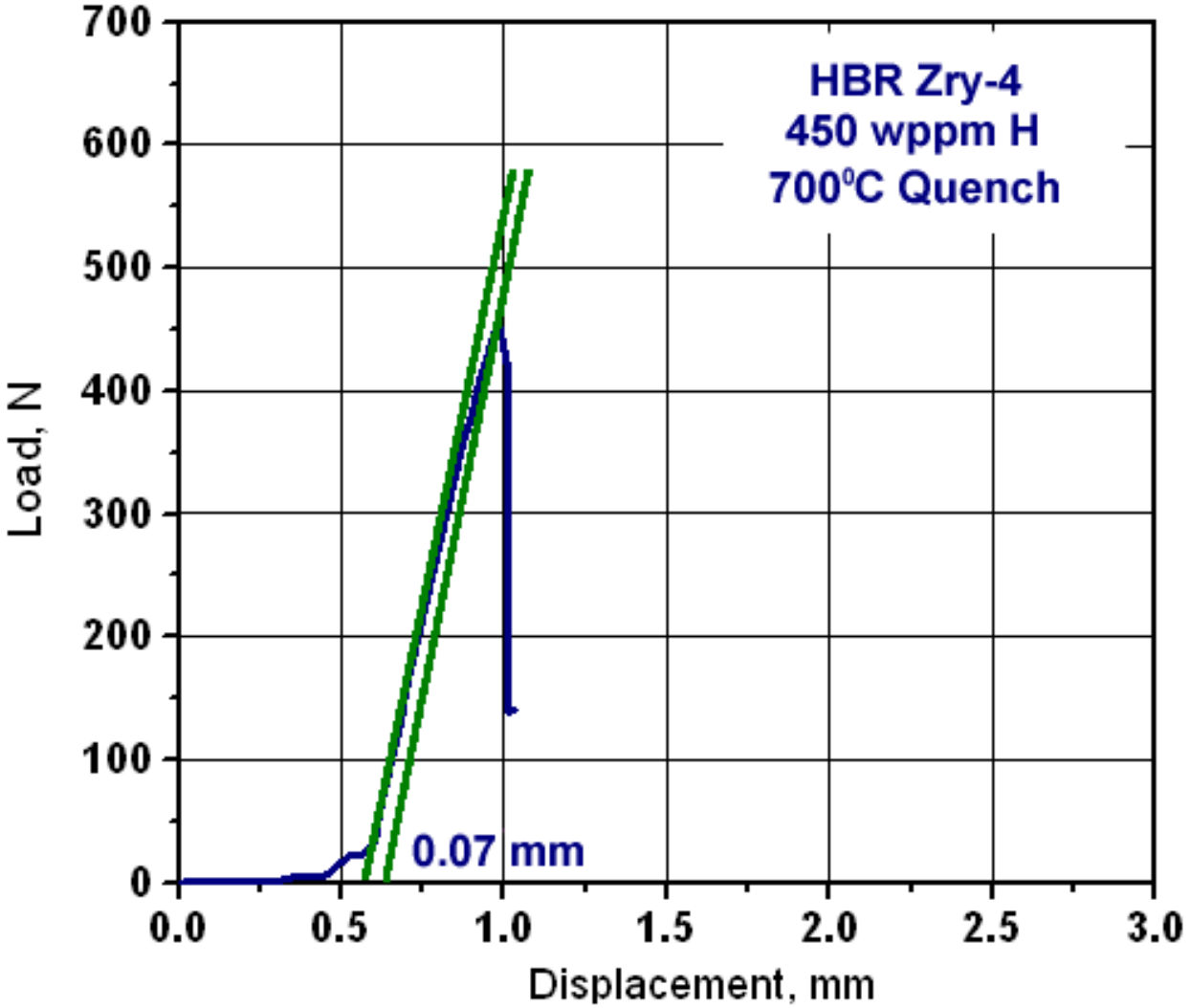

Figure C-3. Load-displacement results for ring #1 (8.08 mm long) of a prehydrided (450 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and quenched at 700 ºC. The post-test sample had through-wall cracks at load and support locations. Offset displacement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

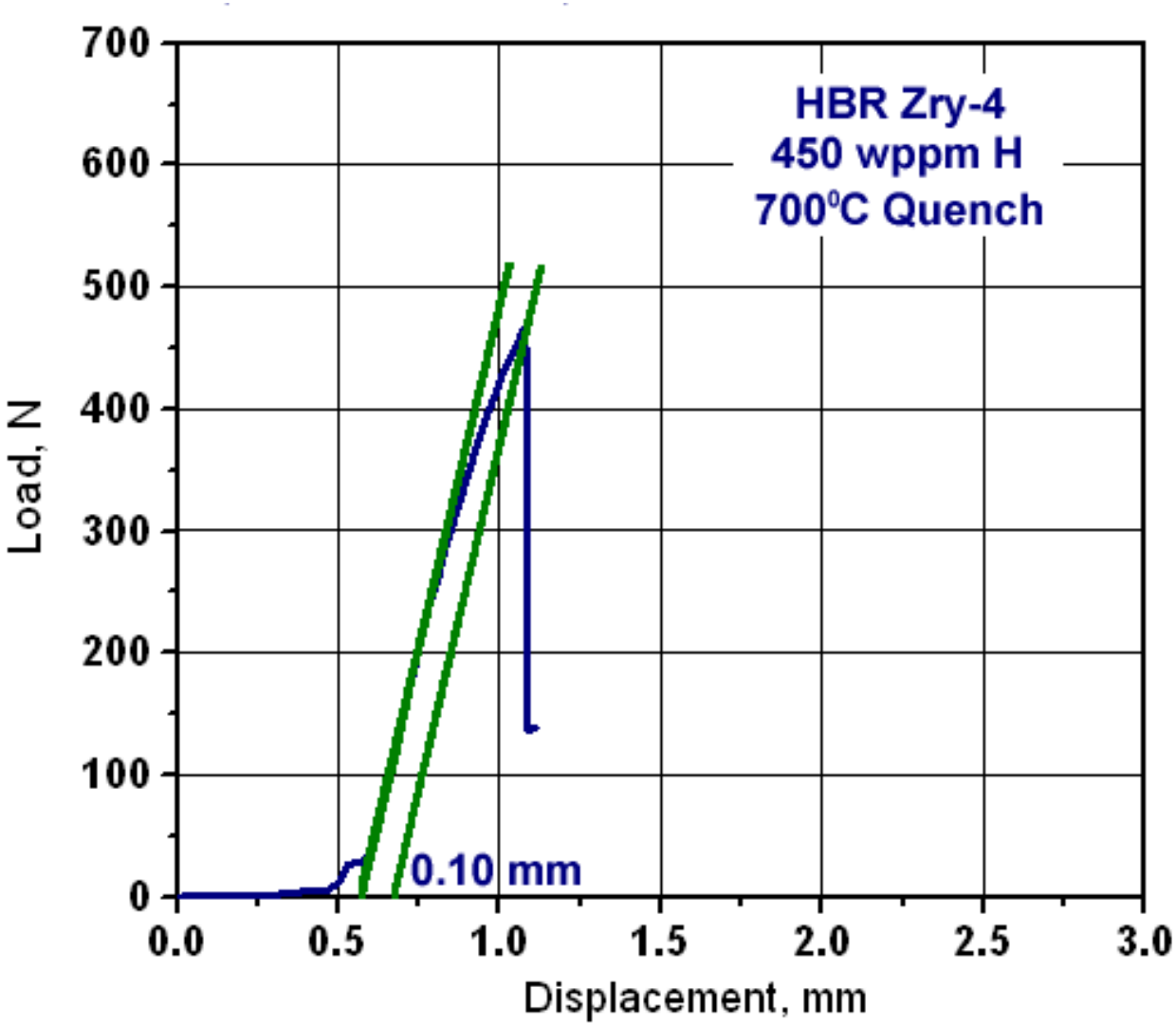

Figure C-4. Load-displacement results for ring #2 (7.30 mm long) of a prehydrided (450 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and quenched at 700 ºC. The post-test sample had tight through-wall cracks at load and support locations. Offset and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

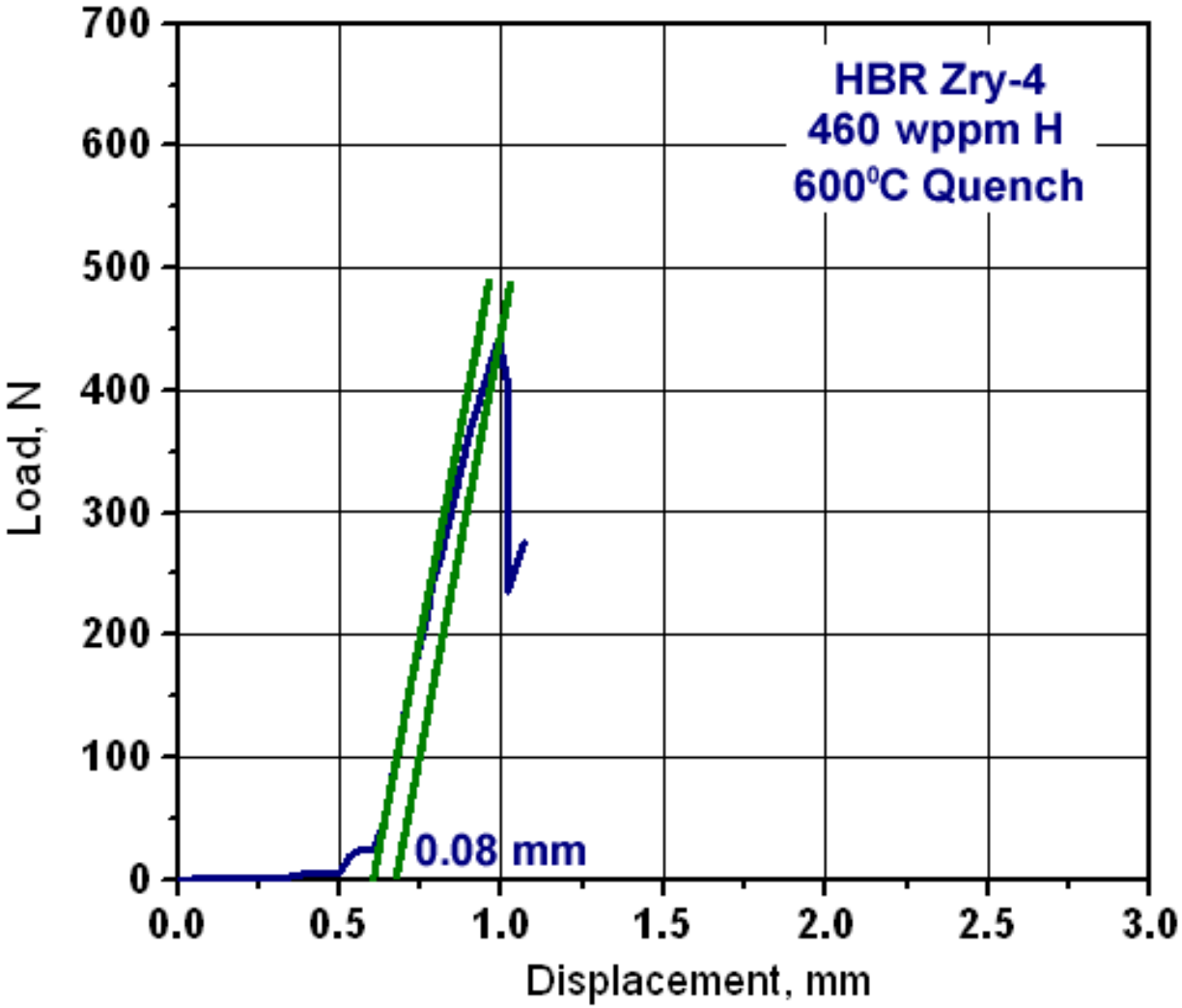

Figure C-5. Load-displacement results for ring #1 (7.93 mm long) of a prehydrided (460±30 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and quenched at 600 ºC. The post-test sample had a tight through-wall crack at the support location. Offset and permanent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

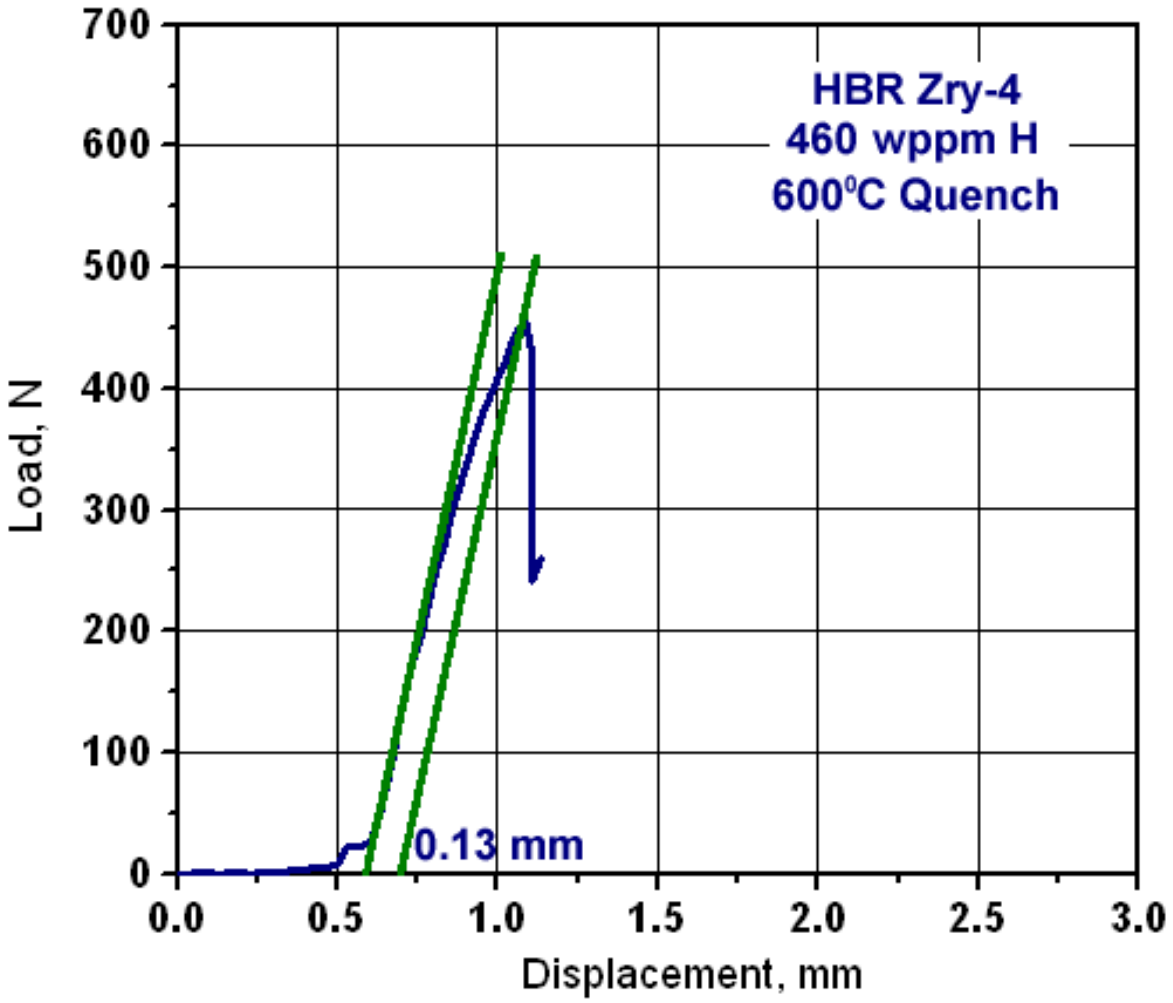

Figure C-6. Load-displacement results for ring #2 (7.05 mm long) of a prehydrided (460±30 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and quenched at 600 ºC. The post-test sample had a through-wall crack at the loading location. Offset and permanent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

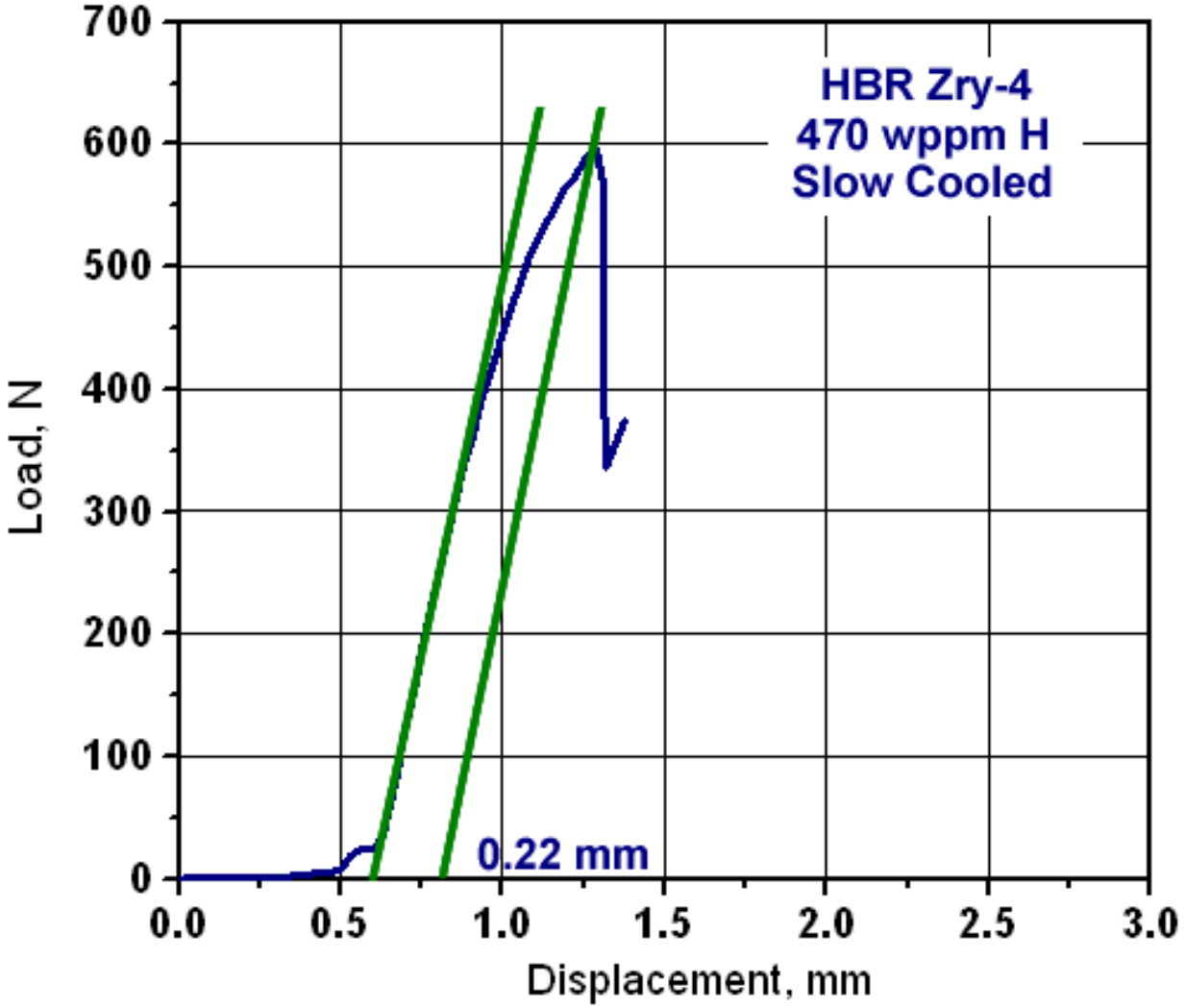

Figure C-7. Load-displacement results for ring #1 (8.04 mm long) of a prehydrided (470±30 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and cooled to room temperature without quench. The post-test sample had a tight through-wall crack at the support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

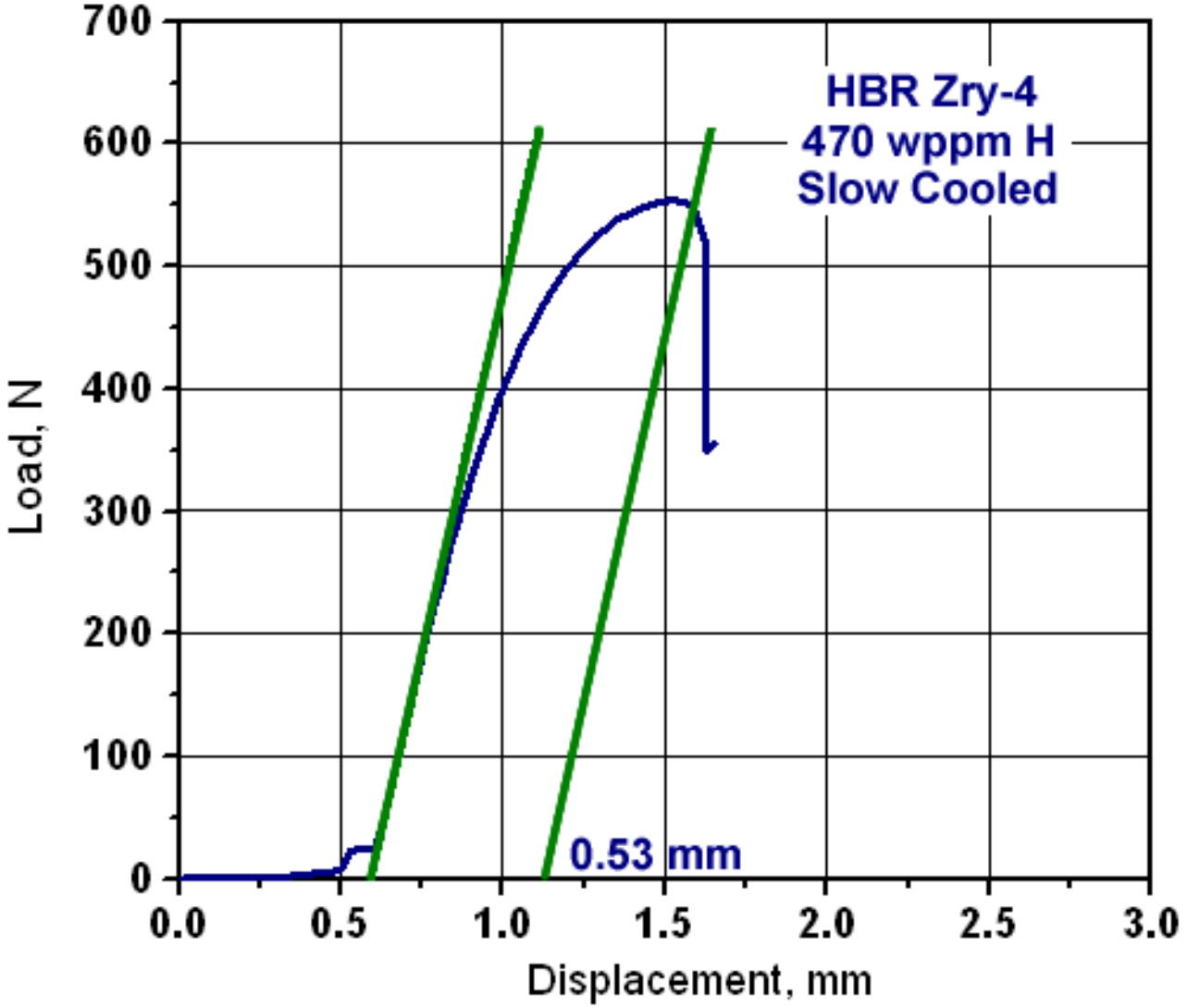

Figure C-8. Load-displacement results for ring #2 (7.05 mm long) of a prehydrided (470±30 wppm) HBR 15×15 Zry-4 sample oxidized to 6% CP-ECR at a maximum oxidation temperature of 1,200 ºC and cooled to room temperature without quench. The post-test sample had a tight through-wall crack at the loading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

APPENDIX C REFERENCES[1]

C-1. U.S. Nuclear Regulatory Commission (NRC), NUREG/CR-6967, "Cladding Embrittlement during Postulated Loss-of-Coolant Accidents," Washington, DC, July 2008.

[1] All NRC documents that are publicly available may be accessed through the Electronic Reading Room on NRC's public Web site at http://www.nrc.gov/reading-rm/doc-collections/ and through the NRC's Agencywide Documents Access and Management System (ADAMS) at http://www.nrc.gov/reading-rm/adams.html The documents can also be viewed online or printed for a fee in the NRC's Public Document Room (PDR) at 11555 Rockville Pike, Rockville, MD. For problems with ADAMS, contact the PDR staff at 301-415-4737 or (800) 397-4209; fax (301) 415-3548; or e-mail pdr.resource@nrc.gov.

APPENDIX D

RELATIONSHIP BETWEEN OFFSET STRAIN AND PERMANENT STRAIN

For as-fabricated cladding compressed at room temperature (RT) or at 135 degrees Celsius (C) and at 0.033 millimeters/second (mm/s) to a total displacement of 2 mm, the difference between offset displacement and permanent displacement is less than or equal to 0.2 mm (0.008 in.). 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 percent. Table D-1 shows the results of these tests.

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

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 less than or equal to 1,200 degrees C (2,192 degrees F), 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 (0.02 in.). 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 (0.0004 in.).

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 percent) and permanent (d_p/D_o in percent) strains for permanent strains in the range of 1.0 to 2.3 percent. Figure D-1 summarizes the data reported in Refs. D-1, D-2, D-3 and in Table D-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 percent) are from pre-hydrided Zircaloy (Zry)-4 and high-burnup Zry-4 and ZIRLO™ samples. Low values of permanent strain at intermediate CP-ECR levels (10–18 percent) are from high-burnup ZIRLO™ and M5® samples. Low values of permanent strain at high ECR values (15–20 percent) are from as-fabricated cladding materials. The following equation gives the best linear fit to the data:

$$\delta_p/D_o - d_p/D_o = 0.25 + 0.0863 \text{ CP-ECR} \quad \text{(D1)}$$

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

$$\delta_p/D_o - d_p/D_o = 0.41 + 0.1082 \text{ CP-ECR} \quad \text{(D2)}$$

Because of the large data scatter in Figure D-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 D2 to 1.0 percent:

$$\delta_p/D_o \geq 1.41 + 0.1082 \text{ CP-ECR} \quad \text{(D3)}$$

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 D3. Similarly, the limit calculated from the right-hand side of Equation D3 should also be rounded to the nearest tenth of a percent.

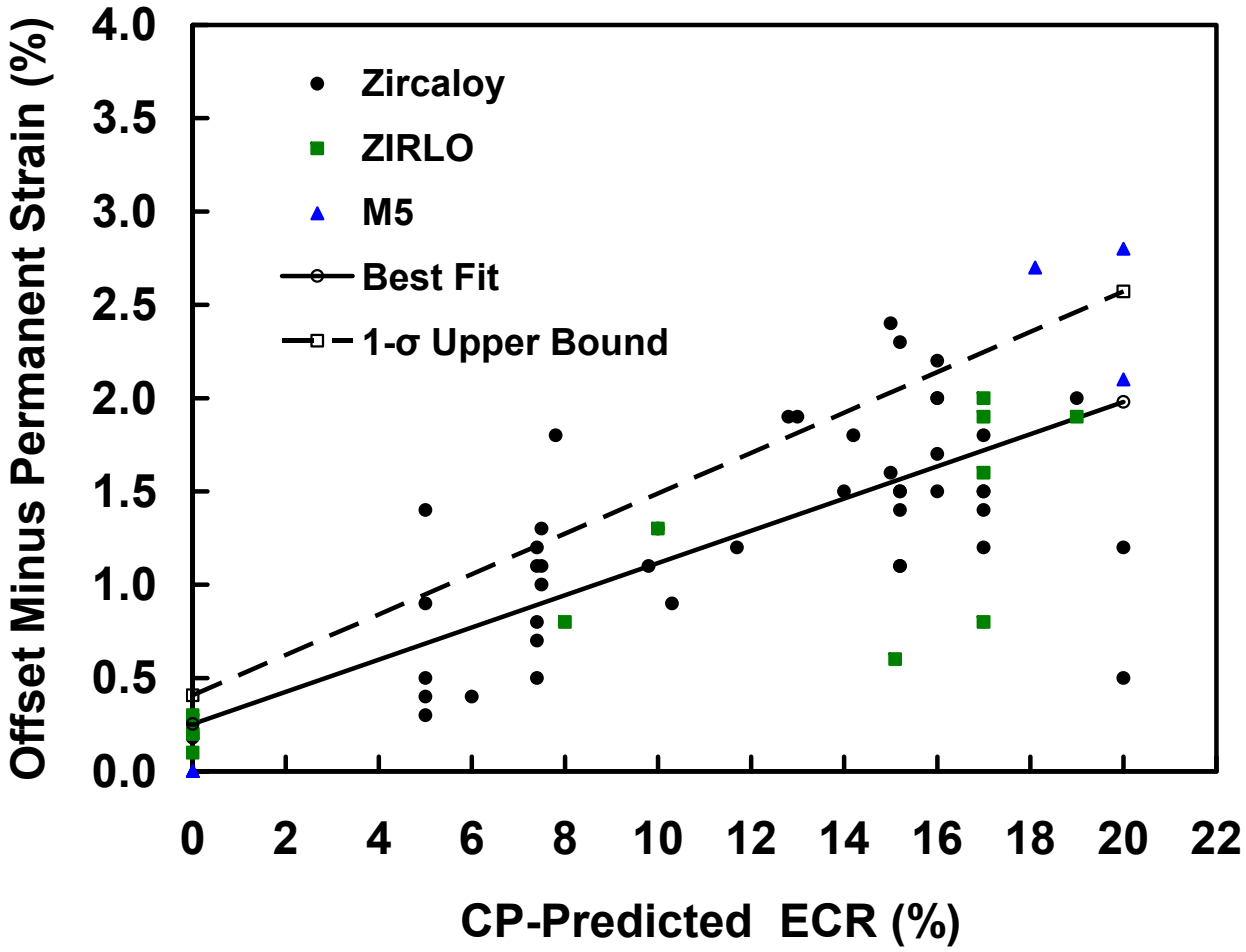

Figure D-1. Difference in offset strain and permanent strain as a function of calculated oxidation level (CP-ECR) for permanent strains near the embrittlement threshold (1.0 to 2.3%) for as-fabricated, prehydrided, and high-burnup cladding alloys oxidized at 1,200 ºC and ring-compressed at room temperature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

APPENDIX D REFERENCES[1]

D-1. U.S. Nuclear Regulatory Commission (NRC), NUREG/CR-6967, "Cladding Embrittlement during Postulated Loss-of-Coolant Accidents," Washington, DC, July 2008.

D-2. Yan, Y., 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 the U.S. Nuclear Regulatory Commission, April 17, 2009 (ADAMS Accession No. ML091200702).

D-3. Regulatory Guide 1.224, "Establishing Analytical Limits for Zirconium-Alloy Cladding Material," DATE

[1] All NRC documents that are publicly available may be accessed through the Electronic Reading Room on the U.S. Nuclear Regulatory Commission's (NRC's) public Web site at http://www.nrc.gov/reading-rm/doc-collections/ and through the NRC's Agencywide Documents Access and Management System (ADAMS) at http://www.nrc.gov/reading-rm/adams.html The documents can also be viewed online or printed for a fee in the NRC's Public Document Room (PDR) at 11555 Rockville Pike, Rockville, MD. For problems with ADAMS, contact the PDR staff at 301-415-4737 or 800-397-4209; fax 301-415-3548; or e-mail pdr.resource@nrc.gov.