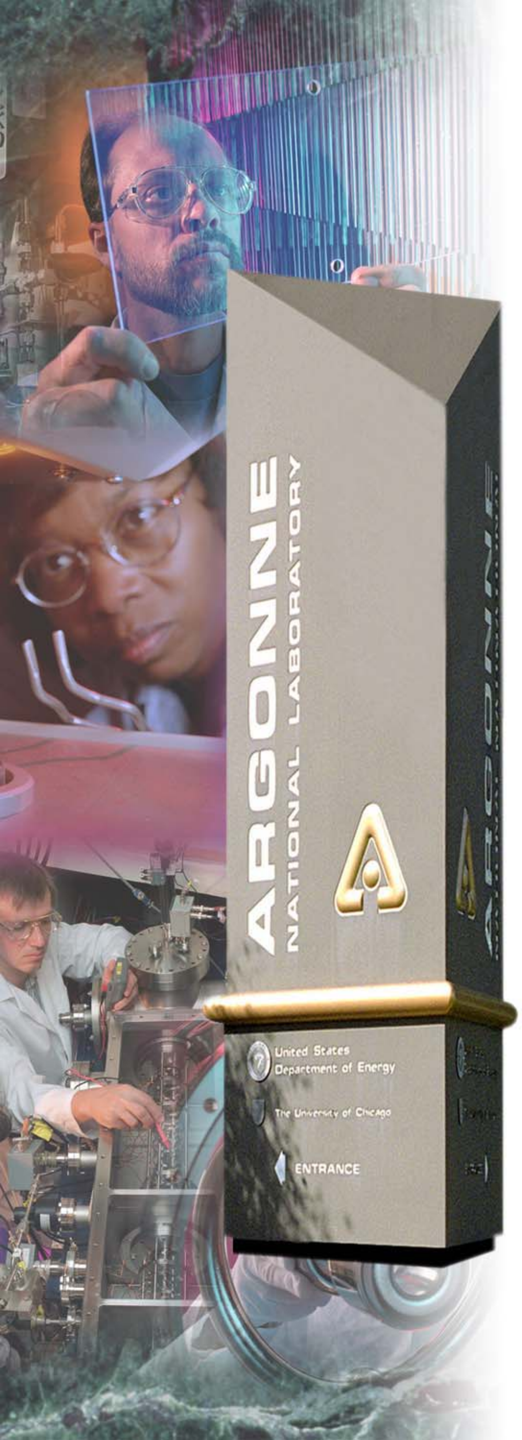


H.B. Robinson High-Burnup PWR Oxidation and Post-Quench Ductility

M. Billone, Y. Yan, T. Burtseva and H. Chung
Energy Technology Division
Argonne National Laboratory
February 10, 2005

Argonne National Laboratory

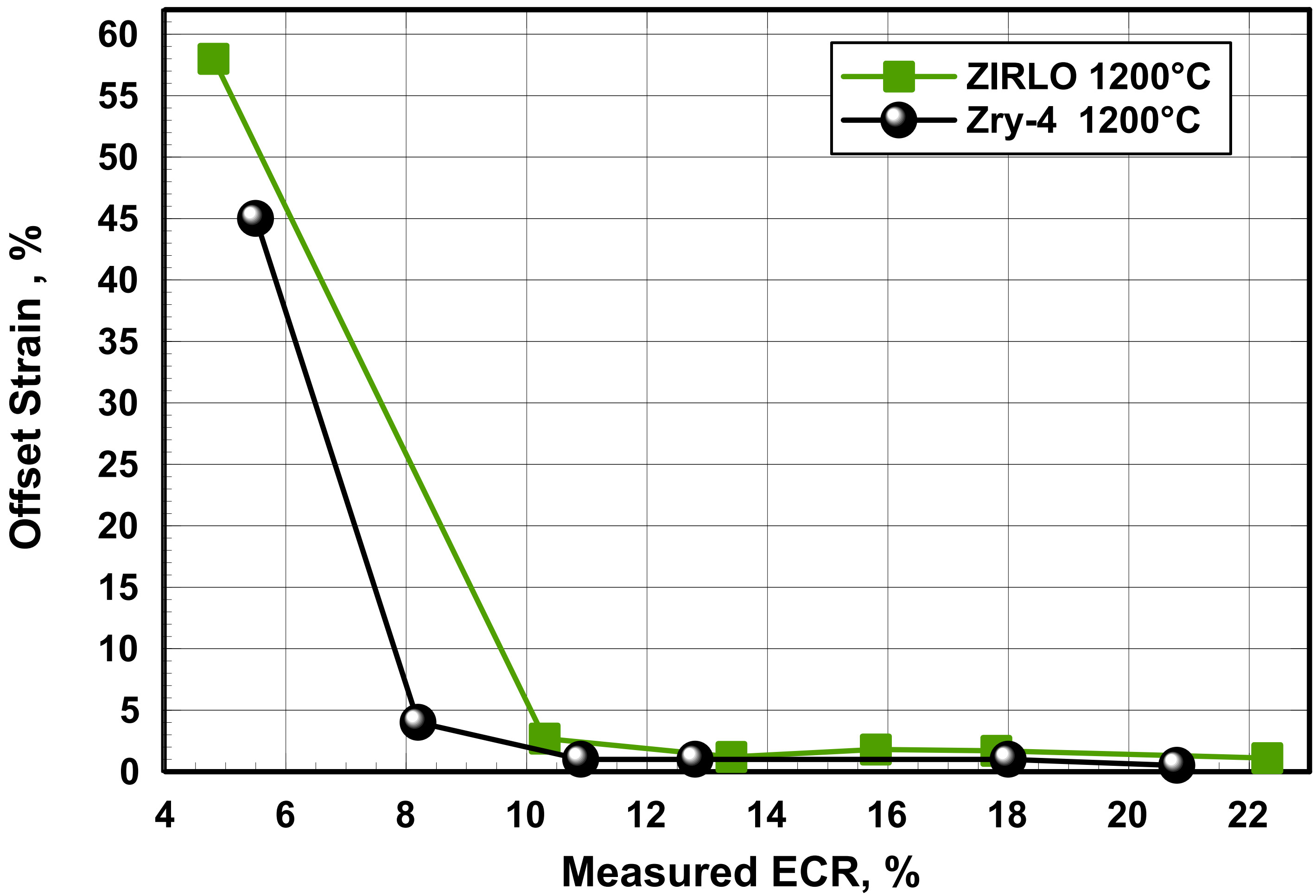
Office of Science
U.S. Department of Energy

A U.S. Department of Energy
Office of Science Laboratory
Operated by The University of Chicago

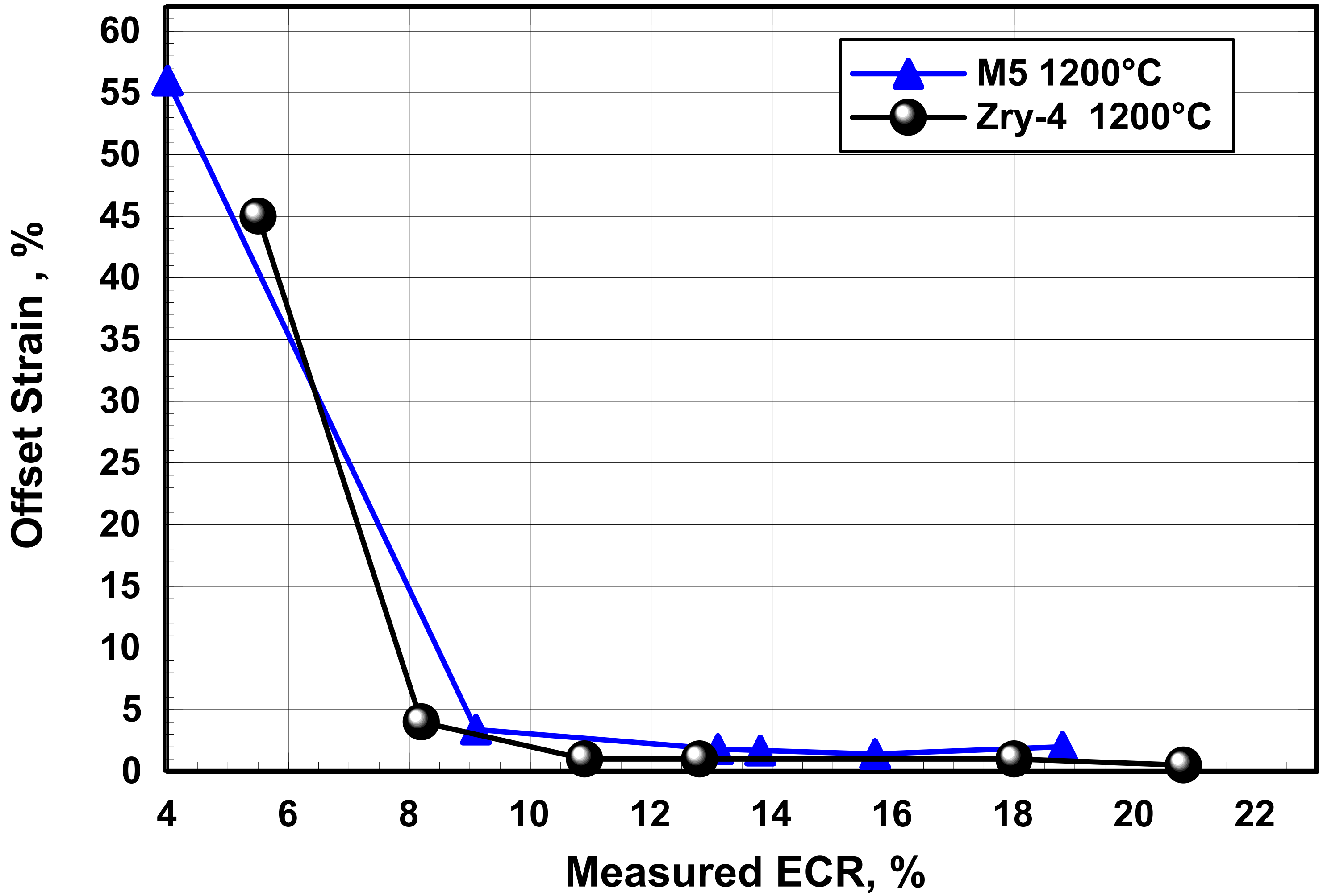
Summary of ANL Results

- **Advanced Alloy Program (NSRC-2004, Billone)**
  - 1000 & 1100ºC oxidation, slow-cooled to 800ºC & quenched
    Good RT ductility to >17% Cathcart-Pawel (CP)-ECR (Zry-4, ZIRLO, M5)
  - 1200ºC-oxidized, slow-cooled to 800ºC & quenched (Zry-4, ZIRLO, M5)
    RT embrittlement at ≈9% ECR (Zry-4) & ≈12% ECR (ZIRLO & M5)
    Significant ductility improvement at 135ºC (embrittlement ECR>17%)
    Severe embrittlement (135ºC): pre-H Zry-4 (<10% ECR, >300 wppm H)
  - Plan to test high-burnp ZIRLO and M5
- **LOCA Integral Test Results at 1204ºC (NSRC-2004, Yan)**
  - High-burnup BWR Zry-2 (significant embrittlement in balloon region)
    Non-uniform wall-thinning, 2-sided oxidation, high secondary H pickup
  - High-burnup PWR Zry-4 (test details to be determined from data below)
    Baseline ductility data for as-fabricated and prehydrided Zry-4 rings
    Data for high-burnup Zry-4 rings oxidized at ≤1206ºC to 3-10% CP-ECR

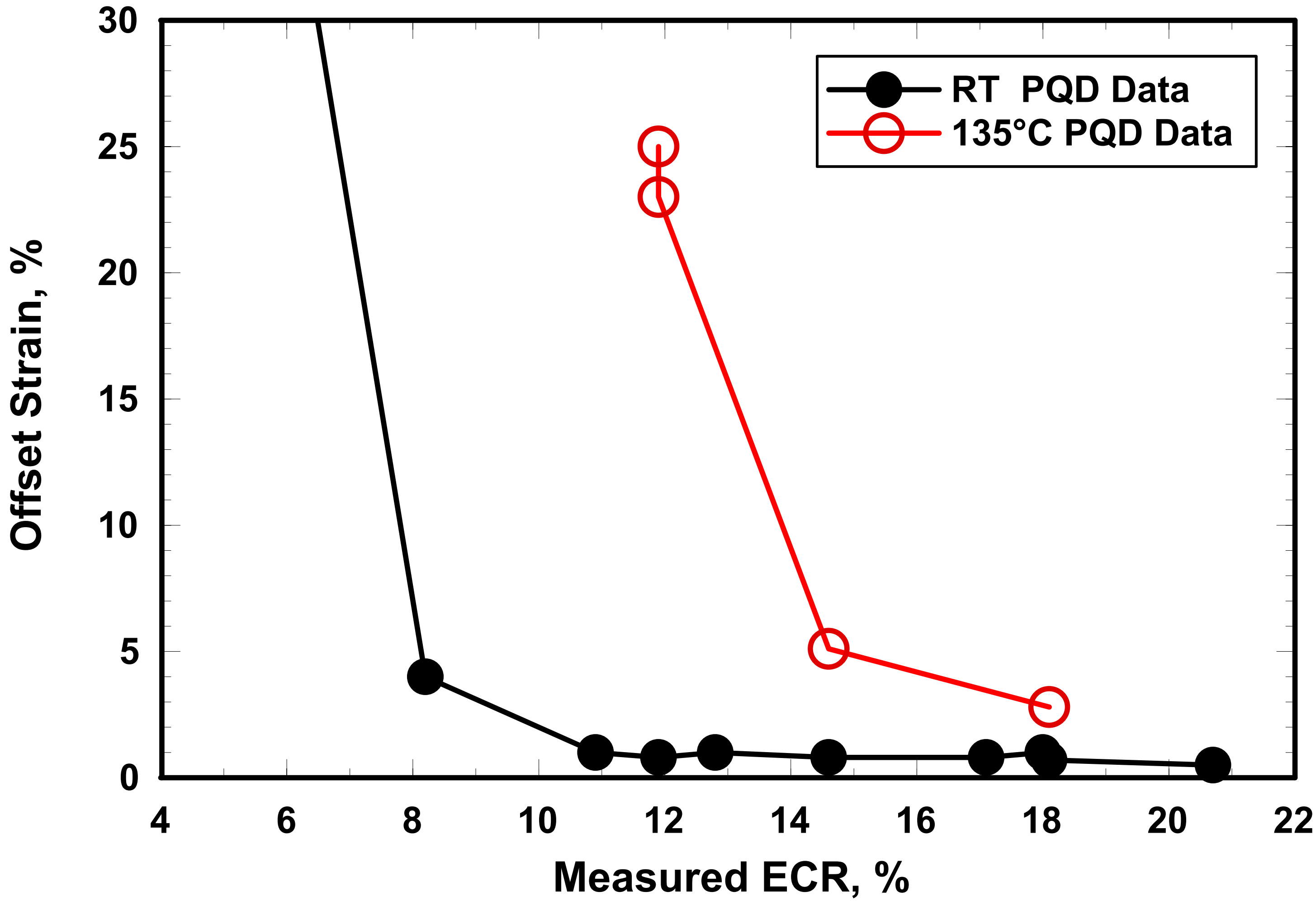
RT Offset Strain for 1200ºC-Oxidatized ZIRLO vs. Zry-4



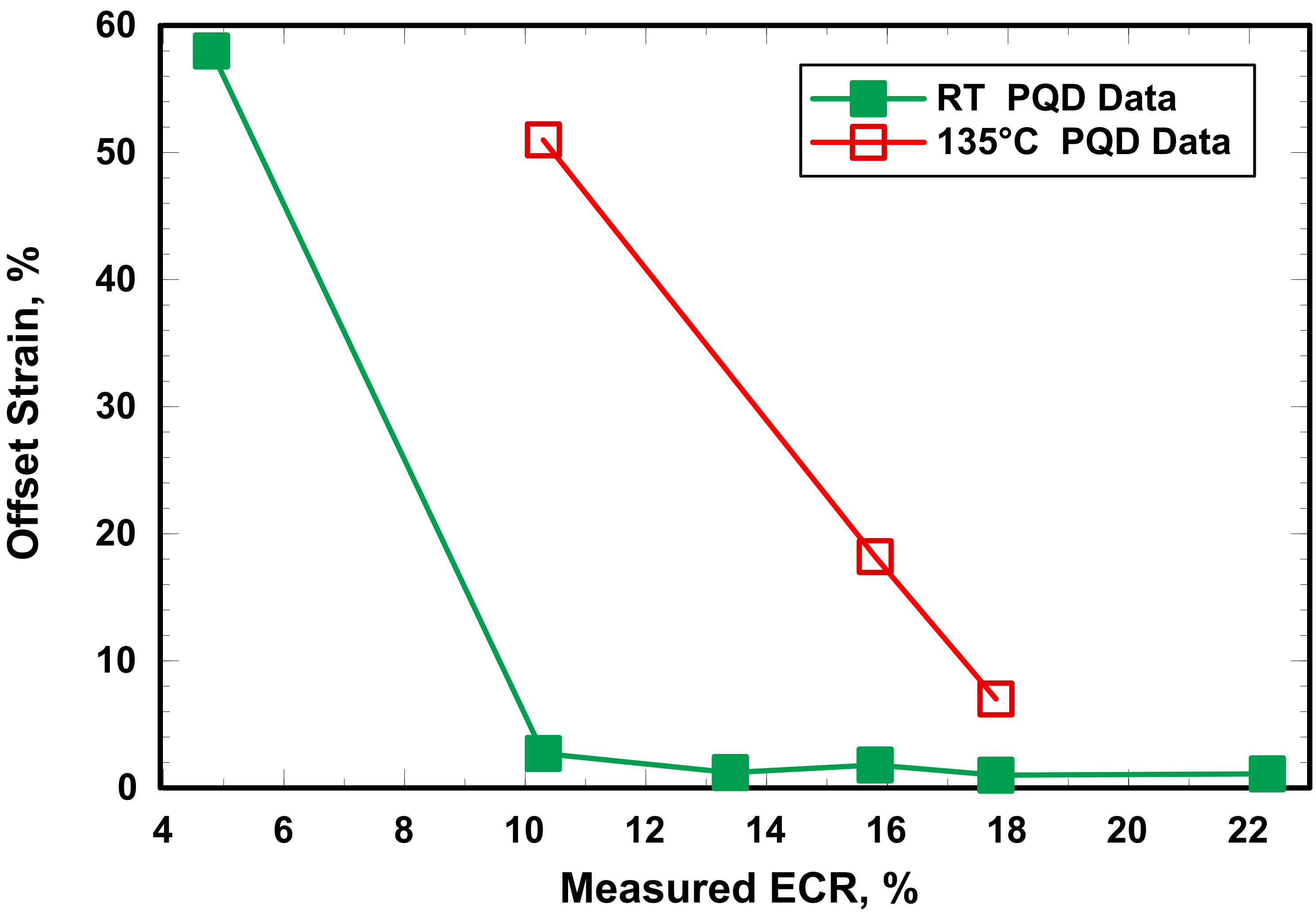
RT Offset Strain for 1200ºC-Oxidatized M5 vs. Zry-4



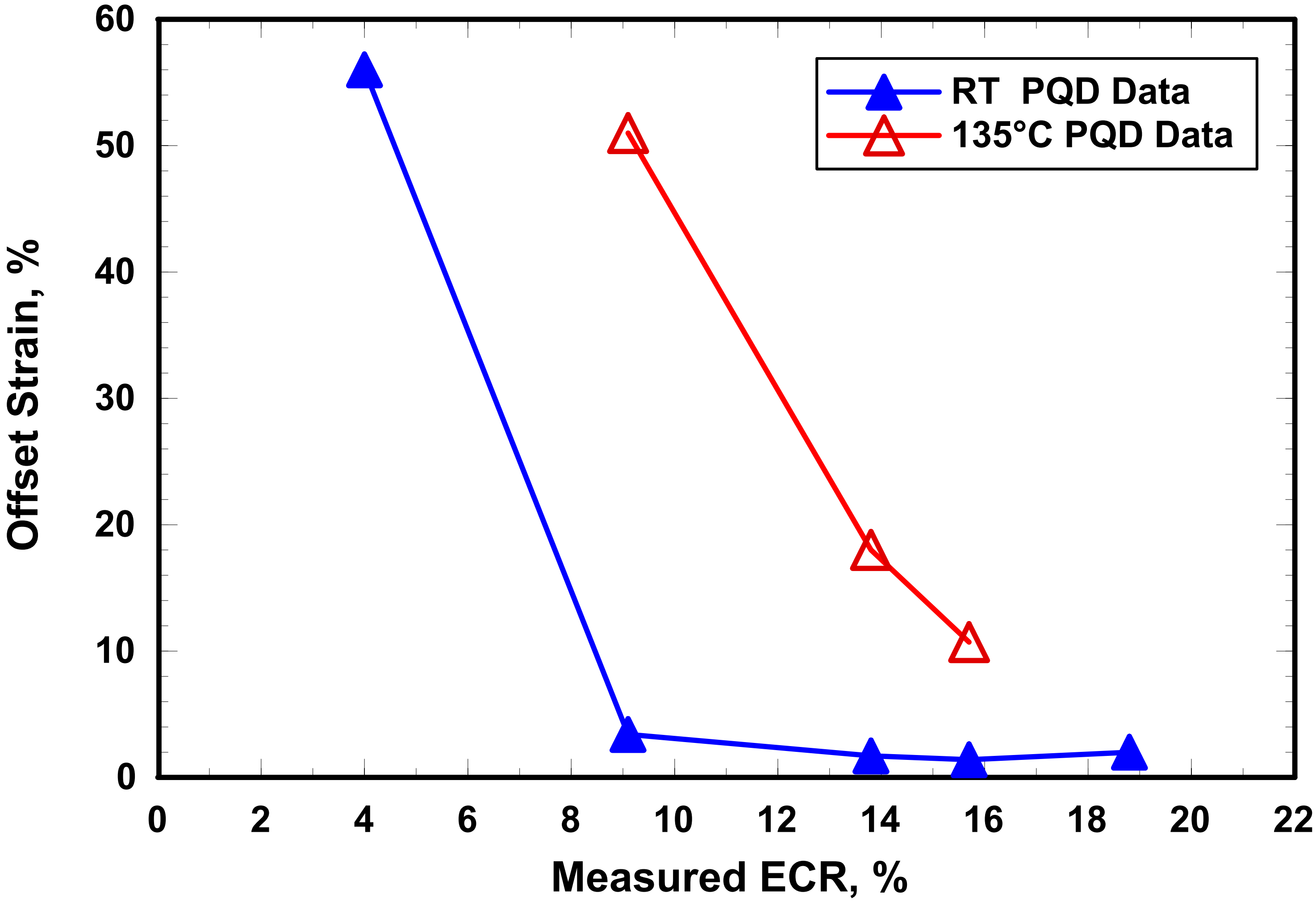
135ºC Offset Strain for 17x17 Zry-4 Oxidized at 1200ºC



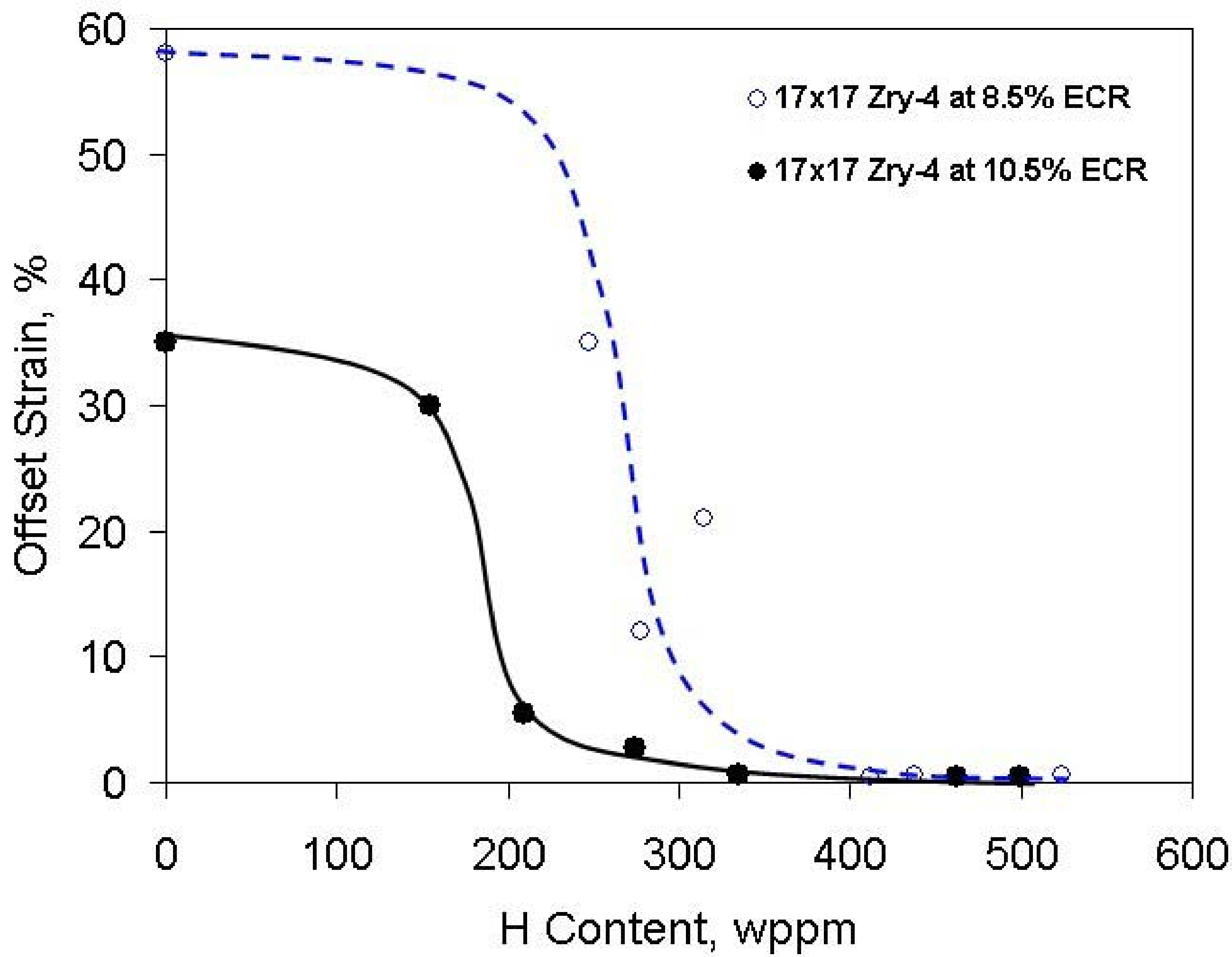
135ºC Offset Strain for 17x17 ZIRLO Oxidized at 1200ºC



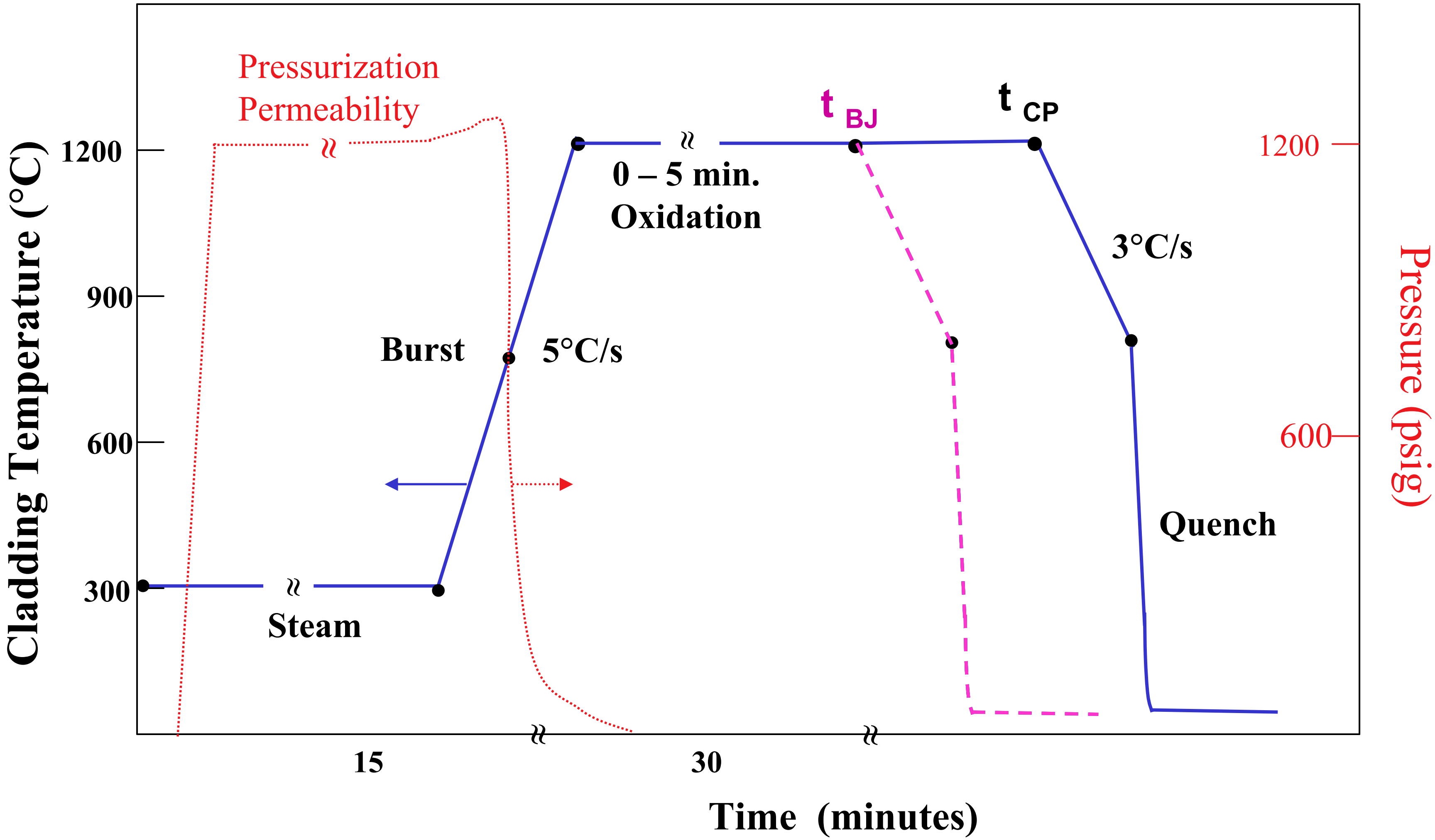
135ºC Offset Strain for 17x17 M5 Oxidized at 1200ºC



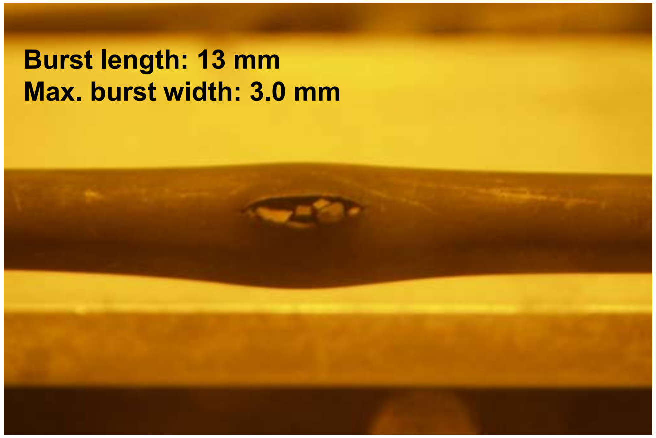
135ºC Offset Strain for Prehydrided 17x17 Zry-4 Oxidized at 1200ºC



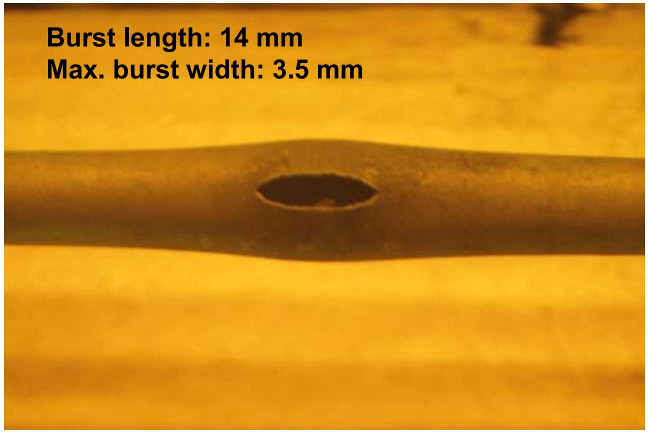
LOCA Integral Test Sequence & Time



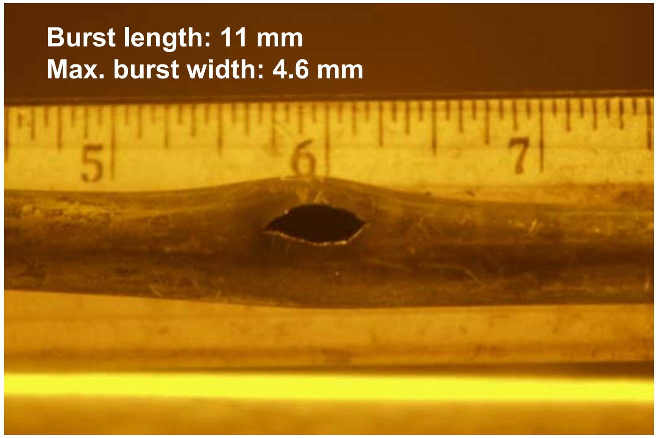
Balloon and Burst Regions for High-burnup Tests



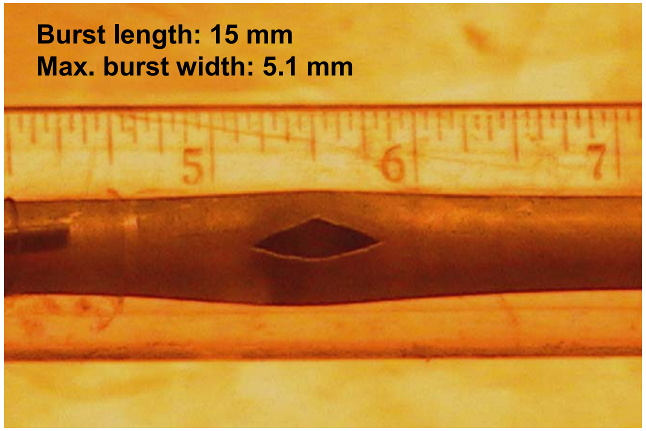
ICL#1: Ramp-to-Burst test conducted in argon



ICL#2: LOCA sequence with 5-minute oxidation at 1204ºC and slow-furnace cooling

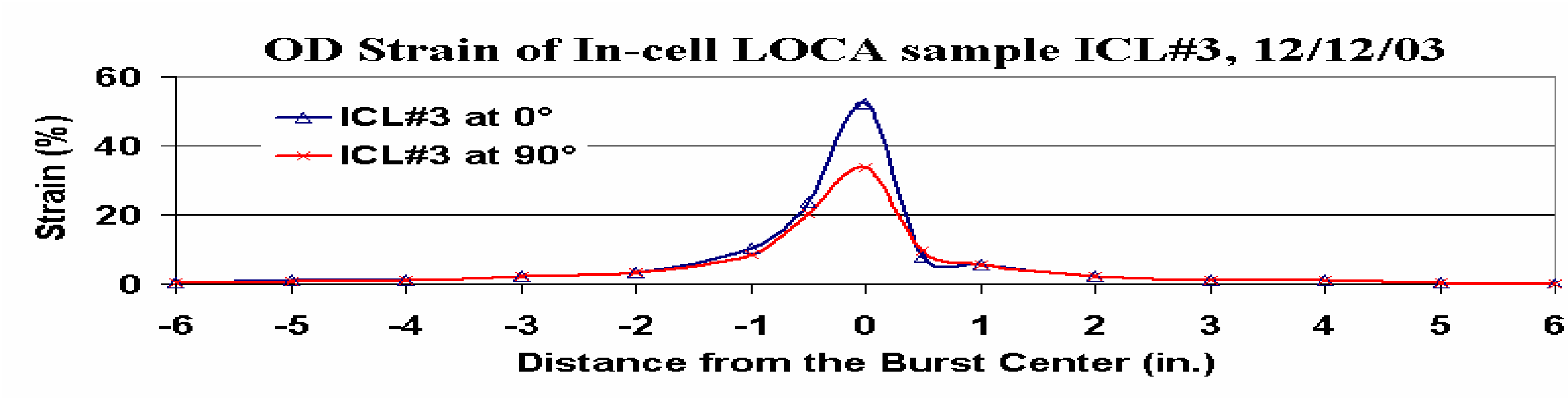


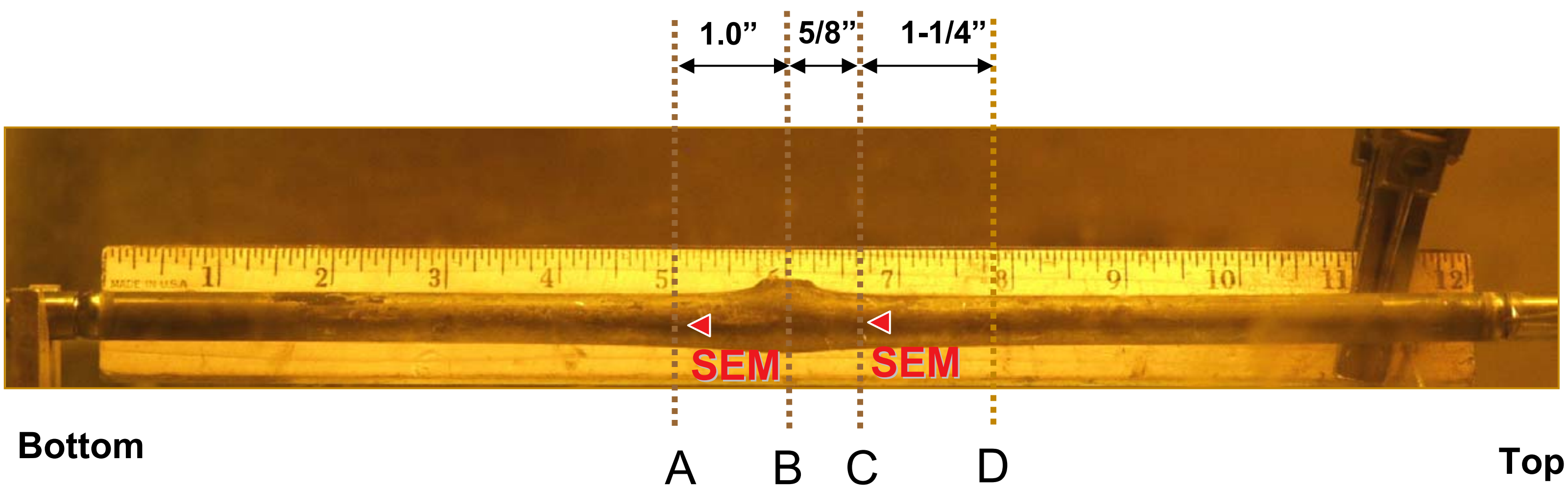
ICL#3: 5-min. oxidation at 1204ºC fFollowed by quench at 800°C (quartz tube failed at 480°C)



ICL#4: Full LOCA sequence (5-minute oxidation at 1204ºC) with quench at 800°C

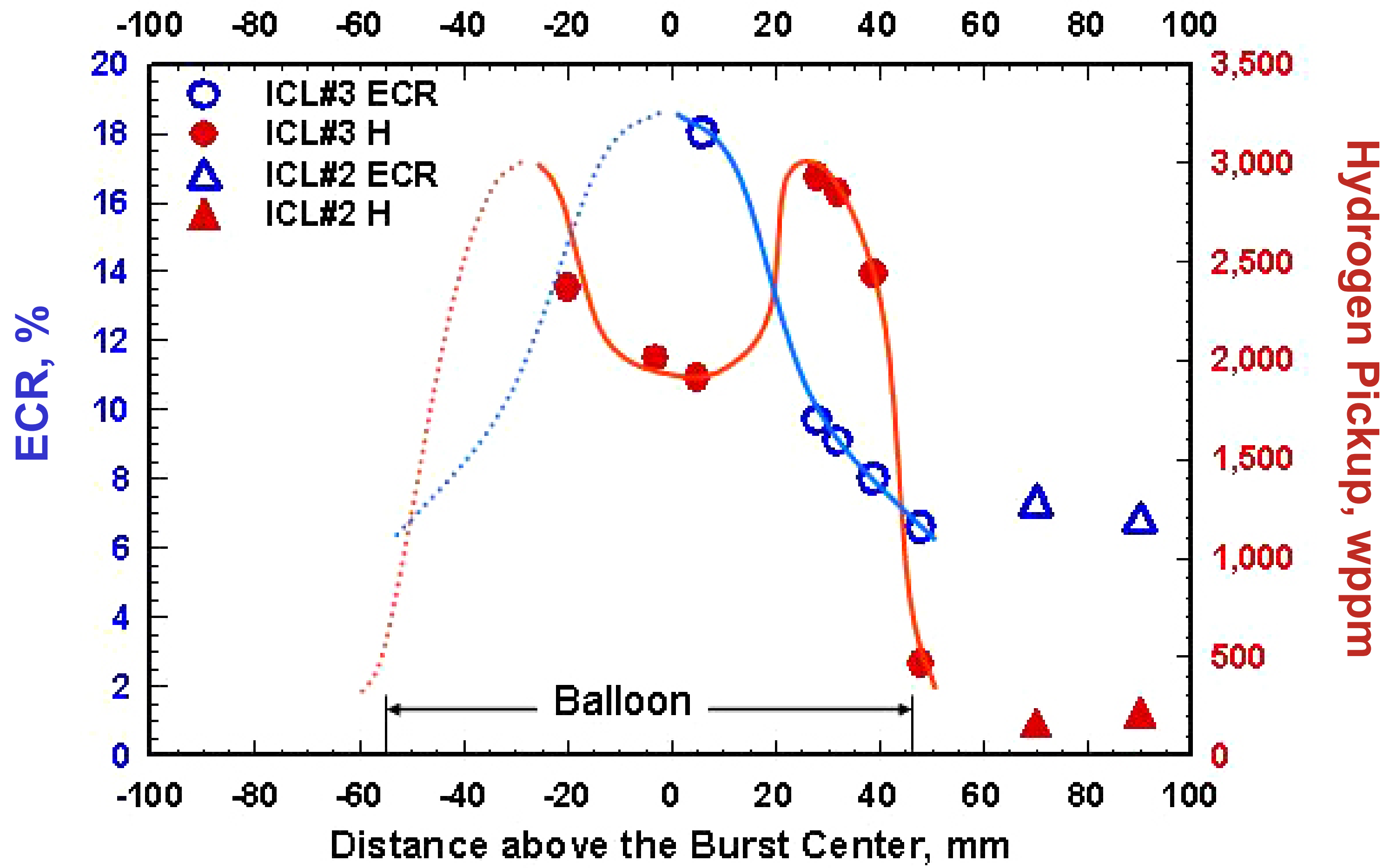
Post-test Characterization for ICL#3 Specimen



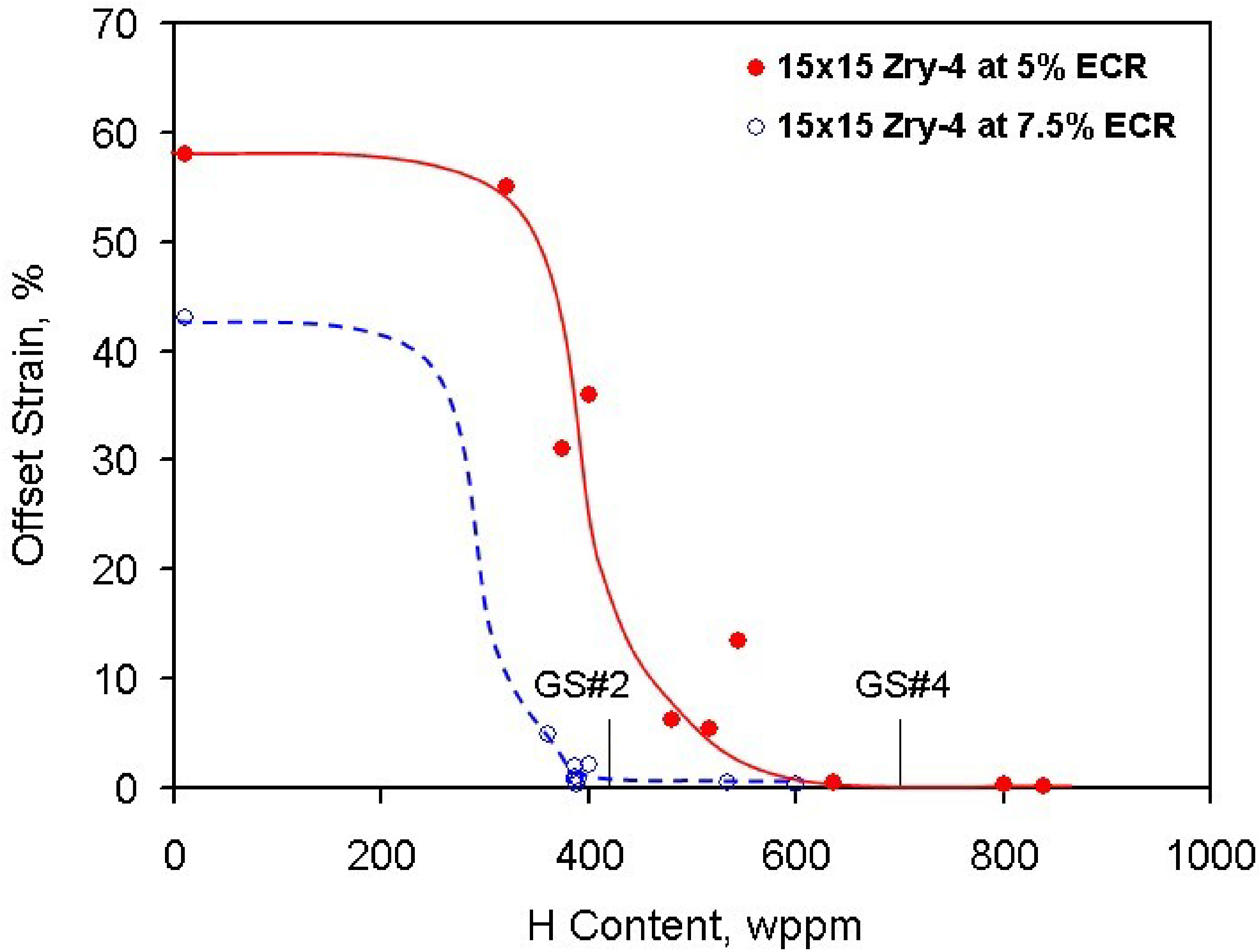


Sample ICL#3 was broken at locations A, B and C during the sample handling before the sectioning was performed at location D.

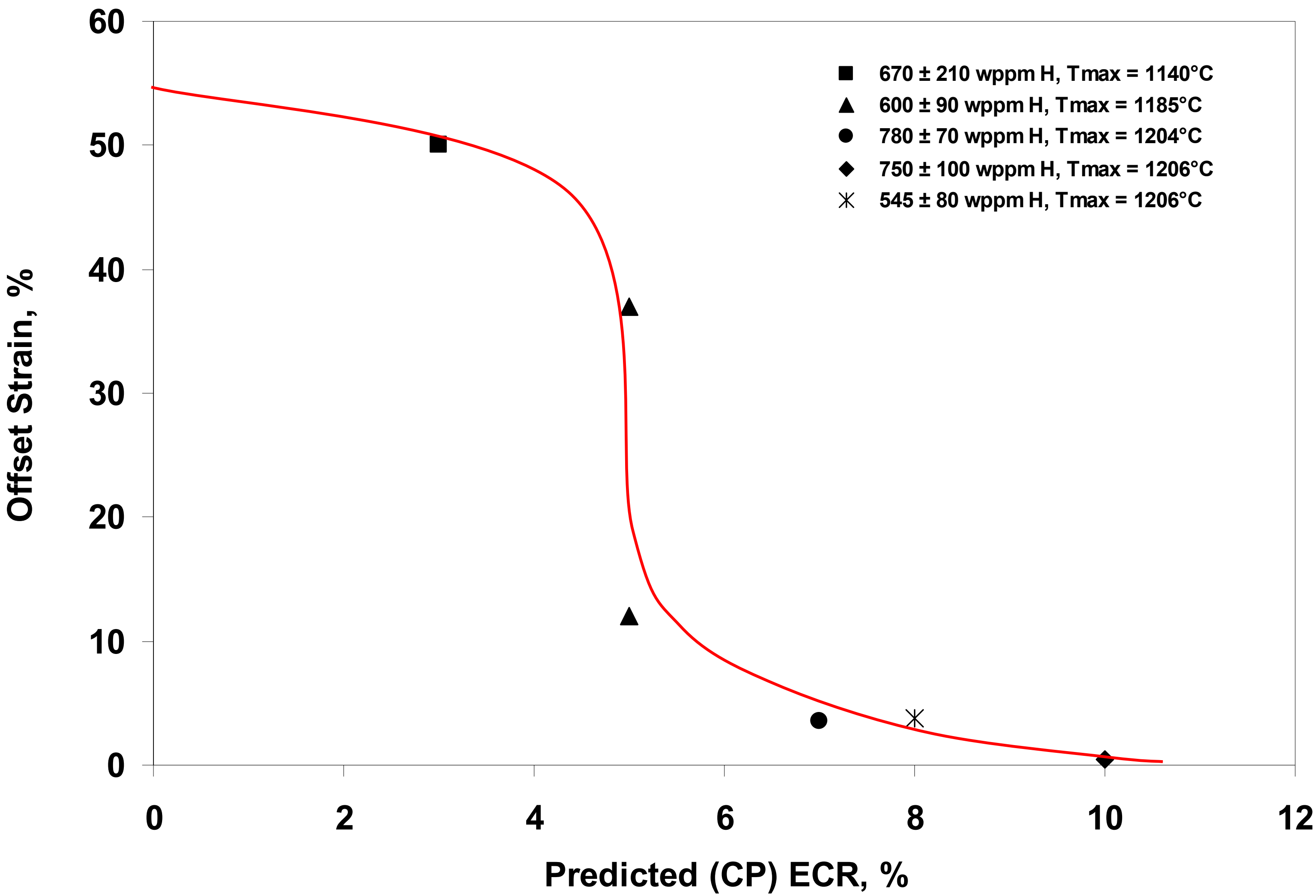
H and O Analyses of ICL#2 and ICL#3



Post-Quench Ductility at 135ºC vs. Hydrogen Content for Prehydrided 15x15 Zry-4 Oxidized at 1204±10ºC



Summary of Post-Oxidation Ductility (Offset Strain) at 135ºC vs. CP-ECR for High-Burnup Zry-4 Cladding



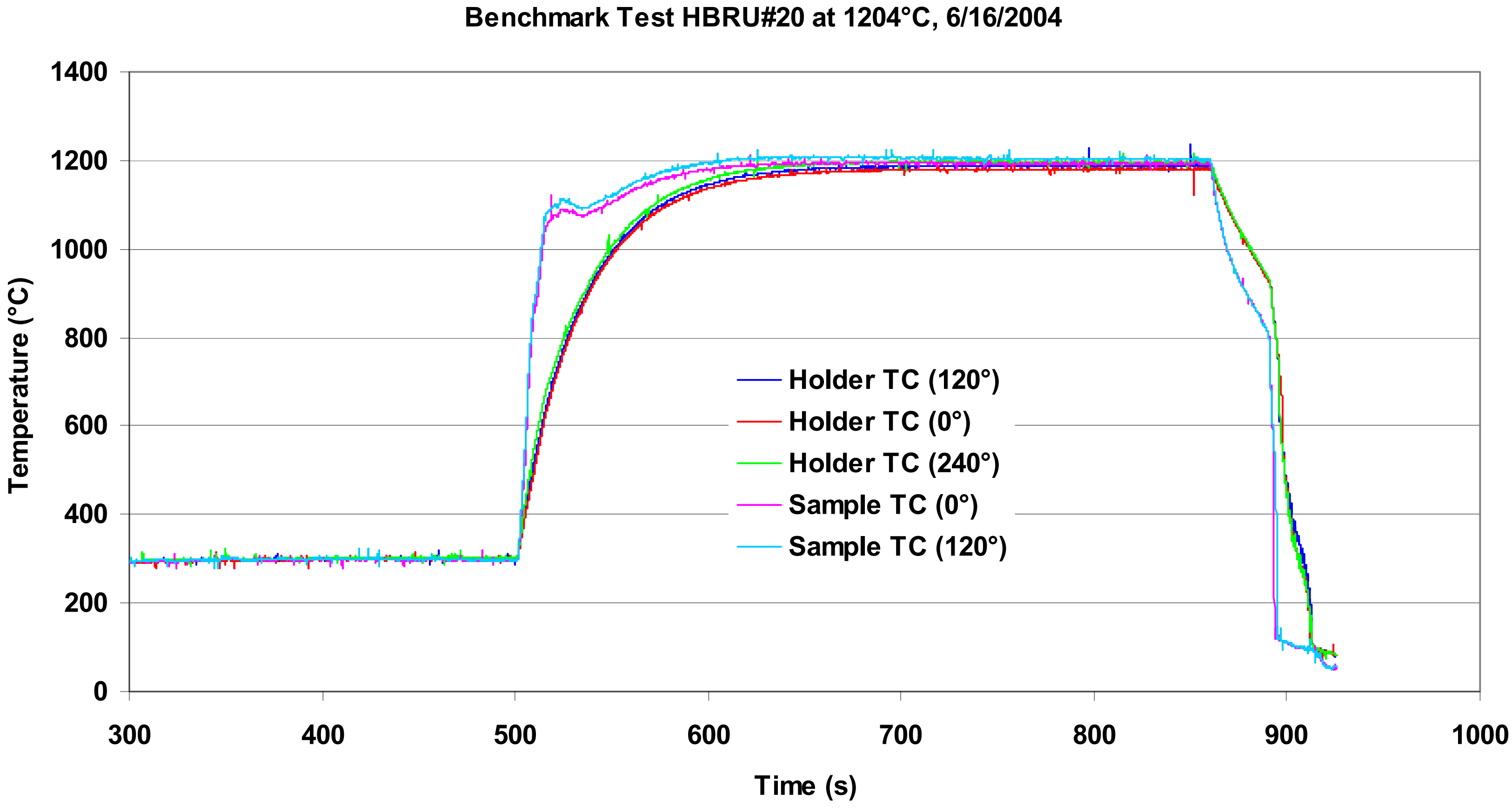
Summary of ANL Testing Program for Non-Deformed 15×15 Zry-4 Rings

- **Baseline Data for Non-Irradiated 15×15 Zry-4 Cladding**
  - Weight gain (1204ºC); PQD vs. ECR, H-content & test temperature
- **HBR Rod Selection (F07) and Characterization**
  - Gamma scanning, corrosion thickness, hydride morphology
  - Hydrogen- and oxygen-content profiles (axial and circumferential)
- **HBR F07 Oxidation Test Samples and Results**
  - 25-mm-long samples cut and defueled from F07 midplane (-40 to 100 mm)
  - Two-sided steam oxidation tests conducted at 1204ºC T_{max} to
    3, 5, 7, 8, 10% ECR without quench
    8% ECR with quench (to be conducted)
- **Post-Oxidation (POD) and Post-Quench Ductility (PQD)**
  - POD offset & permanent strains for ring-compression tests at 135ºC
  - PQD offset & permanent strains for ring-compression tests at 100-135ºC

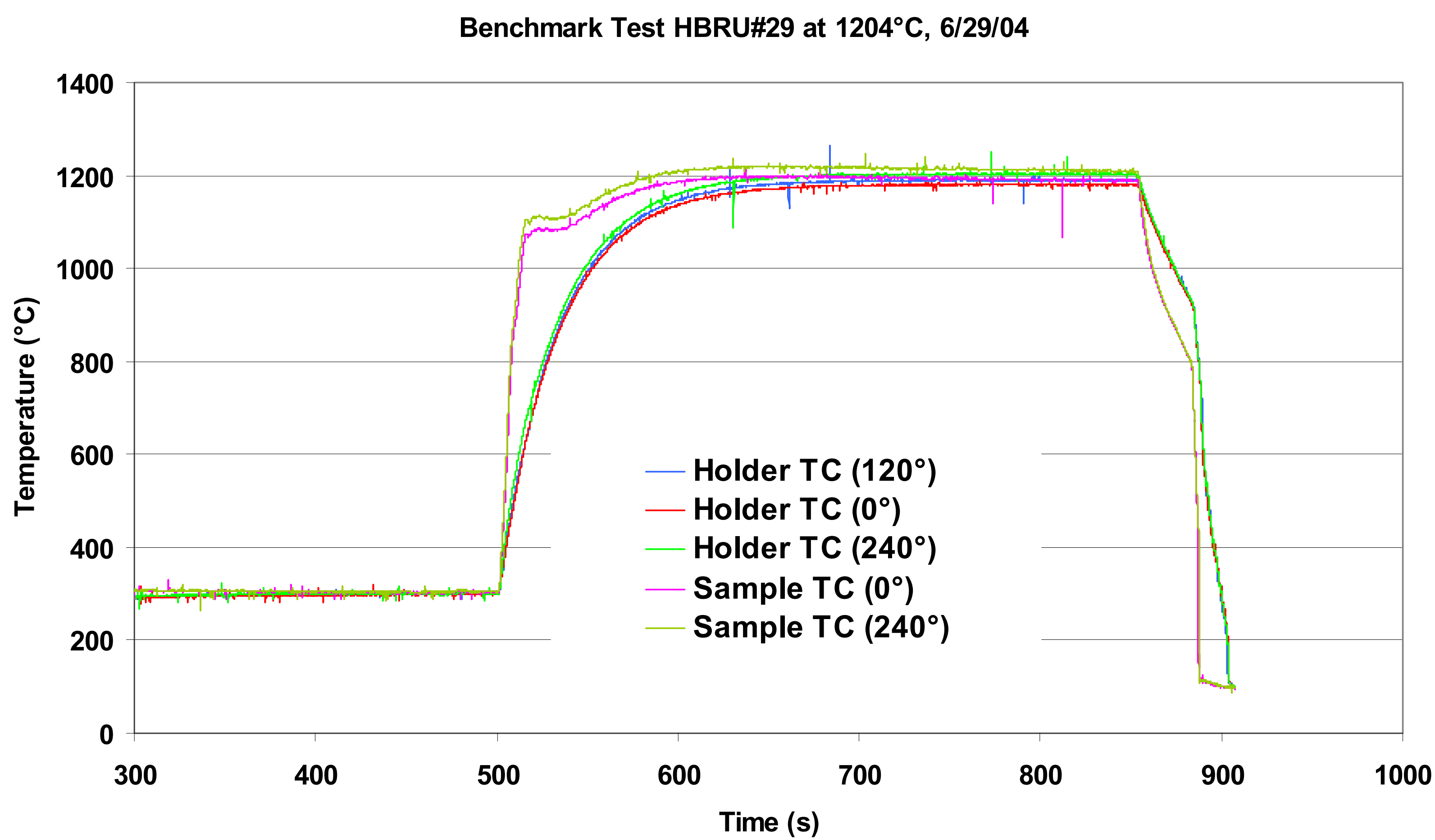
Baseline Data for Non-irradiated 15×15 Zry-4 Cladding

- **Weight Gain due to Steam Oxidation at 1204ºC**
  - Thermal benchmark results with 3 TCs welded onto sample and holder
    Subsequent tests conducted with 3 TCs welded onto holder above sample
  - Data are in excellent agreement with Cathcart-Pawel (CP) correlation
    Hydrogen content and quench have no significant effect on weight gain
    Note 2 data points at about 8.7 mg/cm^2 : one with & one without quench
- **Post-Quench Ductility of 1204ºC-Oxidized/Quenched Zry-4**
  - Nonirradiated 15×15 Zry-4 cladding
    Offset & permanent strains vs. ECR based on measured weight gain
    Ductile-to-brittle trans. ECR: 8% at RT, 11.5% at 100ºC, 14% at 135ºC
    Offset strain vs. ECR calculated with CP correlation: trend curves
  - Pre-hydrided, non-irradiated 15×15 Zry-4 cladding; 135ºC tests
    5% CP-ECR: ductile-to-brittle transition H-content ≈ 600 wppm
    7.5% CP-ECR: ductile-to-brittle transition H-content ≈ 400 wppm

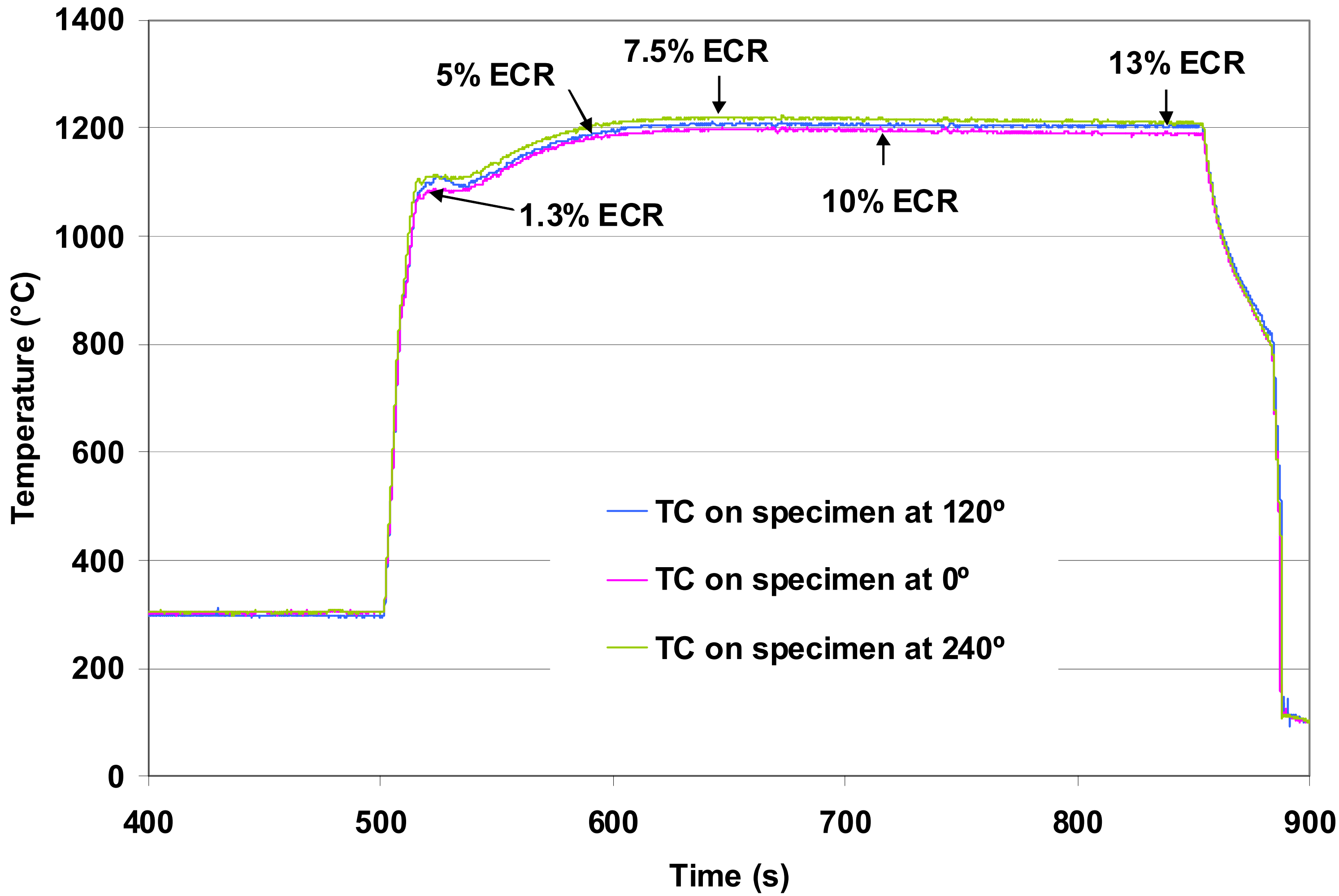
Comparison of Inconel Holder TC Readings vs. Cladding Sample TC Readings



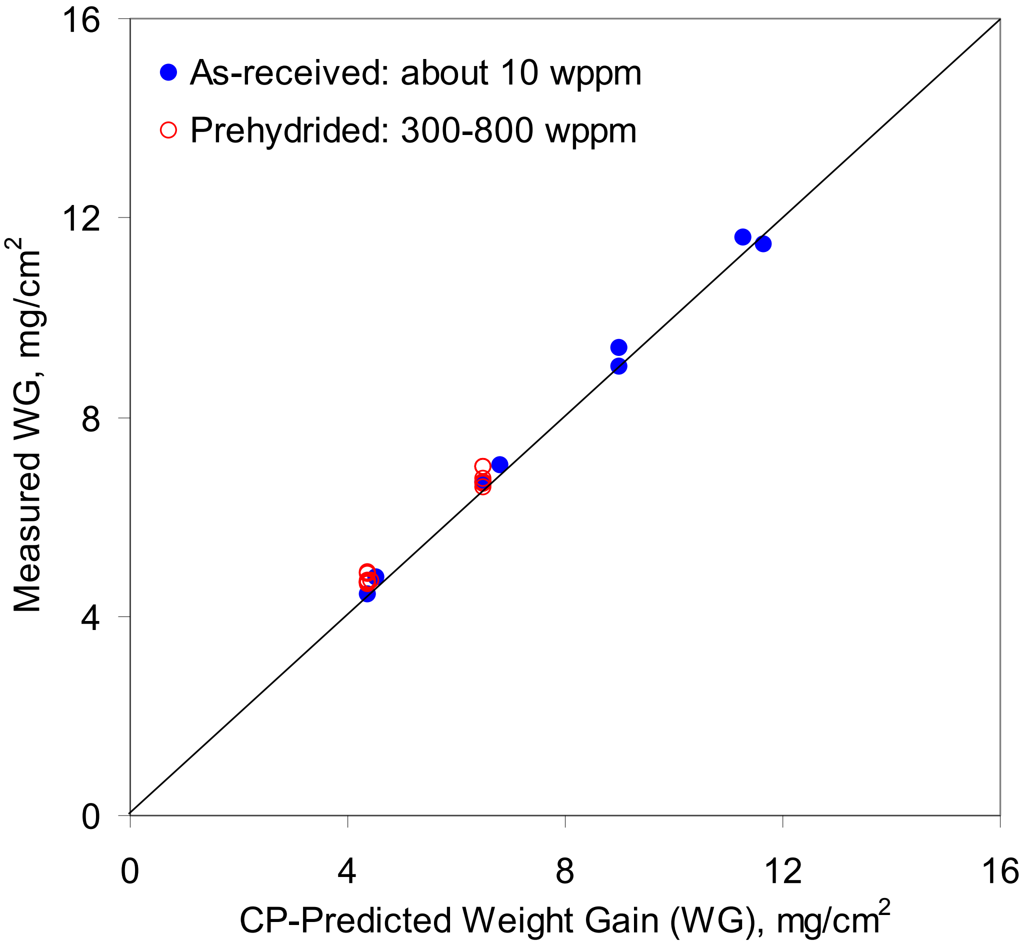
Comparison of Inconel Holder TC Readings vs. Cladding Sample TC Readings (Cont'd)



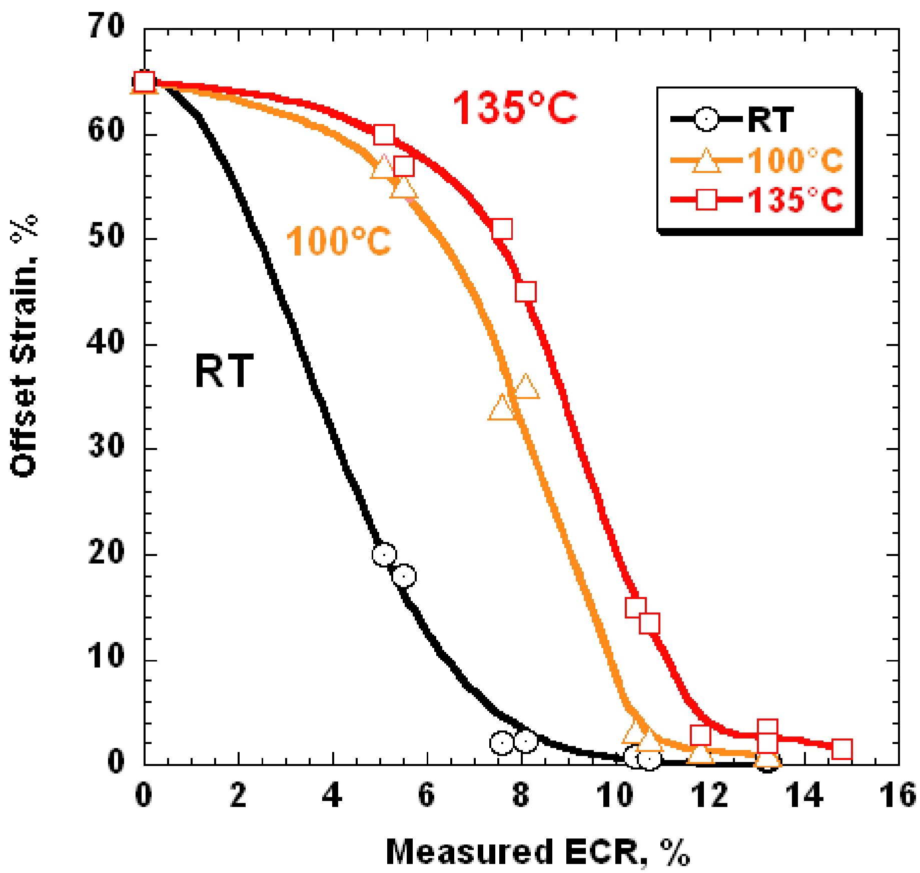
Temperature History for 1204±10°C Steam Oxidation of 15×15 Zry-4 Post-Quench-Ductility Samples



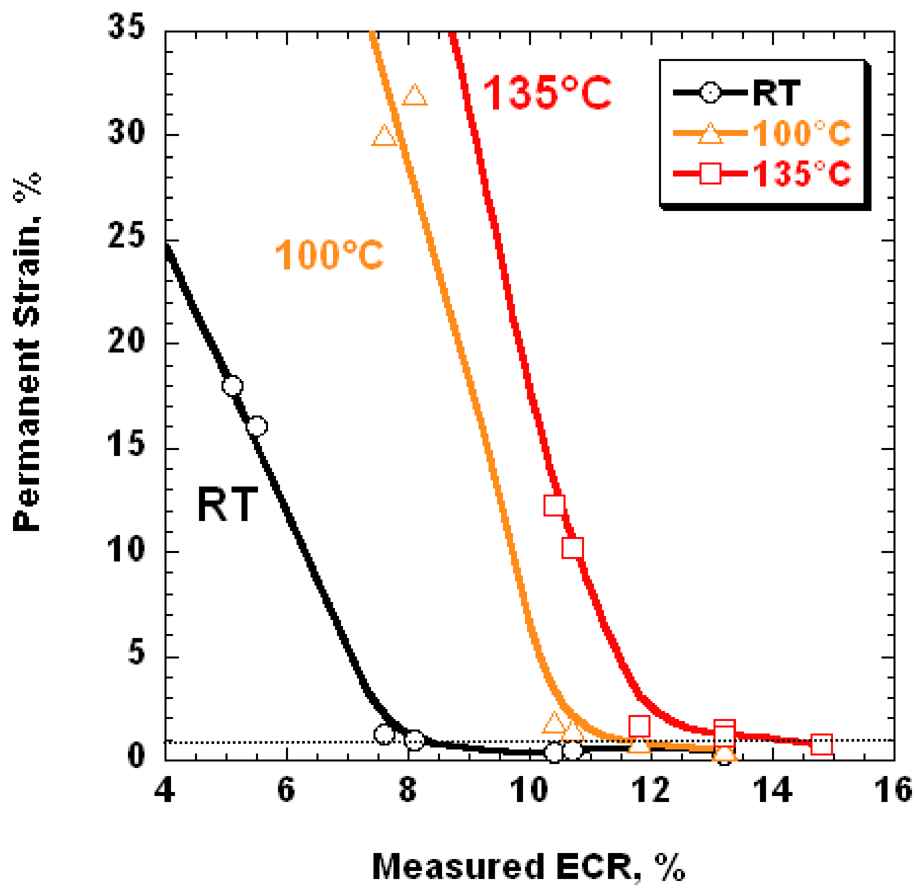
Weight Gain of As-Received & Pre-Hydrided 15×15 Zry-4 Cladding following Steam oxidation at 1204±10°C



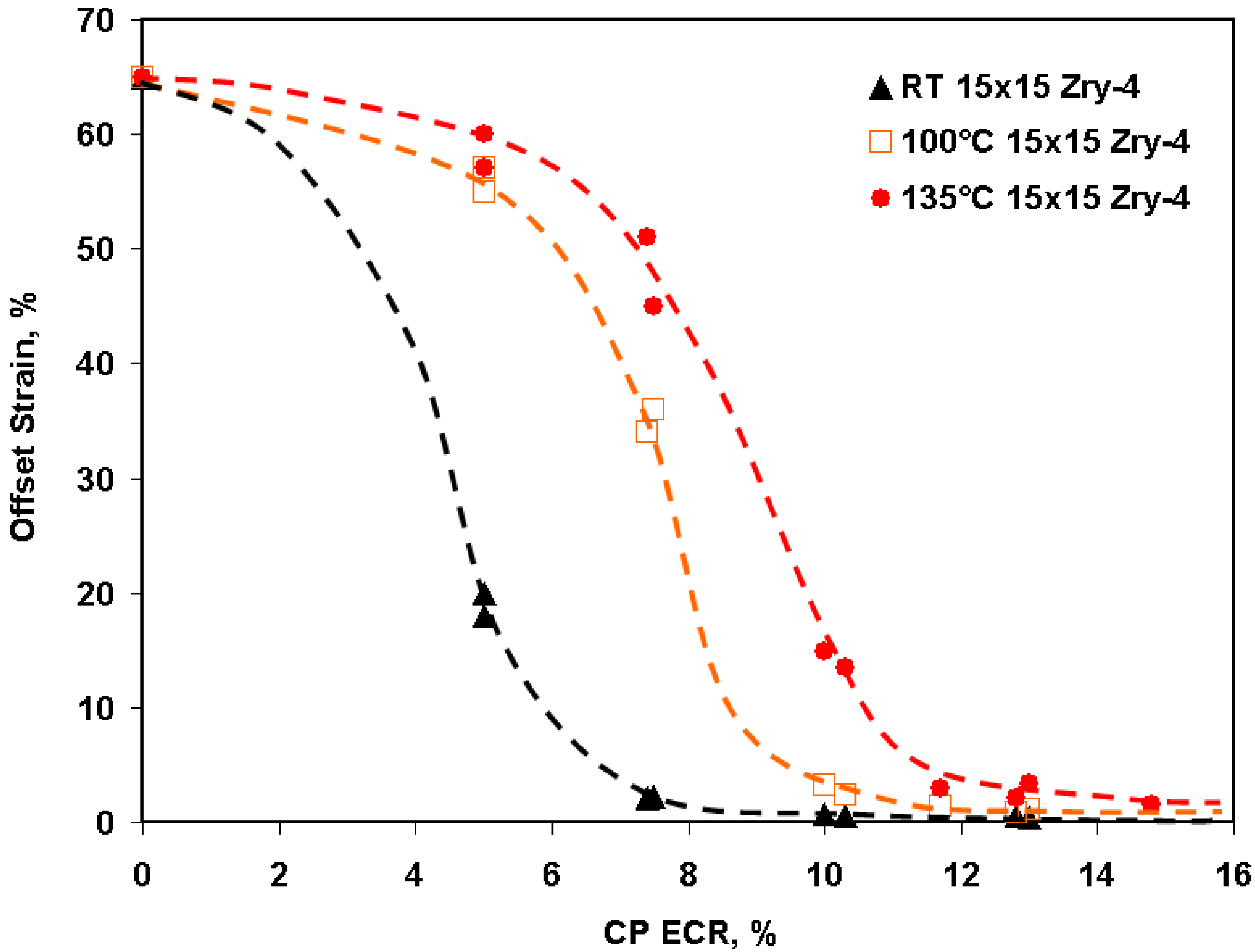
Ring-Compression Offset Strain vs. Measured ECR for 15x15 Zry-4 Oxidized at 1204±10ºC



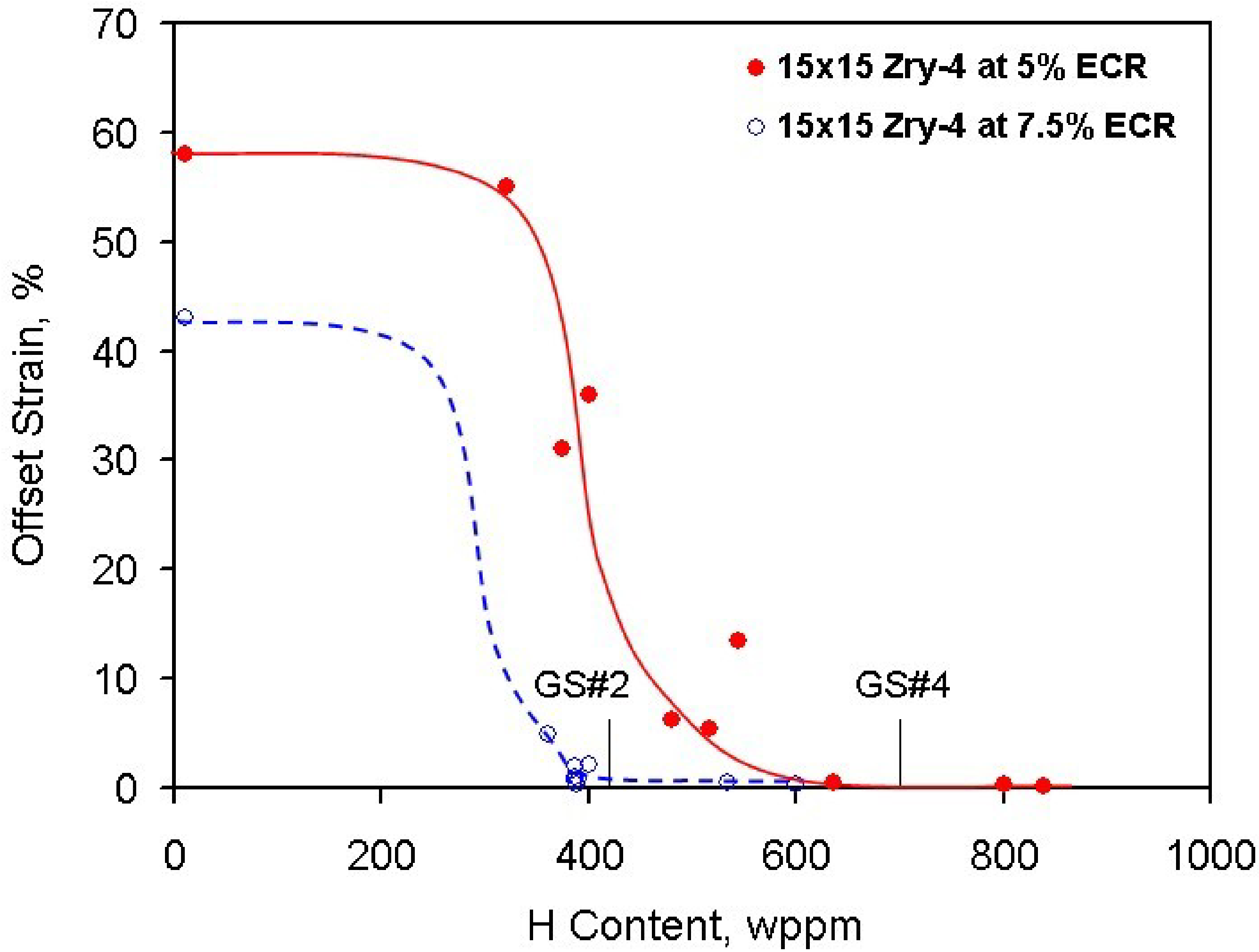
Ring-Compression Permanent Strain vs. Measured ECR for 15x15 Zry-4 Oxidized at 1204±10ºC



Offset Strain vs. CP-ECR for As-Received 15x15 Zry-4 Oxidized at 1204±10ºC



Post-Quench Ductility at 135ºC vs. Hydrogen Content for Prehydrided 15x15 Zry-4 Oxidized at 1204±10ºC



Discussion of Baseline Data for 15x15 Zry-4

- **Weight Gain Data not Plotted in Figure**
  - Results from 0304 test train benchmarked to 1200±17ºC
    Good agreement with CP model for 6 data points at 5-17% ECR
  - Results from 0604 test train also generated at 1174, 1184 & 1194ºC
  - Results from new (1204) test train at 1204±10ºC: 5% & 10% ECR
- **Post-Quench Ductility (PQD) Data: 0604 Test Train**
  - As-Received Zry-4
    PQD (low) the same for 1184-1204ºC oxidation/quench at 13% ECR
    PQD enhancement for 1174ºC oxidation temperature:
    Permanent strain increase: 1.5→3.4% at 135ºC, 0.6→1.0% at 100ºC
  - Pre-hydrided Zry-4 – Low Transition ECR Value Depends on T-Ramp
    At end of 5% ECR oxidation, Avg. T = 1193ºC & Max. T = 1204ºC
    At end of 7.5% ECR oxidation, Avg. T = 1208ºC & Max. T = 1218ºC

HBR Rod Selection for Oxidation, PQD and LOCA Tests

- **Edge-Near-Corner Rods: A02, B01, R01, S02**
  - Characterization of A02 and R01
    Effective flow and moderation area not uniform around rods
    Fuel & cladding temperatures not symmetric with fuel centerline axis
    Significant hoop variation in cladding hydrogen content (≤ ±150 wppm)
    Significant hoop variation in hydride morphology
- **Rods at Corners of Guide Tubes: B05, E02, R05, E14**
  - May have more symmetric cladding temperatures than edge rods
  - Gamma scanning completed for Rod E02 – may be okay for testing
- **Interior Rods not Next to Guide Tubes: F07, G10**
  - Should have axi-symmetric power profile & cladding temperatures
  - Should have more uniform hydrogen content and hydride morphology
  - **Select Rod F07 for initial characterization and testing**

Final Configuration of HBR Rods

Rod B01, Rod E02, Rod R01, Rod A02, Rod S02, Rod B05, Rod R05, Rod F07, Rod G10, Rod E14

	A	B	C	D	E	F	G	H	K	L	M	N	P	R	S
1	FRSC A01	G-38 RA18302 E04	FRSC C01		FRSC E01		FRSC G01		FRSC K01		FRSC M01		FRSC P01	G-38 RA110889 M04	FRSC S01
2	G-38 RA18235 D05	FRSC B02		FRSC D02	G-38 RA18293 F04	FRSC F02		FRSC H02		FRSC L02	G-38 L04	FRSC N02		FRSC R02	G-38 RA19865 N05
3	FRSC A03		G		FRSC E03	G	FRSC G03		FRSC K03	G	FRSC M03		G		FRSC S03
4		FRSC B04		FRSC D04		FRSC F04		G		FRSC L04		FRSC N04		FRSC R04	
5	FRSC A05	G-38 RA18229 D06	FRSC C05		G		FRSC G05	S-31 BL219926 H05	FRSC K05		G		FRSC P05	G-38 RA1[illegible] N06	FRSC S05
6		FRSC B06	G	FRSC D06		FRSC F06	G-38 G06	FRSC H06	G-38 K06	FRSC L06		FRSC N06	G	FRSC R06	
7	FRSC A07		FRSC C07		FRSC E07	G-38 RA18308 F07	FRSC G07		FRSC K07	G-38 L07	FRSC M07		FRSC P07		FRSC S07
8		FRSC B08		G	S-31 E08	FRSC F08		INST		FRSC L08	S-31 M08	G		FRSC R08	
9	FRSC A09		FRSC C09		FRSC E09	G-38 RA18310 F09	FRSC G09		FRSC K09	G-38 L09	FRSC M09		FRSC P09		FRSC S09
10	S-15 BG02438 A10	FRSC B10	G	FRSC D10		FRSC F10	G-38 RA18300 G10	FRSC H10	G-38 K10	FRSC L10		FRSC N10	G	FRSC R10	
11	FRSC A11	G-38 RA18209 D10	FRSC C11		G		FRSC G11	S-31 H11	FRSC K11		G		FRSC P11	G-38 N10	FRSC S11
12		FRSC B12		FRSC D12		FRSC F12		G		FRSC L12		FRSC N12		FRSC R12	
13	FRSC A13		G		FRSC E13	G	FRSC G13		FRSC K13	G	FRSC M13		G		FRSC S13
14	G-38 RA18210 D11	FRSC B14		FRSC D14	G-38 RA18311 F12	FRSC F14		FRSC H14		FRSC L14	G-38 L12	FRSC N14		FRSC R14	G-38 N11
15	FRSC A15	G-38 RA18219 E12	FRSC C15		FRSC E15		FRSC G15		FRSC K15		FRSC M15		FRSC P15	G-38 M12	FRSC S15

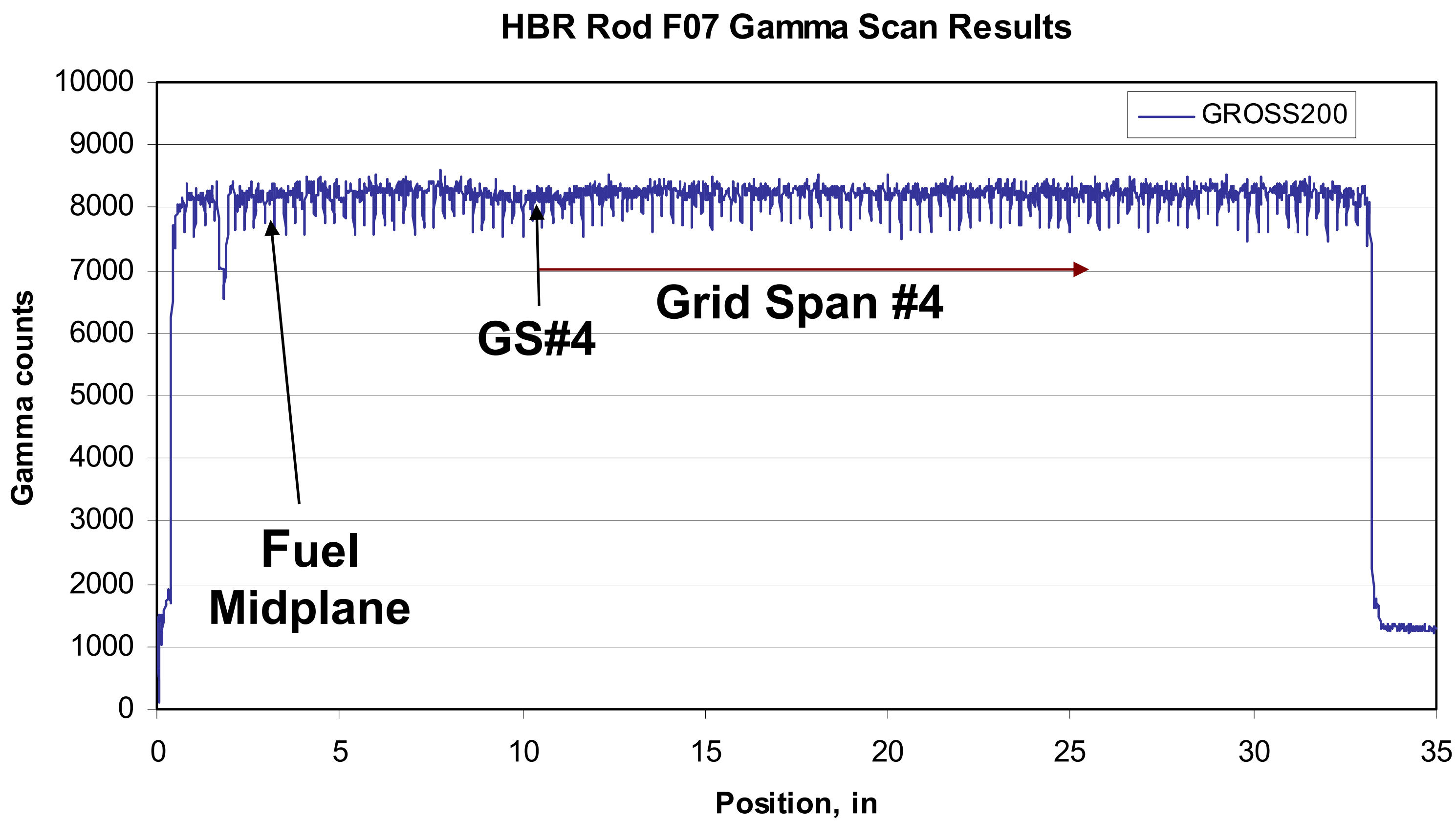
FRSC = Fuel Rod Storage Can, these rods were installed at EOC 14 to extend Assmbly S-15 life.
S-31 = 10 wt% Gadolinia rods originally from Assembly S-31, installed in S-15 at EOC 14.
G-38 = 24 High Burnup rods with 5 cycles of Irradiation, installed for 2 more cycles into S-15 at EOC 14.
The remainder of the rods were from the original Assembly S-15, which was redesignated as S-15H after rebuild.

[shaded box] = Fuel rods removed for Hot Cell Examination

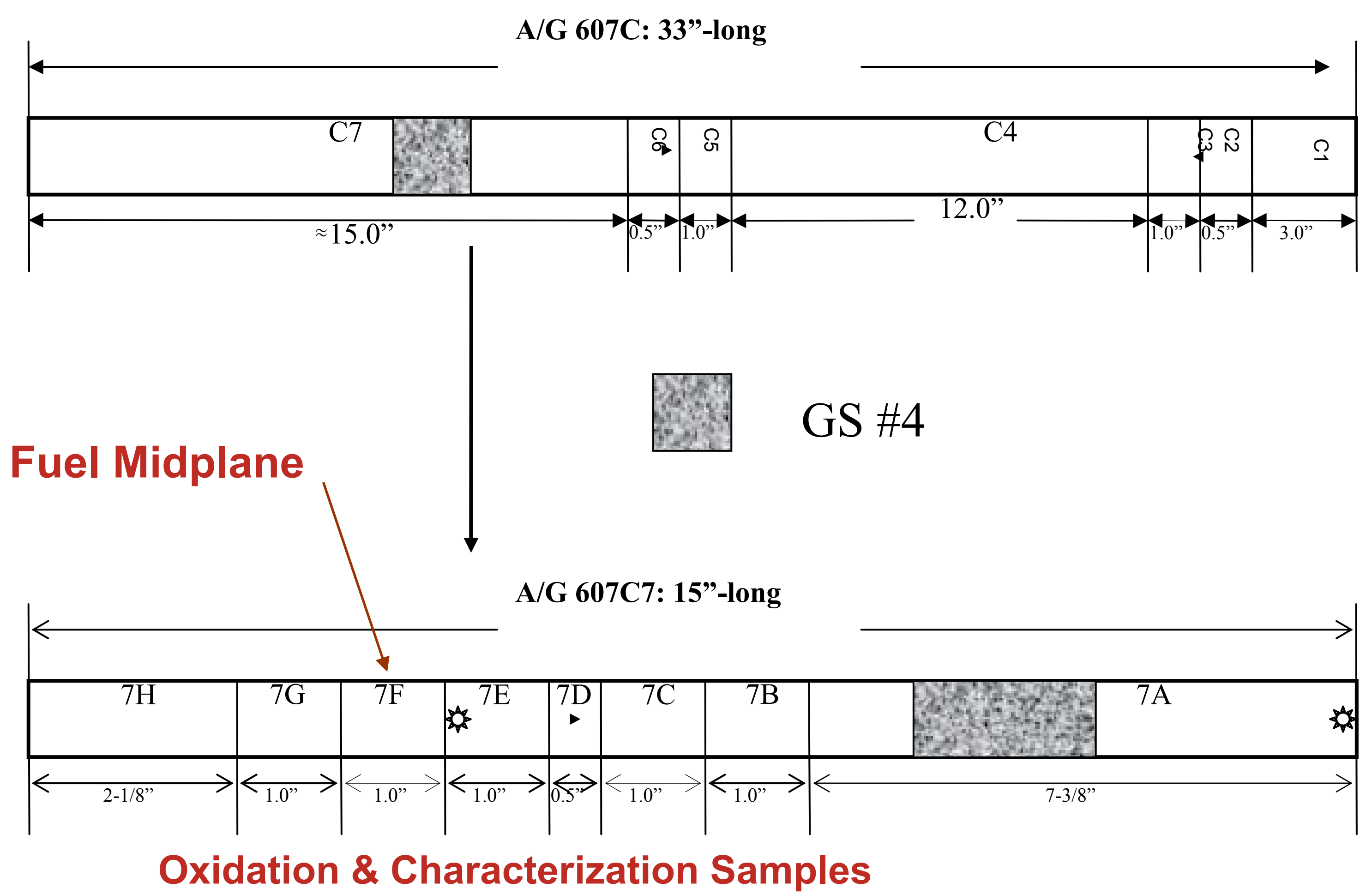
Characterization of HBR Rod F07

- **Gamma Scanning of Middle Segment (C)**
  - Middle of grid spacer #4 is ≈250 mm from bottom of Segment C
  - Fuel midplane is ≈160 mm below center of grid spacer #4
- **Characterization, Oxidation, LOCA Sample Locations**
  - Metallography at 50 mm, 300 mm & 660 mm above fuel midplane
  - H & O analysis at 25 mm, 320 mm and 650 mm above fuel midplane
  - Oxidation samples at -40 to 100 mm above fuel midplane
- **Characterization Results**
  - **Fuel morphology: heat generation and T are not axisymmetric!**
  - **Metallography at +50 mm : oxide layer thickness = 71±5 µm**
    Hydride morphology: relatively uniform in hoop (θ) direction
  - **H-content: 760±60 wppm at +13 mm; 670±80 wppm at +32 mm**
    Too high and too much axial variation: pellet-pellet interfaces??
  - **O-content: 2.08±0.19 wt.% at +35 mm above midplane**

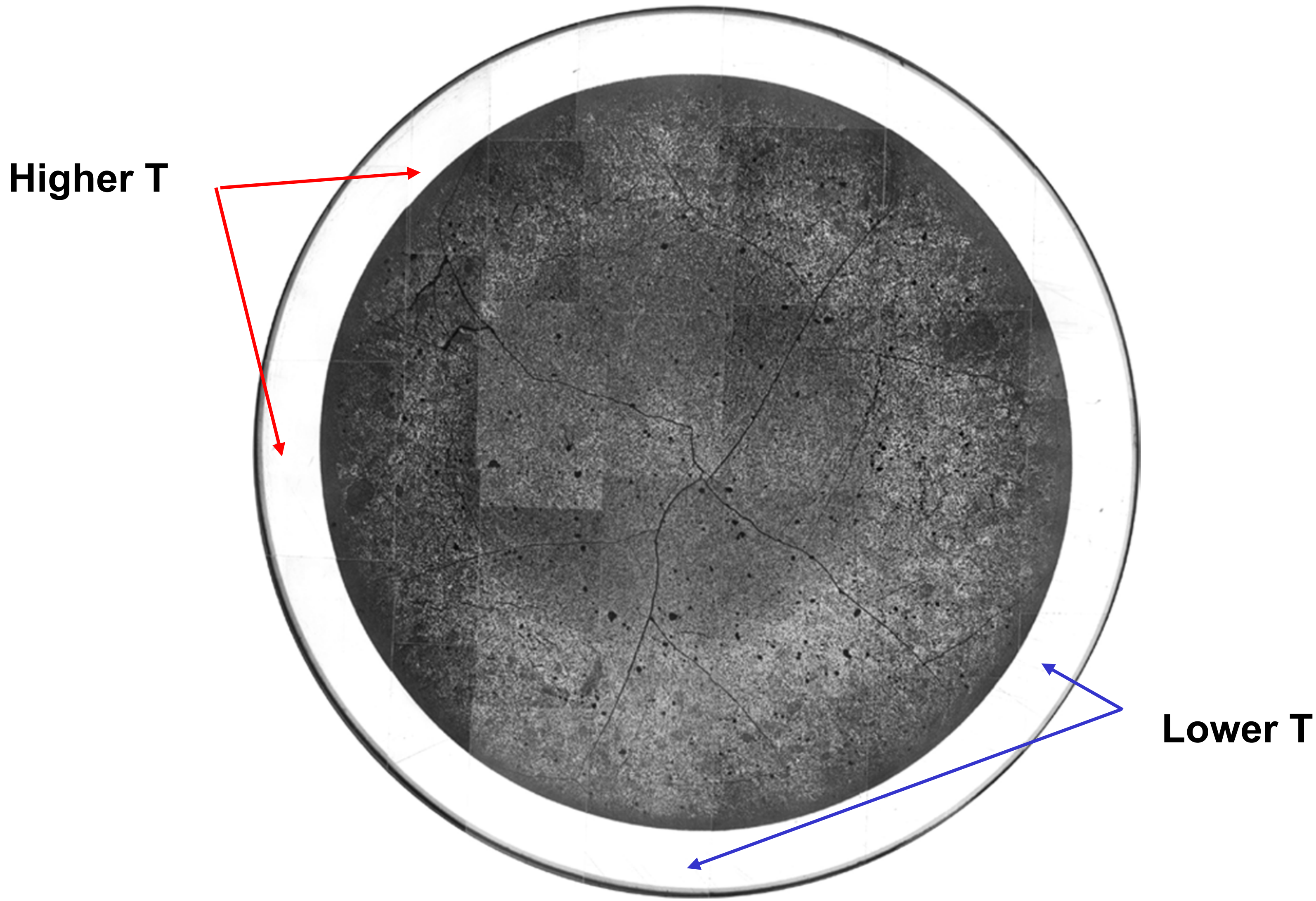
Gamma Scan Results: Mid-segment (C) of HBR Rod F07 Note: Dips at Pellet-Pellet Interfaces Every ≈7 mm



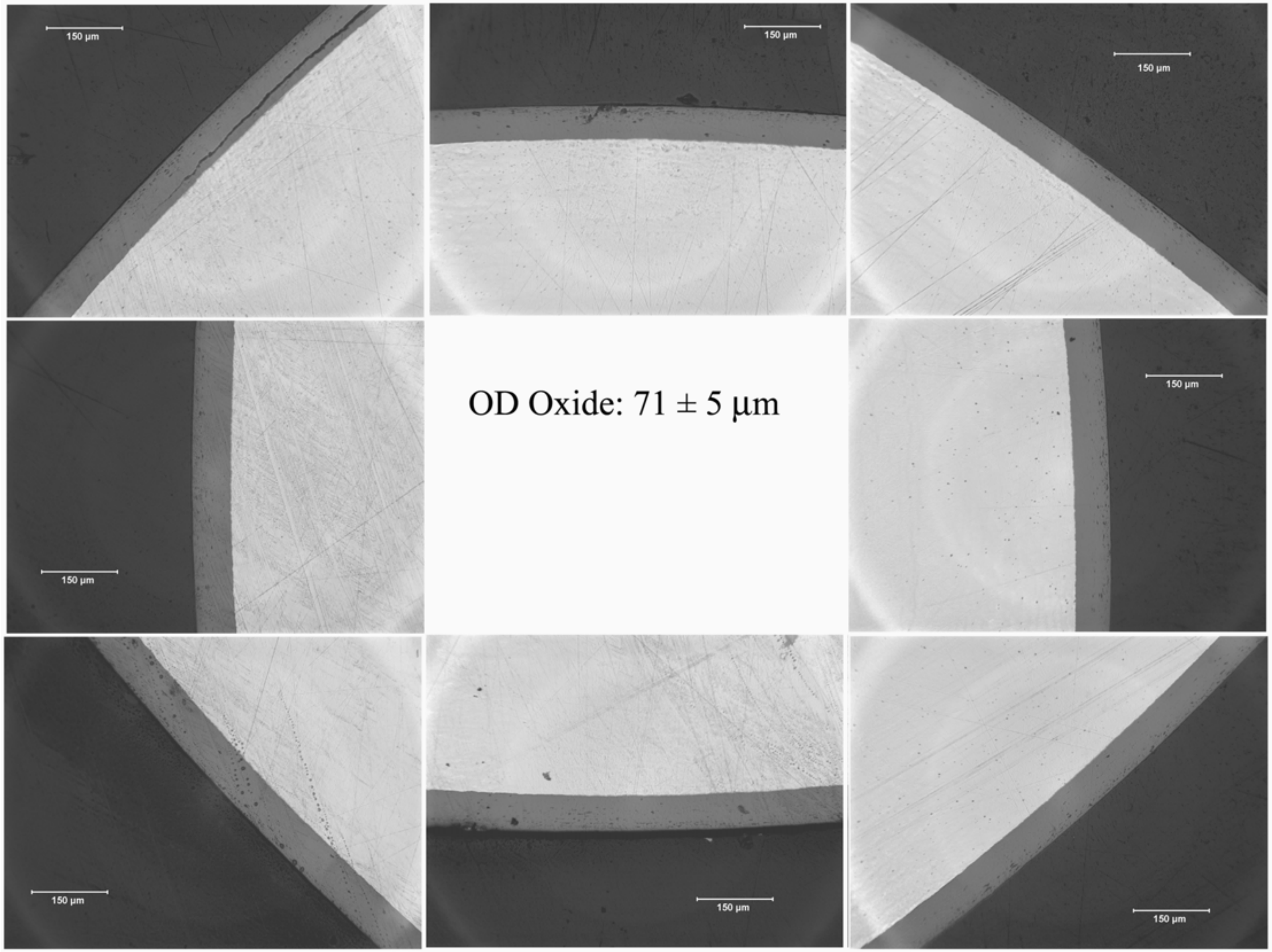
Sectioning Diagram for HBR Rod F07 Segment C



Fuel Mosaic for HBR Rod F07 Cladding at ≈50 mm above Fuel Midplane

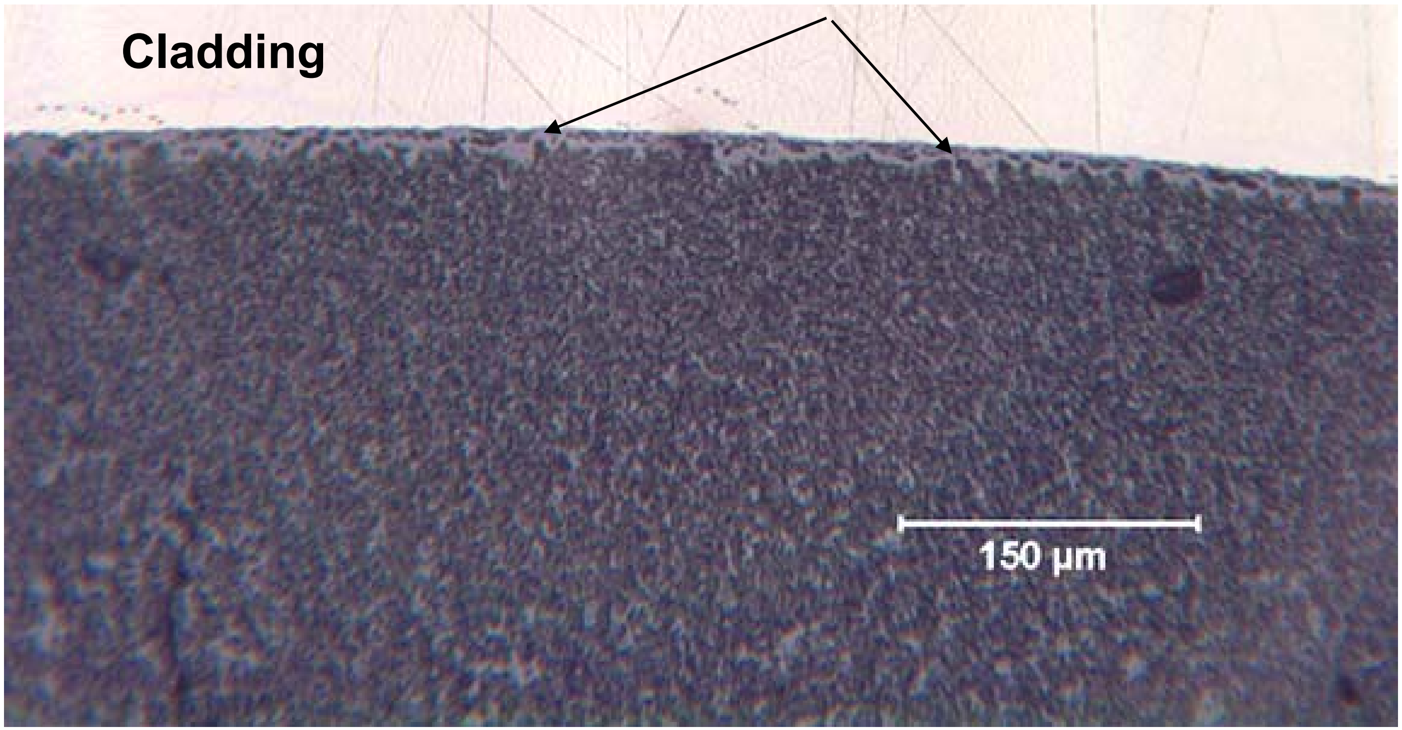


Oxide Layer Thickness for HBR Rod F07 Cladding at ≈50 mm above Fuel Midplane



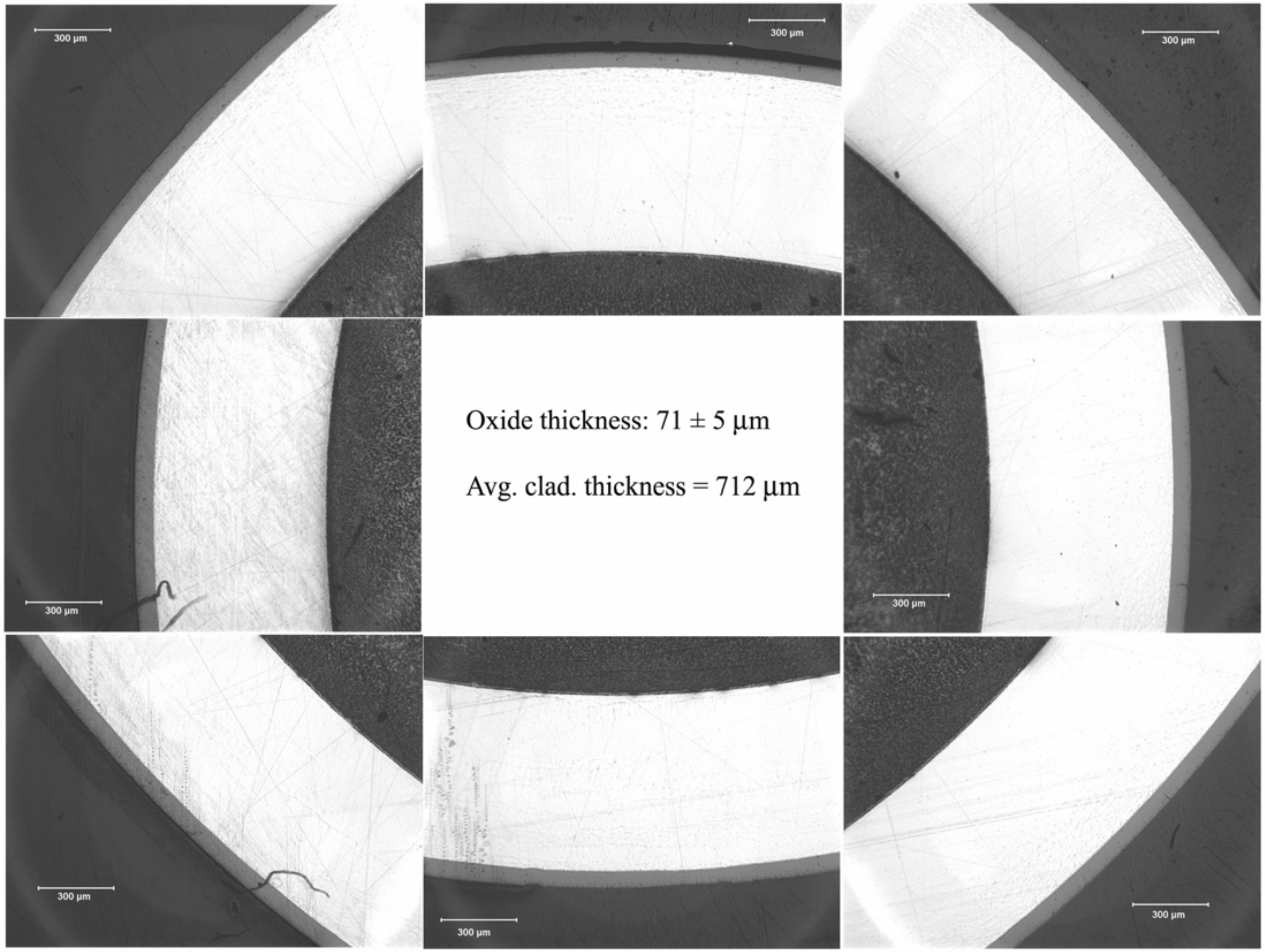
Fuel-Cladding Interface for HBR Rod F07 Cladding at ≈50 mm above Fuel Midplane

Fuel-Cladding Bond: ≈11±4 μm
(Remains after Defueling in RT HNO_3)

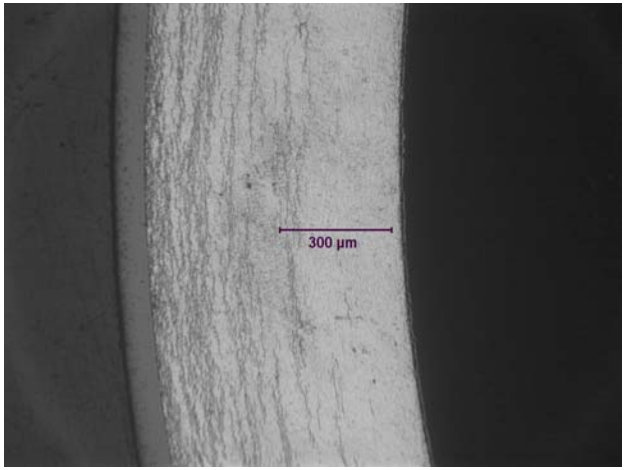


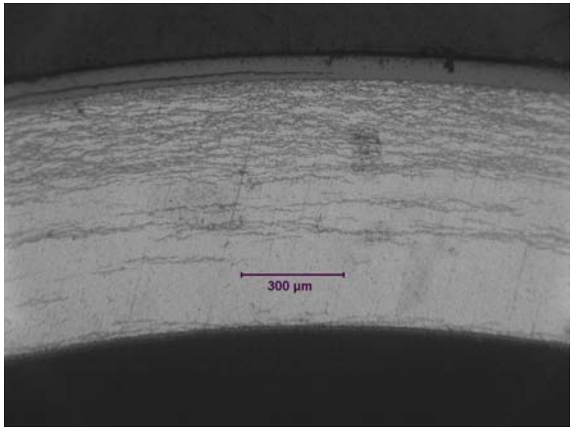
Fuel Rim Region

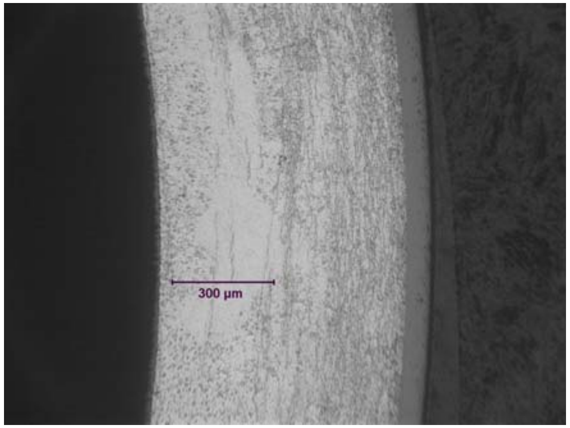
Average Wall Thickness for HBR Rod F07 Cladding at ≈50 mm above Fuel Midplane

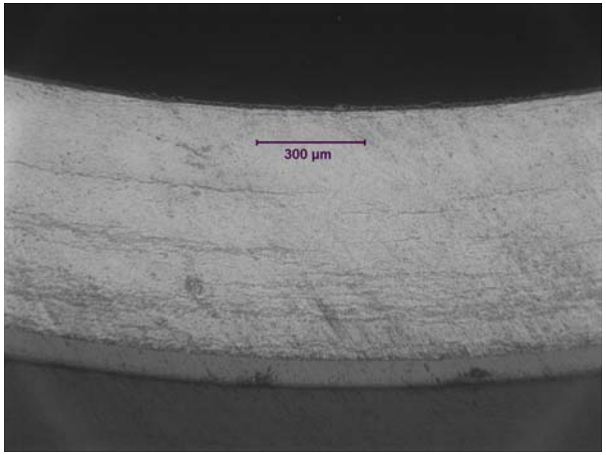


Hydride Morphology for HBR Rod F07 Cladding at ≈50 mm above Fuel Midplane (Low Mag.)

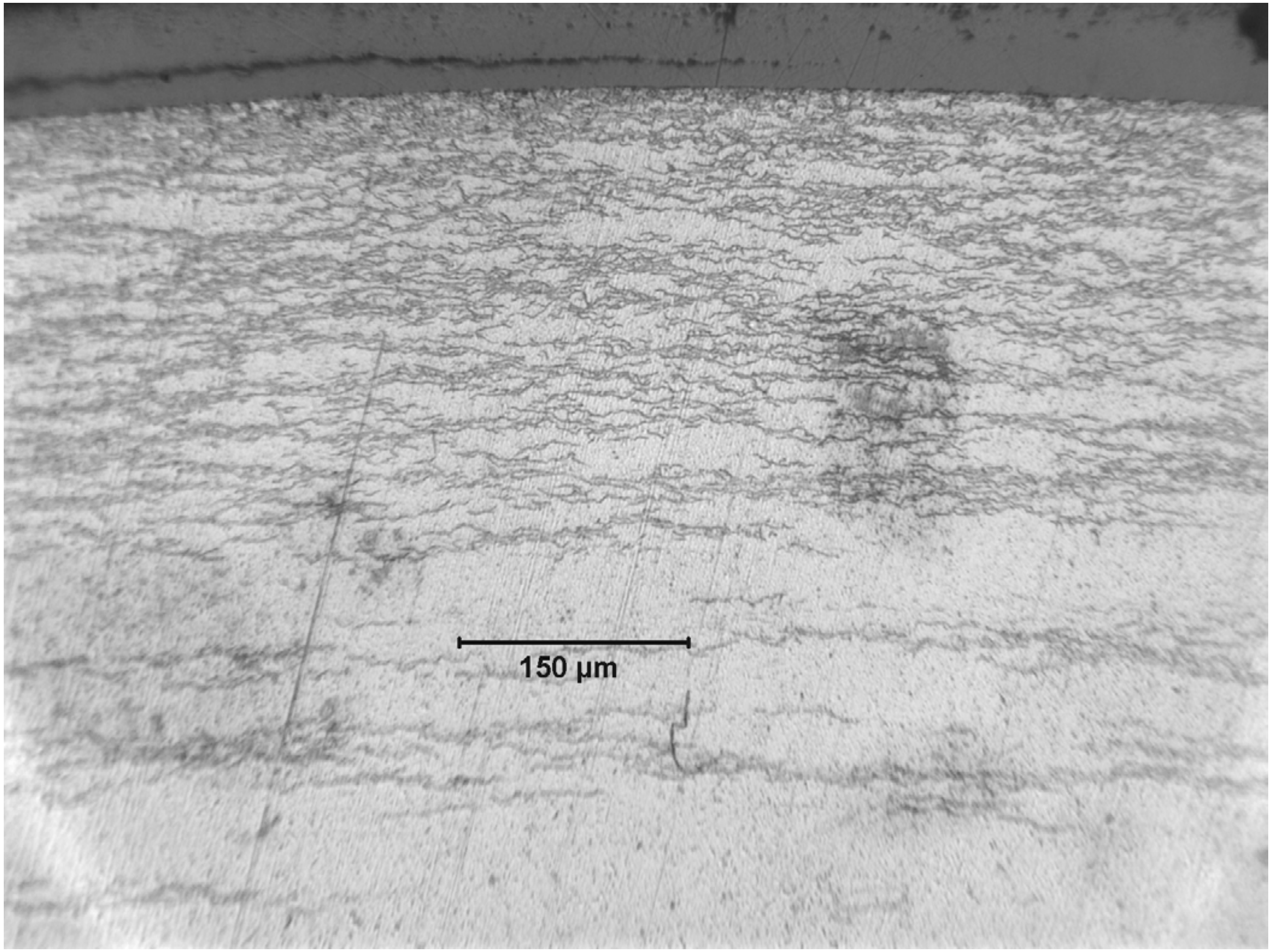




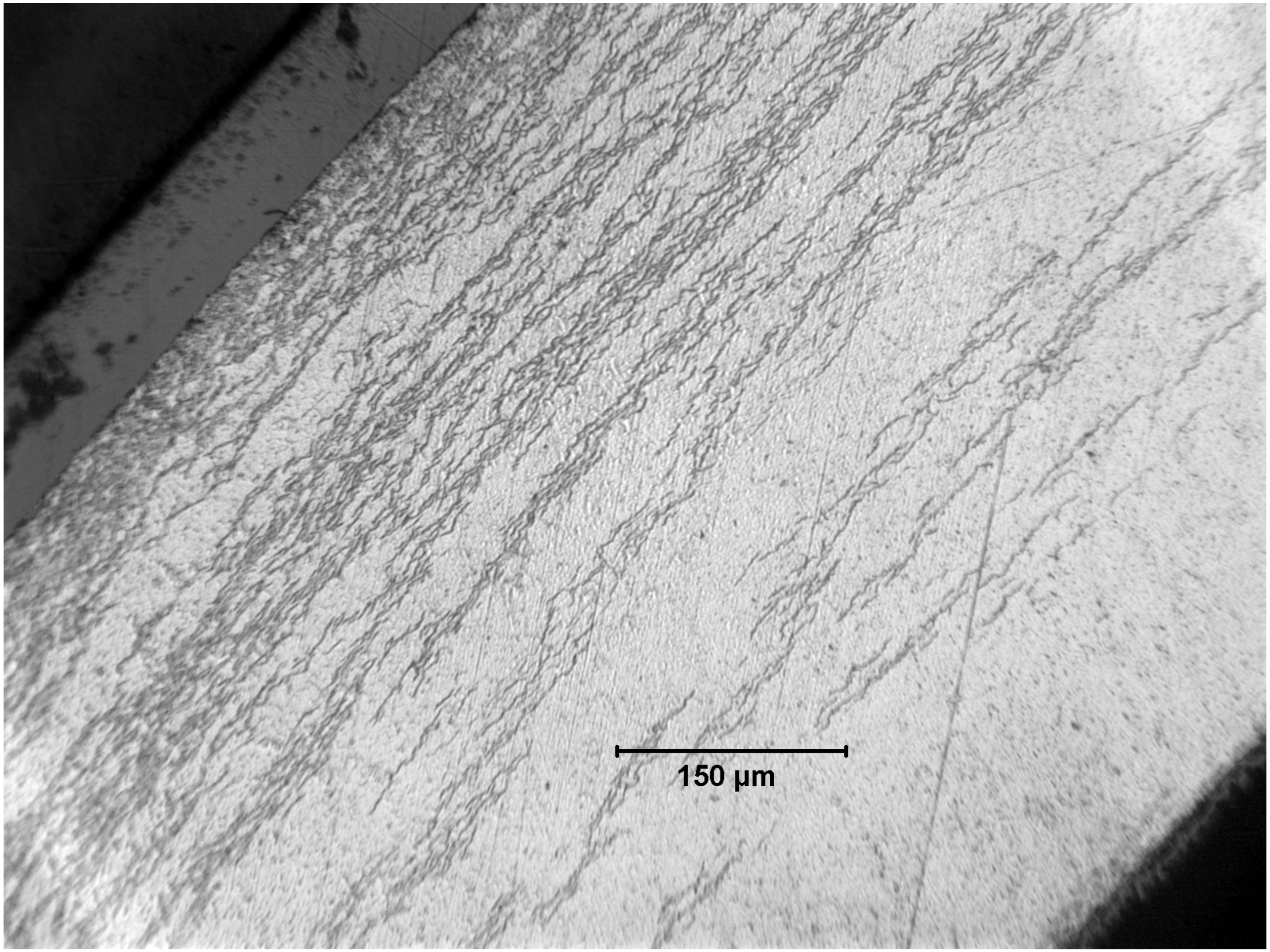




Hydride Morphology for HBR Rod F07 Cladding at ≈50 mm above Fuel Midplane (High Mag.)



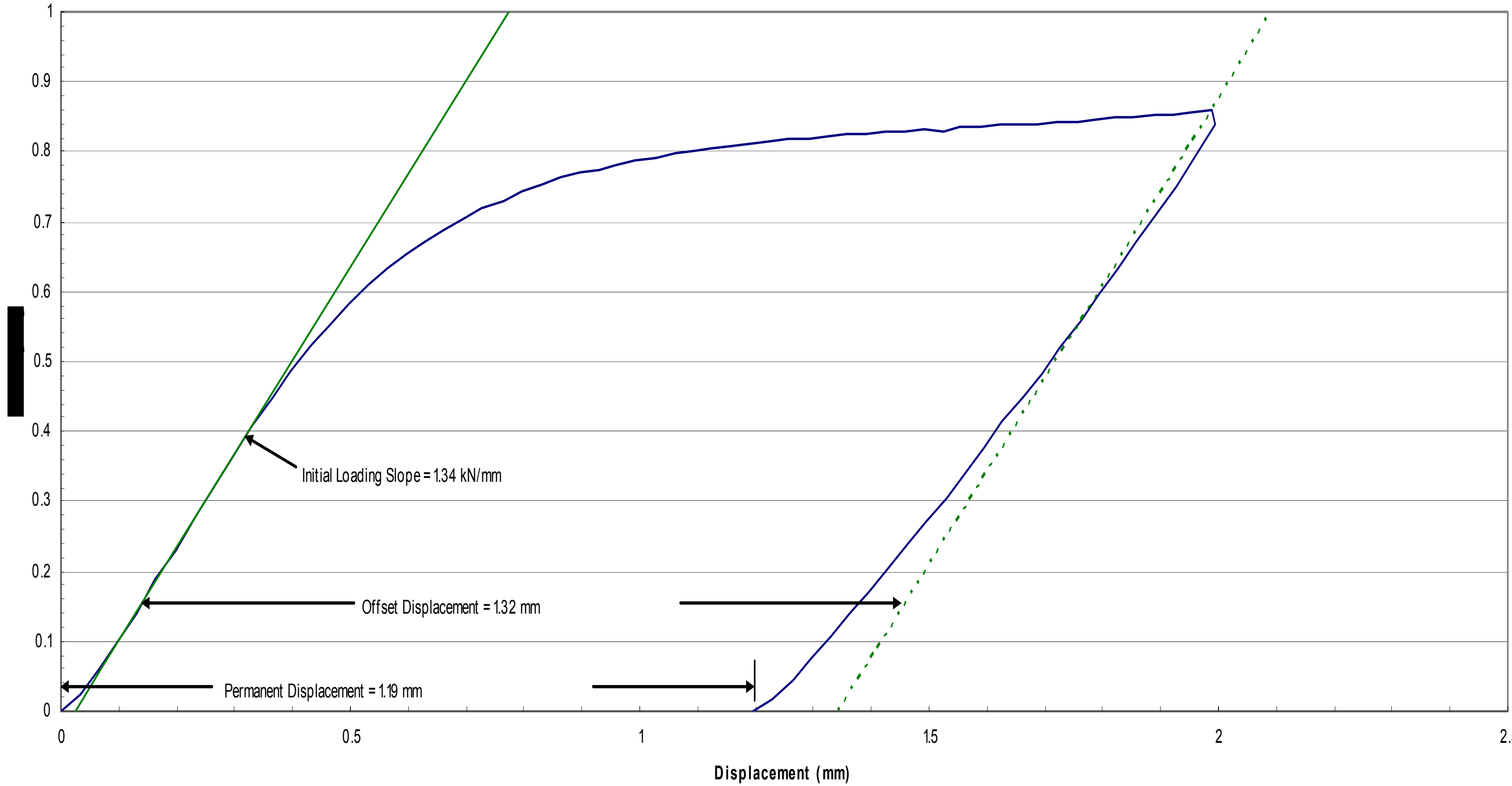
Hydride Morphology for HBR Rod F07 Cladding at ≈50 mm above Fuel Midplane (High Mag.)



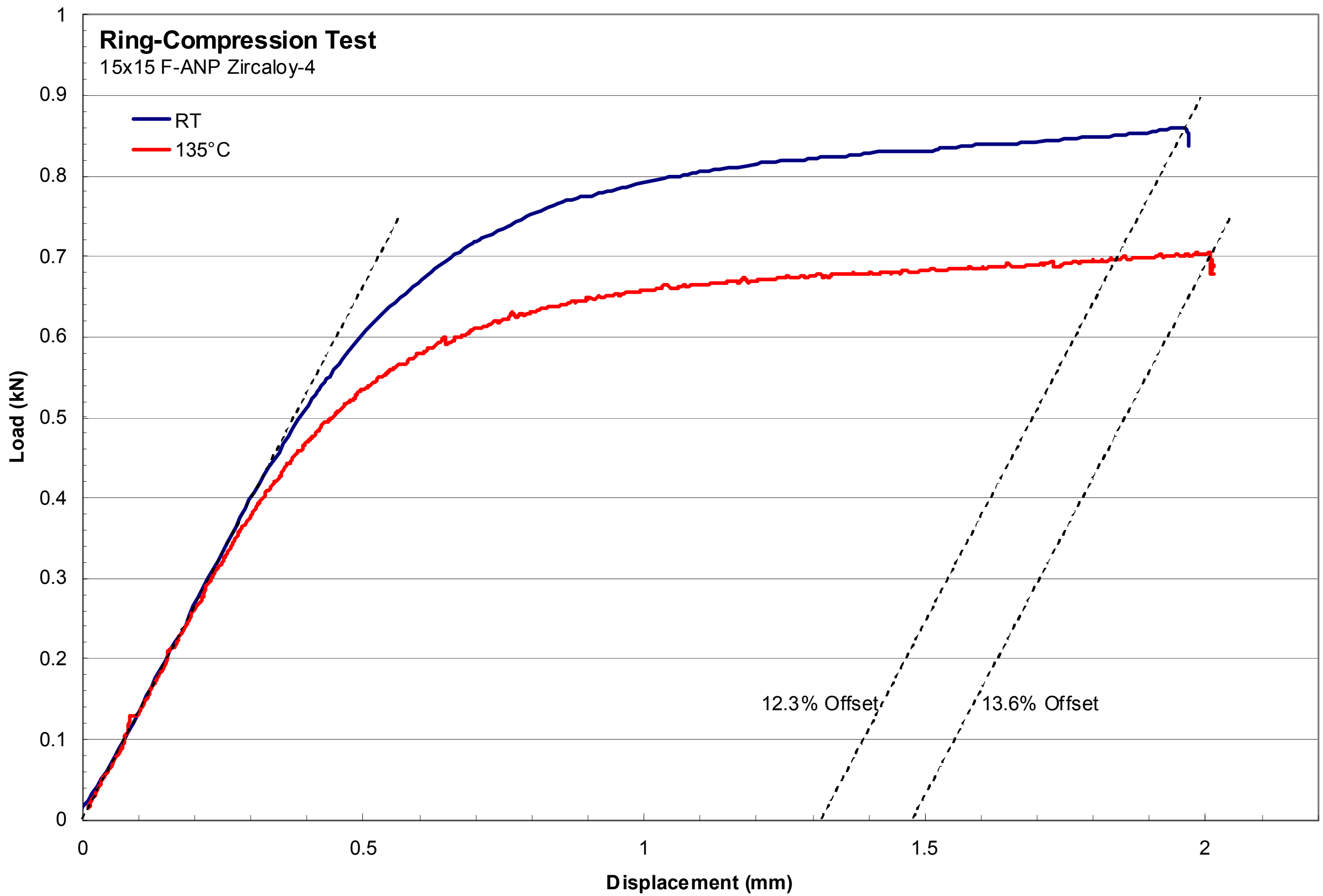
Ring-Compression Ductility of Non-Irradiated, Prehydrided HBR Baseline Cladding (15×15 Zry-4)

- **RT Ring Compression: Non-Irradiated 15×15 Zry-4**
  - 8-mm-long sample displaced to 2 mm and unloading at RT
    Loading stiffness (1.34 kN/mm) consistent with elastic model
    Unloading stiffness < loading stiffness
    Offset displacement is 0.13 mm (1.2%) > permanent displacement
  - 8-mm-long sample displaced to 2 mm and unloading at RT at 135ºC
    Larger decrease in yield strength than in stiffness (Young's modulus)
- **RT Ring Compression: Prehydrided Zry-4**
  - 8-mm-long rings compressed to failure (full-length through-wall crack)
    Circumferential hydrides (≈300 wppm): high ductility = 45%
  - Radial-Hydride-Treated (RHT) samples cooled from 400ºC under σ_θ
    RHT (400ºC→RT at 150 MPa, ≈750 wppm): low ductility = 3.1%
    RHT (400ºC→RT at 190 MPa, ≈300 wppm): brittle = 0% ductility

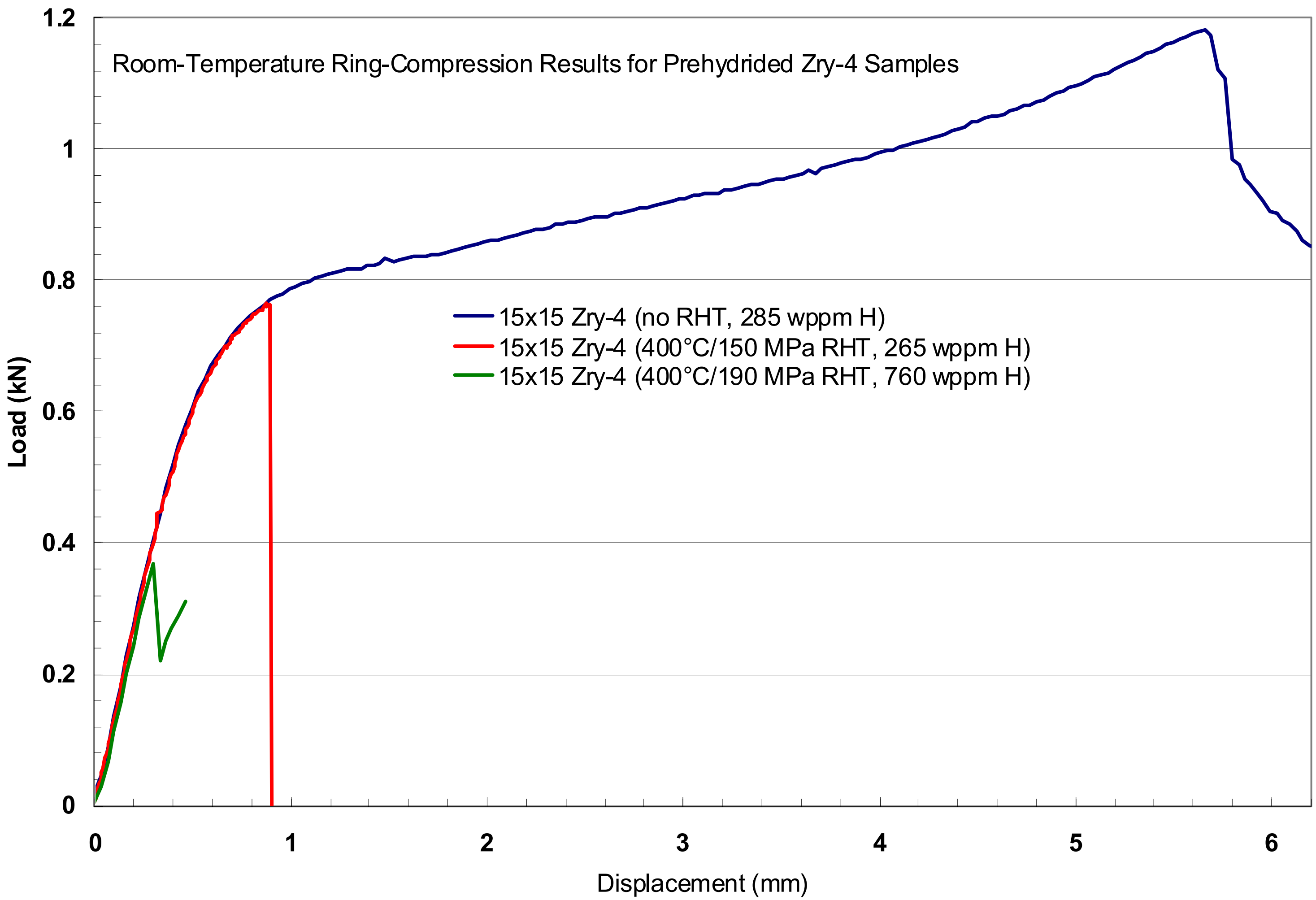
Benchmark RT Ring-Compression Test Results for Non-Irradiated, HBR Baseline 15×15 Zry-4



Comparison of RT and 135ºC Ring-Compression Test Results for Non-Irradiated, HBR Baseline 15×15 Zry-4



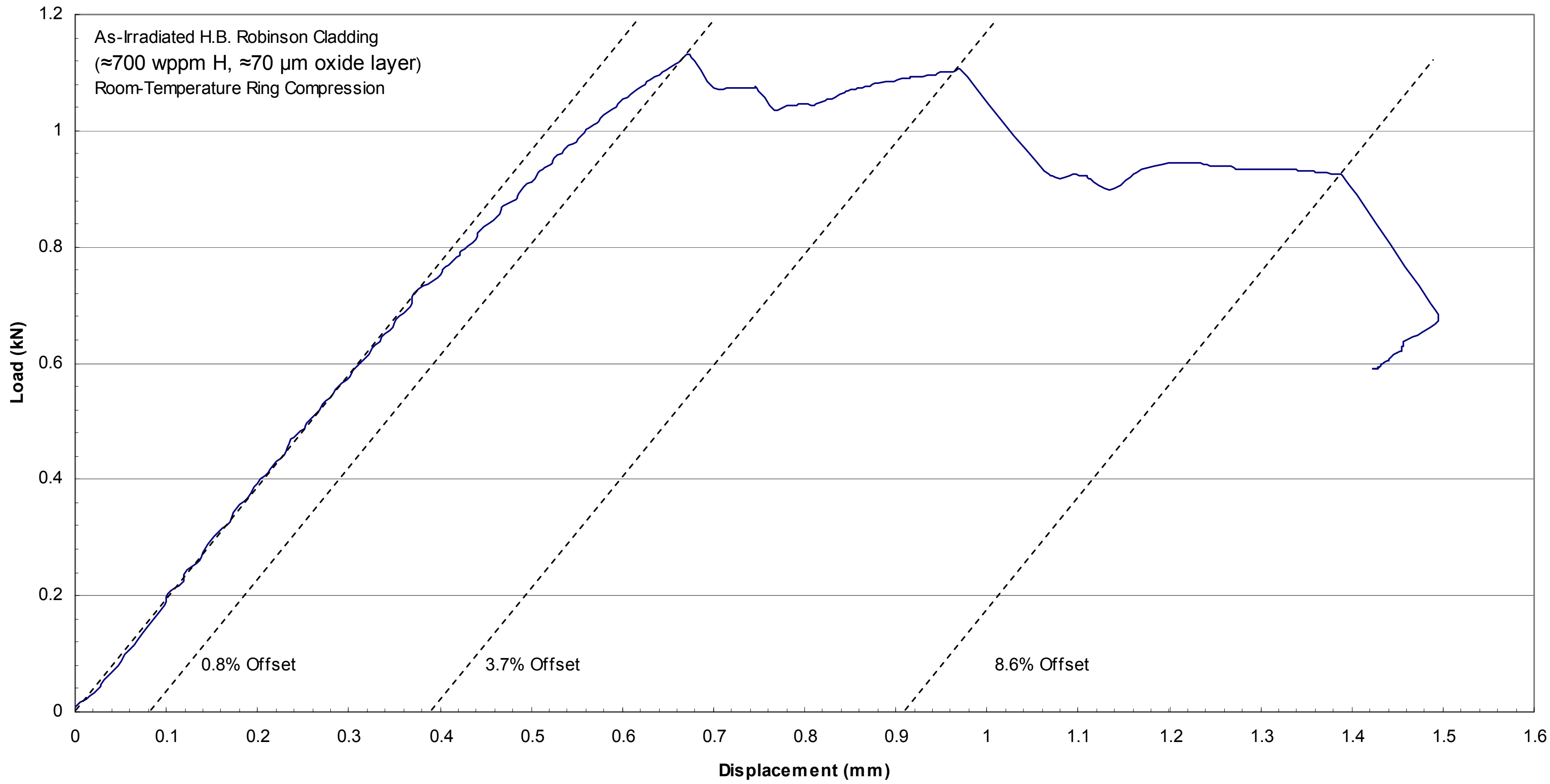
RT Ring-Compression Test Results for Prehydrided, HBR Baseline 15×15 Zry-4



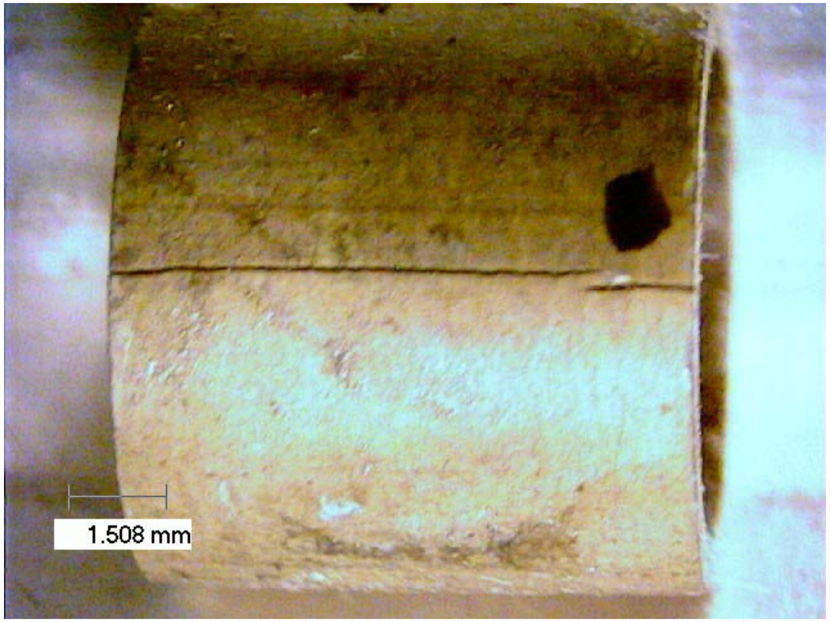
Ring-Compression Ductility of As-irradiated HBR Cladding (15×15 Zry-4)

- **RT Load-Displacement Test Results**
  - 9.4-mm-long ring with ≈70 µm corrosion layer and ≈700 wppm H
    Near midplane of Rod F07 (see specimen 7E in Slide 15)
  - Loading stiffness (1.92 kN/mm) consistent with increased modulus (E)
  - Evidence of small load drops due to oxide cracking from 0.8-3.7% offset
  - 1^{st} large load drop (≈0.2 kN) at 3.7% offset → partial wall crack
  - 2^{nd} large load drop (≈0.2 kN) at ≈8.3% offset → crack extension
    Test stopped at 8.6% offset and 6.3% permanent strains
    Sample crack at 90º from loading line, extending ≈75% from OD to ID
  - Test results for cladding with corrosion layer removed
    Smooth load-displacement curve with load drop at 10.6% offset strain
    2 partial-wall cracks at +90º and -90º extending over full length of ring
- **135ºC Load-Displacement Test Results (more ductile)**

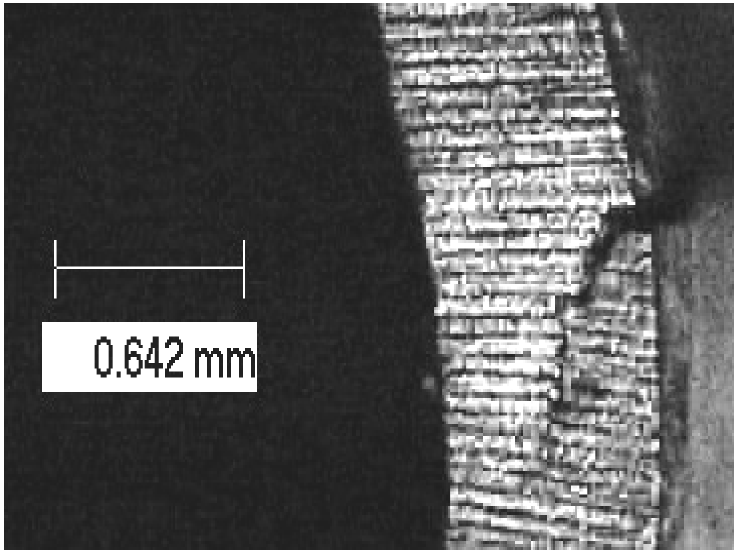
RT Ring-Compression Test Results for High-Burnup HBR Cladding Near Fuel Midplane



Low-Magnification Images of Crack in HBR Sample

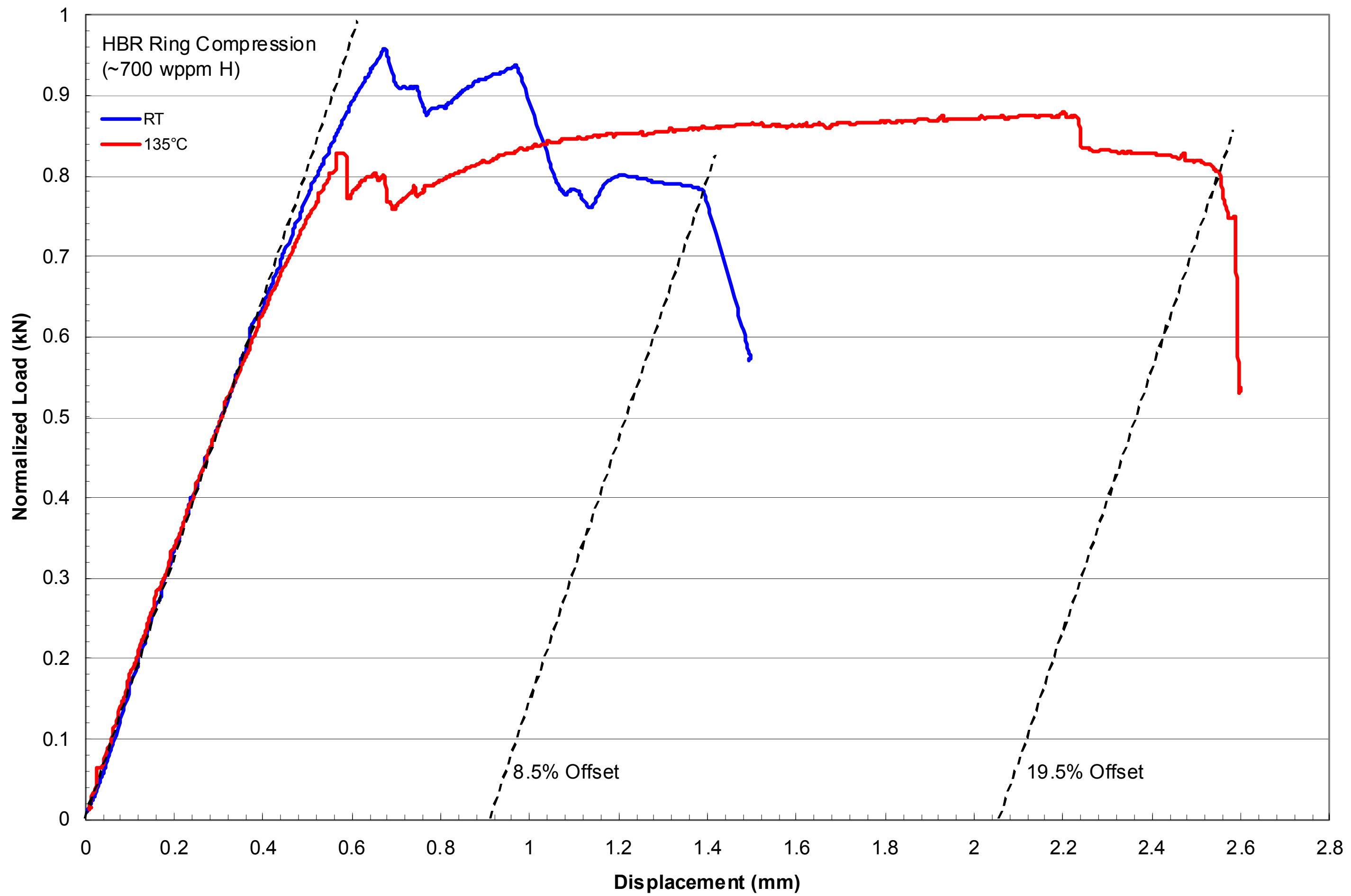


1X View of Crack at 90º from Loading Line

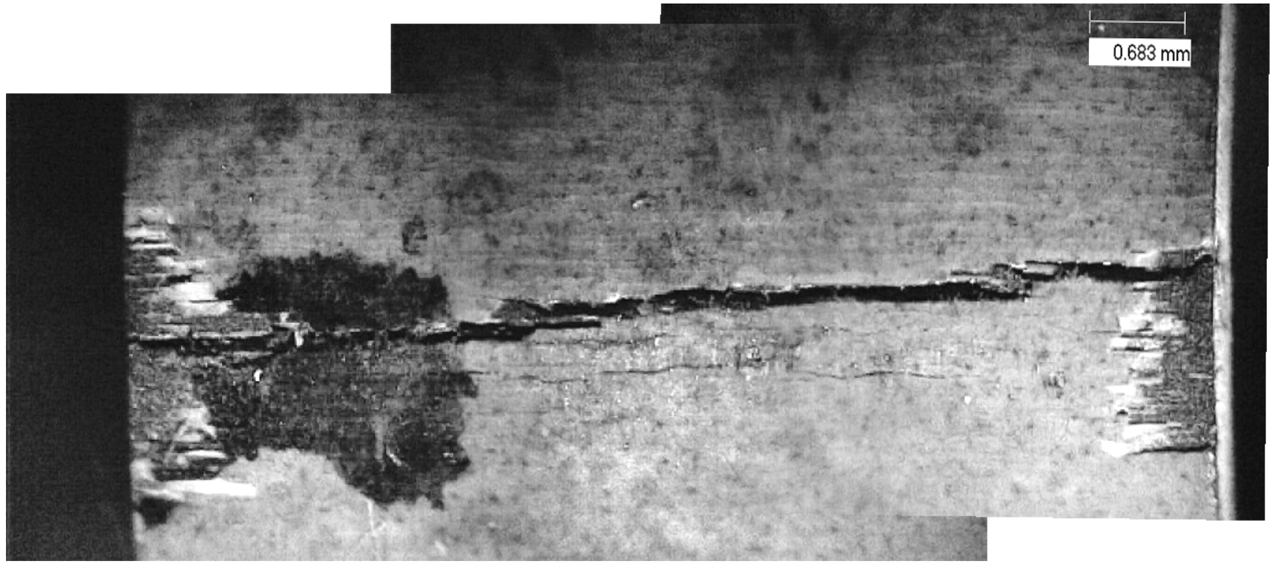


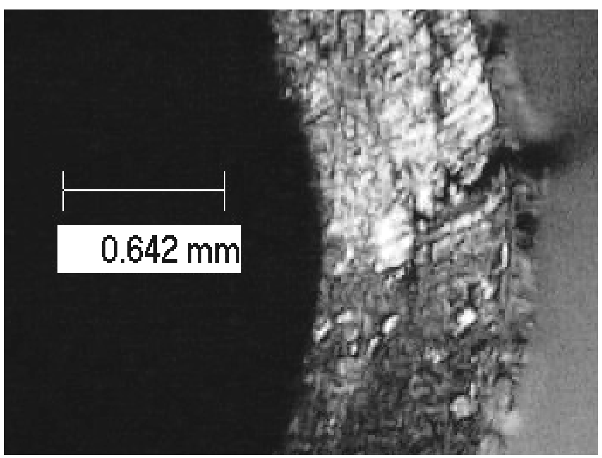
3X View of Crack along Side of Ring

135ºC Ring-Compression Test Results for High-Burnup HBR Cladding Near Fuel Midplane

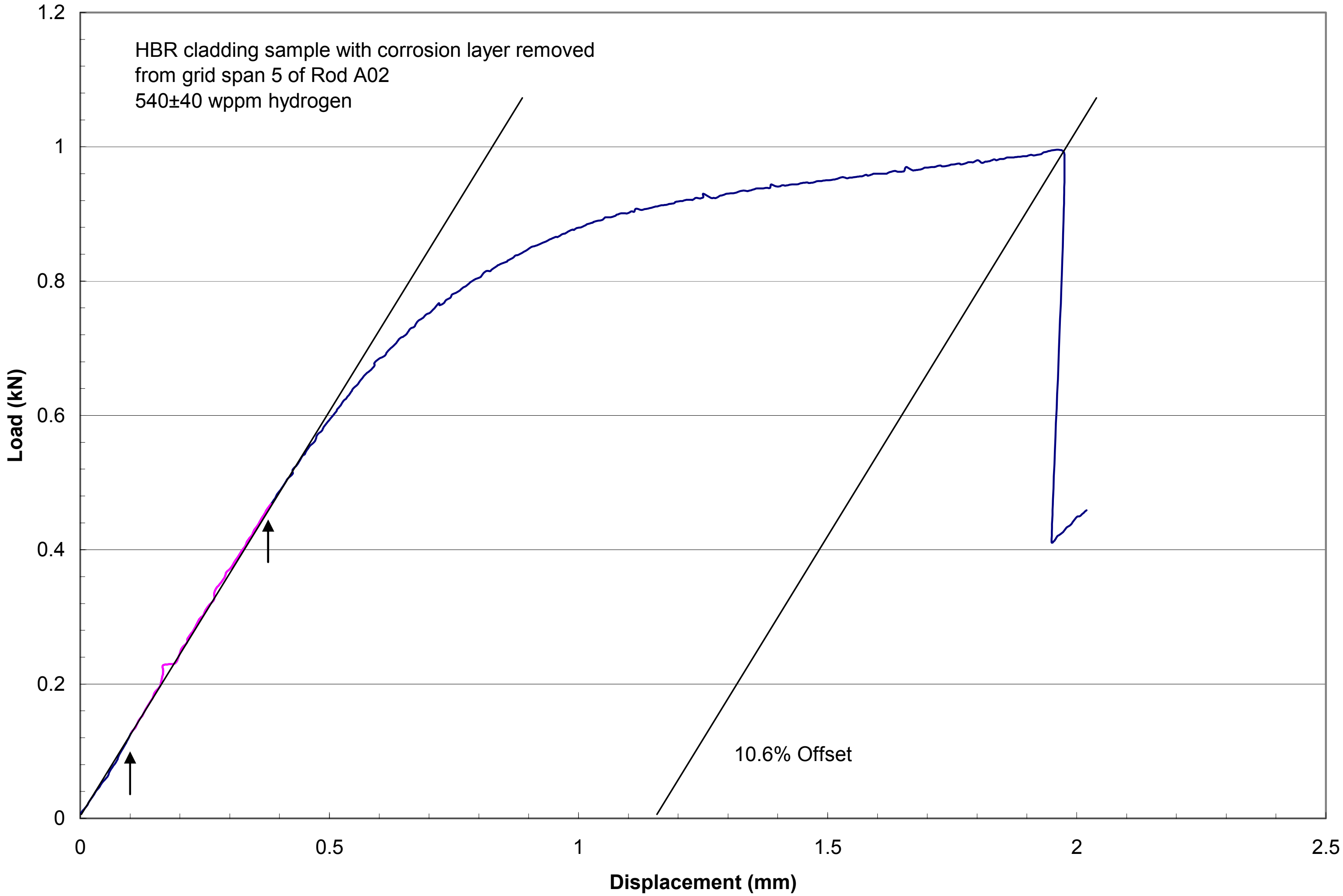


Low Mag. Images of Crack in HBR Ring Tested at 135ºC

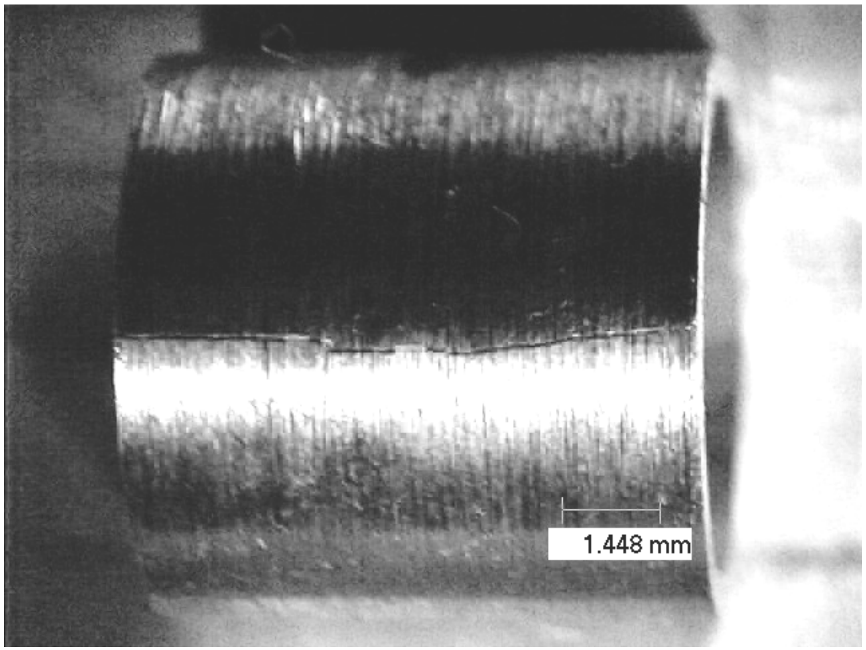




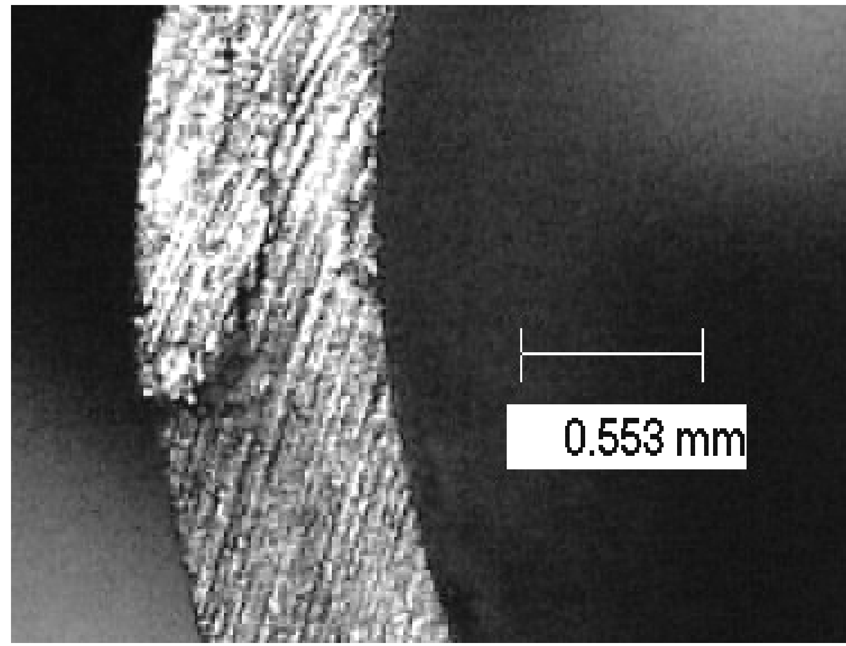
RT Ring-Compression Test Results for High-Burnup HBR Cladding with Corrosion Layer Removed



Low-Magnification Images of Crack in HBR Ring with the Corrosion Layer Removed Prior to RT Testing



1X Image at 90º
from Loading Line



3X Image of Crack Growth
through Side Wall

Discussion of Characterization Results for HBR Rod F07

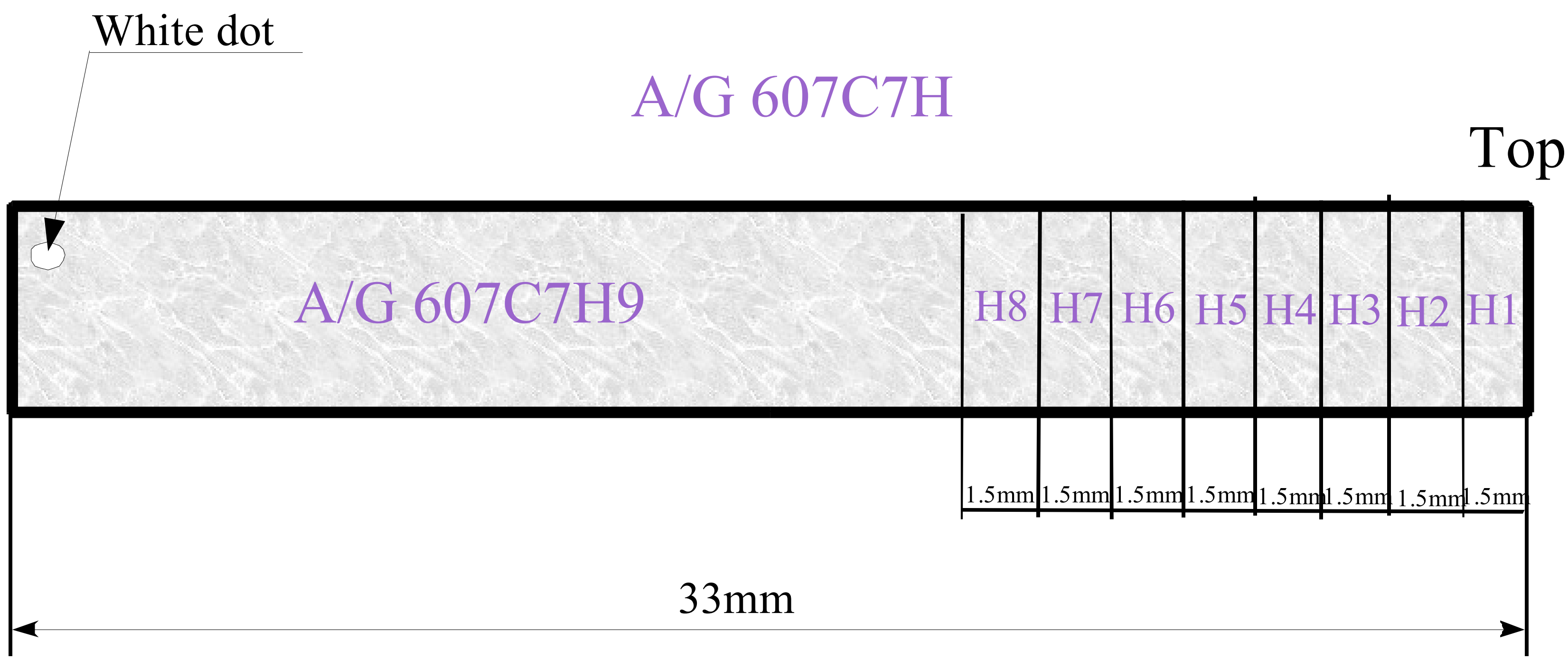
- **Oxide Layer & Oxygen Content near Fuel Midplane**
  - Oxide layer thickness: F07 (71±5 μm) consistent with A02 (70±5 μm)
  - Oxygen content of as-irradiated cladding

    Predicted assuming PB ratio of 1.75: 2.09 wt.%

    Measured at +35 mm: 2.08±0.19 wt.% (good agreement)

    Measured at +10 mm: 1.99±0.40 wt.% (some oxide lost during cutting)
- **Hydrogen Content near Fuel Midplane**
  - Expected = 550-580 wppm based on A02 and R01
  - Measured: 760±60 wppm at +13 mm; 670±80 wppm at +32 mm
  - **Re-measured over 14 mm (7H) just below midplane: 550±90 wppm**
- **Hydride Morphology near Fuel Midplane**
  - Appears symmetric with respect to the fuel centerline axis
  - Large H variations with respect to θ should be visible in metallography

Re-Measurement of Hydrogen Content

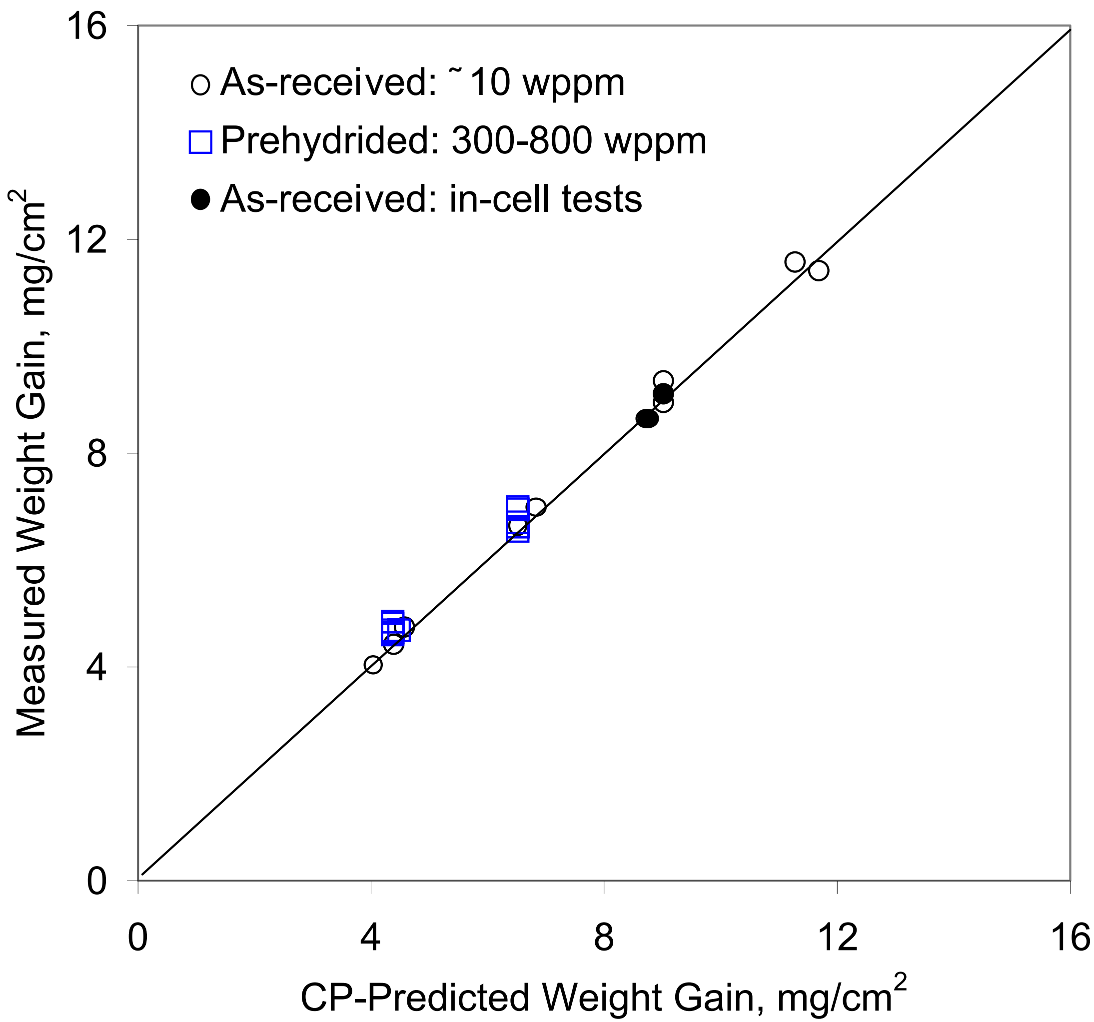


H1: 545±85 wppm H; H2: 530±80 wppm H
H3: 530±90 wppm H; H4: 535±85 wppm H
H5: 555±85 wppm H; H6: 575±80 wppm H
H7: 535±80 wppm H; H8: 565±70 wppm H

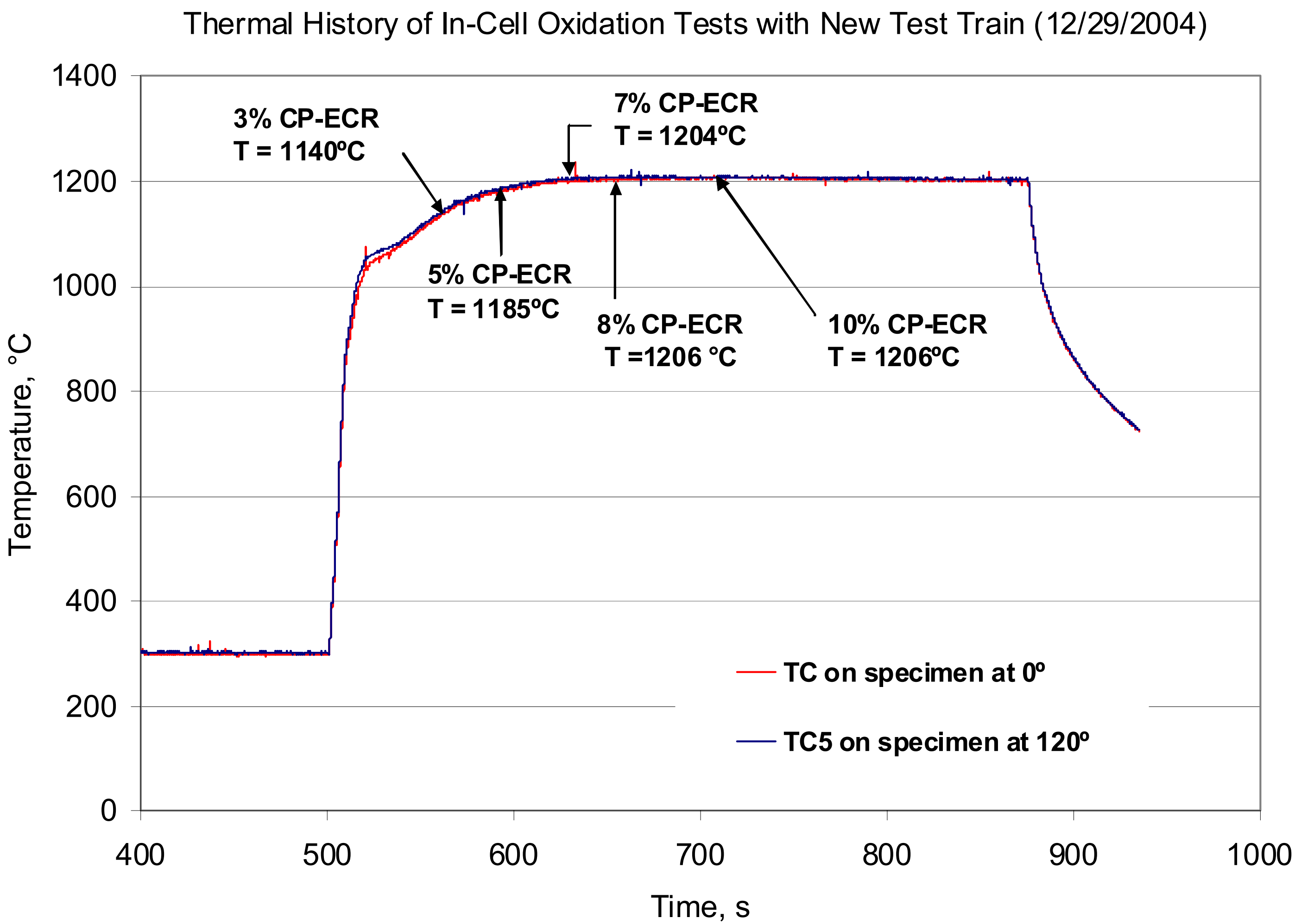
Steam Oxidation Tests of F07 Cladding Samples

- **0604 Test Train Benchmarked In-Cell (Sample Weight Gain)**
  - Nonirradiated 15×15 Zry-4 at 1204ºC to 10% ECR
    Excellent agreement with CP-model and out-of-cell results
  - Thermocouple wires damaged during handling before HBR cladding sample could be run (December 2004)
- **New Test Train (1204) Benchmarks with As-Fabricated Zry-4**
  - Out-of-cell thermal benchmark for sample T vs. control T on holder
  - Out-of-cell weight-gain benchmark at 5% and 10% ECR
  - In-cell weight-gain benchmark at 10% ECR
  - Out-of-cell and in-cell results in excellent agreement with CP model
- **In-cell Oxidation Tests Conducted with HBR F07 Cladding**
  - CP-ECR = 3%, 5%, 7%, and 8% and 10%
  - Metallography to determine steam-oxide, alpha, and beta layers; weight gain; and effects of corrosion layer on weight-gain kinetics

Weight-Gain Results for Nonirradiated 15×15 Zry-4 (0604 and 1204 Test Trains)



New Test Train: Thermal Benchmark Results for Nonirradiated 15×15 Zry-4



Post-Oxidation Ductility of HBR Cladding at 135ºC: 3% CP-ECR Sample

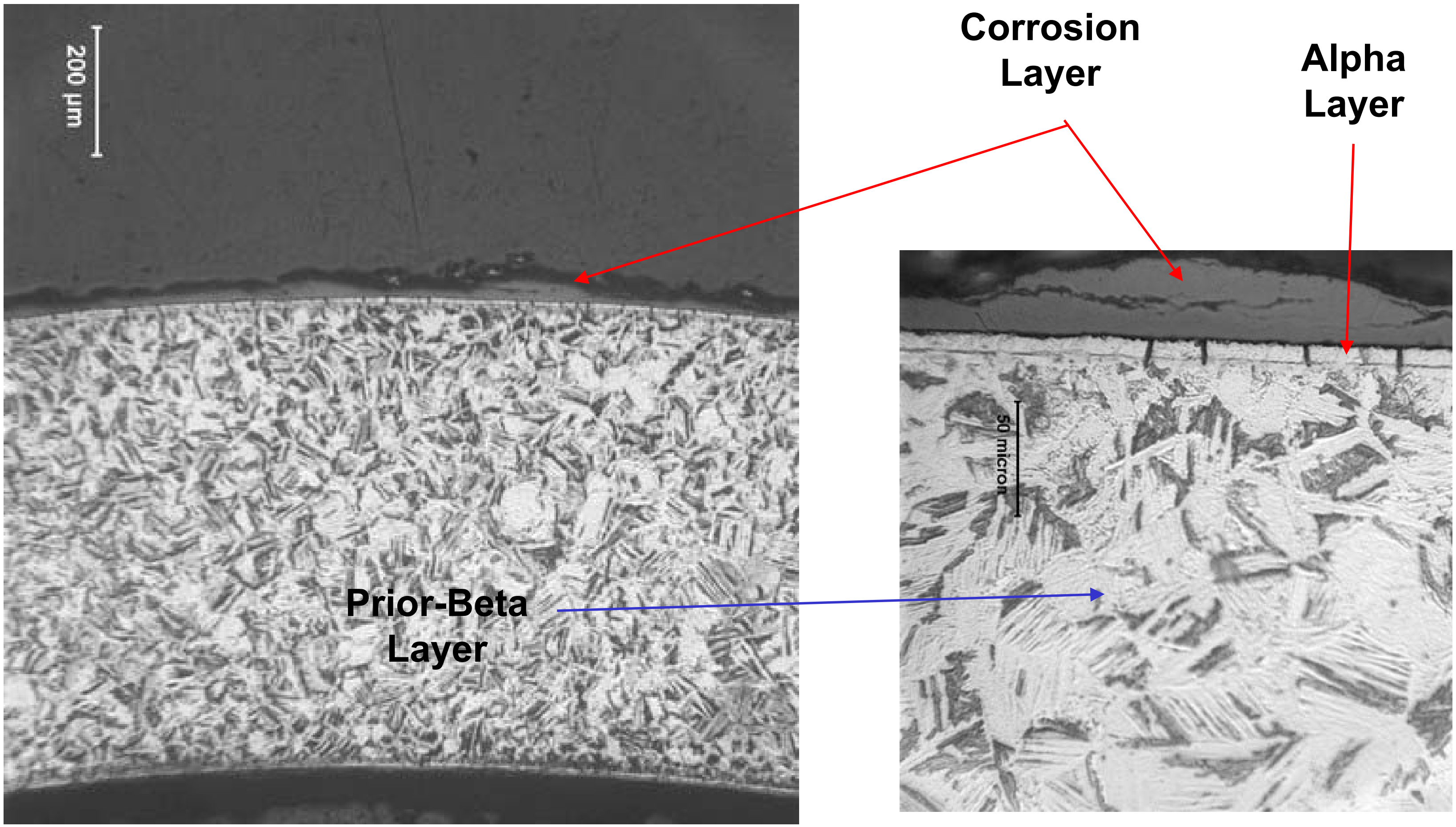
- **3% CP-ECR with T = 1140ºC Prior to Cooling**
  - Post-test hydrogen: 670±210 wppm (2-mm rings sides of sample)
  - Metallography

Outer-surface:	most of corrosion layer is missing;
	no evidence of steam-oxide layer
	O-diffusion: corrosion layer → O-stabilized alpha
	alpha layer ≈ CP model thickness
Inner-surface:	partial fuel-cladding bond layer
	non-uniform steam-oxide layer??
	O-stabilized alpha layer < CP model
Summary:	effects of corrosion layer → ECR_t < CP-ECR

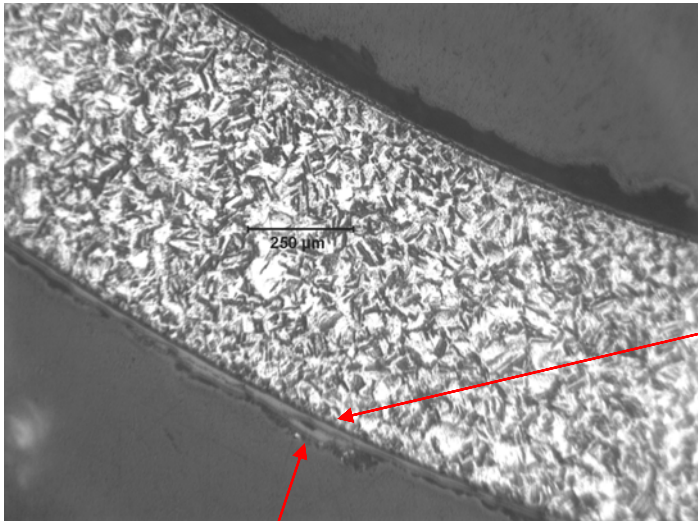
  - Ring-compression ductility > 45% (test stopped before failure)
  - **Assessment: very high ductility; comparable to as-fabricated Zry-4**
  - **No intrinsic hydrogen embrittlement observed**

Metallography of 3% CP-ECR Sample – Outer Surface

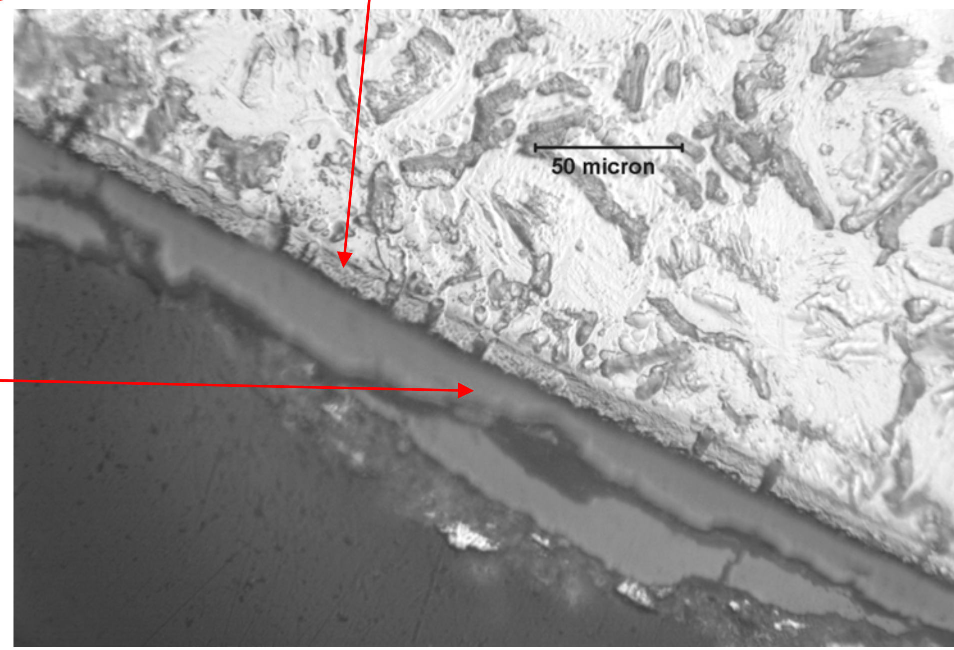
Steam-Oxidation Layer???



Metallography of 3% CP-ECR Sample – Outer Surface (After Grinding off ≈2mm, Polishing and Etching)



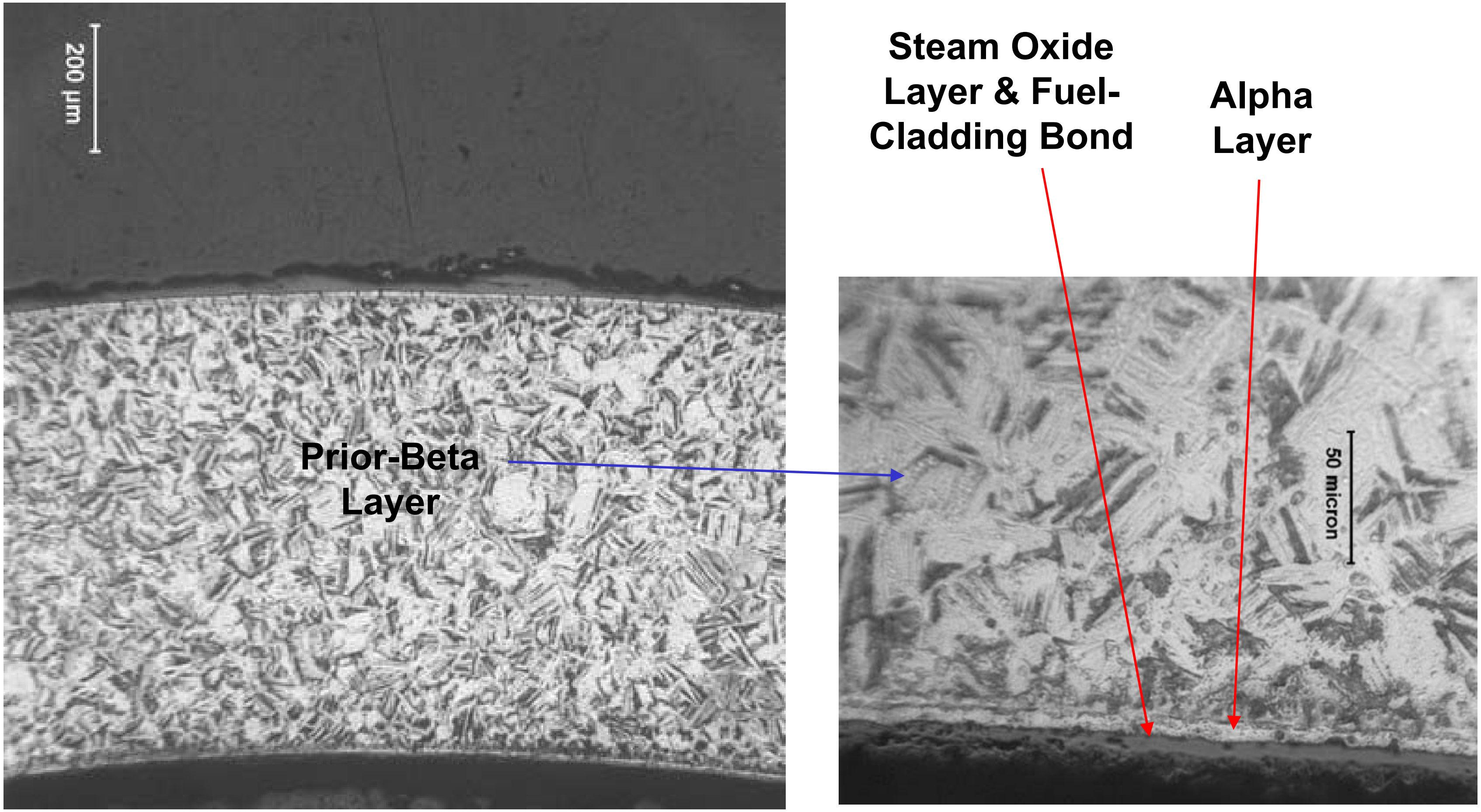
Oxygen-Stabilized Alpha Layer



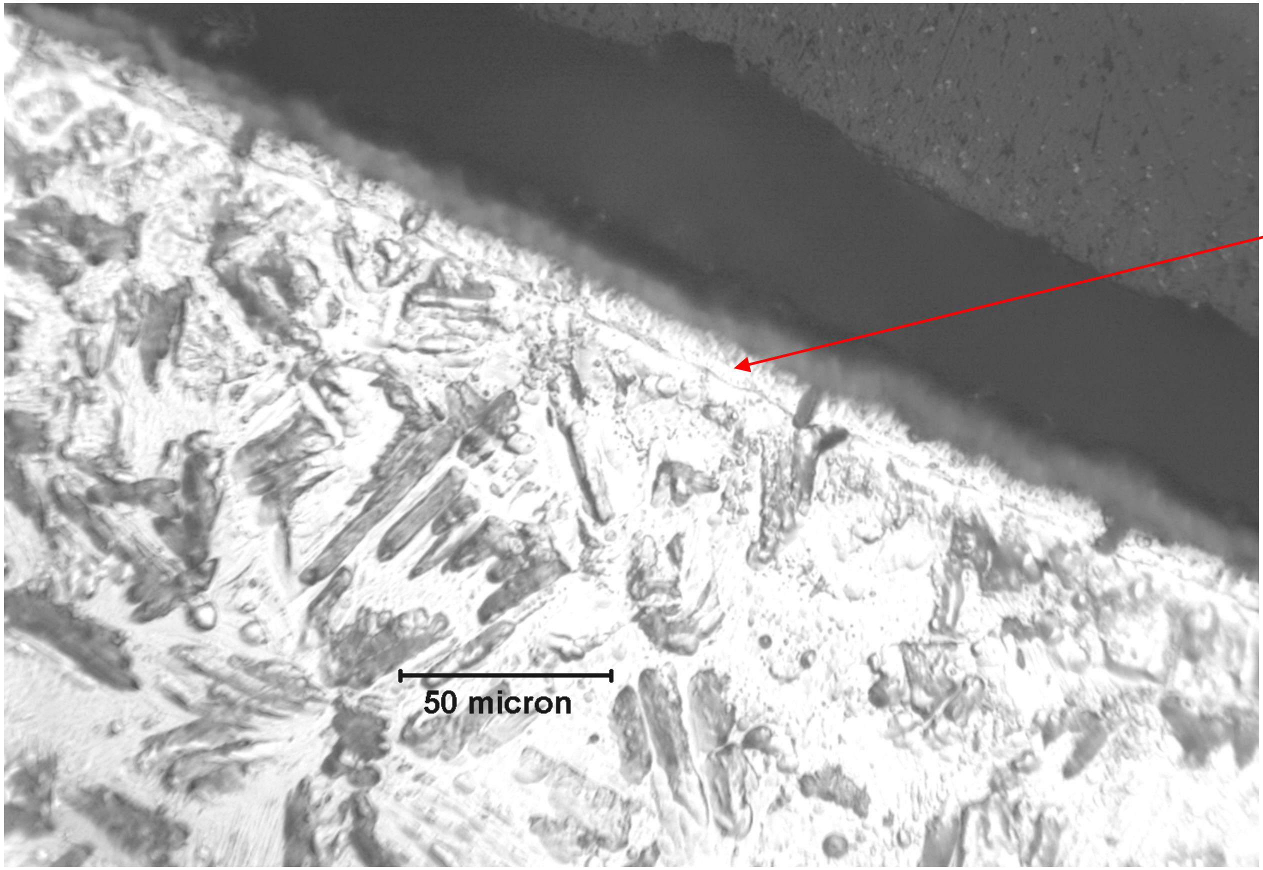
Corrosion Layer

No Steam Oxide Layer

Metallography of 3% CP-ECR Sample – Inner Surface

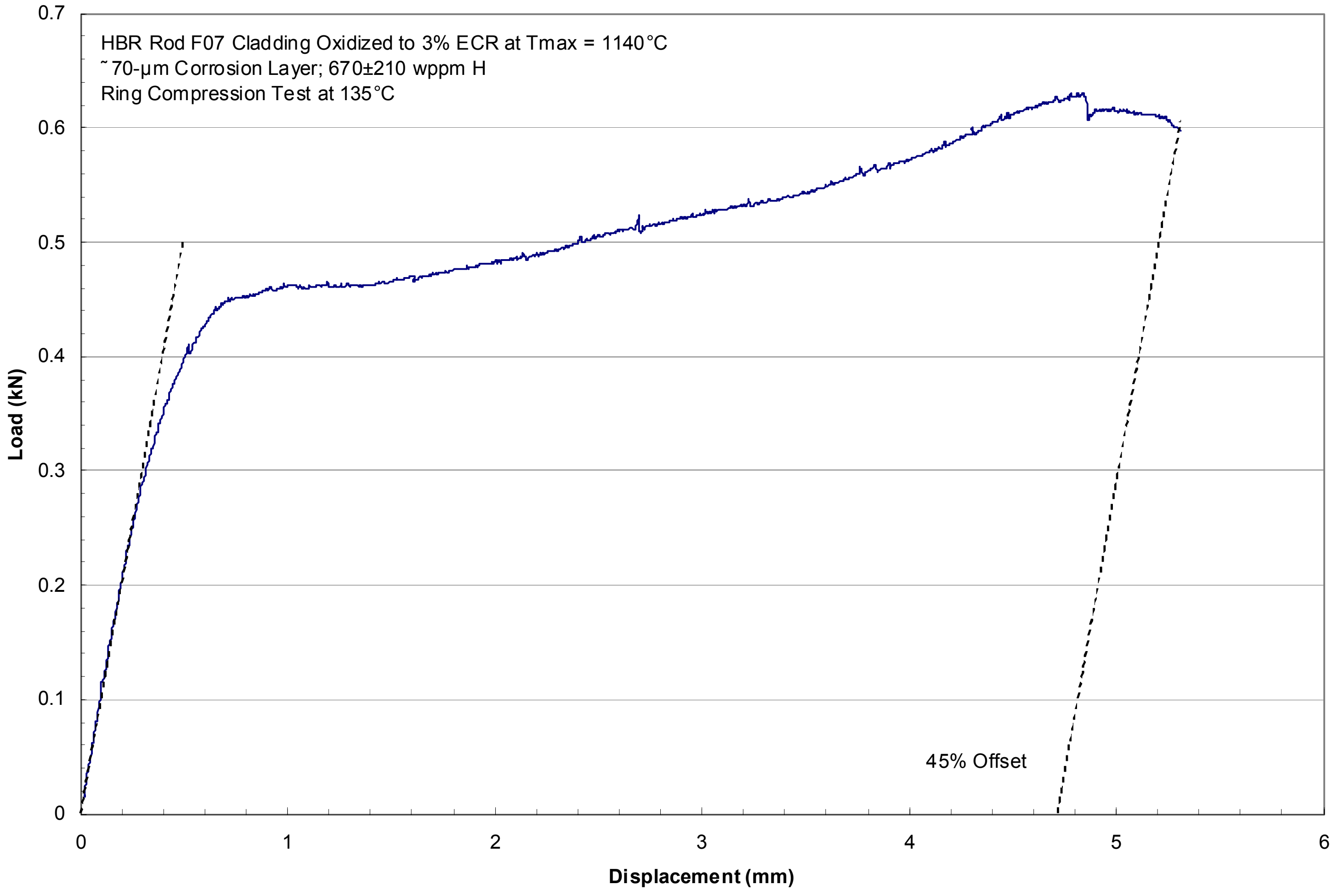


Metallography of 3% CP-ECR Sample – Inner Surface (After Grinding off ≈2mm, Polishing and Etching)



Alpha Layer

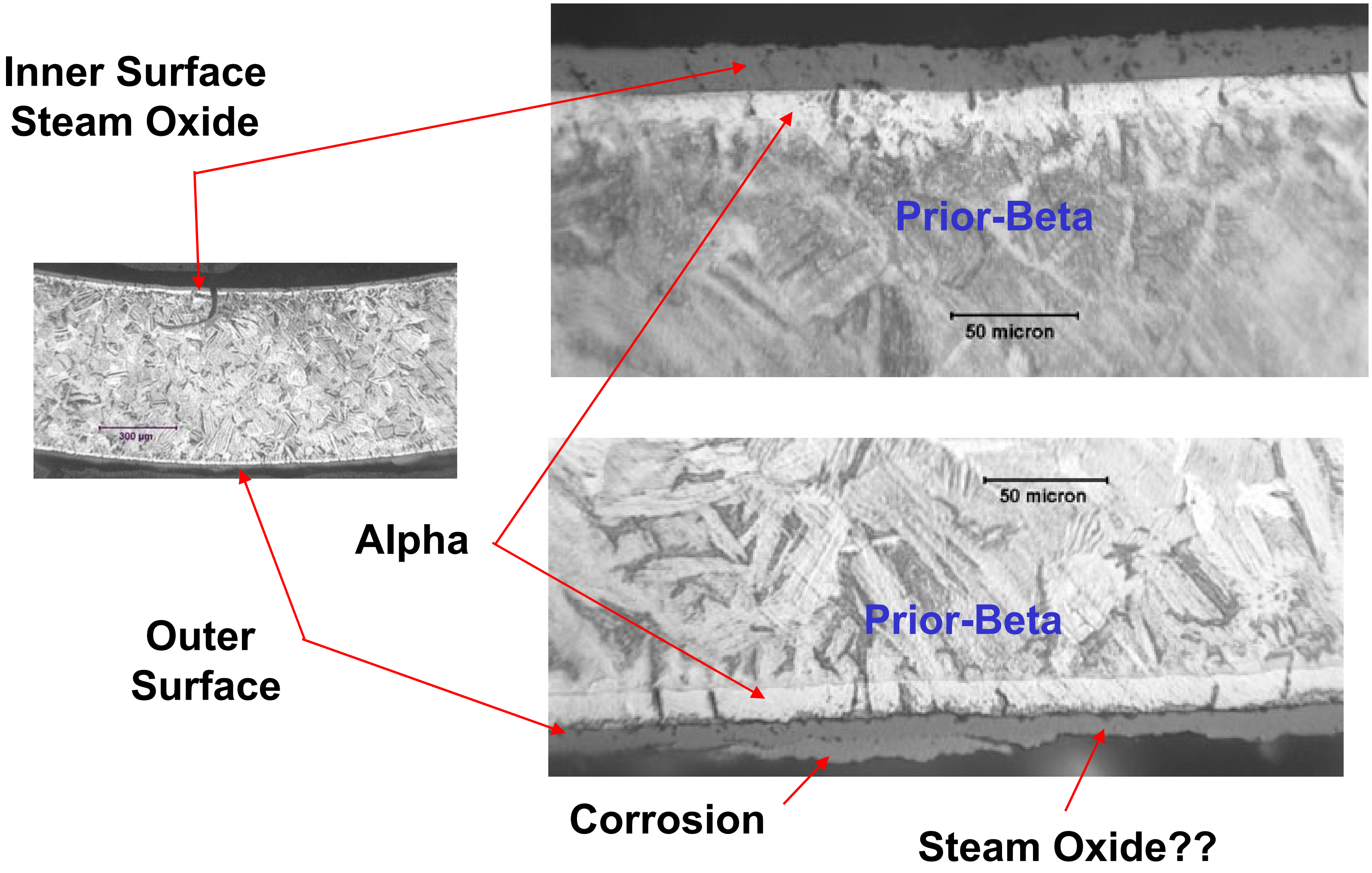
Load-Displacement Curve (at 135ºC) for 3% CP-ECR HBR Zry-4 Ring with 670±210 wppm H (Post-Oxidation)



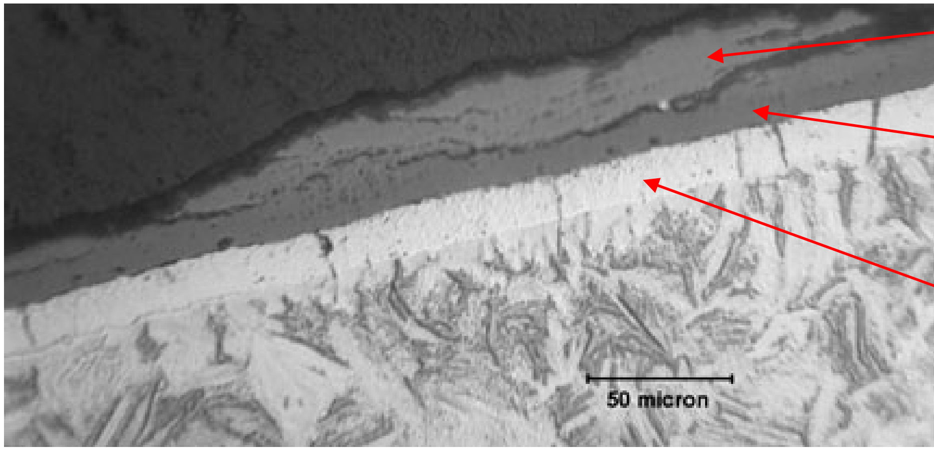
Post-Oxidation Ductility of HBR Cladding at 135ºC: 5% CP-ECR Sample

- **5% CP-ECR with T = 1185ºC Prior to Cooling**
  - Post-test hydrogen: 600±90 wppm (2-mm rings sides of sample)
    Metallography
    Outer-surface: partial corrosion layer, uneven steam-oxide layer
    growth of uniform O-stabilized alpha layer
    alpha layer < CP model, but rate may be similar
    Inner-surface: growth of steam-oxide layer; some fuel-clad. bond
    growth of alpha layer (≈ same as outer surface)
    Summary: effects of corrosion layer → ECR_t < CP-ECR
  - Ring #1: test stopped after 40% load drop at 12% offset strain
    no through-wall crack seen at 3X mag.; need metallography
  - Ring #2: test run to max. displacement; no sharp load drops;
    through-wall crack at bottom; ductility is ≈40%
  - **Assessment: medium-to-high ductility**

Metallography of 5% CP-ECR Sample



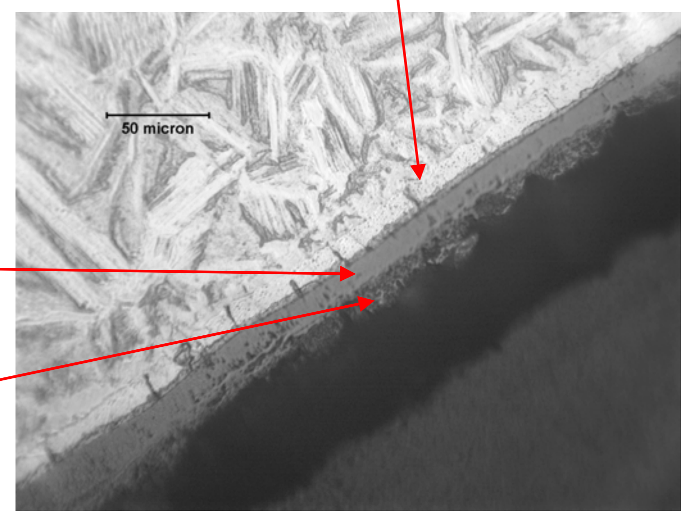
Metallography of 5% CP-ECR Sample (Cont'd)



Corrosion

Steam Oxide

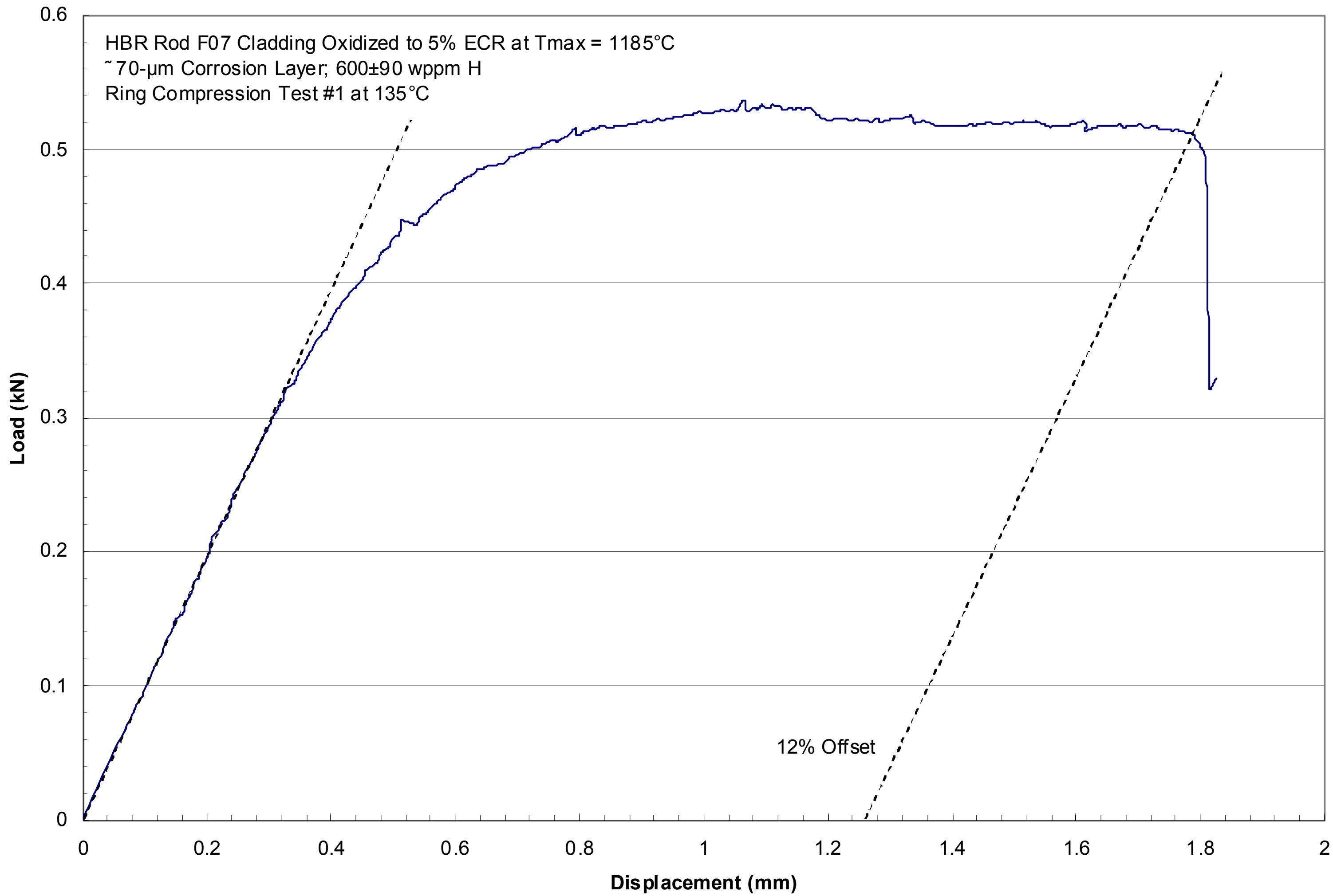
Alpha



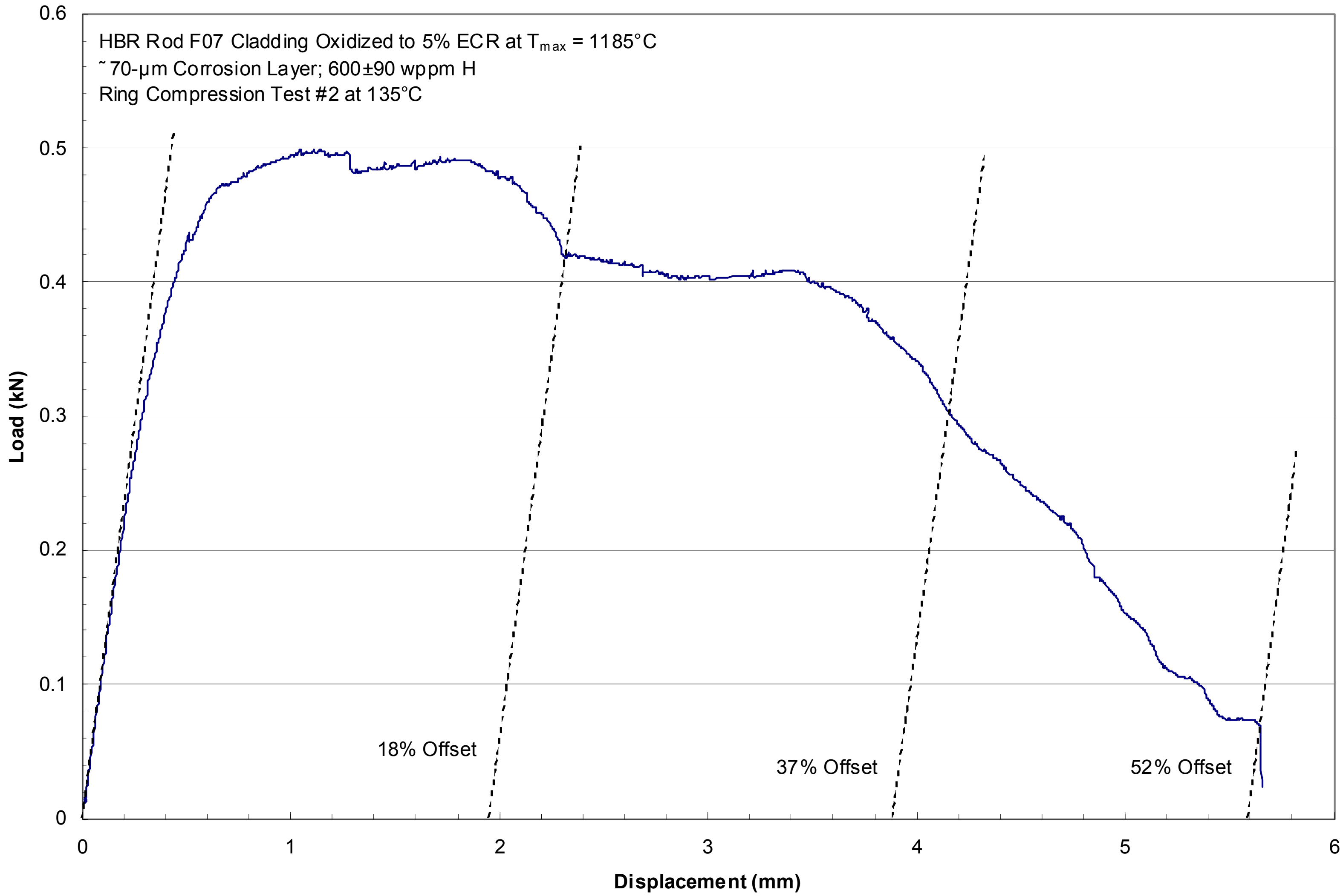
Steam Oxide

Fuel-Cladding Bond

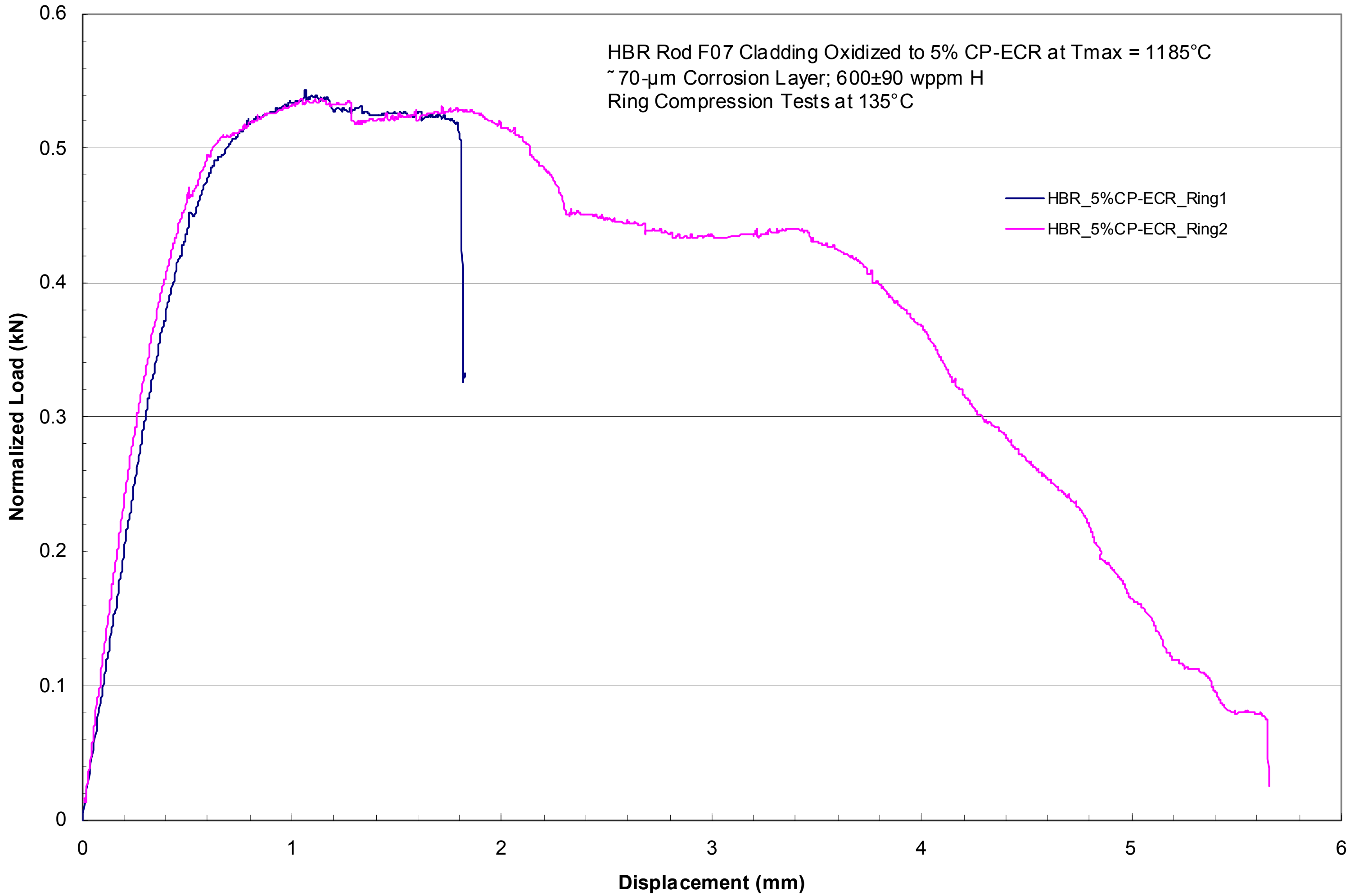
Load-Displacement Curve #1 (at 135ºC) for 5% CP-ECR HBR Zry-4 Ring with 600±90 wppm H (Post-Oxidation)



Load-Displacement Curve #2 (at 135ºC) for 5% CP-ECR HBR Zry-4 Ring with 600±90 wppm H (Post-Oxidation)



Load-Displacement Curves (at 135ºC) for 5% CP-ECR HBR Zry-4 Ring with 600±90 wppm H (Post-Oxidation)



Post-Oxidation Ductility of HBR Cladding at 135ºC: 7% CP-ECR Sample

- **7% CP-ECR with T = 1204ºC Prior to Cooling**
  - Note: sample was heated to ≈900ºC, control TC failed, cooled to 300ºC, reheated to 1204ºC based on working TC
  - Post-test hydrogen: 780±70 wppm (2-mm rings sides of sample)
    too high; probably not all in metal cladding
  - Metallography

    Outer-surface: partial corrosion layer, growth of steam-oxide layer
    growth of uniform O-stabilized alpha layer

    Inner-surface: growth of steam-oxide layer; some fuel-clad. bond
    growth of O-stabilized alpha layer

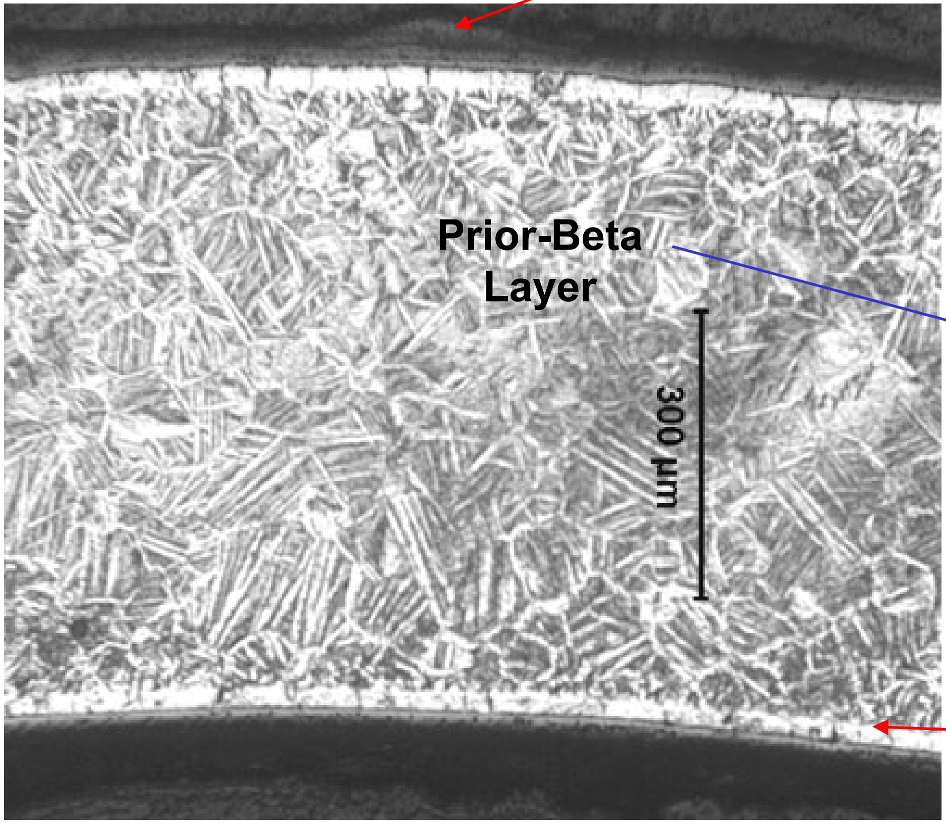
    Summary: effects of corrosion layer → ECR_t < CP-ECR
  - Ring-compression ductility = 3.8% offset strain, 2.6% permanent strain
  - **Assessment: low ductility**

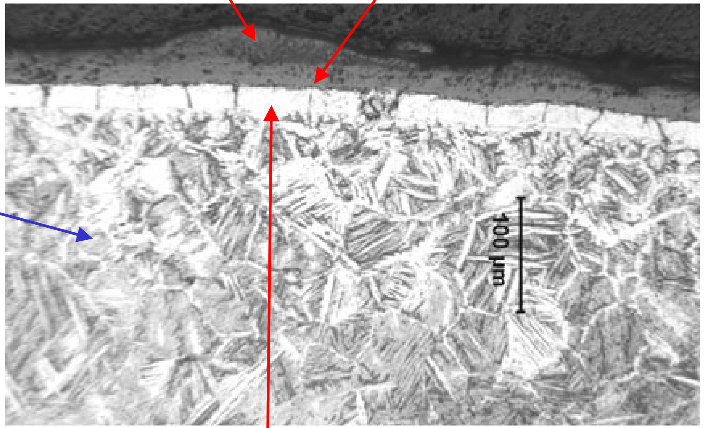
Metallography of 7% CP-ECR Sample – Outer Surface

Corrosion Layer

Steam Oxide Layer

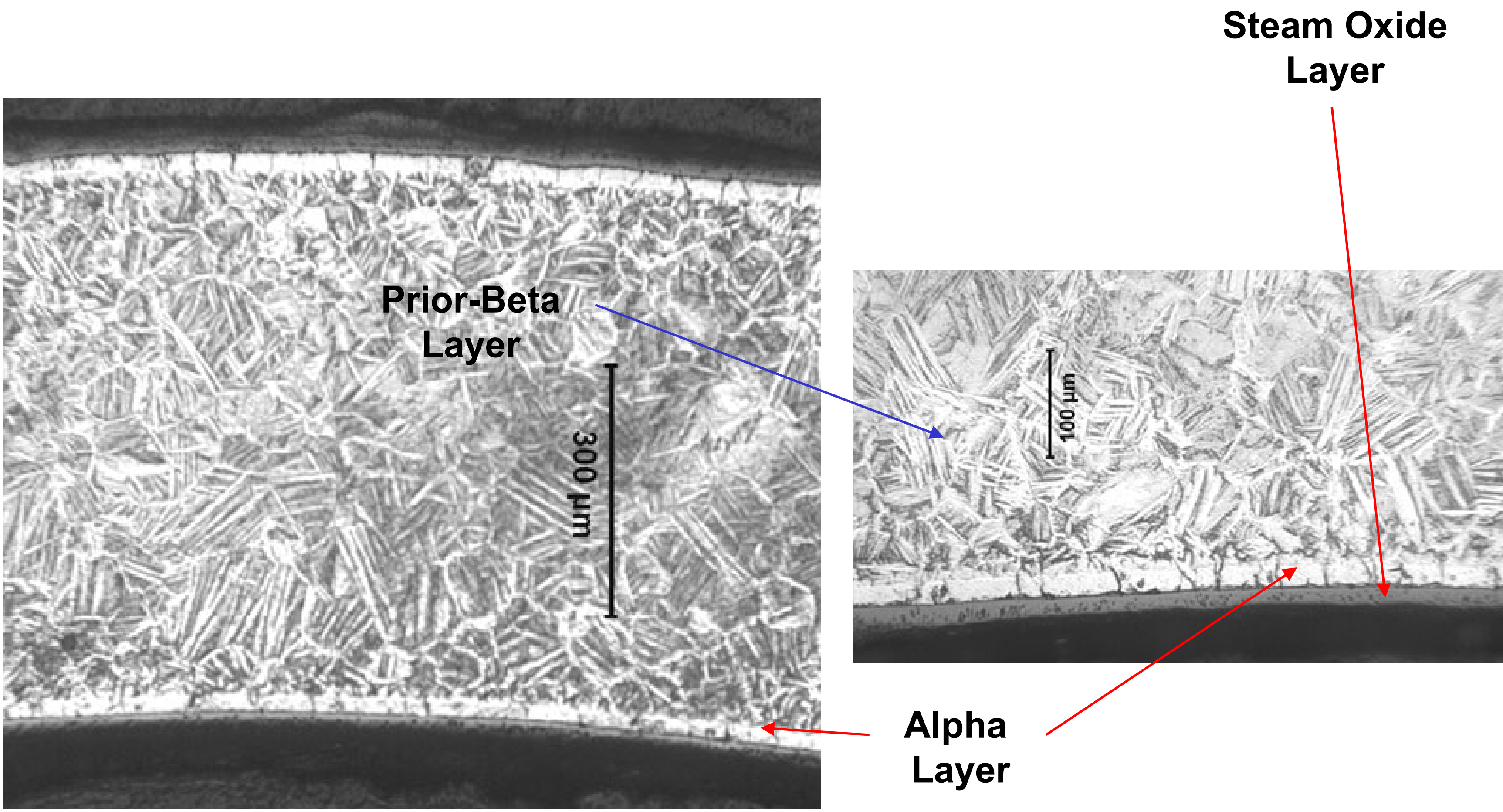
Prior-Beta Layer



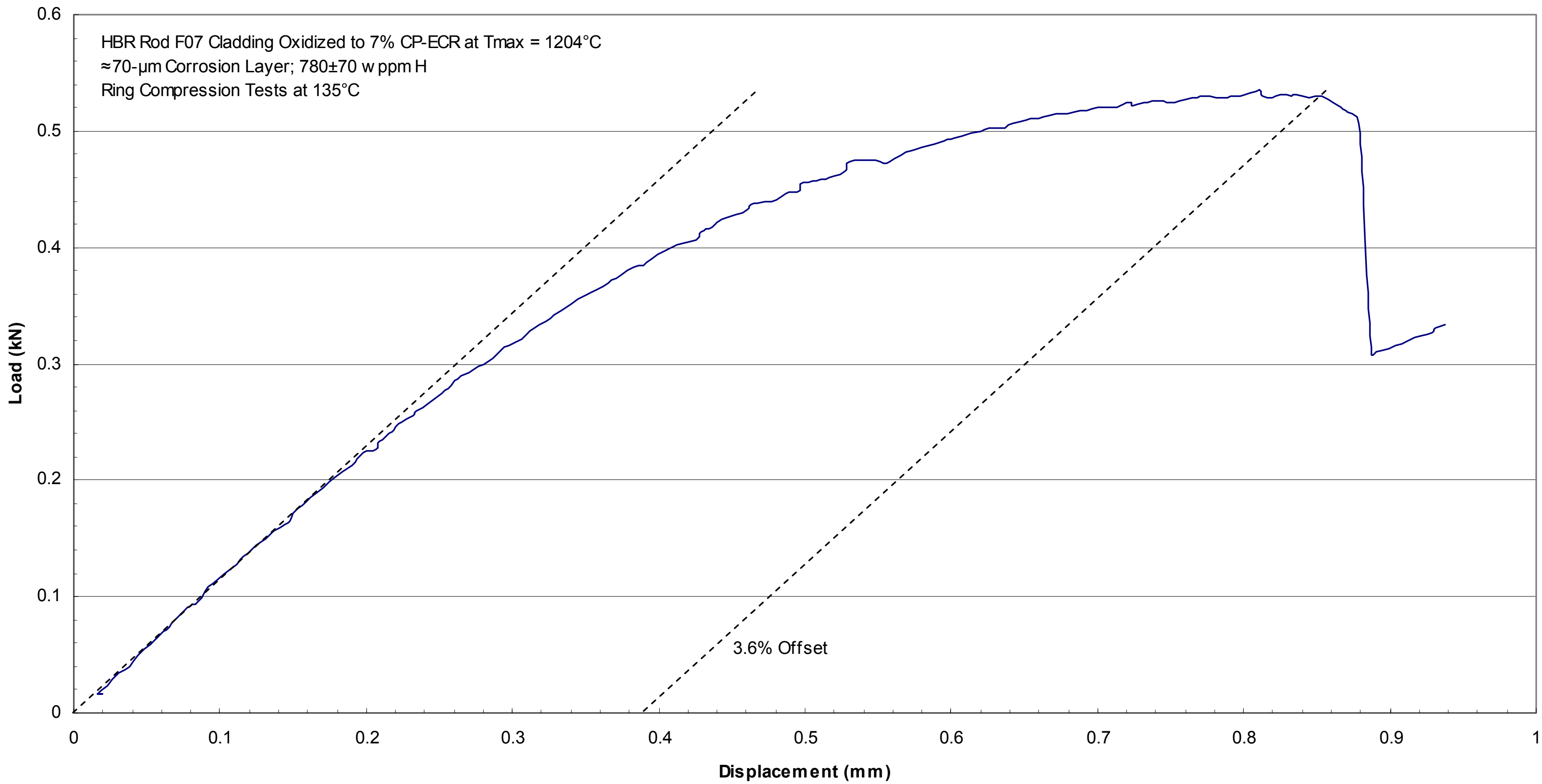


Alpha Layer

Metallography of 7% CP-ECR Sample – Inner Surface



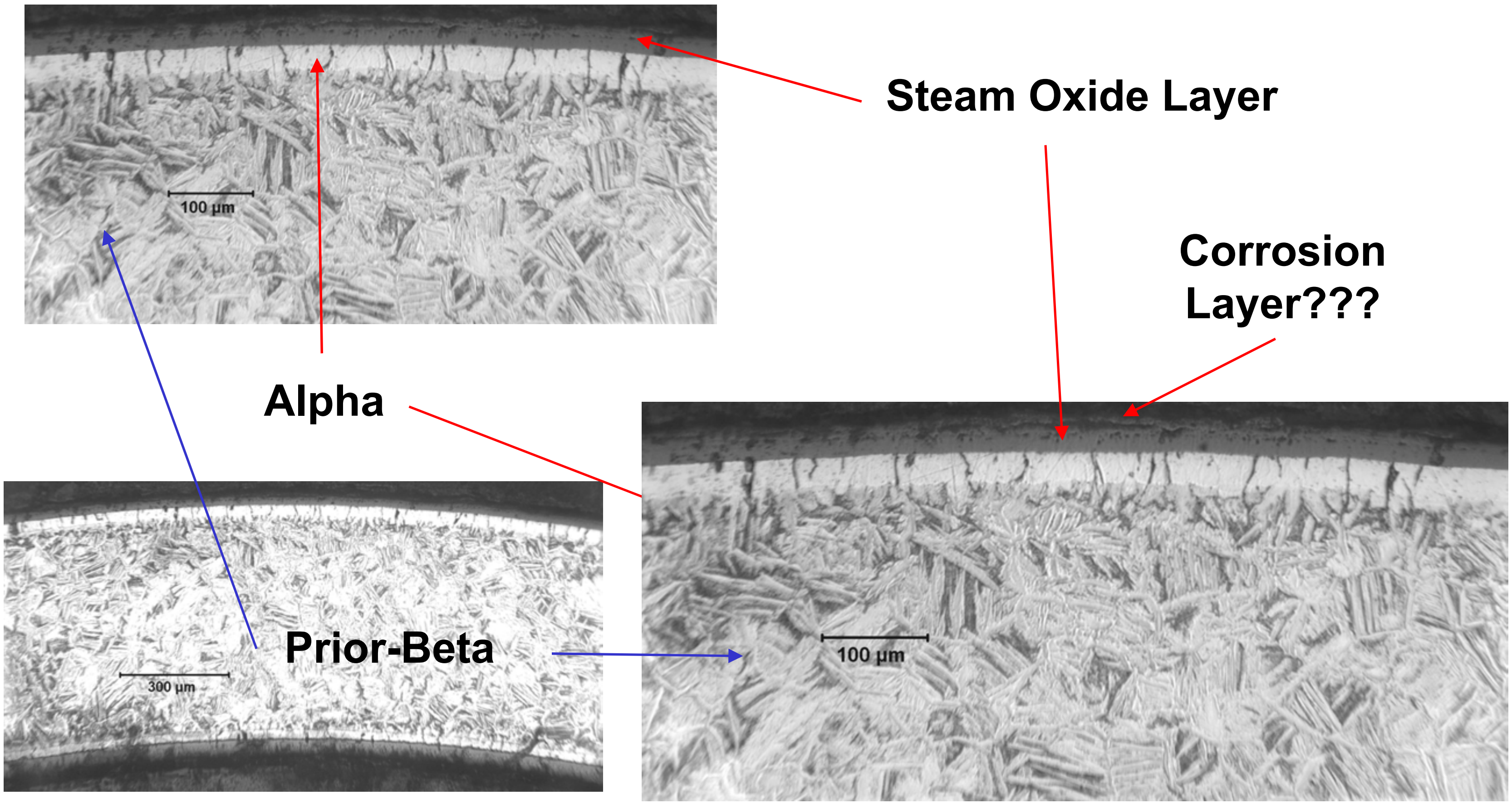
Load-Displacement Curve (at 135ºC) for 7% CP-ECR HBR Zry-4 Ring with 780±70 wppm H (Post-Oxidation)



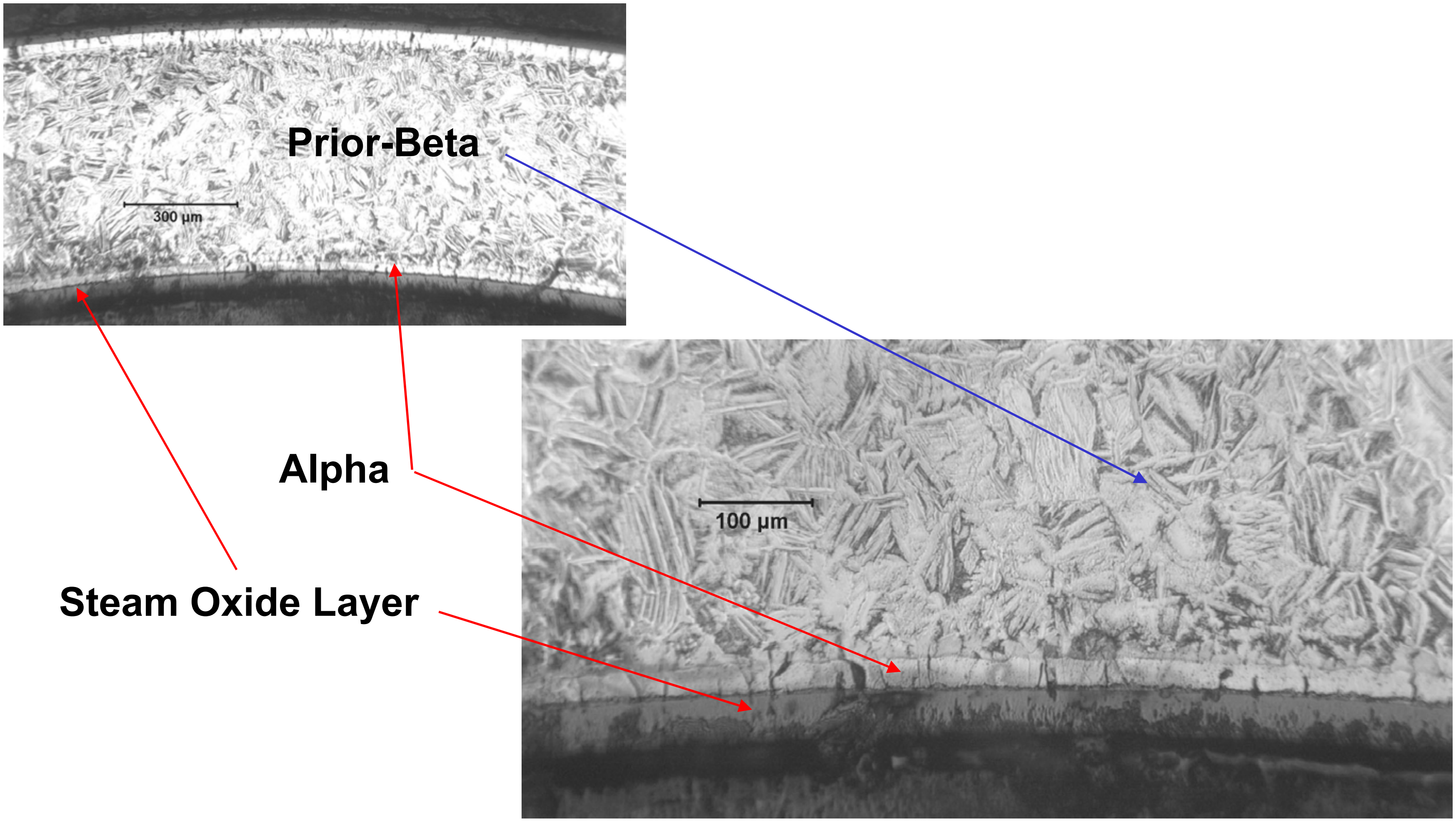
Post-Oxidation Ductility of HBR Cladding at 135ºC: 10% CP-ECR Sample

- **10% CP-ECR with T = 1206ºC Prior to Cooling**
  - Post-test hydrogen: 970±150 wppm (2-mm rings sides of sample)
    750±100 wppm based on 8-mm-long ring
    too high; may not all be in the metal
  - Metallography
    Outer-surface: corrosion layer??, growth of steam-oxide layer
    steam-oxide < O-stabilized alpha layer thickness
    Inner-surface: growth of steam-oxide layer; fuel-clad. bond??
    steam-oxide > O-stabilized alpha layer thickness
    Summary: alpha layer thickness < pre-H, non-irr. sample
    need better metallography → measured ECR
  - Ring-compression ductility = 0.5% offset strain, 0.6% permanent strain
  - **Assessment: very brittle**

Metallography of 10% CP-ECR Sample – Outer Surface

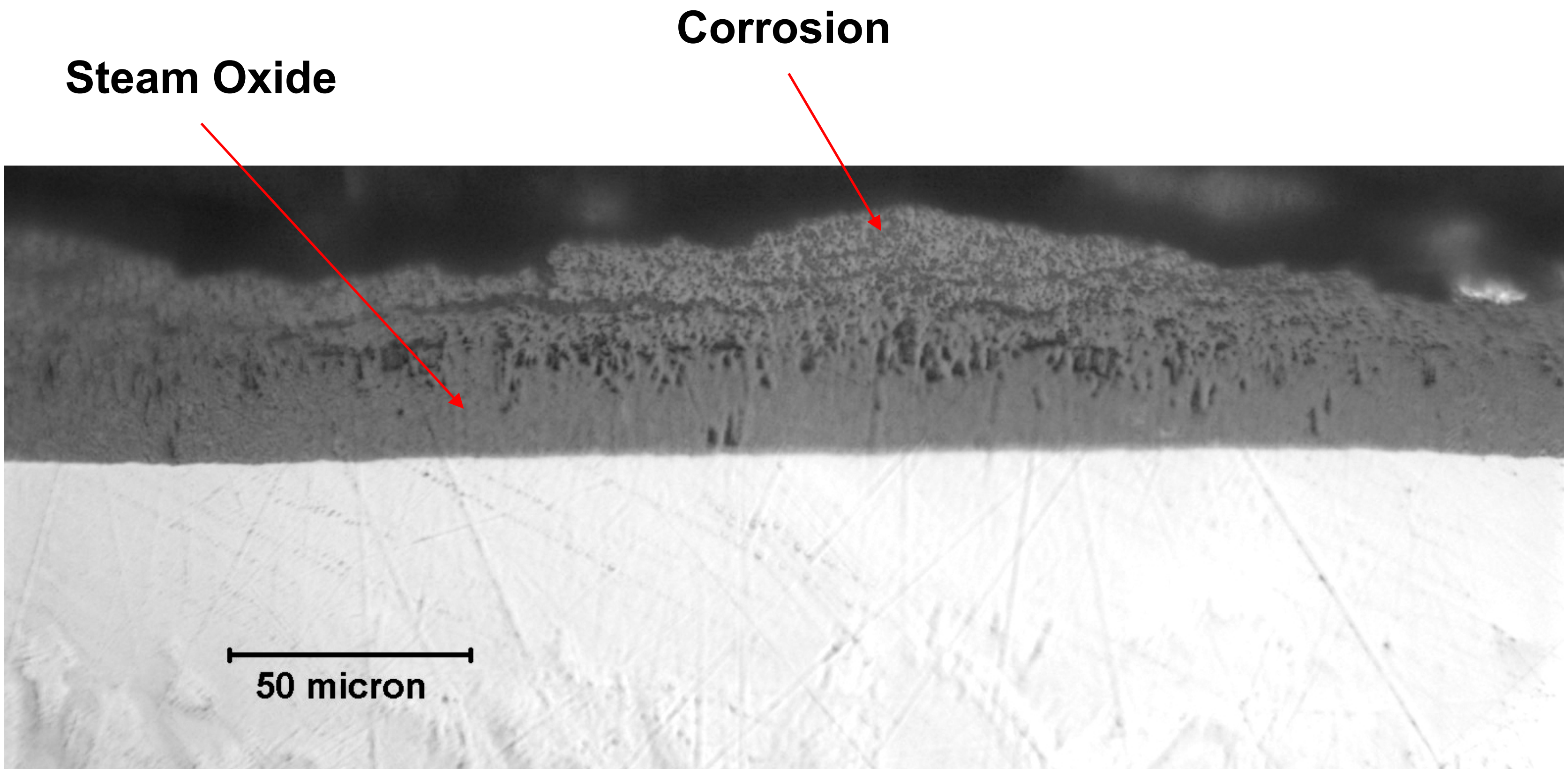


Metallography of 10% CP-ECR Sample – Inner Surface

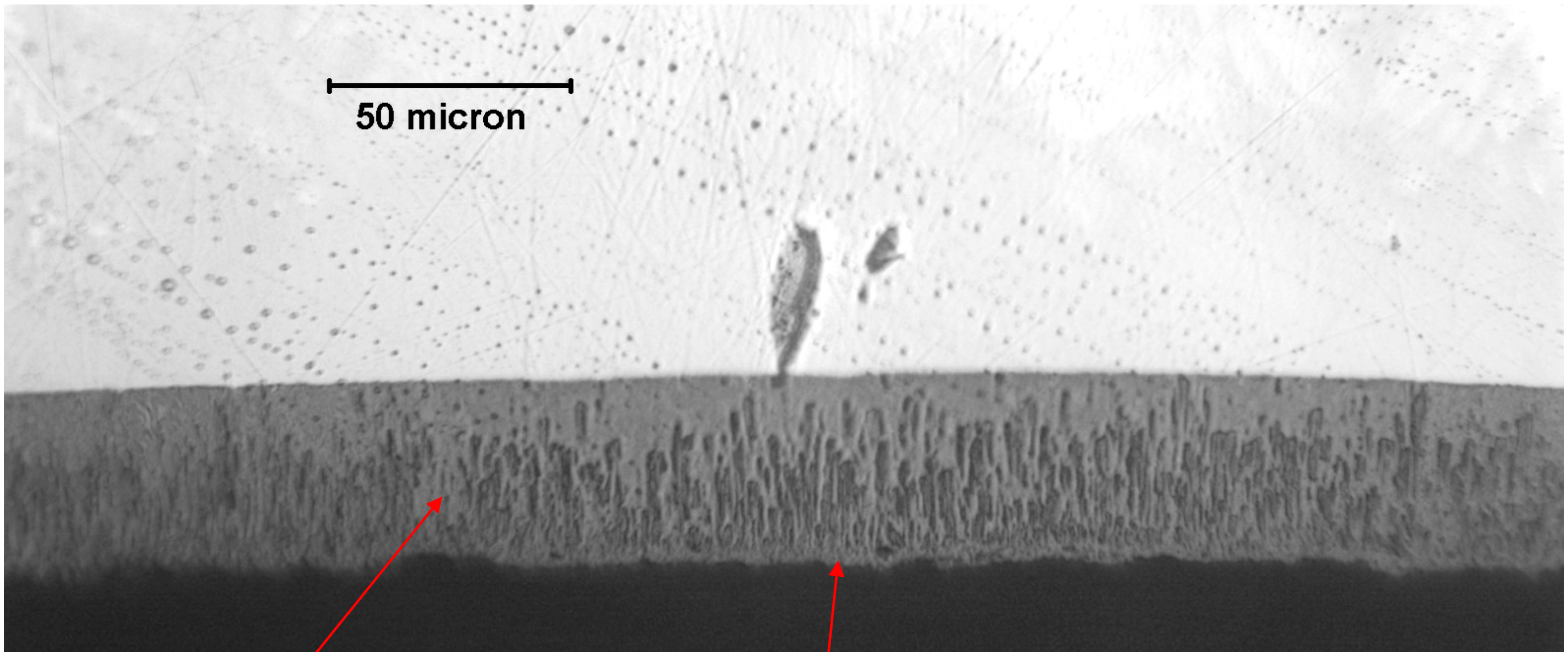


Fuel-Clad Bond???

As-Polished Metallography of 10% CP-ECR Sample Outer Surface



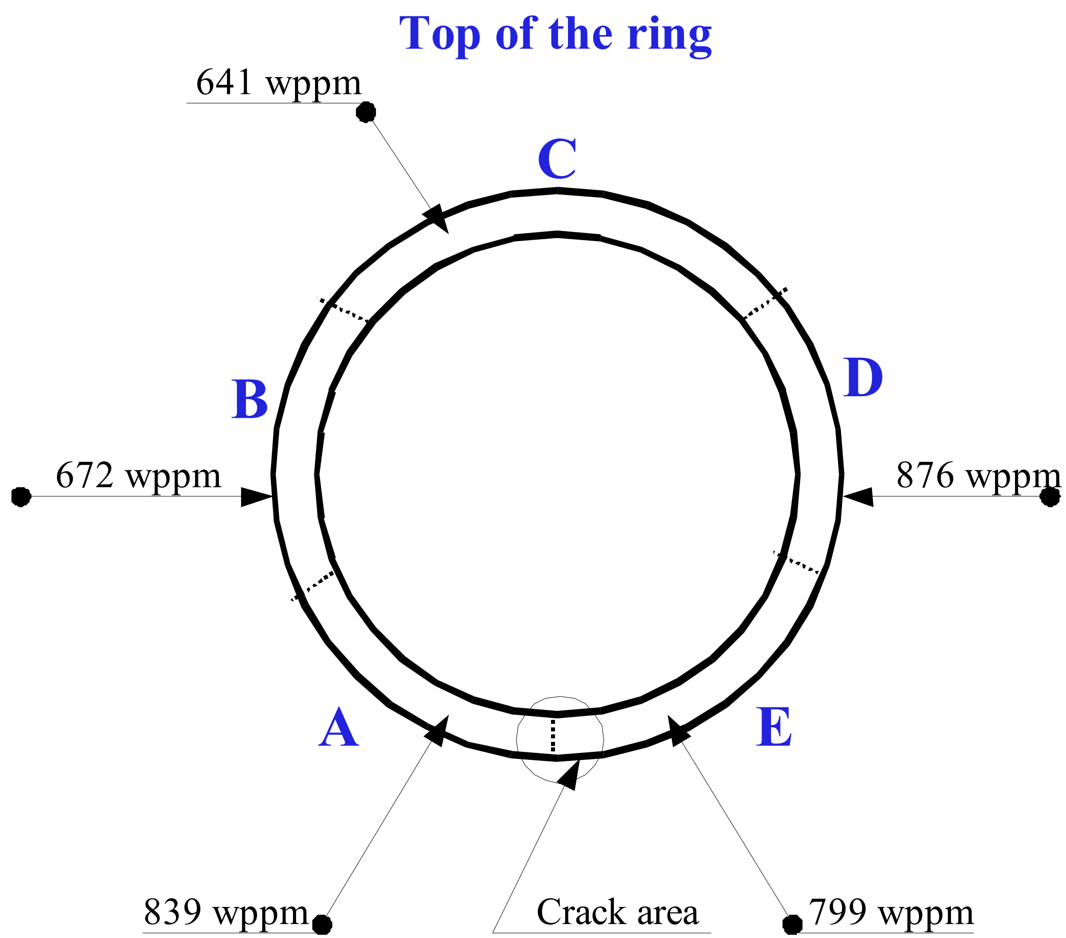
As-Polished Metallography of 10% CP-ECR Sample Inner Surface



Steam Oxide

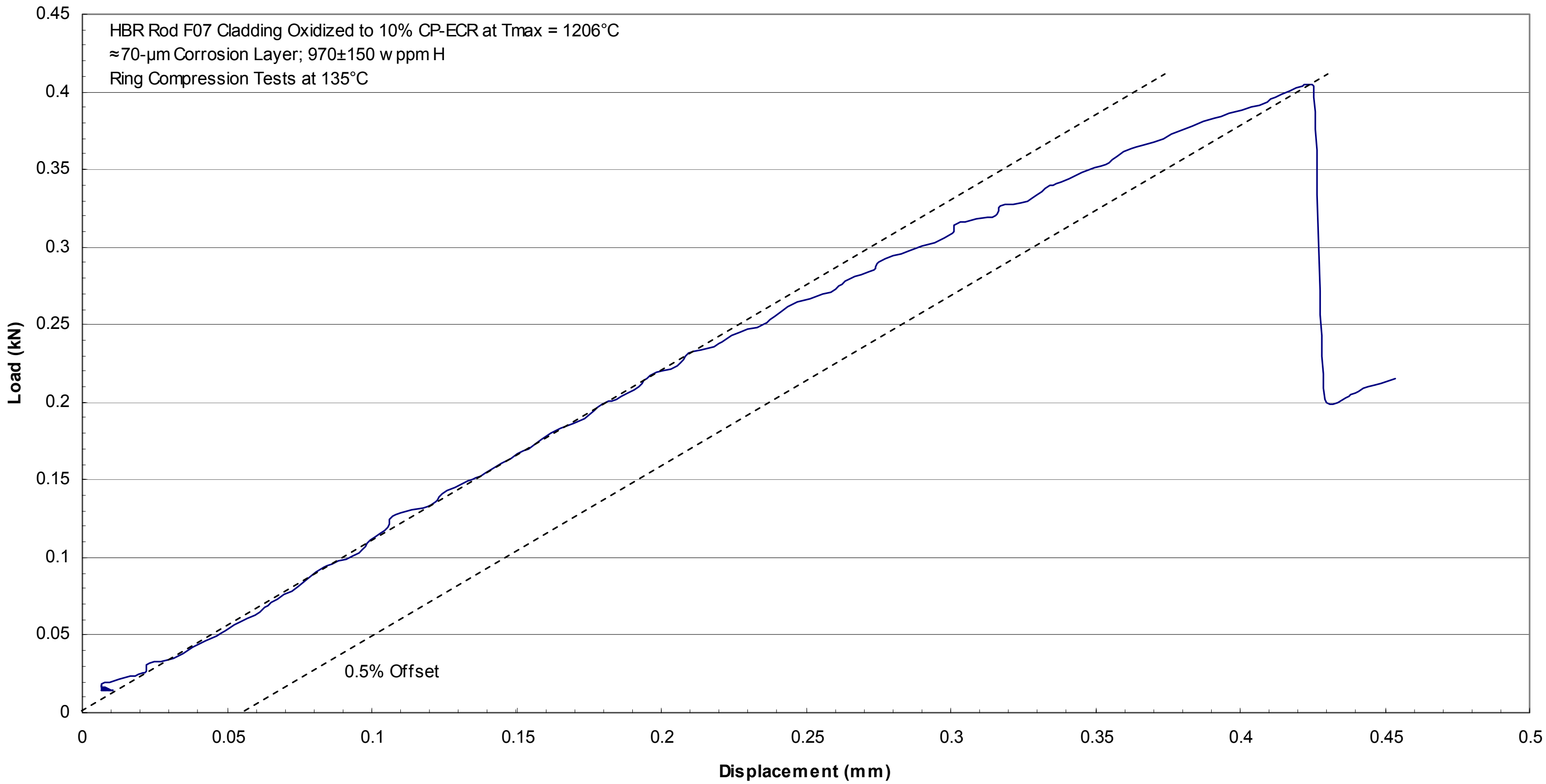
Fuel-Cladding Bond??

Hydrogen Content of 10% CP-ECR Sample

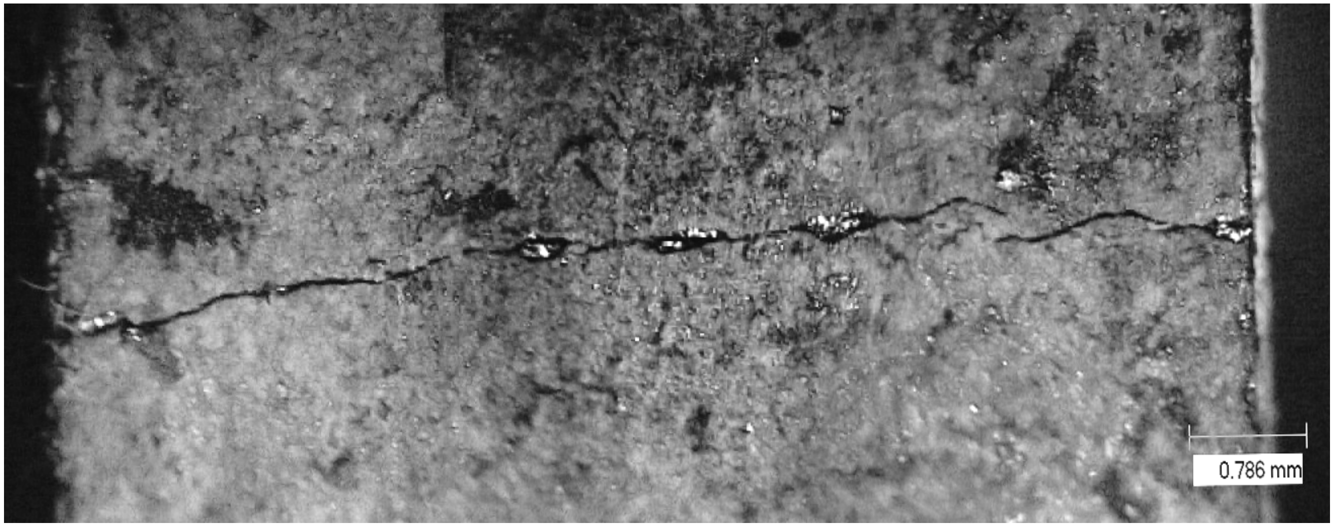


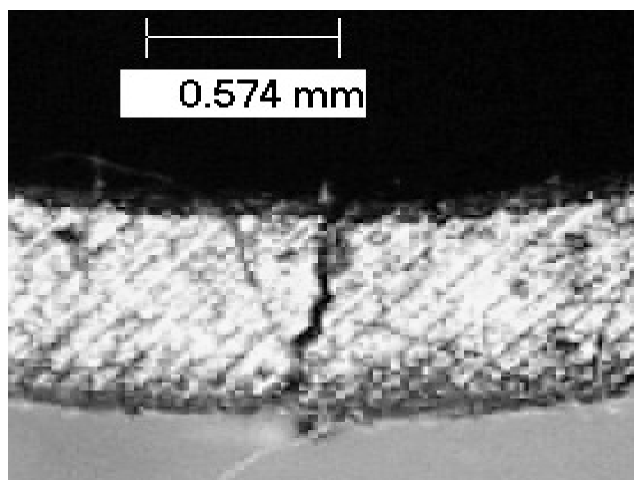
Average Hydrogen Content - 751 wppm

Load-Displacement Curve (at 135ºC) for 10% CP-ECR HBR Zry-4 Ring with 970±150 wppm H (Post-Oxidation)



Low Mag. Images of Crack at Bottom Support Plate in 10% ECR Sample





Post-Oxidation Ductility of HBR Cladding at 135ºC: 8% CP-ECR Sample

- **8% CP-ECR with T = 1206ºC Prior to Cooling**
  - Note: sample was defueled very thoroughly prior to testing
  - Post-test hydrogen: 545±80 wppm (2-mm rings sides of sample)
  - consistent with pre-oxidation H content
  - Metallography (in progress with vacuum-impregnation of epoxy)

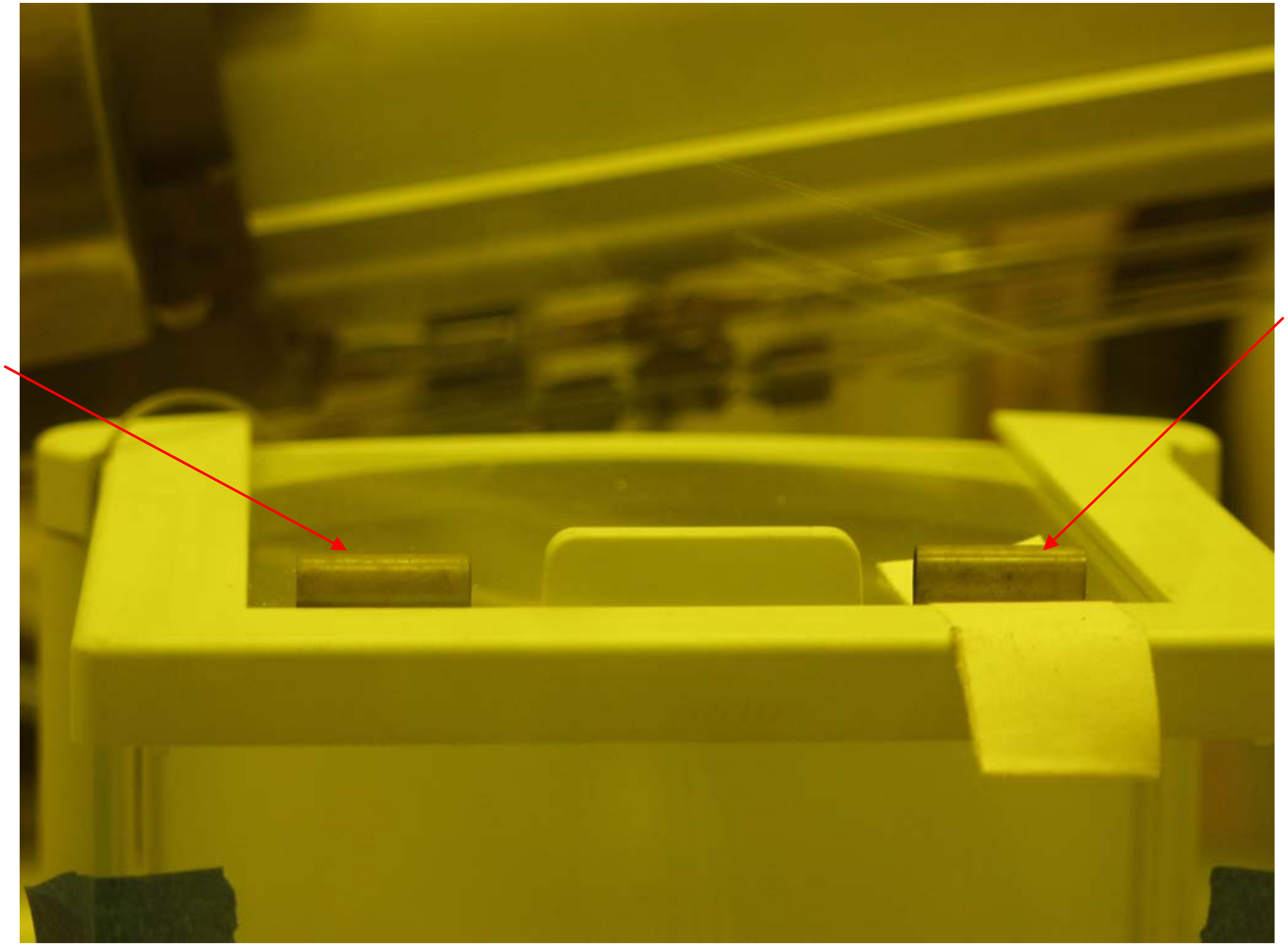
    Outer-surface:

    Inner-surface:

    Summary:
  - Ring-compression ductility = 3.8% offset strain, 2.9% permanent strain
  - **Assessment: low ductility**

Pre-Test Visual Appearance of Sample

Untested Sample



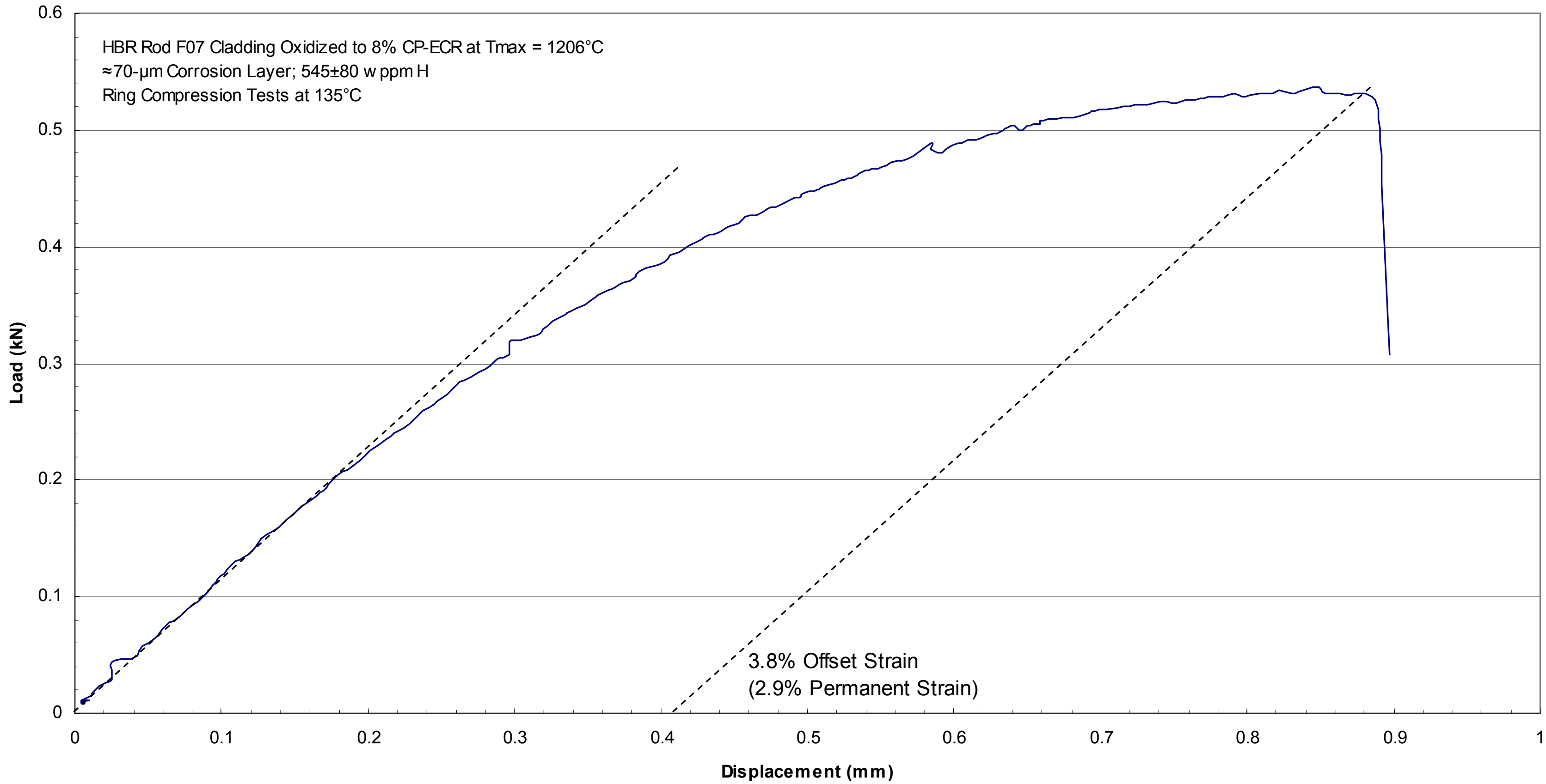
Sample Prior to Testing

Post-Test vs. Pre-Test Visual Appearance of Sample

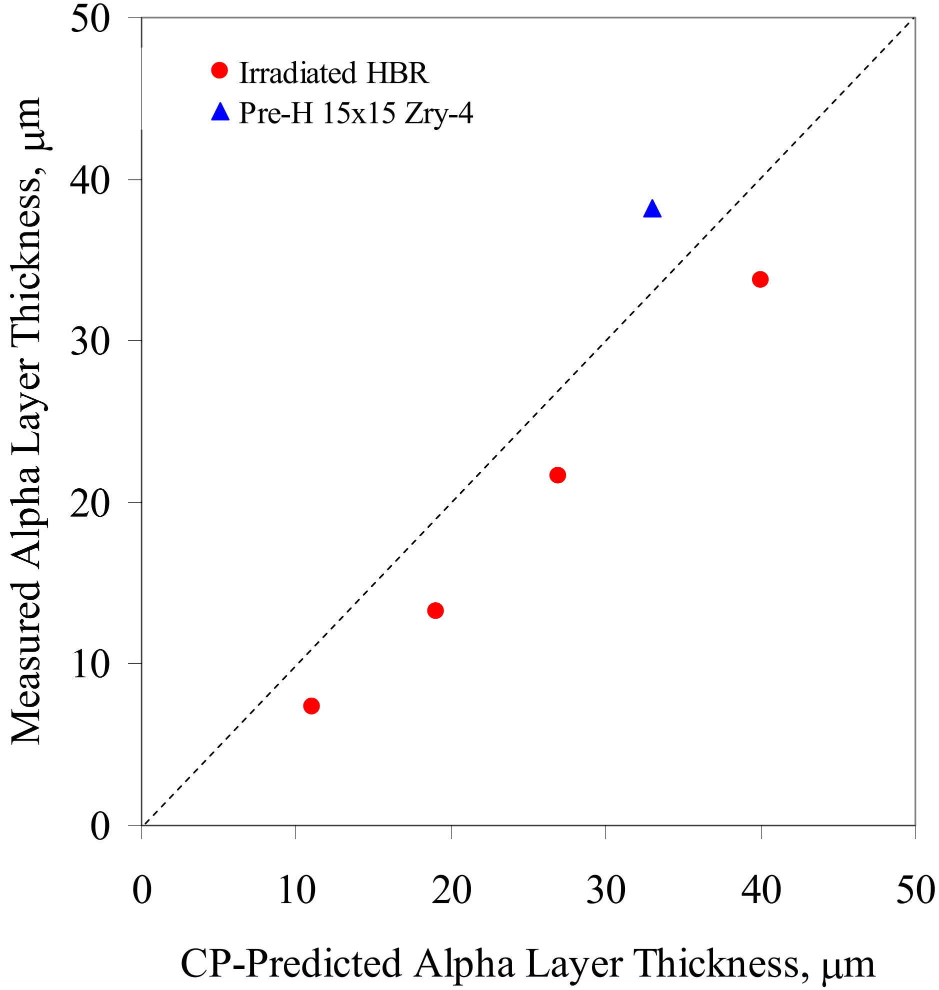
Untested Sample

Sample After Oxidation

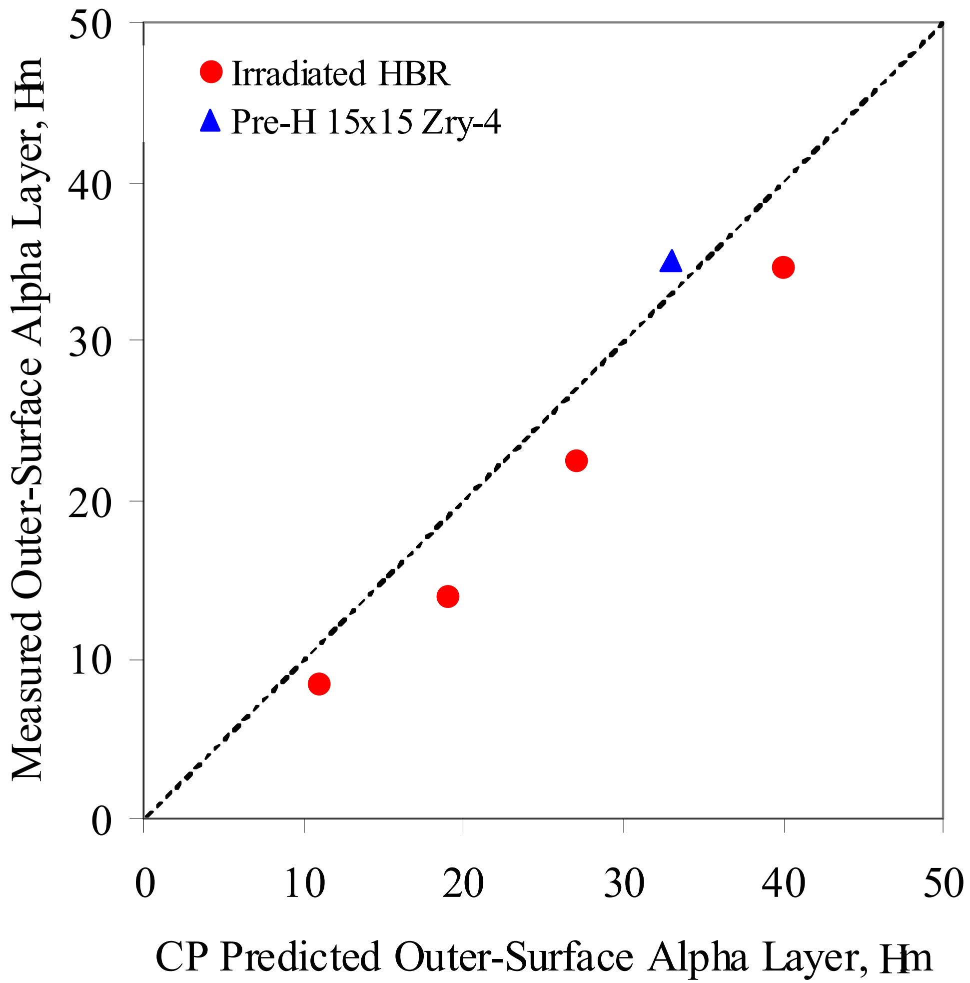
Load-Displacement Curve (at 135ºC) for 8% CP-ECR HBR Zry-4 Ring with 545±80 wppm H (Post-Oxidation)



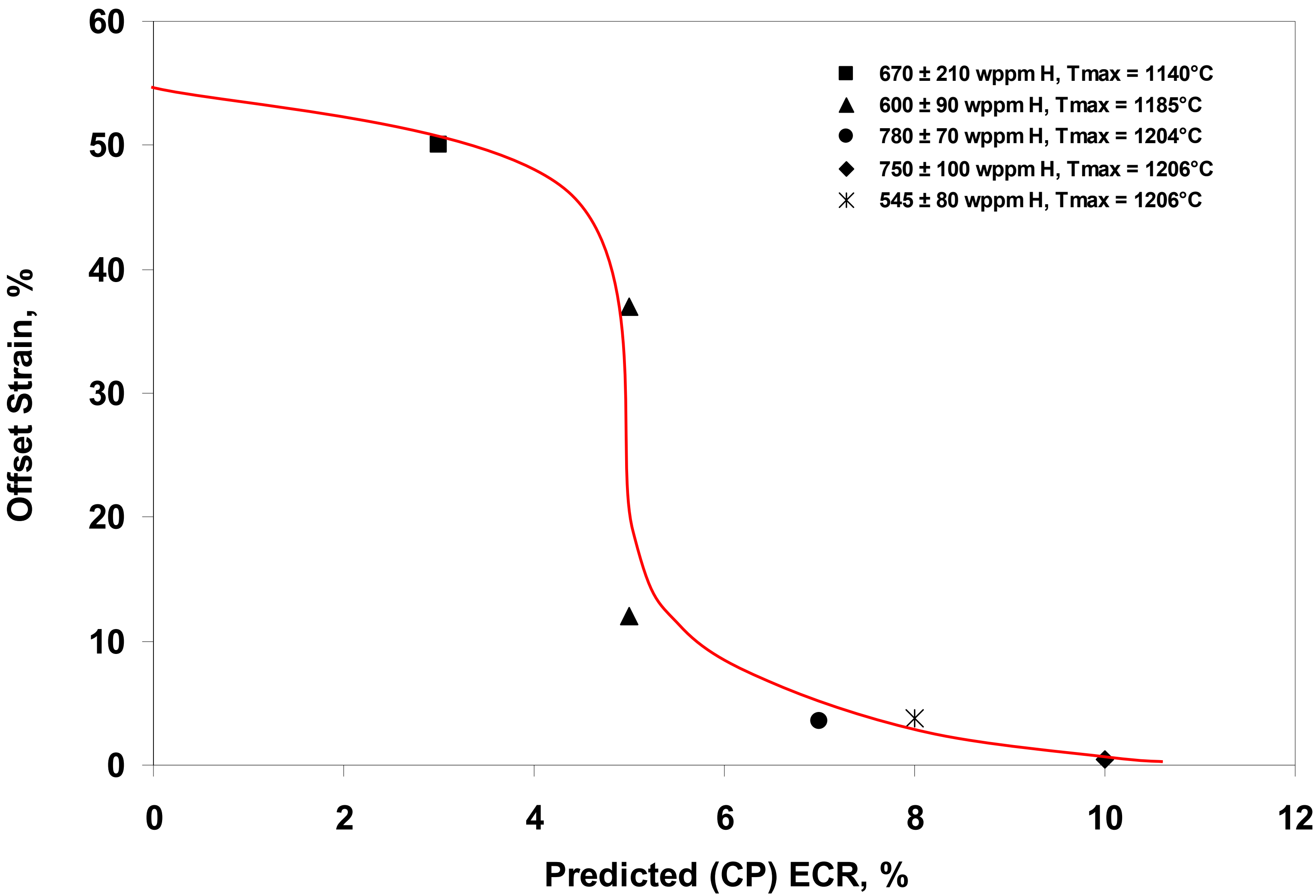
Measured vs. Predicted Average Alpha Layer Thickness Preliminary Results for (OD+ID)/2 Alpha Layer



Measured vs. Predicted Alpha Layer Thickness Preliminary Results for Outer-Surface Alpha Layer



Summary of Post-Oxidation Ductility (Offset Strain) at 135ºC vs. CP-ECR for High-Burnup Zry-4 Cladding



Discussion of Results for High-Burnup HBR Cladding

- **Characterization of As-Irradiated Rod F07 Cladding**
  - Significant hydrogen variation for the 1st 5 defueled segments
  - Expected H content & variation for the next 2 defueled segments
  - Reasonable to assume ductility data correspond to ≈550±100 wppm H
- **Effects of Corrosion Layer on Steam Oxidation Kinetics**
  - Corrosion layer is a source of oxygen for embrittlement and a "partial" barrier to steam oxidation, especially for short times at temperature
  - Metallography of 3-10% CP-ECR samples suggests that corrosion layer acts as partial barrier in slowing down oxidation
  - Better mounting of 8% CP-ECR sample is in progress
  - Other data on effects of corrosion layer

    French data (1990s): PWR Zry-4 corrosion layer is non-protective

    ANL data: BWR Zry-2 (10 μm, ≥300 s at 1204ºC): non-protective

    JAERI data: PWR Zry-4 (20-25 μm): partially protective at 1000ºC

Discussion of Results for High-Burnup HBR Cladding (Cont'd)

- **Effects of Quench**
  - One sample will be oxidized to 8% CP-ECR & quenched (in progress)
- **Effects of Temperature Ramp Rate on PQD**
  - Significant effect expected for low-ECR 2-sided oxidation tests
  - Range of ramp rates reported in the literature

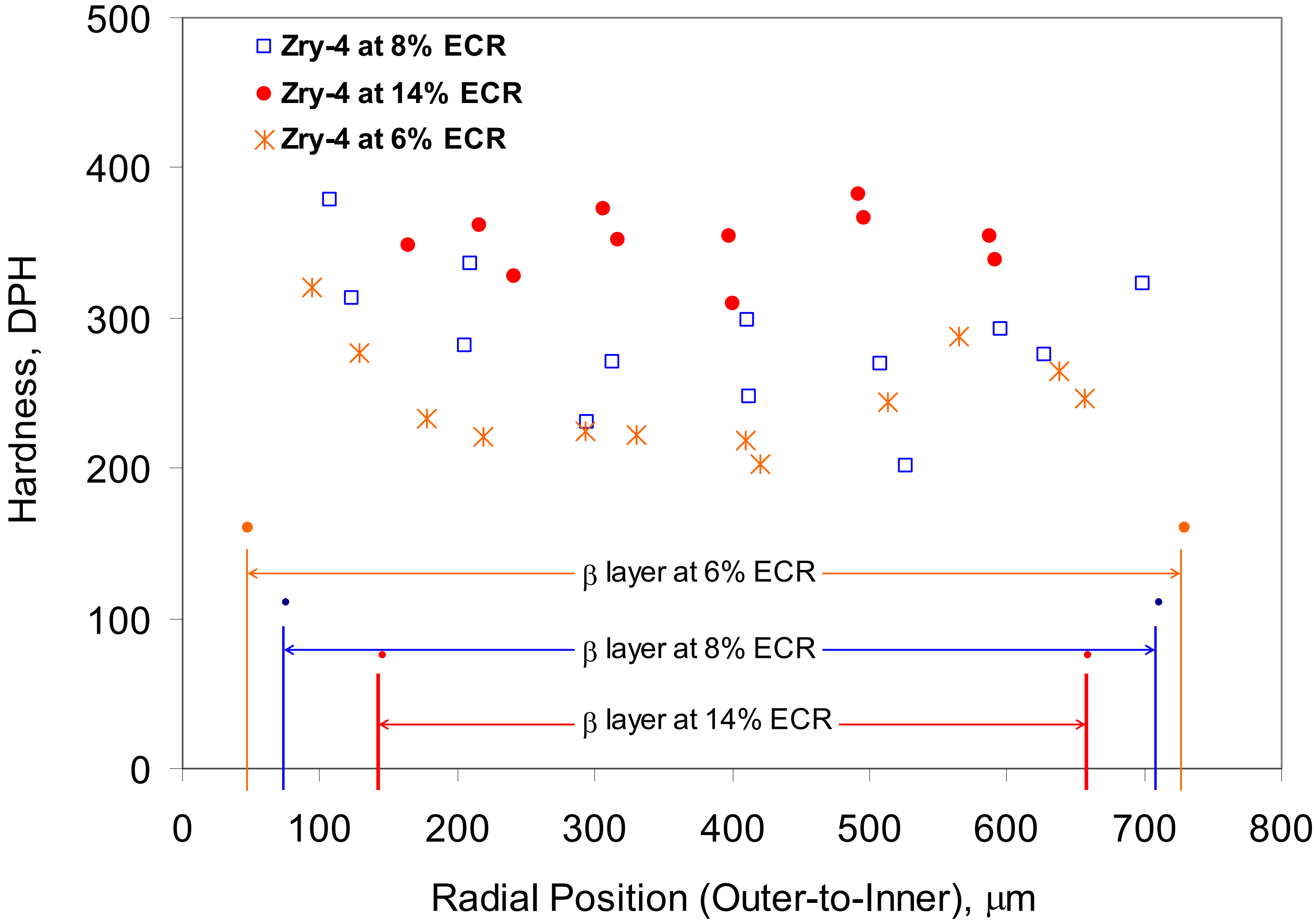
    20ºC/s from 1000 to 1200ºC → 1% ECR for 2-sided oxidation

    0.5ºC/s from 1000 to 1200ºC → 12% ECR for 2-sided oxidation
  - ANL test times at T = 1180-1206ºC (severe embrittlement expected)

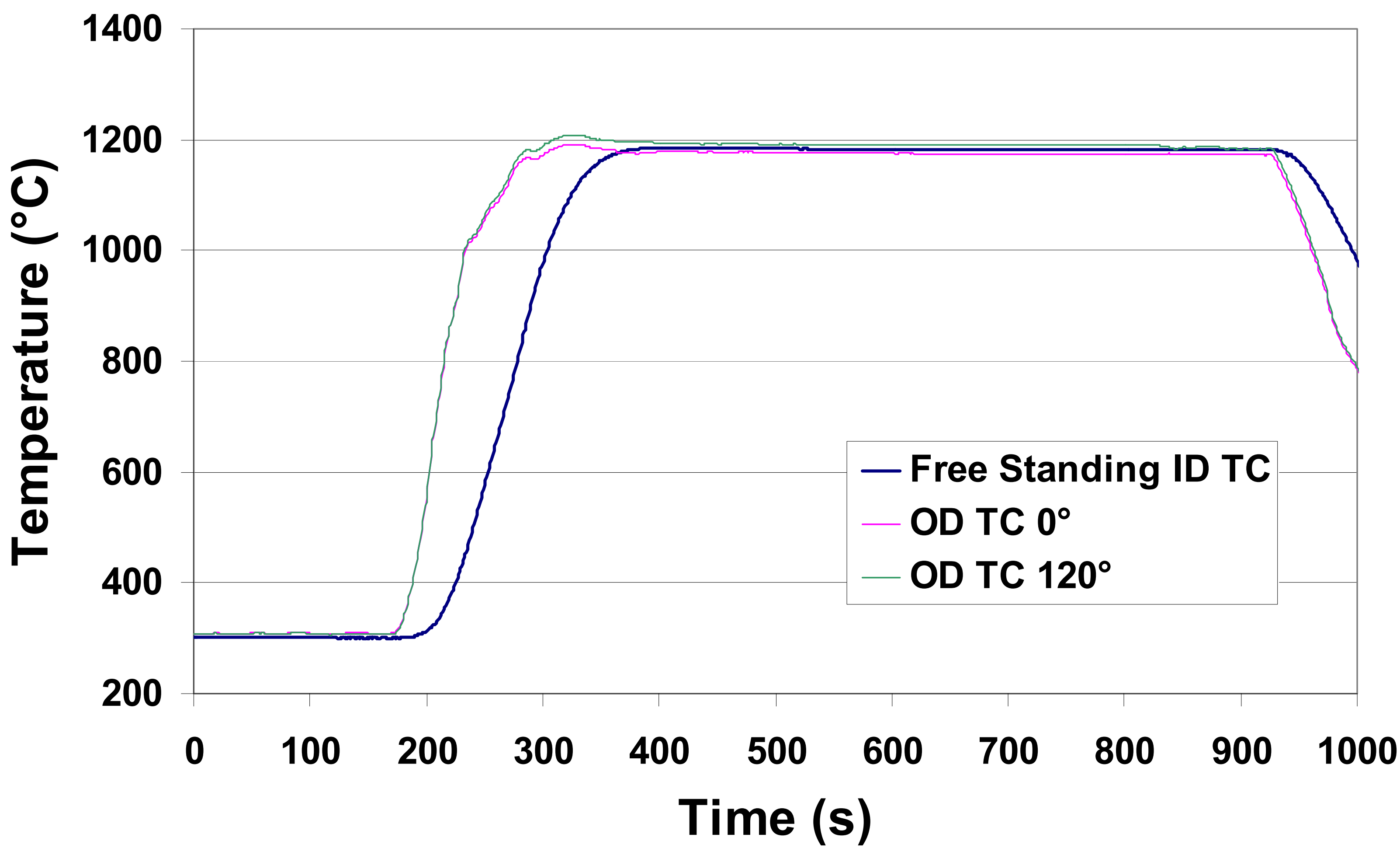
    3% CP-ECR, t = 0 s; 5% CP-ECR, t = 5 s; 7% CP-ECR, t = 44 s

    8% CP-ECR, t = 77 s; 10% CP-ECR, t = 118 s (β-layer saturation)
- **Effects of Corrosion Layer on Cladding Temperature**
  - Low heat of oxidation may result in lower ramp temperatures
  - Adsorption/emissivity coefficients: corrosion < steam oxide layer
  - Neither of these would effect the 1204ºC hold temperature

Microhardness Profiles for 15×15 Zry-4 Oxidized at 1200ºC to 6%, 8% and 14% ECR



Temperature Distribution in 10-minute Test with Unirradiated Zry-2 Sample



Future Plans

- **Post-Quench Ductility of High-Burnup Zry-4**
  - 2-sided oxidation: effects of quench on 8% CP-ECR ductility (2/05)
  - 1-sided oxidation: T_h = 1204°C (3/05), 1000°C & 1100°C (5/05)

    Fast ramp to T_h - 50°C, slower ramp to T_h

    CP-ECR = 3, 5, 7.5, 10% at 1204°C; 5, 10, 13% for 1000 & 1100°C

    Run tests without quench at 800°C; repeat one test with quench

    Ring-compression tests at 135°C & 80-100°C
  - LOCA integral tests (8/05)

    1 test to burst with slow cooling → balloon-burst characterization

    3 tests at 3, 5, 7.5% CP-ECR in non-ballooned region (TBD)

    4-point-bend test (RT), ring-compression tests at 135°C & 80-100°C

Future Plans (Cont'd)

- **Post-Quench Ductility of High-Burnup ZIRLO**
  - Perform Zry-4 oxidation/quench/compression test matrix
    ZIRLO from North Anna → Studsvik → ANL
    1204°C tests (8/05); 1000°C & 1100°C tests after 9/05
    Baseline data on nonirradiated, prehydrided ZIRLO?????
  - Perform LOCA integral test matrix (>9/05)
    Fuel at ≈48 GWd/MTU (<62 GWd/MTU)
- **Post-Quench Ductility of High-Burnup M5**
  - Perform Zry-4 oxidation/quench/compression test matrix
    European M5 → Studsvik → ANL
    1204°C tests (8/05); 1000°C & 1100°C tests after 9/05
    Baseline data on nonirradiated, prehydrided M5 desirable (135°C)
  - Perform LOCA integral test matrix (>9/05)
    M5 from North Anna → (nee ANL-W) → ANL-E