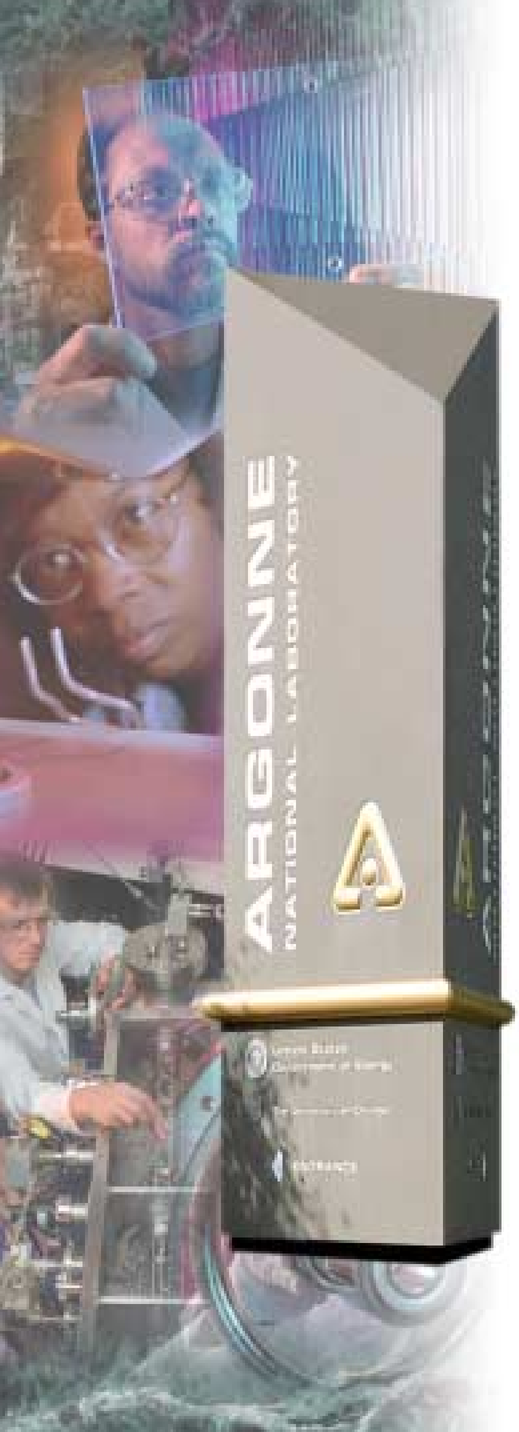

Advanced Alloy Post-Quench Ductility Data

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Review of ANL LOCA and Dry-Cask-Storage Programs
Argonne National Laboratory
July 16-17, 2003

Argonne National Laboratory

Office of Science
U.S. Department of Energy

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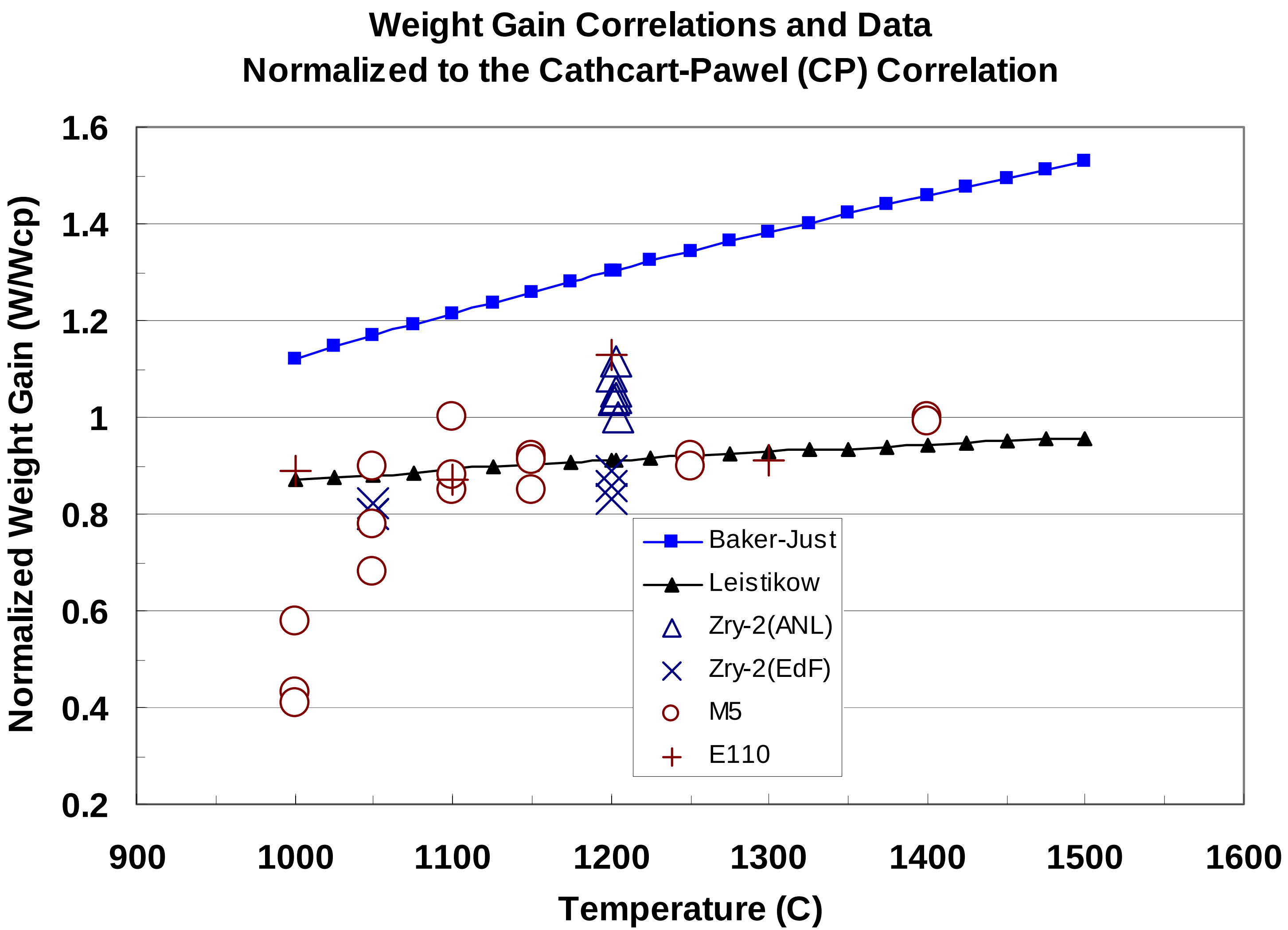
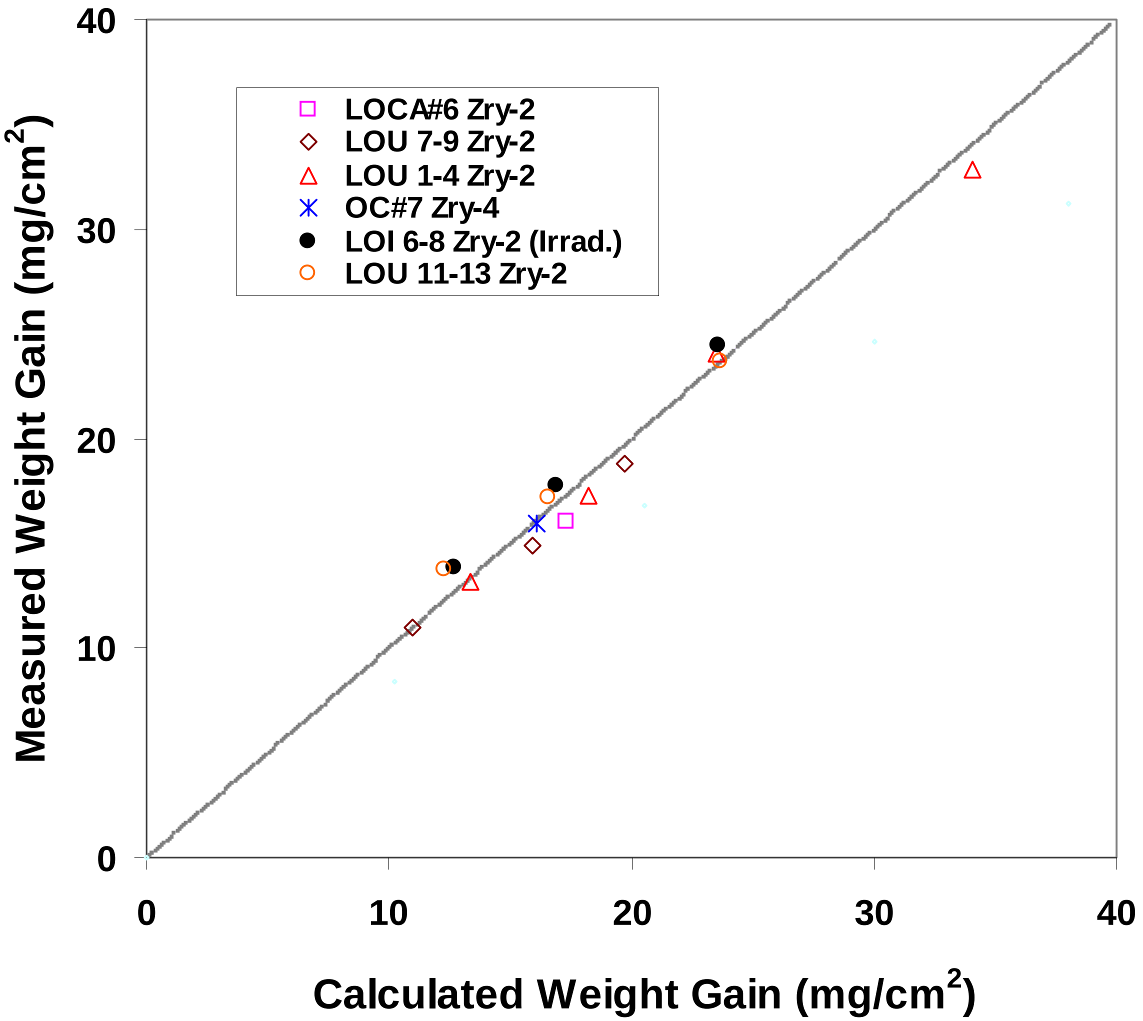
Background on Weight Gain Kinetics From High-Temperature Oxidation in Steam

- **ANL Review of Published Data in Mar.-June 2002**
  - Data were normalized to the Cathcart-Pawel (CP) model
  - All materials exhibited similar weight gain kinetics at 1100-1500°C
  - Weight gain coefficient for M5 ≈ 50% less than Zry-4 at 1000°C
  - **More lab-to-lab variation in data than alloy-to-alloy variation**
  - Westinghouse data: good agreement between ZIRLO and Zry-4; specific T, t and WG were not given; ramp rate (0.56°C/s) too slow; Could not compare W Zry-4 and ZIRLO directly to Zry-2, M5, E110
- **E110 Mystery**
  - Weight gain kinetics consistent with CP model based on protective (tetragonal) oxide layer, but hydrogen pickup is high???
  - Seemingly inconsistent results require in-depth review of E110 data

ANL Review of Published Weight Gain Data



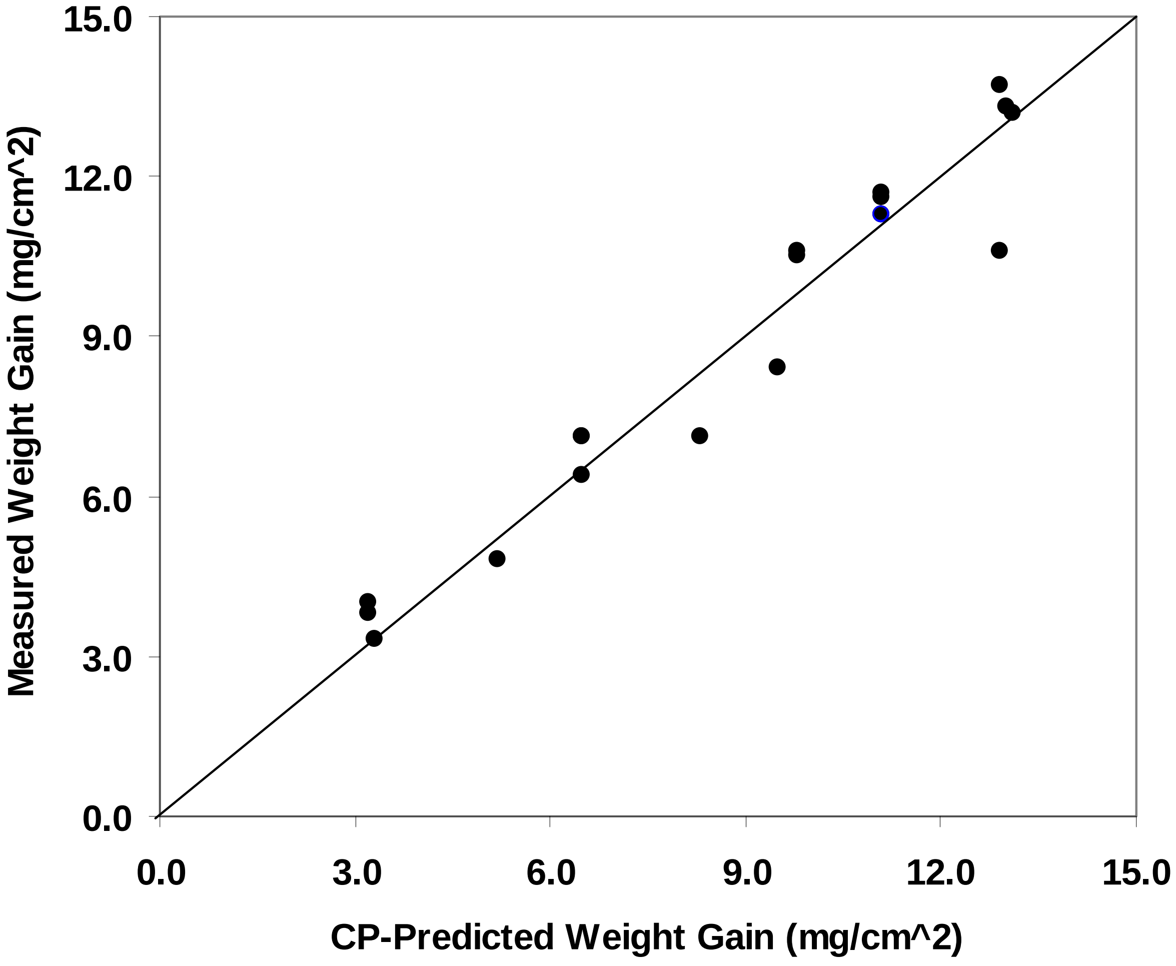
ANL Steam Oxidation Data (One-Sided) at 1200°C Archival and High-Burnup Limerick BWR Cladding



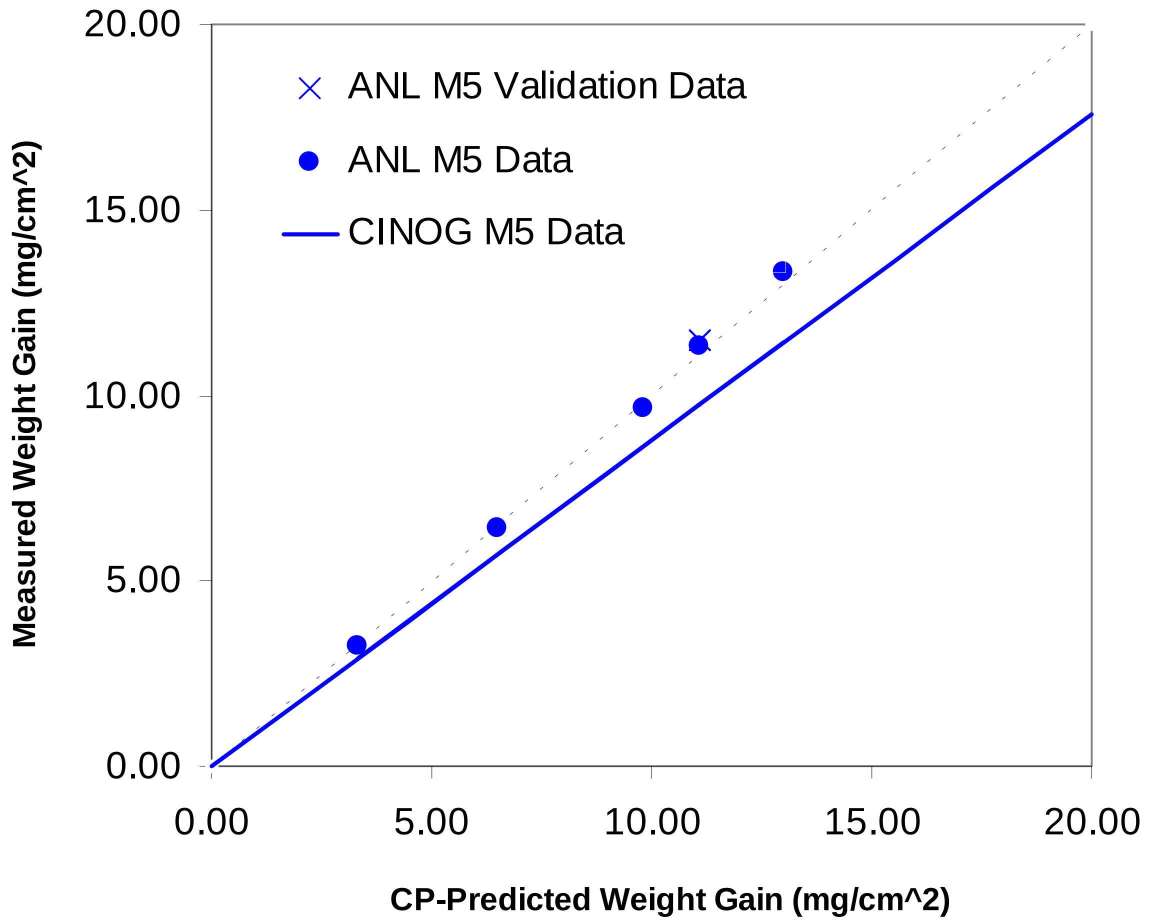
Weight Gain Kinetics from Current ANL Program

- **Weight Gain Kinetics at 1100°C**
  - Zry-4, M5 and ZIRLO data are in agreement with Cathcart-Pawel (CP) model predictions (within ≈ ±10%)
  - Could not get meaningful data from as-received E110 (oxide instability)
  - Data were obtained on polished E110 following ID machining to reduce wall thickness from 0.71 mm to 0.61 mm up to point of oxide instability
- **Weight Gain Kinetics at 1000°C**
  - Zry-4 and ZIRLO tests are in progress
  - Meaningful E110 data for polished E110 with 0.61-mm wall
  - M5 and polished E110 have similar weight gain (WG) kinetics
- **Tests at 1200°C and 1260°C will Follow 1000°C**
- **No Effects of Quench at 800°C on Weight Gain**

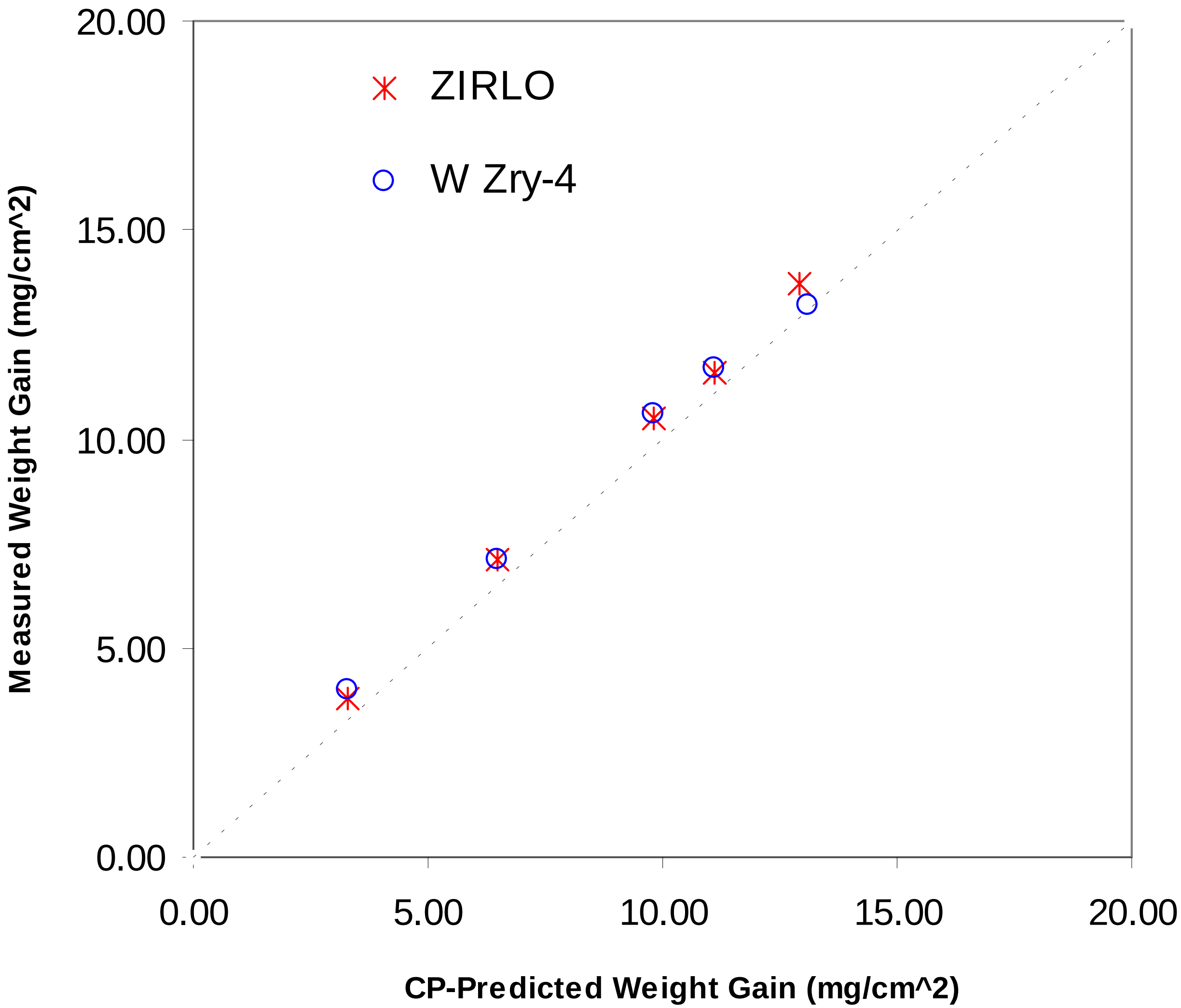
ANL Weight Gain Data for All Alloys at 1100°C



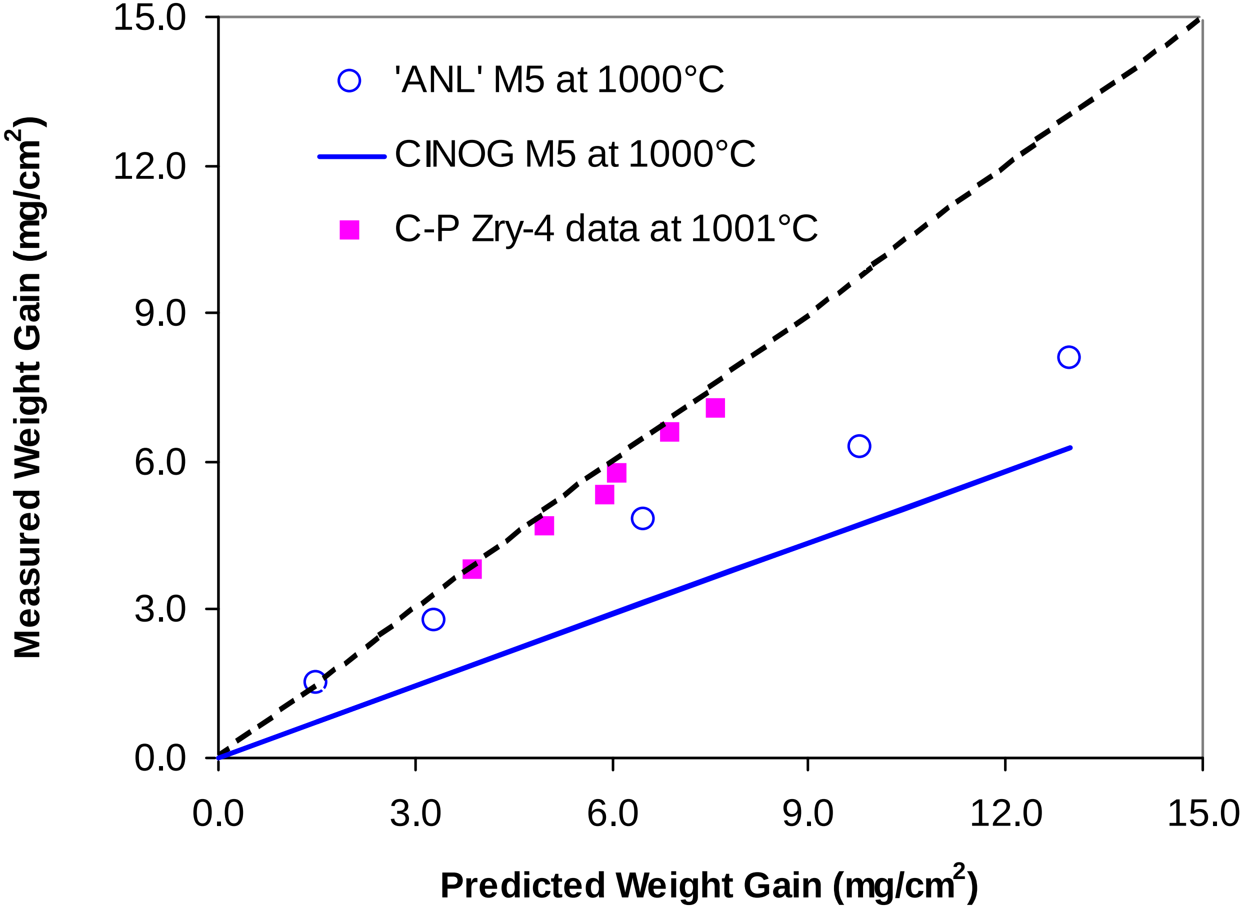
ANL and CINOG Weight Gain Data at 1100°C



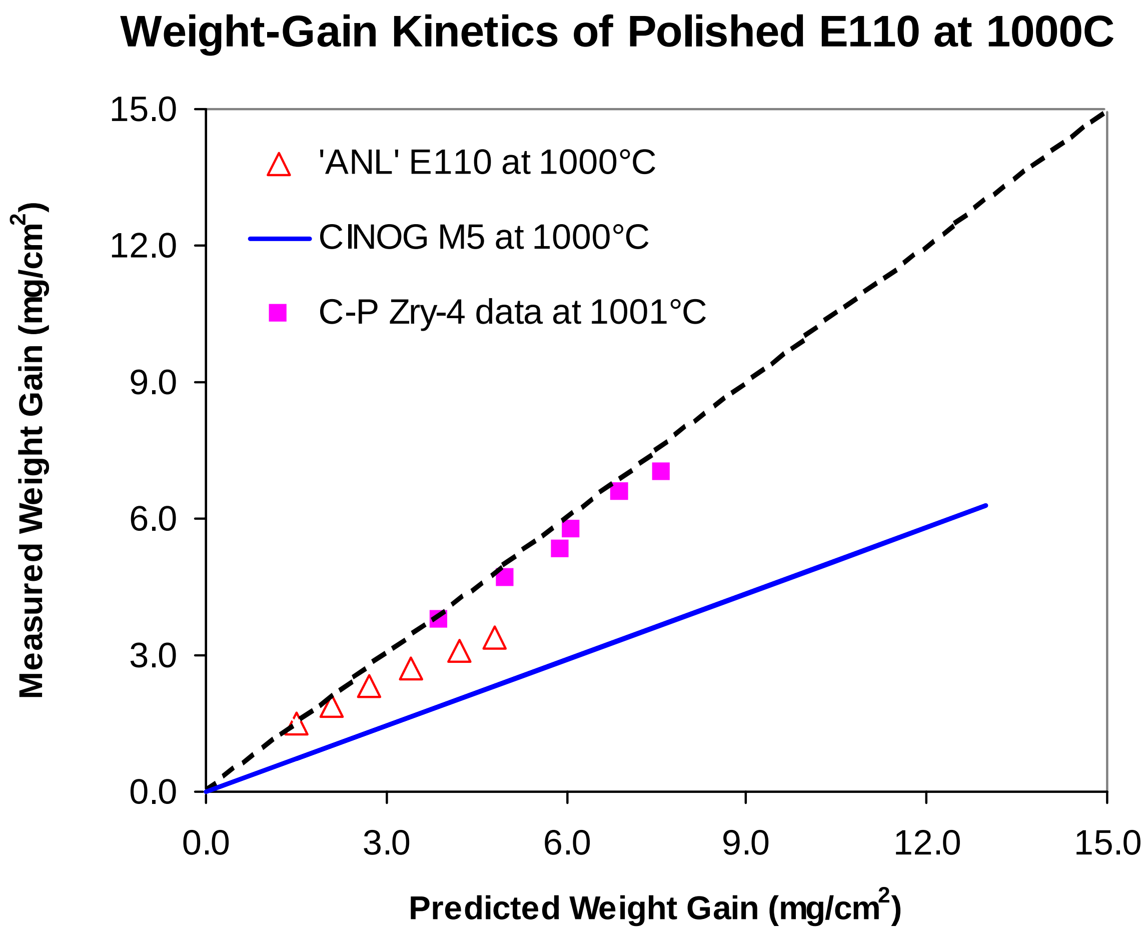
Zry-4 & ZIRLO Weight Gain Data at 1100°C



ANL vs. CINOG M5 Weight Gain Data at 1000°C



ANL Weight Gain Data for Polished E110 at 1000°C



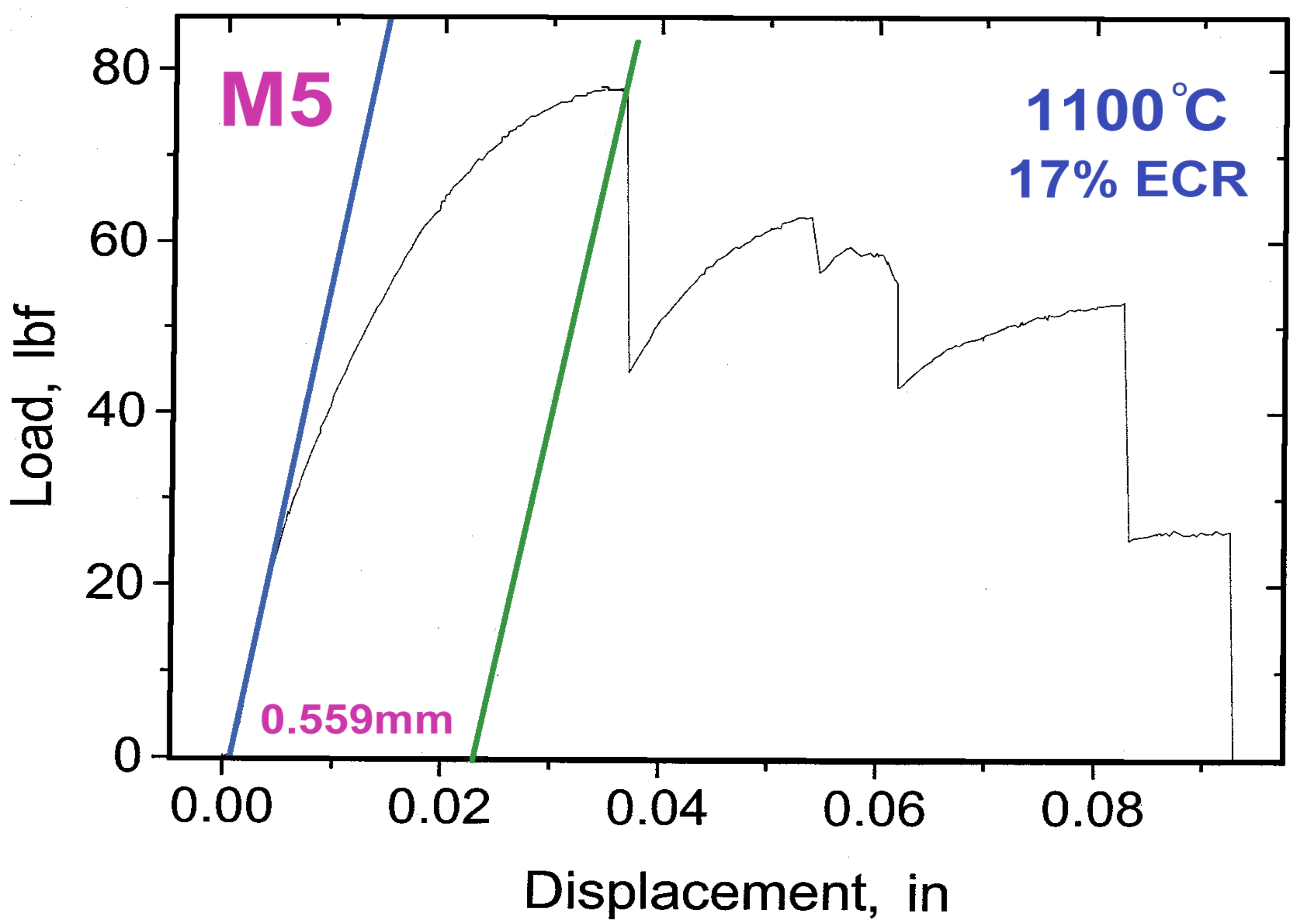
Post-Quench-Ductility Validation Tests

- **Ring Compression Test Parameters and Methodology**
  - RT screening tests at 2 mm/min (0.35%/s) for 8-mm-long rings
  - Measure off-set displacement (δ_p) vs. CP-ECR (5, 10, 15, 17, 20%)
  - Convert to "nominal" strain ($\varepsilon = \delta_p/D_o$) vs. CP-ECR
  - Record measured ECR from measured weight gain and thickness
  - Data can easily be re-plotted vs. measured ECR
- **Validation Results**
  - Results easier to interpret as ductility → 0 (e.g., E110)
  - Zry-4, M5 and ZIRLO all appear to be ductile for CP-ECR up to ≈18%
  - Results do not appear to be dependent on quench vs. slow cooling
  - E110 shows expected embrittlement vs. CP-ECR at 1000°C
  - Correlation between E110 hydrogen content and ductility at 1000°C

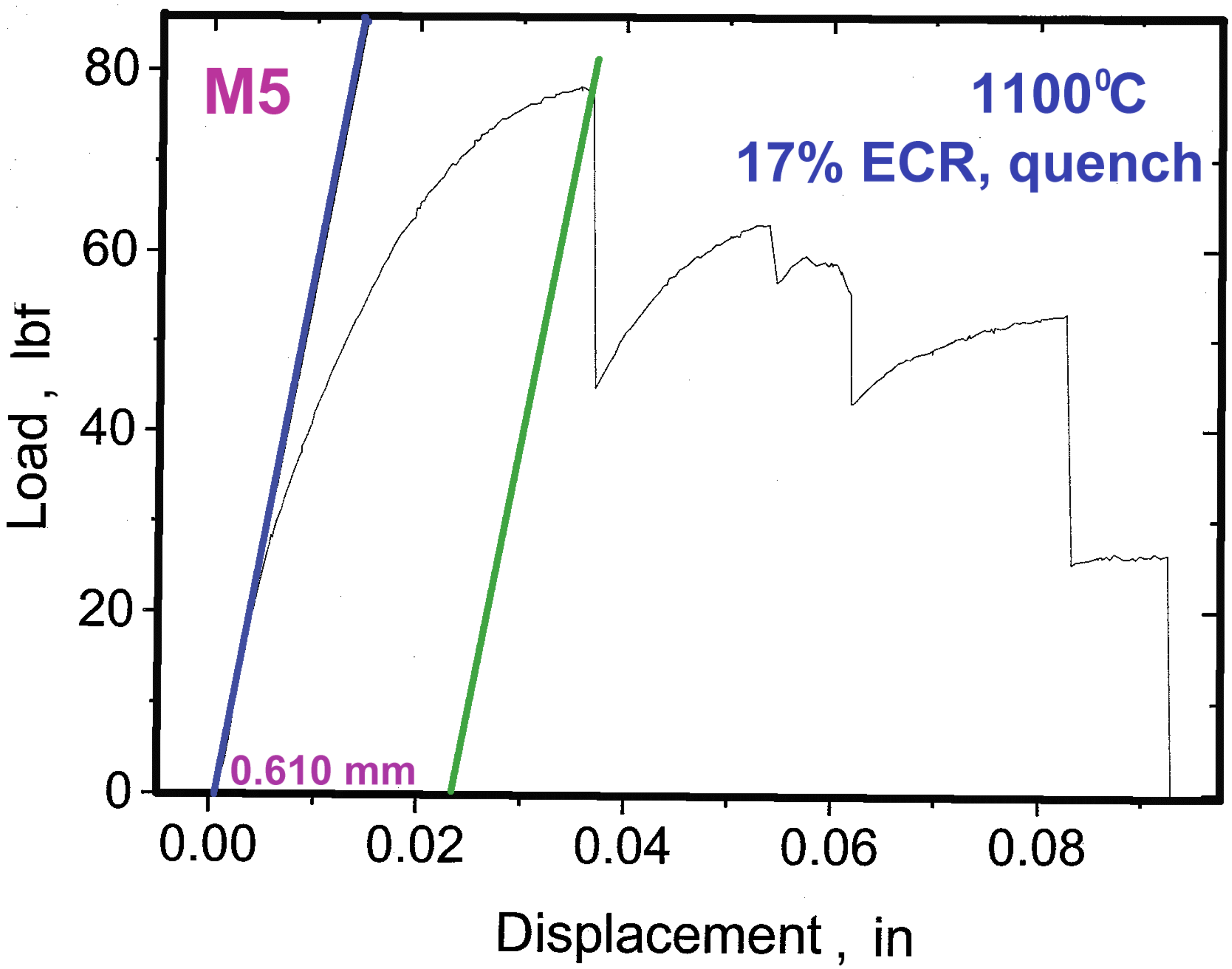
Validation RT Ring Compression Results for M5

Test ID	T °C	CP ECR, %	Data ECR, %	δ_p mm	δ_p/D_o %
MU#0	---	---	---	High	High
MU#10	1100	12	12	1.17	12.3
MU#15	1000	17	12	0.33	3.5
MU#25	1100	17	17.7	0.56	5.9
MU#26 (Quench)	1100	17	17.7	0.61	6.4
MU#12	1100	18	17.8	0.33	3.5

M5 after 18% Measured ECR at 1100°C (No Quench)



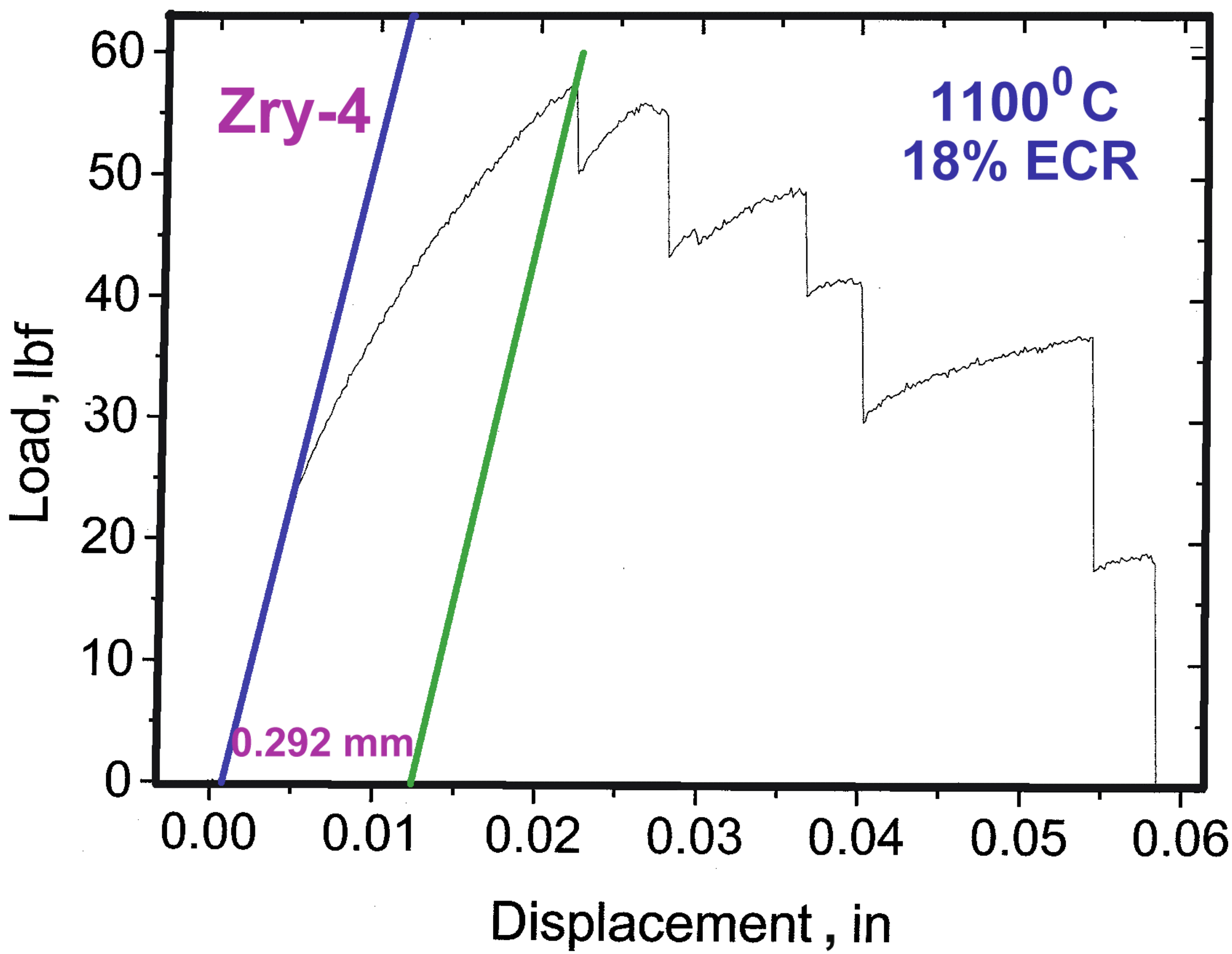
M5 after 18% Measured ECR at 1100°C (Quench)



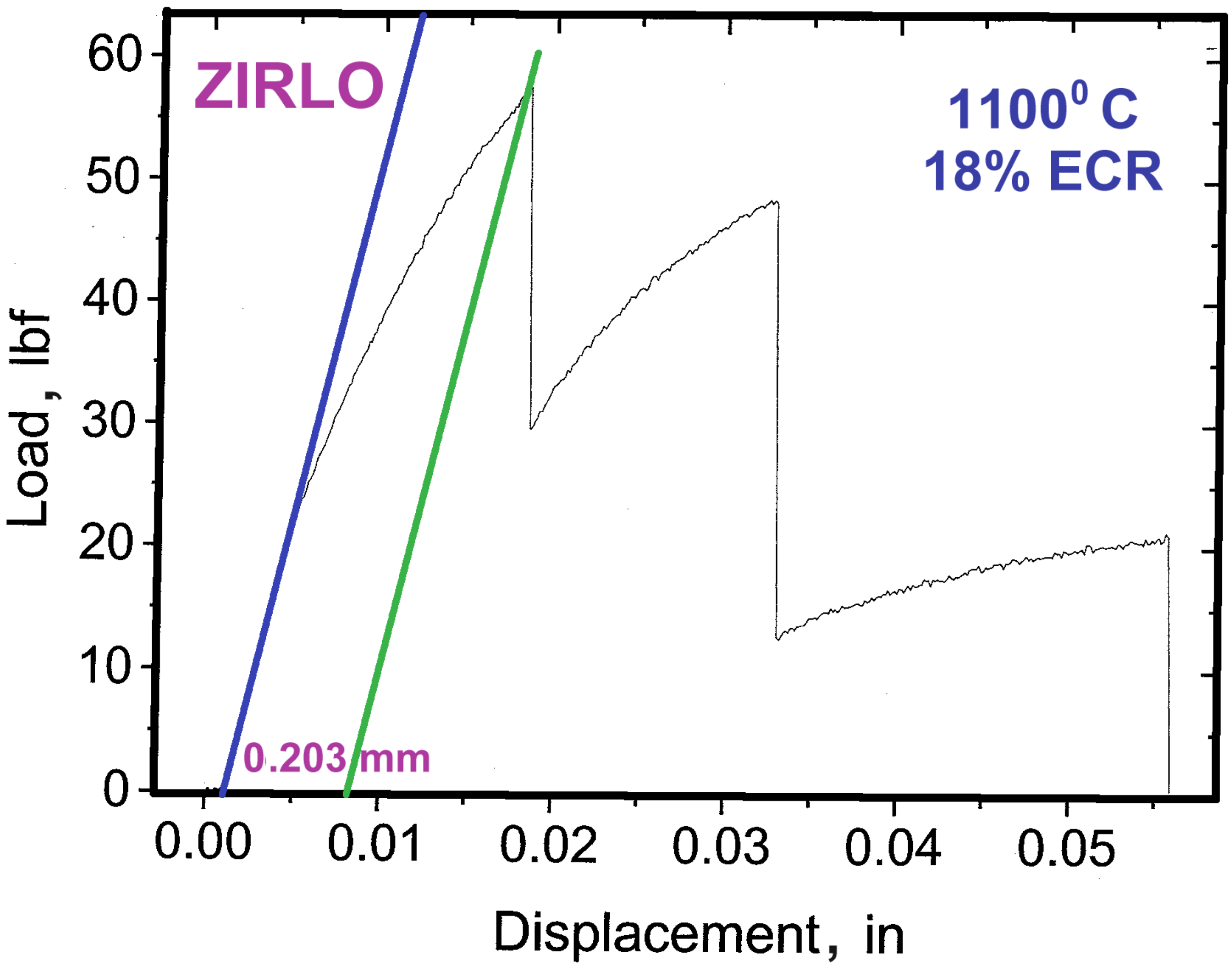
Validation Ring Compression Results at RT

Material	T °C	CP ECR, %	Data ECR, %	δ_p mm	δ_p/D_o %
Zry-4	1100	18	19	0.292	3.1
ZIRLO	1100	18	20	0.203	2.1

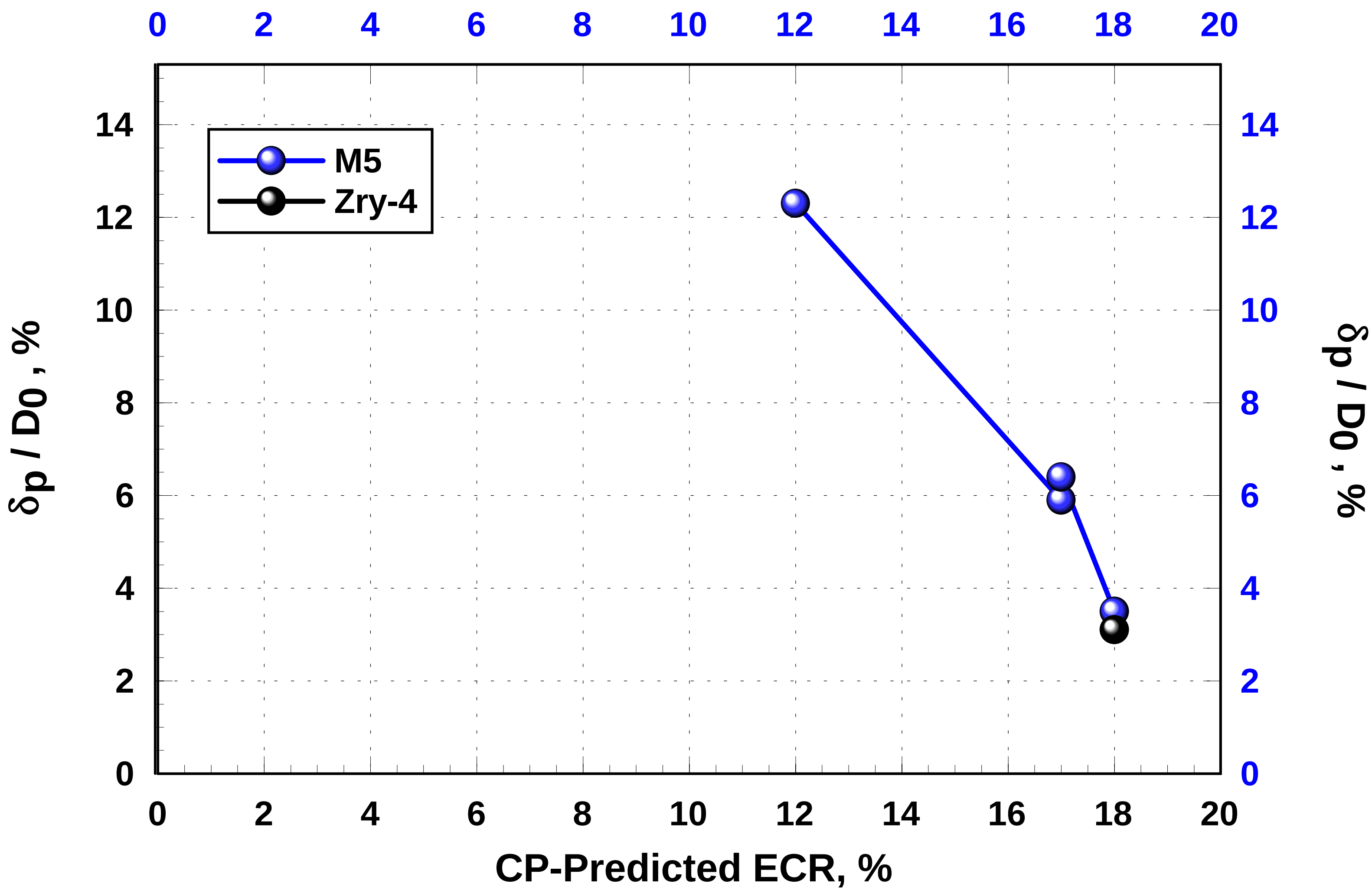
Zry-4 after 19% Measured ECR at 1100°C (No Quench)



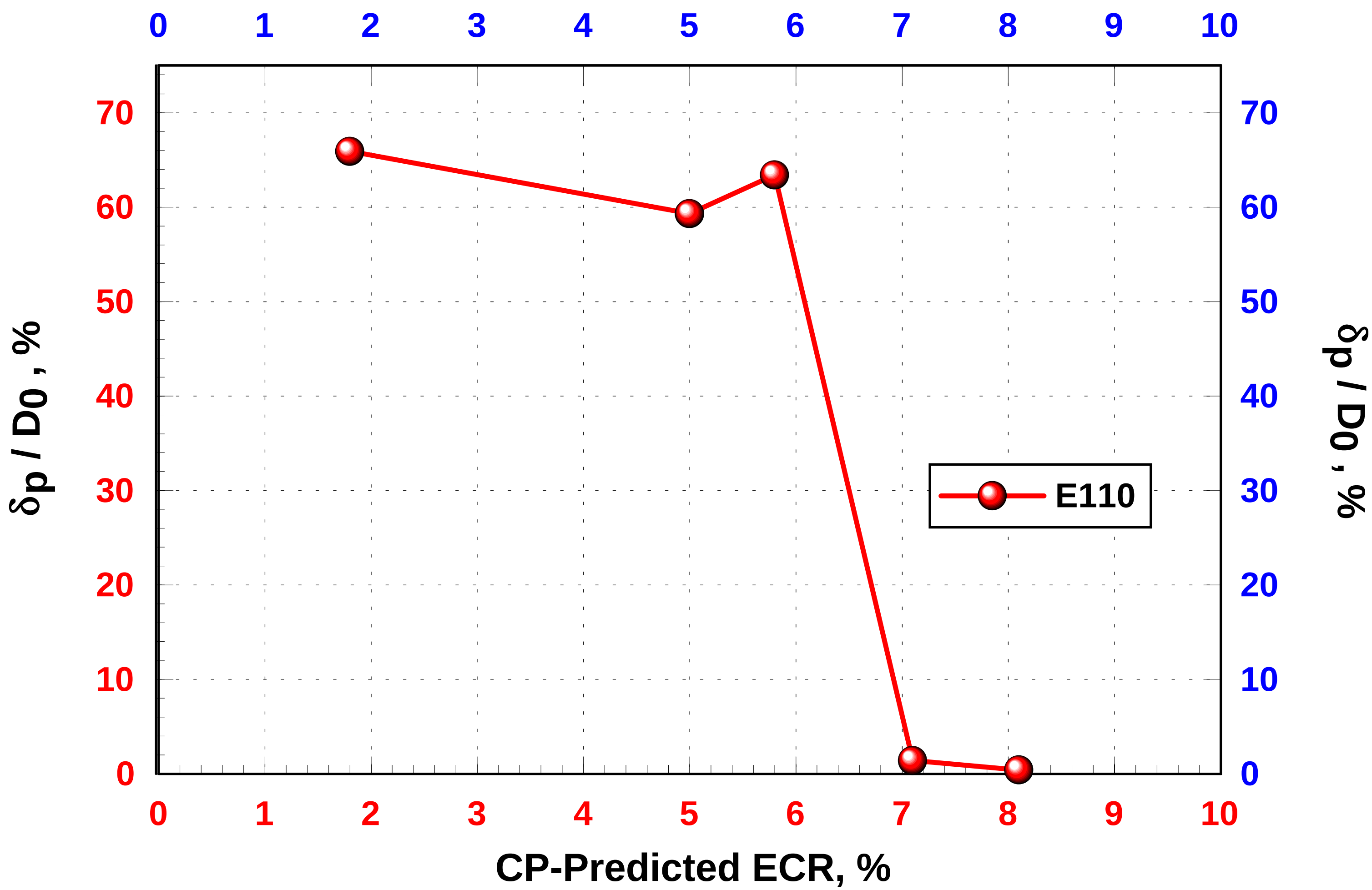
ZIRLO after 20% Measured ECR at 1100°C (No Quench)



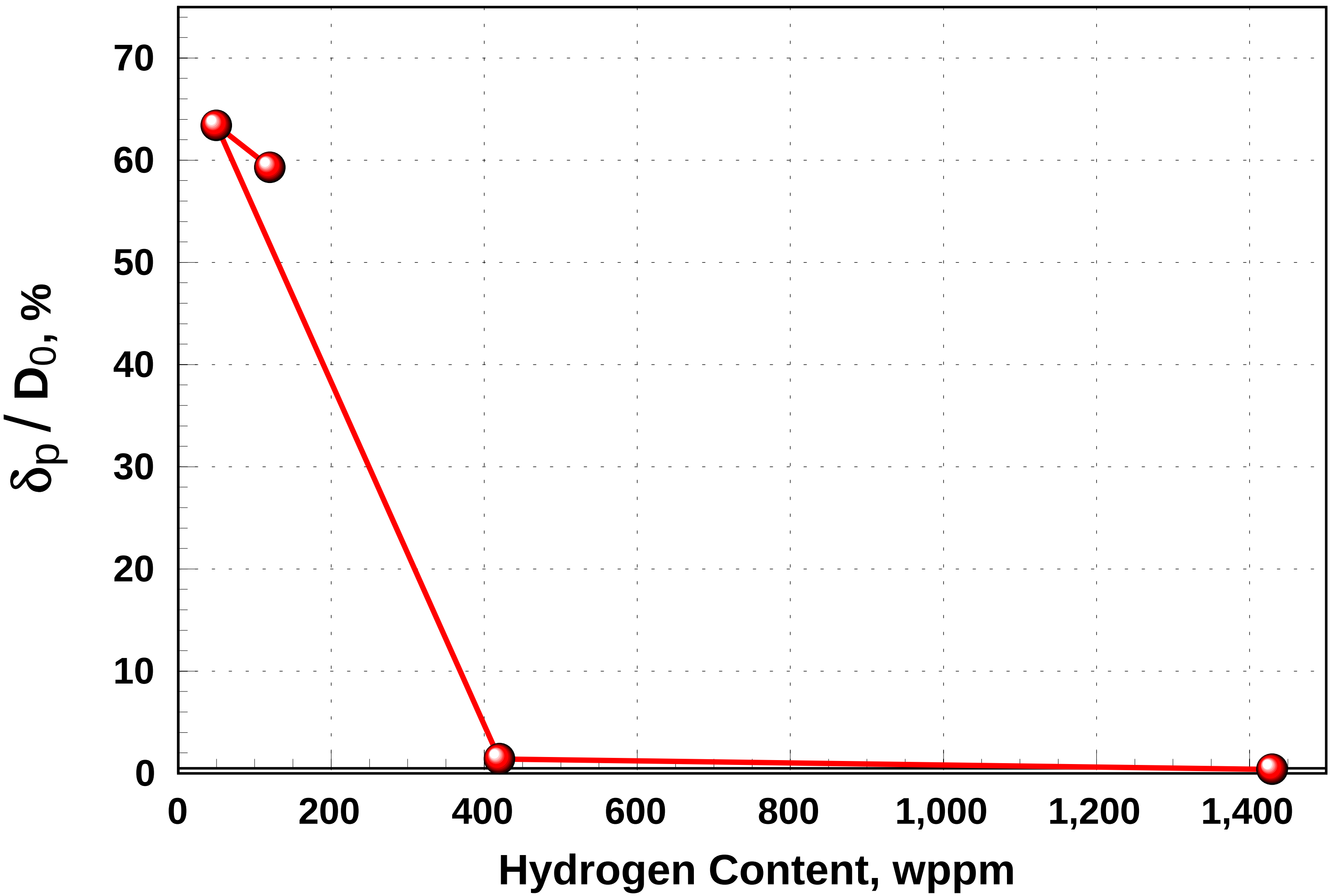
RT Post Quench Ductility for 1100°C Samples



RT Post Quench Ductility: 1000°C E110 Samples



RT Ductility vs. H-Content: 1000°C E110 Samples



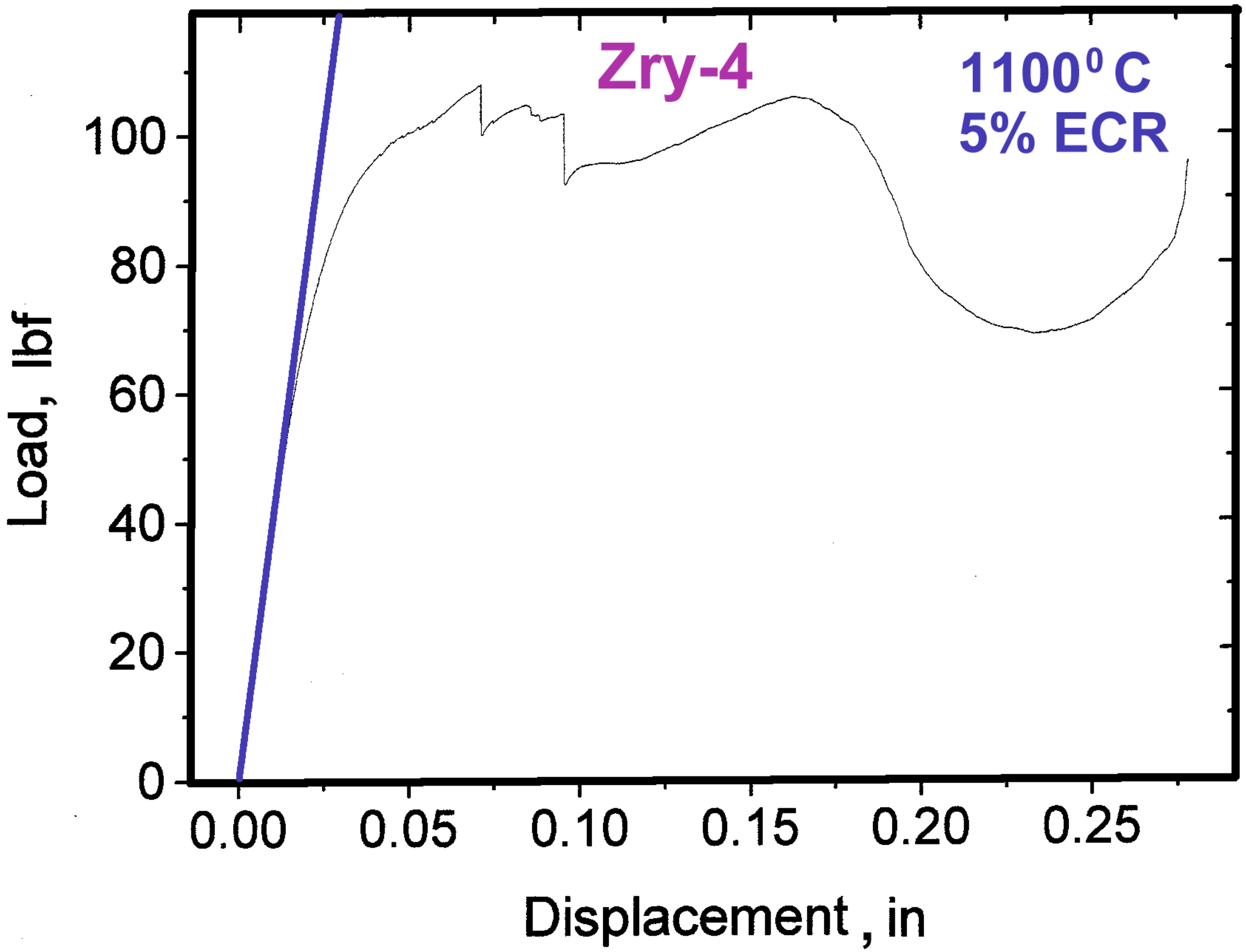
Room-Temperature Ring-Compression Data

- **Tests are in Progress for Zry-4, ZIRLO and M5**
  - 1100°C oxidation/quench and ring-compression tests completed
  - 1000°C oxidation/quench tests have been initiated
  - 1200°C tests will follow 1000°C tests; 1260°C follow 1200°C
- **Results for 1100°C Tests**
  - ZIRLO vs. Zry-4
  - M5 vs. Zry-4

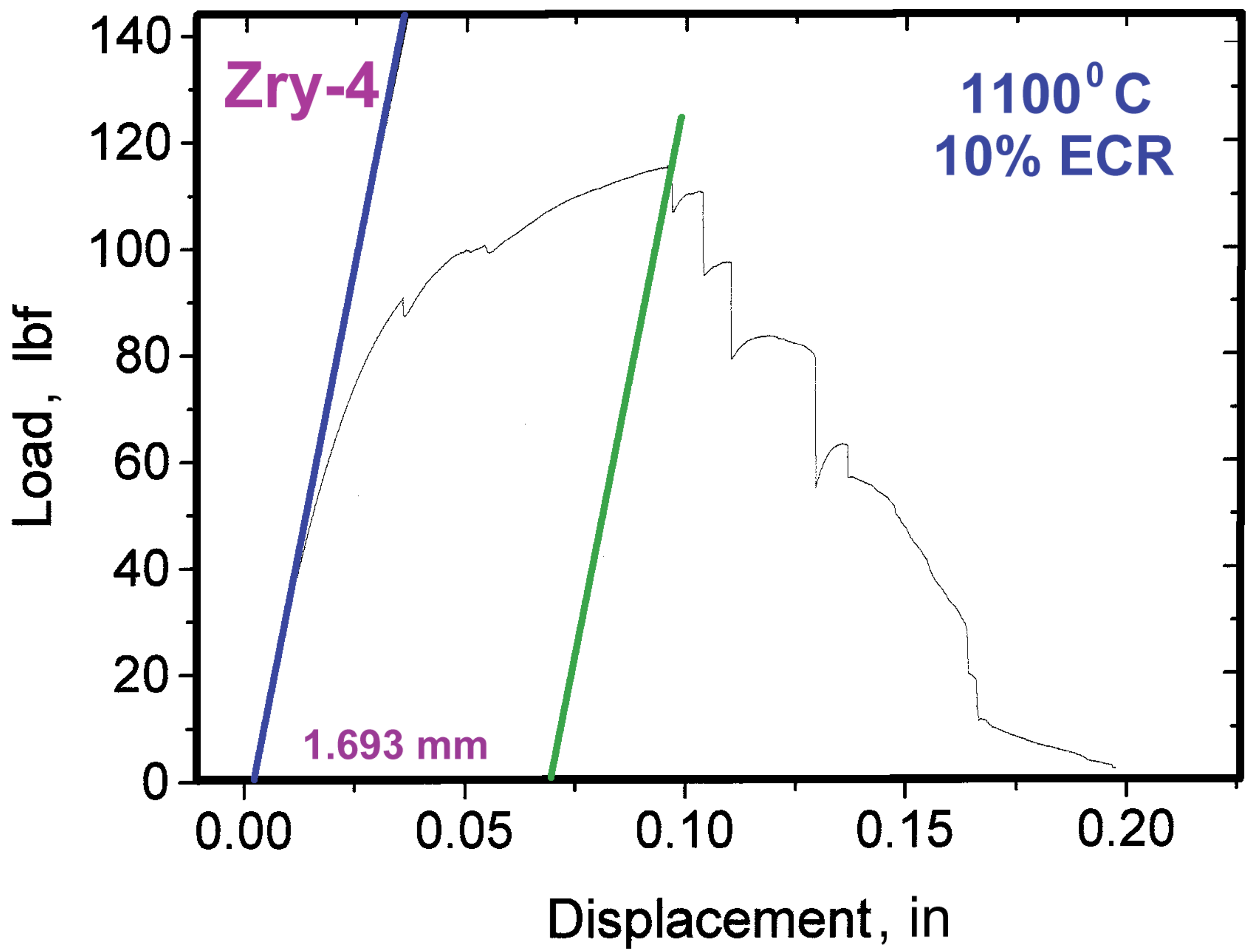
RT Post-QUENCH-Ductility Data for 1100°C Samples

CP ECR, %	Material	Measured ECR, %	δ_p, mm	δ_p/D_o,%
5	Zry-4	6.2	Ductile	Ductile
	ZIRLO	5.9	Ductile	Ductile
	M5	4.7	Ductile	Ductile
10	Zry-4	10.9	1.693	17.8
	ZIRLO	10.9	Ductile	Ductile
	M5	9.2	1.168	12.3
15	Zry-4	16.3	0.476	5.0
	ZIRLO	16.1	0.483	5.1
	M5	13.9	0.709	7.5
17	Zry-4	18.0	0.381 to 0.559	4.0 to 5.9
	ZIRLO	17.9	0.330	3.5
	M5	16.2	0.381	4.0
20	Zry-4	20.3	0.445	4.7
	ZIRLO	21.1	0.318	3.3
	M5	19.2	0.170	1.8

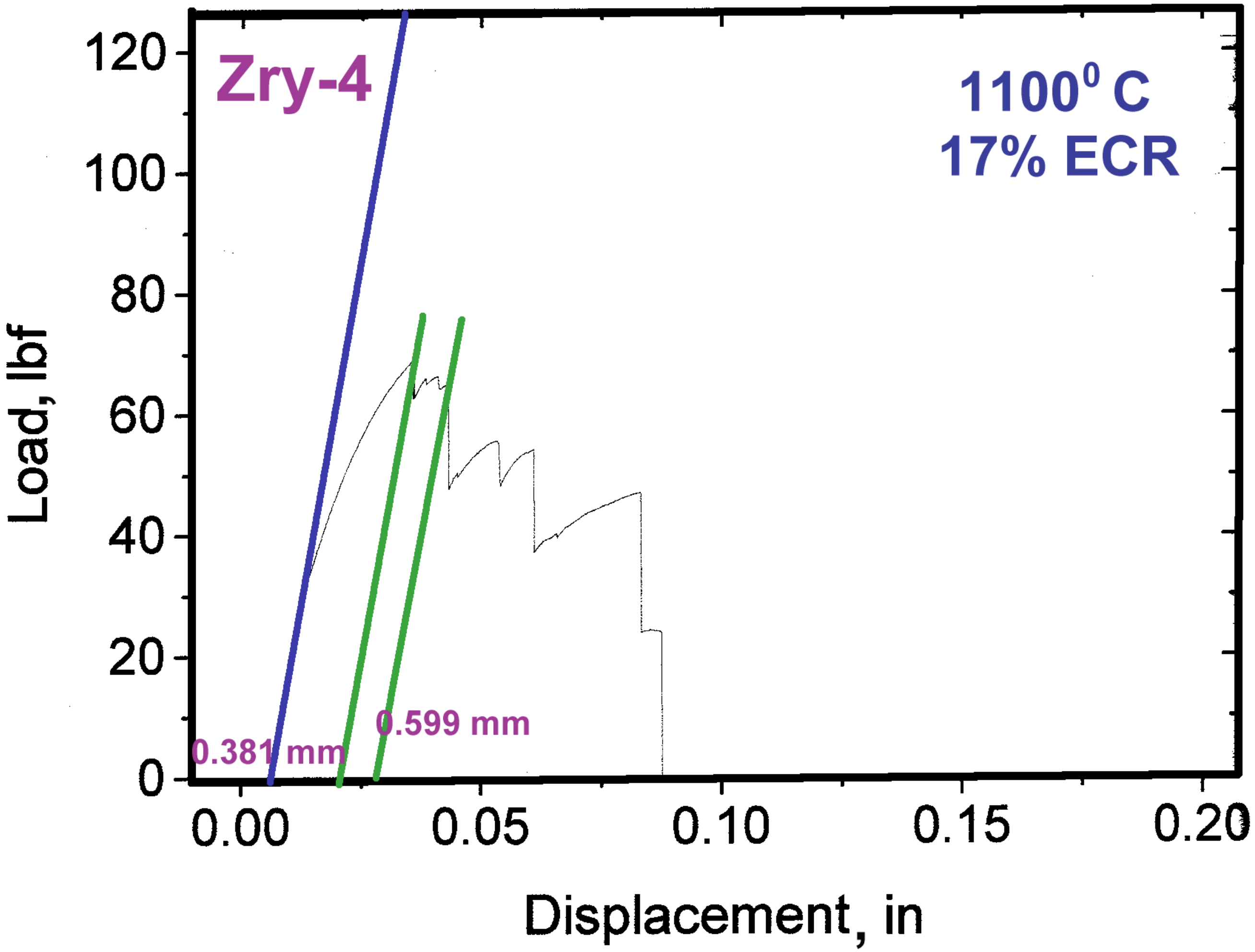
RT Load-Displacement for Zry-4 after 5% ECR at 1100°C



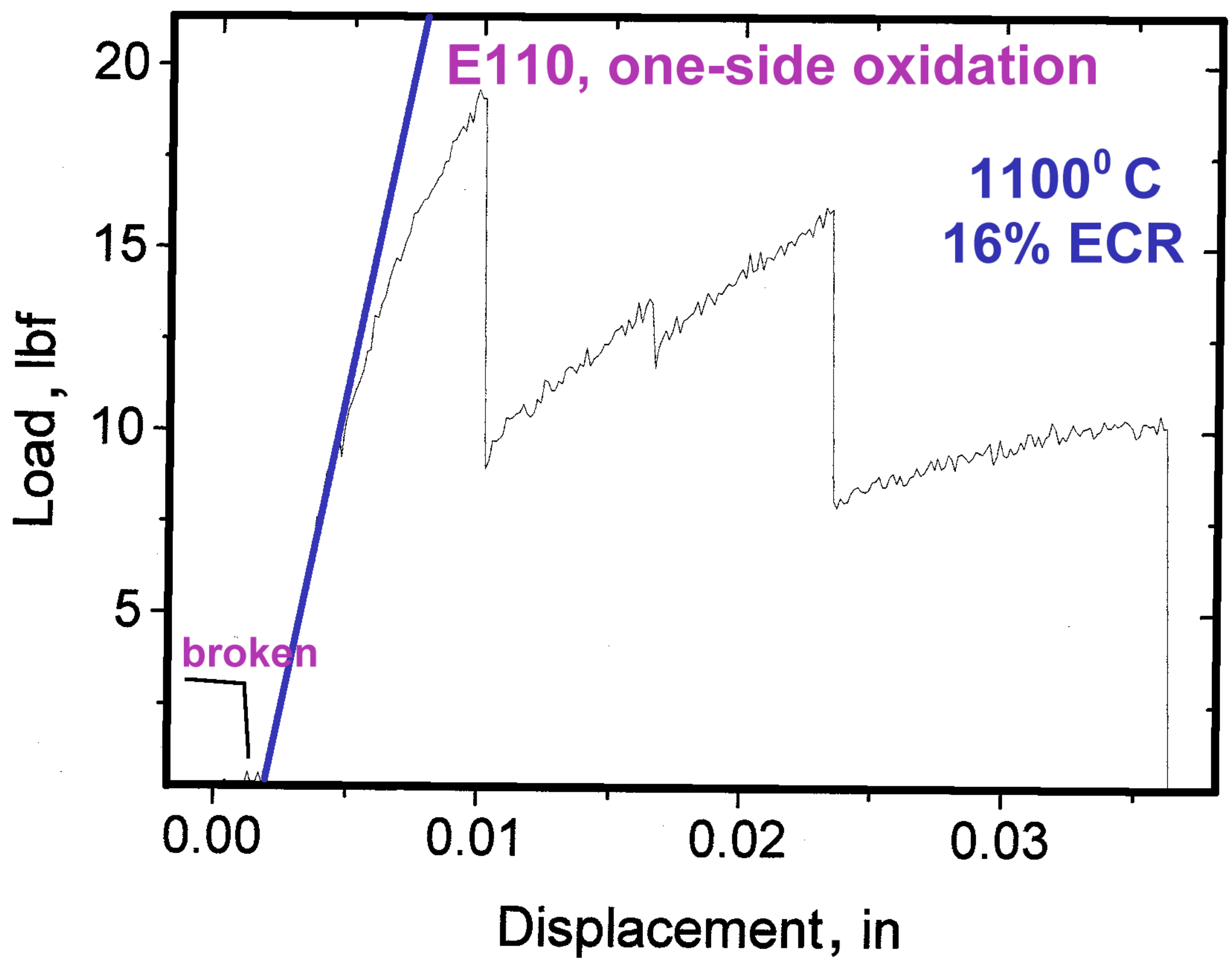
RT Load-Displacement: Zry-4 after 10% ECR at 1100°C



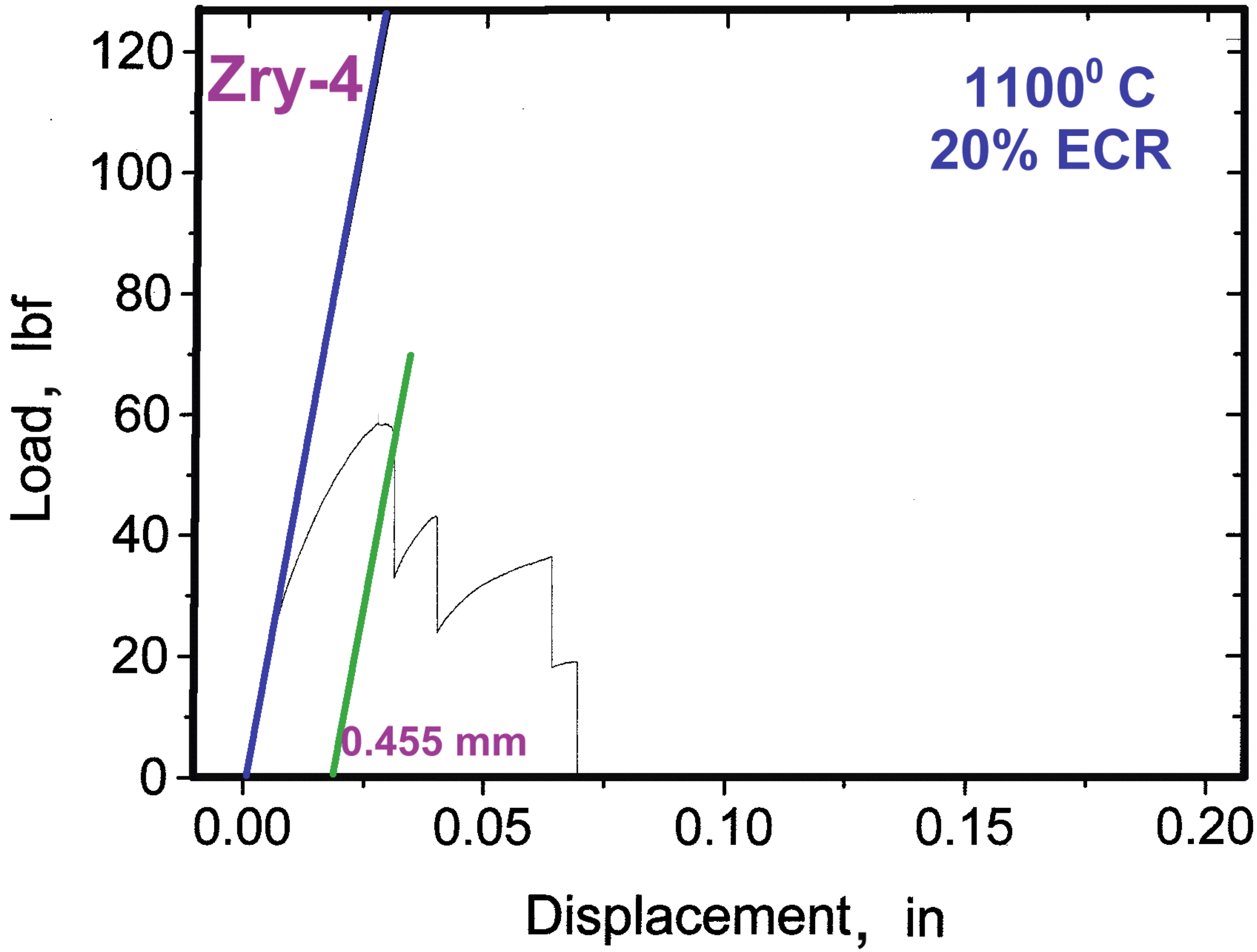
RT Load-Displacement: Zry-4 after 17% ECR at 1100°C



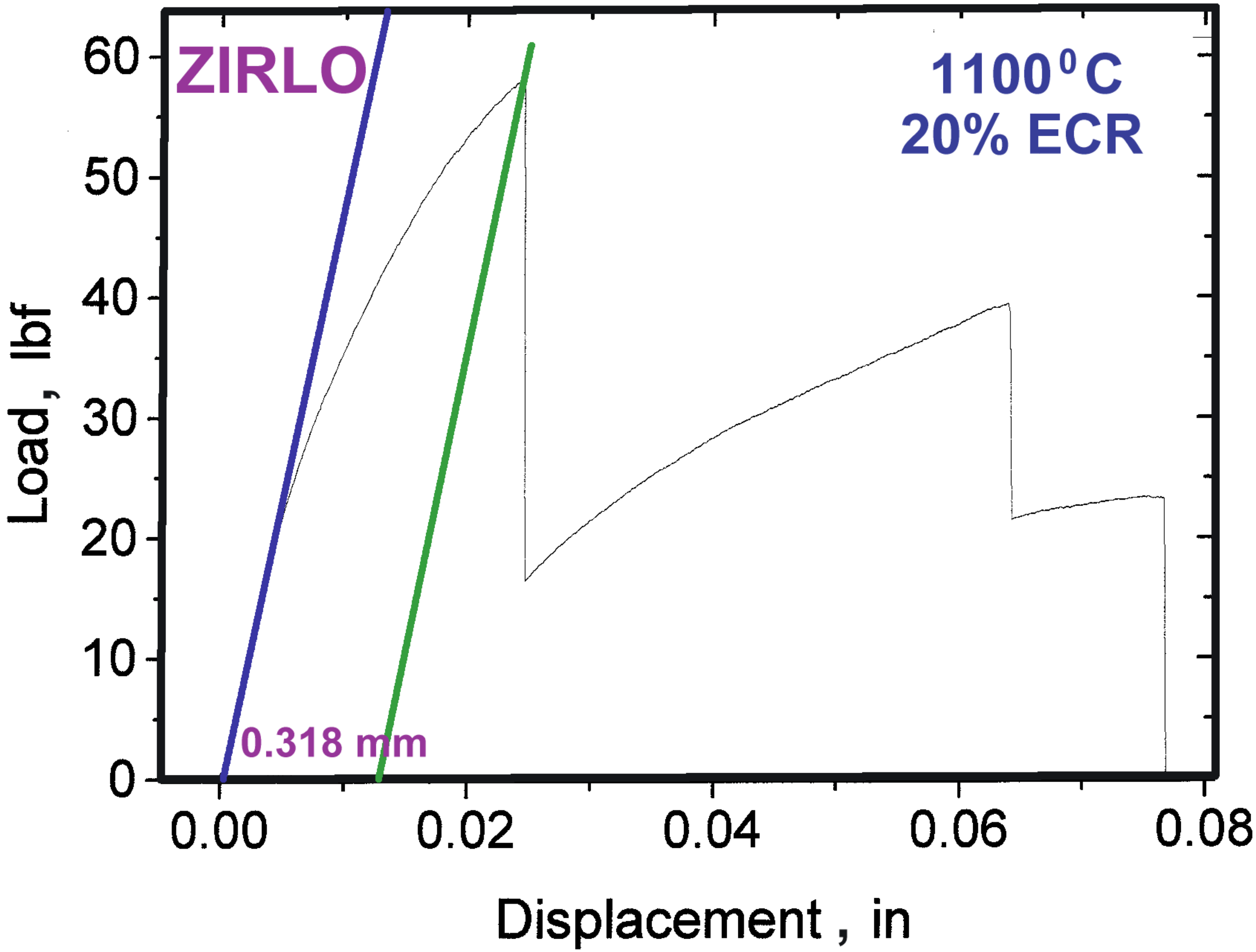
RT Load-Displacement: E110 after 16% ECR at 1100°C



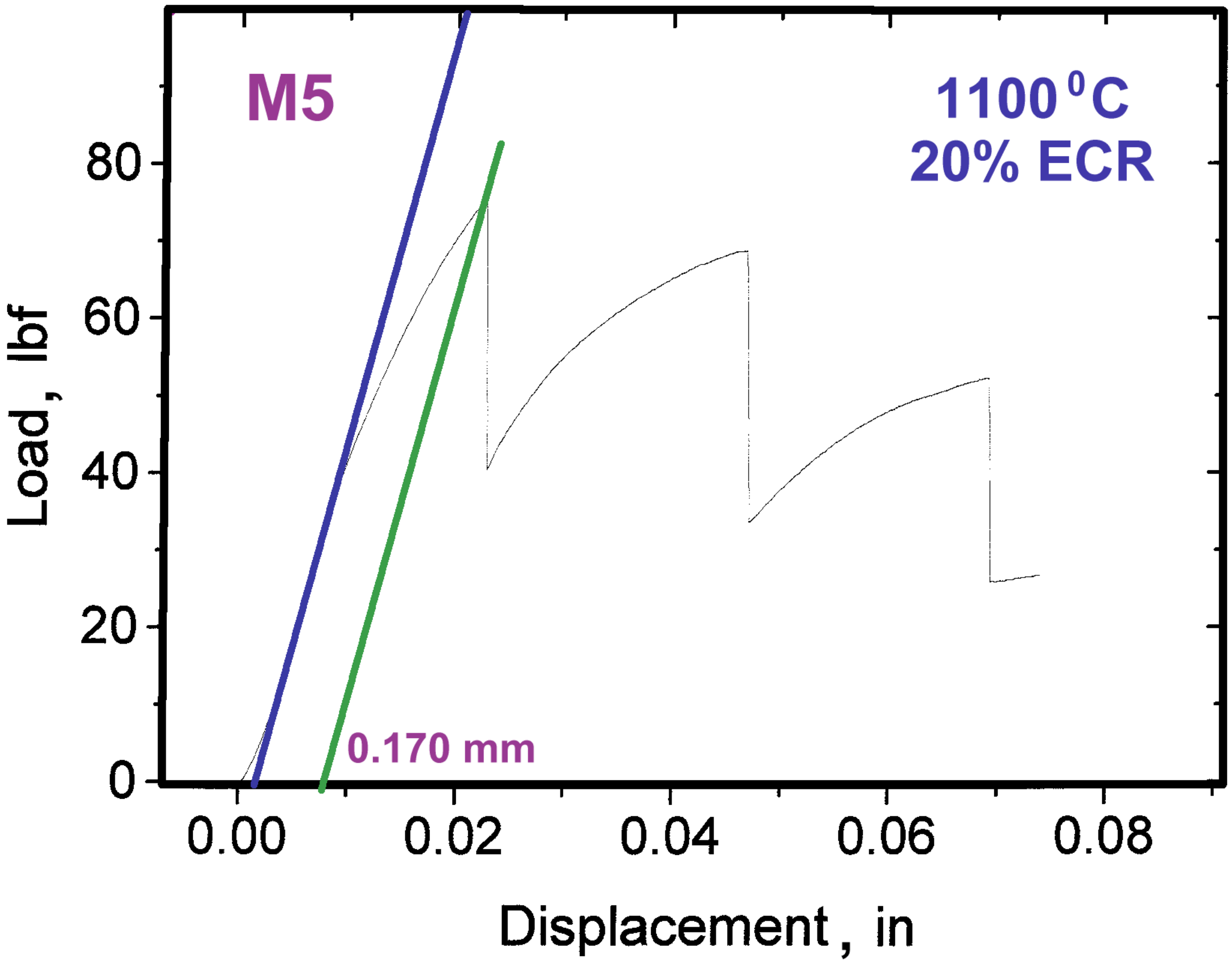
RT Load-Displacement: Zry-4 after 20% ECR at 1100°C



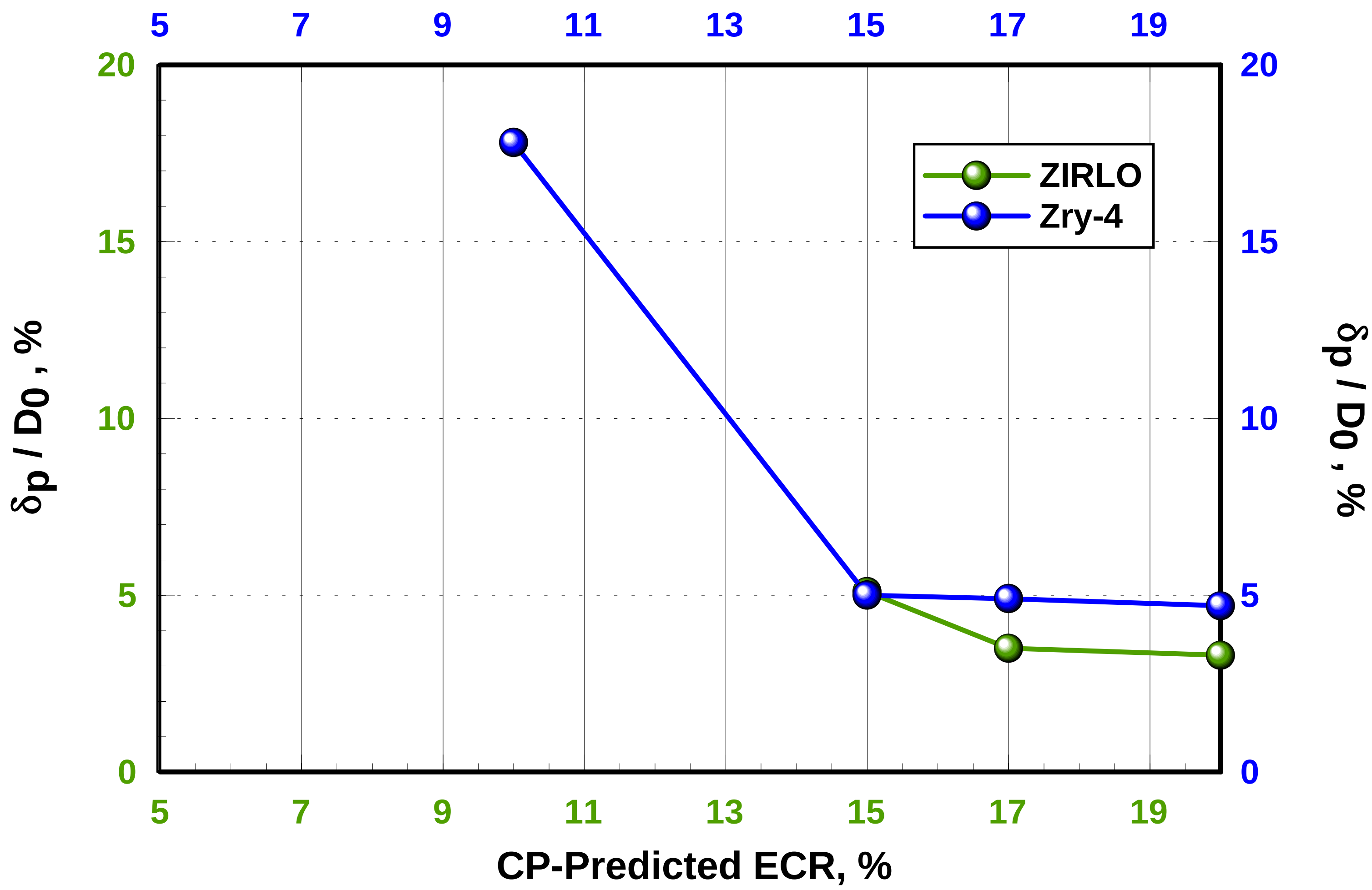
RT Load-Displacement: ZIRLO after 20% ECR at 1100°C



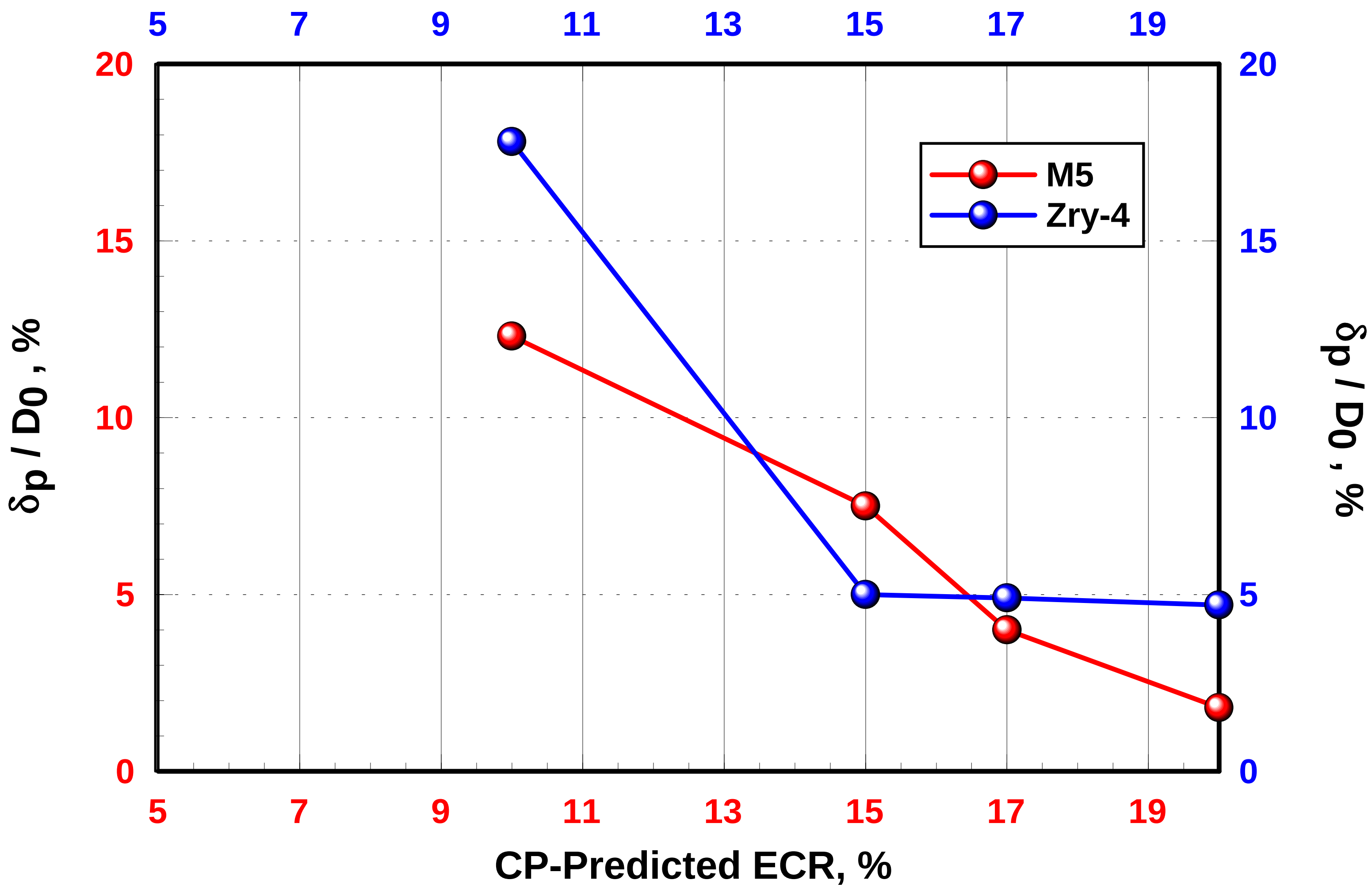
RT Load-Displacement: M5 after 20% ECR at 1100°C



RT Ring-Compression Ductility of ZIRLO vs. Zry-4 Samples Oxidized at 1100°C and Quenched at 800°C



RT Ring-Compression Ductility of M5 vs. Zry-4 Samples Oxidized at 1100°C and Quenched at 800°C



E110 Mystery

- **Instability of Oxide Layer Confirmed at Low Test Times**
  - Alloy is more "challenged" at 1000°C than at 1100°C
    - *1100°C: nodular oxidation → oxygen + hydrogen embrittlement*
    - *1000°C: delamination/spallation → hydrogen embrittlement*
  - Performance at 950°C may be worse than at 1000°C
  - Roughness, grooves, TCs, ends are initiation sites for oxide transition (black to white) and instability: disturbance of compressive stress field
- **Studies of Surface Roughness and Surface Chemistry**
  - Surface polishing significantly improves E110 oxidation performance
  - Etching ($HF+HNO_3+H_2O$), polishing/etching, and etching/polishing

    Etching as-received E110 significantly degrades initial oxide (due to F)
- **Bulk Chemistry, Metallography, SEM, TEM Results**
  - In progress: indication of non uniform distribution of Nb-particles

Summary of Post-Quench-Ductility Program

- **Current Oxidation/Quench Study: As-Received Cladding**
  - Basically oxygen-induced embrittlement of Zry-4, ZIRLO, and M5
  - All 3 alloys retain ductility after 1100°C oxidation to 20% ECR
  - H- and O-induced embrittlement of E110 confirmed at 1000-1100°C
- **LOCA Integral Tests of As-Received Cladding**
  - Oxygen embrittlement within burst region
  - Hydrogen (up to 3500 wppm) embrittlement of balloon-neck region
  - Test will challenge M5 and ZIRLO, as well as Zry-4 and Zry-2
- **Further Study of Hydrogen/Oxygen Embrittlement**
  - Consider testing prehydrided advanced alloys
  - Consider testing high-burnup advanced alloys with H from corrosion
  - Compare prehydrided unirradiated cladding to high-burnup cladding