July 28, 2015

MEMORANDUM TO: Timothy J. McGinty, Director
Division of Safety Systems
Office of Nuclear Reactor Regulation

FROM: Paul M. Clifford, Sr. Technical Advisor ***/RA/***
Division of Safety Systems
Office of Nuclear Reactor Regulation

SUBJECT: AUDIT REPORT: APPLICABILITY OF 50.46C POST QUENCH DUCTILITY ANALYTICAL LIMITS TO WESTINGHOUSE OPTIMIZED ZIRLO™ CLADDING MATERIAL

In a letter dated June 29, 2015, Westinghouse Electric Company (WEC) submitted a comment on Draft Guide (DG)-1263, "Establishing Analytical Limits for Zirconium Based Alloy Cladding." DG-1263 provides an acceptable approach for developing analytical limits for peak clad temperature and time-at-temperature for zirconium-alloy cladding materials corresponding to the ductile-to-brittle transition to demonstrate compliance with the proposed 10 CFR 50.46c Emergency Core Cooling System performance rule. In their comment, WEC requests that Optimized ZIRLO™ material be included as one of the acceptable cladding materials for which Figure 2 of DG-1263 is applicable. The purpose of this audit is to review the post quench ductility (PQD) empirical database supporting the applicability of Optimized ZIRLO.

On July 16, 2015, the U.S. Nuclear Regulatory Commission staff conducted an audit of the WEC ZIRLO® and Optimized ZIRLO PQD experimental procedures and empirical dataset. The audit was held at the WEC offices in Rockville, Md.

Based upon the results of the audit, the staff recommends accepting WEC's proposal to include Optimized ZIRLO as one of the acceptable cladding materials for which Figure 2 of DG-1263 is applicable. The attached audit report provides the basis for the staff's decision.

Attachments:
As stated

CONTACT: P. M. Clifford NRR/DSS,
(301) 415-4043

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AUDIT REPORT:

APPLICABILITY OF 50.46C POST QUENCH DUCTILITY ANALYTICAL LIMITS TO WESTINGHOUSE OPTIMIZED ZIRLO™ CLADDING MATERIAL

Purpose:

In a letter dated June 29, 2015 (Reference 1), Westinghouse Electric Company (WEC) submitted a comment on Draft Guide (DG)-1263, "Establishing Analytical Limits for Zirconium Based Alloy Cladding." DG-1263 provides an acceptable approach for developing analytical limits for peak clad temperature and time-at-temperature for zirconium-alloy cladding materials corresponding to the ductile-to-brittle transition to demonstrate compliance with the proposed 10 CFR 50.46c Emergency Core Cooling System (ECCS) performance rule. In their comment, WEC requests that Optimized ZIRLO™ material be included as one of the acceptable cladding materials for which Figure 2 of DG-1263 is applicable. The purpose of this audit is to review the post quench ductility (PQD) empirical database supporting the applicability of Optimized ZIRLO.

Audit and Participants:

On July 16, 2015, the U.S. Nuclear Regulatory Commission (NRC) staff conducted an audit of the WEC ZIRLO® and Optimized ZIRLO PQD experimental procedures and empirical dataset. The audit was held at the WEC offices in Rockville, Md. The following individuals participated in the audit.

- Paul Clifford, NRC/NRR/DSS (Lead)
- William MacFee, NRC/NRR/DPR
- Michelle Bales, NRC/RES/DSA
- Andrew Mueller, WEC
- David Mitchell, WEC
- Andrew Bowman, WEC

Scope:

During the audit, the NRC performed the following activities:

- Reviewed PQD testing procedures to ensure consistency with DG-1262, and considering anticipated updates planned for RG 1.223.
- Reviewed methods used to interpret measured offset strain and ductile to brittle transition (DBT) to ensure consistency with DG-1262, and considering anticipated updates planned for RG 1.223.
- Reviewed extend of empirical database to ensure consistency with DG-1262, and considering anticipated updates planned for RG 1.223.
- Compared WEC ZIRLO and Optimized ZIRLO PQD database to existing Argonne National Labs (ANL) PQD database.
- Determined applicability of acceptable PQD analytical limits (Figure 2, DG-1263, expected to remain unchanged in RG 1.224) to Optimized ZIRLO.

ENCLOSURE

Discussion:

The NRC high burnup Loss Of Coolant Accident (LOCA) research program conducted at ANL included as-fabricated, hydrogen pre-loaded, and irradiated segments of Zry-2, Zry-4, ZIRLO, and M5® cladding material. One of the principle findings of the research program was that hydrogen absorbed during steady-state water-side corrosion has a significant effect on the rate of cladding embrittlement under high temperature steam oxidation LOCA conditions. This is referred to as hydrogen-enhanced beta layer embrittlement. In addition, the research program identified that alloying chemistry, at least among the cladding materials tested, had only a minor effect on embrittlement. NUREG/CR-6967 (Reference 2) documents the research conducted at ANL.

Based upon the ANL research, Figure 2 of DG-1263 provides acceptable analytical limits for peak clad temperature and time-at-temperature, expressed as a function of cladding hydrogen content. WEC requests that Optimized ZIRLO material be included as one of the acceptable cladding materials for which Figure 2 of DG-1263 is applicable (Reference 1). In support of this request, WEC has completed extensive PQD testing on both ZIRLO and Optimized ZIRLO cladding material. WEC's experimental procedures and protocols were based, in part, on those employed in the ANL research program and the acceptable protocols documented in DG-1262 and DG-1263. In response to public comments, both DG-1262 and DG-1263 were revised and will be issued as RG 1.223, "Determining Post Quench Ductility," and RG 1.224, "Establishing Analytical Limits for Zirconium-Alloy Cladding Material," respectively. As a result of the evolution experimental protocols, those employed by WEC may differ from current standards and these differences need to be understood and dispositioned.

Section C.1.B of RG 1.224 (draft) provides an acceptable methodology for demonstrating consistency with the existing database and analytical limits (i.e., Figure 2) for new alloys. The guidance (draft) states:

> The analytical limit defined in Figure 2 of this RG can be established for zirconium-alloy cladding materials not tested in the NRC's LOCA research program by demonstrating comparable post-quench cladding performance with the analytical limit defined in Figure 2. It is acceptable to demonstrate consistency by showing that data generated with an NRC-approved experimental technique to identify the ductile-to-brittle transition (DBT CP-ECR) for the cladding material is greater than or equal to the analytical limit provided in Figure 2. The DBT CP-ECR should be identified for as-received cladding material and for at least two hydrogen content levels: (1) within 100 wppm of max hydrogen content specified at EOL and (2) within 100 wppm of half max hydrogen content specified at EOL material. For new cladding alloys that meet conditions described below, it is acceptable to test only un-irradiated cladding samples that are pre-charged with hydrogen.

This guidance has evolved. In DG-1263, the magnitude of testing required to demonstrate applicability was larger, requiring testing every 100 wppm hydrogen. In RG 1.224, the requirement for testing irradiated cladding segments is waived if the new alloy is determined to be "similar" in alloying content and manufacturing to those tested at ANL. Table 1 provides the nominal composition of the commercial cladding alloys tested at ANL. Differences between the ZIRLO cladding segments tested at ANL and commercial Optimized ZIRLO cladding consist of (1) a reduction in the Sn content from 1.1% to 0.7% nominal and (2) a change in the final heat treatment from cold worked stress relief (CWSR) to partially recrystallized annealed (pRXA). The remaining manufacturing processes (e.g., source material, reduction, intermediate

annealing) are consistent. Based on the observations from the ANL research program, the reduction in Sn content is expected to have a minor effect, if any, on the rate of embrittlement. Furthermore, due to the LOCA temperature excursion (above $\alpha \rightarrow \alpha+\beta$ phase transition temperature), the final heat treatment is expected to have no impact. As a result, the staff finds that Optimized ZIRLO meets the intent of the draft guidance and no irradiated testing is necessary.

In Reference 1, WEC provides a compilation of the ZIRLO and Optimized ZIRLO PQD database plotted as a function of CP-ECR and cladding corrected hydrogen. Figure 1 shows the extent of the WEC empirical database along with the NRC analytical limit. Plotted are Ring Compression Test (RCT) results for ZIRLO and Optimized ZIRLO cladding in as-fabricated and preloaded with hydrogen to 100, 200, 300, 400 and 600 wppm target levels. Testing was conducted at 1200°C and 1125°C peak clad temperature. Solid symbols denote brittle results and open symbols denote ductile results. Each data point is the average of three ring compression tests with each ring cut from a common specimen of cladding following oxidation and quench. ZIRLO and Optimized ZIRLO cladding specimens were oxidized side by side. Based upon the empirical database, WEC concludes (Reference 1):

> "Examination of the summary data demonstrates that the PQD performance of both ZIRLO and Optimized ZIRLO cladding are nearly identical and that the same ECR limits as a function of hydrogen should be applicable to both alloys."

Demonstrating equivalent PQD performance of a new alloy to one within the ANL database is not a strategy defined in the draft guidance. As a result, the staff's audit is to investigate whether WEC's overall approach, including experimental protocols, extent of investigation, data reduction, and interpretation, met the intent of draft guidance (i.e. sufficient experimental evidence of applicability).

In Reference 1, WEC states that the applied test protocols followed the guidance in DG-1262 with a few exceptions. With respect to deviations, WEC stated:

> "All exceptions from DG-1262 were carefully evaluated for potential impact on the validity of results. Where needed over checks were used to insure accurate and consistent test conditions."

During the audit, the staff asked WEC to explain deviations from DG-1262 and their potential consequences on test results.

- With respect to preparing specimens, WEC described the process used to pre-load the specimens with hydrogen to target concentrations. The process produced good uniformity on hydrogen across a 12 inch tube. Small segments were cut from in between test specimens for hydrogen analysis (for planning oxidation target CP-ECR).
- With respect to conducting steam oxidations, WEC used a relatively large, vertical resistance furnace which is allowed under DG-1262 (differs from ANL test). The uniform heating length is over 6-8 inches, whereas specimens are less than 3 inches. Three specimens were oxidized at once, along with three instrumented specimens. The thermocouples (TCs) provided online temperature measurements to assist the technician and record temperature histories. Rate of insertion, soak time, and rate of withdrawal were timed. DG-1262 required temperature calibration test prior to data generation testing, however WEC utilized an alternative approach in which TCs were present during data generating tests, but on samples adjacent to data generating

specimens. WEC indicated that the large uniform heating zone ensured that the TC's accurately reflected adjacent specimen temperatures. Type K TCs were used instead of Type S TCs recommended by DG-1262. Durability of Type K TCs at high temperature (1200°C) was considered and the TC lifetime was restricted to ensure accurate readings. In addition, WEC used Type S TCs to verify accuracy of the Type K TCs used in their furnace before testing as well as after reaching the WEC imposed restricted lifetime. WEC indicated that Type K TCs tend to drift low if beyond high temperature lifetime. These low reading would promote higher furnace temperatures and more conservative test results. WEC did not observe significant errors in any of the verification checks conducted at the WEC imposed restricted lifetime and therefore WEC was confident they met the heating rates, target PCT, and cooling rates prescribed in DG-1262. After reaching 800°C, specimens were dropped into cool water (which achieves a rapid quench).

- With respect to conducting ring compression tests, WEC cut rings to the specified size, measured point of minimum wall thickness and oriented on the test rig in accordance with DG. Ends of specimens were disregarded in accordance with DG. The loading rate was within the band allowable by DG-1262. WEC stated that there were no deviations from the DG-1262 guidance with respect to ring compression testing.
- With respect to data reduction and DBT, WEC measured hydrogen from 2 rings from each specimen. DG-1262 did not address hydrogen variability. RG 1.223 does recommend hydrogen measurements of every ring. Based on the uniform hydrogen and minimal span in data, WEC believes testing all rings is unnecessary. In accordance with DG-1262, each specimen was cut into multiple rings with the ends discharged. Offset strain was measured for each RCT sample and averaged. The hydrogen content was measured, corrected for weight gain (growth of oxide layer), and averaged. The average offset strain and corrected, average hydrogen content were then used, along with the DG-1262 ductility criterion below, to assess the DBT.

$$\text{Average Measured Offset Strain} \geq 1.41 + 0.1082 * \text{CP-ECR}$$

Based upon the consistency with DG-1262 and justified exceptions, the staff finds the PQD experimental protocols and resulting empirical database acceptable.

The extent of the WEC ZIRLO and Optimized ZIRLO PQD empirical database, shown below, is depicted in Figure 1. WEC has followed the data requirement guidance in DG-1263, which was subsequently reduced in RG 1.224 (draft). The magnitude of Optimized ZIRLO data (i.e., number of tests at each hydrogen level) exceeds the data requirements in current draft guidance and is therefore acceptable.

PQD Test	Number of RCT Tests
Optimized ZIRLO at 1200°C	135
Optimized ZIRLO at 1125°C	51
ZIRLO at 1200°C	108
ZIRLO at 1125°C	51

Figure 2 depicts the ZIRLO PQD RCT data from the ANL research program and from the WEC database. Examination of that data reveals good agreement at 300 wppm where common data exists. Note that the ANL data is from irradiated cladding segments, whereas the WEC data is from unirradiated tubing pre-loaded with hydrogen. This comparison provides further assurance that the testing procedures did not introduce a bias in the results nor that testing of irradiated samples were necessary.

In their comment, WEC requests that Optimized ZIRLO material be included as one of the acceptable cladding materials for which Figure 2 of DG-1263 is applicable. It is important to note that Figure 2 of DG-1263 is divided into two distinct regions. Below 400 wppm hydrogen, the analytical limit is applicable up to 1204°C (2200°F). Above 400 wppm hydrogen, the analytical limit is only applicable up to 1121°C (2050°F). So, in order to assess the applicability of Figure 2, we must divide the WEC database.

Figure 3a shows the WEC ZIRLO and Optimized ZIRLO PQD RCT database for tests conducted at 1200°C along with the original ANL database. Examination of this figure up to 400 wppm hydrogen reveals the following: (1) the performance of ZIRLO and Optimized ZIRLO is nearly identical, (2) reasonable agreement between ANL and WEC data, and (3) reasonable agreement between DBT as a function of cladding hydrogen for WEC database and analytical limit. Based upon this comparison, the staff finds the Figure 2 (DG-1262) analytical limit applicable to Optimized ZIRLO up to 400 wppm hydrogen.

PQD RCT tests conducted at approximately 100 wppm hydrogen do not exhibit the same definitive DBT as test results at higher hydrogen concentration. Examination of Figure 3a reveals that this is true for both the ANL database (irradiated M5® specimens) and the WEC database (hydrogen pre-loaded ZIRLO and Optimized ZIRLO specimens). Ductile and brittle specimens appear on both sides of the analytical limit. Since the acceptable analytical limit was based on a fit of the data, as opposed to a lower bound, this common occurrence of both ductile and brittle test results above and below the line are judged acceptable.

Figure 3b shows the WEC ZIRLO and Optimized ZIRLO PQD RCT database for tests conducted at 1125°C along with the original ANL database. Examination of this figure beyond 400 wppm hydrogen reveals the following: (1) the performance of ZIRLO and Optimized ZIRLO is nearly identical, (2) reasonable agreement between ANL and WEC data, and (3) DBT as a function of cladding hydrogen for WEC database is conservatively higher than analytical limit. Based upon this comparison, the staff finds the Figure 2 (DG-1262) analytical limit applicable to Optimized ZIRLO beyond 400 wppm hydrogen.

Conclusions:

In a letter dated June 29, 2015 (Reference 1), WEC submitted a comment on DG-1263, which provides an acceptable approach for developing analytical limits for peak clad temperature and time-at-temperature for zirconium-alloy cladding materials corresponding to the ductile-to-brittle transition to demonstrate compliance with the proposed 10 CFR 50.46c ECCS performance rule. In their comment, WEC requests that Optimized ZIRLO material be included as one of the acceptable cladding materials for which Figure 2 of DG-1263 is applicable.

On July 16, 2015, the NRC staff conducted an audit of the WEC ZIRLO and Optimized ZIRLO PQD experimental procedures and empirical dataset. The audit was held at the WEC offices in Rockville, Md. The purpose of this audit was to review the PQD empirical database supporting the applicability of Optimized ZIRLO.

Based upon the information described above, the staff finds that Figure 2 of DG-1263 is applicable to Optimized ZIRLO cladding material. As a result, NRR/DSS recommends updating the draft RG to reflect this finding.

References:

1. Westinghouse Letter LTR-NRC-15-58, "Submittal of Comment for Docket ID NRC-2012-0043 on Draft Regulatory Guide DG-1263, 'Establishing Analytical Limits for Zirconium Based Alloy Cladding'," June 29, 2015.

2. NUREG/CR-6967, "Cladding Embrittlement During Postulated Loss-of-Coolant Accidents," Argonne National Laboratory, July 2008.

Table 1: Nominal Composition of Commercial Cladding Alloys Tested at ANL
(Source: Table 1 of NUREG/CR-6967, Reference 2)

Element	Zircaloy-2[a]	Zircaloy-4[a]	ZIRLO[b,c]	M5[d]	E110[e]
Sn, wt.%	1.45	1.45	1.1	--	--
Nb, wt.%	--	--	1.1	1.0	1.0
Fe, wt.%	0.14	0.21	0.1	0.038	0.009
Cr, wt.%	0.10	0.10	--	--	--
Ni, wt.%	0.06	--	--	--	--
O, wt.%[f]	0.125	0.125	0.120	0.135	0.06
Zr	Balance	Balance	Balance	Balance	Balance

[a]ASTM B811 [3].
[b]R. Comstock et al. [4].
[c]W. Leech [5].
[d]J-P. Mardon et al. [6].
[e]P. V. Shebaldov et al [7].
[f]Oxygen is considered an alloying element in these alloys.

Figure 1: WEC PQD RCT Results for ZIRLO and Optimized ZIRLO
(Source: Figure 1 of Reference 1)

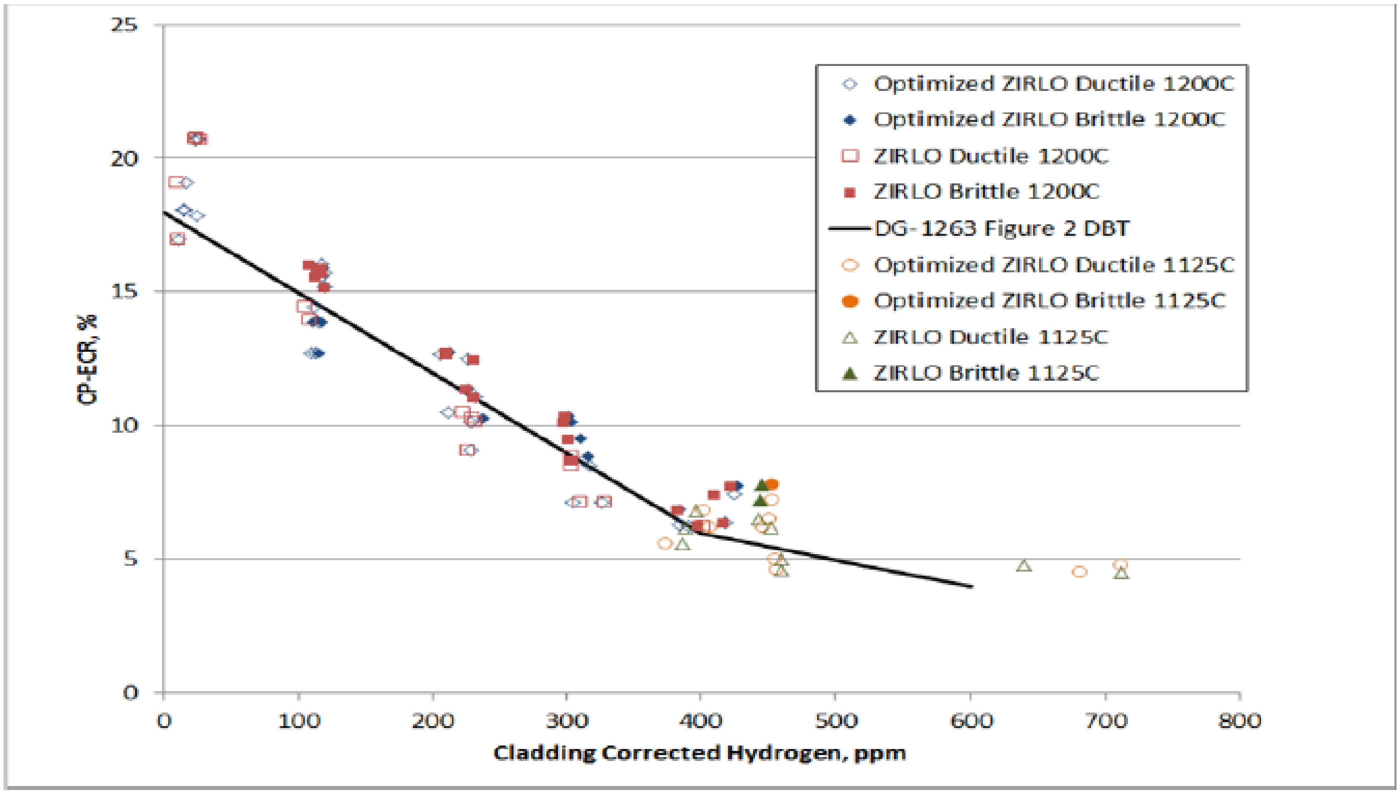

Figure 2: Combined ANL and WEC PQD RCT Results - ZIRLO

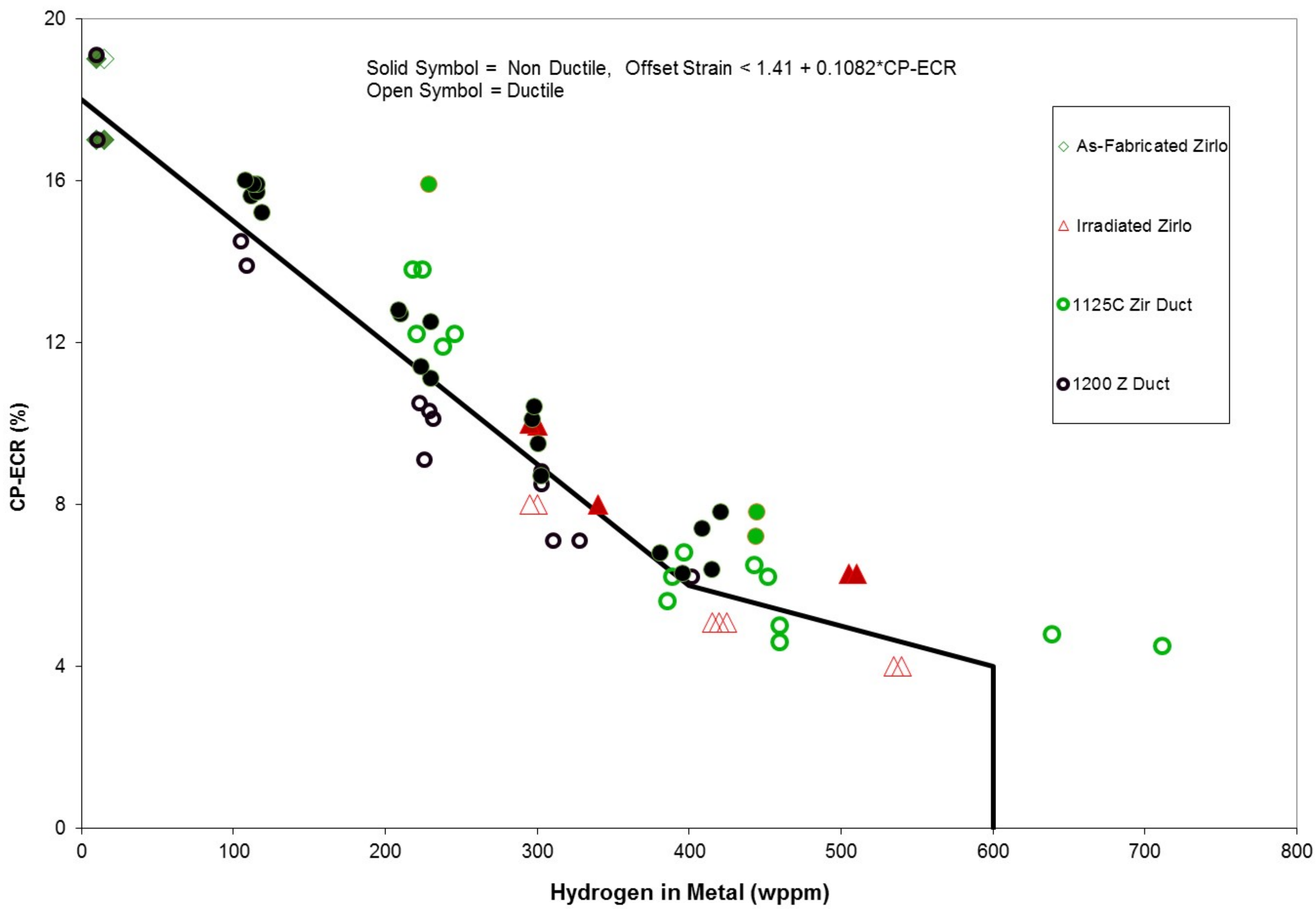

Figure 3a: Combined ANL and WEC PQD RCT Results – All Alloys
(WEC Database at 1200°C)

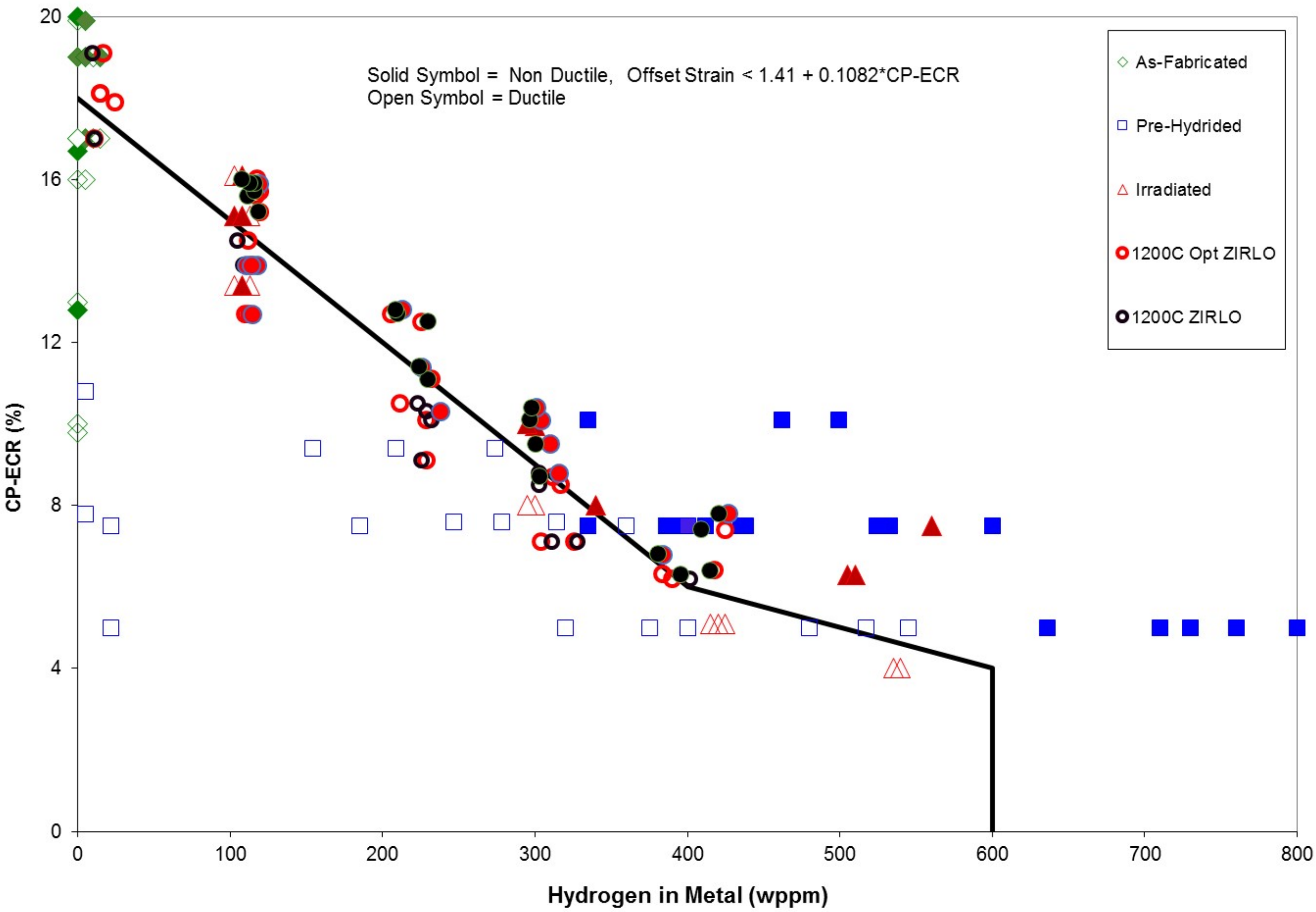

Figure 3b: Combined ANL and WEC PQD RCT Results – All Alloys
(WEC Database at 1125°C)