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Subject: Westinghouse Results from Study on Impact of Specimen Preparation on Breakaway Oxidation (Non-Proprietary)

Per your request, Westinghouse is providing to NRC the Westinghouse results from the study on the impact of specimen preparation on breakaway oxidation. Breakaway oxidation testing at Argonne National Laboratory (ANL) and Westinghouse identified a difference in the minimum time to reach breakaway for ZIRLO[™] cladding with ANL reporting a time of 3000 seconds and Westinghouse reporting a minimum time greater than 5400 seconds. This difference prompted additional testing at ANL and the Westinghouse Science and Technology Department (STD) to determine the source of the discrepancy. As a first step, tests were performed at the two facilities to compare the impact of specimen preparation and cleaning on the test results.

The samples were tested in the high-temperature steam oxidation facility at Westinghouse STD with ten tests performed over the temperature range of 960 °C to 1020 °C for nominal exposure times of 4400 seconds. Visual comparison of the specimens revealed the following:

- Specimens from the common cladding lot that were prepared by ANL and STD were comparable in appearance for all tests.
- Heat-up rate is a significant variable for establishing the time to reach breakaway oxidation. The high ramp rates in the ANL tests and lower ramp rates in the STD tests support the observation of shorter times to breakaway oxidation from tests performed at ANL.

The above results identify ramp rate as a significant test parameter that can impact the time to breakaway oxidation, with higher ramp rates correlating with shorter times to breakaway oxidation. An effort to establish an appropriate ramp rate for breakaway oxidation testing is being initiated at Westinghouse.

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Very truly yours,

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Impact of Specimen Preparation on Breakaway Oxidation (Non-Proprietary)

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Impact of Specimen Preparation on Breakaway Oxidation

Executive Summary

The onset of breakaway oxidation of zirconium alloy cladding has been identified as a potential mechanism for loss of cladding ductility during a small break loss of coolant accident (LOCA) (Ref. 1). Breakaway oxidation testing at Argonne National Laboratory (ANL) and Westinghouse identified a difference in the minimum time to reach breakaway for ZIRLOTM cladding with ANL reporting a time of 3000 seconds (Ref. 1) and Westinghouse reporting a minimum time greater than 5400 seconds (Ref. 2). This difference prompted additional testing at ANL and the Westinghouse Science and Technology Department (STD) to determine the source of the discrepancy. As a first step, tests were performed at the two facilities to compare the impact of specimen preparation and cleaning on the test results.

Two sets of specimens from a common ZIRLO cladding lot were prepared for testing with one set prepared at ANL and the second set prepared at STD. In addition, ZIRLO specimens were also prepared at STD from a second ZIRLO lot that was used for the prior breakaway tests at Westinghouse. The three specimen types were simultaneously tested in the high-temperature steam oxidation facility at STD with ten tests performed over the temperature range of 960 °C to 1020 °C for nominal exposure times of 4400 seconds.

Breakaway oxidation was assessed by the visual appearance of the specimens with black oxide corresponding to pre-breakaway and non-black (e.g., light green) oxide indicative of breakaway oxidation. Visual comparison of the specimens revealed the following:

- The specimens from the common cladding lot that were prepared by ANL and STD were comparable in appearance for all tests. This result indicates that any differences in the preparation of the specimens at ANL and STD were not significant contributors to the onset of breakaway oxidation.
- Heat-up rate is a significant variable for establishing the time to reach breakaway oxidation as the extent of breakaway oxidation was greatest for samples that were rapidly heated to temperature. The high ramp rates in the ANL tests (Ref. 1) and lower ramp rates in the STD tests (Ref. 2) support the observation of shorter times to breakaway oxidation from tests performed at ANL.

The above results identify ramp rate as a significant test parameter that can impact the time to breakaway oxidation, with higher ramp rates correlating with shorter times to breakaway oxidation. An effort to establish an appropriate ramp rate for breakaway oxidation testing is being initiated at Westinghouse.

Background

The onset of breakaway oxidation of zirconium alloy cladding has been identified as a potential mechanism for loss of cladding ductility during a small break loss of coolant accident (LOCA) (Ref. 1). The ductility loss is attributed to the rapid pickup of hydrogen associated with breakaway oxidation. Testing performed at Westinghouse identified the minimum time to breakaway for ZIRLO[™] cladding to be greater than 5400 seconds (Ref. 2). The time was significantly longer than the 3000 seconds reported by Argonne National Laboratory (ANL) (Ref. 1). This difference in breakaway time prompted a meeting on May 13, 2008 between ANL (Dr. M. Billone and Dr. Y. Yan), Westinghouse (Dr. R. Comstock, Dr. R. Baranwal, Mr. R. Buechel, and Mr. A. Atwood), and a representative (Mr. H. Scott) from the NRC. The purpose of the meeting was to review experimental procedures at both facilities and to identify potential reasons for the difference in the minimum time to breakaway oxidation.

One action from the meeting was to evaluate the impact of specimen preparation and cleaning on the test results. Specimens cleaned at ANL would be tested at Westinghouse and specimens cleaned at Westinghouse would be tested at ANL. This memo documents the breakaway oxidation tests that were performed at Westinghouse.

Experimental

Test facility

The high-temperature steam test facility at Westinghouse that was used for the breakaway oxidation tests reported in Ref. 2 was modified during the fall of 2008. The modifications were performed to provide increased flexibility in the heating and cooling of test specimens. A schematic drawing of the modified facility is shown in Figure 1. The primary modifications included the following:

- The Alloy 690 tube that served as the specimen steam chamber was replaced with a quartz tube. The inside diameter of the Alloy 690 tube was 0.675 inch compared to 2.0 inches for the quartz tube. The larger cross-section of the quartz tube required an increase in the flow rate of the water. A typical water flow rate for production of steam in the quartz tube was about 6.5 g/minute.
- A significant increase in the specimen heating rate was achieved by heating the quartz tube steam chamber to the target test temperature prior to insertion of the test specimens. After establishing steam flow through the quartz tube and stabilizing the six-inch long uniform temperature zone, specimens were dropped into the hot zone. This allowed specimens to reach temperature in about one minute. The typical time to reach temperature in the prior facility was about five minutes.

All breakaway oxidation tests for comparing the behavior of ANL and STD prepared specimens were performed in the modified test facility.

Test Specimens

Table 1 identifies the material that was exchanged between Westinghouse and ANL. ANL sent STD ten one-inch ZIRLO cladding specimens that were prepared at ANL. These specimens were from ZIRLO cladding provided to ANL by Westinghouse Nuclear Fuel (Columbia). The material was from lot

S69-8568Z and was identified by ANL as 2008 ZIRLO. None of the ten specimens were engraved as sample identification was provided by placing each specimen in a labeled plastic bottle.

In addition, ANL sent a length of cladding from the same material (2008 ZIRLO) to STD. STD prepared ten one-inch long test specimens from the cladding to provide comparison of specimen cleaning from a common cladding lot. These specimens were sectioned and prepared for testing according to procedures described below.

In exchange, STD prepared twenty one-inch long test specimens from a single cladding tube from ZIRLO lot S73-2102Z. Ten of the twenty specimens were randomly selected and sent to ANL (Ref. 3) for testing while the remaining ten were retained for testing at STD. In addition, a twelve-inch length from the same cladding tube (lot S73-2102Z) was sent to ANL (Ref. 4) so they could prepare additional specimens according to their procedure. Breakaway oxidation tests were previously performed at STD on specimens from this lot with the results reported in Ref. 2.

Sample Preparation at STD

Test samples were sectioned from cladding to nominal lengths of one inch using an abrasive cutoff wheel. The sample ends were prepared by deburring and grinding on 600 grit silicon carbide paper. Guidance for the specimen cleaning was obtained from the recommended practice for preparing zirconium alloy specimens for autoclave testing (Ref. 5). The steps included the following: ultrasonic cleaning in acetone, ultrasonic cleaning in an Alconox solution, and extensive rinsing in flowing deionized water followed by hot deionized water. The samples were cooled to room temperature in deionized water and then dipped in alcohol and blow dried. Specimens from the two lots (2008 ZIRLO and S73-2102Z) were prepared and cleaned at different times to maintain lot identity. The samples were placed in plastic boxes with prelabeled slots to maintain the identity of individual specimens. Following cleaning, specimens were handled only with tweezers and gloves.

High Temperature Steam Oxidation

The high-temperature steam oxidation tests were performed in the modified test facility described above. Each test contained the three specimens listed in Table 1. These included one specimen prepared at ANL from 2008 ZIRLO and two specimens prepared at STD (2008 ZIRLO and lot S73-2102Z). Prior to testing, each specimen was weighed and measured to obtain dimensions for the calculation of the surface area.

Oxidation runs were performed as described below:

- The specimens were slid onto the thermowell and suspended in the steam chamber above the hot zone of the furnace.
- The bottom end of the quartz tube was placed in water to 'seal' the end from ingress of air. An argon purge of the quartz tube was started.
- Water flow through copper tubing wrapped around a cartridge heater was initiated for the production of steam. The bypass valve was opened to vent the steam to air.

- The furnace was heated to the specified temperature. After the furnace reached temperature, the argon purge was stopped and the bypass valve was turned to direct the steam into the steam chamber.
- After reaching a stable test temperature, the specimens were dropped into the hot zone of the furnace. The hold time was started when the temperature was within 5 °C of the target temperature. [Note: ANL typically measured time from the beginning of the temperature ramp from 300 °C (Ref. 1).]
- After holding at temperature for the specified time, the furnace was turned off and the clamshell furnace was opened to cool the specimens.
- After cooling to room temperature, the specimens were removed from the quartz tube and weighed.

Thermocouples placed inside the thermowell were used to record temperature at ten-second intervals for each LOCA test. Plots of the temperature histories are shown in Figure 2. Time at t = 0 seconds corresponded to the samples being dropped into the hot zone of the furnace. This can be seen by the sudden drop in the temperature due to the insertion of cold specimens. However, the temperature quickly rose due to the low mass of the tubing. For all tests, the furnace temperature was set below the target temperature as the temperature overshot during sample heat-up due the exothermic oxidation reaction. The controller setpoints were then adjusted to maintain a constant temperature or to raise the temperature to the required test temperature.

Table 2 summarizes the details of the thermal history for each test. Four of the tests (LOCA 117, LOCA 118, LOCA 119, and LOCA 131) were heated directly to the target temperature. The time for the thermowell thermocouples to reach the target temperature was about one minute. Two tests (LOCA 126 and LOCA 127) were also heated directly to the target temperature but were ramped to temperature at a slower rate. The remaining tests (LOCA 128, LOCA 129, LOCA 130, and LOCA 132) were heated rapidly to an intermediate temperature in the range of 977 °C to 988 °C and held for about five minutes prior to ramping to the target temperature.

Results

Specimen weight gains following exposure to high-temperature steam are tabulated in Table 3 and the visual appearance of the specimens is shown in Figure 3. For the four tests below 1000 °C (LOCA 126, LOCA 118, LOCA 127, and LOCA 119), all specimens were black with no visual evidence of breakaway oxidation. There was no identifiable difference between the ANL and STD prepared specimens. In addition, weight gains from the 2008 ZIRLO samples prepared by ANL and STD were comparable as shown in Figure 4.

The two tests performed at 1000 °C (LOCA 128 and LOCA 117) exhibited large differences in the visual appearance of the specimens. All three specimens from LOCA 128 were black with no visual evidence of breakaway oxidation. This was in sharp contrast with the 2008 ZIRLO specimens from LOCA 117 which exhibited light green oxide indicative of breakaway. The breakaway oxide on 2008 ZIRLO material occurred at the ends of the specimens as well as isolated 'nodules' away from the ends. The specimen from S73-2102Z (LOCA 117) showed minor breakaway oxide on the specimen ends as well as one tiny 'nodule' in the middle of the specimen that suggested the potential onset of breakaway oxidation. A comparison of the temperature histories show that LOCA 117 was heated directly to the test temperature

of 1000 °C while LOCA 128 was heated to 977 °C and held for 250 seconds prior to heating to 1000 °C. A direct comparison of the temperature histories is shown in Figure 5a.

A similar comparison can be made between the two tests (LOCA 130 and LOCA 131) heated to 1020 °C. LOCA 130 was heated to 988 °C prior to heating to the test temperature of 1020 °C. The 2008 ZIRLO specimens were characterized by breakaway oxidation of the specimen ends as well as some isolated 'nodules' within the center of the tube. The ZIRLO sample from S73-2102Z (LOCA 130) had more breakaway oxide associated with the ends but no 'nodules' within the center of the sample. The LOCA 131 specimens were heated directly to 1020 °C and exhibited significantly more breakaway oxide than the LOCA 130 specimens. Both 2008 ZIRLO specimens from LOCA 131 were nearly 100% covered by light green oxide while S73-2102Z exhibited light green oxide on the ends as well as numerous nodules within the center of the tube. A comparison of the temperature ramp to the 1020 °C test temperature is shown in Figure 5b.

The final two tests (LOCA 132 and LOCA 129) were heated to intermediate temperatures prior to heating to the target temperatures of 1005 °C and 1010 °C, respectively. Both 2008 ZIRLO specimens heated to 1005 °C exhibited breakaway oxide on the tube ends as well as isolated 'nodules' away from the ends while S73-2102Z had only minor breakaway oxide on the tube ends. The three samples heated to 1010 °C exhibited light green, breakaway oxide on the sample ends. However, both 2008 ZIRLO samples also had isolated 'nodules' of light green oxide in the center of the specimens. It should be noted that the samples heated to 1010 °C were held at temperature for only 3810 seconds while the remaining tests (with the exception of LOCA 118) were at temperature for nominal times of 4400 seconds.

For the purpose of this study, only a visual examination of the specimens was performed. Prior test results reported in Reference 2 showed that the visual appearance of the oxide was a valid indicator of breakaway oxidation. In the prior study, black oxide was indicative of protective behavior while non-black oxide was indicative of breakaway oxidation. Breakaway oxide in Zircaloy-4 was gray while breakaway oxide in ZIRLO was tan. In all instances, the presence of gray or tan oxide was associated with circumferential cracking of the oxide and high hydrogen pickup while black oxide was free of cracks and resulted in low hydrogen values (e.g., < 200 ppm).

The breakaway oxide on ZIRLO in the current round of experiments was light green and similar in color to the breakaway oxide formed on ZIRLO tubes tested at ANL. The current STD oxidation tests and the ANL tests were both performed inside quartz tubes while the prior oxidation tests at STD that produced tan oxide on ZIRLO were performed inside an Alloy 690 tube. The difference in the color of the breakaway oxide on ZIRLO may be a result of the different containment material used for the prior and current steam tests at STD. Oxide color is typically due to point defects in the oxide which may be influenced by the quartz or Alloy 690 oxidation chamber.

Discussion

The primary objective of this study was to compare the breakaway oxidation results between ANL- and STD-prepared specimens. Material identified as 2008 ZIRLO was from a single tubing lot and provided a one-to-one comparison of the ANL and STD cleaning procedures. Examination of these samples in Figure 3 showed that the visual appearance of the ANL and STD cleaned specimens from 2008 ZIRLO was similar for all tests. This suggests that differences in sample cleaning between ANL and STD were

not a significant contributor to the difference in the minimum time to breakaway oxidation reported by ANL and Westinghouse.

While the current objective was to compare cleaning procedures, the global objective was to identify differences between the ANL and STD testing that would impact the time to breakaway oxidation. The results from the current testing provided some insights into potential differences.

The prior tests at STD (Ref. 2) had much slower heating rates than the current testing and the testing performed by ANL (Ref. 1). As mentioned previously, test specimens were placed inside an Alloy 690 tube that was loaded into a preheated furnace. The substantial increase in mass from the Alloy 690 tube resulted in slower heat-up rates than can be achieved in the current test facility. Table 4 documents the heat-up times for two tests (LOCA 42 and LOCA 43) that were performed in the prior test facility at nominal temperatures of 1000 °C and 1020 °C. The ramp times were about five minutes.

Figure 6 compares the heat-up portion of the breakaway oxidation tests performed in both the current and prior test facilities. LOCA 117 and LOCA 131 exhibited very rapid heating to the target temperatures of 1000 °C and 1020 °C, respectively, while LOCA 42 and LOCA 43 were heated directly to the target temperatures but at significantly lower rates. The ZIRLO samples from LOCA 117 and LOCA 131 exhibited breakaway oxidation as shown in Figure 3 while the samples from LOCA 42 and LOCA 43 were black with pre-breakaway oxide (Ref. 2).

The above comparison demonstrates that the heating rate plays a significant role in the onset of breakaway oxidation. The breakaway oxidation tests performed by ANL utilized heat-up rates that ramped samples to temperature from 300 °C in about 80 to 90 seconds (Ref. 1). The ramp times were slightly longer than those for LOCA 117 and LOCA 131 where samples reached temperature in about 60 seconds. In contrast, the times to reach temperature for LOCA 42 and LOCA 43 were about 300 seconds. The higher ramp rates in the ANL tests compared to those in the STD tests reported in Reference 2 would likely contribute to the shorter times to breakaway oxidation that were reported by ANL.

The specific role of ramp rate on breakaway oxidation times requires further study. LOCA 128 and LOCA 130 were rapidly heated to an intermediate temperature prior to a slower ramp for the final 20 °C to 30 °C to the target temperature (see Figure 6). This suggests that the ramp rate near the critical temperature for breakaway oxidation may be an important parameter as there was a significant reduction in the extent of breakaway oxidation for LOCA 128 and LOCA 130 relative to samples heated directly to temperature (LOCA 117 and LOCA 131).

This sensitivity to heat-up rate supports the use of prototypic rates for breakaway oxidation tests. Heat-up rates for small break LOCA are in the range of about 1.1-2.8 °C/sec (2-5 °F/sec) as reported in Reference 6. A comparison of temperature ramps used in the breakaway oxidation tests (LOCA 117 and LOCA 42) and small break LOCA (Ref. 6) is shown in Figure 7. LOCA 117 corresponds to the very rapid heating in the current study while LOCA 42 shows the slower heating rate that was used in the prior tests at Westinghouse (Ref. 2). The heat-up rates for small break LOCA are lower than those used in LOCA 42. Additional testing is planned at Westinghouse to establish appropriate, realistic heat-up rates for laboratory evaluation of breakaway oxidation times.

A secondary observation from this series of breakaway oxidation tests was a qualitative comparison of the results from the 2008 ZIRLO material and lot S73-2102Z. Visual examination of the post-test specimens in Figure 3 for several tests showed some sensitivity in the extent of breakaway oxidation between the two lots included in the study. The largest difference between the lots was observed for two tests (LOCA 117 and LOCA 131) with the very high heat-up rates. The difference in the visual appearance of the two lots was reduced for the tests that were heated to an intermediate temperature prior to ramping to the final test temperature. While variation in the extent of breakaway oxidation between the two lots was observed, these observations may be confounded by the fact that the heat-up rates that were used are not representative of those attained during a small break LOCA. Once an accepted testing protocol is established using prototypic heat-up rates, additional testing should be performed to investigate potential variation in breakaway times between lots.

One test parameter that has not been explored relative to its impact on breakaway oxidation is steam flow rate. While the flow rate of 6.5 g/minute is believed to be sufficiently high to avoid steam starvation, testing over a range of flow rates is planned to confirm that breakaway oxidation times are insensitive to flow rates that preclude steam starvation. Flow rate will be included as an additional parameter in the planned Westinghouse tests to establish appropriate, realistic conditions for laboratory evaluation of breakaway oxidation times.

Conclusions

A series of breakaway oxidation tests were performed on STD and ANL prepared specimens over the temperature range of 960 °C to 1020 °C to determine the impact of cleaning procedure on breakaway oxidation times. The following conclusions can be made from the study.

- There was no observed difference in the breakaway oxidation behavior of samples prepared by STD or ANL from a common lot (2008 ZIRLO) of material. The specimens were tested in the same steam oxidation run to minimize variations in test conditions.
- No breakaway oxidation was observed in specimens that were oxidized at temperatures from 960 °C to 990 °C for nominal times of 4400 seconds. However, samples tested in the temperature range of 1000 °C to 1020 °C exhibited varying degrees of breakaway oxidation.
- The extent of breakaway oxidation was greatest for samples that were rapidly heated to 1000 °C and 1020 °C. Breakaway oxidation was not observed at 1000 °C and was significantly reduced at 1020 °C when the heat-up rate was reduced. Based on these observations, heat-up rate is a significant variable for establishing the time to reach breakaway oxidation.
- Future breakaway oxidation testing at Westinghouse will be directed at establishing an appropriate heat-up rate for determining minimum times to breakaway oxidation.

References:

1. M. Billone, Y. Yan, T. Burtseva, R. Daum, 'Cladding Embrittlement During Postulated Loss-of Coolant Accidents,' NUREG/CR-6967, July 2008.

- 2. Letter from J. A. Gresham to USNRC, "Updated Westinghouse Breakaway Oxidation Testing/Behavior," LTR-NRC-08-29, June 12, 2008.
- 3. Letter from R. J. Comstock to M. Billone of ANL, STD-MCE-08-46, August 8, 2008.
- 4. Letter from R. J. Comstock to M. Billone of ANL, STD-MCE-08-63, October 16, 2008.
- 5. ASTM G2, "Standard Test Method for Corrosion Testing of Products of Zirconium, Hafnium, and Their Alloys in Water at 680 °F or in Steam at 750 °F."
- Letter from J. A. Gresham to USNRC, "Westinghouse Comments on the Technical Basis for New Performance-Based Emergency Core Cooling System Requirement," LTR-NRC-08-42, Sept. 2, 2008.

| LOCA Test | Target Temp | ANL Prepared | STD Prepared | |
|-----------|-------------|----------------------------|-------------------------|-----------|
| | (°C) | (2008 ZIRLO ¹) | 2008 ZIRLO ¹ | S73-2102Z |
| LOCA 117 | 1000 | IPS 107 A2 | STD 1 | 101 |
| LOCA 118 | 980 | IPS 107 A3 | STD 2 | 102 |
| LOCA 119 | 990 | IPS 107 A4 | STD 3 | 103 |
| LOCA 126 | 960 | IPS 107 A5 | STD 4 | 104 |
| LOCA 127 | 980 | IPS 107 A6 | STD 5 | 105 |
| LOCA 128 | 1000 | IPS 107 A7 | STD 6 | 106 |
| LOCA 129 | 1010 | IPS 107 A8 | STD 7 | 107 |
| LOCA 130 | 1020 | IPS 107 A9 | STD 8 | 108 |
| LOCA 131 | 1020 | IPS 107 A10 | STD 9 | 109 |
| LOCA 132 | 1005 | IPS 107 A11 | STD 10 | 110 |

Table 1Test specimens for breakaway oxidation tests

¹ Westinghouse lot S69-8568Z

| Furnace | | Intermediate Hold Temperature | | | Final Hold Temperature | | | |
|----------|----------|-------------------------------|------------|--------|------------------------|------------|--------|--|
| Test | Pre-Heat | Ramp | Avg. Temp. | Hold | Ramp | Avg. Temp. | Hold | Comments |
| | (°C) | (sec.) | (°C) | (sec.) | (sec.) | (°C) | (sec.) | |
| LOCA 126 | 935 | - | - | - | 250 | 959 | 4350 | Slower heating to final hold temp. |
| LOCA 118 | 970 | - | - | - | 70 | 980 | 3720 | Rapid heating to final hold temp. |
| LOCA 127 | 950 | - | - | - | 270 | 980 | 4370 | Slower heating to final hold temp. |
| LOCA 119 | 980 | - | - | - | 70 | 990 | 4320 | Rapid heating to final hold temp. |
| LOCA 128 | 970 | 80 | 977 | 250 | 130 | 1000 | 4410 | Rapid heating to inter. temp./ slower heating to final hold temp. |
| LOCA 117 | 990 | - | - | - | 60 | 1002 | 4330 | Rapid heating to final hold temp. |
| LOCA 132 | 980 | 60 | 988 | 350 | 90 | 1005 | 4470 | Rapid heating to inter. temp./ slower heating to final hold temp. |
| LOCA 129 | 980 | 80 | 987 | 280 | 130 | 1010 | 3830 | Rapid heating to inter. temp./ slower heating to final hold temp. |
| LOCA 130 | 980 | 70 | 988 | 310 | 210 | 1020 | 4340 | Rapid heating to inter. temp./ slower heating to final hold temp. |
| LOCA 131 | 1010 | - | - | - | 70 | 1020 | 4380 | Rapid heating to final hold temp. |

 Table 2
 Summary of thermal histories for steam oxidation tests

| Test | Temp. | Time (sec) | Surface | Lot Sample | | Meas. Wt. Gain (mg/dm^2) |
|-------------------|--------------|------------|------------|------------|-------------|----------------------------|
| | | | ANL Clean | 2008 ZIRLO | IPS 107 A5 | 638 |
| LOCA126 | 959 | 4350 | | 2008 ZIRLO | STD 4 | 648 |
| | | | STD Clean | \$73-2102Z | 104 | 659 |
| | | | ANL Clean | 2008 ZIRLO | IPS 107 A3 | 732 |
| LOCA118 | 980 | 3720 | | 2008 ZIRLO | STD 2 | 740 |
| | | | STD Clean | S73-2102Z | 102 | 771 |
| | | 4370 | ANL Clean | 2008 ZIRLO | IPS 107 A6 | 791 |
| LOCA127 | 980 | | | 2008 ZIRLO | STD 5 | 777 |
| | | | STD Clean | S73-2102Z | 105 | 815 |
| | | 4320 | ANL Clean | 2008 ZIRLO | IPS 107 A4 | 861 |
| LOCA119 | 990 | | | 2008 ZIRLO | STD 3 | 848 |
| | | | STD Clean | S73-2102Z | 103 | 890 |
| | | 4410 | ANL Clean | 2008 ZIRLO | IPS 107 A7 | 1000 |
| LOCA128 | 1000 | | STD Clean | 2008 ZIRLO | STD 6 | 953 |
| | | | | S73-2102Z | 106 | 975 |
| LOCA117 1002 4330 | | ANL Clean | 2008 ZIRLO | IPS 107 A2 | 1095 | |
| | 1002 | 4330 | | 2008 ZIRLO | STD 1 | 1116 |
| | | | STD Clean | S73-2102Z | 101 | 1022 |
| | | | ANL Clean | 2008 ZIRLO | IPS 107 A11 | 1142 |
| LOCA132 | 1005 | 4470 | | 2008 ZIRLO | STD 10 | 1075 |
| | | | STD Clean | S73-2102Z | 110 | 1052 |
| | LOCA129 1010 | 3830 | ANL Clean | 2008 ZIRLO | IPS 107 A8 | 1028 |
| LOCA129 | | | | 2008 ZIRLO | STD 7 | 1005 |
| | | | STD Clean | S73-2102Z | 107 | 1023 |
| | 1020 | 4340 | ANL Clean | 2008 ZIRLO | IPS 107 A9 | 1212 |
| LOCA130 | | | | 2008 ZIRLO | STD 8 | 1188 |
| | | | SIDClean | S73-2102Z | 108 | 1320 |
| | 1020 | 4380 | ANL Clean | 2008 ZIRLO | IPS 107 A10 | 1509 |
| LOCA131 | | | | 2008 ZIRLO | STD 9 | 1520 |
| | | | SIDClean | \$73-2102Z | 109 | 1424 |

Table 3Measured weight gains of ZIRLO following exposure to high-temperature steam.

LTR-NRC-09-24 NP-Enclosure

| Test | | Final Hold Temperature | | | |
|---------|--------------|------------------------|------------|--------|--|
| | ZIRLO | Ramp | Avg. Temp. | Hold | |
| | Sample | (sec.) | (°C) | (sec.) | |
| LOCA 42 | S73-2102Z 52 | 330 | 999 | 4320 | |
| LOCA 43 | S73-2102Z 53 | 270 | 1014 | 4320 | |

Table 4Thermal history from prior steam oxidation tests (Ref. 2)

LTR-NRC-09-24 NP-Enclosure



Figure 1 Schematic drawing of modified high temperature steam oxidation facility at Westinghouse STD.

LTR-NRC-09-24 NP-Enclosure



Figure 2 Temperature histories for breakaway oxidation tests.



Figure 3 Visual appearance of specimens following exposure to high-temperature steam.



Figure 4 Comparison of weight gains from 2008 ZIRLO samples prepared by ANL and STD. Samples were exposed to high-temperature steam as documented in Table 3.



Figure 5 Comparison of temperature histories of LOCA breakaway oxidation tests. Time t = 0 corresponds to the insertion of the specimens into the hot zone of the furnace.a) comparison of temperature ramp of tests heated to 1000 °C and b) comparison of temperature ramp of tests heated to 1020 °C.



Figure 6 Comparison of heat-up rates used for breakaway oxidation tests. Time t = 0 corresponds to the insertion of the specimens into the hot zone of the furnace.
a) nominal temperature of 1000 °C; modified test facility (LOCA 117) and prior test facility (LOCA 42), and b) nominal temperature of 1020°C; modified test facility (LOCA 131) and prior test facility (LOCA 43).



Figure 7: Comparison of temperature ramps in laboratory breakaway oxidation tests (LOCA 117 and LOCA 42) and small break LOCA (Ref. 6). (For laboratory tests, t = 0 seconds corresponds to insertion of the specimens into the furnace.)