AN ANALYSIS OF STATIC LOADING RESULTS ON SLOTTED RING SAMPLES TO ALLOW FOR FURTHER INVESTIGATION OF STRESS CORROSION CRACKING

***J. Metzler, G.A. Ferrier, M. Farahani, P.K. Chan, and E.C. Corcoran**
Royal Military College of Canada, Kingston, Ontario, Canada
*(Joseph.Metzler@rmc.ca)

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Abstract

Stress corrosion cracking can cause failures of CANDU® Zircaloy-4 fuel sheathing. A series of static loading tests were performed on slotted ring samples in support of ongoing efforts to analyze the effects of iodine concentration, temperature, and stress levels on the corrosion of Zircaloy-4. The corrosive degradation of Zircaloy-4 was evaluated using deflection measurements. A regression analysis determined that iodine concentration and temperature have had a linear effect on deflection results thus far, while the stress level has not.

1. Background

Stress corrosion cracking (SCC) is a phenomenon that can cause failures in CANDU® fuel sheathing. Three criteria are required for SCC: 1) a susceptible material; 2) a critical level of strain in the material; and 3) an environment that permits SCC [1]. When Zircaloy-4, the material of CANDU fuel sheathing, is strained and exposed to sufficiently high iodine concentrations and temperatures, it will likely experience SCC [2].

During reactor operation, Zircaloy-4 sheathing is strained by thermal expansion of UO_2 fuel pellets following a power ramp. As the fuel pellets expand, fission products (including iodine) are released from the UO_2 pellet that subsequently interact with Zircaloy-4 at the pellet-sheath interface where the temperature can exceed 300 °C. This environment satisfies the conditions required for SCC and can lead to failures. Current SCC mitigation strategies have proven to be effective, largely preventing failures (0.01% failure rate) that would allow fission products to escape into the reactor coolant flow and create a safety hazard for workers. SCC is being investigated to potentially develop strategies that would allow CANDU reactors to operate at a higher burnup or with increased load cycling.

The experiments described in this paper continue the work of Quastel *et al.* [3] to create a baseline of static loading results based on experiments developed by Wood *et al.* [4]. These data can be compared against results investigating alternative SCC mitigation strategies and the results being collected by Ferrier *et al.* [5] using a dynamic loading apparatus.

2. Experimental Results and Analysis

A series of static tests were performed with varying iodine concentrations, wedge sizes, and temperatures. In the static tests, slotted ring samples were cut from Zircaloy-4 sheaths and loaded onto static wedges of a known width, creating a maximum tensile stress opposite from the slot. The loaded ring samples are placed into an evacuated capsule at a set temperature of at least 300 °C with a known iodine concentration for five days.

After each static test, the ring samples were removed from the capsule, separated from their corresponding wedge, cleaned, and subjected to elastic loads to evaluate their mechanical resistance. The corresponding ring deflections were recorded using a deflection measurement apparatus as discussed by Quastel *et al*. Equation 1 displays the expected deflection of a ring sample, D_y, when placed under a mass load (F_y = mg) where R is the radius, l is the width, E is the Young's modulus, and t is the ring thickness.

$$D_y = \frac{36\pi R^3 F_y}{Elt^3} \tag{1}$$

Small changes in the thickness of the ring can be measured since the deflection experienced by a ring is proportional to the inverse cube of its thickness (Equation 1). Thus, deflection measurements can be used to quantify the severity of the corrosive attacks actively reducing the sample thickness. Measurements were performed at 22 distinct test conditions (Table 1).

Table 1: Summary of static loading deflection results (*N* = number of samples, δ is average deflection).

Test condition	***Iodine*** ± 10 / mg	***Wedge*** ± 0.05 / mm	***Temperature*** ± 10 / °C	*N*	δ ± 0.05 / mm for a 95 g load
1	1530	6	300	4	1.06
2	1530	7	300	4	1.01
3	1530	9	300	12	1.10
4	1530	12	300	4	1.04
5	1530	9	375	4	1.27
6	1530	12	375	4	0.98
7	255	4.5	300	4	0.82
8	255	6	300	4	0.85
9	255	7	300	4	0.86
10	255	9	300	8	0.94
11	255	12	300	3	0.93
12	255	6	375	4	0.94
13	255	7	375	4	0.96
14	0	4.5	300	4	0.80
15	0	6	300	4	0.90
16	0	7	300	4	0.92
17	0	9	300	8	0.90
18	0	12	300	4	0.87
19	0	6	375	4	0.90
20	0	7	375	4	0.96
21	0	9	375	4	0.94
22	0	12	375	4	0.86

To develop a more complete and quantifiable understanding of how temperature, wedge size, and iodine level affect deflection results, a multivariable linear regression was performed using Microsoft Excel™. The regression was performed to model the deflections experienced by each ring when loaded with 95 g. The statistics for each parameter from the preliminary regression results are shown in Table 2. The regression model also produced a y-intercept value of 0.7 with a standard error of 0.09 and a *p*-value of 5.3E-12.

Table 2: Preliminary regression results for investigated parameters.

Parameter	Coefficients	Standard Error	*p*-value
Iodine	0.0001	1.4E-05	4.7E-16
Temperature	0.0006	0.0003	0.02
Wedge Size	0.002	0.004	0.7

The *p*-value in Table 2 represents the likelihood of a parameter not having a significant linear effect on deflection results. This value must be less than 0.05 for the parameter to be accepted into the model at the 95% confidence level. Since the *p*-value for wedge size was too high (0.7 > 0.05), it was removed from the model and the regression was updated with temperature and iodine as the parameters of interest (Table 3). The regression model also has a y-intercept value of 0.7 with a standard error of 0.08 and a *p*-value of 6.3E-13.

Table 3: Updated regression results for investigated parameters.

Parameter	Coefficients	Standard Error	*p*-value
Iodine	0.0001	1.3E-05	3.9E-17
Temperature	0.0006	0.0003	0.02

All of the parameters in the updated regression results have a *p*-value of < 0.05, which indicates that they can all be accepted with 95% confidence. Figure 1 through Figure 3 display the deflection results against each specific parameter, with error bars representing a standard deviation from the results in either direction. These figures are used to visualize/confirm the results from the regression analysis. There appears to be a significant increase in deflection at high levels of iodine (Figure 1), a possible increase in deflection at higher temperatures (Figure 2), and no apparent effect on deflection from changes in wedge size (Figure 3). These observations correspond well with the *p*-values found in the original regression results. In light of the large overlap in error bars in Figure 2 a two-tailed, type three, T-test was performed for the temperature results to confirm that the ring deflections at 300 °C and 375 °C are distinct (at a 95% confidence level). Also, it should be noted that the seemingly lower deflection value corresponding to the 4.5 mm wedge in Figure 3 is most likely caused by the fact that only two runs were completed with 4.5 mm wedges, both at low temperatures and low levels of iodine. Similarly, the seemingly higher value corresponding to the 9 mm wedge in Figure 3 is likely caused by the large number of runs performed at high temperatures and high levels of iodine while using 9 mm wedges.

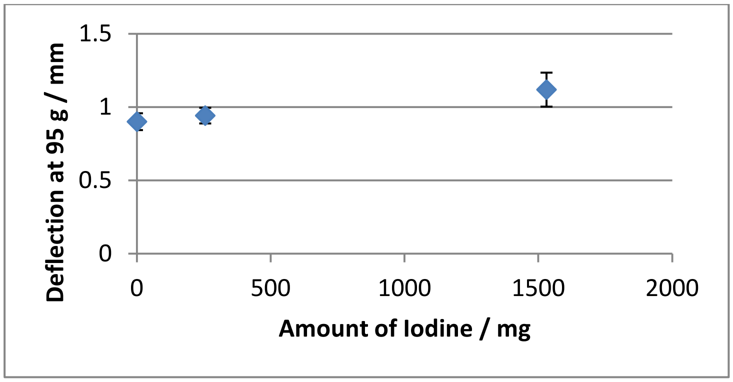

Figure 1: Average deflection results at 300 °C of ring samples on 9 mm wedges vs. iodine.

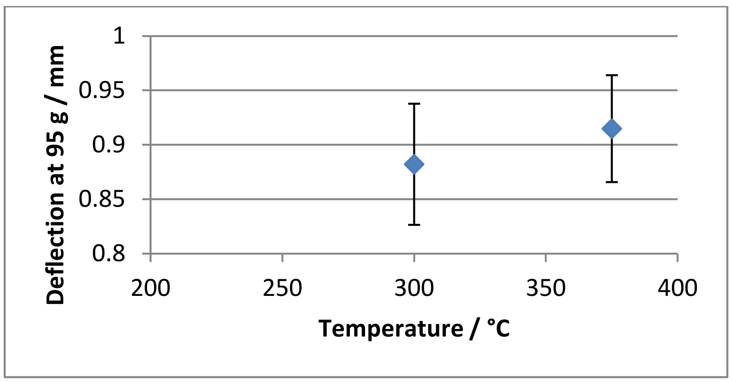

Figure 2: Average deflection results for all samples tested without iodine vs. temperature

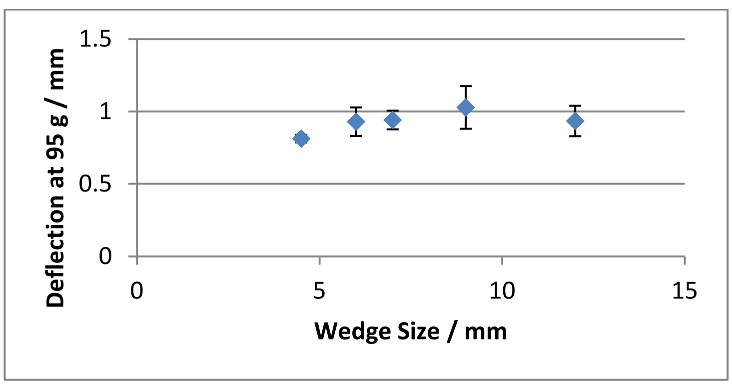

Figure 3: Average deflection results for experiments performed vs. wedge size.

To visualise the accuracy of this model, a plot was developed to compare the experimental and regression results. Figure 4 displays the average experimental deflection and the expected deflection according to the updated regression model at all six combinations of iodine level and temperature. To analyze the accuracy of this model, summary statistics from the excel regression were included into Figure 4. The adjusted R^2 value above 0.5 shows that the regression model produced results reasonably similar to the average experimental results.

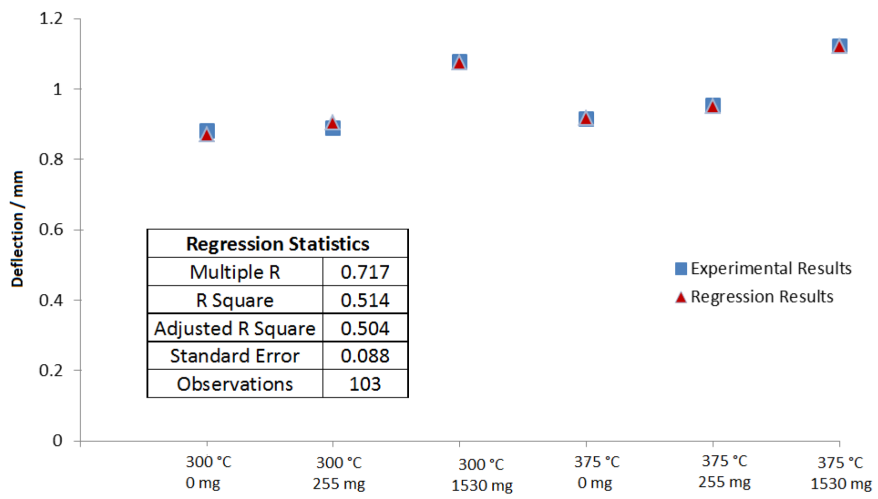

Figure 2: Comparison of experimental and regression results. The accuracy of the predictions without considering wedge size further proves the insignificant effect of wedge size on deflection seen thus far.

3. Conclusion

A series of static loading experiments were performed to create a baseline data set for future dynamic loading and alternative SCC mitigation investigations. Statistical analysis showed that the amount of iodine included and the temperature of the experiments have had a significant effect on deflection results thus far. It has also been shown that the stress level caused by varying the size of the wedges used in experiments has not been having a linear effect on deflection results.

4. References

[1] R. Cottis, "Guides to good practice in corrosion control - stress corrosion cracking," *National Physical Laboratory*, 2000.

[2] H. Rosenbaum, "The interaction of iodine with Zircaloy-2", General Electric Company, Pleasanton, California, 1966.

[3] A.D. Quastel, E.C Corcoran, and B.J. Lewis, "The effect of oxidized UO2 on iodine-induced stress corrosion cracking of fuel sheathing," in 12th International Conference on CANDU Fuel, Kingston, Canada, 2013.

[4] J. Wood, "Factors affecting stress corrosion cracking of Zircaloy in iodine vapour," *Journal of Nuclear Materials*, vol. 45, pp. 105-122, 1972.

[5] G.A. Ferrier, J. Metzler, M. Farahani, P.K. Chan, and E.C. Corcoran, "Dynamic thermo-chemo-mechanical strain of zircaloy-4 slotted rings for evaluating strategies that mitigate stress corrosion cracking," Pacific Basin Nuclear Conference, Vancouver, Canada, 2014.