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In-Situ Environmentally Induced Cracking in sCO_2

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ABSTRACT

Supercritical Carbon Dioxide (sCO_2) has been identified as a candidate fluid for advanced power cycles. The sCO_2 Brayton cycle is considered for a wide variety of power conversion systems such as nuclear, geothermal, coal, natural gas, and any system that converts heat energy into electrical energy. The need for corrosion data on energy systems materials is a primary concern for developing more efficient power production facilities. Various institutions have examined the corrosion resistance of applicable materials; however, to date, there are zero publications on in-situ crack growth in high temperature and pressure sCO_2. This project examines the ability to collect in-situ stress corrosion cracking and low cycle fatigue data in environments that energy system materials would experience. The elevated temperatures (500-800 °C) and high pressures (up to 25 MPa) of these cycles make it challenging to use traditional test methods; therefore, the tests are being performed using new equipment recently developed at Idaho National Laboratory. The corrosion resistance of unstressed materials, tested at these temperatures and pressures will be displayed, as well as the calibration procedure and set up of the cracking data collection device.

INTRODUCTION

The *in situ* performance of materials in sCO_2 environments is largely unexplored due to the challenges associated with both high temperature, high pressure experiments. To date, there is no available published literature on stress corrosion cracking (SCC) in sCO_2. There is also a lack of published literature for corrosion fatigue in sCO_2 for both post exposure analysis and in situ measurements. However, literature does exist for both types of corrosion for the similar supercritical water reactors (SCWR); see references [1-16]. Research for SCC in SCWR, [1-3], reveals many insights about research in SCC for sCO_2. First, the crack growth rates did not appear to be collected in situ, instead they used constant load or extension rate, and they compared results to in air results. The research for corrosion fatigue in SCWR [16] revealed that in situ results were easily obtained for the comparative air exposure via direct current potential drop (DCPD), but the crack growth rate of the SCWR exposure was determined post exposure.

Crack growth rates are particularly important to component designers for materials selection and lifetime predictions. We are using a novel loading mechanism and crack growth measurement technique to perform SCC and low cycle corrosion fatigue testing in a small volume envelope to allow for in-situ testing in existing sCO_2 autoclave chambers. This technology was originally designed by Idaho National Laboratory (INL) to perform SCC testing inside the Advanced Test Reactor. This system uses a bellows, actuated by high pressure gas, to apply a load to the specimen. The force from the expanding bellows is transmitted to the test specimen by a scissor-like mechanism, Figure 1. There is, therefore a direct correlation between the pressure applied to the bellows and the load applied to the specimen. By alternating the pressure inside the bellows with atmospheric pressure, it is possible to apply cyclic loading to the specimen. Constant load, constant stress intensity factor tests are also possible. The crack growth is measured *in situ* by direct current potential drop technique. The whole unit can fit inside the sCO_2 corrosion loop currently functioning at Oregon State University (OSU). The pressurized line and direct current potential drop leads will pass through the top cover of the test section of the sCO_2 loop.

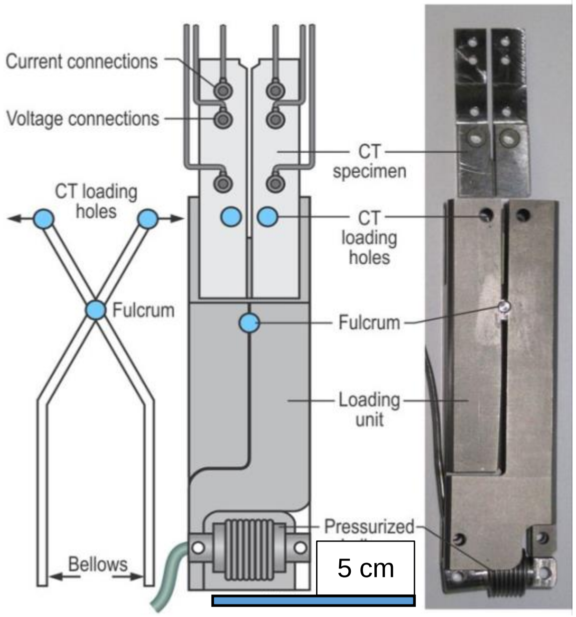


Figure 1: *In situ* crack growth measurement [17]

Significant work has been performed to assess the readiness of the bellows system. The bellows system has been calibrated against a load cell to relate sample strain to the pressure in the bellows. Figure 2 shows a typical compact tension specimen used in the bellows actuator. The specimen has long legs for the attachment of the voltage and current leads. By using a DCPD system, a micro-current can be passed through the compact tension (CT) specimen and the potential drop can be measured across the sample. As the cross section of the CT specimen decreases, the electrical potential also decreases between the leads in a predictable way.

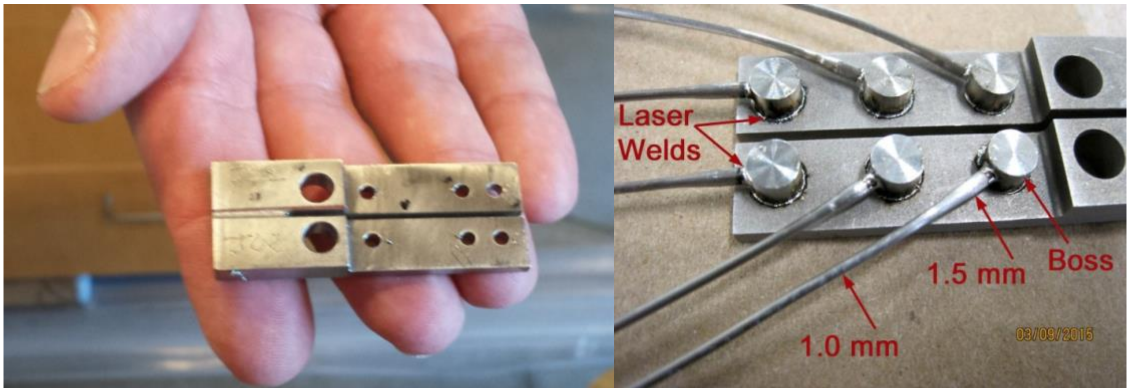


Figure 2: CT specimen for bellows actuated system (left) without leads (right) with leads

RESULTS AND DISCUSSION

The first step in utilizing the pressurized loading device was to calibrate the system to be comparable to conventional crack propagation data. The pressure applied to the bellows, therefore, needed to be correlated to an equivalent force. The calibration was performed with two complementary methods. Since we possess two loading devices, each pressure-loader was calibrated with one of the methods. Figure 3 displays the first calibration where the load-strain data collected on the specimen is compared to the bellows pressure-strain data. The pressure-load relation that was derived from the strain gauge calibration is displayed as Equation 1. Equation 1 corresponds to load experienced from the pressure acting on the geometry of Pressure-Loader 1.

$$Force\ [kN] = 1.26 * Pressure[KSI] - 143 \tag{1}$$

The second calibration performed was to assess the role of temperature on the pressure/load relationship. Figure 5 shows the experimental set up of the load frame used to calibrate the bellows system. The bellows actuator is surrounded by heating tape in this figure for the temperature calibration test. The load recorded at incremental pressures is shown in Figure 6 for three different temperatures. It is clear from this data that the pressure/load relation does not change with temperature in the range tested. A relation for the pressure loader was developed and displayed in Equation 2. Equation 2 corresponds to load experienced from the pressure acting on the geometry of Pressure-Loader 2.

$$Force\ [kN] = 1.5 * Pressure[KSI] \tag{2}$$

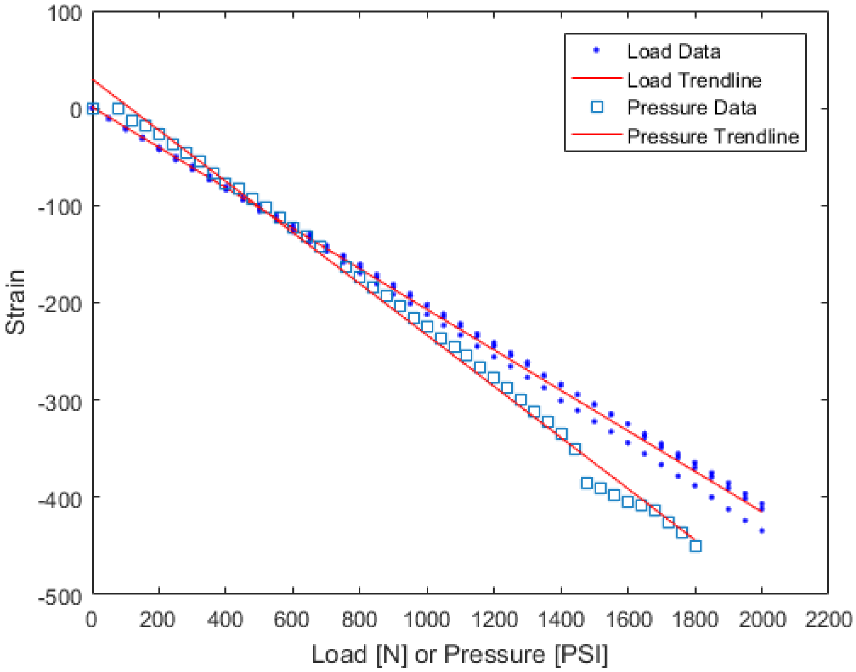


Figure 3: Pressure and load vs. strain

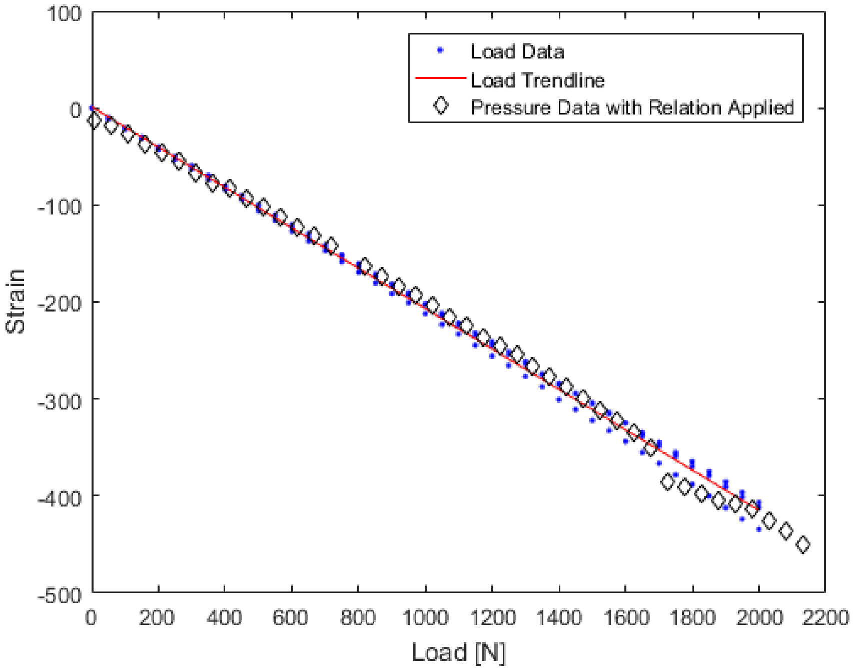


Figure 4: Pressure/load relationship applied

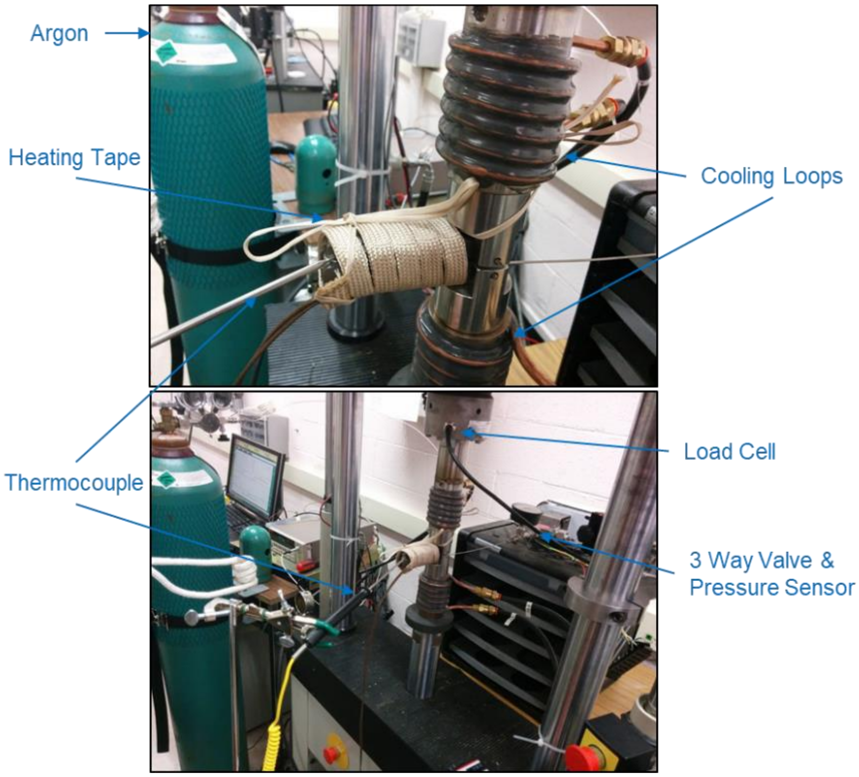


Figure 5: Load frame set up for bellows pressure/temperature calibration

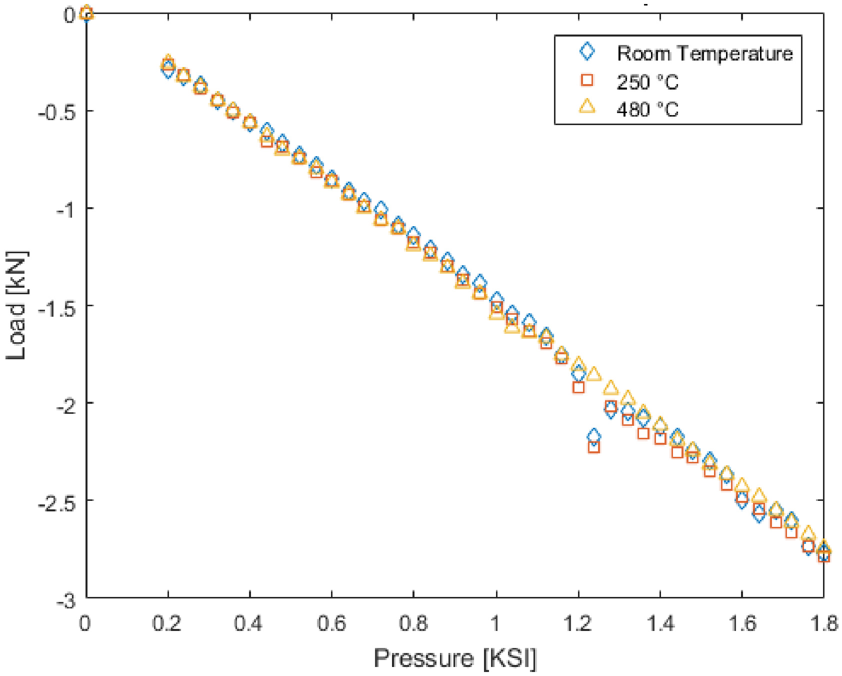
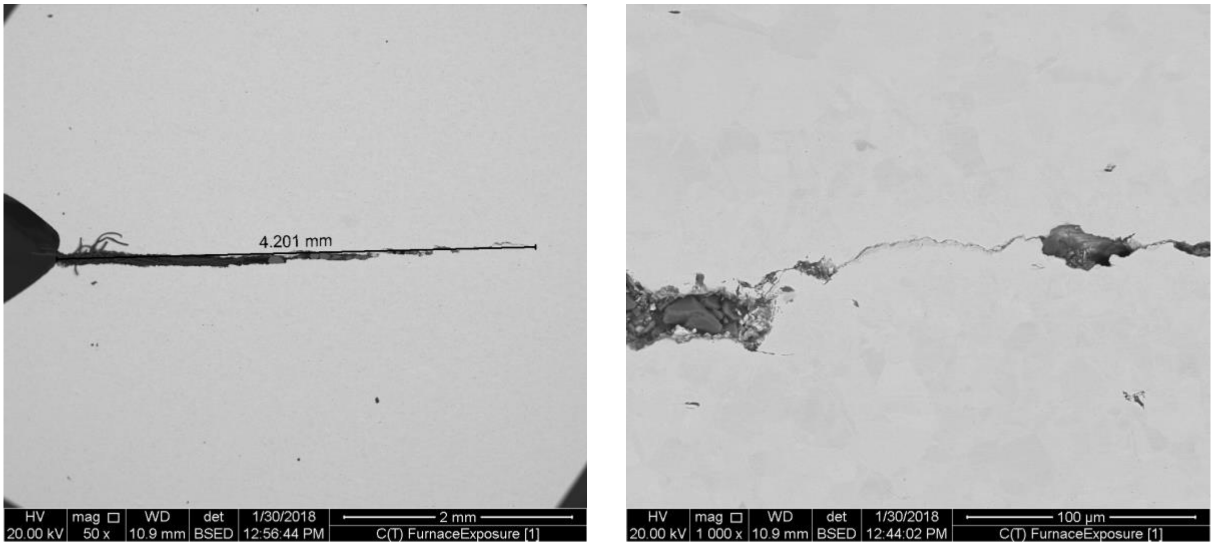


Figure 6: Pressure/load relationship with temperature

The first set of testing was exposure at elevated temperature in an ambient pressure air environment. The exposure was carried out on a pre-cracked sample (1 mm pre-crack) of alloy 316 at 350°C. Pressure-Loader 2 was used for this experiment. The bellows was attached to an Argon source and set to an alternating pressure of 0-2000 psi, which according to Equation 2 is equivalent to 0-3 kN force on the specimen. The frequency of this force was a full cycle every 30 sec (.03 Hz). The sample was tested for 5 hours and the resulting crack cross-section can be seen in Figure 7. The raw voltage data collected from the exposure is displayed in Figure 8.



Crack Length

Near Crack Tip

Figure 7: Crack after 5 hours of exposure to 350°C and ambient pressure air

At the end of the 5 hour exposure, the bellows failed, due to rupture. Two likely causes are overextension due to the large crack that formed or thermal cycling due to the cooler incoming Argon. The size of this crack (4.2 mm) is large for 5 hours of exposure, implying that future data may be collected with considerably lower load and frequency. We do not necessarily need high frequency because the goal is to test under constant stress intensity factor, which is close to no cycling. This will be good for the bellow; however, this is not necessarily good for the noise because the electrical noise does not come from the bellow. Under a constant load, the system will have to be able to detect smaller fluctuations and the electrical noise may become an issue. Ideally, decreasing the load and the cycle frequency would cause the crack to progress over a longer time interval which will decrease the Argon consumption, prolong the life of the bellows, and decrease noise of the voltage readings. This initial test also demonstrates the need for the Argon to be preheated prior to entry into the bellows to limit the thermal cycling it is exposed to.

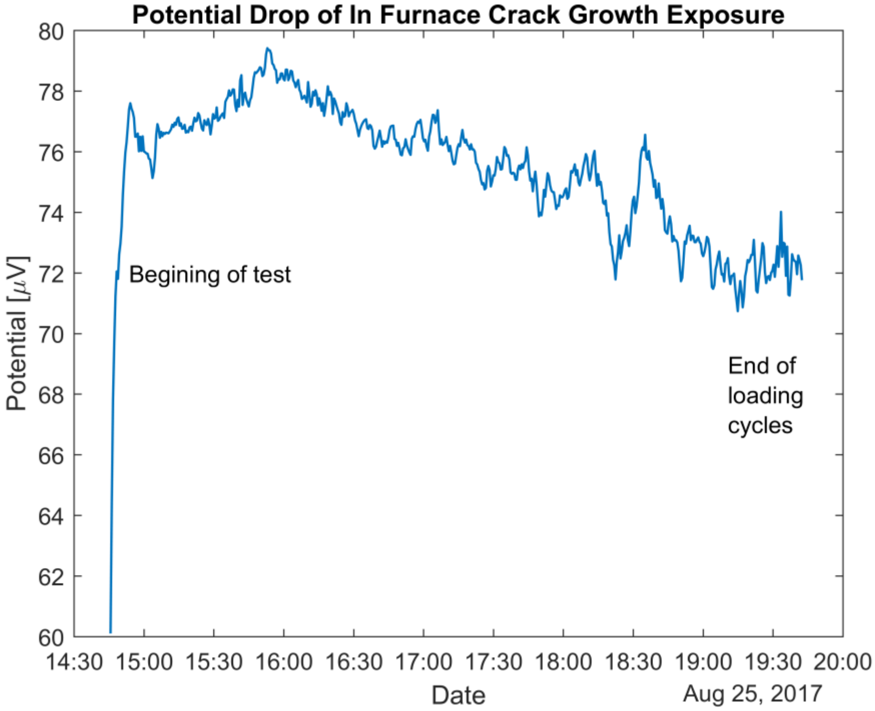


Figure 8: Raw voltage readings for air furnace exposure

CONCLUSIONS

Pressure-load relations were developed for the two mechanical pressure-loaders at OSU. These relations are linear and do not appear to apply loads that vary with temperature. Pressures applied to the bellows can be correlated to load applied to the CT samples.

The beginning steps to perform in situ sCO_2 crack growth exposures are underway. Further ambient pressure air furnace exposures are required. Load and cycle frequencies will need to be adjusted in order to increase crack growth time from hours to days or weeks. This decrease in cracking rate will allow more accurate collection of data as well as preserve the integrity of bellows and Argon consumption.

Once conditions for ambient air furnace exposures are optimized, the system can be transferred into the OSU sCO_2 autoclave. Electrical connections and pressure tubing can be inserted into the autoclave via feedthrough in order to maintain external pressure and collect in situ sCO2 environmentally induced cracking data.

REFERENCES

[1] L. Fournier, D. Delafosse, C. Bosch, and T. Magnin, “Stress corrosion cracking of nickel base superalloys in aerated supercritical water,” in *2001 NACE Corrosion Conference*, (Houston, TX), NACE, 2001.

[2] H. Je and A. Kimura, “Stress corrosion cracking susceptibility of candidate structural materials in supercritical pressurized water,” *Journal of Nuclear Materials*, vol. 455, no. 1, pp. 507–511, 2014. Proceedings of the 16thInternational Conference on Fusion Reactor Materials (ICFRM-16).

[3] G. Was, P. Ampornrat, G. Gupta, S. Teysseyre, E. West, T. Allen, K. Sridharan, L. Tan, Y. Chen, X. Ren, and C. Pister, “Corrosion and stress corrosion cracking in supercritical water,” *Journal of Nuclear Materials*, vol. 371, no. 1,pp. 176 – 201, 2007. Nuclear Fuels and Structural Materials 1.

[4] S. Teysseyre “Supercritical Water Reactors,” in *Nuclear Corrosion Science and Engineering* published by Woodhead ed. Damien Ferron (2012).

[5] S. Teysseyre, G. S. Was and J. Busby, “Stress Corrosion Cracking Behavior of Cast Stainless Steels,” Proc. 14th Int’l Conf. *Environmental Degradation* of Materials in Nuclear Power Systems – Water Reactors, TMS, Virginia Beach, VA (2009)

[6] S. Teysseyre and G.S. Was, “SCC crack growth rates of austenitic alloys in supercritical water,” *NACE Corrosion 2008*, paper 08428

[7] Q. J. Peng, S. Teysseyre, P. L. Andresen and G. S. Was, “Stress Corrosion Crack Growth in 316 Stainless Steel in Supercritical Water,” *Corrosion*, No 11, vol. 63 (2007) 1033-1041.

[8] S. Teysseyre, Z. Jiao, E. West, G.S. Was, “Effect of Irradiation on Stress Corrosion Cracking In Supercritical Water”, **J***. Nucl. Mat.* , Vol 371 (2007) 1-3 pp 107-117.

[9] G. S. Was, P. Ampornrat, G. Gupta, S. Teysseyre ,E. A. West, T. R. Allen, K. Sridharan, L. Tan, Y. Chen, X. Ren, and C. Pister, **“**Corrosion and Stress Corrosion Cracking in Supercritical Water”, *J. Nucl. Mater*., Vol 371 (2007), pp176-201

[10] S. Teysseyre and G.S. Was, “Stress Corrosion Cracking of Neutron-Irradiated Stainless Steel in Supercritical Water.”, *Proc. 13th Int’l Conf. Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors*, TMS, Whistler, BC, Canada (2007)

[11] E.A. West, S. Teysseyre, Z. Jiao, G.S. Was, “Influence of irradiation induced microstructure on the stress corrosion cracking behavior of austenitic alloy in supercritical water”, *Proc. 13th Int’l Conf. Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors*, TMS, Whistler, BC, Canada (2007)

[12] S. Teysseyre, Q. Peng, C. Becker, G.S Was, “Facility for stress corrosion cracking of irradiated specimens in supercritical water”, *J. Nucl. Mat.* , Vol 371 (2007) pp 98-106.

[13] G. S. Was, S. Teysseyre and Z. Jiao, "Corrosion of Austenitic Alloys in Supercritical Water," *Corrosion*, No. 11, vol. 62 (2006) 989-1005.

[14] S. Teysseyre, G.S. Was, “Stress Corrosion Cracking of Austenitic Alloys in Supercritical Water”, *Corrosion*, No. 12, vol. 62 (2006) 1100-1116.

[15] G. S. Was, S. Teysseyre, “Challenges and recent progress in corrosion and stress corrosion cracking of alloys for supercritical water reactor core components”, *Proc. 12th Int’l Conf. Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors*, TMS, Warrendale, PA (2005)

[16] Y. Yi, B. Lee, S. Kim, and J. Jang, “Corrosion and corrosion fatigue behaviors of 9cr steel in a supercritical water condition,” *Materials Science and Engineering: A*, vol. 429, no. 1, pp. 16-168, 2006.

[17] A. J. Palmer, J. L. Rempe, and S. P. Teysseyre, “Adaption of Crack Growth Detection Techniques Developed to US material Test Reactors,” in *Proceedings of ICAPP 2014*, ICAPP, 2014.

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