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Hydrogen Fracture Toughness Tester Completion

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HYDROGEN FRACTURE TOUGHNESS TESTER COMPLETION

I. SUMMARY

The Hydrogen Fracture Toughness Tester (HFTT) is a mechanical testing machine designed for conducting fracture mechanics tests on materials in high-pressure hydrogen gas. The tester is needed for evaluating the effects of hydrogen on the cracking properties of tritium reservoir materials. It consists of an Instron Model 8862 Electromechanical Test Frame; an Autoclave Engineering Pressure Vessel, an Electric Potential Drop Crack Length Measurement System, associated computer control and data acquisition systems, and a high-pressure hydrogen gas manifold and handling system. The tester is located in Cell 1 of an existing SRNL facility, Building 774A which is designed for safely conducting experiments with high pressure hydrogen gas. During FY15, the tester was completed for conducting initial tests on materials in gaseous environments. These activities included: (1) Relocation and installation of Instron load frame control system for remote operation outside of the high-pressure cell; (2) Designing, fabricating and installing mechanical hardware on the load frame for conducting fracture mechanics tests on samples contained in a high-pressure hydrogen enviroment; (3) Installing high-pressure tubing, pressure gage, and automatic valve and connections to high-pressure manifold; (4) Installing other associated fracture mechanics hardware including an internal load cell, displacement gage, crack length monitoring wiring, thermistor and electrical feedthroughs; (5) Installing a custom DC Potential Drop Crack Length Monitoring System; (6) Developing and installing a LabView® data acquisition system; (7) Conducting leak tests of pressure vessel and high-pressure manifold; (8) Developing procedures for vessel assembly/disassembly, sample insertion 7 removal and conducting fracture mechanics tests; and, (9) Conducting a fracture mechanics test within the vessel under pressure.

This report documents fulfillment of the requirements for the Enhanced Surveillance Campaign (ESC) FY15 Level 2 milestone, 5313, to “Complete Hydrogen Fracture Toughness Tester (HFTT).”

II. BACKGROUND

Tritium reservoirs are constructed from welded forged stainless steels and filled and stored at the Savannah River Site. One of the primary interests of the Savannah River Site’s Enhanced Surveillance Campaign is to measure hydrogen and tritium effects on the materials of construction of tritium reservoirs. The purpose is to provide relevant fracture toughness values for use by the Design Agencies for fracture modeling, reservoir life prediction, and safety margin calculations.

It has become increasingly clear that a facility at SRNL was needed for measuring fracture toughness properties of tritium reservoir materials in high-pressure gas (1-3). This is particularly true for evaluating hydrogen effects on cracking when hydrogen can more readily diffuse through the material (1). For example, weldments of stainless steel have a two-phase microstructure consisting of ferrite and austenite. Hydrogen can diffuse much more rapidly through the ferrite which can affect the cracking properties. Aluminum alloys are under evaluation for future reservoir systems. Hydrogen diffuses through aluminum much more rapidly than in stainless steel. Also, additive manufacturing is being considered as an alternative manufacturing technique for the construction of tritium reservoirs. The microstructure of Type 304L stainless steel, fabricated in this way will have more of the high-diffusivity ferrite phase than in the purely austenitic steels fabricated using conventional forging and machining. For these reasons, the capability for conducting cracking tests in high-pressure hydrogen gas was needed.

The purpose of this report is to describe the FY15 activities that were done to complete the installation of the HFTT in a high-pressure cell in SRNL. The report also lays out activities and deliverables for FY16.

III. HFTT Completion Activities

The HFTT was located in Cell 1 of 774A during FY14 (3). This Cell is designed for conducting experiments with high-pressure hydrogen and became available after hydride development programs were completed or moved to another laboratory. The HFTT consists of a mechanical testing frame equipped with a pressure vessel with electrical ports and pull rods with sliding seals for conducting fracture mechanics experiments in high-pressure gas (3). The system will be capable of conducting a variety of mechanical property measurements; however, it is chiefly designed for conducting ASTM fracture toughness measurements (4). It is a two-column load frame that features high stiffness and precision alignment. It has low speed capabilities, ideal for hydrogen embrittlement testing, and uses an electromechanical actuator. The floor-mounted systems is ideal for hydrogen-embrittlement studies. Testing speeds range from 1 mm/hr to 100 mm/min. and loads up to 100 kN. Figure 1 shows the Instron hardware, Autoclave vessel, and manual control system within the cell. Figure 2 shows the existing high-pressure hydrogen gas manifold, control valves, gas cylinders, vacuum pumps and a high-pressure gas compressor.

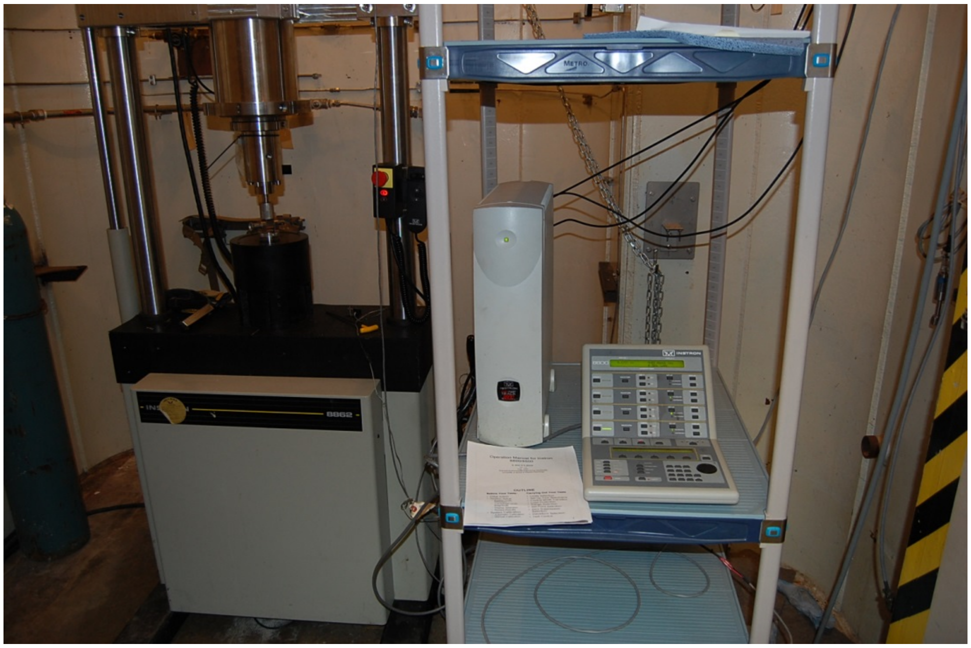


Figure 1. Instron load frame, Autoclave vessel and manual controls inside Cell 1.

Figure 2. Part of existing Cell 1 manifold including compressor and vacuum pump. Not shown are gas cylinders, automatic control valves and high-pressure manifold.

One of the key activities during FY14 was to design and fabricate the required mechanical hardware for conducting fracture mechanics tests. Two clevis grips were fabricated for connecting the vessel cap to the external load cell and upper crosshead and the lower pull rod with sliding seal to the Instron actuator. These grips are shown in Figures 3 and 4. The upper grip allows for the cap to be removed during specimen insertion and removal operations. The pin is removed and the upper crosshead raised up and out of the way so that the cap can be unscrewed. The crosshead is then lowered and used to raise the cap and specimen load-train up and out of the vessel (Figure 5). The lower grip is used for connecting to the lower pull rod. The lower pull rod is designed with a sliding seal and a threaded internal connection that connects to and disconnects from the specimen load train within the vessel.

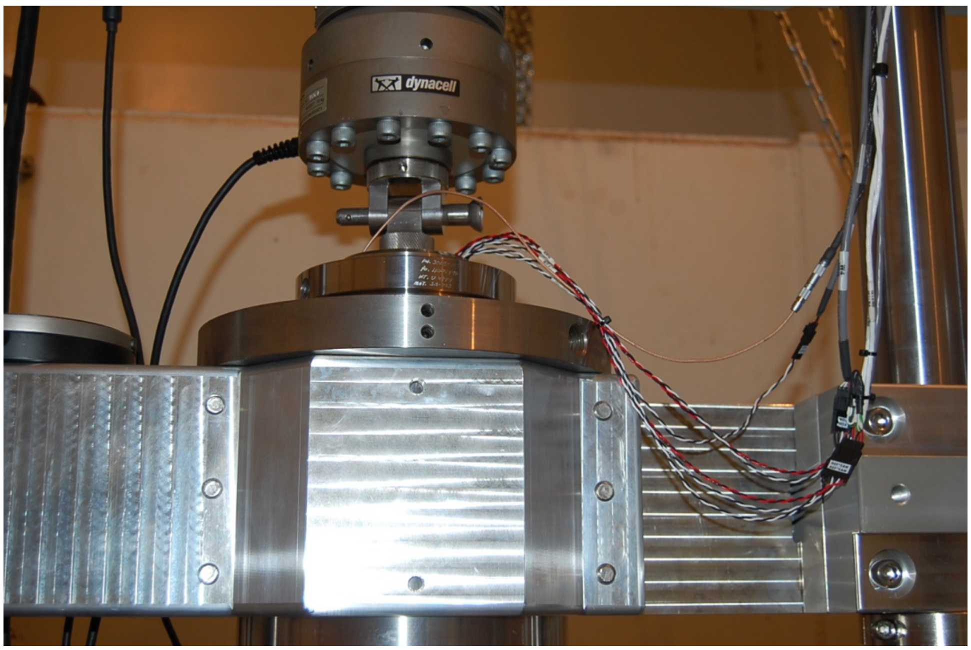


Figure 3. Detail of upper clevis grip designed for removal of vessel cap during specimen insertion & removal.

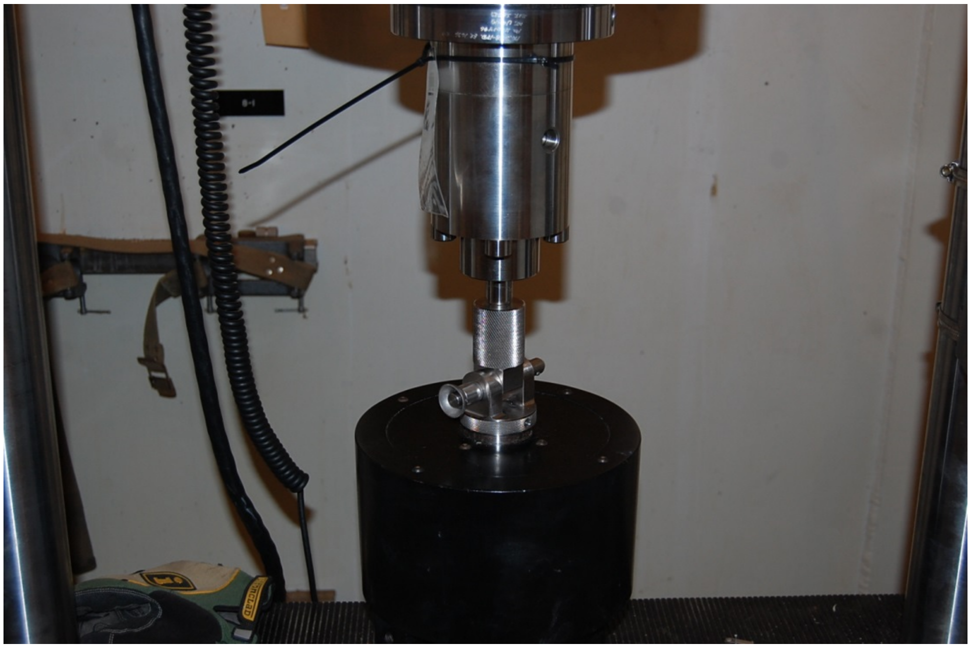
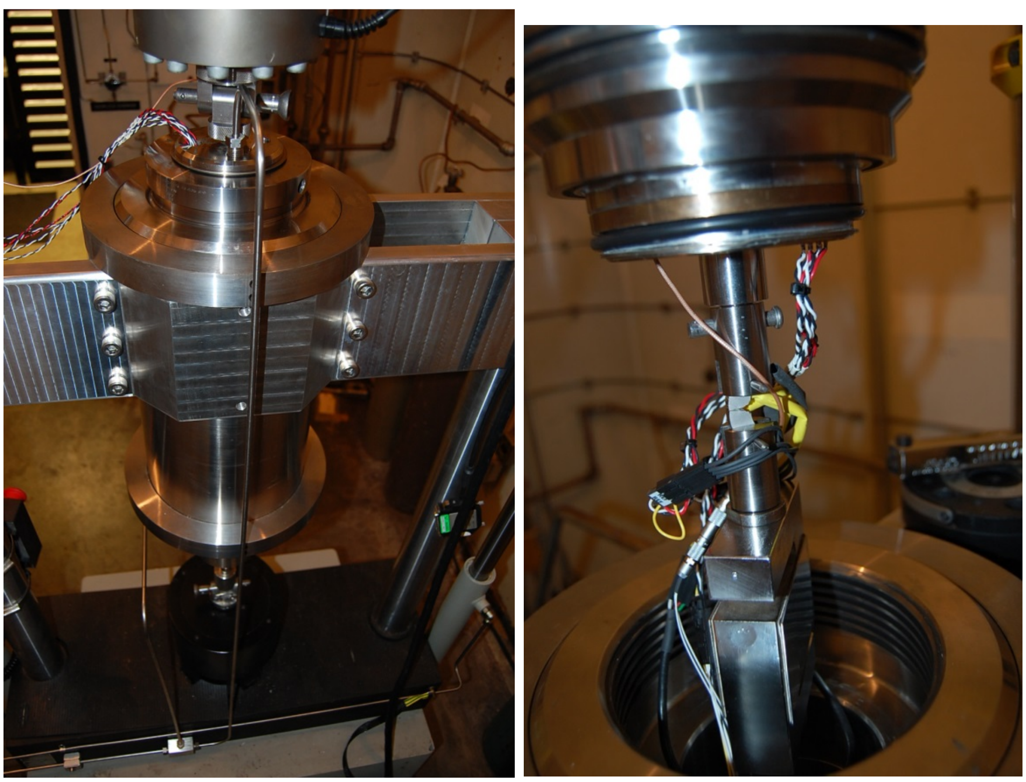


Figure 4. Detail of lower clevis grip designed for connecting lower pull rod to Instron actuator.



(a) (b)

Figure 5. (a) High-pressure connections to Autoclave vessel and (b) Arc-shaped-specimen installed in grips and raised out of vessel.

Another activity conducted during FY14 was to fabricate and install the high-pressure tubing, pressure gage, and automatic valve and connections from the vessel to high-pressure manifold. Along with these hardware additions, the details of the vessel evacuation, hydrogen flush and fill operations were developed. The gas handling procedures are very similar to procedures that have been safely used for years for hydrogen charging samples at elevated pressures and temperatures in the same facility. The details will be listed in a new work instruction that details the vessel assembly and disassembly, sample insertion and removal, and the gas handling operations. Figure 5 shows the high pressure connection to the autoclave and a specimen wired up for the fracture mechanics test in line with the internal load cell before it is lowered into the vessel for the test in the high pressure gas environment.

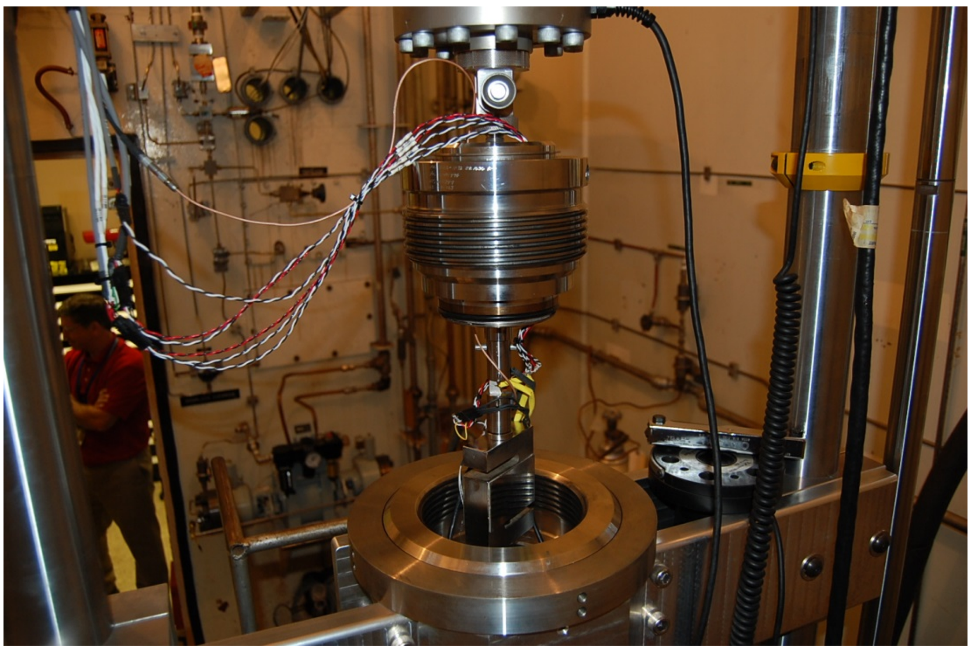


Figure 6. Specimen as connected to external electrical feedthroughs and installed in grips connected to vessel cap.

The fracture mechanics experiment in high pressure gas requires associated hardware within the Autoclave and the ability to read the data signals with a high-pressure electrical feedthrough. An internal load cell is required to measure load on the specimen that is independent of pressure and volume changes during the mechanical test. Crack length monitoring wiring is needed on the specimen and a reference specimen. The crack length monitoring is a four-probe technique that alternates the flow of a DC current in and out of each specimen while measuring voltage across the crack mouth. Crack length changes are inferred from the specimen resistance changes. Also required is a gage for specimen displacement and thermistor for temperature monitoring. The 15 signal wires were designed with connections that are easily snapped together for easy specimen insertion and removal and are shown in Figure 6.

A custom-designed DC Potential Drop Crack Length Monitoring System that has been successfully used in the past was modified for use within the high-pressure cell. Spot welding is used to attach four wires to each specimen under test that mate up to the external feedthroughs. A new LabView® data acquisition system was purchased and assembled. The custom software program was developed for collecting, sample voltage, reference voltage, external and internal load cells, clip gage and actuator displacement, temperature, and pressure. Figure 7 shows the typical display that is used during the fracture mechanics tests to monitor the various data channels. Figure 8 shows the Instron Control hardware and the Potential Drop Crack Length Measurement System Hardware that is designed to remotely conduct an experiment from the control room outside of the high-pressure cell.

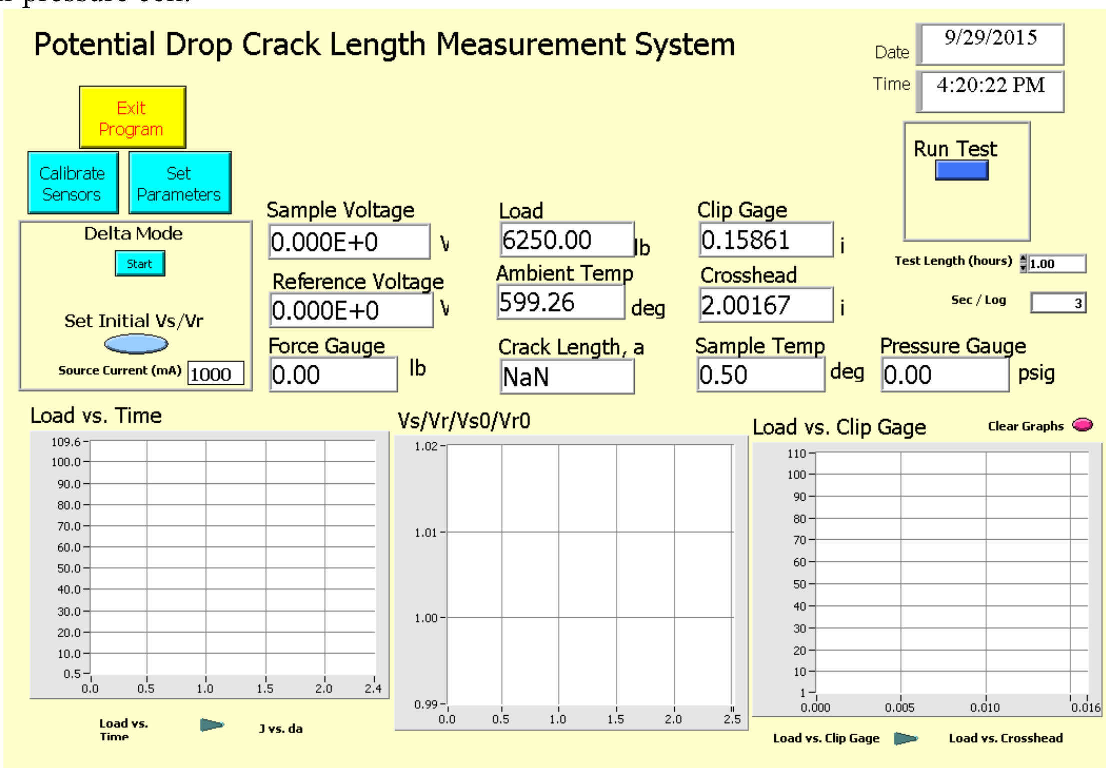


Figure 7. Computer display showing custom LabView® data acquisition system for collecting, sample & reference voltage, external and internal load cell, specimen and actuator displacement, temperature, and pressure signals.

Figure 8. Instron computer control located outside of Cell 1. Also shown is the data acquisition systems and Electric-Potential-Crack-Length-Measurement system.

A fracture mechanics demonstration test was conducted to verify operation of the completed system. A Type 21-6-9 stainless steel specimen was wired up and installed into the specimen grips attached to the underside of the vessel cap. The internal load cell was hung from the bottom of the specimen and connected to the internal bottom pull rod. All signal wires were connected up to the external feedthroughs and verified. The specimen was lowered into the vessel and the vessel sealed closed. The top of the vessel was reconnected to the upper crosshead and the bottom pull rod connected between the internal pull rod and the bottom actuator. The vessel was then backfilled with helium gas at 145 psi and a fracture mechanics test conducted. Figure 9 shows the measured load-displacement diagram measured during the test along with the crack length monitoring signals. The data were used to calculate and determine the ASTM fracture toughness properties shown in Figure 10.

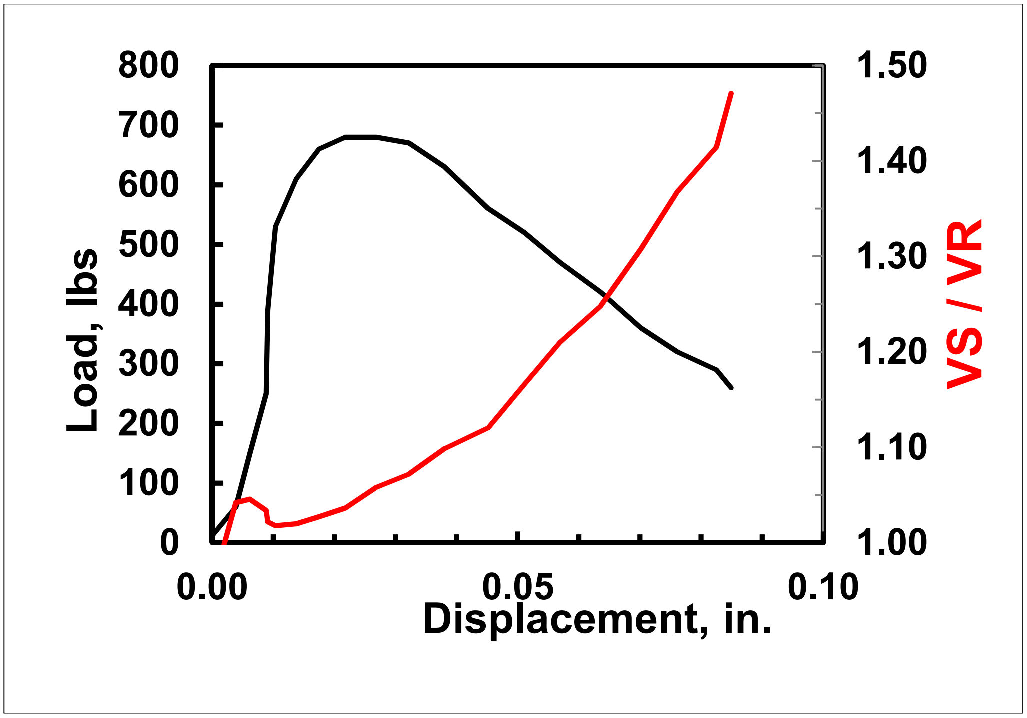


Figure 9. Example of data collected during fracture mechanics test conducted within the Autoclave vessel under 145 psi helium gas.

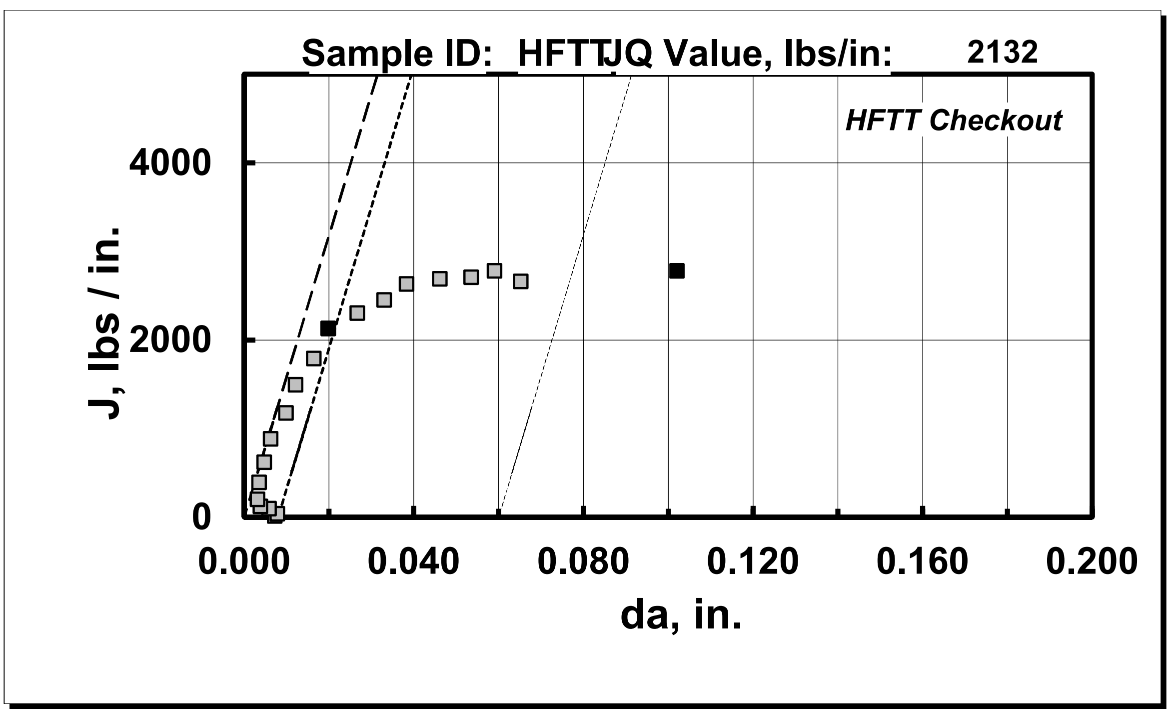


Figure 10. ASTM Fracture Mechanics Test Result Showing J-Integral vs Change In Crack Length (da). The Intercept of the Curve with an Offset Line is Used to Determine the Material Fracture Toughness Value (2132 lbs/in).

IV. FY16 Plans and Deliverables

A number of tasks are planned for FY16. First, leak rates during fracture mechanics tests at various elevated pressures will be closely monitored and characterized. Small gas leakage is expected from the sliding seals during the mechanical test. These leak rates will be quantified and used to monitor seal wear and aging. The vendor will be consulted for seal replacements and schedules. If required, an automatic control valve operation will be incorporated into the system control system to maintain constant pressure during the fracture toughness tests.

Once a number of trial tests are completed, the fracture toughness properties of block, cup and stem forgings in high-pressure hydrogen will be measured. Samples have been prepared from Types 304L and 316L stainless steel stem, block, and cup forgings. The data collected from the tests in high-pressure hydrogen will be compared with data collected during from tests on similar samples that have been exposed to tritium gas.

Another goal is to measure fracture toughness properties of Type 304L steel and its weldments in high-pressure hydrogen. These properties have been measure for hydrogen-charged specimens tested in air but not for specimens tested in high pressure gas. The fracture toughness properties could be significantly different for samples tested in high pressure gas because of the fast hydrogen diffusion paths associated with weld ferrite in the microstructure.

A number of upcoming material studies may require alternative specimen designs. Round and Square Compact tension specimens may require different specimen grips for tests within the high-pressure vessel. These grips and pull rods will be designed and fabricated. Also, improved displacement measurement techniques for tests in high-pressure gas will be required. These techniques will also be developed as more experience with the system is acquired.

Table 1 shows the FY16 Plans and Deliverables, to be accomplished as part of the ongoing ESC-funded aging studies effort.

Table I HFTT FY16 Plans and Deliverables

Task Description	1Q	2Q	3Q	4Q
Quantify leak rates from during fracture mechanics tests at elevated pressures				
If required, develop automatic control valve operation based for constant tests at constant pressure				
Measure fracture toughness properties of block, cup and stem forgings in high-pressure hydrogen.				
Measure fracture toughness properties of Type 304L steel and its weldments in high-pressure hydrogen.				
Fabricate Specimen Grips for Compact Tension Specimens				
Improve displacement measurement techniques for tests in high-pressure gas.				

VI. SUMMARY

A new capability for measuring the fracture toughness properties of materials in high-pressure hydrogen gas is installed in Laboratory 774A-Cell 1. The HFTT includes an Instron Model 8862 load frame and an Autoclave Engineering pressure vessel operating at pressures up to 20,000 psi. A pressure vessel mounting bracket was installed on the machine and then the Autoclave Engineering pressure vessel was mounted onto the machine. The HFTT is operated remotely from a control room and will be used for conducting fracture mechanics tests in high-pressure hydrogen gas. The operations for vessel assembly/disassembly, sample insertion/removal, and vessel pressurization were demonstrated. Initial fracture mechanics test procedures were developed and tests conducted in helium gas while operating remotely. Tests in high-pressure hydrogen gas are scheduled for FY16.

VI. ACKNOWLDEGEMENTS

Jim Wilderman was invaluable for his help in getting the HFTT in operation and assisting with the assembly/disassembly, specimen insertion/removal and test procedure development. Annamarie MacMurray, Chris Schifer, and Dean Thompson designed the pull rods and mechanical test fixtures and Annamarie was extremely helpful in coordinating the project completion activities. John Mcintosh built the data acquisition systems and wrote the LabView software for conducting the experiments. The R&D Machine Shop and High Pressure Laboratory provided rapid turnaround and response in mechanical hardware fabrication and testing.

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