

LWRS Fuels Pathway: Engineering Design and Fuels Pathway

Initial Testing of the Hot Water Corrosion System

John Garnier and Kevin McHugh

September, 2012



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Abstract

The Advanced LWR Nuclear Fuel Development R&D pathway performs strategic research focused on cladding designs leading to improved reactor core economics and safety margins. The research performed is to demonstrate the nuclear fuel technology advancements while satisfying safety and regulatory limits. These goals are met through rigorous testing and analysis. The nuclear fuel technology developed will assist in moving existing nuclear fuel technology to an improved level that would not be practical by industry acting independently. Strategic mission goals are to improve the scientific knowledge basis for understanding and predicting fundamental nuclear fuel and cladding performance in nuclear power plants, and to apply this information in the development of high-performance, high burn-up fuels. These will result in improved safety, cladding, integrity, and nuclear fuel cycle economics. To achieve these goals various methods for non-irradiated characterization testing of advanced cladding systems are needed. One such new test system is the Hot Water Corrosion System (HWCS) designed to develop new data for cladding performance assessment and material behavior under simulated off-normal reactor conditions. The HWCS is capable of exposing prototype rodlets to heated, high velocity water at elevated pressure for long periods of time (days, weeks, months). Water chemistry (dissolved oxygen, conductivity and pH) is continuously monitored. In addition, internal rodlet heaters inserted into cladding tubes are used to evaluate repeated thermal stressing and heat transfer characteristics of the prototype rodlets. In summary, the HWCS provides rapid ex-reactor evaluation of cladding designs in normal (flowing hot water) and off-normal (induced cladding stress), enabling engineering and manufacturing improvements to cladding designs before initiation of the more expensive and time consuming in-reactor irradiation testing.

Summary

The Hot Water Corrosion System (HWCS) apparatus was successfully designed, constructed and demonstrated to have sufficient flexibility to access the performance of a variety of advanced cladding materials and designs. Data from the HWCS tests will support down-selection of these materials and designs and provide baseline data for future evaluation of clad performance under irradiation. Several preliminary runs were conducted to assess system performance, and issues were resolved. Operational testing of the HWCS has been initiated with the first 3-day and 10-day runs. Test runs of 3 days, 6 days, and 10 days were concluded which included a Zircaloy-4 rodlet for baseline measurements and two SiC CMC hybrid tubes (SiC CMC external over-braid with Zircaloy-4 liner tube). The prototype testing also provided an opportunity for establishing operational procedures and personnel training and will establish standard test procedures.

Acknowledgements

In the achievement of the goals of the Advanced LWR Nuclear Fuel Development R&D pathway, rigorous testing and analysis is necessary including out-of-reactor systems such as the hot water corrosion system (HWCS) designed to develop new data for cladding performance assessment and material behavior under simulated normal and off-normal reactor conditions. It is the subject of this technical report. The Co-PIs Drs. Kevin McHugh and John Garnier appreciate this opportunity to acknowledge various team members during design, set-up and initial operation of the hot water corrosion system (HWCS) including technical instrumentation support from James Lee and Matt Weseman; INL machining support led by Adam St. Clair; Michael Teague (Modeling); Dr. Isabella Van Rooten (Characterization); Dr. Shannon Bragg-Sitton (current Program Manager), and Dr. George Griffith (former Program Manager). A special thanks to summer interns Jessie Johns (Texas A&M) and Tristan Griffith (Skyline high school).

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ACRONYMS

ATR	Advanced Test Reactor
CMC	Ceramic Matrix Composites
CuO	Copper (II) Oxide
DAS	Data Acquisition System
DO	Dissolved Oxygen
EDS	Energy Dispersive Spectroscopy
HNO ₃	Nitric Acid
HWCS	Hot Water Corrosion System
LED	Light Emitting Diode
LWR	Light Water Reactor
LWRS	Light Water reactor Sustainability
NO	Nitric Oxide
SEM	Scanning Electron Microscope
SiC	Silicon Carbide
SS	Stainless Steel
VAC	Voltage-Alternating Current
Zr-4	Zircaloy-4
Zr-702	Zircadyne® Alloy

1. Apparatus Design and Function

In support of the LWRS Program, a Hot Water Corrosion System (HWCS) has been designed and constructed at the Idaho National Laboratory to characterize the corrosion behavior, wear behavior, thermal properties, and chemical stability of cladding materials and cladding designs that are leading candidates for advanced fuel cladding applications. The system can assess the performance of these cladding materials and designs under a variety of simulated flow and internal heating conditions that mimic operational reactor conditions. A design drawing is shown in **Figure 1**, a photograph of the operational system is provided in **Figure 2**, and a schematic drawing of the flow loop is given in **Figure 3**.

The HWCS system is designed to expose cladding materials to heated, pressurized water ranging from 70 to 212°F (21 to 100°C), pressures to 40 psig (276 kPa), and volumetric flow rates up to about 400 gpm (1,514 l/min). Flow velocity and Reynolds number at the exposed surface of the cladding samples are adjustable over a wide range. A variety of diagnostic sensors are used to evaluate temperature, system pressure, volumetric flow rate, and water chemistry characteristics. The system can be divided into the flow loop (plumbing) subsystem, instrumentation/data acquisition subsystem, and power control/ interlock circuitry subsystem. In general, the HWCS is designed for the following modes of operation:

1. Corrosion-only: heated water with internal rodlet heating available.
2. Thermal stress: heated water with internal rodlet heating.
3. Thermal conductivity/heat flux: variable water temperature with internal rodlet heating.

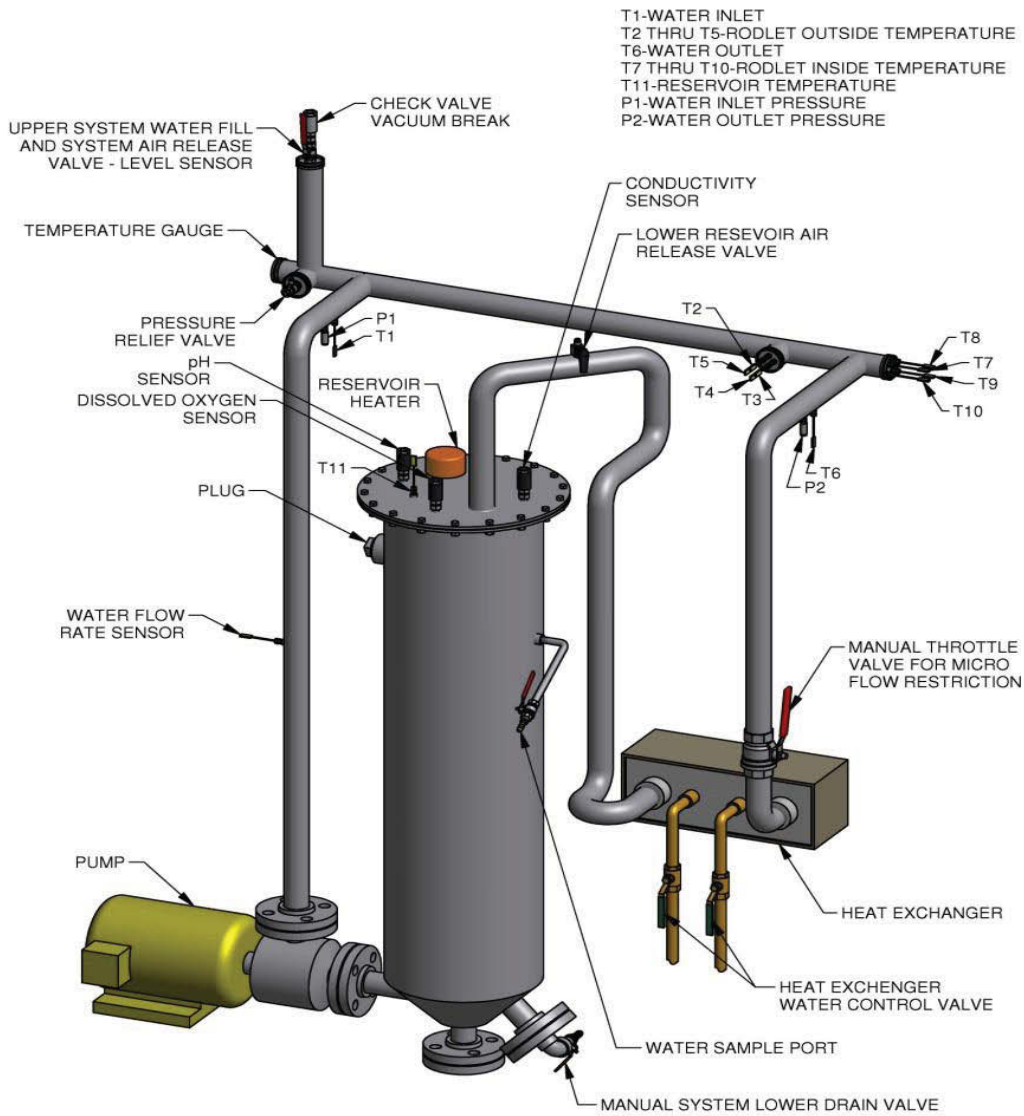


Figure 1. Design drawing of the HWCS apparatus.

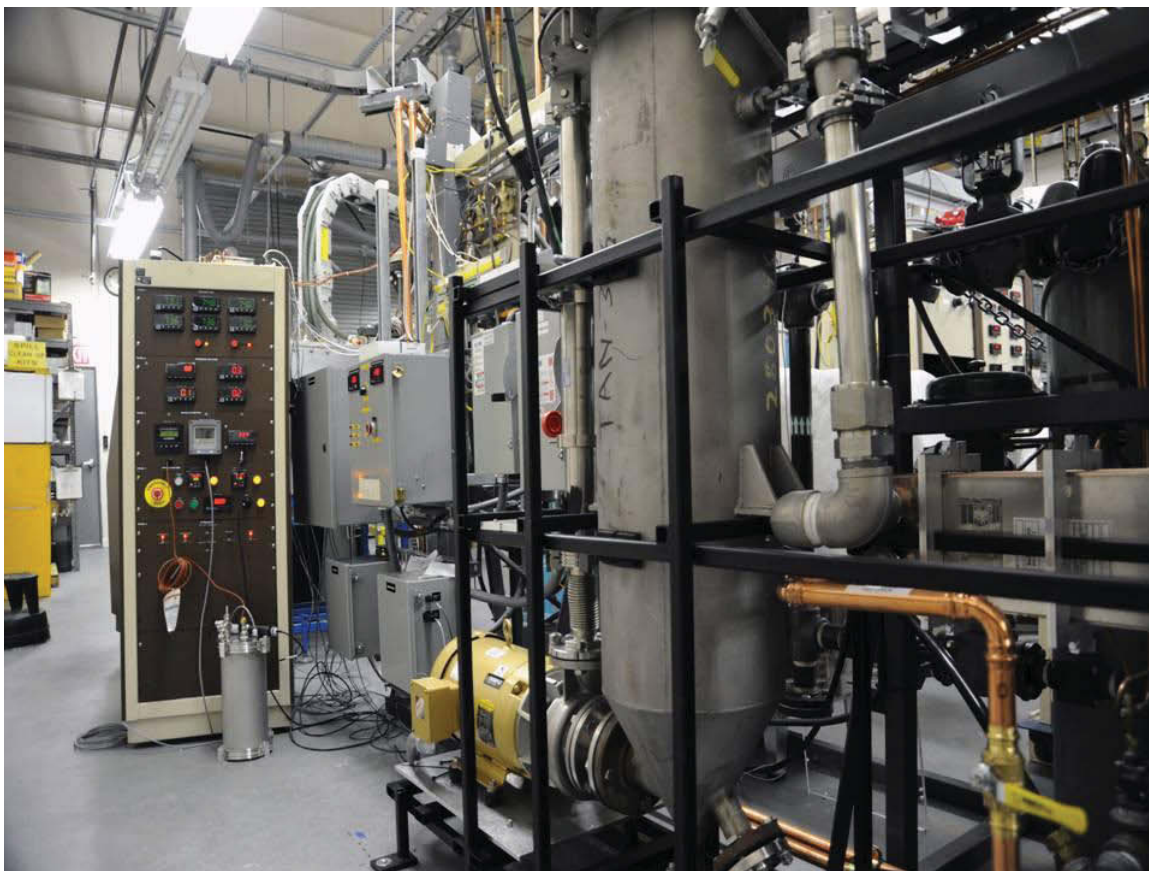


Figure 2. Photograph of the HWCS during operation.

Tests can be run in an automatic or an “unattended” condition for long periods of time (e.g., hours/days/weeks) with minimal operator supervision, while automatically recording relevant data from system sensors. Operator pre-set alarms and safety interlocks are used to activate water pump and heater shutdown should an off-normal event occur (such as a power outage or water leak). Set points are established for water level, temperature, pressure, and flow rate.

Thermal stress tests incorporate internal cartridge heaters to impart thermal stress to the tube walls, similar to the conditions in an operating reactor. Thermal stress tests require manual operation to vary the internal rodlet temperature via heater current adjustments. Individual heater elements can be operated at up to 1 kW. A summary of the main experimental parameters and operational ranges of the HWCS are given in Table 1.

Table 1: HWCS Test Parameters and Ranges.

#	Test Parameter	Range	Comments
1	Test section	2.5 inch ID x 38 inch long	Test section can accommodate internal flow constrictors such as sample baskets used at the ATR. Currently up to four samples. Larger diameter piping samples (3 to 20 in.) allows more test.
2	Test Time	unlimited	System can operate unattended indefinitely.
3	Water Temperature	70 to 212 °F	Broad range to simulate reactor input temperature to fuel rods under steady-state and off-normal conditions. Flexibility for thermal stress and heat transfer measurements.
4	Volumetric Flow	0 to 400 gpm	Low flow (2.5 to 29 GPM) and high flow (25 to 400 GPM) turbine sensors. Flow velocity past samples is geometry dependent.
5	Flow velocity	0.1 to 100 ft/s	Calculated from sample geometry and measured volumetric flow rate.
6	Reynolds Number	laminar to turbulent	Reynolds number determined using calculated flow velocity in sample test section and hydraulic diameter.
7	Water pressure	0 to 40 psig	Pump operating pressure limited to about 40 psig. Plumbing rated to several hundred psig. Pump water head capacity 135 to 70 head feet.
8	Water chemistry	pH: 0 to 14. DO: 0 to 1000 ppm Conductivity: 0 to 1000 µS/cm.	On-line, continuous monitoring of pH, conductivity and dissolved oxygen. Off-line analysis to calibrate sensors. Operational range depends on selected probe range. Ion concentrations and impurities evaluated off-line.
9	Thermal Stress, heat flux and Conductivity of Cladding	70 to 1830 °F	1 kW internal heaters. Simultaneous measurement of internal and external surfaces of cladding.

1.1 Flow Loop Subsystem

The flow loop subsystem consists of a closed-loop piping system with a pump, reservoir, heat exchanger, pressure and vacuum relief valves, fill port and drain, viewport, and horizontal test section which can contain up to four prototype cladding rodlets of lengths up to ~24 inches.

About 30 gallons (114 liters) of high-purity water is introduced into the system through a port. Venting ports are used to degas the system as water is heated to the desired temperature and flow rate.

The piping through which the water flows is constructed primarily of stainless steel alloys 304 and 316L. Viton o-rings provide leak-tight seals between piping sections. Water is moved through the 2.5 inch piping where the samples are tested using a high capacity, re-circulating pump (Chemflo 6) capable of moving 450 gallons per minute at 60 psig. The pump is powered by a 10 HP motor.

The water chemistry, temperature, and flow dynamics in the test section of the loop are currently established to be representative of the primary coolant loop of INL's ATR reactor. These parameters can be varied to simulate other reactor conditions. Temperature compensated sensors mounted in the 20 gallon reservoir continuously monitor pH, dissolved oxygen and conductivity. This water can be readily changed as necessary or sampled while the HWCS is operational by filling a sampling vessel using a valve located in the reservoir. This sampling vessel is usually used to obtain water for off-line measurement of the concentrations of dissolved ionic species and solid particulate levels. It is also equipped with calibrated pH, DO, and conductivity probes to verify accuracy of the probes mounted in the reservoir.

Custom hardware (referred to as the "basket") has been installed in the horizontal test section to establish the flow velocity and Reynolds number consistent with the ATR. To accomplish this, a 35" long x 2.25" OD 6061 Al rod (**Figure 4**) was machined with three evenly spaced, parallel, 0.5" diameter through holes (**Figs. 4b** and **4c**), and installed in the HWCS to house the individual rodlets. This geometry is the same (diameter and length) as an existing basket in the ATR. Aluminum spacers (**Figure 4d**) are placed in the flow channels of the basket upstream of rodlet samples to provide water flow conditions found in the ATR.

Figure 5a is a photograph of rodlet samples inside the flow channels of the aluminum basket. Each rodlet passes through the water tight seal of a 3/8" ultratorr fitting which in turn are mounted on a removable flange, allowing easy retrieval of the rodlets (**Figure 5b**). **Figure 5c** shows an assembly of two SiC CMC rodlets and one Zr-4 rodlet removed from the basket. Welded end caps on the inlet end of each sample help locate them along the centerline of the flow channels.

Each tubular rodlet can be fitted with a 1 kW internal cartridge heater to simulate heat produced by fission. These heaters find utility during thermal stress, thermal conductivity and heat flux measurements as well as corrosion evaluation. A heat exchanger, situated downstream of the cartridge heaters, is used to extract this additional heat from the system, while a 4.5kW heater, located in the reservoir, maintains the water temperature at the inlet to the rodlets at the desired value.

Water flow through each channel of the basket was measured using the assembly shown in **Figure 5d**. O-rings sealed the inlet end of each tube match the ID of the flow channels in the

basket. The volume of water discharged through each flow channel per unit time, was found to be the same. The total volume of water discharged per unit time was used to verify calibration of the volumetric flow meter.

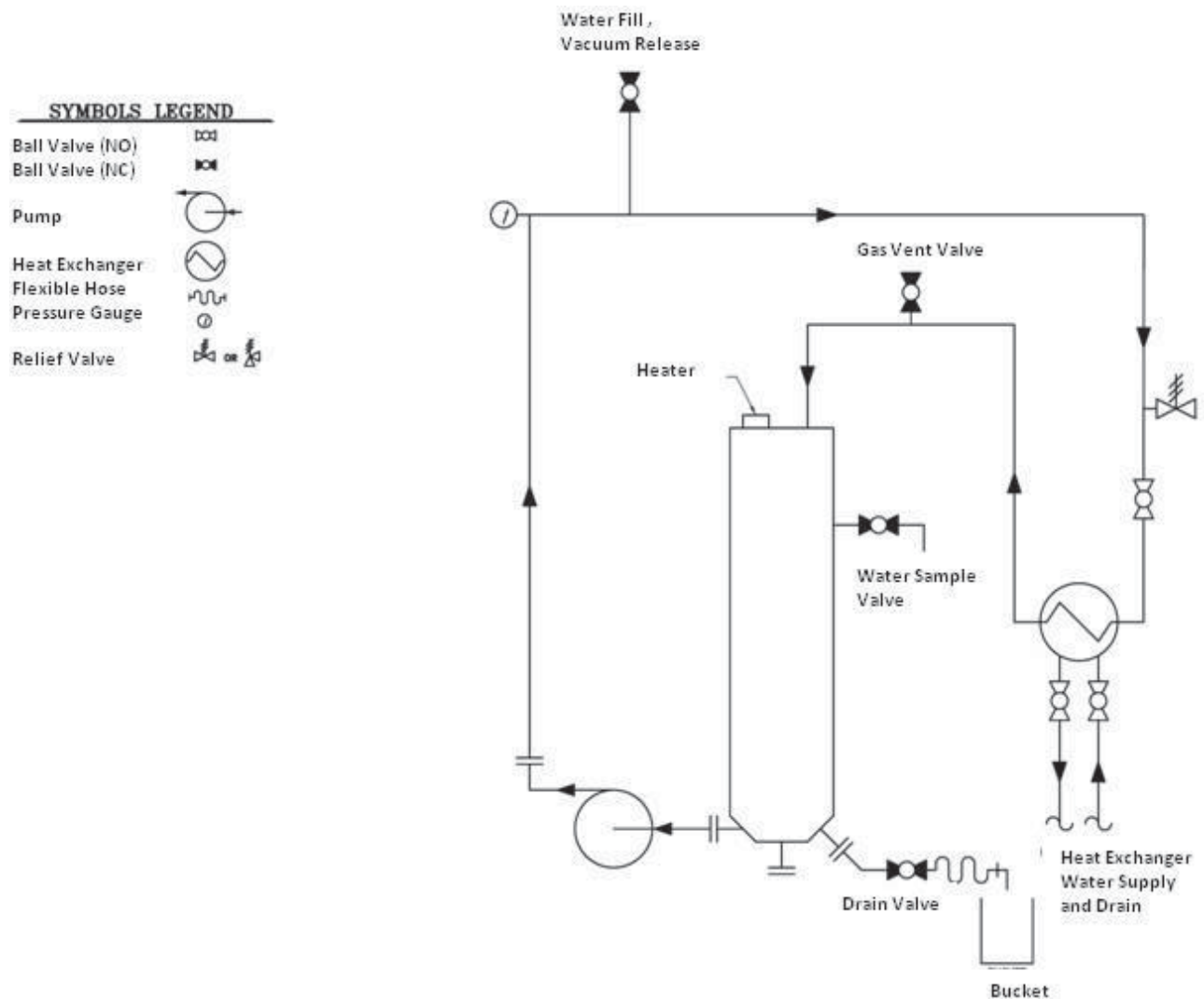


Figure 3. Schematic diagram of the flow loop.

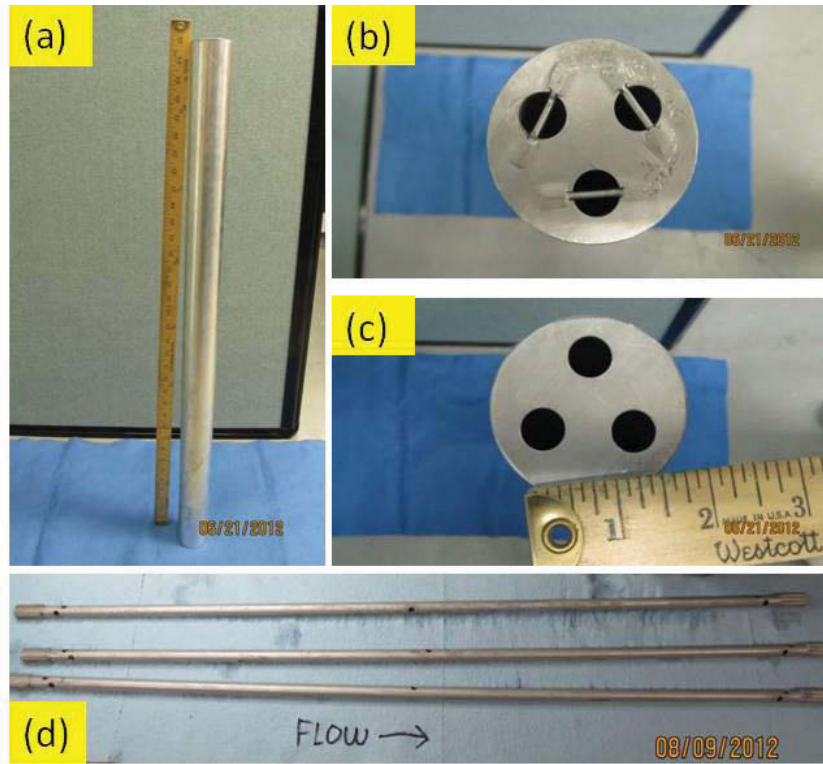


Figure 4. Aluminum “basket” design used to simulate flow conditions in the ATR. (a) Photograph illustrating overall length. (b) Inlet to basket. Pins are used to retain inserts during placement. (c) Outlet of basket showing flow channel geometry. (d) Aluminum spacers.



Figure 5. (a) Two rodlet samples are placed inside flow channels of basket. (b) Rodlets adaptor flange installed in system. (c) Two SiC CMC samples and one Zr-4 sample after removal from system. (d) Diagnostic tool used to measure flow through each of the basket channels.

A fluid dynamics code was developed to calculate the Reynolds number and flow velocity at the center of the rodlets when placed inside the Al basket. The code assumes steady-state, incompressible, streamline and irrotational flow while solving the mass conservation equation. Using geometry inputs for the rodlets, flow channels of the basket, and bypass gap (if any), the code calculates the required volumetric flow rate for the desired flow velocity or Reynolds number. A screen-capture image of the code is shown in **Figure 6**.

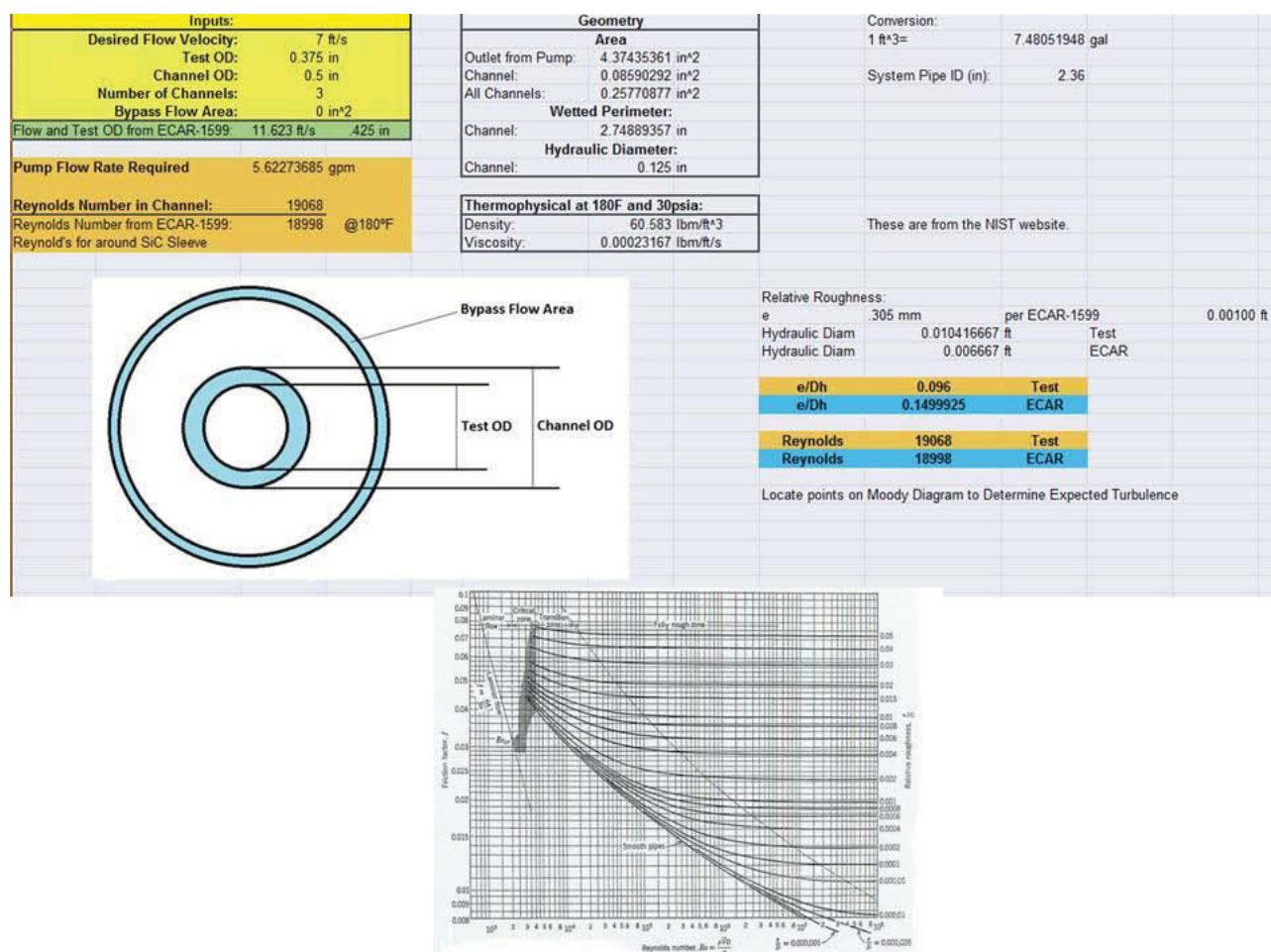


Figure 6. A screen-capture image of the computer code used to calculate Reynolds number and flow velocity past the rodlets.

1.2 Instrumentation/Data Acquisition Subsystem

A variety of sensors and other diagnostic tools are employed in the HWCS to continuously monitor system parameters. These tools, described below, evaluate temperature, pressure, flow, and certain chemistry characteristics of the water (conductivity, pH, and dissolved oxygen). In addition, water samples are periodically extracted and evaluated off-line for concentrations of

ionic species and solid particulates. Data from these sensors will support down-selection of advanced cladding materials and cladding designs for future evaluation of cladding performance under irradiation.

1.2.1 Temperature, Pressure, Flow

Eleven Type K grounded junction thermocouples are employed to measure temperatures at various locations in the HWCS. Thermocouples located upstream and downstream of the rodlets measure the water's temperature rise due to heat input from the 1 kW internal rodlet heaters. The temperature of the water contained in the 20 gallon reservoir is measured and is used for closed-loop control of heat flux into the system via a 4.5 kW immersion heater also located inside the reservoir. This heater establishes a constant water temperature at the inlet to the rodlets. In addition, each rodlet is equipped with a thermocouple to measure the internal wall temperature of the cladding. Simultaneously, the outside wall temperature of each rodlet is measured using retractable thermocouples. The temperature gradient across the cladding is used to determine thermal stress characteristics, thermal conductivity and heat flux performance of a particular rodlet design. These characteristics are influenced by the materials and surface roughness of the cladding design. All temperature signals are cataloged by the data acquisition system.

Two pressure sensors (Omega PX209-60G5V), one located upstream and one downstream of the rodlets, provide a measure of the pressure drop across the rodlets. A high flow turbine meter (Omega FTB-410A) provides volumetric flow measurement up to 400 GPM, while a low flow turbine meter (Omega FTB-405A) provides greater reading sensitivity for flow rates up to 29 gpm. Flow velocity and Reynolds number at the rodlets are not directly measured, but are calculated based on the geometry of the rodlets, basket and measured volumetric flow rate, as described above.

1.2.2 Water chemistry

The primary coolant loop of a LWR utilizes very high purity water. Careful monitoring and control of water chemistry is critical for several reasons:

1. To minimize corrosion of reactor components including fuel cladding. Impurities in the water, pH conditions, oxygen level, etc., can greatly affect corrosion behavior.
2. To minimize radiation levels in a reactor facility. Some of the natural impurities and most of the corrosion products become highly radioactive after exposure to the neutron flux in the core region. If not removed, these soluble and insoluble substances may be carried to all parts of the system.
3. To minimize fouling of heat transfer surfaces. Corrosion products and other impurities may deposit on core surfaces and other heat transfer regions, which result in decreased heat transfer capabilities by fouling surfaces or blockage of critical flow channels.

Targeted water chemistry parameters, such as pH, are chosen based on the materials used in the reactor. For example, reactors containing Al components, such as the ATR, typically operate with lower pH water (pH~5) than most commercial reactors to minimize aluminum corrosion. The basket (see **Figure 4**) used to house the cladding samples in the HWCS, and to establish the appropriate water flow dynamics near the cladding samples, is constructed of aluminum alloy 6061, as it is in the ATR. Consequently, the chemistry of the nano-pure water added to the HWCS loop is modified to closely simulate typical ATR water chemistry: pH= 4.8-5.4, conductivity= 2.0-5.0 $\mu\text{S}/\text{cm}$, and dissolved oxygen = 7.0-13.0 ppm (lower is desirable). This is accomplished by treating the nanopure filtered water with a small amount (4 $\mu\text{l}/\text{gal}$) of a dilute solution of nitric acid.

No in-line treatment of water quality is currently employed in the HWCS to help stabilize water chemistry because the primary function of the system is to evaluate the corrosion and erosion behavior of candidate fuel cladding materials. Note, however, that in the ATR, several practices are used to purify the water in the systems and water used as makeup. Deaeration is used to strip dissolved gases, filtration is effectively used to remove insoluble solid impurities, and ion exchange systems remove undesirable ions and replace them with acceptable ions.

Chemistry changes to the water due to corrosion or leaching are documented. Temperature compensated probes mounted in the reservoir of the HWCS are used for continuous in-line measurement of conductivity (Rosemont Analytical 400 sensor), pH (Rosemont Analytical 3500VP sensor), and dissolved oxygen (Rosemont Analytical HX438 sensor). Water samples periodically drawn from the system are also evaluated for conductivity (Omega CDCE90-0001), pH (Omega PHE-7531-15-PT1K-PPS) and dissolved oxygen (Omega DOE-45PA) to verify calibration of the in-line sensors. Concentrations of ionic species are evaluated by periodically extracting water samples and measuring these concentrations using ion chromatography, emission spectroscopy, etc. Of particular interest are cation species such as Cl^- , NO_3^- , and OH^- ; anions such as Na^+ , Al^{3+} , Cu^{2+} and $\text{Fe}^{2+, 3+}$, as well as particulate concentrations.

1.2.3 Data Acquisition and Readouts

An instrumentation rack (**Figure 7**) houses digital readouts for each of the 0-5 VDC or 4-20mA signals from the various transducers (temperature, flow, pressure, pH, conductivity and dissolved oxygen). A readout is also provided in the instrumentation panel for the flow velocity by applying a scaling factor to the measured volumetric flow rate. The signals are filtered and sent to a computer equipped with Labview™ data acquisition software. The data acquisition system displays the signals superimposed on a schematic drawing of the apparatus. A screen-capture image of the display is illustrated in **Figure 8**.



Figure 7. (a)HWCS instrumentation and control rack. (b) Run time meters.

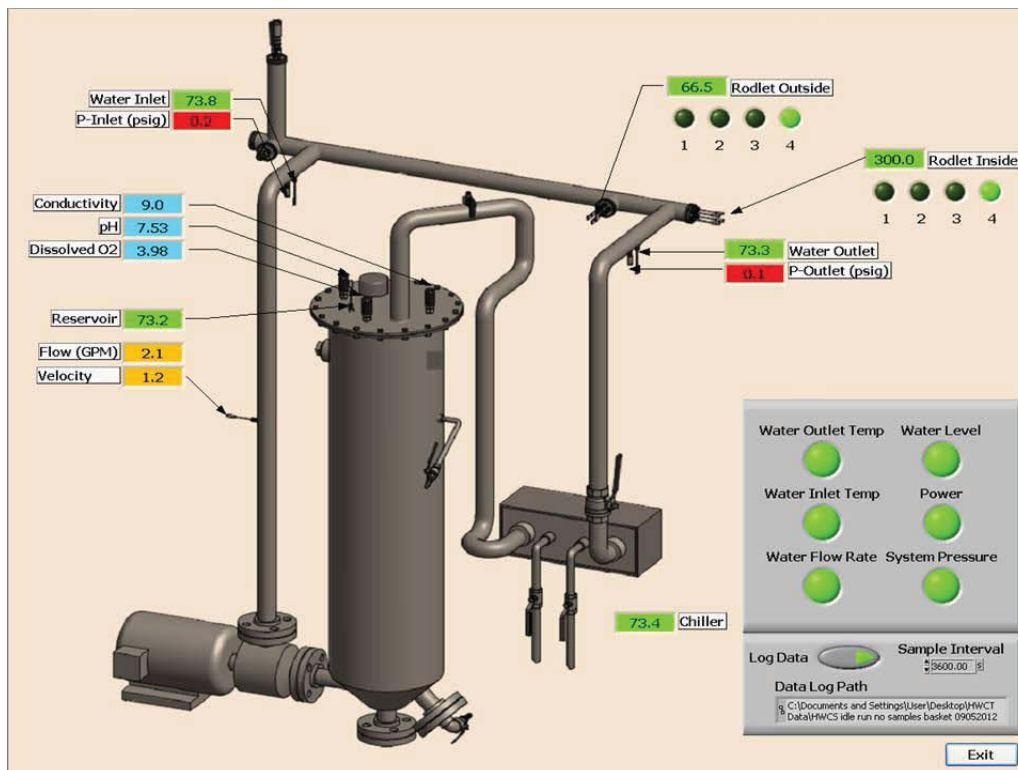


Figure 8. Screen-capture image of the data acquisition as it is currently displayed in the HWCS. Green lights in the lower right corner indicate the interlocks are active. An individual light will turn red alerting the operator of the location of a tripped interlock.

1.3 Control Circuitry and Interlock Subsystem

The instrumentation rack also houses control circuitry to power the pump and heaters as well as interlock features to protect the equipment during off-normal events and unattended operation. The 4.5 kW immersion heater (Watlow BLR710L5-21), located in the 20 gallon reservoir, is powered by a 3-phase, 480 VAC solid state relay that is controlled by a proportional-integral-derivative controller (Watlow Model PM6C1CJ). The PID controller attempts to minimize the difference between a measured inlet temperature to the rodlets and a desired setpoint by controlling the output voltage to the heater. Similarly, a 208 VAC, three-phase solid state relay/PID unit controls power input to the cartridge heaters that internally heat the rodlets. However, in this case, a 3-phase variac is used to limit current to the individual heaters, and provide greater rodlet temperature control and flexibility during operation.

The interlock circuitry is designed to de-energize power to the pump and heaters in the event of an off-normal incident. The power latches off and must be reset by an operator. An LED light displays where a failure occurred in the system. For example, if a plumbing leak occurs, the water level sensor will trip and an LED light will illuminate for that sensor. The interlocks protect the system in the event any of the following occur: power outage, low water flow rate, water over-temperature at the inlet to the rodlets, water over-temperature at the outlet from the rodlets, low water level in system, and system over-pressure. In conjunction with the latter, a check valve will discharge water to a drain if the pressure reaches unsafe levels. Dedicated switches mounted on the front panel allow each interlock to be overridden. Schematic diagrams of the HWCS control circuitry are provided in Appendix A.

2. Preliminary Runs and System Assessment

Prior to operation and data collection, a readiness check and calibration of all sensors, readouts, and data acquisition signals was performed. Thermocouple signals (emf voltages) to the instrumentation rack and data acquisition system (DAS) were simulated using millivolt input signals from a constant voltage source corresponding to 180°F. Instrumentation gains for each meter were optimized for that value. After installation of the basket, water flow measurements through each channel were conducted using the test device shown in **Figure 5d**. The gain for the volumetric flow meter was adjusted to maximize accuracy for flows similar to those used at the ATR, as suggested from turbulence and flow velocity calculations using the computer code described above (**Figure 6**). Water conductivity and pH probes were calibrated against commercial stock solutions. 10 $\mu\text{S}/\text{cm}$ solution was used for conductivity and pH 7 and pH 4 buffer solutions were used to calibrate pH probes. Commercial solution of 0 ppm oxygen was used to calibrate the lower limit of dissolved oxygen, while a known upper limit (water saturated with air) was used to set the upper limit. For all trial runs, water added to the HWCS was nanopure, filtered water with a small amount of dilute HNO_3 added to adjust the pH. Water

chemistry measurements at room temperature were typically pH~ 5, dissolved oxygen of ~5 ppm, and conductivity of ~3 $\mu\text{S}/\text{cm}$.

During an initial HWCS evaluation test, the system was run with stainless steel rodlet samples rather than Zr or SiC CMC rodlets. Two SS-304 and one SS-316L samples were loaded into the Al basket. The system was run for several days using a relatively high water inlet temperature of 180°F to accelerate corrosion affects. A similar discoloration pattern of each SS sample was observed (**Figure 9a**). The portion of the rodlets inside the aluminum basket was covered with a pink corrosion product that was somewhat loosely adherent (**Figure 9b**). During removal, portions had flaked off revealing the underlying silver rodlet. This suggested the pink surface coating had plated onto the surface of the steel. Outside the aluminum basket, corrosion of the SS-304 was the typical tan-colored discoloration (**Figure 9c**). This corrosion product was strongly adherent and thinner. No corrosion was observed outside the flowing water, as expected. While the overall corrosion patterns were similar, the amount of corrosion at the two flow channel locations was somewhat different. This suggested that the flow rate of water was somewhat different through the channels.

The corrosion products on the 316L rodlet were similar to those found on the SS-304 rodlets. Photographs are shown in **Figure 10** for the leading edge inside the aluminum basket (**Figure 10a**), outside the basket but in the flowing water (**Figure 10b**), and outside the flowing water (**Figure 10c**). Scanning electron microscopy (SEM) images of the corrosion products were obtained and energy dispersive spectroscopy was used to obtain information on their chemical composition.

SEM photomicrographs of the pink discoloration of **Figure 10a** are shown in **Figure 11** at several magnifications. The overall surface morphology is best illustrated at 200X in **Figure 11a**. Note the “bare” areas where the coating had dislodged. When viewed at progressively higher magnifications, the pink corrosion product was seen to consist of uniform, well-developed equiaxed crystals of about 1 μm diameter (**Figures 11b,c**). A side view of the coating is shown in **Figure 11d**.

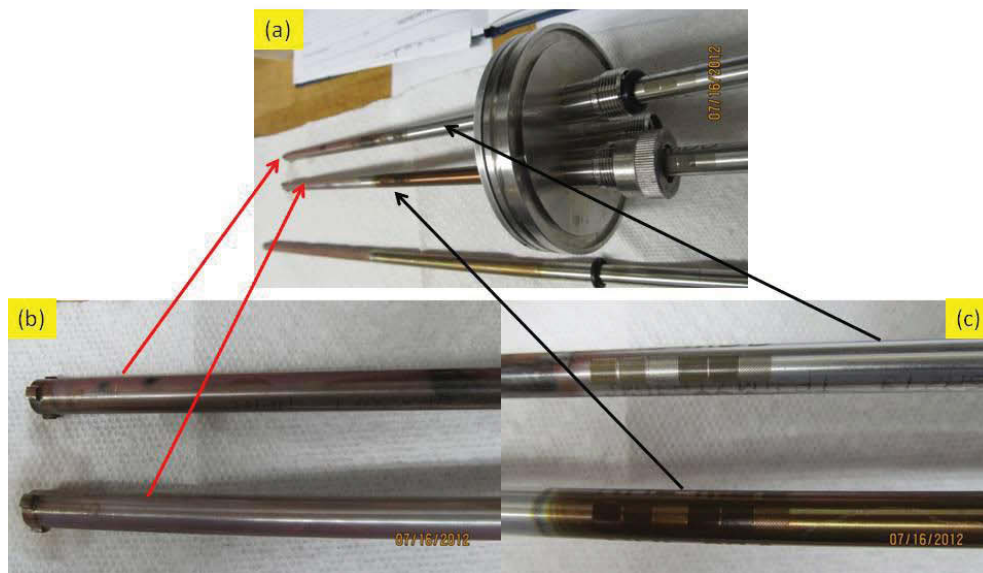


Figure 9. Corrosion patterns observed on SS-304 rodlets following 3-day run at 180°F. (a) Rodlets in sample holder after removal from the HWCS. (b) “Pink” corrosion on samples located in the 6061 aluminum basket. (c) “Tan” corrosion of rodlets outside basket but in water.

Energy dispersive spectroscopic (EDS) analysis of the crystals indicated the main constituent was elemental copper (**Figure 11**). Copper oxide and aluminum oxide impurities are also observed. The origin of the copper was the heat exchanger. The manufacturer used a copper brazing material used to join parallel stainless steel sheets inside the heat exchanger. It is postulated that two types of corrosion took place to give rise to the plating behavior. The first of these involved gradual dissolution of the copper brazing material at the heat exchanger by the weak nitric acid solution inside the heat exchanger:



The other corrosion reaction involved a galvanic reaction in which copper ions were reduced to elemental copper. Copper ions contained in solution plated out onto the SS (cathode) inside the aluminum basket (anode) of a Al/SS galvanic couple. Subsequent analysis of the inner diameter of the stainless steel housing for the aluminum basket also revealed the presence of plated copper and a black coating of cupric oxide. This elevated temperature corrosion test was successful at locating potential corrosion sources in the HWCS.

The heat exchanger was removed from the system for subsequent runs and replaced with a stainless steel pipe wrapped with a copper coil. In addition, the observed difference in the amount of corrosion on each rodlet led to an improved method to measure the flow (see **Figure 5d**), and ultimately, to a procedure to calibrate the volumetric flow meter.

Figure 13 illustrates, at several magnifications, SEM photomicrographs of the morphology of the tan colored corrosion on the stainless steel rodlets outside the aluminum basket. EDS elemental analysis (**Figure 14**) is consistent with a thin iron oxide film, and a background signal

of 316 stainless steel, which arises due to inherent sampling limitations of EDS. These results are typical of oxidized 316 stainless steel.

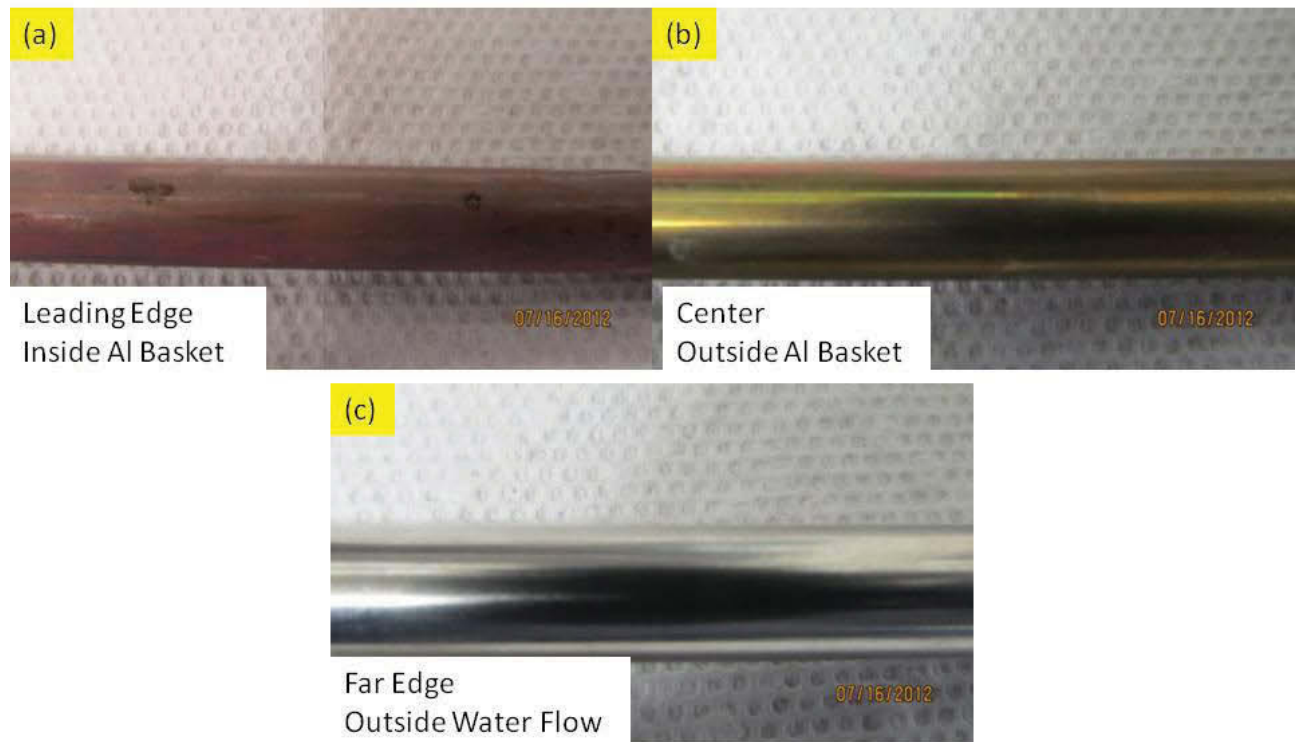


Figure 10. Photographs of corrosion patterns observed on a SS-316L rodlet following the 3-day run at 180°F. (a) “Pink” corrosion on samples located in the 6061 aluminum basket. (b) “Tan” corrosion outside basket but in water. (c) Corrosion was not observed on sample outside water flow.

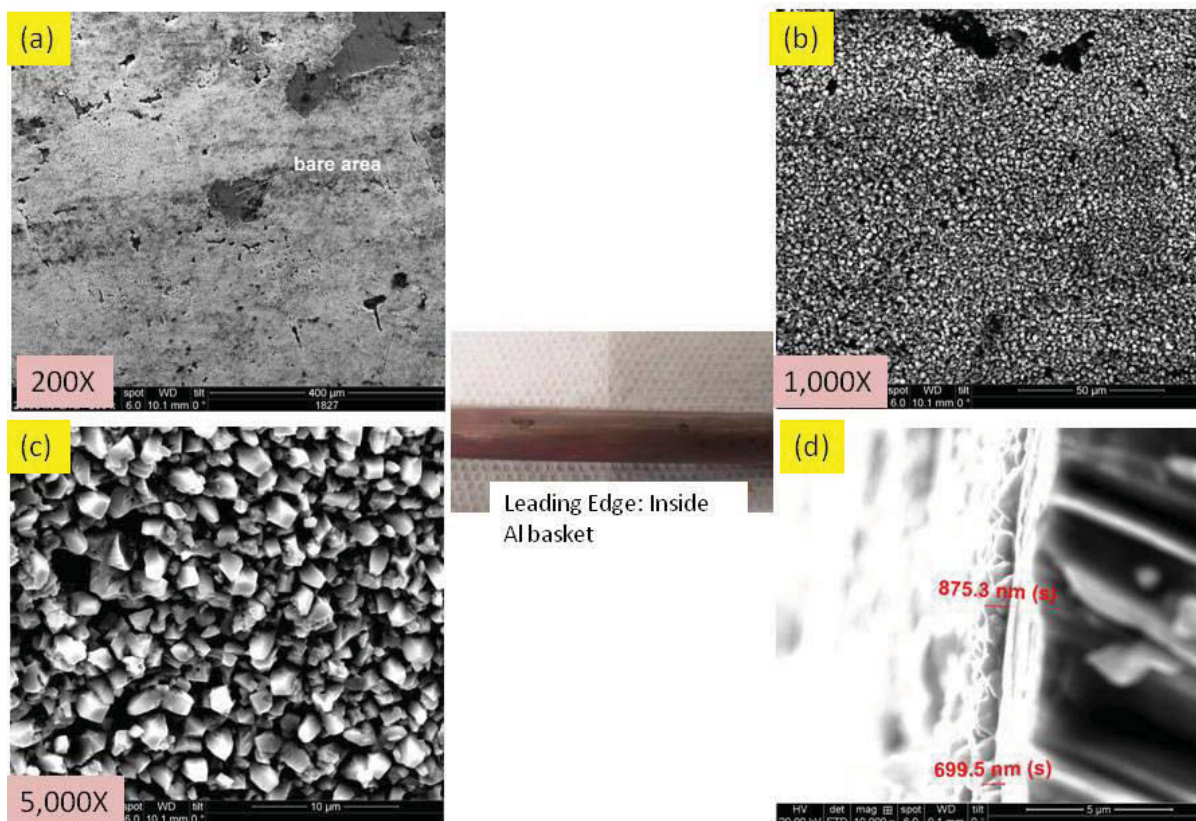


Figure 11. SEM photomicrographs showing the surface morphology of the “pink” corrosion product observed on stainless steel rodlets located inside the aluminum basket. (a) 200X photomicrograph illustrating overall surface appearance. Note “bare” areas where the coating had dislodged. (b) View at 1,000X. (c) 5,000 X photomicrograph showing equiaxed crystal growth on the surface of the rodlets. (d) Side view illustrating the coating thickness is approximately 800 nm thick.

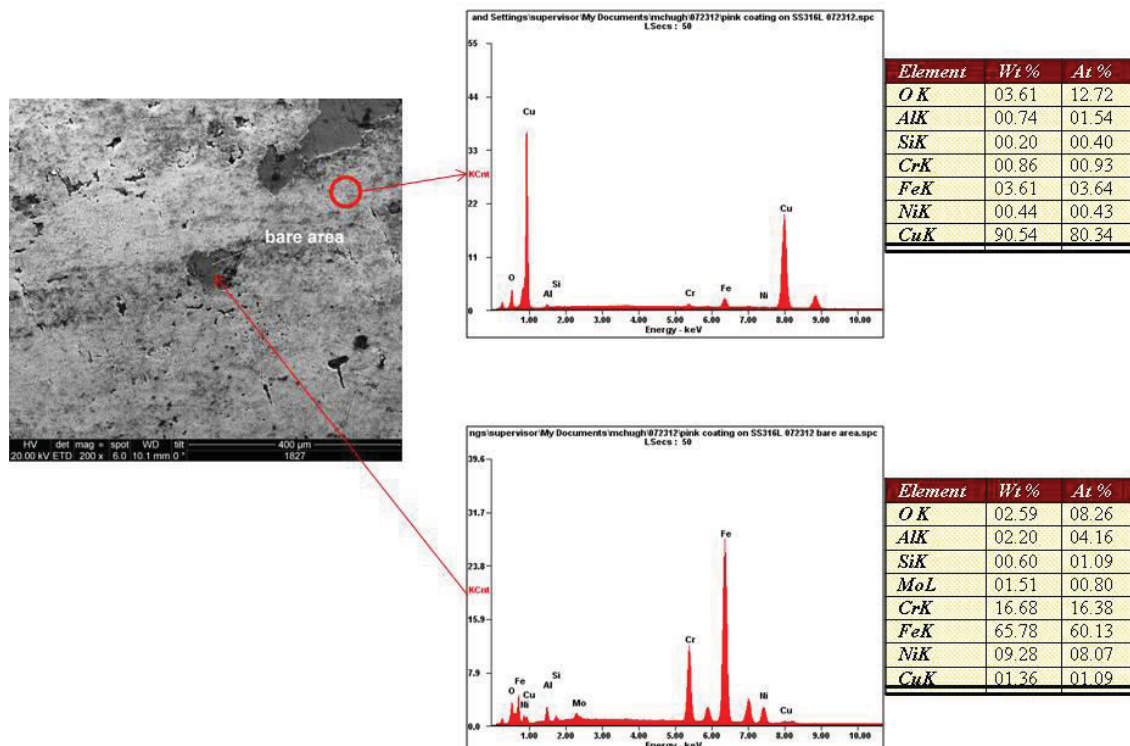


Figure 12. EDS analysis of “pink” coating and underlying stainless steel on the portion of rodlet inside the aluminum basket.

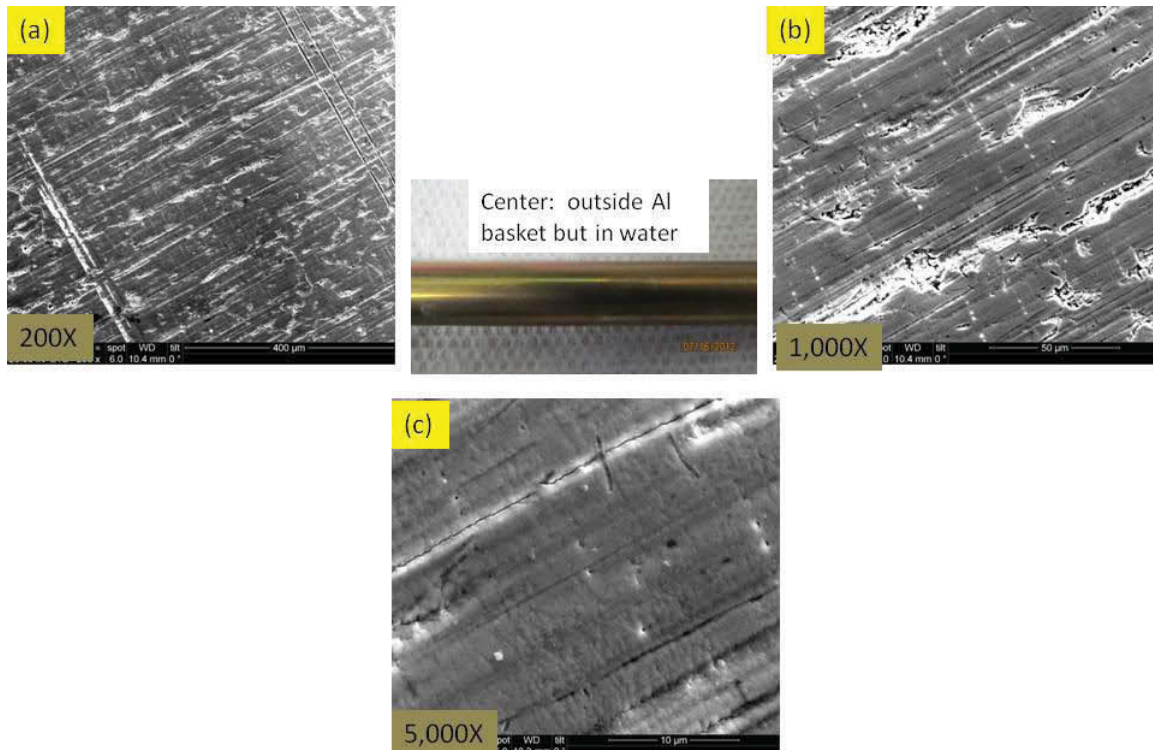


Figure 13. SEM photomicrographs illustrating the surface morphology of the “tan” corrosion product formed on stainless steel rodlets immersed in flowing water outside the basket.

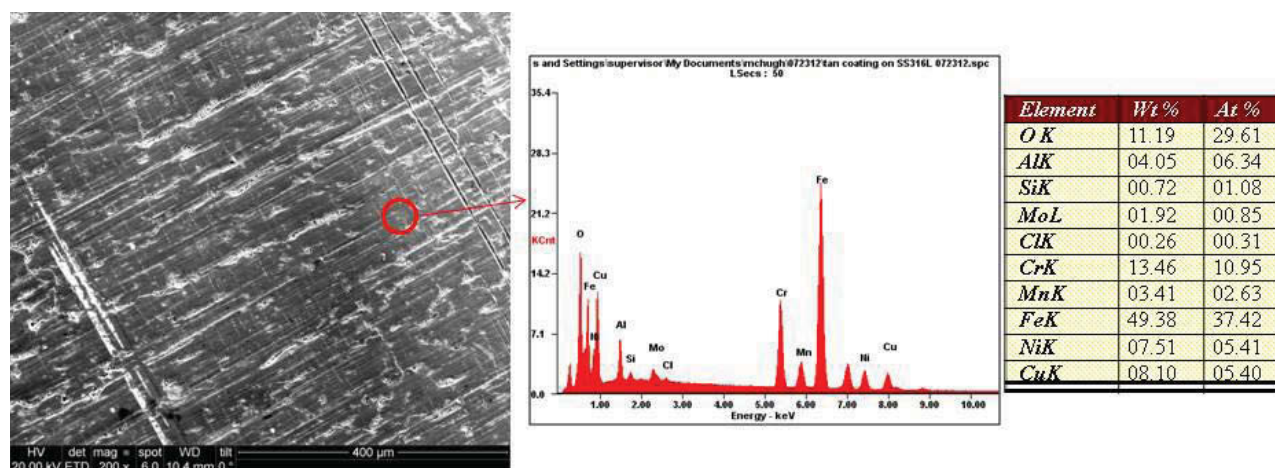


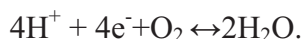
Figure 14. EDS analysis of the “tan” corrosion product on the portion of rodlet immersed in flowing water outside the aluminum basket.

After removing the source of copper ions, the system was drained and refilled with fresh water. Several subsequent runs were then conducted:

1. A three-day run at 140°F without rodlets was conducted to further passivate the aluminum basket. This run did suggest that a small amount of residual copper persisted in the system. The system was drained and refilled with fresh water.
2. A four-day run at 125°F with two Zr-702 rodlets and one SS-316L rodlet installed in the Al basket. The samples were removed and examined. No notable oxidation of the samples was observed. The system was drained and refilled with fresh water.
3. A one-day run at 125°F with one Zr-702 rodlet, one Zr-4 rodlet and one SS-316L rodlet installed in the Al basket. The samples were removed and examined. No notable oxidation was observed. A small discoloration at the upstream end of the SS-316 suggested that perhaps not all of Cu contamination was purged from the system. As a result, the basket was removed, the internal piping of the system and basket were cleaned, and the system reassembled. A low-flow volumetric flow meter was installed as well as an all-stainless heat exchanger.

Throughout these preliminary runs, the pH, conductivity, and dissolved oxygen (DO) of the water were recorded by the data acquisition system. No significant variations from run to run were found and a consistent pattern to the data was observed. Typical plots of inlet pressure to the basket, and water chemistry values are summarized in Figure 14 during a three-day basket passivation run. At the start of the run, water pH = 5.1, dissolved oxygen = 5.5 ppm, and conductivity = 3.2 μ S/cm. The plot of pressure at the inlet to the basket (**Figure 15a**) closely mirrors the plot of dissolved oxygen (**Figure 15b**) as a function of time. The solubility of oxygen

is pressure and temperature dependent. As the temperature rises, dissolved gas initially comes out of solution, and the level is seen to drop from the initial value of 5.5 ppm to about 4 ppm. In a closed system such as this, the pressure increases which forces gas in trapped pockets back into solution according to Henry's Law, and the dissolved oxygen level rises. For safety reasons, the system is periodically degassed which results in a sudden drop in pressure and reduction in oxygen. The response time of the oxygen probe lags behind that of the pressure probe, and the DO during the first hour appears to gradually rise. (At high data accumulation rates the plots more closely mirror one another). After about one-two hours, the water temperature reaches its steady-state value and both pressure and DO smoothly follow one another as periodic degassing continues. After an initial rise in conductivity, as temperature reaches steady-state, the conductivity plot versus time levels off at a constant value of about 3.8 $\mu\text{S}/\text{cm}$. (**Figure 15c**). pH, on the other hand, slowly rises as oxygen is depleted from the system by hydrogen ion (and corrosion reactions) according to:



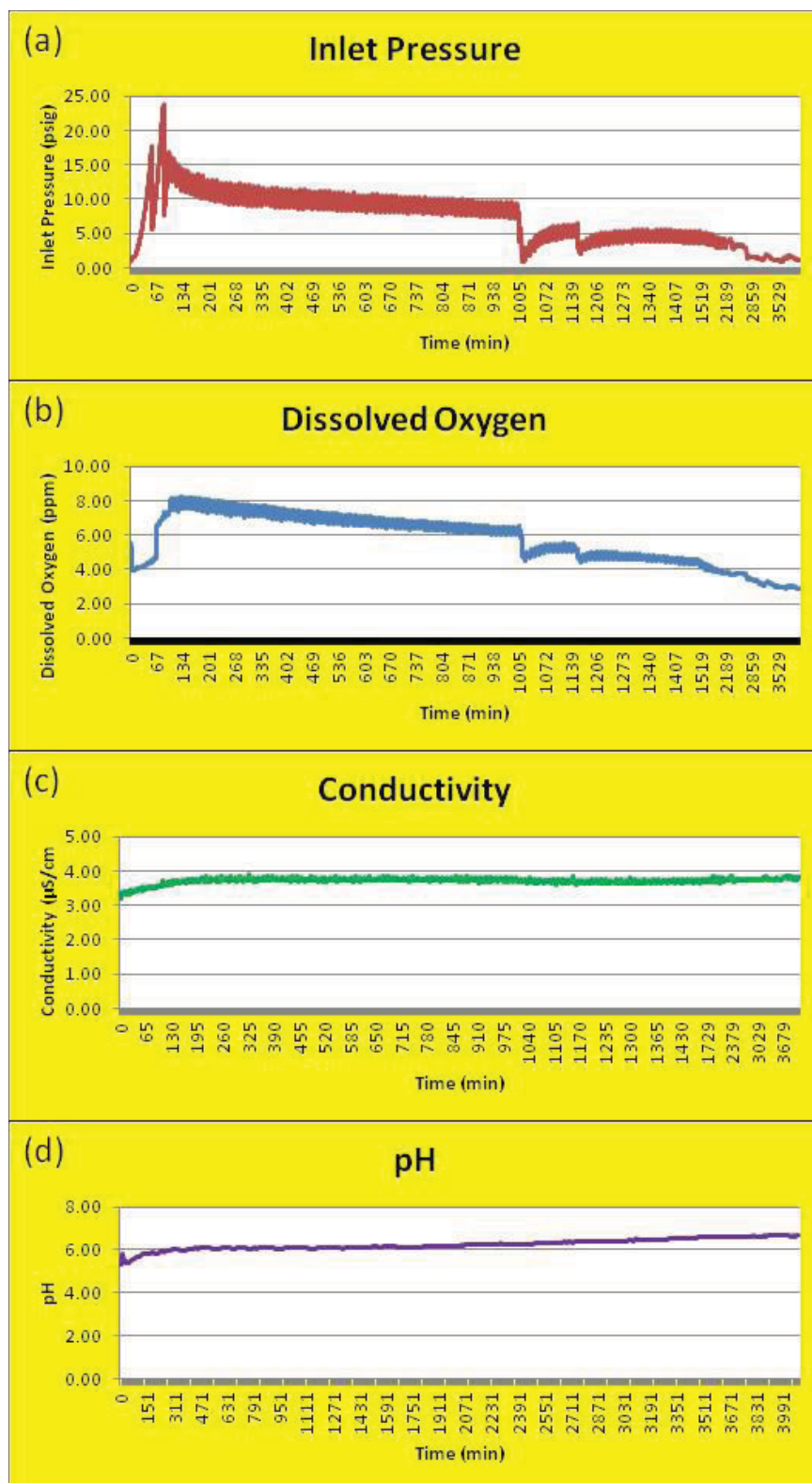


Figure 15. Water chemistry plots during a three-day aluminum basket passivation run. (a) Inlet pressure. (b) Dissolved oxygen. (c) Conductivity. (d) pH.

3. SiC CMC/Zr-4 Cladding Sample Run

The preliminary runs of the HWCS were designed to assess the overall performance of the system, isolate potential problems, and allow operators to gain experience. Methods used to calibrate instruments, general observations, etc., were documented in laboratory notebooks. A user's operating checklist was prepared to aid the operator by providing a list of suggestions and reminders when setting up an experiment and standard maintenance practices. These checklists are provided in Appendix B. A run sheet template, shown in Appendix C, was also prepared to provide a standard format for entering data for each experimental run.

As mentioned above, prior to running the CMC rodlets, the system plumbing housing the basket were removed and cleaned with Scotch Brite abrasive cloth and water. A low-flow volumetric flow sensor was installed, and the location of a pressure relief valve was changed to upstream of the basket.

The HWCS tests on advanced cladding designs were conducted with the following samples:

1. A Zr-4 rodlet.
2. A Zr-4 rodlet wrapped with one ply silicon carbide fiber-wrapped ceramic matrix composite (SiC CMC).
3. A Zr-4 rodlet wrapped with two plies of SiC CMC.

The SiC CMC material was supplied by Physical Sciences, Inc. Flow conditions consistent with ATR operation were established, and water was heated to 125°F. In all, the samples were subjected to a ten-day run under these conditions, but they were removed for inspection after three days, and again after an additional three days. Photographs of the samples following the initial three-day run are shown in **Figure 16**. Oxidation of the Zr-4 rodlets was not observed, and the CMC wraps showed no signs of delamination, fraying, cracking, visible oxidation or other defects. However, a very small amount of particulate debris was found on the CMC samples. This was traced to residual scotch brite abrasive used to clean the piping, and a small amount of aluminum oxide which originated from the basket. These materials were removed, the samples were re-inserted, and the system was run for an additional three days after reaching steady-state conditions. Photographs of the samples are shown in **Figure 17**. Again, no corrosion of Zr-4 was observed, and no deterioration of the SiC CMC wraps was observed. The samples were reinserted into the system, steady-state flow at 125°F was resumed, and the test sequence was completed. Photographs of the samples after the ten-day run are shown in **Figure 18** and **Figure 19**. No change in the condition of the Zr-4 or SiC CMCs was observed.

Changes in water chemistry with time exhibited the same pattern discussed previously. The oxygen concentration gradually decreased, pH gradually rose, and conductivity remained fairly constant. Water samples were periodically extracted for off-line analysis of ion and particulate concentrations. The turbidity of the water also increased with time as the concentration of aluminum oxide particles rose. This is to be expected as the system lacks a particulate filter and

ion exchange unit. Oxidation of aluminum is minimized if the pH is maintained at about 5. It should be noted that the flow conditions for the three rodlet samples was slightly different due to small differences in outer diameter. The Zr-4 rodlet with the smallest diameter would have seen a somewhat larger volume of water throughout the test, and the 2-ply SiC wrapped rodlet a somewhat smaller volume of water. This can be remedied in future runs by boring the basket flow channels to provide consistent gaps.



Figure 16. Photographs of Zr-4, SiC CMC rodlet samples following a three-day test run at 125°F. (a) Longitudinal view. (b) End-on view. (c-f) Various longitudinal views. (f) A small amount of debris from water collected on SiC CMC. No corrosion of Zr-4 was observed.

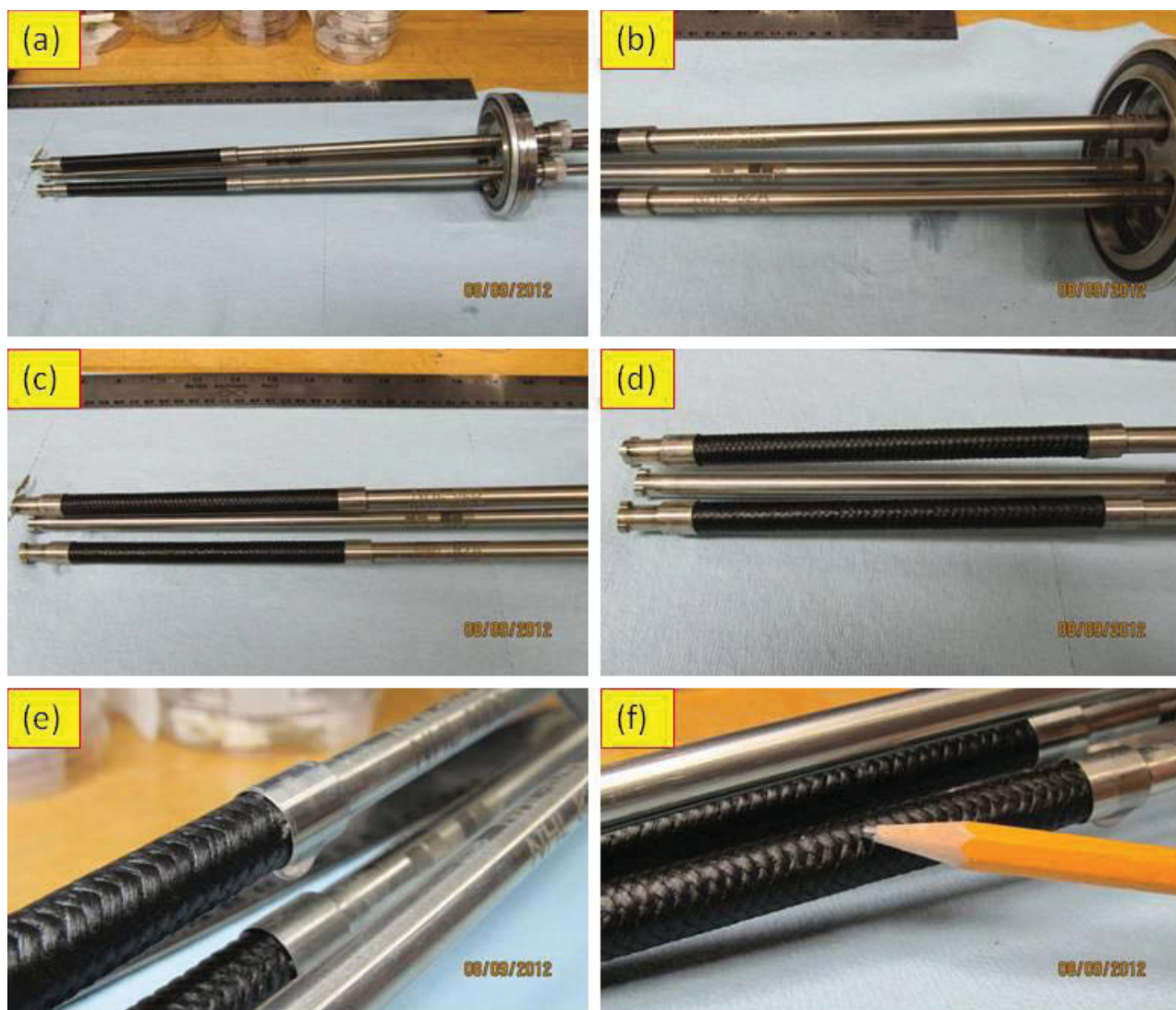


Figure 17. Photographs of Zr- 4 and SiC/CMC rodlet samples after six days at 125°F. (a-d) Longitudinal views of samples. (e,f) Close-up views of samples.



Figure 18. Photographs of Zr- 4 and SiC/CMC rodlet samples following the ten-day test at 125°F. (a) Sample holder removed from system. (b) Upstream end of samples. (c) Center of samples. (d,e) Downstream end of rodlets. (f) Edge view of rodlets.



Figure 19. Magnified visual examination shows no evidence of corrosion products on the 2 hybrid CMC and Zr-4 test samples. Note that minor particulate debris on the CMC samples was removed at 3-days prior to re-insertion for the remainder of the 10-day run.

4. Recommendations for Future Technical Improvements to the HWCS

- Degassing of the water added to the test loop can be accomplished by preheating the water at atmospheric pressure or by exposing the water to reduced pressure/vacuum. This would decrease the oxygen content of the water and reduce the need for degassing during operation. Steady-state operating conditions could be reached more quickly in the system.
- Increase the pressure rating of the system to allow operation under conditions that simulate flow/temperature conditions of a broader range of reactors.
- Incorporate hardware to control oxygen level at fixed, steady-state levels, including elevated O₂ levels to potentially enhance corrosion rate. This could be accomplished by introducing premixed gases (e.g., N₂-O₂) or other oxidizers.
- Modify basket design to provide equal gaps with test samples and equal water flow past test samples.
- Install an ion exchange water treatment system and a particulate filtration system to more closely simulate water treatment operations in nuclear reactors. This is needed for long-term runs with chemically-controlled water. In other tests it is desirable to allow water chemistry to fluctuate on its own, to evaluate leaching etc. from rodlets.
- Upgrade heat exchanger.

5. Summary

The HWCS apparatus was successfully designed, constructed and demonstrated to have sufficient flexibility to access the performance of a variety of advanced cladding materials and designs (**Figure 20**). Data from the HWCS tests will support down-selection of these materials and designs and provide baseline data for future evaluation of clad performance under irradiation. Several preliminary runs were conducted to assess system performance, and issues were resolved. Operational testing of the HWCS test system has been initiated with the first 3-day and 10-day runs. Test runs of 3 days, 6 days, and 10 days were concluded which included a Zircaloy-4 rodlet for baseline measurements and two SiC CMC hybrid tubes (SiC CMC external over-braid with Zircaloy-4 liner tube). The prototype testing also provided an opportunity for establishing operational procedures and personnel training and will establish standard test procedures.

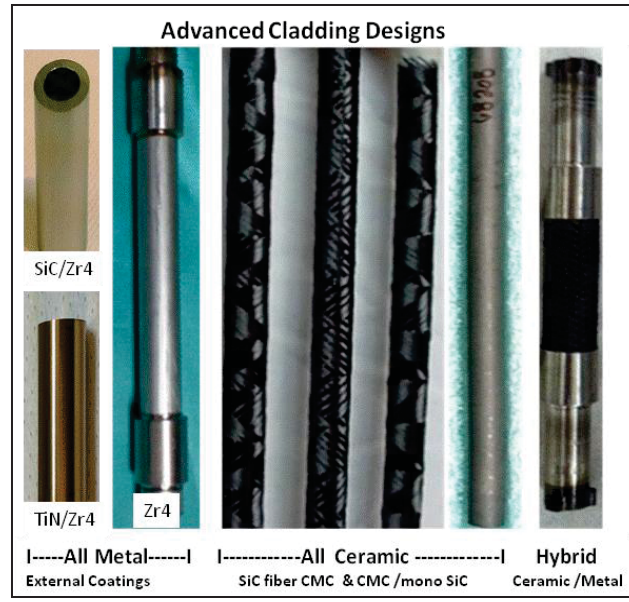
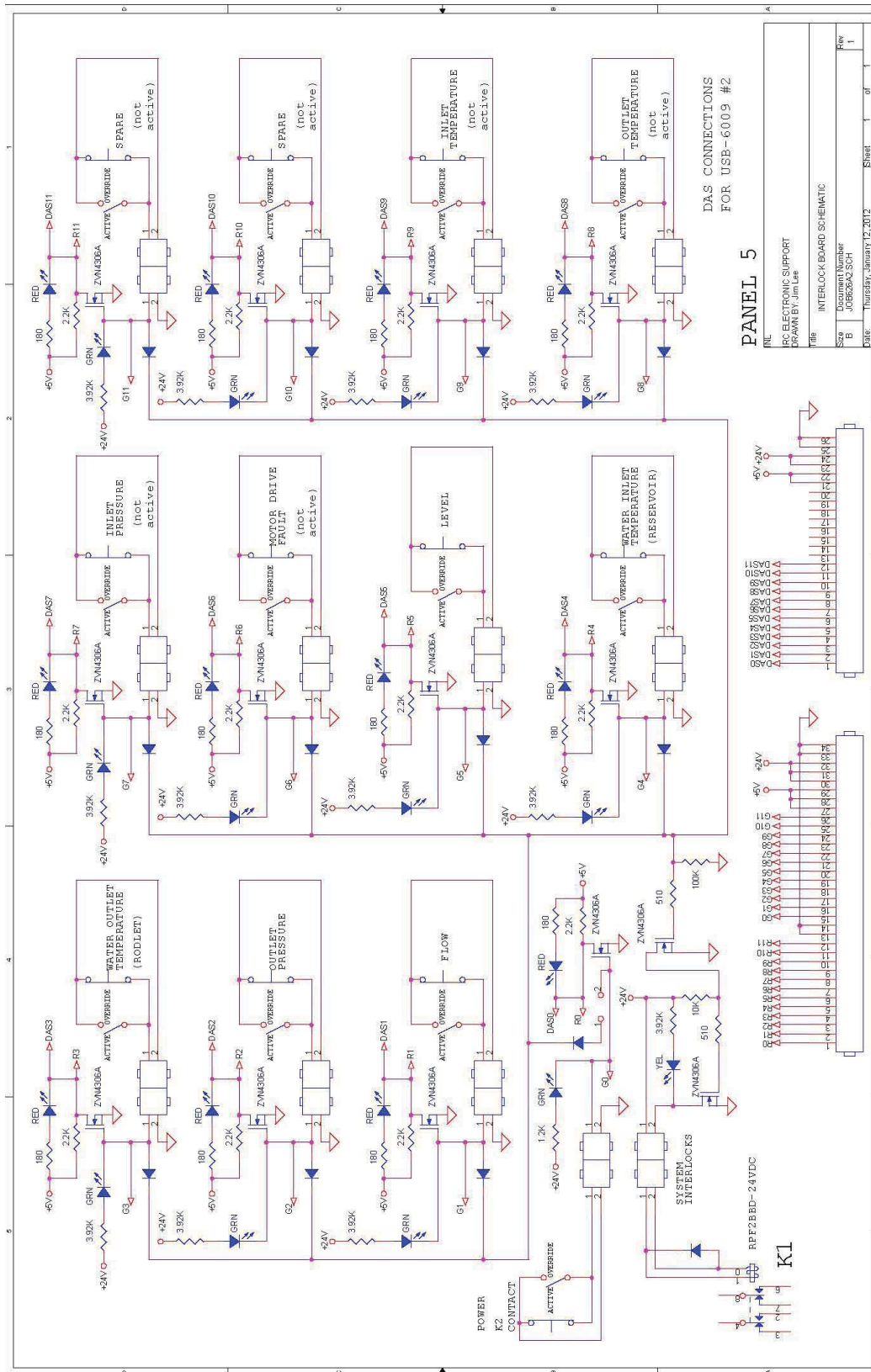
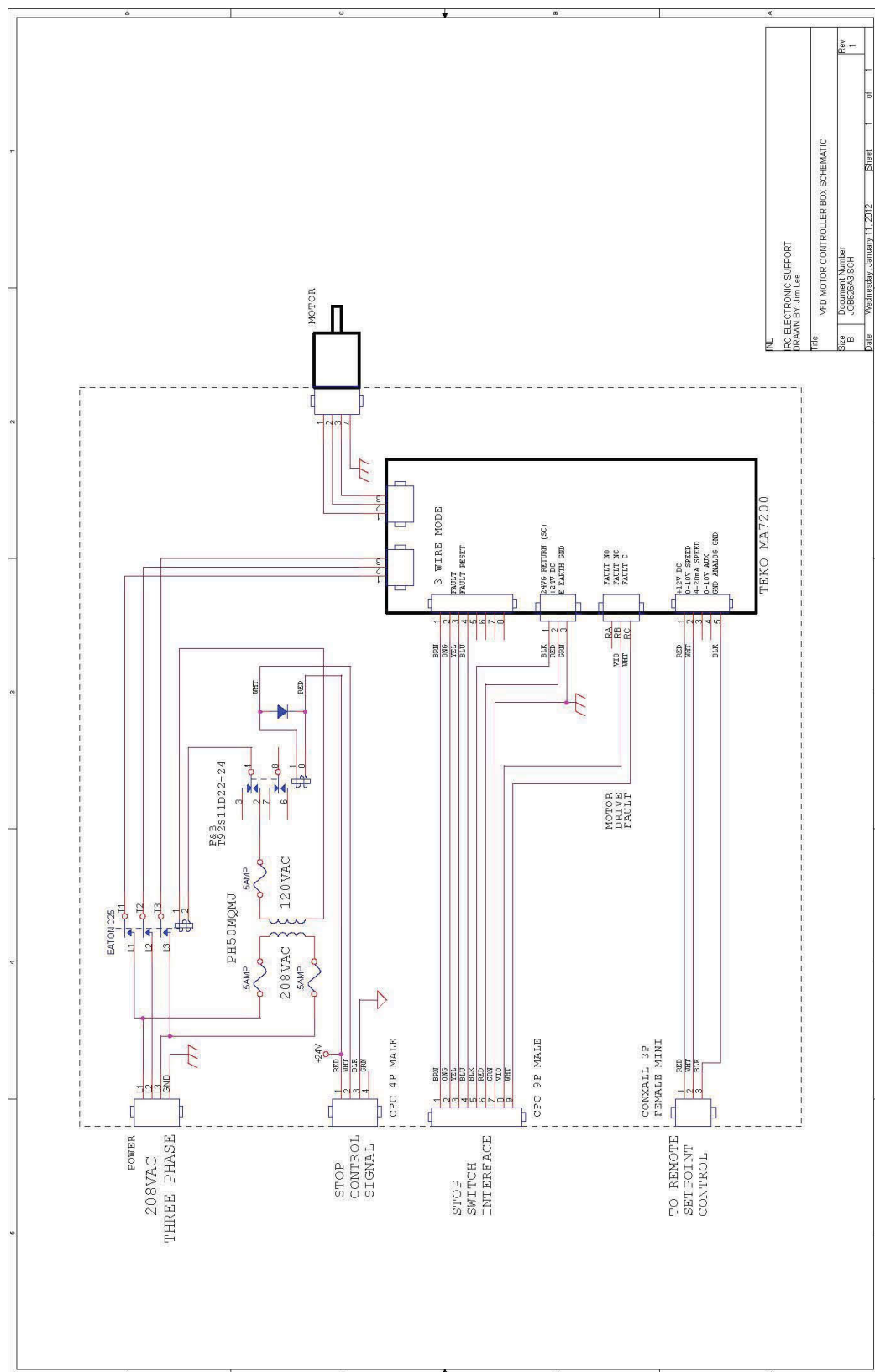


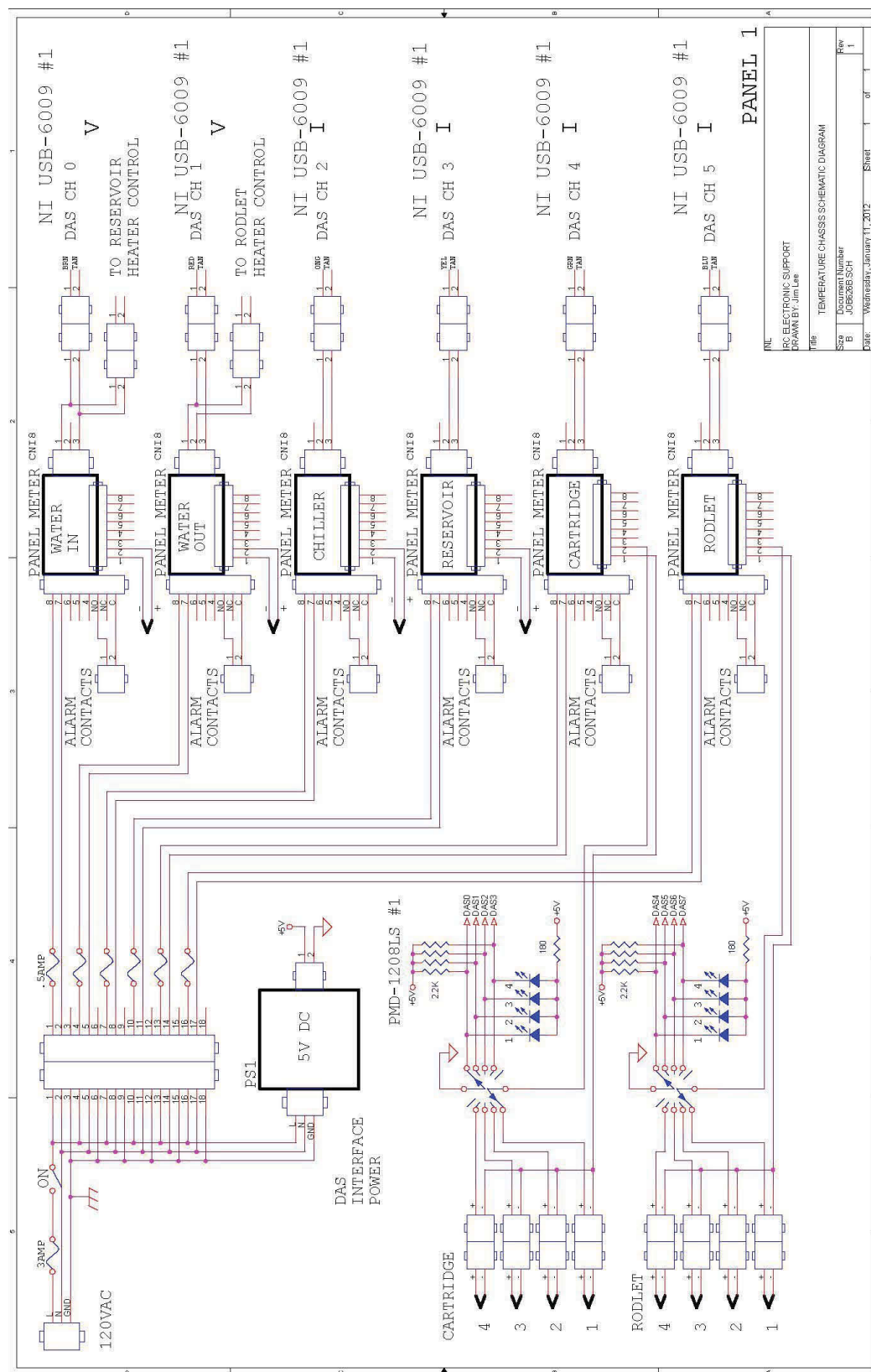
Figure 20. Examples of various advanced cladding designs: All metal, all ceramic and hybrid (ceramic/metal).

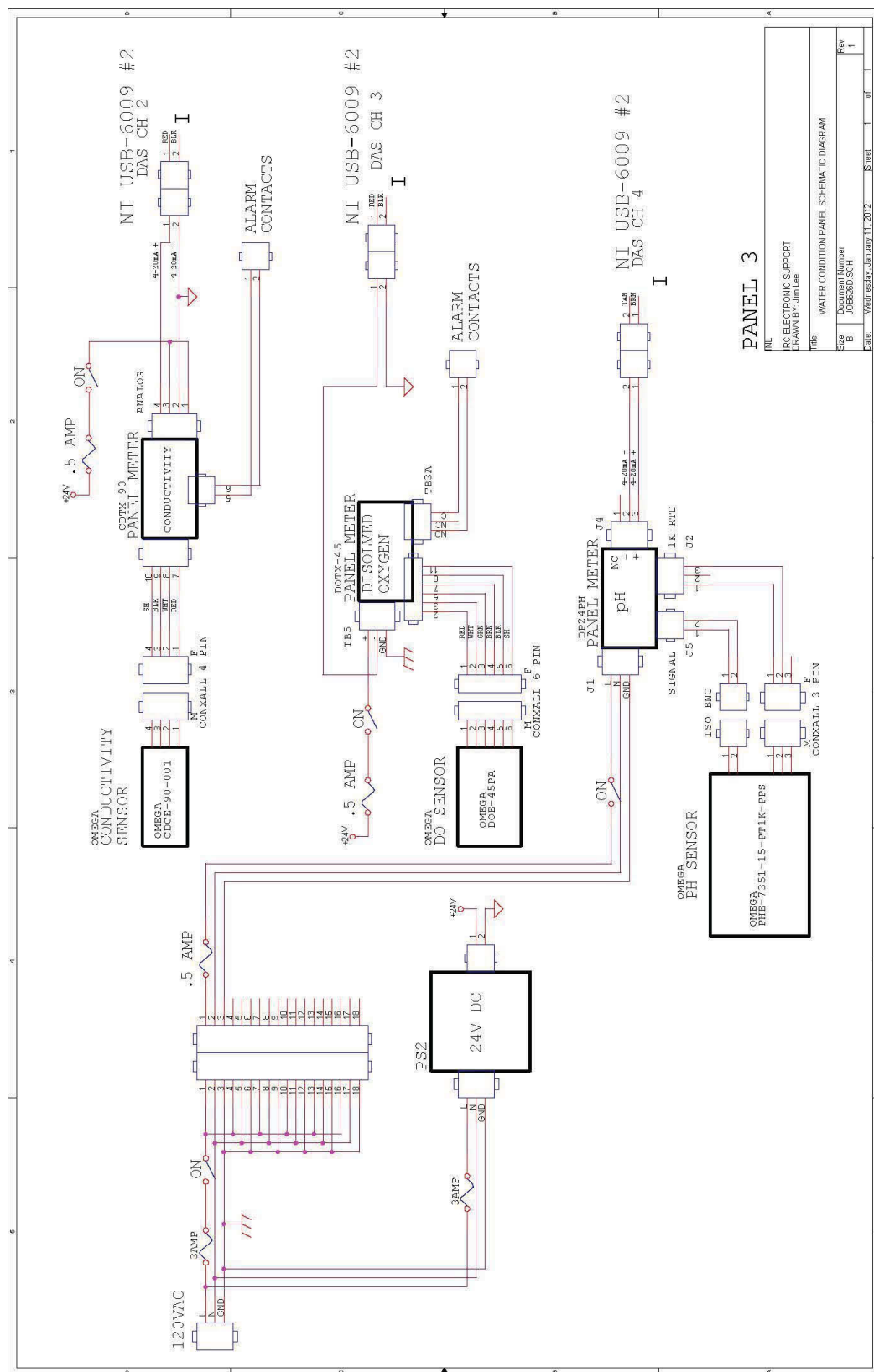
Appendix A

Electronic Diagrams for Control Panel and Interlock Circuitry



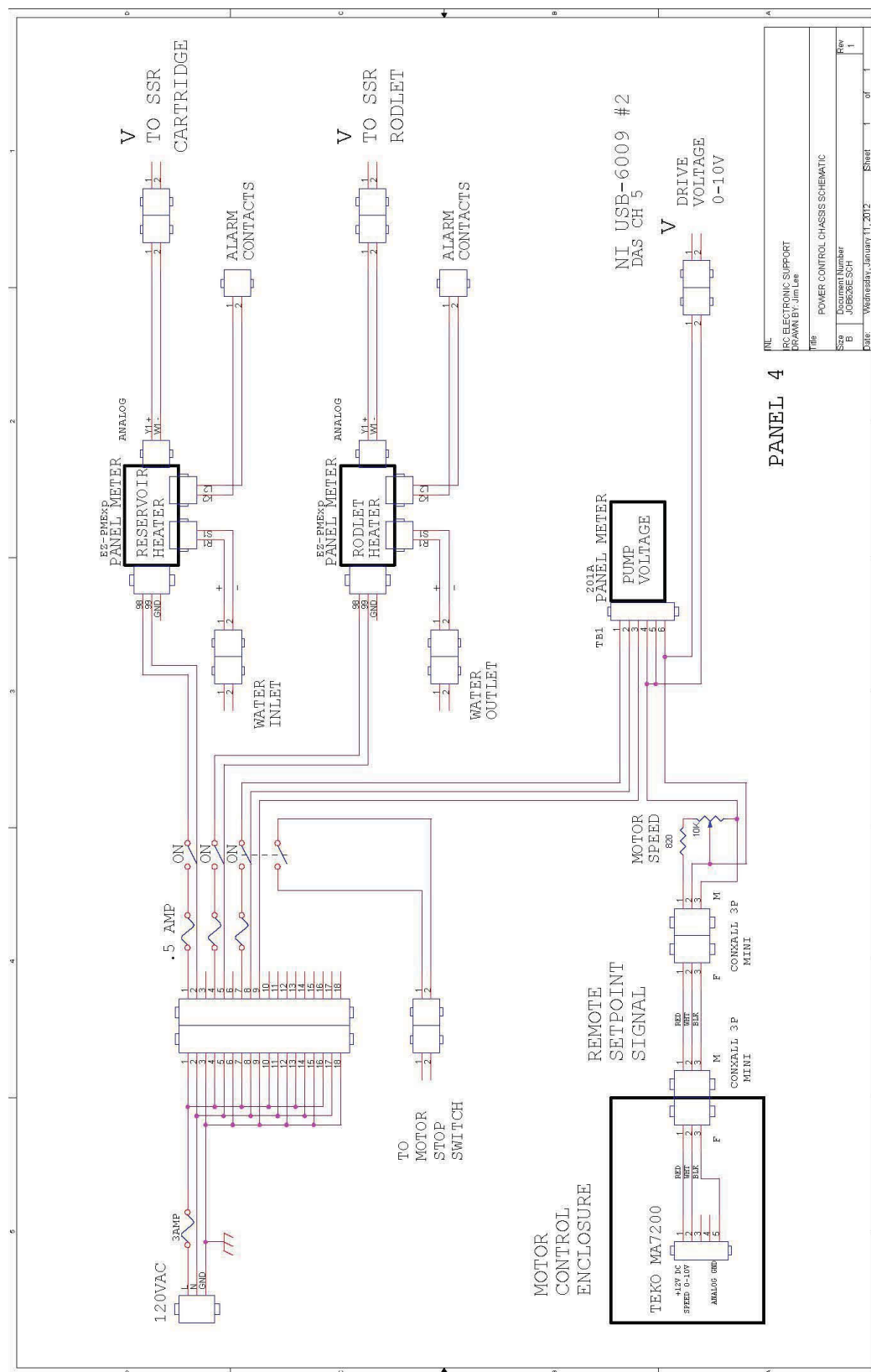






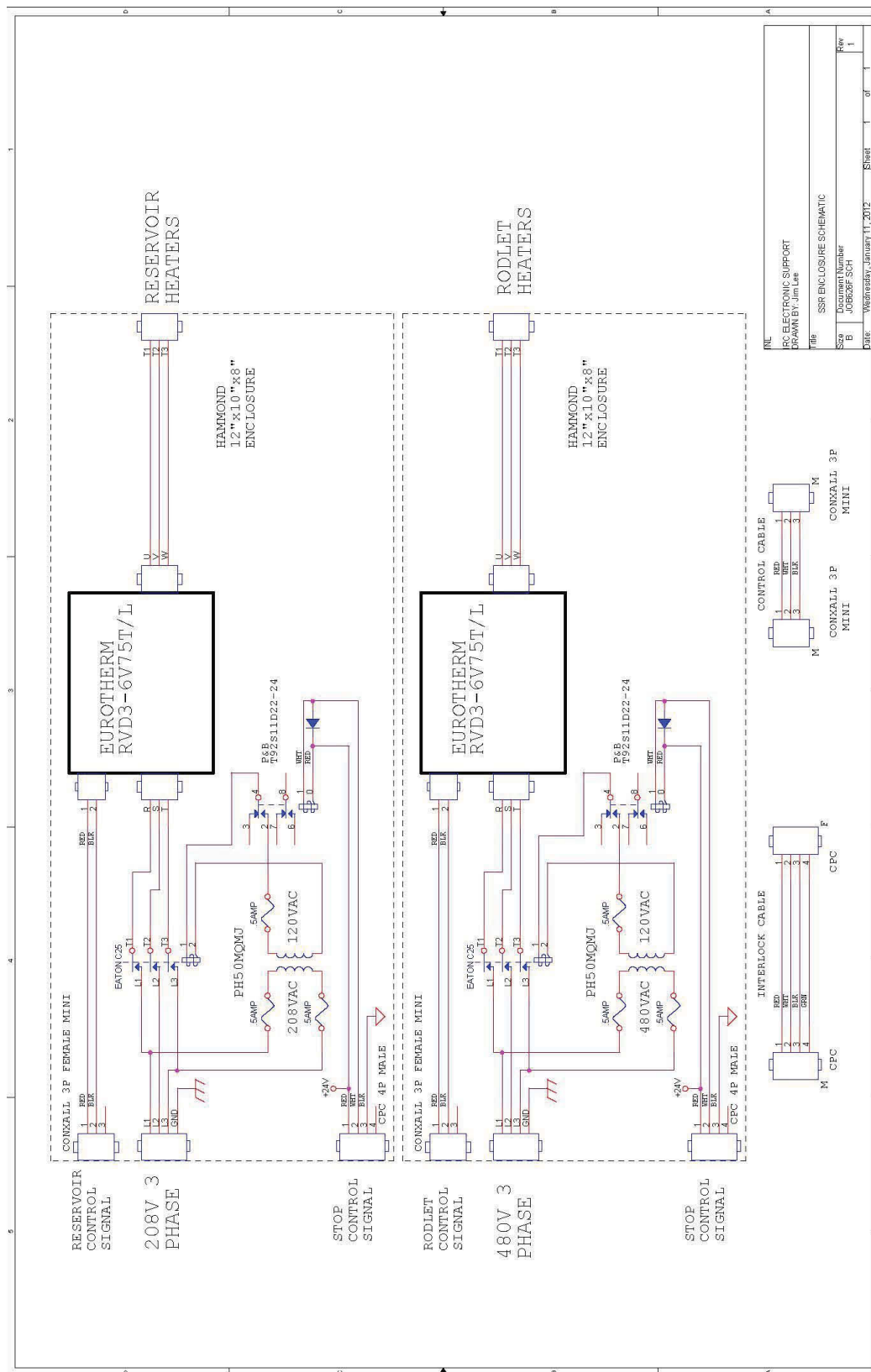
PANEL 3

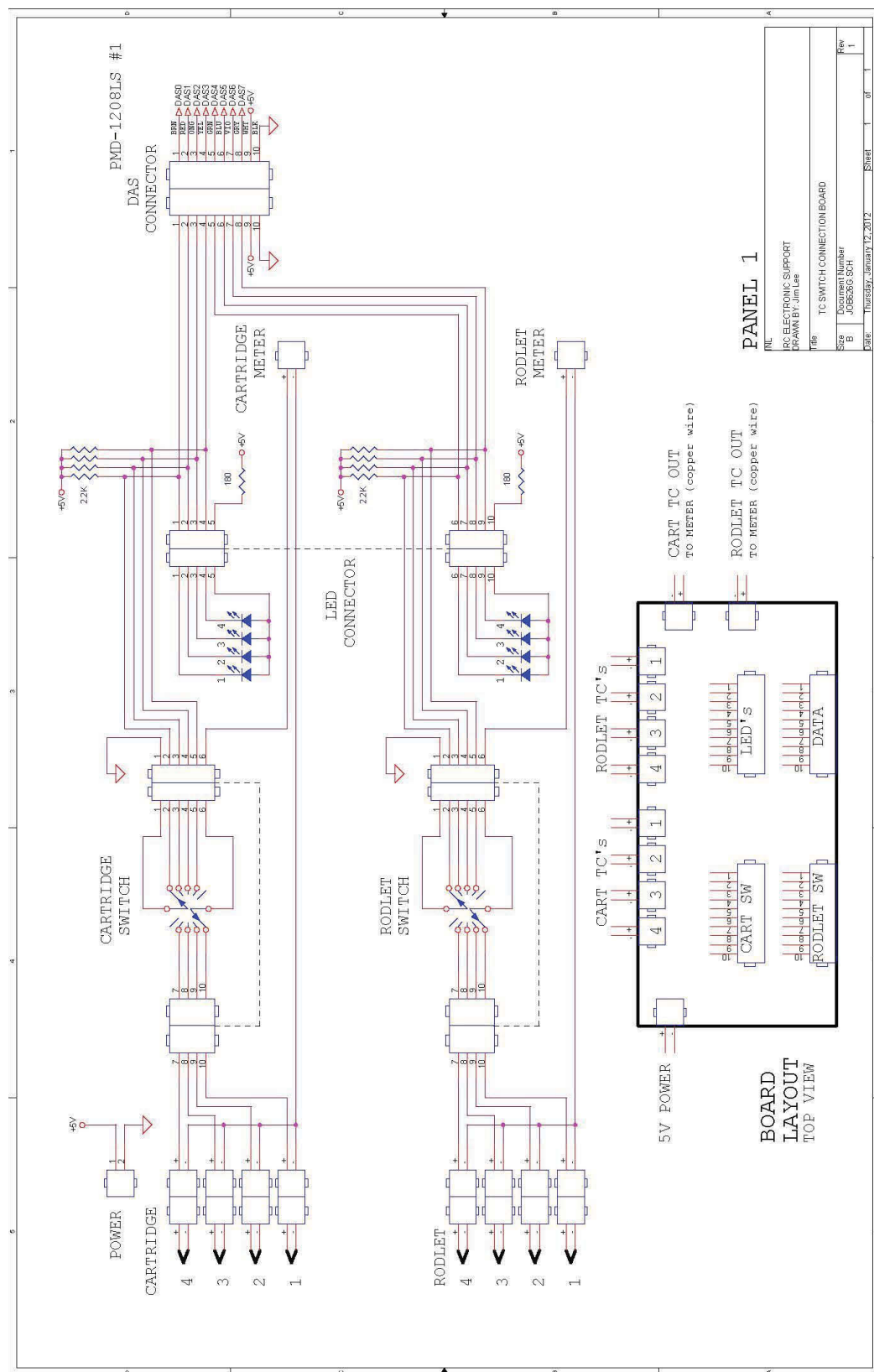
Rev	1
Doc Number	J060605-001
Doc Title	WATER CONDITION PANEL SCHEMATIC DIAGRAM
Doc Date	Wednesday, January 11, 2012
Doc Size	1 of 1



PANEL 4

Rev	1
Doc Number	J060825-01
Doc Title	POWER CONTROL CHASSIS SCHEMATIC
Doc Date	Wednesday, January 11, 2012
Doc Size	1 of 1





Appendix B

OPERATING CHECK LIST FOR BCTC Bay 4 HOT WATER CORROSION TEST SYSTEM

READY THE TEST SAMPLES FOR INSERTION

#	OPERATION	CHECK	COMMENTS
1	Received tagged test rodlet samples, verify supporting paperwork		
2	Log in test samples into the Bay 4 LWRS logbook, store in LWRS cabinet		
3	Measure surface roughness of each rodlet, record in logbook and on run sheet		
4	Load rodlets onto end support rings and through end flange		
5	If rodlet heaters inserted, attach heater wires to labeled end plugs (1,2,3,4)		Used only in internal stress tests
6	Check on need for an additional tube sleeve insert used (Reynolds number)		
7	Check on supply of ATR water in 50 gallon vessel		Refill with ATR water only
			PAGE 1 OF 3

SYSTEM IS IN OFF CONDITION. TEST SAMPLES TO BE LOADED			
#	OPERATION	CHECK	COMMENTS
1	Turn off main power breakers to water pump and heater. Disconnect plugs		
2	Check water level. Drain to below test section before opening		This drains about 2 gallons
3	Re-check water valves to "off/closed position "on all drain/ re-fill valves		
4	Re-position for sleeve clearance water and rodlet thermocouples		
5	Review Test Procedure Chart for test sample ID and orientation position		
6	Insert tube sleeve and test samples. Reposition thermocouples per Run Sheet		
7	Bolt on the specimen end flange		
8	If running thermal stress experiment, plug specimen wires into heater plug		plug in only if running stress test
9	Re-fill with ATR water only system to top, open pipe air vent (top of reservoir), close water return to reservoir top valve.		ATR water in the 50 gallon reservoir. Use pump
10	Connect plugs and at breakers turn on main power to system and pump		
11	Run pump at low speed (3 to 4 ft/second) for 5 minutes		
12	Water drip check: Using wrenches re-check all clamps around the system piping		
13	Stop pump and re-vent the air release valve above reservoir		
14	Add additional water at system fill point		typically 1 cup is needed.
15	Review Test Procedure Chart (water T, time, flow rate, sampling rate)		
16	Per Run chart: Manual dial velocity to run water flow rate: (e.g. 12.1 ft/second)		
17	Wait 2 minutes for system flow stabilization		
18	Set water temperature controller (180 degrees F)		
19	Re-check monitor outputs and alarm settings:		
	water thermocouples, water flow, water pressure		
	Water chemistry (future): pH, O2		
20	In-situ water chemistry sensors: pH, O2, conductivity		
21	Laptop recorder: Enter test run information; sampling rate (e.g. 1 /		
22	Initiate laptop recorder to begin recording data		
23	Zero Re-set the three (3) Run Clock timers: total time, run time, time		
24	Manually open chiller valves (1/8)		Low cooling flow needed (1/8 turn)
25	Turn on water heater: Water T rises to 180 F. Surfaces will be HOT		No heater; T= 84 F @ 12.1 ft/s
26	Re-check data logger for test information recording		
27	Water drip check: Using wrenches re-check all clamps around the system piping		Continue visual until water T = 180 F and stabilizes for 5 minutes
28	Position plastic shields in front / sides of the system.		Surfaces will be at 180 F
29	System is in run mode		
			PAGE 2 OF 3

SYSTEM IS IN 24/7 TEST RUNNING MODE			
#	OPERATION	CHECK	COMMENTS
	Daily system check (24 hours)		Frequency TBD
1	Review Test Procedure Chart (water T, time, flow rate, sampling rate)		Adjust water velocity as needed
2	Check all system gauges / monitors/ data acquisition system		
	water thermocouples, water flow rate, water pressure		T and flow within ATR limits ?
	Water chemistry (future): pH, O2		Values within ATR limits ?
3	Re-move plastic shields in front / sides of the system.		Surfaces will be at 180 F
4	Water drip check: Using wrenches re-check all clamps around the system piping		Add ATR water ? See 4A 7 day check
5	Position plastic shields in front / sides of the system.		Surfaces will be at 180 F
6	Leave data acquisition system on		
7	If ATR water is to be added, reduce pump speed to trigger "trip condition"		
	Reduce pump speed to trigger "off" condition		
	Open fill reservoir at top. Add ATR water		
	Open reservoir vent then close		
	Re-set trip override on pump. Restart pump		
	Set pump speed (12.1 ft/second)		
8	Check in-situ water chemistry sensors readings: pH, O2, conductivity		Values within ATR limits ?
9	Laptop recorder: Enter test run information; sampling rate (e.g. 1 / minute)		
10	Repeat daily. Note any changes on the Run Sheet and HWCTS logbook		Adjust as needed. (e.g. water T, flow rate)
11	Back to beginning --> Daily system check (24 hours)		Frequency TBD
	Every 7 days (160 to 180 hours)		Frequency TBD
1	Leave data acquisition system on		
2	Turn off the water heater.		Do not change water T set-point
3	Re-move plastic shields in front / sides of the system.		Surfaces will be at 180 F
4A	Stop pump by lowering speed to "trip" to off condition , turn off the circuit breaker		
	Decant ATR water sample into Remote pH container and measure water chemistry (pH, O2, conductivity)		Decant tube static water; fill sampling bottle 1/2 gallon ATR
4B	Per Run Sheet: Re-position thermocouples; unbolt flange; remove rodlets an note positions; re-measure surface roughness; replace rodlets in same positions; bolt on flange		Record surface roughness data on Run sheet
	Add additional ATR water at system fill point		
	Re-vent the air release valve above reservoir		
	Open fill reservoir at top. Add ATR water		
	Open reservoir vent then close		
	Re-set trip override on pump. Restart pump, Set pump speed (12.1 ft/second)		
5	Position plastic shields in front / sides of the system.		Surfaces will be at 180 F
6	Re-check status of data acquisition system		
7	Back to beginning --> Daily system check (24 hours)		Frequency TBD

Appendix C

Sample Run Data Sheet

BCTC Bay 4 HOT WATER CORROSION TEST SYSTEM: HWCTS Test Run Sheet										PAGE	1 OF 1
CHECK	RUN NUMBER:	1	RUN DURATION (TOTAL HRS.) 3 DAYS = 72 HOURS		72	ATR HOT WATER AND FLOW	YES	THERMAL STRESS	NO		
	RUN START Date:					INL Set-up and Run Operator:					
x	RODLET MANUFACTURER:					Log-in sample IDs into LWRS log-book in storage cabinet					
x	TEST RODLET LOCATION		1 2 3								
x	TOP RODLETS		1		SAMPLE #:	4	SAMPLE #: NA				
x	BOTTOM RODLETS		2		SAMPLE #:	3	SAMPLE #:				
CHECK	AS-RECEIVED INDIVIDUAL TEST SAMPLE COMMENTS										
	Sample	RODLET TYPE				APPEARANCE	PHOTO	Talsurf SURFACE ROUGHNESS	3D Tomography		
	#	Zr4	SS	CERAMIC	HYBRID						
	x	1	X								
	x	2				X					
x	3				X						
	4										
CHECK	STARTING TEST CONDITIONS										
	WATER TEMPERATURE (degree F)				125				TRUE	TRUE	
	FLOW RATE (FEET/SECOND)				12.1		CALCULATED		TRUE	TRUE	
	SAMPLE BASKET				ALUMINUM				YES	NO	
	WATER CHEMISTRY				pH= 5 to 8; pH =		O2=	Conductivity =			
x	REYNOLDS NUMBER (BASKET INSERT)				Re =		CALCULATED				
CHECK	STARTING SYSTEM SENSOR ALARM LIMITS										
	Sensor	Low	High	RE- CHECK	Operator Action			Special Actions for Thermal Stress Tests			
	Water Level		NA	x	disconnect to trip. Then re-set			Same			
	Water Temperature	NA	195 F	x	Set at heater controller.			Will need to lower main heater and increase chiller flow			
	Flow Velocity (Ft/s)	3	26	x	Needs to be scaled to basket. set manually at knob			This will be reduced to ca. 2-3 ft/sec			
	ATR Water Sample			x	Ph (5 TO 8) set aside 1 gallon						
	On-line Ph	not active	not active	x							
	On-line O2	not active	not active	x							
	Water Pressure	NA	20 psig	x	Re-check sensor set. With Basket in place large delta P Manual relief at 20			Same			
	Chiller By-pass Low	NA	T = 200 F	not used	low flow alarm trigger by T sensor			Same			
	Chiller By-pass High	NA	T = 210 F	not used	high flow alarm trigger by T sensor. Emergency by-pass chiller water - NOT SET			Same			
	DATA LOGGER: RE-SET /TURNED ON				Yes or No		Data Rate:	Data logger sample rate at 60 s lead to data stop at 21/2 days. Changed rate to 600 s.			
	x	RUN INITIATE	PUMP ON	HEATER ON	LEAK CHECK	IS THE DATA LOGGER ON ?		Y OR N			
	Date	RUN Time (HR:MIN)	TOTAL	Flow (ft/s)	Water T (deg F)	Water pH	Data Logger	Comments		OPERATOR	
	0:00:00	0					ADD WATER and REMOVE ENTRAINED AIR				
1 day							TAKE MANUAL WATER SAMPLE				
							ADD WATER and REMOVE ENTRAINED AIR				
2 day							TAKE MANUAL WATER SAMPLE				
							ADD WATER and REMOVE ENTRAINED ARI				
3 day							TAKE MANUAL WATER SAMPLE				
							ADD WATER and REMOVE ENTRAINED AIR				