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Abstract – *An integrated laboratory scale, 15 kW high-temperature electrolysis facility has been developed at the Idaho National Laboratory under the U.S. Department of Energy Nuclear Hydrogen Initiative. Initial operation of this facility resulted in over 400 hours of operation with an average hydrogen production rate of approximately 0.9 Nm^3/hr. The integrated laboratory scale facility is designed to address larger-scale issues such as thermal management (feed-stock heating, high-temperature gas handling), multiple-stack hot-zone design, multiple-stack electrical configurations, and other "integral" issues. This paper documents the initial operation of the ILS, with experimental details about heat-up, initial stack performance, as well as long-term operation and stack degradation.*

I. INTRODUCTION

The recent escalation in crude oil and gasoline prices, along with worries concerning the reliability of crude oil supplies, have created a strong interest in developing hydrogen as a second energy carrier for the non-electrical market. The goals of a hydrogen-based energy economy are reduced oil consumption, foreign energy independence, and reduced greenhouse gas emissions. Since hydrogen is an energy carrier and not an energy source, attaining these goals is conditional upon development of suitable renewable energy sources and/or nuclear energy to power water-splitting technologies for carbon-free hydrogen production.

Water-splitting for hydrogen production can be accomplished via high-temperature electrolysis or thermochemical processes, using high-temperature nuclear process heat. In order to achieve competitive efficiencies, both processes require high-temperature operation (~850°C). High-temperature electrolytic water-splitting supported by nuclear process heat and electricity has the potential to produce hydrogen with an overall system efficiency near those of the thermochemical processes [1], but without the corrosive conditions of thermochemical processes and without the fossil fuel consumption and greenhouse gas emissions associated with hydrocarbon processes.

The Idaho National Laboratory (INL), in conjunction with Ceramatec Inc. (Salt lake City, USA) has been researching the use of solid-oxide fuel cell technology to electrolyze steam for large-scale nuclear-powered hydrogen production. The scope of activities includes computational fluid dynamics modeling [2], process flow sheet analyses, and experimental testing [3, 4, 5, 6]. Experimental testing has followed a logical progression in scale. Button cell (~2 watt) and short stack (~500 watt) tests have primarily concentrated upon quantifying material and cell performance and have not addressed larger-scale issues such as thermal management (feed-stock heating, heat recuperation, and high-temperature gas handling), hydrogen recycle, multiple-stack hot-zone design, multiple-stack electrical configurations, and other "integral" issues. For example, in button cell and bench-scale stack testing, steam is introduced into the inlet gas stream by saturating a carrier gas via a heated humidifier. Furthermore, the cell or stack is located inside of a furnace and the inlet gases are heated to the stack inlet temperature by the same furnace. This approach for steam production and feed-stock heating is not realistic for larger scales of electrolysis.

An integrated laboratory scale (ILS) high-temperature electrolysis facility has been developed at the Idaho National Laboratory under the DOE Nuclear Hydrogen Initiative. The ILS is designed to address larger-scale issues not addressed at the smaller bench scale. Initial operation of this facility resulted in over 400 hours of operation with an average hydrogen production rate of approximately 0.9 Nm^3/hr. For the multiple-stack tests performed in the ILS facility, the inlet gas flows are preheated upstream of the primary hot zone to the electolyzer operating temperature of 800 C. In the initial ILS operation, neither heat recuperation nor hydrogen recycle were incorporated. The system is designed to allow later incorporation of recuperative heat exchangers and hydrogen recycle. The use of recuperation minimizes the net heat addition required to heat the process gas streams up to the high temperature electrolysis (HTE) operating temperature. In addition, steam was introduced to the system using a steam generator rather than via heated water bath as used in smaller scale tests.

The ILS facility has been designed for an ultimate nominal hydrogen production rate of 14.1 kW based on lower heating value (LHV, equal to 120 MJ/kg for hydrogen) [1], or 4735 Normal (273°K, 1 atm) L/hr. The initial ILS single module implementation was designed for ~5 kW hydrogen production. This paper documents the initial operation of the ILS, with experimental details about heat-up, initial stack performance, as well as long-term operation and stack degradation. A more complete description of the facility and initial operation can be found in [7].

II. OVERVIEW OF FACILITY

The piping and instrumentation schematic for the ILS single-module experiment with no heat recuperation or hydrogen recycle is shown in Fig. 1. The electrolysis module requires a support system supplying electrical power for electrolysis, a feedstock gas mixture of hydrogen and steam, a sweep gas, and appropriate exhaust handling. In particular, this system must include means for controlled steam generation, mixing hydrogen with the steam, feedstock and product dewpoint measurements, heating the feedstock and sweep gas to the appropriate electrolysis temperature (via a superheater), cooling the electrolysis product stream, condensing any residual steam out of the product stream, and venting the hydrogen product. The final ILS support system will consist of three parallel systems that supply feedstock, sweep gas streams, and electrical power basically independent of each other to each of three modules. All three modules will be located within a single hot zone. The facility is designed to accommodate later incorporation of heat recuperation and hydrogen recycle capabilities. To aid in interpretation of Fig. 1, the hydrogen / steam feedstock is represented by the color magenta, the product stream by orange, the inlet sweep gas by green, and the outlet sweep gas by blue.

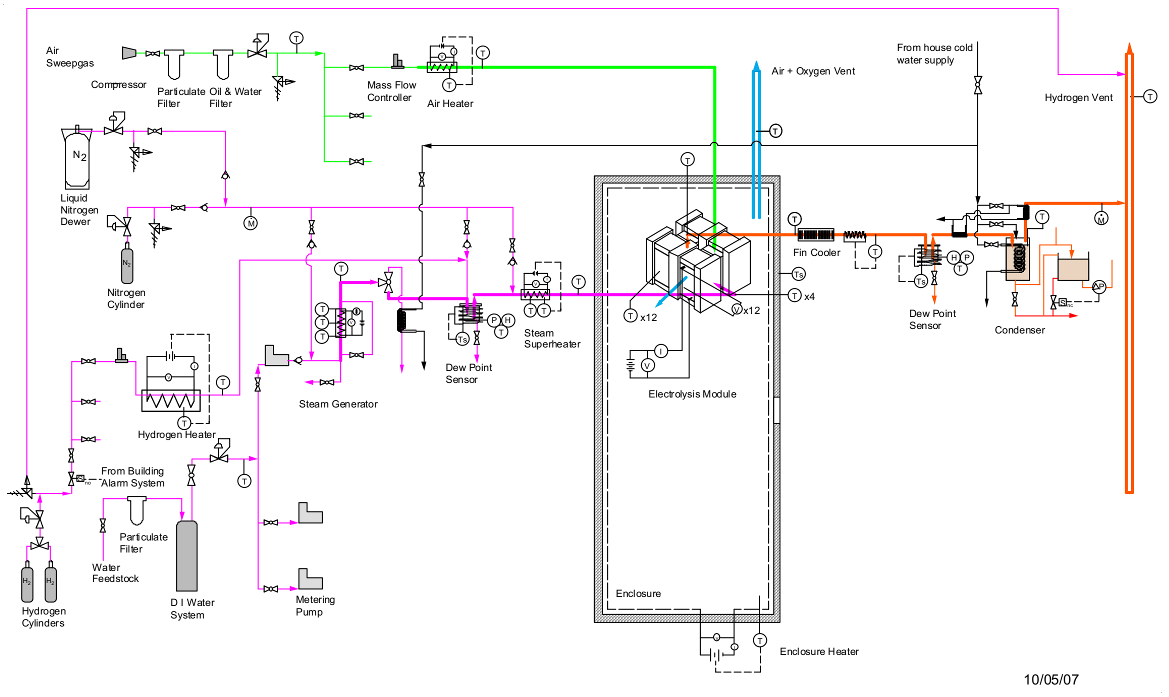

Fig. 1. ILS single module piping and instrumentation schematic.

Fig. 2. Hot zone enclosure with one module installed.

Liquid water feedstock is fed at a controlled rate into the system by means of a positive-displacement metering pump. The water is then vaporized and slightly superheated in an inline electrically-powered steam generator. The steam generator was fabricated by attaching a combination of fifteen 200 and 300 watt clamp-on electric heaters to the outside of a 1" diameter stainless steel tube. The heaters are covered with 2" of thermal insulation, then topped by an aluminum covering. The heaters are spaced such that a higher heat flux is obtained in the boiling region and lower heat flux in the single-phase regions. The heaters are all wired in parallel so that each operates at the same voltage. The tube interior is filled with a copper foam material which reduces flow perturbations and increases temperature uniformity in the boiling region. The outlet temperature is controlled by carefully adjusting the input power supplied by a DC power supply to obtain the desired superheat temperature.

The slightly superheated steam exiting the steam generator is mixed with hydrogen, which is required on the inlet side of the stack in order to maintain reducing conditions at the steam/hydrogen electrode. In the initial ILS configuration (prior to the implementation of hydrogen recycle), the inlet hydrogen will be supplied from a compressed gas bottle. The hydrogen flow rate is controlled by a mass-flow controller and the data acquisition / control system (DACS). The inlet hydrogen must be heated to the steam generator outlet temperature in order to prevent cooling of the steam and possible steam condensation. This is accomplished by temperature-based feedback control of the hydrogen preheater powered by a DC power supply in conjunction with the DACS. Downstream of the mixing point, the temperature, pressure, and dewpoint of the steam/hydrogen gas mixture are measured. The absolute pressure is directly measured at the dewpoint measurement station in order to allow for accurate determination of the steam mole fraction. Precise measurement of the dewpoint and pressure allows for independent determination of the inlet gas composition.

A high-temperature electrically powered inline superheater then boosts the feedstock stream to the final electrolyzer operating temperature, 800° - 830°C. Heat is supplied from six semi-cylindrical ceramic-fiber heaters with embedded coiled elements. Each heater section is capable of providing 1800 watts of power when operated at 240 volts, but they are operated at a much lower voltage for this application. Power is supplied to the heaters from 3.3 kW DC power supplies. Heater power is feedback-controlled based on thermocouples located inside the ceramic fiber heaters. Two inch thick high-temperature thermal insulation is wrapped around the heaters and covered with an aluminum skin.

The primary material of construction for the low-temperature tubing and components upstream of the superheater is 316 stainless steel. For high temperatures such as 800°C, Inconel 600 tubing is used within the superheater and air heater.

The electrolysis module is mounted in the hot zone enclosure (Fig. 2) where it is maintained at the desired operating temperature using radiant heaters installed in the sides and top of the removable lid. As explained in references [8, 9], when the electrolysis process is operated below the thermal neutral voltage (voltage at which stack ohmic heating balances the endothermic heat requirement, V_{tn} = 1.287 V/cell for 800°C operating temperature), heat must be added to overcome the endothermic reaction heat requirement. At thermal neutral conditions, the module operation is adiabatic and isothermal. If, however, the module is operated above the thermal neutral voltage, heat must be removed from the system.

The base of the hot zone enclosure consists of a stainless steel plate covered with several inches of high-temperature insulation, as shown in Fig. 2. The module rests on top of the insulation. The process streams, power leads, and instrumentation access the module through holes in the bottom plate and insulation. A stainless-steel lid covers the hot zone enclosure and is sealed against the bottom plate with an O-ring. The radiant heater panels are powered by a DC power supply, feedback-controlled based on a thermocouple mounted inside the enclosure. The lid is attached to screw-drive rods on each end, driven by an electric motor, which allow for convenient raising and lowering of the lid.

The gas mixture exiting the electrolyzer will be significantly enriched in hydrogen, typically to at least 50% hydrogen mole fraction, with the remainder being residual steam. The product stream is first cooled via a natural-convection air-cooled heat exchanger. The product stream temperature exiting this cooler is controlled such that no condensation can occur. Then the product gas mixture enters the outlet dewpoint measurement station. As discussed previously, the measurement of both inlet and

Fig. 3. INL ILS facility, with major components labeled.

outlet dewpoint temperatures allows for direct determination of the steam consumption rate, and the corresponding hydrogen production rate. This rate can be compared to the electrochemical hydrogen production rate determined from the stack electrical current. The outlet hydrogen/steam flow then enters a condenser where the vast majority of the residual steam is removed. The rate of water condensation is monitored via tank level, providing an additional independent measure of steam consumption. At this point, the product stream will be ambient-temperature, saturated hydrogen gas, with about 2.7% residual water vapor. The flow rate of this product gas is measured with a low-pressure-drop mass flow transducer. Comparison of the condensate and hydrogen product mass flow rates with the electrolyzer inlet mass flow rates helps quantify any stack leakage that may occur. The hydrogen product is then vented from the building.

Air is used as a sweep gas to remove excess oxygen from the ILS system. Filtered compressed air flows through a mass-flow controller and into an electrically-powered heater to preheat the inlet air to the stack operating temperature. Downstream of the electrolyzer, the hot oxygen-enriched air stream is then vented from the building to the environment.

Nitrogen gas can be injected directly into the steam superheater. This feature is used during startup until the superheater outlet temperature reaches about 400ºC to preclude any liquid entering the electrolysis module. During some scenarios, nitrogen gas may continue to be injected during steady state operation. For instance, if a module is found to be particularly leaky, nitrogen can be used to increase the average molecular weight of the gas mixture and hence reduce hydrogen diffusion rates. The nitrogen can be supplied from either a compressed gas cylinder or from a liquid nitrogen Dewar.

Detailed process flow sheets were developed for the ILS design using the commercial system-analysis code UniSim. These flow sheets include all of the components present in the actual ILS facility such as pumps, heaters, condensers, and the electrolyzer. Since the electrolyzer is not a standard UniSim component, a custom one-dimensional electrolyzer model was developed for incorporation into the overall process flow sheet. This electrolyzer model allows for the determination of the H_2 production rate, average Nernst potential, cell operating voltage, gas outlet temperatures, and electrolyzer efficiency for any specified inlet steam, hydrogen, and sweep-gas flow rates, current density, cell active area, and

TABLE I

Component identifiers for Fig. 2.

ID	Component
1	Electrolysis stacks / module
2	Hot zone enclosure lid
3	Power supply and instrument racks
4	Electrical distribution cabinets
5	Data acquisition and control monitors
6	Deionized water system
7	Water supply metering pump
8	Steam generator
9	H_2 preheater
10	Steam and H_2 superheater
11	Air compressor
12	Air heater
13	Product finned cooler
14	Steam condenser
15	Condensate tank
16	H_2 mass flow meter
17	H_2 vent
18	Air and O_2 vent
19	Dew point sensor

external heat loss or gain. The model includes a temperature-dependent area-specific resistance (ASR) that accounts for the significant increase in electrolyte ionic conductivity that occurs with increasing temperature. Details concerning this one-dimensional model and its implementation in UniSim have been reported in [8, 9].

All of the system components and hardware were mounted on a skid that is 16 ft. long by 10 ft wide. A photograph of the ILS skid with the components identified is presented in Fig. 3. The components are listed in Table I by identification number. A custom LabView (National Instruments) program was developed for ILS data acquisition and instrument control using SCXI data acquisition hardware.

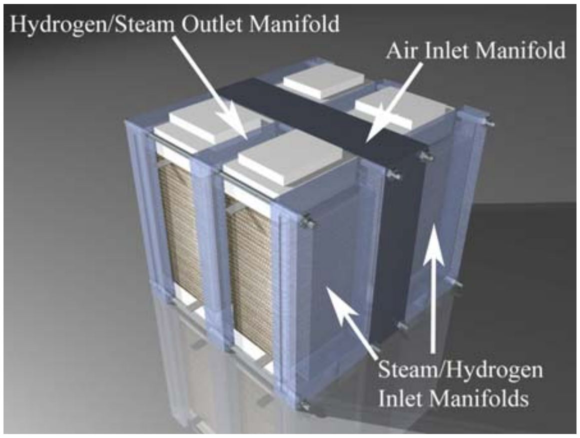

Fig. 4. ILS 4-stack module.

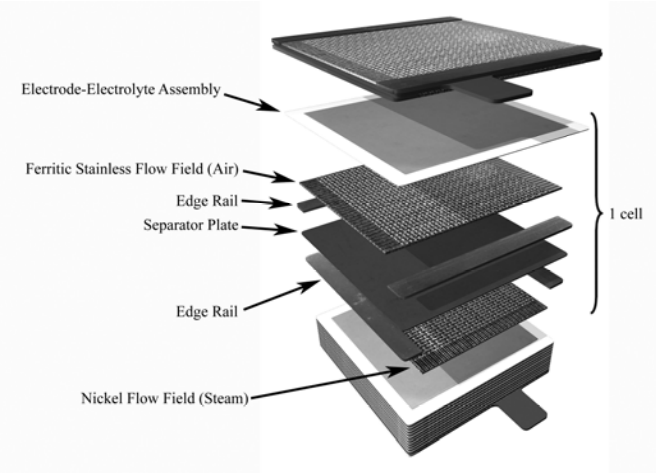

Fig. 5. Diagram of solid-oxide stack components.

II. ELECTROLYSIS MODULE

Planar stacks used for testing by the INL are fabricated by Ceramatec, Inc., of Salt Lake City, UT. They have an active area of 64 cm^2 per cell, providing a total active area of 3840 cm^2 in a sixty-cell stack. They are designed to operate in cross flow, with the steam / hydrogen gas mixture entering the inlet manifolds on the right and left sides in Fig. 4, and exiting through the outlet manifold visible in Fig. 4. Airflow enters through an air inlet manifold (Fig. 4) and exits through the front and back open faces directly into the hot zone enclosure.

The internal components of the stack are shown in Fig. 5 and are comprised as follows. The interconnect plate is fabricated primarily from ferritic stainless steel. It includes an impermeable separator plate (~0.46 mm thick) with edge rails and two corrugated flow fields, one on the sweep-gas side and one on the steam / hydrogen side. The height of the flow fields is 1.0 mm. Each flow field includes 32 perforated flow channels across its width to

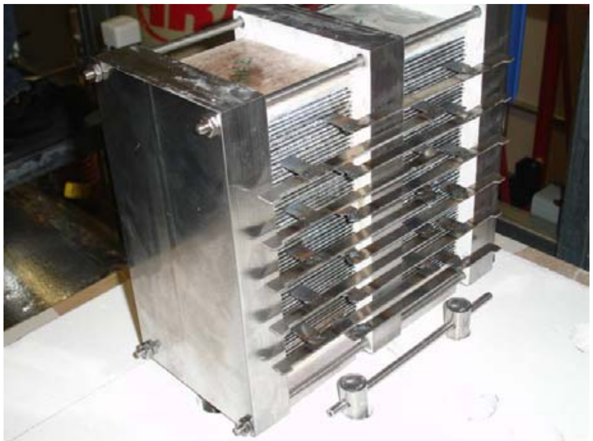

Fig. 6. One-half of ILS module showing electrical interconnections.

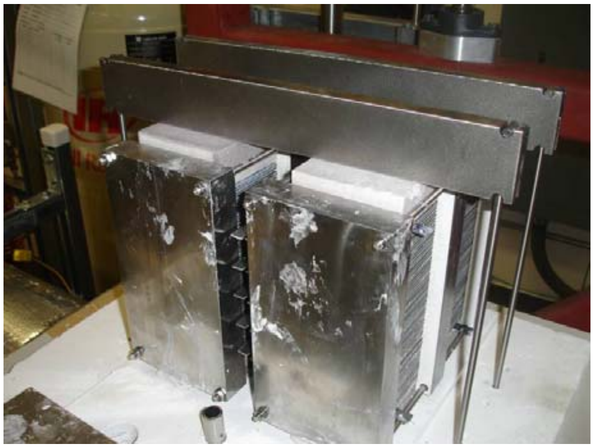

Fig. 7. ILS module with spring-loaded compression bars.

provide uniform gas-flow distribution. The steam / hydrogen flow fields are fabricated from nickel foil. The air-sweep flow fields are made from ferritic stainless steel. The interconnect plates and flow fields also serve as electrical conductors and current distributors. To improve performance, the sweep-side separator plates and flow fields are surface-treated to form a rare-earth stable conductive oxide scale. A perovskite rare-earth coating is also applied to the separator plate oxide scale by either screen printing or plasma spraying. On the steam / hydrogen side of the separator plate, a thin (~10 μm) nickel metal coating is applied.

The electrolyte is scandia-stabilized zirconia, ~140 μm thick. The sweep-side electrode (anode in the electrolysis mode) is a strontium-doped manganite. The electrode is graded, with an inner layer of manganite/zirconia (~13 μm) immediately adjacent to the electrolyte, a middle layer of manganite (~18 μm), and an outer bond layer of cobaltite. The steam / hydrogen electrode (cathode in the electrolysis mode) is also graded, with a nickel cermet layer (~13 μm) immediately adjacent to the electrolyte and a pure nickel outer layer (~10 μm).

The electrolysis module is composed of four stacks, each consisting of sixty cells (Fig. 4). Each pair of stacks is called a half module (Fig. 6). To preclude the loss of an entire stack if a single cell fails, the four stacks are electrically interconnected at every fifth cell. This is done by first electrically interconnecting the pair of stacks in each half module (Fig. 6), and then interconnecting the two half modules when they are in final position. When the two half-modules are placed back-to-back a common air inlet plenum for all four stacks is formed. Spring loaded bars are placed over the stacks to maintain a compressive load on the stacks during operation (Fig. 7). Power leads to each stack, intermediate voltage taps and interior thermocouples were then attached, and subsequent sealing of gaps completed the installation (Fig. 8).

A summary of the operating parameters and nominal predicted performance characteristics of the ILS for one module is provided in Table II. Three modules will be incorporated in the final ILS configuration, each of which will include 4 stacks of 60 cells each, totaling 720 cells. The nominal performance of the 3-module system can be

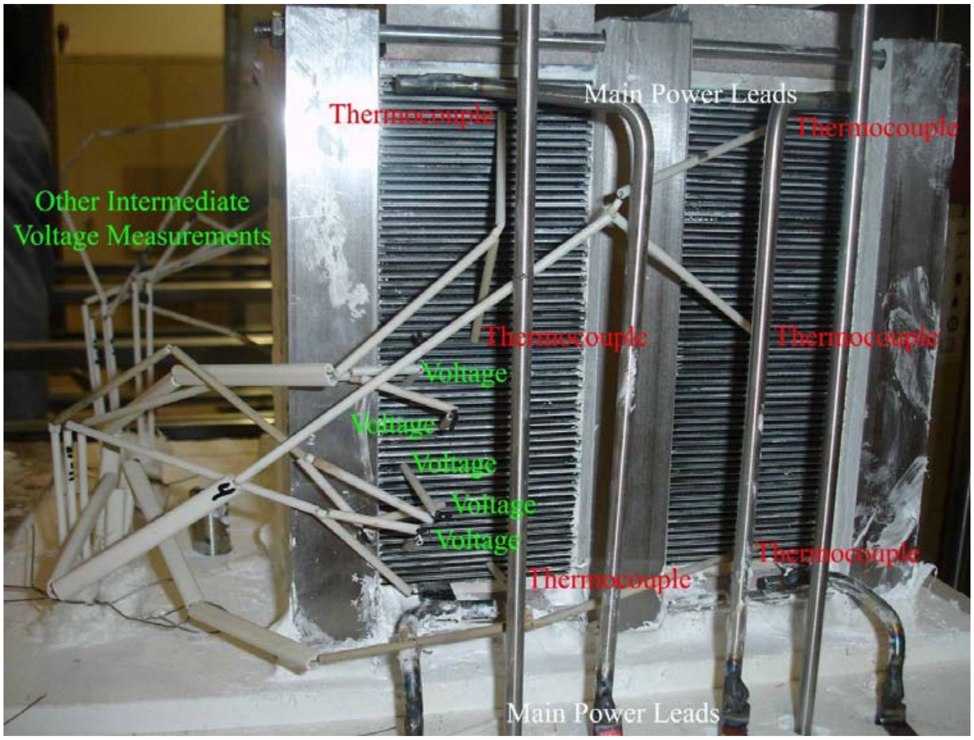

Fig. 8. Final installation of ILS module with instrumentation and power attachments.

TABLE II.

ILS single module design parameters.

Independent Design and Operational Parameters	
active cell area	64 cm^2
cells per stack	60
number of stacks	4
stack operating temperature	830°C
steam utilization	50%
stack operating voltage	77 V
per-cell ASR	1.5 Ωcm^2
inlet steam mole fraction	0.9
inlet hydrogen mole fraction	0.1
Anticipated Performance Values	
per-cell operating voltage	1.283 V
current density	0.25 A/cm^2
stack power	1232 W
total power (electric)	4.85 kW
inlet hydrogen flow rate	5.8 NLPM
inlet steam flow rate	53 NLPM
inlet liquid water flow rate	0.7 g/s
air flow rate	22.6 NLPM
hydrogen production rate	1578 NL/hr
heating value of hydrogen produced	4.7 kW (LHV)

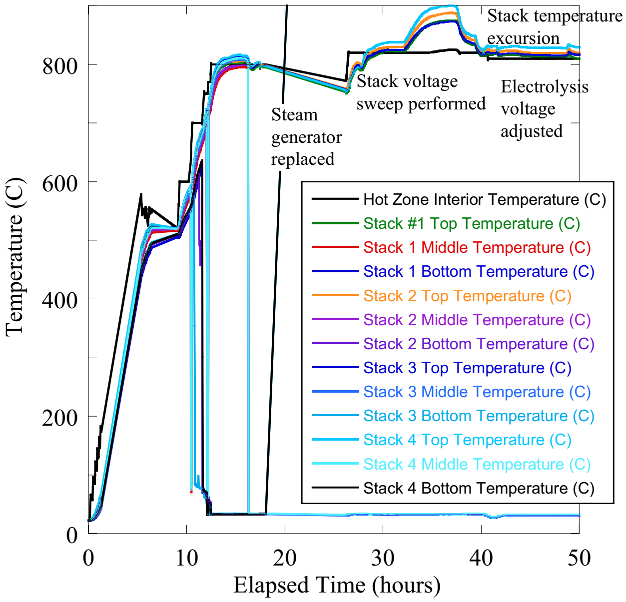

Fig. 9. ILS initial module heatup temperature trace.

scaled from Table II. All three of the ILS modules will be incorporated into a single hot zone for the ILS.

The per-cell ASR value of 1.5 Ωcm^2 in Table II represents a conservative value that has already been validated in stack tests. Note that for nominal operating conditions with a modest current density of 0.271 A/cm^2 and the flow rates shown, a hydrogen production rate in excess of 1700 NL/hr could be expected. Actual test results showed average H_2 production rates lower than this, but peak H_2 production rates were higher.

III. ILS TEST RESULTS

Fig. 9 presents the hot zone and stack internal temperatures for the heatup period as well as the initial voltage sweep discussed below. Steps in the hot zone temperature correspond to points in the heatup procedure where changes in gas flow settings were made.

The hot zone cool-down between 6 and 9 hours elapsed test time was due to installation of a computer-operated relay to disengage the ILS module power supply from the module. To accurately measure the open cell potential of the ILS module, the module must be in the open–circuit condition. Furthermore, installation of the relay reduces the chances of accidentally allowing the module to operate in fuel cell mode, with the power supply serving as a current sink.

The second large data perturbation between approximately 18 and 26 hours elapsed test time was due to installation of new clamp heaters on the steam generator. Several internal stack thermocouples were lost at approximately 17 hours as well.

Finally, there is a slight internal stack temperature excursion at approximately 33 to 38 hours elapsed test time. Some steam condensate formed in an uninsulated line leading from the hot zone steam/H_2 outlet to the product cooler. This condensate eventually imposed a backpressure upon the module, causing some H_2 to leak and subsequently combust (at operating temperature, the hot zone is well above the auto-ignition temperature of hydrogen, so any mixing of hydrogen and air results in a diffusion flame). The heat of combustion, in turn, forced a module temperature increase. Since the ASR is inversely related to temperature, the increased operating temperature reduced the effective ASR and allowed greater electrical current throughput and higher H_2 production. During the temperature excursion, internal module temperatures approached 900ºC and the module H_2 production rate peaked at over 2 Nm^3/hr. Lowering the module electrolysis voltage reduced the internal temperatures. Addition of insulation to the line eliminated the condensate problem in that part of the ILS system.

III.A. ILS Module Voltage Sweep

At approximately 27 hours elapsed test time the ILS module performance was tested by sweeping the module power supply voltage over the range of 50 to 79 V (0.83 V/cell to 1.32 V/cell). This range corresponds to operation from the open-cell voltage to slightly above the thermal neutral voltage. The operating conditions for the ILS

TABLE III.

Operating conditions for ILS module voltage sweep.

Hot zone temperature	820 C
Inlet water mass flow rate	34 ml/min
Inlet H_2 flow rate	5.4 Nl/min
Inlet N_2 flow rate	5.4 Nl/min
Inlet Air flow rate	25 Nl/min
Predicted OCV	50.5 V
Measured OCV	49.6 V
Predicted inlet dew point	90.3 C
Measured inlet dew point	91.3 C
Outlet dew point at OCV	90.2 C

module voltage sweep are listed in Table III. The corresponding voltage / current (VI) or polarization curve is displayed in Fig. 10. The average per cell ASR for the initial ILS module, represented by the average slope of the VI curve, was measured to be 2.38 Ωcm^2. This value was significantly higher than the design value of 1.5 Ωcm^2, but was not unexpected. Subcontractor Cermatec Inc, the manufacturer of the ILS module, expected lower performance from this particular module due to manufacturing difficulties they had encountered. After testing samples of cells manufactured since this module, Ceramatec is certain that these problems have been solved and future modules should exhibit higher performance.

Stack internal temperatures initially decreased during the voltage sweep, due to the endothermic heat of reaction for water splitting. Once the operating voltage exceeded the thermal neutral voltage (77V for 60 cells), outlet gas temperatures exceeded inlet values.

Fig. 11 presents inlet and outlet dewpoint temperatures and the hydrogen production rate for the ILS initial single-module sweep. The inlet gas dew point remained essentially constant at 89.6°C throughout the duration of the sweep. The outlet stream dew point temperature decreased continuously through the sweep as the operating voltage and stack current increased. The straight black line in Fig. 11 represents the hydrogen production rate based on electrolysis current, while the green trace is the hydrogen production rate based on the difference between inlet and outlet dew points. Agreement between the two independent measurements of hydrogen production was generally excellent. At the highest current levels, H_2 production rates exceeded 1.5 Nm^3/hr (25 Nlpm).

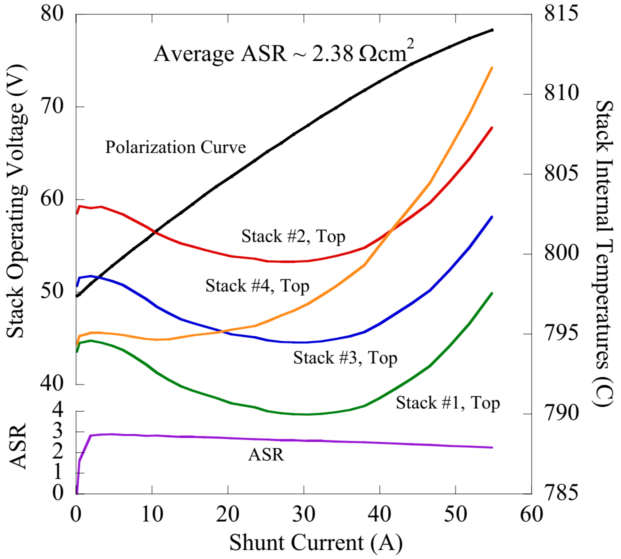

Fig. 10. ILS module voltage sweep / polarization curve.

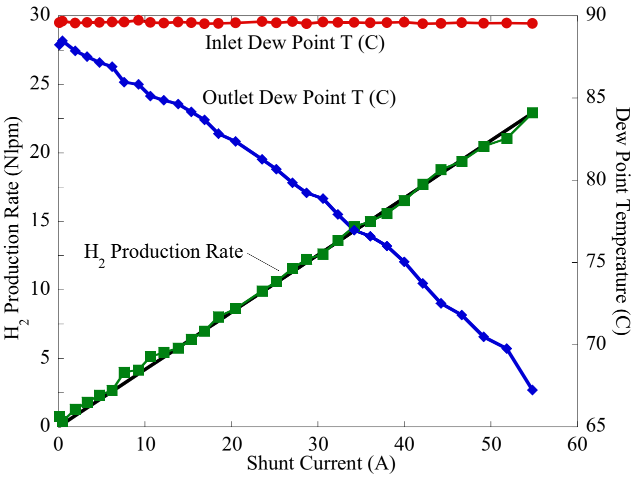

Fig. 11. ILS module voltage sweep hydrogen production rates and dew points.

III.B. ILS Module Long Duration Test

After performing a voltage sweep, the ILS operating parameters were set to the conditions listed in Table III. Fig. 12 presents the complete time history of module voltage, current, and H_2 production rate. The test was concluded after 420 hours duration. This was not due to any equipment failure. It was the opinion of the researchers that since the stack performance degradation had essentially stopped after about 250 hours, there was little more to be learned from the test.

Over the period of the test, the H_2 production rate dropped from over 1.5 Nm^3/hr to a steady value of 0.7

TABLE III

Long duration operating conditions for ILS module.

Hot zone temperature	810-820 C
Inlet water mass flow rate	34 ml/min
Inlet H_2 flow rate	5.4 Nl/min
Inlet N_2 flow rate	5.4 Nl/min
Inlet Air flow rate	25 Nl/min
Measured inlet dew point	91.3 C
Module operating voltage	78 V

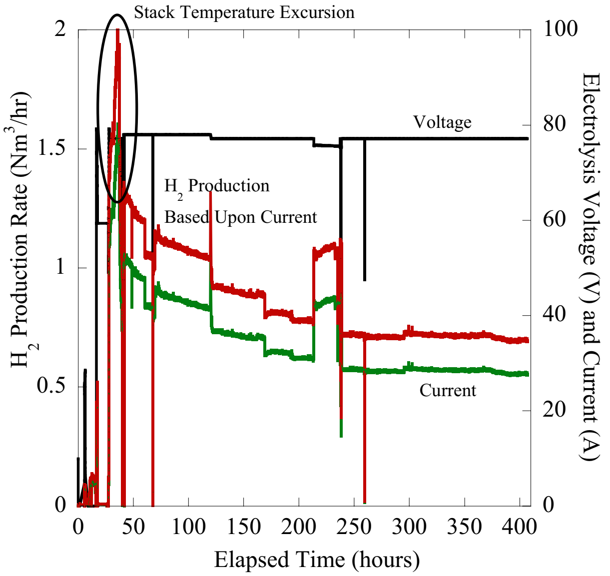

Fig. 12. Complete electrical history of ILS module.

Nm^3/hr. An issue that arose during the course of long-duration testing concerned the intra-stack temperatures. The thermocouples inserted into the stack were 0.020" outer diameter, inconel-sheathed, ungrounded Type K. While inserted, these thermocouples are in direct contact with the metallic air flow fields, which are at an elevated voltage potential whose magnitude is dependent upon which cell they are part of. Over time, the internal insulation of one such thermocouple deteriorated, eventually to the point that it was no longer ungrounded. When grounded, the thermocouple introduced a bias voltage into the DACS, which could be greater than 60 V. For short stack testing in the past, this was never a problem since the bias voltage would be much smaller. Such a large bias voltage affected other readings within the DACS. Eventually, all intrastack measurements were disconnected to eliminate the bias voltage from the DACS. To solve this problem in the future, a separate isolated DACS will be used for intrastack temperature measurements.

Once all test objectives had been successfully met, the test was terminated in a controlled fashion. Fig. 13 is a post-test photograph of the ILS initial electrolysis module after 420 hours of operation. No significant signs of corrosion are evident.

Fig. 13. Post test photograph of the ILS initial module.

IV. SUMMARY

The first test of the INL ILS facility was conducted with one electrolysis module in place. Initial module testing continued for 420 hours. A module polarization curve was generated, which showed that the module performance was lower than the design specification. This lower performance, however, was expected and explained by fabrication difficulties at the subcontractor's facilities. The test-average H_2 production rate was approximately 0.9 Nm^3/hr, with a peak measured value of over 2 Nm^3/hr. Significant module performance degradation was observed over the first 250 hours, after which no further degradation was noted for the remainder of the test.

Several relatively minor problems were encountered during testing, but were resolved without significant impact upon the test. These problems, plus the operational experience gained from the test, have identified several modifications that will be incorporated into the facility components to improve reliability and ease of operation for future long-term testing. The next phase of testing, planned for 2008, will incorporate 3 modules for an estimated H_2 production rate of over 4.7 Nm^3/hr.

ACKNOWLEDGMENTS

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