
High-Temperature Electrolysis Test Stand

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Subcontractor:
OxEon Energy, LLC, North Salt Lake, UT (provider of HTE stacks and technical support)

Project Start Date: August 1, 2016
Project End Date: Project continuation and direction determined annually by DOE

Overall Objectives

- Deploy a 25 kW high-temperature electrolysis (HTE) flexible test facility at the Idaho National Laboratory (INL) Energy Systems Laboratory.
- Integrate the HTE system with co-located thermal energy systems, including a high-temperature, high-pressure water flow loop and a thermal energy storage system.
- Integrate the HTE test station with co-located digital real-time simulators for dynamic performance evaluation and hardware-in-the-loop simulations.
- Perform HTE stack testing using hardware obtained from industry partners; focus on flexible intermittent and reversible operation and the effects of flexible operation on long-term performance.
- Work with HTE industry partners to demonstrate performance of flexible intermittent operation of large HTE systems.

Fiscal Year (FY) 2019 Objectives

- Complete full 25-kW system installation and individual component testing.

- Perform integrated system shakedown testing.
- Establish industry subcontract for delivery of initial 5-kW solid oxide electrolysis cell (SOEC) stack.
- Perform initial SOEC stack testing at 5-kW scale.
- Document 5-kW stack performance via voltage-current sweeps and long-term operation.
- Demonstrate overall facility system performance.
- Demonstrate system ramping response in accordance with the FY 2019 Annual Operating Plan milestone.
- Restart 5-kW testing to determine the effects of thermal cycling on stack performance.
- Perform additional long-term stack testing for a minimum of 200 hours, assuming the stack performance is satisfactory.
- Complete the details of the September 30, 2019, Annual Operating Plan milestone including the digital real-time simulator communication

Technical Barriers

This project addresses the following technical barriers from the Technology Validation section of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan¹:

- (G) Hydrogen from Renewable Resources
- (H) Hydrogen and Electricity Co-Production.

Contribution to Achievement of DOE Milestones

This project will contribute to achievement of the following DOE milestones from the Technology Validation section of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration Plan:

- Milestone 3.5: Validate distributed production of hydrogen from renewable liquids at a

¹ <https://www.energy.gov/eere/fuelcells/downloads/fuel-cell-technologies-office-multi-year-research-development-and-22>

projected cost of \$5.00/gge and from electrolysis at a projected cost of \$3.70 with an added delivery cost of <\$4/gge (4Q, 2018).

- Milestone 3.9: Validate large-scale system for grid energy storage that integrates renewable hydrogen generation and storage with fuel cell power generation by operating for more than 10,000 hours with a round-trip efficiency of 40% (4Q, 2020).

FY 2019 Accomplishments

- Completed installation and individual component testing of full 25-kW HTE system.
- Completed integrated system shakedown testing.
- Completed initial HTE 5-kW stack procurement with OxEon Energy.
- Completed initial testing of 5-kW stack, including performance characterization and long-term test of 100 hours.
- Demonstrated all operational and safety interlock features of the 25-kW test facility, including hydrogen recycle.
- Demonstrated data communication and HTE system control via the digital real-time simulator link in the INL Systems Integration Laboratory.
- Restarted the 5-kW HTE test after 2 months, verifying no stack performance degradation associated with thermal cycling.
- Performed additional long-term testing for more than 500 hours with good stack long-term performance

INTRODUCTION

High-temperature electrolysis of steam for hydrogen production is an advanced water splitting technology that exhibits high electric-to-hydrogen efficiency, especially when coupled to an integrated high-temperature process heat source. INL developed a world-class HTE laboratory and test capability under the DOE Office of Nuclear Energy Nuclear Hydrogen Initiative during the 2002–2012 time period. The focus of the current project is to establish a new HTE research and demonstration facility at INL to enhance the existing INL core capability in HTE and to support systems integration, systems operation, HTE model validation, and technical performance characterization of advanced hydrogen production by high-temperature water splitting. The initial thrust of this project is the development of a 25-kW flexible test station to support integrated operation of state-of-the-art HTE stack technologies from multiple industry partners. Establishment of the 25-kW HTE system will be followed by deployment of a test skid with infrastructure support for large-scale turnkey HTE systems up to the 250-kW scale.

The new HTE test capability has been designed for integrated operation with the INL Power Systems Test Bed (comprising real-time digital/real-time simulation units and a renewable-power microgrid) and the forthcoming Dynamic Energy Transport and Integration Laboratory (DETAIL). The HTE system will be co-located with a Thermal Energy Distribution System (TEDS) that is currently in procurement, with funding from the DOE Office of Nuclear Energy, under the Nuclear-Renewable Integrated Energy Systems initiative. TEDS will have its own high-temperature (up to 340°C) heater to support independent operation as a heat source. It will also include a large (200 kW·h) thermal energy storage system based on a packed-bed thermocline tank with a solid alumina particulate sensible heat storage medium. System integration will enable assessment and characterization of dynamic HTE operation to simulate load leveling capability with intermittent power from renewables and a fluctuating demand profile. This project leverages emerging and demonstrated high-temperature water splitting by HTE and high-temperature solid oxide cell technology, which may include reversible HTE/solid oxide fuel cell operation.

APPROACH

The new HTE technology validation capability under development at INL will include both a 25-kW flexible HTE test station plus infrastructure support for up to 250-kW HTE turnkey systems. At the 25-kW scale, this approach will enable thermal integration with co-located thermal energy sources as well as integrated operation with the INL Power Systems Test Bed. The 25-kW system will be flexible, allowing HTE operation from the 5-kW to the 25-kW scale, with support for intermittent and reversible operation. INL will work with

various industrial partners to supply the HTE stacks and to design the test matrices. The 250-kW infrastructure installation will support demonstration and testing of industry-supplied pilot-scale turnkey systems with grid integration and variable operation.

The flexible HTE test station has been designed to support HTE operations in the 5 to 25 kW range. At the 25-kW scale, the hydrogen production rate will be approximately 135 SLPM or 12.1 gm/min. For a typical solid oxide electrolysis cell (SOEC) active area and current density of 144 cm² and 0.67 A/cm², respectively, and four stacks, the current and voltage would be 96.5 A and 65 V per stack. The test station includes four DC power supplies, each rated at 100 V and 100 A to supply electrolysis power to the stacks. The HTE system was assembled on a skid with all of the hydrogen-containing components positioned inside of a ventilated enclosure with an active gas monitoring system that is interlocked to the hydrogen production process for safety. The ventilation duct is fitted with an air-flow switch. In the event of a loss of ventilation air exhaust flow, the HTE system will be placed into a safe mode via the system interlock controller.

The HTE system includes hydrogen recycle, which provides a steady flow of hydrogen to the inlet side of the stacks to prevent oxidation of the steam/hydrogen electrode. During startup, hydrogen is supplied from compressed gas cylinders, but for long-term operation, a fraction of the hydrogen that is produced by steam electrolysis is recycled from the electrolyzer outlet flow back to the inlet after residual steam is removed by a combination of low-pressure condensation, compression, and high-pressure condensation. Condensation is aided by cooling the condenser units and the counterflow heat exchanger using chilled water at 5°C delivered from a refrigerated chiller. During long-term operation, recycled hydrogen is drawn from the electrolytically produced hydrogen at the stack outlet, compressed, and stored in a hydrogen recycle storage tank from which it is fed back into the inlet side of the stacks via the hydrogen mass flow controller.

Additional safety features associated with hydrogen recycle include a check valve that prevents backflow of air from the hydrogen exhaust vent. This valve closes if the recycle compressor is running and there is not enough hydrogen exhaust flow coming from the HTE stacks to support the compressor flow requirement. In this case, a pressure transducer in the hydrogen recycle line will indicate low pressure and will shut off the recycle compressor to avoid any possibility of mixing air into the hydrogen recycle tank. In addition, an oxygen sensor is installed on the hydrogen storage tank inlet line that will also shut off the hydrogen recycle compressor if oxygen is detected.

An overview photograph of the installation is provided in Figure 1. Major system components include a large top-hat furnace, steam generator/superheater, air compressor, chiller, hydrogen compressor, air-cooled hydrogen outlet finned tube array, water-cooled condensers, a hydrogen storage tank, plus associated instrumentation including mass flow controllers, pressure transducers, and thermocouples. The ventilation exhaust duct from the experiment enclosure is visible in the top of Figure 1. A blower that is mounted outside of the building draws air from the surrounding high-bay laboratory through the experiment enclosure, through the exhaust duct to the outside blower. The hydrogen vent line can also be seen in the top of Figure 1. The steam generator is an inductively heated unit that delivers high-temperature superheated steam (up to 900°C) directly from an inlet flow of room-temperature liquid deionized water. The HTE system will also include a heat exchanger option (Phase II) for process-heat-based steam generation when the DETAIL thermal network is complete. The furnace that will house the HTE stacks has a moveable top hat that lifts up to expose the hot zone for installation of test articles. The electrolysis stacks are positioned within the hot zone for testing at 800°C.



Figure 1. Overview of the 25-kW HTE experiment skid and enclosure within the INL Systems Integration Laboratory.

The inlet flow to the electrolysis stacks will include steam plus hydrogen. Hydrogen must be included on the inlet side in order to maintain reducing conditions on the electrolysis cell cathodes. During startup, hydrogen will be supplied from compressed gas cylinders, but for long-term operation, a fraction of the hydrogen that is produced by steam electrolysis is recycled from the electrolyzer outlet flow back to the inlet after steam is removed by a combination of low-pressure condensation, compression, and high-pressure condensation. Condensation is aided by cooling the condenser units and the counterflow heat exchanger using chilled water at 5°C delivered from a refrigerated chiller. A finned-tube air-cooled natural convection heat exchanger is used for the initial cooling of the outlet hydrogen/steam flow that exits the hot zone at high temperature. Nitrogen is included as an inert carrier gas and a purge gas. Air is supplied to the anode side of the HTE stacks as a sweep gas to remove electrolytically produced oxygen from the anode side of the stacks. Compressed air is produced by an air compressor, metered by a precision mass flow controller, and preheated to 800°C using a high-temperature in-line gas heater prior to entering the hot zone.

Two 50-cell solid oxide electrolysis stacks supplied by OxEon Energy were installed in the INL High-Temperature Electrolysis 25-kW Research and System Integration Facility in late May 2019 (Figure 2 and Figure 33). Each stack has 50 electrolyte-supported cells (~150 μm thickness) with an active area of 110 cm^2 . Overall stack dimensions are 12.5 cm x 13.5 cm x 16.1 cm high. The steam/hydrogen electrode material is nickel-zirconia cermet and the air electrode material is lanthanum strontium cobalt ferrite. The stacks are a scaled-up version of smaller stacks that were developed for NASA Jet Propulsion Laboratory to demonstrate oxygen production from the Martian atmosphere on the Mars 2020 rover. These stacks incorporate two important innovations for the integrated system application. The interconnects are fabricated using powder metallurgy techniques from chrome-iron-yttria powders resulting in a coefficient of thermal expansion (CTE) that matches the CTE of the electrolytes. This matched CTE ensures that all the stack materials undergo the same thermal expansion/contraction during heat-up and cool-down such that the stacks can withstand multiple thermal cycles with minimal performance penalty. In addition, the matched CTE enables the use of glass seals, resulting in a hermetic stack.

The stacks are internally manifolded for the steam/hydrogen flow and externally manifolded for air sweep gas flow.

The stack installation process included welding of the four Inconel bus bars to the top and bottom stack plates. These bus bars supply electric power to the stacks from the DC power supplies during electrolysis operations. The stacks were powered independently by two Lambda 100 V, 100 A DC power supplies. The stacks are internally manifolded for the steam/hydrogen flow, which is supplied to the two stacks in parallel via two base manifold plates and interconnected tubing. The seal between the stack base plates and the base manifold plates is accomplished using a thermiculite-based seal material developed specifically for solid oxide stacks (Flexitallic 866 LS). The seals and stacks were maintained in mechanical compression throughout the test using dead weights. The stacks were instrumented with intermediate voltage taps every five cells (Figure 2b). The inlet flows of steam/hydrogen and air enter the stack after passing through coiled tubing inside the furnace to ensure that these gases enter the stacks at the desired stack operating temperature of 800°C (Figure 3).

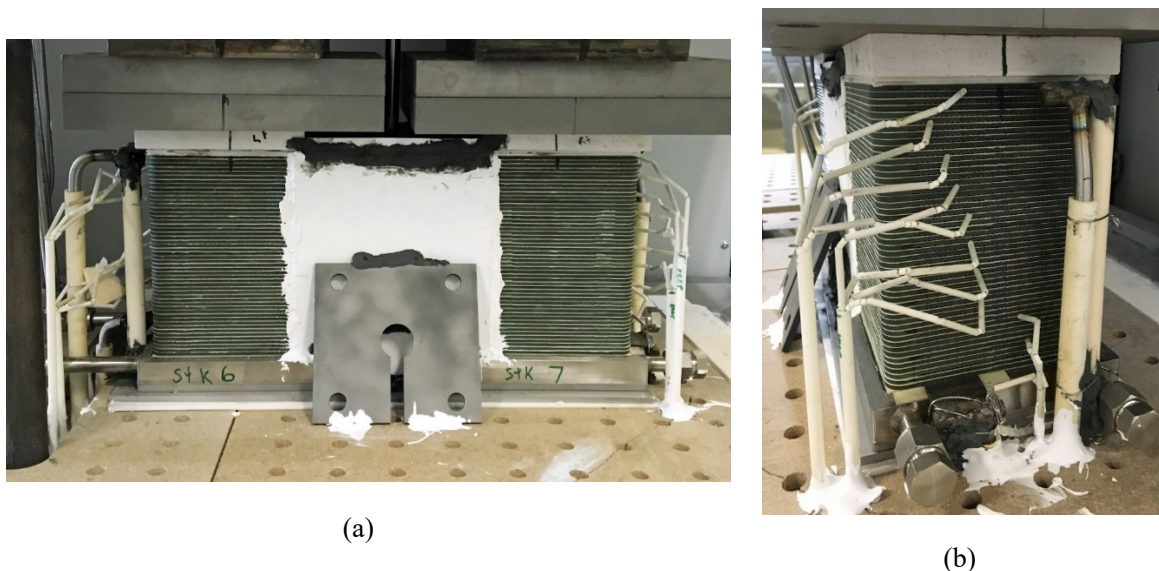


Figure 2. (a) High-temperature electrolysis stacks, 2.5 kW per stack installed in INL furnace; (b) intermediate voltage taps



Figure 3. 5-kW stack installation in INL furnace showing coiled process gas inlet lines and dead weights

RESULTS

Following stack heat-up and electrode reduction, initial stack performance characterization was determined using cyclic voltammetry from open-cell conditions (~42 V/stack) to slightly above thermal neutral (~65

V/stack), which corresponds to 100% electrical efficiency. For the initial test series, these sweeps were performed at the start of testing and after long-term (~100 h) operation. Results of these sweeps are presented in Figure 4. This figure also shows the electrolysis power as a function of current for one stack during the V_i sweeps. Note that the maximum power is significantly greater than 2.5 kW/stack. The cyclic voltammetry indicates better performance at the start of testing compared to after long-term testing, indicating some initial degradation.

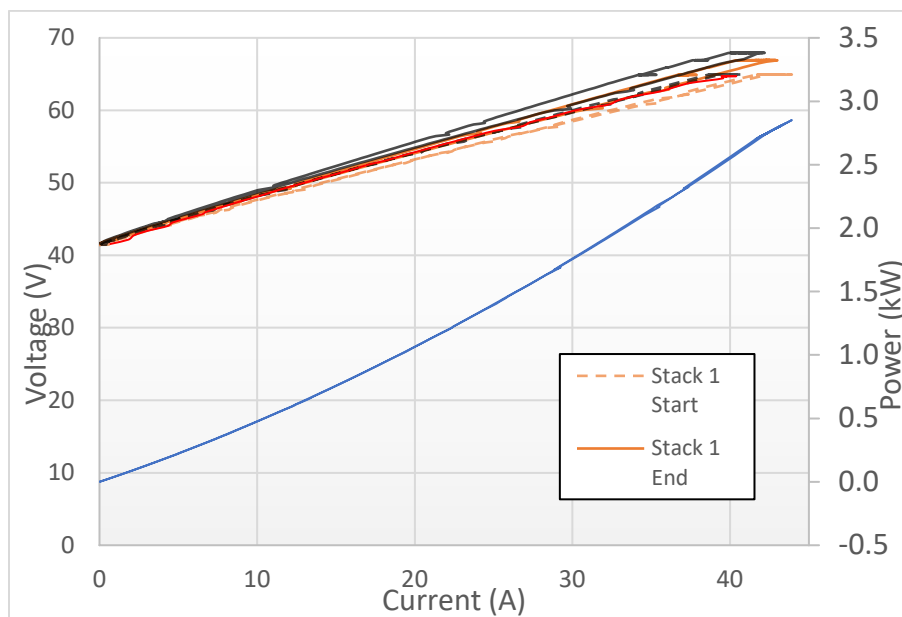


Figure 4. Results of initial and final cyclic voltammetry for the two OxEon stacks

Results of long-term testing are presented in Figure 5. For the initial long-term testing (first 100 h), the stacks were operated at a constant power supply voltage of 58 V. Constant voltage operation is preferable for the electrolysis mode of operation because it fixes the thermal operating condition of the stack: endothermic, exothermic, or thermal neutral. At constant stack voltage, any decrease with time indicates performance degradation. Some degradation is evident in the results presented in Figure 5. Initial stack operation was terminated after ~100 hours of initial operation in order to address an issue with the furnace. One of the bottom heating zones was not working, resulting in a vertical temperature gradient in the stack, with higher temperatures at the top of the stack and lower temperatures in the bottom cell groups. This issue was resolved during shutdown, along with other minor operational issues that were encountered during initial testing. Operation of the 5-kW stack was restarted in September 2019. Post-restart V_i sweeps were performed, followed by long-term operation for an additional 450 hours. Results of one of the post-restart V_i sweeps were added to Figure 4 for comparison with the initial sweeps. Remarkably, this post-restart sweep is virtually identical to the sweep obtained at the end of the initial 100 hours of testing, indicating no performance loss associated with a full thermal cycle.

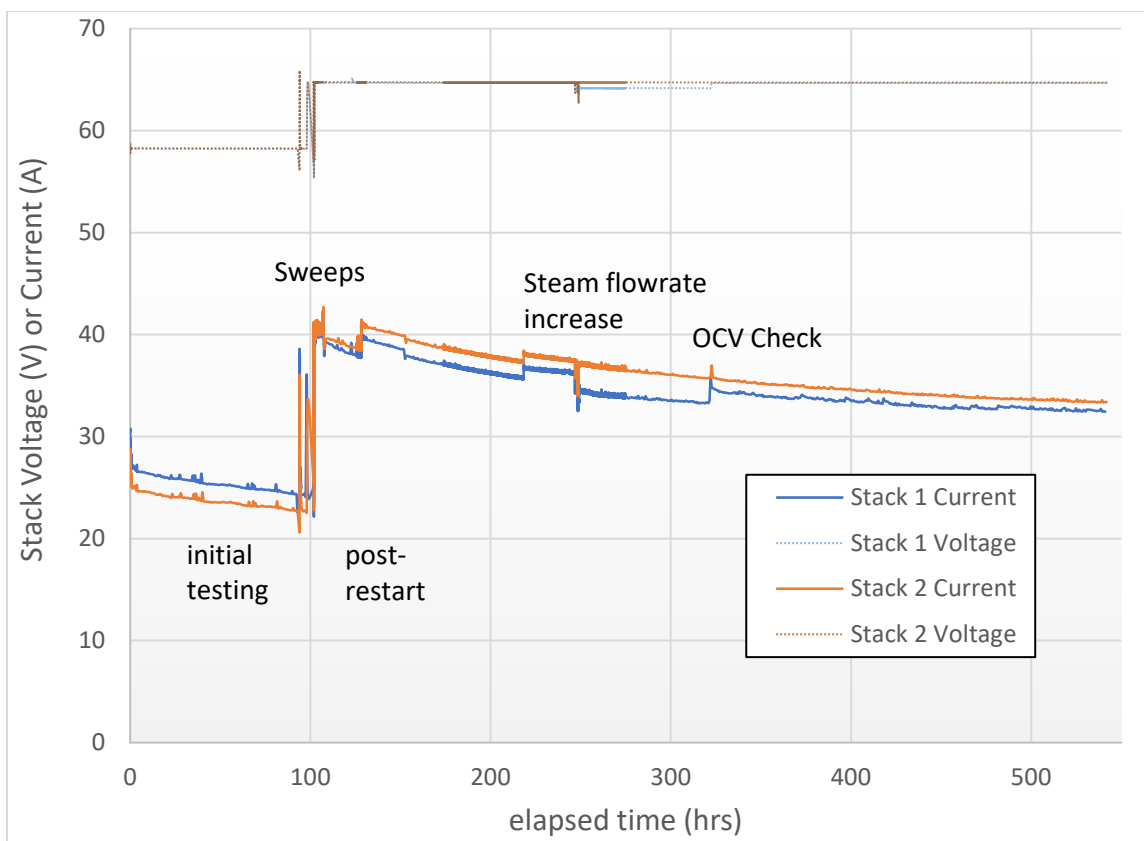


Figure 5. Results of long-term testing, OxEon stacks

Long-term post-restart test results were appended to the results obtained during the initial 100 hours of operation, as shown in Figure 5. Total long-term test time is now nearly 550 hours. This test was terminated at 550 hours due to a power failure that occurred during the night, placing the system in a safe shutdown mode and initiating a cooldown. The system will be checked out and restarted again to determine the effects of thermal cycling and to obtain additional long-term performance and degradation data. The stacks were operated at an overall stack voltage of 58 V during the initial 100 hours of operation. With a more uniform stack temperature distribution, the stack operating voltage was increased to 65 V during post-restart long-term testing, yielding higher current densities and correspondingly higher hydrogen production rates. Performance degradation, as indicated by decreasing stack current with time, continued during post-restart testing. The degradation rate appears to be slowing significantly with time, especially after 450 hours of operation.

Hydrogen production rate is directly proportional to stack current and is equal to 3.61 kg /day at an average stack current of 40 A, such as after stack restart. Total stack power at this point was 5.2 kW. Stack efficiency is inversely proportional to operating voltage. Therefore, for constant-voltage operation at the thermal neutral voltage, the stack efficiency is fixed at 100%, but the hydrogen production rate drops as the stack current and stack power decrease. At the end of the test at 550 hours, the stack current had dropped to an average of 32.9 A and the corresponding hydrogen production rate had dropped to 2.97 kg/day at a total stack power of 4.28 kW

CONCLUSIONS AND UPCOMING ACTIVITIES

Operation of the new INL 25-kW flexible HTE test station has been demonstrated at the 5-kW scale with a total operational time of ~550 hours to date. The system performed well, and all operational and safety features were verified. Results of stack testing indicated good initial stack performance followed by performance degradation. The degradation rate slowed significantly during long-term testing, especially after ~450 hours of running time.

Additional testing with the current 5-kW stacks will be done during Q1 of FY 2020. INL will also be procuring additional stacks from OxEon to enable operation of the test stand at its full rated capacity of 25 kW.

FY 2019 PUBLICATIONS/PRESENTATIONS

1. J.E. O'Brien, "A 25 kW High Temperature Electrolysis Facility for Flexible Hydrogen Production and System Integration Studies," 2nd International Conference on Electrolysis, Loen, Norway, June 2019.
2. S.M. Bragg-Sitton, R.D. Boardman, C. Rabiti, and J.E. O'Brien, "Reimagining Future Energy Systems: Maximizing Energy Utilization via Integrated Nuclear-Renewable Energy Systems," *International Journal of Energy Research*, submitted for review, July 2019.