Standard Operating Procedure

(SOP)

Large-Area Single-Cell SOEC Testing

Test ID: HTE-P-25

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Revision History

This page documents the revisions over time to the SOP. The most recent iteration should be listed in the row space, with consecutive versions following.

Date of Revision	Page(s)/Section(s) Revised	Revision Explanation
1/28/2021	all	First draft

3. Procedures

a. Scope and Applicability – This SOP describes processes for performing single-cell testing of large-area, electrolyte-supported and anode-supported solid oxide electrolysis cells (SOECs).

b. Summary of Method – A large-area electrolyte-supported (or anode supported) planar cell, with air and fuel electrodes on opposite sides is sealed within a pair of testing manifolds, which is then heated to the testing temperature. For SOFC mode testing, a fuel mixture of hydrogen and nitrogen is fed to the fuel electrode and air is fed to the air electrode. In SOFC mode testing, a (positive) current is applied to the electrodes such that oxygen transports through the electrolyte and forms water at the fuel electrode, and the voltage is measured (and resulting power calculated). For SOEC mode testing, a fuel mixture of steam and hydrogen is fed to the fuel electrode and air is fed to the air electrode. In SOEC mode testing, a (negative) current is applied such that oxygen is removed from steam (generating hydrogen) and is transported through the electrolyte to the air electrode, and the resulting voltage is measured.

c. Definitions

1. Anode is the fuel side for SOFC testing and the air side for SOEC testing. Cathode is the air side for SOFC testing and the fuel side for SOEC testing. To minimize confusion, air-side and fuel-side terminology is used in this SOP.

2. Active cell area – in this SOP, active cell area is defined by the area of the current collector meshes, not electrode area. The electrodes might be oversized to make it easier for opposite electrodes to completely overlap. In some cases, the total electrode area and active electrode area are the same, but this is not a necessity for this test.

3. OCV is open circuit voltage (or open circuit potential) and is the cell voltage with fuel and air on opposite sides of the cell when no current is applied to the cell.

4. sccm – standard cubic centimeter per minute (0 °C reference).

5. Current Density – current divided by active cell area.

d. Health & Safety Warning – Avoid touching hot surfaces. Practice appropriate caution with electrical equipment. Use Proper PPE when working with electrode powders (review SDS documentation).

e. Cautions – n/a.

f. Interferences – n/a.

g. Personnel Qualifications / Responsibilities – The user should have either an A.A.S or B.S. degree in an engineering discipline.

h. Equipment and Supplies – the following is required:

1. A power supply
2. A data acquisition system
3. Testing manifolds, cells & accessories:
    a. 5x5 cm^2: https://fuelcellmaterials.com/products/equipment/planar-cell/5-cm-x-5-cm-test-fixture-kit/
    b. 10x10 cm^2: https://fuelcellmaterials.com/products/equipment/planar-cell/10-cm-x-10-cm-test-fixture-kit/
4. A testing furnace, ideally with electronic control – an acceptable testing furnace product is here:https://fuelcellmaterials.com/products/equipment/planar-cell/10-cm-x-10-cm-test-system/
5. A mass flow controller or pump for precisely delivering a controlled amount of water. Also need appropriate means for converting water to steam.
6. A mass flow controller for delivering a controlled amount of hydrogen.
7. A mass flow controller for delivering a controlled amount of nitrogen.
8. A mass flow controller for delivering a controlled amount of air.
9. A planar cell to be tested having either 5 x 5 cm or 10 x 10 cm dimensions with electrodes on both faces.
    a. Acceptable electrolyte substrates are here: https://fuelcellmaterials.com/products/substrates/hionic/hionictm-planar-substrate/
    b. Often times, the electrolyte material and substrate are experimental materials that are being tested.
    c. Acceptable anode-supported bi-layer substrates are here: https://fuelcellmaterials.com/products/cells/anode-supported-cells/anode-electrolyte-planar-bi-layer/.
    d. Often times, the anode-supported bi-layer substrates are experimental components that are being tested.
    e. An acceptable air side electrode material is: LSCF: https://fuelcellmaterials.com/products/powders/cathode-powders/cathode-lanthanum-based/lanthanum-strontium-cobalt-ferrite-premium-powder/.

f. Another acceptable air side electrode material is LSCF/GDC: https://fuelcellmaterials.com/products/powders/cathode-powders/cathode-mixed/lscf-gdc-composite-cathode-powder/.

g. An acceptable fuel side electrode material is NiO/YSZ powder: https://fuelcellmaterials.com/products/powders/anode-powders/anode-mixed-nickel-electrolyte/nickel-oxide-ysz-anode-powder-for-coating-applications/.

h. Another acceptable fuel side electrode material is NiO/GDC powder: https://fuelcellmaterials.com/products/powders/anode-powders/anode-mixed-nickel-electrolyte/nickel-oxide-gdc-anode-powder-for-coating-applications/.

i. Often times, the air-side and fuel-side electrode materials are experimental materials that are being tested.

j. Note that air-side and fuel-side electrode powders need to be made into inks for electrode deposition via screen printing or painting. An appropriate ink vehicle is: https://fuelcellmaterials.com/products/inks/ink-vehicle/ink-vehicle/.

k. Acceptable (complete) electrolyte supported cells include: https://fuelcellmaterials.com/products/cells/electrolyte-supported-cells/nextcell-hp-electrolyte-supported-planar-cell/.

l. Acceptable (complete) anode supported cells include: https://fuelcellmaterials.com/products/cells/anode-supported-cells/anode-supported-planar-cell/.

10. Seal gaskets (two required per test) are cut to target size. Pre-cut seal gaskets are available here: https://fuelcellmaterials.com/products/equipment/test-accessories/seals/test-fixture-seals/

11. Corrugated silver mesh current collectors for air side of cell. Pre-cut and pre-corrugated silver mesh current collectors are available here: https://fuelcellmaterials.com/products/equipment/test-accessories/silver-mesh/

12. Contact paste for the air electrodes:

    a. An acceptable air-side contact paste can be made from $(La_{0.8}Sr_{0.2})_{0.95}CoO_{3-X}$ (LNC) powder: https://fuelcellmaterials.com/products/powders/cathode-powders/cathode-lanthanum-based/lanthanum-nickel-cobaltite-cathode-powder/.

    b. Another acceptable air side contact paste can be made from $(La_{0.8}Sr_{0.2})_{0.95}CoO_{3-X}$ (LSC) powder: https://fuelcellmaterials.com/products/powders/cathode-powders/cathode-lanthanum-based/lanthanum-strontium-cobaltite-cathode-powder/.

c. Often times, the air side contact paste materials are experimental materials that are being tested.

d. The air-side contact paste materials need to be made from powders into inks by mixing with an ink vehicle, for subsequent deposition via painting. An appropriate ink vehicle is: (https://fuelcellmaterials.com/products/inks/ink-vehicle/ink-vehicle/.

13. Corrugated nickel mesh current collectors for fuel side of cell. Pre-cut and pre-corrugated nickel foam current collectors are here: https://fuelcellmaterials.com/products/equipment/test-accessories/nickel-mesh/

14. Contact paste for the fuel electrode (Ni metal ink). An acceptable fuel side contact paste is here: https://fuelcellmaterials.com/products/inks/anode-inks/ni-metal/nickel-ink/. Note that the composition and particle size of fuel side contact paste materials often is an experimental material that is being tested. An appropriate ink vehicle for converting contact materials into inks is: https://fuelcellmaterials.com/products/inks/ink-vehicle/ink-vehicle/.

15. Lead wire. Acceptable lead wire materials include platinum, silver, or Inconel (0.001" to 0.002" diameter).

i. Step by Step Procedure – The procedure below applies for testing of planar electrolyte supported cells of 100 cm^2 (10 x 10 cm) with 34 cm^2 (6.2 by 5.5 cm) electrode (active) area. The procedure can be modified accordingly if testing smaller (5 x 5 cm cells). The procedure also can be modified for anode supported cells (this likely would involve lower testing temperatures).

Pre-Loading Preparation

1. Read through this entire procedure to become familiar with process prior to beginning.
2. Corrugate silver mesh current collectors and nickel foams to the same height as the seal gasket.
3. Cut silver mesh to 62.0 x 55.5 mm.
4. Cut nickel foam to 62.0 x 55.5 mm.
5. Alternative to (2) through (4) is to purchase pre-cut cut-to-size and pre-corrugated current collector meshes (see above).
6. Cut two 3-foot lengths of 0.001" to 0.002" diameter lead wire to serve as voltage taps.
7. Cut seal gaskets to required geometry or purchase commercial pre-cut seals (see above).
8. Prepare necessary contact paste inks using three-roll mill or Thinky mixing or procure commercial inks (see above).
9. Locate desired cell to be tested. Cell should be 10 by 10 cm area with electrodes with 6.2 cm by 5.5 cm on opposite sides.

Cell Loading

The procedure below is for 10 x 10 cm cells with 34 cm^2 active area electrodes. Specified material quantities should be adjusted based on actual cell active area.

1. Paint active area of fuel side manifold with 0.50 grams of Ni metal ink.
2. Trim nickel foam current collector (if necessary) to assure fit within the seal area.
3. Place seal gasket on top of manifold.
4. Place nickel foam on active area of fuel-side manifold and lightly press into place.
5. Evenly paint fuel-side (green) active area of cell with 1.5 grams of Ni ink.
6. Make a small spiral into voltage wire and place in the middle of the nickel foam on the fuel-side manifold.
7. Carefully align the cell and place the anode side against the nickel foam and wire in place on the fuel-side manifold.
8. Lightly holding the cell in place, paint the air-side (black) active area of the cell with 2.5 grams of air-side contact paste.
9. Check the size of the silver meshes against the seal on the cathode manifold to assure no overlap and good fit within active area. Trim if necessary.
10. Make a small spiral into second voltage wire and place in the middle of the painted cathode active area.
11. Place seal gasket on top of air-side of the cell ensuring the pattern matches that of the manifold gas holes.
12. Place the silver mesh onto the air-side active area and lightly press into place.
13. Paint the active area of the air-side manifold with 0.50 grams of air-side contact paste ink.
14. Carefully place air-side manifold in place on cell, aligning active areas as much as possible.
15. Place the single cell / manifold assembly into place in the furnace.
16. Place the cell weight mechanical load 13.5-pound steel bar on top of manifold.
17. Place appropriate insulation in place to separate fuel-side and air-side manifold tubing.
18. Place sleeve insulation on anode and air-side voltage taps to isolate from the furnace and one another.
19. Slowly and carefully lower furnace lid.
20. Connect the fuel-side and air-side inlet tubing and current cable connections.
21. Check that air-side current cable is unplugged from control box (and load box component is off) to assure no current is passing through the system during heat-up.
22. Program furnace to heat up to 850 °C at 1 °C/min and hold at 850 °C.

23. Increase air-side and fuel-side flow rates to appropriate desired flowrates for heat-up:
    a. Air Side: air to 350 sccm.
    b. Fuel Side: dry N_2 to 350 sccm.
24. Start furnace.
25. Allow cell and furnace to heat-up (typically, overnight).

Cell Reduction

The procedure below is for 10 x 10 cm electrolyte supported cells with 34 cm^2 active area electrodes. Gas flows should be adjusted based on actual cell active area.

1. Switch on load box component of control box and record Open Circuit Voltage (OCV).
2. Being sure not to allow a short in the cell, connect the anode exhaust tube to the copper tubing/condenser.
3. Maintain 350 sccm N_2 on the anode.
4. Increase cathode air flow to 500 sccm.
5. Allow system to sit for 30 minutes (or until OCV begins to stabilize).
6. Increase the H_2 to 150 sccm, air to 750 sccm and reduce the N_2 to 300 sccm.
7. Hold flows for 30 minutes so the OCV can stabilize.
8. Increase the H_2 to 250 sccm, air to 1000 sccm and reduce the N_2 to 200 sccm.
9. Hold flows for 30 minutes so the OCV can stabilize.
10. Increase the H_2 to 350 sccm, air to 1250 sccm and reduce the N_2 to 0 sccm.
11. Hold flows for 30 minutes so the OCV can stabilize.
12. Finally increase the H_2 to 450 sccm and the air to 1500 sccm maintaining the N_2 at 0 sccm.
13. Once at the desired first condition of 450 sccm H_2 and 0 sccm N_2 on the anode and 1500 sccm air on the cathode, allow system to sit for 30 minutes to assure steady state.

Cell Testing (SOFC Mode)

1. Record the OCV at the first condition. OCV should be at least 1.0 V at this condition (ideally > 1.1 V).
2. Obtain polarization curve:
    a. Step current in 0.5 amp, 30-second increments, and record voltages.
    a. Continue increasing current until voltage reaches pre-specified level (e.g., 0.7 volts), fuel utilization reaches a pre-set maximum (e.g., 80%), or instabilities are observed.
    b. Note any strange behavior or unusual performance.
    c. Reduce current gradually to zero (approximately one amp per minute).

3. Transition cell to second condition and complete VIR curve.
4. Repeat for all desired fuel and air flow conditions at this temperature. Note that the fuel and air flow conditions often are experimental variables that are tested. Example conditions at any temperature may include (for example):

Fuel Side H_2 flow (sccm)	**Fuel Side N_2 flow (sccm)**	**Air Side Air flow (sccm)**
450	0	1500
250	250	1500

5. Transition anode flow back to first condition (450 sccm H_2 and 0 sccm N_2).
6. Cool furnace to next testing temperature (typically 800 °C) and allow system to reach steady state.
7. Repeat various anode flow conditions recording OCV and completing a polarization curve at each condition.
8. Transition anode flow back to first condition (450 sccm H_2 and 0 sccm N_2).
9. Cool furnace to next testing temperature (typically 750 °C) and allow system to reach steady state.
10. Repeat various anode flow conditions completing a polarization curve at each condition.
11. Complete preliminary data analysis of cell's performance to determine next tests to be completed.
12. Transition cell to desired conditions following similar procedures to those used during reduction and testing (for flow rate and temperature changes).
13. Set the cell to the desired constant current or constant voltage conditions and start lifetime test.
14. Check that lifetime test is running properly.

Cell Testing (SOEC Mode)

1. Record the OCV at the first fuel cell condition (450 sccm H_2 and 0 sccm N_2) as a check on cell integrity. OCV should be at least 1.0 V at this condition (ideally > 1.1 V). Note that alternative fuel cell mode fuel-side and air-side gas compositions and flow rates can be used – the key is that OCV values from beginning of testing should be directly comparable.
2. Reverse electrical leads.
3. Transition gas flows to those required for SOEC testing. Note that the fuel and air flow conditions often are experimental variables that are being tested. Example conditions are as follows:

$H_2O:H_2$ Ratio	Fuel Side H_2O flow (sccm, liquid)	Fuel Side H_2 flow (sccm)	Air Side Air flow (sccm)
3/1	0.362	150	600
1/1	0.201	250	500

4. Obtain polarization curve:
    a. Step current in 0.5 amp, 30-second increments, and record voltages.
    b. Continue increasing current until voltage reaches pre-specified level (e.g., 1.4 volts), steam conversion level reaches a pre-set maximum (e.g., 80%), or instabilities are observed.
    c. Note any strange behavior or unusual performance.
    d. Reduce current gradually to zero (approximately one amp per minute).
5. Transition cell to second condition and complete polarization curve.
6. Repeat for all desired conditions at this temperature.
7. When all conditions have been tested, transition fuel flow back to first fuel cell condition (450 sccm H_2 and 0 sccm N_2).
8. Cool furnace to next testing temperature (typically 800 °C) and allow system to reach steady state.
9. Obtain polarization curves at each of the targeted fuel and air side composition and flow rate conditions.
10. Transition anode flow back to first fuel cell condition (450 sccm H_2 and 0 sccm N_2).
11. Cool furnace to next testing temperature (typically 750 °C) and allow system to reach steady state.
12. Obtain polarization curves at each of the targeted fuel and air side composition and flow rate conditions.
13. Complete preliminary data analysis of cell's performance to determine next tests to be completed.
14. Transition cell to desired conditions following similar procedures to those used during reduction and testing (for flow rate and temperature changes).
15. Set the cell to the desired constant current or constant voltage conditions and start lifetime test program.
16. Check that lifetime test is running properly.

Shut Down Procedure

1. Transition cell to 350 sccm dry N_2, 100 sccm H_2, and 500 sccm air.
2. Switch load box component of control box off.

3. Program furnace to cool at 3 °C/min to room temperature (20 °C).

Breakdown Procedure (once cell is shut down and cooled)

1. Disconnect fuel and air inlet and outlet tubes.
2. Disconnect voltage taps and current cables.
3. Disassemble and clean manifolds via sand-blasting.