Standard Operating Procedure

(SOP)

Solid Oxide Electrolysis Button Cell Testing

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Rev 1

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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
04/11/2019	All	First Release

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3. Procedures

a. Scope and Applicability – The purpose of this SOP is to describe the method for testing electrode/electrolyte supported solid oxide electrolysis cells with nickel based cathode materials. The best practice recommendations apply to single cells for the performance evaluation of materials.

b. Summary of Method – Test rig is cleaned and checked before using. Prior prepared solid oxide electrolysis cell is connected to a current collector meshes using respective electrode inks. The sealing is achieved by using glass paste/ring followed by appropriate heat treatment. On cathode steam, hydrogen and inert gas mix was provided using *Bronkhorst® controlled evaporator and mixer*. The electrochemical performance of the cell is monitored using Zahner IM6e electrochemical work station or Solatron 1260 Frequency Response Analyzer interfaced with Solatron 1287 A pontentiostat/galvanostat controlled by software.

c. Definitions –solid oxide electrolysis cell (SOEC); hydrogen (H_2); platinum (Pt); open circuit voltage (OCV).

d. Health & Safety Warning –Only trained person should be operating the test rig and cell fabrication equipment. Appropriate safety measures for remediation of hazard and risks associated with powders and solvents, electrical equipment, hot surfaces of the furnace, and flammable (Hydrogen and Carbon Monoxide (CO)) /asphyxiant gases. Refer to safe working instructions, personal protective equipment guidelines, HSE practices and compliance requirements in your lab.

Special safety precaution must be observed as CO is toxic gas (hazardous chemical) with Inhalation of concentrations of 4000 ppm in air could prove fatal in less than one hour. Safety Data Sheet must be referred prior to use.

All sensors should be checked for current calibration. Gas safety interlocks must be fully operational. Confirm LED indicators on gas safety panel before use.

e. Cautions – Special gas safety precautions must be observed as CO is a toxic gas (hazardous chemical) when electrolysis of CO_2 is to be performed with CO_2/CO gas mix. Other precautions: Hot surfaces, flammable/asphyxiant gases, hazardous chemicals

f. Interferences – Any electromagnetic interference associated with impedance measurement at lower frequencies due mains power supply, induction due to lead wires and furnace windings.

g. Personnel Qualifications / Responsibilities – The experiment requires basic skills in ceramic processing, dry-pressing, high-temperature sintering, and use of impedance spectroscopy equipment along with gas safety training.

h. Equipment and Supplies – Pre-Fabricated Solid Oxide Electrolysis Cells, Au paste (prepared with 3 microns gold powder dispersed in organic solvent), Au wires, *Zahner Im6E* electrochemical test station or *Solatron 1260* Frequency Response Analyzer interfaced with *Solatron 1287A* pontentiostat/galvanostat controlled by software, Test Rig with temperature controlled furnace and enclosure with gas extraction.

i. Step by Step Procedure

1. Before start, check the test rig for mechanical integrity of the alumina tube, test fixture and thermocouples positioning.

2. For benchmarking tests and validation of test protocol, a pre-fabricated YSZ electrolyte supported cell with screen printed Ni-YSZ cathode and LSCF anode with ceria barrier layer should be used. Alternatively, Ni-YSZ electrode supported cell with ~10 µm 8YSZ electrolyte can be used with LSCF anode with ceria barrier layer. Typical electrode and electrolyte shape is circular. Cell can be made *in-house* or purchased from commercial manufacturer (e.g., FCM Materials, USA or Fiaxell SOFC Technologies, Switzerland)

3. Cell sealing: Either of glass seal, ceramic or gold seals can be used. Depending upon type of sealing used (glass, gold or ceramic) and the method of seal formation (*in-situ or pre-formed*), the appropriate seal application procedures and heating/cooling cycles to be followed. (*See **Appendix** for the typical cycles for each seal type*).

4. For current collection, Pt or Ni (0.5 to 1 mm thick) wires connected (welded) to Ni mesh for SOEC cathode (hydrogen electrode) and Au mesh for SOEC anode (oxygen electrode) should be used. The area of current collector mesh should be same as active electrode area. Use of Ag mesh should be avoided for long term testing and for the testing of oxygen electrode performance as Ag may provide additional catalytic activity.

5. A current collection mesh specifications such as aperture, wire length and open area should be reported along with the result. *For example, Platinum Mesh, Nominal Aperture: 0.4mm, Wire diameter: 0.1mm, Wires/inch: 52, Open area: 63%, Purity: 99.9%, Type: Plain weave mesh.* For long term testing (> 24 hrs) use of Pt or Ni mesh/foam is recommended.

6. Some fixtures designs allows current collection directly from the fixture itself where Ni or alloys are used as fixture components. In that case, a manufacturer recommended procedure needs to be followed for both sealing and current collection. The examples of commercial fixture include *Fiaxell SOFC Technologies, Switzerland*.

7. Once seal is formed (*ex situ or in situ*), the cell should be heated to operating temperature (3°C/min). During heating, the either air or inert gas such Argon should be used as a purge gas on both air and fuel electrode side. Inert gas is necessary when Ni based materials are used for fixture or Ni meshes current collector.

8. For YSZ electrolyte-supported cells, an operating temperature of 800°C should be used. For electrode-supported cells, including metal supported cells, the operating temperature of 650°C can be used as a starting point.

9. At the operating temperature, the anode chamber gas should be swapped to air, and the cathode chamber gas to be swapped to fuel gas. For SOEC, it is suggested that sealing to be first evaluated using 2-3% humidified hydrogen. The target OCV with air and humidified hydrogen should be above 1.1 V for a button cell after 1 hr.

10. For electrode-supported cells, if a full reduction of the Ni-YSZ support is to be achieved before electrolysis, the cathode should be treated with hydrogen for 1 hr while monitoring cell ohmic resistance at a single frequency (~ 500 Hz or 1 kHz). Otherwise, further reduction of cathode may be achieved *in operando* during cell loading.

11. Once target conditions are reached, cell should be left in a steady state for 1 hr before starting the measurement.

12. For cell testing, the mixture of steam and inert gas along with hydrogen should be introduced through pre-heated inlet with appropriately positioned *dew point sensor*. In absence of the sensors, the thermodynamical data may be used to determine steam concentration, where the temperature of humidifier, pressure, and gas volume and flow rates can be used to estimate steam content.

13. During the test, the temperature of inlet gas and outlet gas should to be monitored using in-line thermocouples.

14. Procedure for cell testing and electrochemical chacareisation is outlined in *Data Acquisition, Calculations & Data Reduction Requirements* section.

- Troubleshooting

In the event of unusual results, check the gas flows and system temperature, and reset the electrochemical data acquisition system.

Monitor the OCV to make sure cell/seal is not cracked. If leak through sealing is suspected the in-situ heat treatment cycle may be repeated.

If with OCV does not drop below 1 V after steam is introduced (with target steam content above 50%), the actual steam concentration at electrode can be lower than the target. In such even due point sensor/steamer needs post-test calibration.

- Data Acquisition, Calculations & Data Reduction Requirements

Data acquisition is using electrochemical work station. Most of the modern potentiostat have OEM provided software for data monitoring, control and collection.

1. For I-V curves, scan rate 5 mV/S is recommended for button cell with voltage varied from OCV to 1.4 V. I-V curves should be taken after cell conditioning. For button cells, conditioning involves a loading of cell at 1.25 V for 1 hr at least. After conditioning, cell should be left at OCV conditions for 15 minutes before running I-V curve.

2. EIS should be taken at OCV and as well as under loading condition. For EIS at loading condition, cell should be loaded at constant voltage for 30 minutes before recording EIS at that voltage. (*EIS at constant current may lead to excessive voltage being imposed on cell*). Recommended amplitude is 10 to 50 mV.

3. The exhaust gas analysis should be performed to using *steam trap* to obtain dry hydrogen for Gas Chromatography analysis. The Faradaic efficiency of a typical cell (with YSZ electrolytes) is expected to be $\geq$ 90%.

4. After electrochemical characterization, cell may loaded at below thermos-neutral voltage (< 1.28 V at 800°C), e.g., 1.1-1.2 V. (SOE operation in sub-Thermo-neutral mode is sought where waste heat is available). Calculate the degradation based on the initial and final current at particular voltage.

5. If long term testing is intended, minimum of 100 hrs of operation should be targeted in either constant voltage or constant current mode chosen based upon initial performance.

6. Reportable from the test:

- details of test fixture (ceramic/metallic, use code for type used)
- Full details of the fabricated SOEC such as electrode/electrolyte thickness and nominal electrode porosity.
- pressure
- temperature (furnace, specimen and gases at inlet and outlet)
- flow rates
- gas concentration
- Differential pressure (if any)
- Electrochemical characterization data
- Steam utilization for cell area > $3cm^2$

- Computer Hardware & Software – Z View and Zplot (Scribner Associates Inc.) installed on compatible Windows OS.

j. Data and Records Management

All data should be backed up as text file as well as in format readable by software. Process conditions along with timing should be noted in Lab book. The readouts from gas flow meters/ Steamer and furnace temperature controller can be integrated using LabVIEW software/or excel Macro.

4. Quality Control and Quality Assurance Section

- Calibrate electrochemical work station using manufacturer provided test circuits),

- Thermocouple readouts, and flow meters should also be periodically tested-using manufacturer recommended procedures.

- Repeated experiments are required to confirm the quality of the data.

5. Reference Section

Not applicable for this SOP.

Appendix

Sealing procedure for glass and gold seals are as below:

I. *For glass sealing*: Glass sealing may be formed in-situ or pre-formed externally:

(a) For *in-situ* glass seal, a glass ring or a paste of *Schott G018-311* glass may be used. Such seals are commercially available from *Schott.* A typical heating cycle is: 100°C/0.5 h (5°C/min), 180°C/0.2 h (3°C/min), 720°C/1h (10°C/min) and reach operating temperature of the cells at 3°C/min. During sealing either air or argon atmosphere may be use for purging the fixture. If use paste is desired a glass powder may be mixed with organic binder (*Ferro Corporation/Schott)* should be mixed

(b) For pre-formed glass seal: The glass seal using glass powder slurry prepared with commercial materials such as *Schott GM31107* glass powder mixed with organic binder in 4:1 wt% followed by a thermal cycling to obtain gas tight seal (*refer to instruction provided by Schott for pre-formed seal*).

II. Alternatively, ceramic sealant such as *Ceramabond® (Aremco)* can be used, and procedure provided by manufacturer may be followed for pre-formed ceramic seal.

III. Gold paste or gold ring can be used for sealing which involves heating cell close to melting temp of gold (1050°C) using 2°C/min ramp rate, and holding at 1050°C for 20 minutes, and cooling to operating temperature (2°C/min). The ramp rates and hold time are indicative and may change with fixture design and flow rates of gases.