Standard Operating Procedure

(SOP)

Leak test for SOECs

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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
04/15/2019	All	First Release

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3. Procedures

Note the following procedures are primarily for planar type SOFC and SOEC cells and stacks where the cell is sealed onto a coated metallic window frame. The sealed cell/window frame assembly was then repeatedly stacked up to form a multi-cell stacks. For tubular type SOFC and SOEC, the current procedures may not be applicable.

a. Scope and Applicability – the purpose of this procedure to identify if any leaks are present in the system.

b. Summary of Method

c. Definitions
SOEC – solid oxide electrolysis cell
SOFC – solid oxide fuel cell
LSCF – lanthanum strontium cobaltite ferrite
LSM – lanthanum strontium manganate
OCV – open circuit voltage
YSZ – yttria-stabilized zirconia

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

e. Cautions
Hot surfaces, flammable gases, hazardous chemicals

f. Interferences

g. Personnel Qualifications / Responsibilities

h. Equipment and Supplies

i. Step by Step Procedure

<u>Leak Test of Cell Sealed onto Coated Metallic Window Frame Plate</u>

A typical example of a 2”x2” LSM-based YSZ electrolyte cell glass sealed onto an aluminized SS441 window frame plate is shown in Figure 1. The leak test of this geometry is pretty straight forward by using iso-propanol, instead of designing a test fixture and pressurize with inert gas and monitor the pressure decay over a period of time. One could design a test fixture for leakage determination; however, the window frame often showed some warpage after sealing at elevated temperature. Hence the attachment

of a test fixture frame onto the warped cell/window frame could potentially cracking the glass seal and causing the leak. For simple leak test with iso-propanol, one would:

1. Place two flat spacers of same thickness on a flat surface (Figure 1, A)
2. Place the sealed cell/window frame plate on top of the spacers with cathode side cavity facing up (Figure 1, B)
3. Add some iso-propanol liquid into the cathode cavity to cover the whole cathode surface, but not overflowing (Figure 1, C)
4. Wait for about 3-5 minutes for iso-propanol to penetrate through defected glass seal (you may need to refill some if the ambient temperature is too high that the alcohol evaporates fast
5. Carefully take up the window frame plate and drain the iso-propanol from one of the four corners
6. Flip the other side and look for wet spots. If there is no wet spot, the seal is hermetic (Figure 2, A) or liquid spot showing a leaked seal (Figure 2, B). Note the penetrated liquid could also come from a cracked cell where the glass seal was good; however, such a cell still would not be suitable for testing.

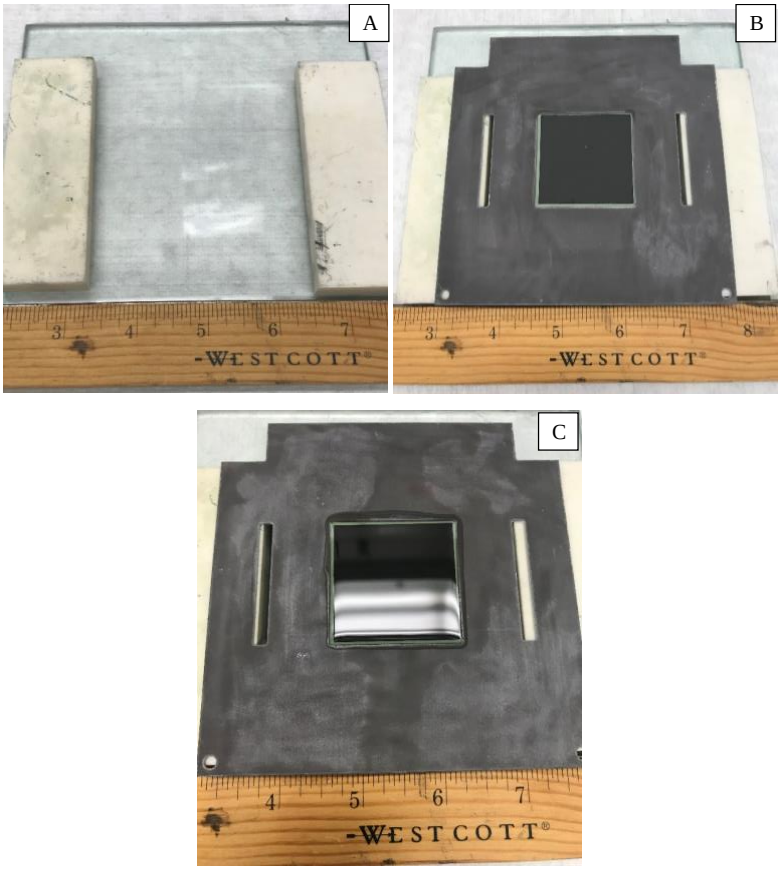


Figure 1. Leak test of sealed cell to window frame plate: A) a flat glass plate with 2 same thickness spacers; B) a sealed cell/window frame plate with cathode cavity facing up; C) iso-propanol filled in the central cavity.

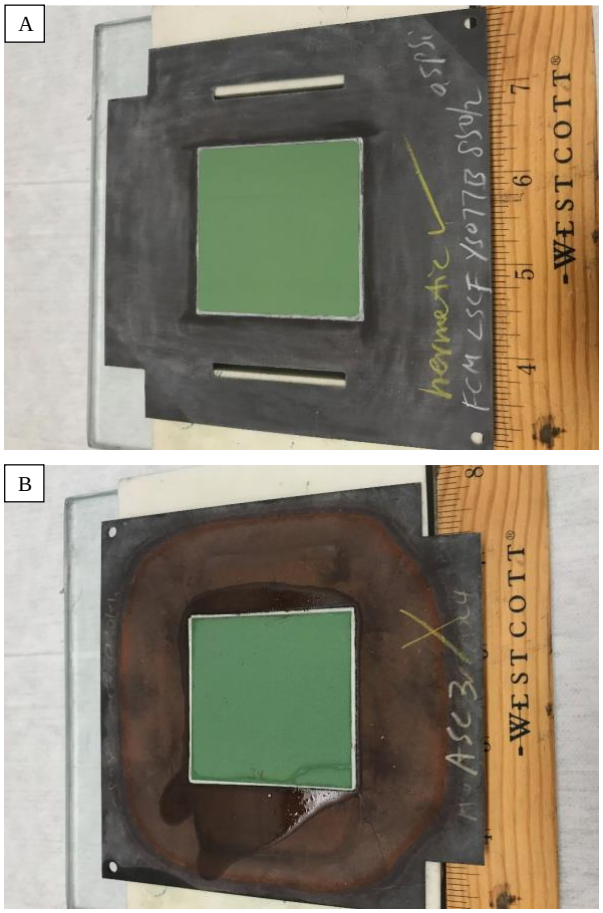


Figure 2. Leak test results of sealed cell to window frame plate observation at anode side after ~5 minutes of iso-propanol penetration at cathode side: A) hermetic seal where no iso-propanol was found; B) a leaked seal where iso-propanol was found at anode side.

Leak Test of Assembled Cell in Stack Test Fixture

The following leak test procedures are for assembled cell/window frame plate with interconnect plates and gas manifold or end compressive loading plates. In other words, it is designed for a "stack" type assembly; however, the concept and procedures may be adopted for typical small-sized "button" cells where metallic interconnect plates and end compressive loading plates may not be needed. At Pacific Northwest National Laboratory (PNNL) we have developed a simple generic stack test fixture for routine testing/validation of novel materials for cathode contact, Cr-mitigation, degradation of standard LSM or LSCF based YSZ thin electrolyte cells (2"x2"), and environmental moisture effects, etc. A typical example of all parts used for a single cell testing in the generic stack test fixture is shown in Figure 3 where the sealed cell/window frame plate (Figure 3, A) is sandwiched between the cathode and anode interconnect plates (Figure 3,

B) with Ag/mica compressive seals (Figure 3, C), and compressed between the end plates of Inconel for fuel and porous alumina for air (Figure 3, D). The outgoing gases were immersed in a water bubbler with water of a few inches head. The leak test procedures are described below which contains the first part of cross-bubbling test and the second part of OCV.

Cross-Bubbling Leak Test

A typical full view of the generic stack test is shown in Figure 4, A where the cell was under compressive loading and the exhaust of fuel and air are immersed in the water bubblers (Figure 4, B) with water head of 0.5-1 inch (Figure 4, C). After the final stack firing to form bonding/contact of contact materials the cell was cooled to operation temperature (e.g., 800^{o}C), and the simple leak test was conducted before reducing the anode. The procedures can be described as below:

1. Apply air to the cell at a reasonable flow rate while no gas flowing in the fuel side. A typical air flow rate for a 2"x2" LSM-based anode-supported YSZ thin electrolyte cell with an active cathode of 16 cm^2 could be 800-900 sccm (standard cubic centimeter per minute). To minimizing potential thermal shock for large size cell, the air flow rate should increase in a gradual manner from zero to maximum. For example in 30 minutes for maximum of 800-900 sccm.
2. Make sure the water head of the exhaust air bubbler and exhaust fuel bubbler is about 0.5-1 inch, this would present least resistance for leak bubbling if cell seal failed or cell fractured during assembling or final stack firing.
3. Visual examination of the exhaust fuel bubbler. If see bubbling, it suggests leaks although it will not tell whether the leak was caused by (1), cell to window frame glass seal failure, (2), cell fracture (often caused by warped window frame plate under stack's compressive loading), or (3) perimeter mica seal failure. Note it is not common to have the cross-bubbling if the leak was only due to mica failure.
4. Shut down air flow, wait for 5-10 minutes till no bubbling in exhaust air bubbler.
5. Turn on gas flow (air or nitrogen for un-reduced anode) in fuel side in a similar manner as 1.
6. Watch for cross-bubbling, if exhaust air bubbler shows bubbling then there is leak
7. It is expected no leak if no cross-bubbling occurred.
8. The cell may still be operable if the cross-bubbling is very minute, like a few bubbles per a few second when the outer diameter of metal tubing inside the bubbler is 1/8".

OCV Leak Test

In addition to the previous mentioned cross-bubbling method to detect cell leakage, one could also using open circuit voltage (OCV) as an alternative for leakage test. However, the cell needs to be fully reduced and more time consuming. Once the cell being fully reduced, one can flow the desired fuel (e.g., H_2:N_2=1:1) and air at reasonable flow rates. For example a 2"x2" cell with active cathode area of 4 cm x 4 cm, one can flow 450 sccm of fuel (H_2:N_2=1:1) and 900 sccm air. Depending on your system and furnace, it may

take some time to reach this final flow rate since one would need to reduce the potential thermal shock by changing flow rate drastically. Once the flow rate being stabilized for 30 minutes, and cell temperature remains fairly constant, one can then use OCV to determine if the cell leaks or not by comparing the measured OCV with the calculated Nernst voltage at specified temperature and oxygen partial pressures in fuel and air (dry or moist). If the measured OCV is >=20 mv lower than the Nernst voltage, it is expected some leakage occurred. This information can also be used to confirm with previously mentioned cross-bubbling technique before cell being fully reduced. It needs to be cautioned that if one would also like to run cross-bubbling after cell being fully reduced, care must be taken to avoid potential explosion or cell damage when air may be introduced to fully reduced anode.

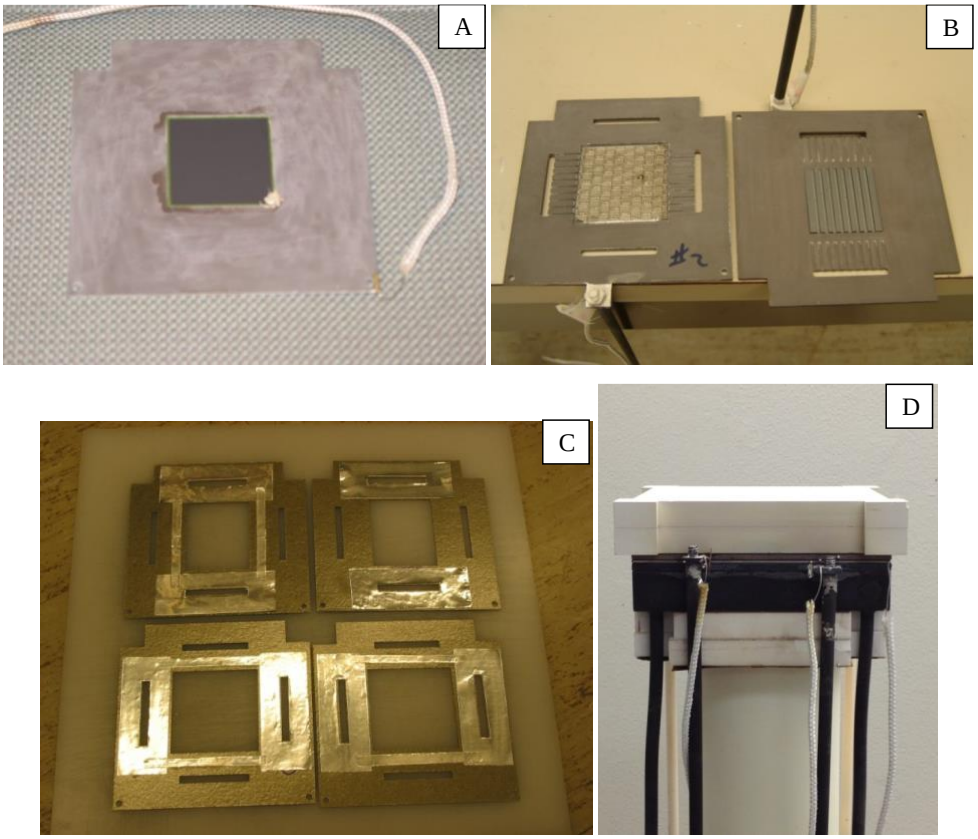


Figure 3. Typical SOFC single cell assembly for testing where the ceramic cell was pressed between the porous alumina top block (for air in and out) and Inconel600 bottom block (for fuel in and out). The photo also shows two current rods and two Pt wire leads for voltage sensing. The assembly is sitting on an alumina pipe with insulation inside, and will be placed inside the split cylindrical furnace for high temperature testing. A, cell/window frame plate, B, interconnect plates for anode and cathode side, C, compressive Ag/mica for perimeter seal, D, assembled view.

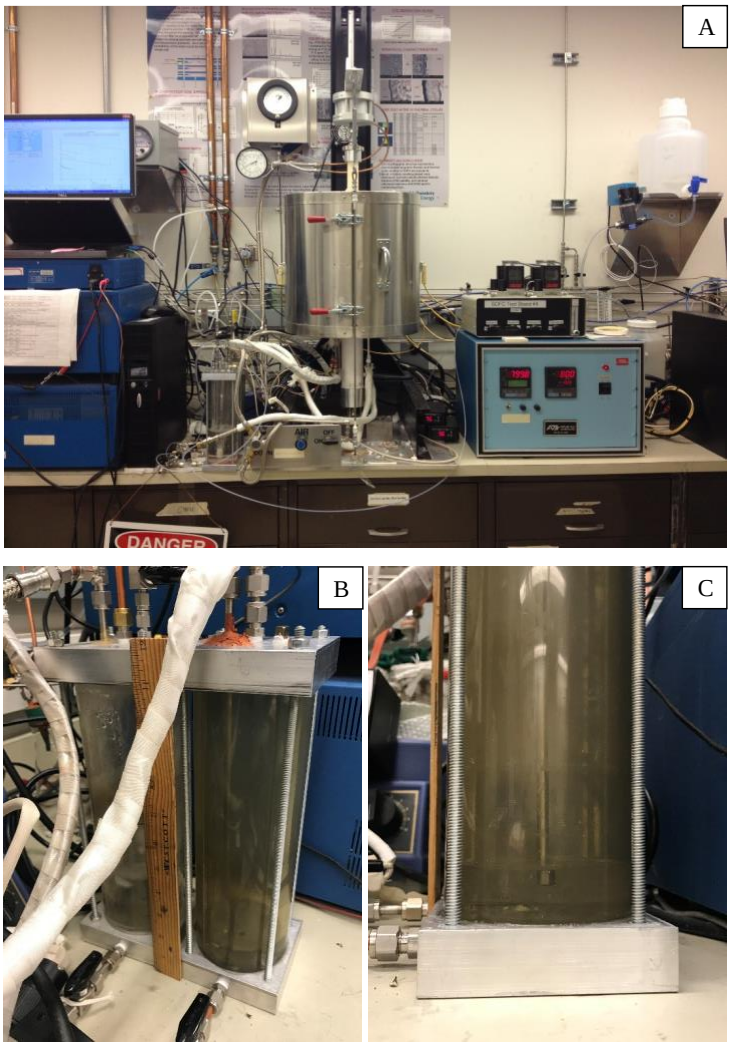


Figure 4. Actual set up for a generic stack test, A. full view of the experimental set-up, B, view of the two exhaust water bubblers (one for fuel and one for air), and C, enlarged side view of the bubbler with water at ~0.5-1 inch head.

4. Quality Control and Quality Assurance Section

5. Reference Section