Standard Operating Procedure

(SOP)

Contact Layer Bonding Strength and Thermal Cycle Stability

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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

The following procedure is to evaluate bonding strength of the contact layers by an uniaxial tensile test. This procedure is primarily for planar type SOFC and SOEC cells and stacks where each cell is sealed onto a coated metallic window frame. The sealed cell/window frame assembly is then repeatedly stacked up to form a multi-cell stack. For tubular type SOFC and SOEC, the current procedure may not be applicable.

For large size cells and stacks, mechanical properties play an essential role. The oxygen electrode contact layer is the most critical as compared to hydrogen electrode contact layer. For hydrogen electrode contact layer, the contact bonding is expected to be metallurgical and, hence, strong; typically, a NiO contact layer is used on the NiO/YSZ electrode or the support. For oxygen electrode, an oxide-based (perovskite) contact layer is used. When using brittle ceramics, like LSM, LSC, etc. (i.e., the compound similar to the oxygen electrode), the contact strength is weak, less than 1-2 MPa at room temperature. This is because of very poor necking between the particles after final stack firing temperature well below 950^{o}C and due to significant porosity in the contact paste because of the presence of large amount of volatile organic (binders).

a. Scope and Applicability – the purpose of this procedure to perform mechanical property test for contact bonding strength and thermal cycle stability

b. Summary of Method

c. Definitions
SOEC – solid oxide electrolysis cell
SOFC – solid oxide fuel cell
LSC – lanthanum strontium cobaltite
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

1. Laser cut the bilayers to desired dimensions of 1 inch x1 inch (Figure 1, A).
2. Prepare contact paste and transferred into a syringe.
3. Laser cut mica spacer (e.g., 20 mils thick mica of 1 inch x1 inch size and a central hole with diameter of 0.5 inch) (Figure 1, B).
4. Place mica spacer on top of bilayer (Figure 1, C).
5. Apply contact paste into the central cavity and use a razer blade to press the paste and fill the cavity (Figure 1, D).
6. Dry in oven for ~1 hour.
7. Sinter the dried sample to final stacking firing temperature and time.
8. After sintering and cooling, glue the sample (with mica still on the sample) using fast curing epoxy on aluminum test blocks (Figure 2, A).
9. After epoxy curing for 15 to 30 minutes at room temperature (depending on the fast curing info of the epoxy), test the uniaxial tensile strength with a typically cross-head speed of 0.5 mm/min and a self-aligning fixtures to minimize type II or type III fracture (Figure 2, B).
10. Use optical microscopy to examine the fracture surface to confirm the fracture is along the contact and cathode interface.
11. Calculate the apparent contact strength with maximum load divided by the apparent contact area.
12. Due to the brittle nature of ceramics, it is advised to test 5 samples of each formulation and at each condition and calculate the average value.

Thermal cycle stability test will depend on the particular application and a temperature profile chosen.

1. Place sintered samples in the furnace.
2. Pre-set temperature cycle profile: specify heating/cooling rate, soak time, and a number of thermal cycles. 10 is a good number of thermal cycles.
3. After completing thermal cycling, prepare test samples as listed above, Steps 8-11, and measure the apparent strength (step 11).

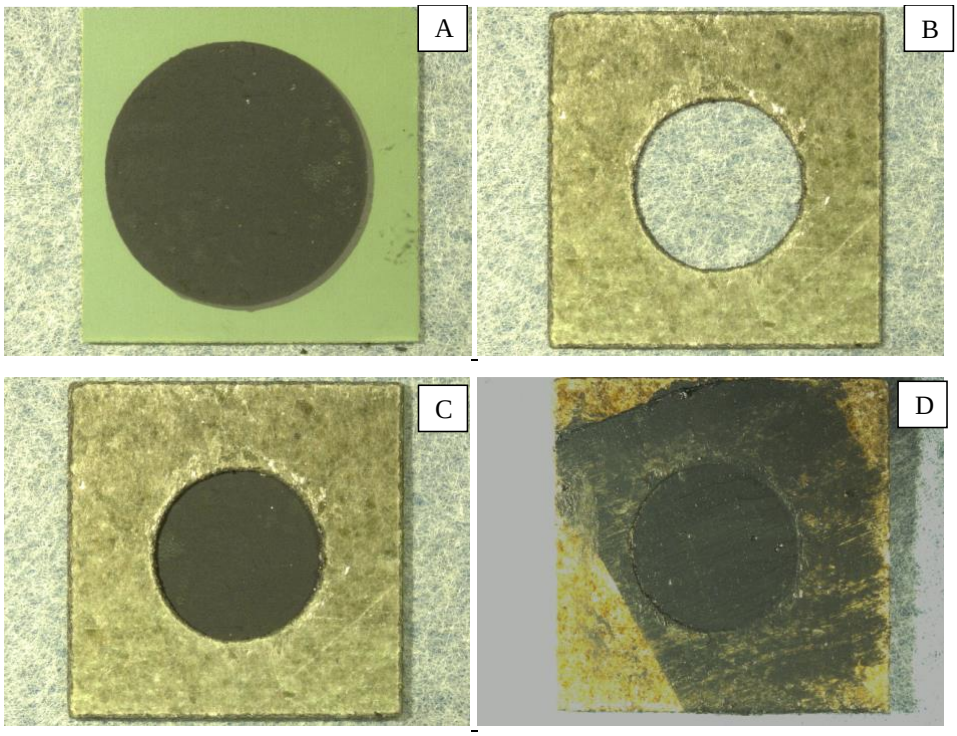


Figure 1. Preparation of bilayer joining for contact strength: (A) plain bilayer with LSM cathode; (B) a mica mask with a central hole; (C) mica mask placed on top of bilayer; (D) as-sintered bilayer with contact paste. Note the contact strength was then tested with Al fixtures bonded on the top and bottom surface with epoxy and tested in uniaxial tension at room temperature.

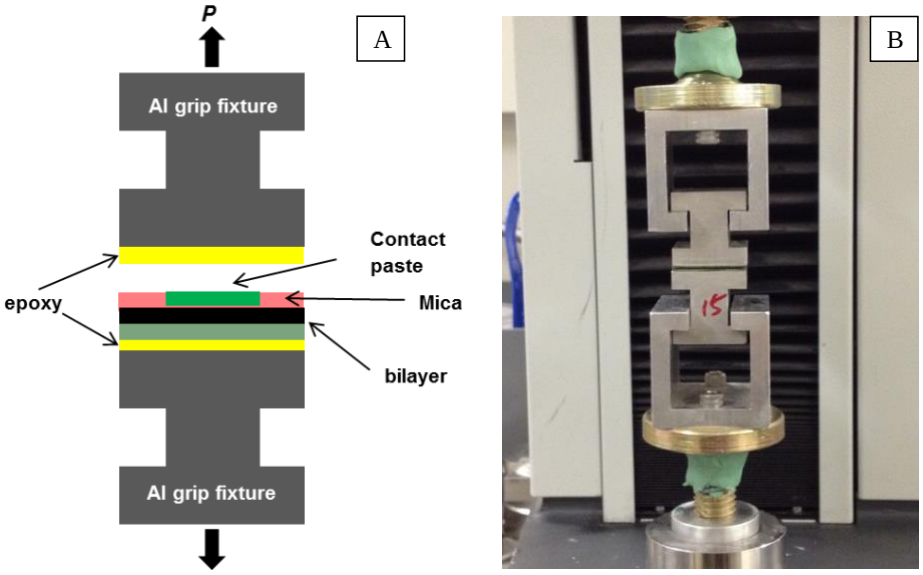


Figure 2. A) Schematic drawing shows the preparation of strength test of contact bonding; B) actual assembly for uniaxial tensile testing.

4. Quality Control and Quality Assurance Section

5. Reference Section