Standard Operating Procedure (SOP)

Measurement of Area Specific Resistance (ASR) of the interconnect plates for solid oxide cells

Test ID: HTE-P-18

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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

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3. **Procedures**

a. Scope and Applicability – The purpose of this SOP is to describe a method for the measurement of ASR of the interconnect plates used for stacking solid oxide electrolysis or solid oxide fuel cells. The procedure applies to planar interconnect plates prior to the machining of flow channels.

b. Summary of Method – A method is based upon a simple four-point conductivity measurement procedure using two current probes and two voltage probes welded to interconnect alloy. The measurements are done in dry air using spring loaded fixture placed in alumina or quartz tube of a tube furnace. Examples of the interconnect materials can be Crofer®22APU, Hastelloy, Haynes230 and related high temperature alloys with or without protective spinel coating. Typical spinel coating could be Mn-Co spinels, used to avoid migration/evaporation of Cr from the alloys. Schematic of the proposed test setup is shown below in Figure 1.

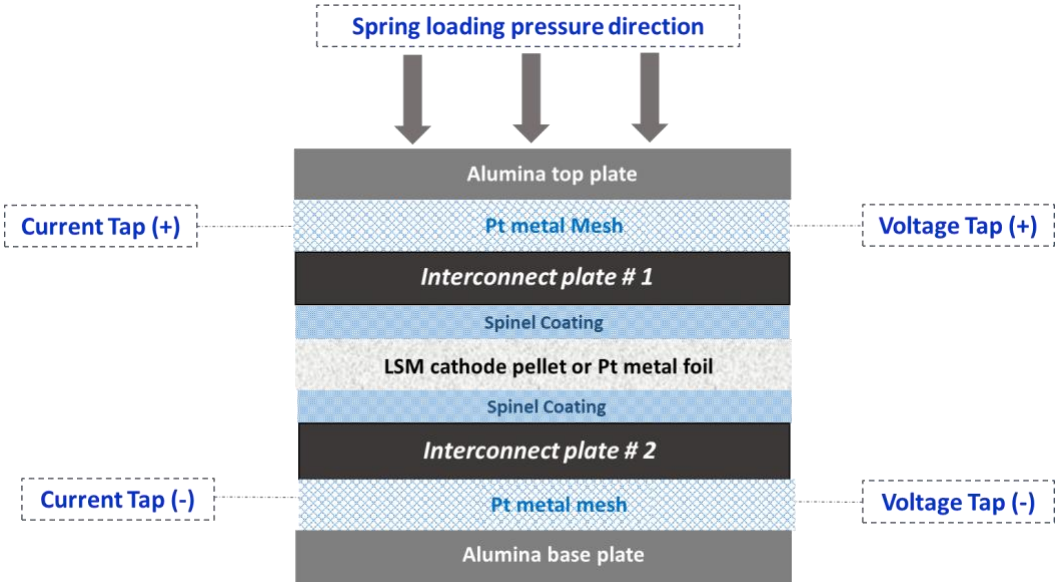


Figure 1: Schematic of proposed specimen assembly for Measurement of Area Specific Resistance (ASR) of the interconnect

c. Definitions –Area Specific resistance (ASR); platinum (Pt); Digital Multimeter (DMM)

d. Health & Safety Warning – Only trained personnel should operate the test rig and cell fabrication equipment. Appropriate safety measures for remediation of the hazards and risks associated with 1. Ceramic powders and solvents, 2. Electrical equipment, 3. And Hot surfaces of the furnace. Refer to safe working instructions (SWI), personal protective equipment guidelines, HSE practices and environmental compliance requirements in the lab.

e. Cautions – Hot surfaces, Irritant chemicals (refer to SDS for ceramic powders), flammable gases (for heat treatment of spinel coating)

f. Interferences –Seebeck effect

g. Personnel Qualifications / Responsibilities – The experiment requires basic skills in ceramic processing, high-temperature furnace operation, and use of electrical devices (e.g., DC power supply and multi-meter or galvanostat/potentitostat)

h. Equipment and Supplies –

1. Two square interconnect plates (1.5 cm (L) 1.5 cm (W) ~ 1mm thick). Other dimensions may be used as per test fixture design.
2. Pt Paste (Engelhard 6082 from Heraeus Precious Ltd. or equivalent)
3. Pt wires (1 mm)
4. Test fixture with furnace and furnace controller. The test fixtures can be purchased commercially or built in house. Examples of commercial fixtures include, *Probostat* from Norecs, Fiaxell or Green Lights Innovation. The general guidelines for typical button/coupon test fixture design for SOFC can be found in Reference [1].
5. Pt foil with same dimensions (L and W) as the interconnects under test or a ceramic cathode material pellet ((1.5 cm (L) 1.5 cm (W) ~ 0.5 to 1mm thick)
6. Precision current source (e.g. Keithley 6000) and DMM (e.g. Fluke 8588A Ref) or electrochemical test unit (e.g Gamry 6000, PAR Versatstat, Solatron 1286, Zahner IM6, Autolab from Biologic, Ivium)

i. Step by Step Procedure

1. Cut two square plates of the interconnect materials cut to the dimensions and clean ultrasonically and degrease.
2. Plates should be acid-treated with 1N Nitric Acid (HNO_3) or 1N Phosphoric Acid (H_3PO_4) for 1 minute followed by washing with DI water to remove any scale if any.
3. If spinel coating is desired, spinel coating procedure should be followed. If the interconnects are to be tested without coating to understand scaling behavior, go to step 6.
4. The spinel coating ink can be prepared by mixing spinel oxide (e.g $MnCo_2O_4$) with organic binder (e.g. ESL-V400-A) with 60% solid loading. A ball milling for 12 hrs is recommended. The spinel ink is then screen printed or brush painted on one of the surfaces of both the interconnects. (*The coating ink preparation procedure may be varied depending upon spinel material and desired coating characteristics*). Nominal coating thickness desired is about 10-15µ.

5. After screen printing, the spinel coating should be heat treated in a forming gas for 1h followed by heat treatment in air at 1000°C for 3 h to form a uniform spinel layer.
6. Once both interconnect specimens are coated (top surface only), both the specimens should be spring loaded in the test fixture with a nominal load of 500 g/cm^2. See the **Figure 1** for schematic of suggested assembly.
7. Insert the cathode slab or Pt foil between the interconnect surfaces (with or without spinel) as shown in the **Figure 1**. A typical cathode slab can be prepared by dry pressing the cathode material (e.g LSM) and sintering it to the required densification (~40-50% porosity).
8. A mechanical integrity of the alumina tube, test fixture and thermocouple positioning should be checked before starting experiment. Thermocouples must be placed as close as possible to the specimen but without touching the specimens.
9. Set the furnace temperature to the desired operating temperature. Once the setup has been heated to the target temperature (3°C/min ramp rate), it should be allowed to equilibrate for about 60 minutes in flowing air (50 ml/min). Excessive airflow should be avoided to control surface temperature.
10. At set temperature, ideally OCV across the voltage taps should be zero. Any deviation from the zero voltage should be noted. This voltage is due to the stray effects in the system including *Seeback effect* and should be subtracted from any subsequent voltage measurement. It is termed as *compensation voltage*.
11. Once *compensation voltage* is obtained, I-V characteristics should be obtained either using linear voltage or current sweep. The current should be sourced from Keithley current source and voltage should be measured using Fluke DMM. Refer to manufacturers manuals for the operation instruction.
12. Alternatively, if electrochemical work stations (electrochemical test unit e.g *Gamry 6000, PAR Versatstat, Solatron 1286, Zahner IM6, Autolab from Biologic, Ivium*) are used, a suitable *linear volatge sweep* program can be selected from controlling software. Refer to manufacturers manuals.
13. A scan rate of 5 mV/S is suggested for I-V curves. The upper and lower voltages should correspond to the target SOFC/SOEC operating voltage windows.
14. ASR can be then obtained from I-V curve using the linear portion of I-V curve.
15. The other data can be logged using a LabVIEW interface connected to air flow meters and thermocouple.
16. The data files may be supplied as a supporting information for manuscript or report.
17. For the measurement of ASR of interconnects, minimum test period of 200 hrs is recommended (1000 hrs preferred).

18. The measurements at 3 to 4 different temperatures may be carried out to report an operating window for a particular interconnect materials.
19. Further reading materials related to interconnect materials and key performance indicators can be found in references [2-4].

- **Troubleshooting**

  In the event of unusual results, check the air flows and system temperature, and reset the electrochemical data acquisition system and check the instruments using standard test circuit.

- **Data Acquisition, Calculations & Data Reduction Requirements**

  Data is acquired using electrochemical work station or Digital Multimeter and Labview. Most modern equipment has OEM provided software for data monitoring, control, and collection.
  Reportable from the test:
  - Details of test fixture (ceramic/metallic, use code for type used).
  - Full details of the fabricated cathode, Spinel coating including materials source and processing conditions
  - ASR at various temperatures along with time

- **Computer Hardware & Software** – OEM provided software.

j. **Data and Records Management**

All data should be backed up as a text file as well as in format readable by software. Process conditions and time should also be noted in a lab book.

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.

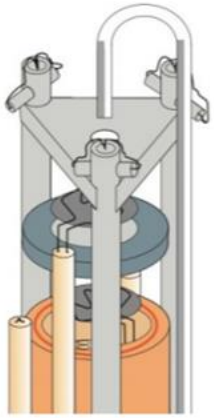
5. Reference Section

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2. Interconnect Materials for SOFC Stacks, Xingbo Liu, Junwei Wu and Christopher Johnson in Solid Oxide Fuel Cells: From Materials to System Modeling, RSC publications, https://doi.org/10.1039/9781849737777

3. Metallic interconnects for SOFC: Characterisation of corrosion resistance and conductivity evaluation at operating temperature of differently coated alloys, Journal of Power Sources, Volume 171, Issue 2, 27 September 2007, Pages 652-662
4. S Elangovan et al. , Metal Interconnects for Solid Oxide Fuel Cell Power Systems SECA Core Technology Program, Phase I Topical Report, https://www.osti.gov/servlets/purl/833893

Appendix

(All images and information is property of respective company or organisation and reproduced here with a reference to the manufacturer's website accessed in March 2020)



Schematic of commercial Test Fixture from NORSEC
(https://www.norecs.com/index.php?page=ProboStat)

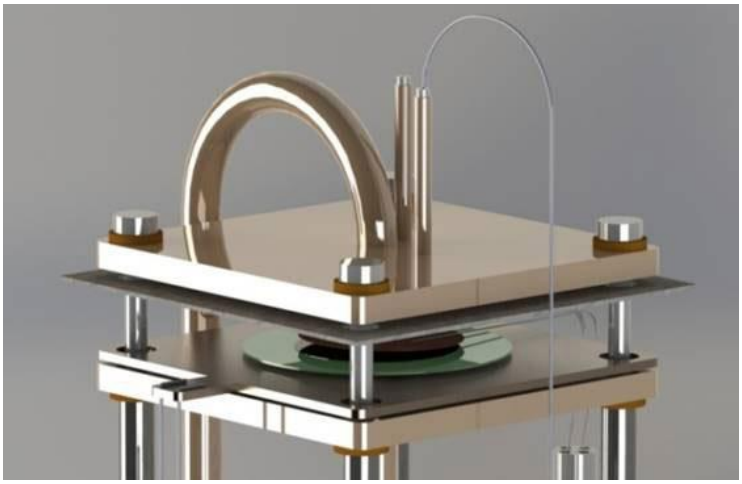


Photo of commercial Test Fixture from Fiaxell
(https://fiaxell.com/)