Standard Operating Procedure

(SOP)

Polarization Resistance Measurements for SOEC

Test ID: HTE-P-10

Rev 1

Dong Ding / Senior Research Engineer	3/27/2019
Ani Kulkarni / Senior Scientist	4/11/2019
Prepared By: NAME / TITLE	Date
Approved By: NAME / TITLE	Date
Approved By: NAME / TITLE	Date

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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 best practices for evaluation of the electrode polarization resistance for symmetrical solid oxide cells using electrochemical impedance spectroscopy technique. It applies to common oxide materials which are used as oxygen evolution electrode (anode) in SOEC.

b. Summary of Method – The method encompasses recommended fabrication techniques for electrolyte supported symmetrical solid oxide (*Three electrode configuration*) cells and impedance spectroscopy of the cells to obtain *Nyquist plot*. The *Nyquist plot* can be fitted with equivalent circuits to obtain the resistance corresponding to the different electrochemical processes. The dependence of electrode resistance on different conditions (temperature, oxygen partial pressure, and humidity) can be studied to explore the optimal operating condition.

c. Definitions

SOEC – Solid oxide electrolysis cell

d. Health & Safety Warning – Appropriate safety measures for remediation of hazard and risks associated with powders and solvents, electrical equipment, hot surfaces of the furnace, and flammable/asphyxiant gases. (*Refer to safe working instructions, personal protective equipment guidelines, HSE practices and compliance requirements in your lab*)
Hydrogen is a flammable gas.

e. Cautions: Hot surfaces, flammable/asphyxiant gases, hazardous chemicals

f. Interferences – For three-electrode configuration, the selection of reference electrode position is critical to correctly collect the electrochemical information. In this protocol, the reference electrode is positioned at the middle edge of the electrolyte pellets, and working and counter electrodes are at the both sides of electrolyte.

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.

h. Equipment and Supplies
Cylinder die, high-temperature sintering furnace, electrode/electrolyte powders, Pt wire, Pt paste, muffle furnace, electrochemical work station with software control, gases and water bubbler.

i. Step by Step Procedure

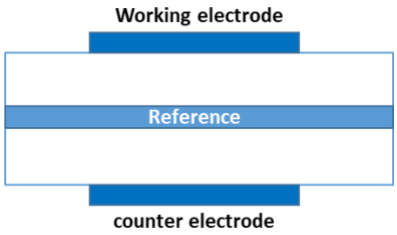
- Instrument or Method Calibration and Standardization – Solartron 1400 series electrochemical work station or equivalent calibrated using OEM supplied test circuit

- Sample Preparation and Analysis

1. The sample fabrication process involves die pressing of electrolyte powder into thick electrolyte green disc followed by sintering at electrolyte sintering temperature (For YSZ 8 it is 1500°C for 4 hrs), The density of electrolyte close to 95% of theoretical density is expected as determined by Archimedes method.

2. On the sintered electrolyte pellet, working and counter electrodes are fabricated using screen printing followed by heat treatment at suitable temperature which is specific to the test electrodes. (For example, For LSM electrode on YSZ electrolyte, heat treatment at 1050°C for 2 hrs is recommended). The electrode shape should be same and point to each other

3. The reference electrode can be a Pt wire (0.25 mm) attached using Pt paste (Engelhard 6082) or electrode materials paste along the circumference line (positioned at middle edge of the pellet at equal distance from the both faces) as shown in Schematic (*not to scale*) below



4. Place the prepared sample in the text fixture. Typically, spring loaded Au meshes connected to Au wired should be used to make electrical connections for working and counter electrode to working and counter lead wire of the potentiostat/Impedance analyzer (e.g. Solatron).

- Troubleshooting
  Avoid short circuit before heating up the furnace by examining the lead wires with multi-meter.

- Data Acquisition, Calculations & Data Reduction Requirements –
  Data acquisition using *Z View and Z plot* software.
  Typically frequency range: 1000000~0.01Hz,
  Excitement voltage (amplitude): 20~50 mV,
  Potentistatic EIS mode.
  Data visualization and Fitting the impedance *Z View*

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

j. Data and Records Management –Real (Z') and imaginary (Z'') impedance data set for replotting the impedance should be saved to hard drive. The data should be normalized to area using software settings or can be done in MS Excel spreadsheet after exporting data from Z view.

   If three electrode arrangement is used, the obtained impedance (Nyquist plot) will reflect polarization resistance associated with working electrode. Estimation of ohmic resistance of the cell from three electrode arrangement is erroneous. For determination of ohmic resistance, a two electrode method (by disconnecting reference electrode connection) should be used.

4. Quality Control and Quality Assurance Section

   Ideally the impedance spectra curves should be smooth and show the semi- or depressed semi- arcs. Repeated experiments are required to confirm the quality of the data points.

5. Reference Section

1. J. Nielsen, J. Hjelm, "Impedance of SOFC electrodes: A review and a comprehensive case study on the impedance of LSM: YSZ cathodes", Electrochimica Acta, 115 (2014) 31-45.

2. Q. Huang, R. Hui, B. Wang, J. Zhang, "A review of AC impedance modeling and validation in SOFC diagnosis", Electrochimica Acta, 52 (2007) 8144-8164.