Standard Operating Procedure

(SOP)

Measurement of Bulk Conductivity of SOEC Materials

Test ID: HTE-P-01

Rev 1

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Date: 04/11/2019

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
4/11/2019	All	First Release

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a. Scope and Applicability

When investigating new materials for solid oxide electrolysis cell (SOEC) components (e.g. electrodes), conductivity measurements are often an important part of the materials characterization profile. Conductivity can be measured as a function of temperature and under controlled oxygen partial pressures (pO_2) or gas compositions of interest.

b. Summary of Method

Dense bars of the material of interest are measured using 4 point conductivity measurements.

c. Definitions

pO_2	oxygen partial pressure
SOEC	solid oxide electrolysis cell

d. Health & Safety Warning

e. Cautions

f. Interferences

g. Personnel Qualifications / Responsibilities

h. Equipment and Supplies

- Fine platinum wire (0.25 mm)
- A >90% dense bar of the material of interest with the approximate dimensions 2 mm x 4 mm x 40 mm.
- Platinum ink
- Diamond saw
- Small paint brush
- Needle nose pliers
- Micrometer calipers
- Tube furnace capable of the temperature range of interest
- Gas source to provide the atmosphere of interest (if controlled atmosphere other than air is desired)
- Potentiostat

i. Step by Step Procedure

1) Instrument or Method Calibration and Standardization – follow instrument's SOP calibration procedure.

2) Sample Collection

3) Sample Handling and Preservation

4) Sample Preparation and Analysis

   a) Perform density measurement on the sample bar using the Archimedes' method. For electrolyte materials theoretical density ≥ 95% is expected.
   b) Use the diamond saw to cut thin notches around the bar approximately 7 mm and 12 mm from the lengthwise center of the bar toward each end. The notches should be shallow, only deep enough to keep a loop of wire tightened around the rod from sliding.
   c) Using a small paint brush, paint a small band of platinum ink (Engelhard 6082 or equivalent) in the notches made in the bar.
   d) Wrap platinum wire (0.25 mm thick) around the bands of platinum ink in the notches and twist the wire to tighten it down with a pair of pliers, taking care not to break the wire. Nominal thickness of bands is 0.5 mm. Silica free masking tapes may be used to control thickness and shape of the bands.
   e) Heat the assembly in a furnace to 900°C for 30 min in air to sinter the Pt ink connecting the wire to the bar.
   f) Check that the wires are still tight around the Pt bands and gently retighten if needed.
   g) Use micrometer calipers to measure the width and thickness of the sample bar and distance between the innermost platinum wire loops around it.
   h) Electrically connect the Pt wires on the bar to the potentiostat with the innermost wires connected to the voltage leads and the outer wires to the current leads.
   i) Place the bar in the tube furnace and introduce the gas for the desired atmosphere (if other than air). The placement of thermocouple should be close to the center of the bar. If pO_2 is to be varied, a suitable purge time should be given to allow equilibrium to be reached. pO_2 should be monitored using calibrated oxygen sensor.
   j) For testing SOE electrodes, the conductivity measurement in presence of steam may be required. If conductivity measurement to be performed in presence of steam, an appropriate "due point sensor" should be installed in the rig.

   [The following can be automated with furnace and potentiostat control software.]

   k) Heat to the desired measurement temperature.
   l) Set the potentiostat to 0.05 to 0.1 V and measure the current, then set it to -0.05 to 0.1 V and measure the current. [The voltages can be varied if needed to keep currents within the measureable range for the instrument but should be controlled below the polarization voltages while testing electrode materials.]
   m) Heat the sample to the next temperature of interest and repeat step l.

5) Troubleshooting

6) Data Acquisition, Calculations & Data Reduction Requirements

Conductivity (σ) values can be calculated from the current (I), voltage (V), and bar dimensions including cross-sectional area (A) and length between inner/voltage wire contacts (d) according to the equation:

$$\sigma = \frac{d\Delta I}{A\Delta V}$$

7) Computer Hardware & Software

8) Data and Records Management

4. Quality Control and Quality Assurance Section

5. Reference Section