Standard Operating Procedure

(SOP)

SOEC Thermal Cycling Protocol

Test ID: HTE-P-21

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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

1. Procedures

    a. Scope and Applicability –

        i. The purpose of this procedure is to establish a common method for thermal cycling SOEC stacks from operational temperature down to room temperature, and then back up to operational temperature again.

    b. Summary of Method –

        i. To safely thermal cycle SOEC stacks, the stacks will need to be electrically disconnected from the power supply, the appropriate gas composition and flow maintained on the cathode side of the cell (fuel side), and an appropriate ramp programmed in to cool or heat the stack at the determined rate.

    c. Definitions –

        i. SOEC – Solid Oxide Electrolysis Cell
        ii. Steam Generator – device to produce steam from water

    d. Health & Safety Warning –

        i. Steam generator shut down or start up – potential burns from hot surfaces
        ii. Power supplies connecting and disconnecting – risk of electric shock
        iii. Hydrogen gas safe use – risk of fire or explosion
        iv. High Temperature kiln – risk of burns from hot surfaces
        v. Heat Traced Lines – risk of burns from hot surfaces

    e. Cautions –

        i. Failure to flow hydrogen to maintain a reduced cathode
        ii. Ramping too quickly
        iii. Condensing water in lines or stack
        iv. Not disconnecting power supply during thermal cycling could result in oxidation of the cathode in the presence of CO2.

    f. Personnel Qualifications / Responsibilities –

        i. Familiarity in the use of hydrogen gas, steam, power supplies, and with high temperature kilns and their controllers, general good laboratory practices.

g. Equipment and Supplies –

i. SOEC Stack and Test Stand
ii. Steam Generator
iii. High purity hydrogen and nitrogen gas
iv. Data Acquisition System to monitor temperatures, voltage, current and gas flows
v. Programmable controller with ramp function for the kiln.

h. Step by Step Procedure-

i. Define operational/thermal cycling needs (application dependent)

ii. Define desired heating and cooling rates for thermal cycling.
    1. OxEon standard heating rate for stacks = 100 °C/hr (1.67 °C/min)
    2. 15 °C/min is considered a rapid heating rate for a HTE stack that allows for reaching operating temperature (e.g. 800 °C) from RT in less than an hour.
    3. Standard stack test kilns may not be able to achieve this rate however (~ 5.5 °C/min average heating rate with full kiln power from OxEon's standard test kilns).
iii. Define desired operating temperature dwell time, stack operating conditions when at temperature, and lag time needed for temperature stabilization.
iv. Define gas flows during cooling and heating (same as operation or shut-off to simulate full system shut-down?)
    1. Safety consideration: If H_2 is flowing below ~400 °C during a thermal cycle there is a risk of building up H_2 in the test kiln and causing a combustion once the kiln is re-heated. Safety measures should be taken to avoid this; OxEon's standard procedure is to cut the flow of H2 at these lower temperatures during thermal cycling.
v. Define number of cycles
vi. Program thermal/operational cycling according to defined needs. Ensure thermal and load cycling are synchronized.
vii. Consider disconnecting the power supply from the stack during cooling and heating to eliminate possibility of a small leakage current that could cause unintended stack degradation.
viii. Analyze degradation vs. OC (operating cycles)
ix. Seals: leak rate measurements after defined cycles
x. Power density degradation for desired operating conditions
xi. ASR (R", aASR, iASR)

1. Fit to power law R”(T) = R0”*(1+((OC-1)/tau)^alpha)

xii. Example OxEon Thermal Cycling Procedure:

1. Remove SOEC stack from operation and Thermal cycle
    a. Sweep current or voltage down in steps
    b. When at zero current disconnect power supply
    c. Turn off steam flow
    d. Adjust hydrogen to 3% of the total flow with balance nitrogen on the cathode side
    e. Ramp kiln controller to cool off at 100C/hr
    f. Start cooling ramp
    g. When the kiln temperature reaches 400C turn off the hydrogen flow and only flow N2 on the cathode side
    h. When the kiln temperature reaches a temperature below 50C then turn off N2 flow

2. Heat SOEC stack and place under load

    a. Flow the desired flow rate of nitrogen on the cathode side
    b. Program kiln controller to heat at a rate of 100C/hr to operating temperature.
    c. Start heating ramp
    d. When the stack reaches 400C, start a flow of hydrogen equal to 3% of the total gas flow on the cathode side.
    e. When the stack reaches the operating temperature, adjust the N2 and H2 to the desired flow rates for operation and add the steam flow.
    f. Connect the power supply to the stack
    g. Sweep the current or voltage in steps to reach the desired operating voltage.

i. Data and Records Management –

    i. Record sweep data and log kiln ramps and gas flows

j. Quality Control and Quality Assurance Section

    i. QC activities are designed to allow self-verification of the quality and consistency of the work. Describe the preparation of

appropriate QC procedures (self-checks, such as calibrations, recounting, re-identification) and QC material (such as blanks – rinsate, trip, field, or method; replicates; split samples; spikes; and performance evaluation samples) that are required to demonstrate successful performance of the method. Specific criteria for each should be included. Describe the frequency of required calibration and QC checks and discuss the rationale for decisions. Describe the limits/criteria for QC data/results and actions required when QC data exceed QC limits or appear in the warning zone (control charts). Describe the procedures for reporting QC data and results.

k. Reference Section

i. Documents or procedures that interface with the SOP should be fully referenced (including version number), such as they relate to the SOP, published literature, or methods manuals. Citations cannot substitute for the description of the method being followed in the organization. Attach any that are not readily available. References should be numbered (1, 2, 3, … etc.) and may be used as a footnote. Use an acceptable format for citing a reference (see examples provided below).

ii. R1. 40 CFR 30, Code of Federal Regulations, "Grants and Agreements With Institutions of Higher Education, Hospitals, and Other Non-Profit Organizations."

iii. R2. ANSI/ASQC E4-1994, *Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs*, American National Standard, January 1995.

iv. R3. U. S. Environmental Protection Agency, 2000a. *Guidance for Data Quality Assessment: Practical Methods for Data Analysis (QA/G-9)*, EPA/600/R-96/084, Office of Environmental Information.