Standard Operating Procedure

(SOP)

SOEC Durability Test Protocol

Test ID: HTE-P-24

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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 defining the durability and long-term degradation rate of SOEC devices.

    b. Summary of Method

        i. Use long-term stack or cell data to produce a non-linear degradation model for better expectations of lifetime performance compared to a simple linear degradation model.

    c. Definitions

        i. SOEC – Solid Oxide Electrolysis Cell
        ii. ASR – Area Specific Resistance – Internal resistance of a SOEC stack normalized for area ($\Omega \cdot cm^2$).
        iii. Apparent ASR – That resistance obtained from the slope of the IV curve of current density (current/active area of the cell) and cell voltage (individual or Stack Voltage/#cells)
        iv. Intrinsic ASR (iASR) – The resistance based on incremental changes that take in to account the localized Nernst potential at each point ($\Omega \cdot cm^2$).
        v. HTE = High Temperature Electrolysis
        vi. SSE = sum of squared errorors

    d. Health & Safety Warnings

        i. One should conduct their own Hazardous Operations review to insure they are practicing safe operations for their facility.
        ii. Some potential hazards:
            1. Steam (pressure hazards and burns)
            2. Electrical Shock (high voltage and current)
            3. Burns from hot surfaces

    e. Cautions

        i. Insure proper polarity connection of the power supply or potentiostat / galvanostat to the SOEC device.

f. Interferences

    i. Measurement equipment should be calibrated in its HTE test location to account for any electrical interferences.

g. Personnel Qualifications / Responsibilities

    i. Familiarity in the use of math and algebra
    ii. Electrochemical testing and good lab practices

h. Equipment and Supplies

    i. Potentiostat / galvanostat for single cells and or DC power supply and current shunt for stacks
    ii. Data collection & analysis software to record voltage/current values and to produce IV plots for a SOEC stack or single cell.
    iii. DC power supply, shunt, volt meter
    iv. Instrument or Method Calibration and Standardization
        1. The potentiosat /galvanostat used to record IV characteristics should be regularly calibrated with a standard to insure accurate measurements.
        2. The calibration method will depend on the particular device, but values should be calibrated in the range of actual measurements expected from the SOEC device and while in the normal test location
        3. Calibrate according to manufacturer specifications.

i. Step by Step Procedure

    i. Degradation & Lifetime Model:
        1. Diffusion limited degradation processes
            a. Oxide scale growth
            b. Cation interdiffusion & reaction layers
        2. Leads to a parabolic rate law
            a. R”(t)=R0”*(1+sqrt(t/**τ**)) **where tau is time to double area specific resistance**
        3. Integrate 1/R”(t) for average lifetime production
            a. Integration substitution variable U=1+sqrt(t/τ)
            b. Lifetime fraction of initial production rate:

$$2*(U-\ln(U)-1)/(t/\tau)$$

ii. Generate Long-Term Data Set:
    1. Operate SOEC to generate degradation data
        a. Minimum 1,000 to 2,000 hours – prefer 4k to 20k hours
        b. Operating on representative conditions of Flow, V-I, Utilization, T
        c. Reduce data to series in (t, iASR) to normalize for operating conditions
        d. If varying T, correct iASR to reference temperature with Arrhenius fit
    2. Plot data series to identify early life artifacts
        a. Initial few 100 hours may exhibit improvement or rapid decay
        b. Eliminate early life data from series to be fit

iii. Parabolic Fit of Long Term iASR
    1. Setup Non-linear Regression of data to extract
        a. Tau and iASR0
    2. Data columns in spreadsheet
    3. Minimize SSE using solver by
        a. Varying Tau and iASR0
    4. Plot Data vs fit
    5. Report Tau & iASR0
    6. Assume lifetime (hours)
    7. Report lifetime average prduction rate

Time, hours	**iASR ohm-cm^2 Data**	**iASR ohm-cm^2 Model Predicted**	**Squared Error**
Time 1	iASR 1	=iASR0*(1+sqrt(Time 1/Tau))	=(iASR1-Model 1)^2
Time 2	iASR 2	Model 2	Squared Error 2
Time 3	iASR 3	Model 3	Squared Error 3
…	…	…	…
Time last	iASR last	Model last	Squared Error last
	iASR0 variable	Tau variable	Sum Squared Error

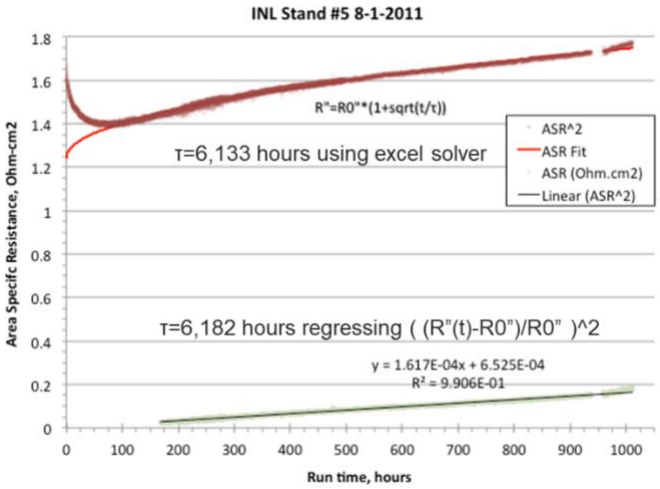


Figure 1 - Example of degradation model fitting to a stack with relatively high degradation.

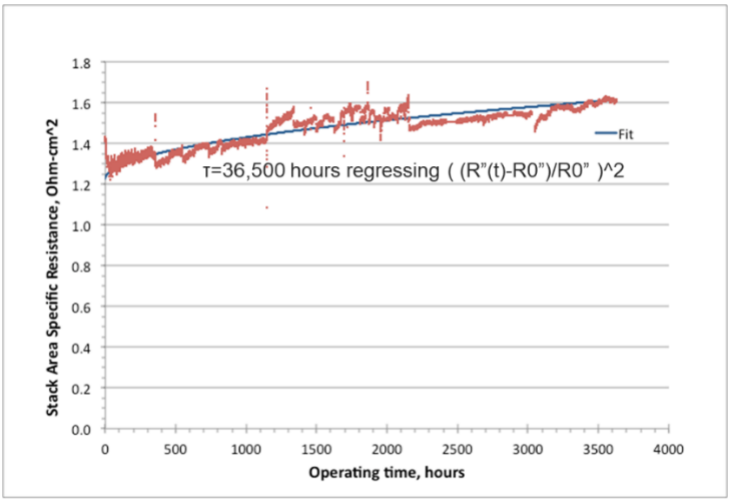


Figure 2 - Example of degradation model fitting to a stack with relatively low degradation.

iv. Computer Hardware & Software

1. Data acquisition hardware and software to record current and voltage measurements.

j. Data and Records Management

i. IV data and calculated results should be recorded and saved in a secure location and backed up in a physically separate location.

2. Reference Section

a. 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).

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

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

d. 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.