Standard Operating Procedure

(SOP)

High-Temperature SOEC Test Termination and Disassembly

Test ID: HTE-P-30

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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.

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3. Abstract

This step-by-step test protocol is presented for the test termination and disassembly of small-to-intermediate planar solid oxide electrolysis cells (SOEC). Careful control of the operating conditions at the final stages of solid oxide cell testing is essential to preserving the cell for postmortem analysis and safety in the laboratory. In an effort to establish a universal benchmark test protocol, appropriate cell cooling and disassembly procedures of SOECs are discussed. Following the presented procedure, one can safely end a test and collect the post-test cell for physical and chemical characterization.

4. Introduction

Increased interest in efficient advanced water-splitting technologies has brought about the development of the SOECs from around the world. However, discrepancies in the SOEC pre-conditioning protocol between research groups in the high-temperature electrolysis (HTE) community make it difficult to guarantee consistency or comparable results. A high priority has been placed on developing accelerated stress test (AST) protocols to reduce test duration and cell/stack fabrication time and cost. Few procedures for testing SOECs have been reported to effectively synchronize the global advancement of high-temperature water-splitting technologies [1], [2]. The focus is primarily on operating conditions/techniques and protocols for AST to better understand degradation phenomena and device lifetime [3], [4], [5]. Fortunately, the development of solid oxide fuel cells (SOFC) has been extensively reported on with start-up and cool-down procedures readily available [6], [7], [8]. By applying similar principles and safe practices, a standard test termination and disassembly was developed in order to create a baseline for SOEC testing.

5. Procedures

a. Scope and Applicability

The proposed protocol is specific to metal-supported SOE cells for non-metallic test fixtures at a variety of operating conditions however, it can be adapted for larger cells with manifolds, interconnects, or flow fields. Not included are cell fabrication, test rig design, cell start up, steam generation, electrochemical techniques, and post-test analysis techniques. The step-by-step procedure was developed for the termination of planar solid oxide electrolysis cell (SOEC) tests, variations may be necessary to accommodate different materials and cell designs.

b. Summary of Method

The protocol is designed for the simple and reproducible cool down and disassembly after completion of SOEC testing. Once returning the cell to open circuit voltage (OCV), the fuel gas is switched to H_2 and the furnace was cooled

to room temperature (RT). After removing the cell from the test rig, it is prepared for posttest analysis.

c. Health & Safety Warning

Users should be trained to be able to safely handle and use hydrogen and high-pressure gas systems, furnaces, and electrical systems. H_2 is explosive in the presence of oxygen, with a flammability limit between 4.0 and 75.0vol% in 1atm of air. Metal tubing and connections are required for H_2 and should be leak-checked before the start of the test. The furnace that the test takes place in should be located within a fume hood in the case of a gas leak. A hydrogen gas sensor mounted in the lab area/fume hood is required for the safety of those occupying the area. A second hydrogen gas sensor is also installed to sample air side gas as part of the safety system which enables an automatic switch from H_2 to forming gas ($5\%H_2/N_2$) during cell cool down. Hazards can also include electrical energy around water, hot steam, and hot surfaces. Research institute, fire marshal, and other regulatory body guidelines should always be followed.

d. Equipment and Supplies

The used equipment during cool down includes a Thermolyne 48000 high-temperature furnace, type-K thermocouple, SOEC, test rig, Honeywell UDC2500 temperature controller, Alicat Scientific mass flow controllers, RKI Beacon 800 hydrogen gas monitor, Swagelok ball valves, REDHAT solenoid valve, and a gas delivery system. A schematic of the alumina tube test rig used in this protocol is pictured in Figure 1 [1], [2], [9], [10], [11]. To disassemble the test-rig and cell, wrenches, cutters, and an Dremel/Milwaukee electric rotary tool with aluminum oxide grinding bits are needed.

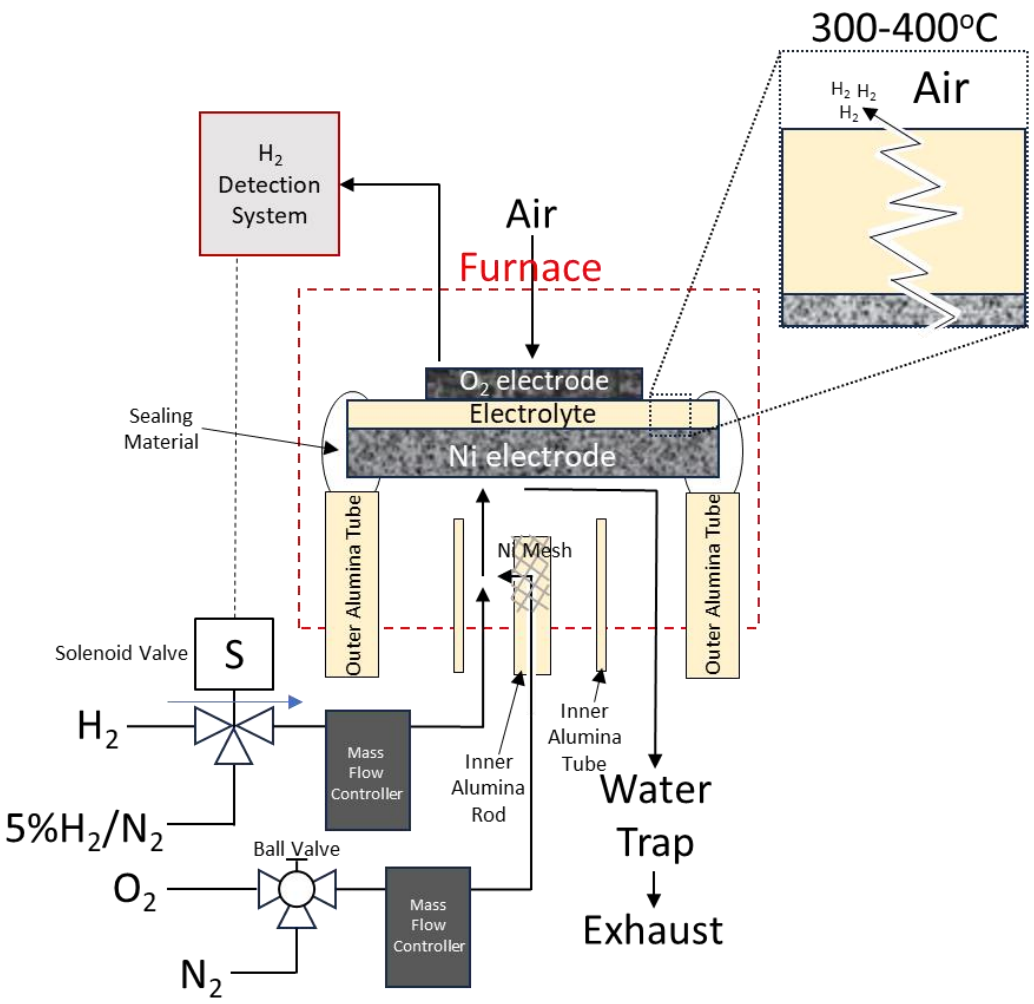


Figure 1. Schematic of SOEC test rig and gas delivery system. Not drawn to scale.

e. Step by Step Procedure

1) Once the current test of a SOEC is deemed to be complete or the performance is too low to warrant further testing, the test is terminated. All potentiostatic/galvanostatic techniques are stopped and the cell is returned to OCV.
2) Switch the fuel gas flow to 100% H_2 while continuing the flow of air to the O_2 electrode. This will preserve the Ni electrode in its metallic form for postmortem analysis.
3) Cool the furnace to RT at 3-5°C/min. As the system cools to 300-400°C, the cell will likely fracture and break due to the mismatch in coefficients of thermal expansion between the Ni-metal supported SOEC and the sealing material/outer alumina tube of the test rig. As H_2 leaks from the fuel side to the air side of the SOEC through micro-cracks, the H_2 detection system is triggered. Power to the solenoid

valve is lost and fuel flow is instantly switched from 100% H_2 to 5%H_2/N_2.

4) Once the temperature controller reads RT, turn the fuel gas flow to 0 sccm.
5) Disconnect the voltage/current leads and the H_2, O_2 and exhaust gas lines from the test rig. Carefully remove the test rig from the furnace.
6) Secure the test rig to a support stand with a rubber gripped clamp.
7) Remove the spring-loaded ceramic air diffuser.
8) Using cutters, cut all current/voltage leads at the cell and glass sealant.
9) Carefully grind the away the glass sealant from the test rig using a rotary tool with sanding bit. Being careful to stay away from the face of the O_2 electrode, the cell should separate from the test rig once enough peripheral sealant if removed.
10) Collect the cell and break in half with your hands if the cell is not already fractured. One piece of the cell will be sent for posttest analysis, while the other will go into storage in case further analysis is required.
11) Label the container of the piece of cell intended for analysis with TEST ID#, and the container with the remainder of the cell meant for storage with TEST ID#, LSL ID# and a few descriptive words about the test/cell.

4. Quality Control and Quality Assurance Section

Regular check of thermocouples, gas flow controllers, hydrogen gas monitoring system should take place to ensure correct readings and operating conditions. Maintaining a reducing atmosphere during cell cool down is imperative to obtaining useful microstructural and chemical information from SEM/EDS images taken of the Ni electrode. For this reason, during cell cooling and subsequent cell fracturing, the timely detection of H_2 by the hydrogen gas monitoring system on the air side of the cell and the ensuing actuation of the solenoid valve to switch the fuel gas from H_2 to 5%H_2/N_2 is critical.

5. Reference Section

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