Standard Operating Procedure

(SOP)

High-Temperature SOEC Test Preconditioning and Startup

Test ID: HTE-P-29

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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

A simple step-by-step test protocol for the preconditioning and startup of small-to-intermediate planar solid oxide electrolysis cells (SOEC) is presented. Careful control of the operating conditions in the initial stages of solid oxide cell testing is essential to maintaining structural and chemical integrity of the cell with consistent results. In an effort to establish a universal benchmark test protocol, appropriate firing and startup procedures of SOECs are discussed. Following the presented procedure, one can achieve a hermetic seal for a high chemical potential while minimizing polarization losses.

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 cell preconditioning and startup protocol was developed in order to create a baseline for SOEC testing.

5. Procedures

a. Scope and Applicability

The proposed protocol is specific to button and rectangular cells 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, steam generation, electrochemical techniques, cell cool down and, post-test analysis techniques. The step-by-step procedure was developed for testing planar solid oxide electrolysis cells (SOEC), variations may be necessary to accommodate different materials and cell designs.

b. Summary of Method

The protocol is designed for the simple and reproducible operation of SOECs under diverse testing conditions. The electrodes of the prefabricated MS-SOEC are

electronically connected to an external potentiostat/galvanostat, followed by heating and curing the glass seal. After reducing the fuel electrode in hydrogen, steam is formed by introducing oxygen and combusting with hydrogen to operate at desired test conditions until test termination.

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 gas sensor is also recommended to analyze the gas from the air electrode. The presence of water or hydrogen in the air side can indicate a fractured cell, in which case a safety mechanism engages a solenoid that switches the fuel side gas to forming gas ($5\%H_2/N_2$). 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 includes a prefabricated SOEC, Thermolyne 48000 furnace, type-K thermocouple, Schott G18 sealing material, test rig, Honeywell UDC2500 temperature controller, Gamry/Arbin potentiostat/galvanostat, Alicat Scientific mass flow controllers, and a stainless-steel gas delivery systems for hydrogen, oxygen and nitrogen. A schematic of the alumina tube test rig used in this protocol is pictured in Figure 1 [1], [2], [9], [10], [11]. Schott sealing glass is used to bond the cell to the alumina test rig, while both electrodes are connected electronically to the potentiostat/galvanostat. Variations to the cell structure or using ceramic cement to seal will require a modified protocol.

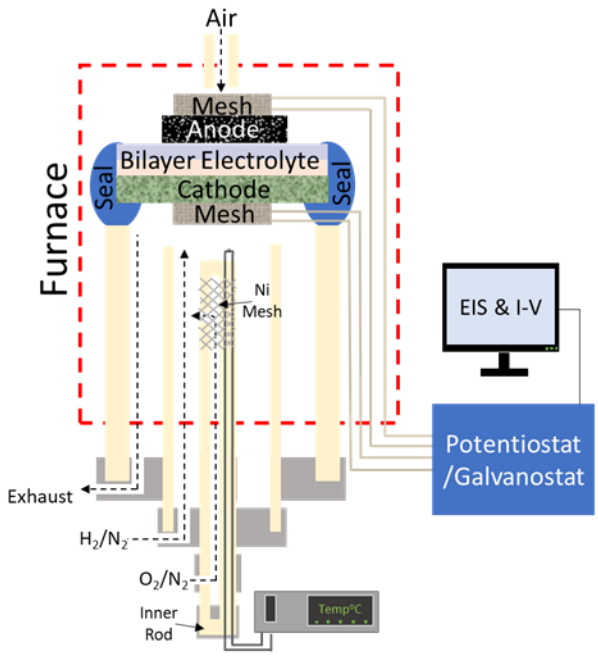


Figure 1. Schematic of button cell sealed test rig in furnace, with electrically connected potentiostat/galvanostat

e. Step by Step Procedure

1) Inspect the test rig for damage, especially along the tube edge that will be in contact with the cell. If necessary, polish the surface of the alumina tube with sandpaper.
2) Inspect the prefabricated SOEC for any damage. This may include fractured cathode/electrolyte layers, off-center printed oxygen electrode, delaminating current collecting mesh, and more. Example of prefabricated button SOEC is pictured in Figure 2.

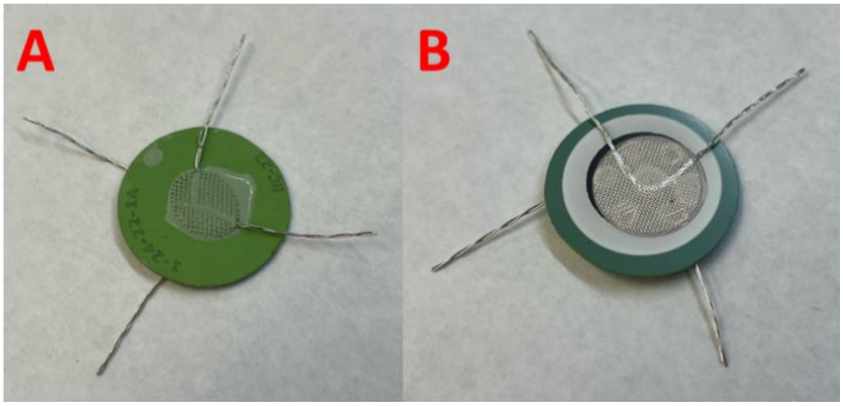


Figure 2. Fuel/cathode side (A) and air/anode side (B) of standard button SOEC

3) Prepare 2 glass sealing gaskets (ID:¾”; OD:1”) by hole-punching tape-caste glass sealing material. Popular sealing materials include alumina and glass

in the form of pastes and tape, a tape cast fiber reinforced G18 (barium-alumina-silicate based glass ceramic) produced by Schott is used in this protocol. Different glass sealing gaskets will be necessary for different cell sizes and test setups.

4) Flip the SOEC with Ni side facing up and bend the current collector leads towards the ceiling. Wet the surface of the Ni electrode and glass sealing gaskets with isopropyl alcohol to improve wetting and place one of the gaskets onto the perimeter of the cell. Fold down the current collector leads onto the first gasket and apply the second sealing gasket directly on top of the first gasket to sandwich the current collector leads.
5) Using tweezers, grasp the cell/gasket assemble and center it on the outer alumina tube of the test rig with the O_2 electrode facing up.
6) Apply compression with finger to O_2 electrode while adding sealing material over the edge of the cell to fill in gaps and secure current leads coming from anode. Sealing the edge of the sell to the alumina tube/test rig is critical to ensuring a barrier from the fuel and air sides of the cell, maintaining an electrochemical potential across the electrolyte.
7) If the SOEC comes premade without a current collector on the O_2 electrode, attach silver current collector with gold paste to the electrode. Stagger the direction of the current leads coming from the cell to allow for working room when spot welding.
8) Fix cell to test rig with spring loaded ceramic air diffuser.
9) Spot weld the current leads to the conductive wires running out of the test rig to be later connected to a potentiostat/galvanostat.
10) Place the test rig into the furnace and secure the fixture before attaching hydrogen, oxygen, and exhaust lines to the test rig. Connect the current leads from the test rig to the potentiostat/galvanostat with alligator clips.
11) Thermal treatment of the G18 glass sealing material is as follows:
    i. 80^{o}C/1hr (3^{o}C/min)
    ii. 850^{o}C/1hr (1^{o}C/min)
    iii. 800^{o}C/4hr (1^{o}C/min)
    iv. Once thermal treatment is complete, set the furnace to the final test temperature (750^{o}C) with a ramp rate of 1^{o}C/min.
12) Monitor and record the OCV change, it should be close to 0V. Flow 200sccm of air to the air electrode and 200sccm of nitrogen to purge the fuel electrode of air.
13) Step-wise, replace nitrogen with hydrogen until the gas flow to the fuel electrode is 100% hydrogen. OCV will continuously increase to >1.1V and fluctuate by ~1%, a lower value or large oscillation could indicate a pin hole or a fractured seal between the air and hydrogen. Depending on the surface area of the fuel electrode, the time necessary to reduce the cathode with vary.

14) Step-wise introduce steam to the fuel electrode by flowing oxygen and combusting with hydrogen.
15) Once OCV has reached equilibrium at the desired test conditions, collect a 0hr I-V curve by ramping current at 0.01A/s until thermoneutral voltage is reached, and back to OCV. Note the current recorded at thermoneutral voltage.
16) Collect electrochemical impedance spectra (EIS) at 0hr at a frequency of 100kHz to 0.1Hz with an amplitude of 20mV.
17) Begin electrochemical durability test by first collecting an I-V curve, then applying a constant thermoneutral voltage and recording the current continuously.
18) Collect EIS periodically.
19) Resume electrochemical durability test.

6. Quality Control and Quality Assurance Section

Regular checks of electrochemical potentiostat/galvanostat, thermocouples, and gas flow controllers should take place to ensure correct readings and testing conditions. Maintaining an intended steam/hydrogen ratio during the test is critical, directly effecting the cells OCV and polarization behavior. The seal between the cell and test rig is equally important to SOEC functionality, it's recommended to use the same amount of sealing material to enhance cell performance repeatability. Table 1 contains the theoretical OCV corresponding to test temperature and steam content of the fuel side gas. Figure 1 a plot of measured OCV from a SOEC during preconditioning and startup.

Table 1. Theoretical OCV (V) of SOEC applying humidified H_2 at 1atm vs. air.

	% H_2O								
Temp (^oC)	10	20	30	40	50	60	70	80	90
700	1.066	1.032	1.009	0.991	0.974	0.957	0.938	0.916	0.882
725	1.060	1.025	1.002	0.983	0.966	0.948	0.929	0.906	0.871
750	1.055	1.019	0.995	0.976	0.958	0.940	0.920	0.897	0.861
775	1.049	1.012	0.988	0.968	0.950	0.932	0.912	0.887	0.851
800	1.043	1.006	0.981	0.961	0.942	0.923	0.903	0.878	0.840

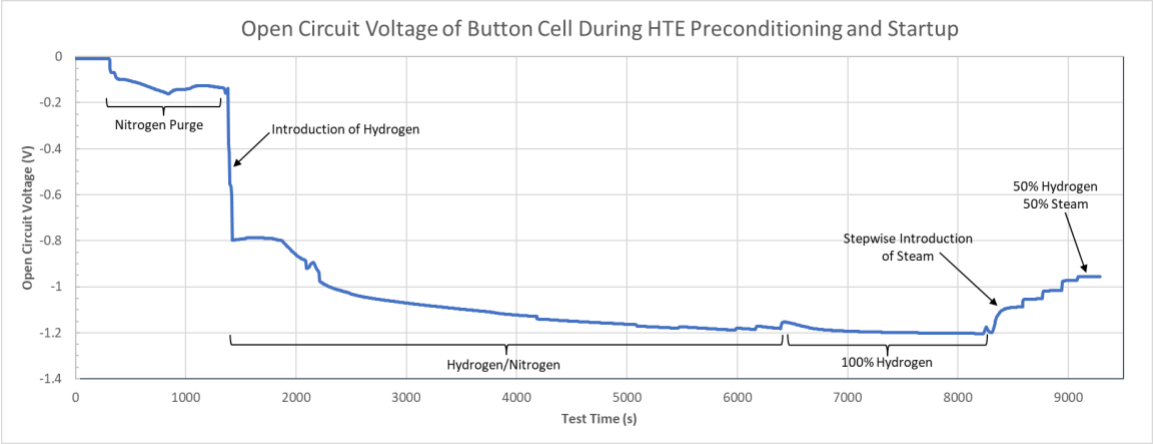


Figure 3. Measured Open Circuit Voltage (OCV) during cell startup and preconditioning.

7. Reference Section

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