Procedure for Segmented Cells Tests at HTceramix for ENDURANCE

Cell specifications:

The segmented cell used in the set-up at HTceramix comprises a continuous anode/electrolyte/barrier layer half-cell and a segmented cathode. The total active area of this cell is 71.4 cm^2. The amount of segments is 20, with 5 segments in the flow direction and 4 segments perpendicular to the flow direction.

The segmented cell is tested in a two-cell stack arrangement, the top cell being the segmented one and the bottom cell being a normal cell with an active area of 80 cm^2.

Mounting:

The procedure for mounting the segmented cell includes proprietary information about the SOLIDpower stack design and is therefore not disclosed.

Gases available for the set-up are:

- Fuel: H_2, N_2, Natural Gas, CH_4, H_2O, CO_2
- Oxidant: compressed Air, N_2

The cell is tested in co-flow configuration.

The oxidant is preheated to 730°C to prevent severe cooling at the cell inlet.

Heating

Prior to heating, the oxidant flow is set to 7600 ml/min air, and the fuel flow is set to 480 ml/min N2 (dry). These flows are the ones normalized to 0°C at 1 atm.

The heating profile is free to choose as long the maximum temperature is kept below 900°C and the heating/cooling rate is kept below 120°C.

Anode reduction

- The reduction is taking place at a cell temperature of 750°C
- Set the fuel flow to 72 ml/min H_2 + 408 ml/min N_2 (dry flow) and wait 10 min
- Set the fuel flow to 144 ml/min H_2 + 336 ml/min N_2 (dry flow) and wait 10 min
- Set the fuel flow to 216 ml/min H_2 + 264 ml/min N_2 (dry flow) and wait 10 min
- Set the fuel flow to 288 ml/min H_2 + 192 ml/min N_2 (dry flow) and wait 1 h
- Check the gas tightness of the set-up is checked by reading the OCV of each segment. An OCV higher than 1.150 mV indicates good gas tightness.

<u>Operation + shut-down</u>

The procedure below is an example of how a test could look like. Depending on specific wishes and the state of the segmented cell during the test, the procedure may be adapted.

- Record an I-V curve of the whole repeat-element under potentiostatic conditions from OCV in discrete steps until 80% fuel utilization is reached. Watch carefully the current density of the outlet segments and interrupt the IV immediately the IV if the current density is continuing to decrease at stabilized potential, or if the current density drops to zero. The nominal potential at 80% FU under such conditions should be above 0.76V.
- Perform mapping of the cell according to the procedure below.

150 hours sequence

- Stabilize the cell for 150 h at 800 mV
- Record an I-V curve of the whole repeat-element under potentiostatic conditions as above.
- Perform mapping of the cell according to the procedure below.

150 hours sequence

- Stabilize further the cell for 150 h at 800 mV
- Record an I-V curve of the whole repeat-element under potentiostatic conditions as above.
- Perform mapping of the cell according to the procedure below.

Sensitivity analysis

- Perform complete sensitivity analysis of the cell according to the procedure below.

500 hours sequence

- Operate the segmented cell (except one segment at the inlet and one segment at the outlet to record OCV) at 800 mV for 500 h
- Record an I-V curve of the whole repeat-element under potentiostatic conditions as above.
- Perform mapping of the cell according to the procedure below.

1000 hours sequence

- Operate the segmented cell (except one segment at the inlet and one segment at the outlet to record OCV) at 800 mV for 500 h
- Record an I-V curve of the whole repeat-element under potentiostatic conditions as above.
- Perform mapping of the cell according to the procedure below.

1000 hours sequence

- Operate the segmented cell (except one segment at the inlet and one segment at the outlet to record OCV) at 800 mV for 1000 h
- Record an I-V curve of the whole repeat-element under potentiostatic conditions as above.
- Perform mapping of the cell according to the procedure below.

1000 hours sequence

- Operate the segmented cell (except one segment at the inlet and one segment at the outlet to record OCV) at 800 mV for 1000 h

- Record an I-V curve of the whole repeat-element under potentiostatic conditions as above.
- Perform mapping of the cell according to the procedure below.

1000 hours sequence

- Operate the segmented cell (except one segment at the inlet and one segment at the outlet to record OCV) at 800 mV for 1000 h
- Record an I-V curve of the whole repeat-element under potentiostatic conditions as above.
- Perform mapping of the cell according to the procedure below.

Final sensitivity analysis

- Perform complete sensitivity analysis of the cell according to the procedure below

Cool-down

- Set the anode flow to 50 ml/min H_2 + 1550 ml/min N_2 (dry flow), stop polarization and disconnect loads
- Cool the cell to room temperature with 120°C/h
- Switch off the gases and the furnace

Mapping

Mapping is meant to obtain impedance spectra for each segment operated under the same condition.

Procedure:

- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.9 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.

Sensitivity analysis

Sensitivity analysis aims for obtaining better resolution for cathode and anode contributions in the impedance spectra.

Prior to starting the measurements, check that the gas flows are set right. The anode flow should be 288 ml/min H_2 + 192 ml/min N_2 (dry flow). The cathode flow should be 7600 ml/min Air.

Procedure:

- Stabilize at 800 mV for 1 h. Adjust the furnace temperature to reach a cell temperature of 750°C, if necessary.
- Record an I-V curve for each segment under potentiostatic conditions from OCV in steps of 50 mV until 80% fuel utilization is reached.
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.9 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.7 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.5 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.2 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Set the anode flow to 384 ml/min H2 + 96 ml/min N_2 (dry flow)
- Stabilize at 800 mV for 1 h. Adjust the furnace temperature to reach a cell temperature of 750°C, if necessary.

- Record an I-V curve for each segment under potentiostatic conditions from OCV in steps of 50 mV until 80% fuel utilization is reached.
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.9 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Set the anode flow to 192 ml/min H_2 + 288 ml/min N_2 (dry flow)
- Stabilize at 800 mV for 1 h. Adjust the furnace temperature to reach a cell temperature of 750°C, if necessary.
- Record an I-V curve for each segment under potentiostatic conditions from OCV in steps of 50 mV until 80% fuel utilization is reached.
- Stabilize at 800 mV for 30 min
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.9 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Set the cathode flow to 6000 ml/min Air + 7600 ml/min N_2
- Stabilize at 800 mV for 1 h. Adjust the furnace temperature to reach a cell temperature of 750°C, if necessary.
- Record an I-V curve for each segment under potentiostatic conditions from OCV in steps of 50 mV until 80% fuel utilization is reached.
- Stabilize at 800 mV for 30 min
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.9 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.7 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.5 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Record an impedance spectrum for each segment between 0.1 Hz and 100 kHz with 10 steps per decade, at 0.2 A galvanostatic load and an AC bias of 0.1 A, while the other segments are at OCV.
- Bring back cathode flow and anode flow to original values.