Thermal and current load testing results for Integrated Planar and short SOFC stacks – Birmingham experience within RealSOFC

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Understanding SOFCs: What can we learn from “advanced methods”

Work Package 1 & Work Task 1.4

Leader for Work Package 1
“Understanding of aging of SOFC for industrial applications”

Partner responsible for Work Task 1.4
“Cycled stack operation for 50 to 100 cycles at defined conditions”

Supported by the European Commission under the 6th Framework Programme
Our work horse AM Inc. Test Rig
Load cycling for IP SOFC of RRFCS Ltd.

Initial tests for 15 cells tube design:

- Attempted 100 full load cycles (actually 93 have been achieved); Results for 50 cycles presented at Grove’2005 as a poster. Full version to appeared in Journal of Power Sources, 2006 (“Cycling studies of solid oxide fuel cells” by W. Bujalski, J. Paragreen, G. Reade, S. Pyke and K. Kendall).

- General findings:
  - The module has shown some increase in resistance at the end of the cycling process. Initial visual inspection of the module in the test box identified a possible defect in one of the cells, however, when the module was removed from the box after completion of additional cycling tests leading to 93 cycles in total no physical damage to cells structure was identified.
  - However, there were some changes in the appearance of the glass selant and/or electrolyte layer.

- Cycling of RR IP SOFC tubes. Work performed within WT 1.4 for two designs of tubes consisting of 15 or 10 dual cells in series.
- The nominal temperature was 900°C but the actual one was closer to 920°C.
- The maximum load applied was 2.7 A i.e specific current load of 0.281 A/cm².
- Fuel: 97%H₂ (3%H₂O) at 1.5 l/min
- Oxidant: Air at 5 l/min

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Load cycling for IP SOFC of RRFCS Ltd.

Clearly some deterioration of Cathode side Pt connection was noticed:

- Possible interaction with glass sealant leading to poor (or partial lost of) electrical connection and increased resistance.

- Cycling of RR IP SOFC tubes. Work performed within WT 1.4 for a tube design consisting of 15 dual cells in series.
- The nominal temperature was 900°C but the actual one was closer to 920°C.
- The maximum load applied was 2.7 A i.e. specific current load of 0.281 A/cm².
- Fuel: 97%H₂ (3%H₂O) at 1.5 l/min
- Oxidant: Air at 5 l/min
Redox cycling for 15 cell tube

Redox cycling approach and results:

- An attempt was made to assess this cycling mode. Several periods of no flow either of fuel or of inert gas on the anode side and with maintaining air flow of 5 l/min on the cathode side were tried out for varying periods lasting from 5 to 30 min thus constituting redox conditions. The tube withstood a number of cycles with no sign of immediate loss of its performance.

- However some sings of damage were apparent at visual inspection stage at Cathode – Pt connection and, as in previous case, there were some changes in the appearance of the interconnect and/or electrolyte layer (see pictures on the next slide). These however could have already happened at the temperature loading part of the test run. More detailed post-mortem examination of the tube is to be carried out by RR to identify the changes to its structure in order to explain the system degradation causes.

- Numerical data did show decline in tube performance but are difficult to link with and to provide clear insight into the process and mode of degradation of the tube.

- In order to link the readings (stack voltage as a function of time and mode of operation or test type and/or its stage) with physical degradation of the tube one will have to stop/pause the experiments when the signs are becoming apparent (rather than to wait till the end of planned run!).
Cycling of RR IP SOFC tubes. Work performed within WT 1.4 for tube consisting of 15 dual cells in series.

- The nominal temperature used was 900°C (but the actual value was different by up to 10°C which was achieved due to repositioning of the thermocouples).

**Cathode-Pt interphase**

**Deterioration of Interconnect and Electrolyte**
Thermal cycling for 10 cell tube

Conditions of test run:

- Temperature cycling (using 1ºC/min heating/cooling rate) and proper, separate Pt wires arrangement was used for voltage measurement:
  - Starting from the nominal operating temperature of 900ºC for the test tube at which reduction was performed followed by 2 hrs stabilisation period at OCV over 40 runs of thermal cycling consisting of a range of temperatures (800 to 950ºC) were carried out accomplishing over 160 I-V curves in total.
  - Then the first I-V baseline curve was collected at that temperature. This was followed by short i.e. 10 hrs “durability” period using 1.2 A current load for comparison of possible degradation processes in the run.
  - The temperatures then, went through a sequence of 950, 900, 850 and 800 and then back to 900ºC allowing 30 mins stabilisation period after reaching the desired level. At each temperature I-V curves were collected (using 0.1 A steps for a period of 40 s and kept for 30 min at this level and was applied for up and down current loads).
  - After 25th (and also the final cycle) 10 hrs durability tests at 1.2 A current load were performed. Throughout the test there were some safety measures in place not allowing the voltage under load to go below its threshold value of 6 V for the tube (i.e. 0.6 V for a single cell). The loads were different for different temperatures i.e. 2.5 A for 900 and 950ºC, 2.2 A for 850ºC and 1.9 A for 800ºC, respectively. The test was run for over 28 days in total i.e. approx. 680 hours. The run was not truly continuous due to factors beyond our control e.g. hydrogen reduction valve failure, furnace over temperature thermocouple failure, scheduled electricity shut down and Easter break. However, all the necessary precautions were taken in order to prevent possible anode oxidation (safe gas used when needed, etc) in between restarts.
Thermal cycling for 10 cell tube

Experimental set up:

- Cycling of RR IP SOFC tubes. Work performed within WT 1.4 for tube design consisting of 10 dual cells in series (4.5 cm² surface area of each single cell).
- The nominal temperature ranged from 800 to 950°C with the actual difference from the target narrowed down to about 10°C. The test temperatures were achieved at controlled level of change at 1°C/min.

10 cell tube in a box

Box inside the furnace

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Cycling of RR IP SOFC tubes. Work performed within WT 1.4 for tube design consisting of 10 dual cells in series (4.5 cm² surface area of each single cell).

The nominal temperature ranged from 800 to 950ºC with the actual difference from the target narrowed down to about 10ºC. The test temperatures were achieved at controlled level of change at 1ºC/min.

General results:

- Perfect results for the first 24 temperature cycles at each temperature used

Qualitative description:

- Virtually the same OVC for all the runs with a little deterioration under load, too
- Sudden change at 25th cycle and since then progressive deterioration of the tube performance (judged by OCV and under full load voltage results)
- Eventually from 31st temperature cycle the voltage values under full load started reaching the safety limit of 6 V i.e. 0.6 V per cell
- Performance was getting worse all the time and the run was stopped after 40th cycle.
Cycling of RR IP SOFC tubes. Work performed within WT 1.4 for tube consisting of 10 dual cells in series.

- The nominal temperature used was 900°C (but the actual value was different by up to 10°C which was achieved due to repositioning of the thermocouples).

Deterioration at the anode and cathode

Cathode-Pt interphase

Deterioration at the anode and cathode
Cycling of RR IP SOFC tubes. Work performed within WT 1.4 for tube consisting of 10 dual cells in series.

The nominal temperature used was 900°C and was very precisely controlled. Temperature cycling was carried out in the range of 900°C to either 200°C or 50°C. IV curves were obtained for each cycle. Temperature ramps as shown in the graph header.

Thermal cycling at 10 C/min up (5 C/min down to 500°C and 2 C/min down to 200 or 50°C)

- Initial IV
- 1st temperature cycle
- 2nd
- 3rd
- 4th
- 5th
- 6th
- 7th
- 8th
- 9th
- 10th
- 11th
- 12th (50°C)
- 13th (50°C)
- 14th
- 15th
- 16th
- 17th
- 18th
- 19th
- 20th
- 21st
- 22nd
Comments on the above results:

General:

- The deterioration in the tube performance can be clearly seen from the graph, however, the rate of degradation slowed down with the number of cycles performed;
- Judging by the voltage drop under \( I_{DP} \) load after the 22 cycles completed to date, the average degradation rate can be calculated as 0.23% per cycle i.e. about 5% overall.
- On average, it has taken about 8 hrs to complete one cycle, thus the run to date has taken about 180 hrs. This can be translated into expected degradation from 16 to 28% per 1000 hrs.

\( I_{DP} \) load used was 2.5 A.
Information obtained from RRFC this type/generation of tubes shows typically degradation rate of 3-5% per 1000 hrs (assessed under \( I_{DP} \) load using steady state test conditions).
Comments on the above results:

General:

- Clearly this mode of operation/testing could potentially provide means of faster testing of new designs and their variations and sought improvements.

- However, it should be noted that:
  - Careful assessments of the modes of degradation need to be established first before a degree of confidence can be assured for the testing and performance predictions on the basis of accelerated ageing results.
  - We have already carried out similar tests under less severe conditions of thermal cycling (temperature ramping rate of 5 C/min were used) with the tube developing a crack after 72 full thermal cycles (this data have to be assessed and compared with the one described above). (see picture below).

- The I_{DP} load used was 2.5 A.
- Information obtained from RRFC this type/generation of tubes shows typically degradation rate of 3-5% per 1000 hrs (assessed under I_{DP} load using steady state test conditions).
- Same intermediate assessment steps were also carried out i.e. durability for 10 hrs at 1.2 A current load between set of runs etc.
Comments on 5 C/min thermal loading;

• The \( I_{\text{DP}} \) load used was 2.5 A.

• Information obtained from RRFC this type/generation of tubes shows typically degradation rate of 3-5% per 1000 hrs (assessed under \( I_{\text{DP}} \) load using steady state test conditions). Conditions used: 5 C/min on heating and 3 C/min (on average) for cooling.

General:

➢ Loss of performance of the order of 10% has been achieved giving average degradation of about 0.14% per cycle and the run lasted about 800 hrs, thus accelerated ageing of the tube of about 12.5 % per 1000 hrs has been achieved.

➢ Again, the adopted procedure seems to work but needs further careful assessment before adoption/acceptance.
Julich short stack experiments

- Temperature of 800 C.
- Flow rates as defined for RealSOFC tests.
- The load used was up to 16 A.
- Mild steel used for the weight (50 kG) did not work well in this hot environment

Julich short stack experimental set up
Remarks on Julich short stack experiments

Some initial work has been carried out using load cycling but

- Due to fuel line failure during the run not reliable information have been obtained
- Problems with controlling the temperature ramps (especially for the cooling cycle) have been experienced and also limit on the load availability (16A) of the present rig prevented us from carrying out the planned experimental work for the time being.

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- Flow rates as defined for RealSOFC tests.
- The load used was up to 16 A.
- Mild steel used for the weight (50 kG) did not work well in this hot environment
More cycling (thermal and current load) has been planned for:

- Second generation of Julich short stack
- RR IP SOFC design:
  - Single tubes
  - Three tube bundle
- Input from partners will be required in order to identify and/or interpret the modes of accelerated aging and degradations of the designs
- Due to substantial capital and revenue funding obtained recently both, two test rigs were purchased and revenue for new manpower have been made available for completing the planned work within the extended RealSOFC programme by the end of 2008.