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

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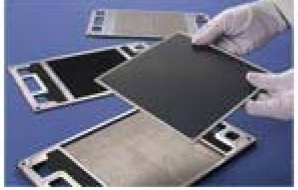

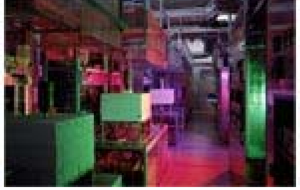


# Work Package 1 & Work Task 1.4

ECN

## Leader for Work Package 1

*"Understanding of aging of SOFC for industrial applications"*

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## Partner responsible for Work Task 1.4 *"Cycled stack operation for 50 to 100 cycles at defined conditions"*



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# Our work horse AM Inc. Test Rig




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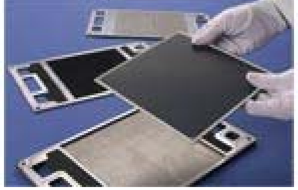
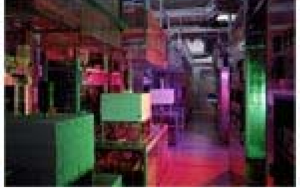





# Load cycling for IP SOFC of RRFCS Ltd.

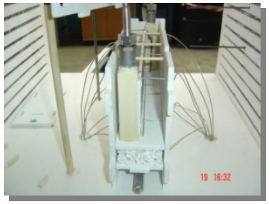
- **Cycling of RR IP SOFC tubes. Work performed within WT 1.4 for two designs of tubes consisting of 15 or 10 dual cells in series.**
- **The nominal temperature was 900ºC but the actual one was closer to 920ºC.**
- **The maximum load applied was 2.7 A i.e. specific current load of 0.281 A/cm$^2$.**
- **Fuel:**

97%$H_2$ (3%$H_2$0) at 1.5 l/min

- **Oxidant:**

Air at 5 l/min

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.



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# Load cycling for IP SOFC of RRFCS Ltd.

- **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 900ºC but the actual one was closer to 920ºC.**
- **The maximum load applied was 2.7 A i.e. specific current load of 0.281 A/cm².**
- **Fuel:**

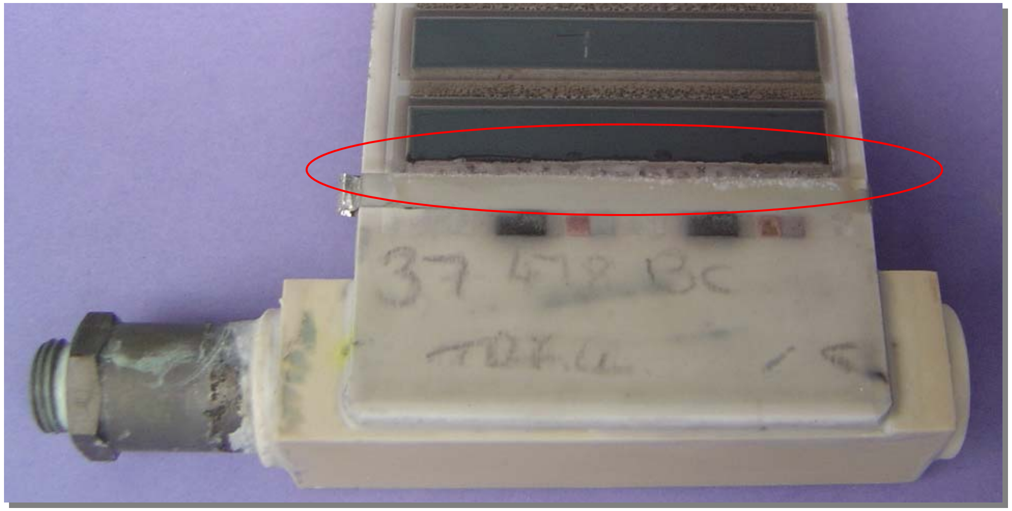
97%$H_2$ (3%$H_20$) at 1.5 l/min

- **Oxidant:**

Air at 5 l/min

## 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.





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# Redox cycling for 15 cell tube

- **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 900ºC (but the actual value was different by up to 10ºC).**

## 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!).



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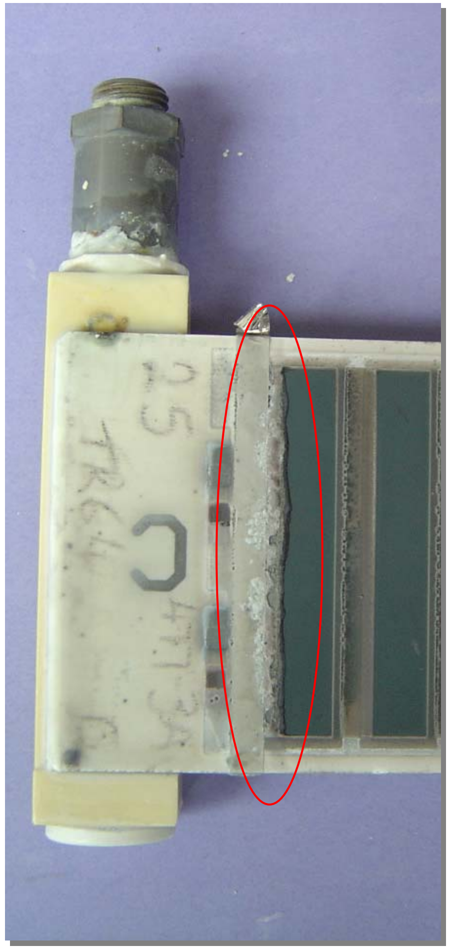
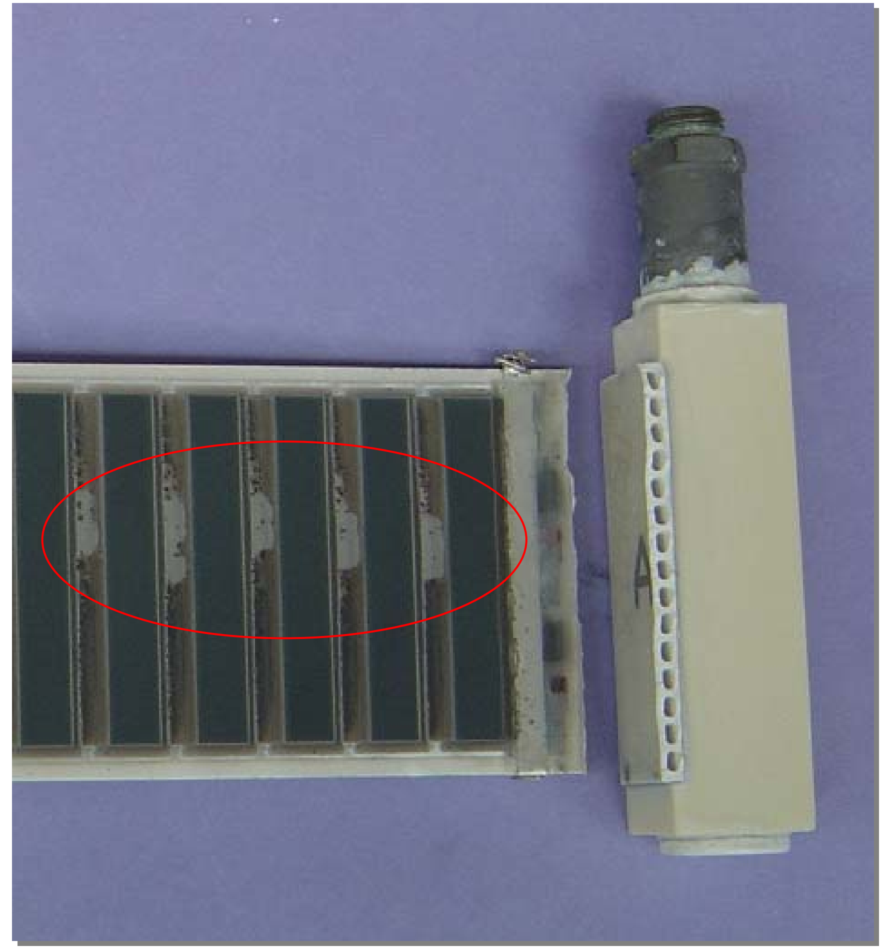
# 15 cell tube after thermal and redox cycling:

- **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 900ºC (but the actual value was different by up to 10ºC which was achieved due to repositioning of the thermocouples).**



*Cathode-Pt* interphase



Deterioration of *Interconnect* and *Electrolyte*



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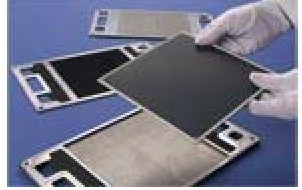

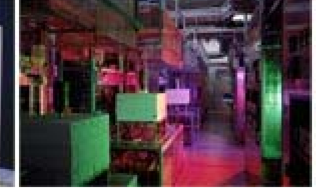

# Thermal cycling for 10 cell tube

- **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.**

## 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

- 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).
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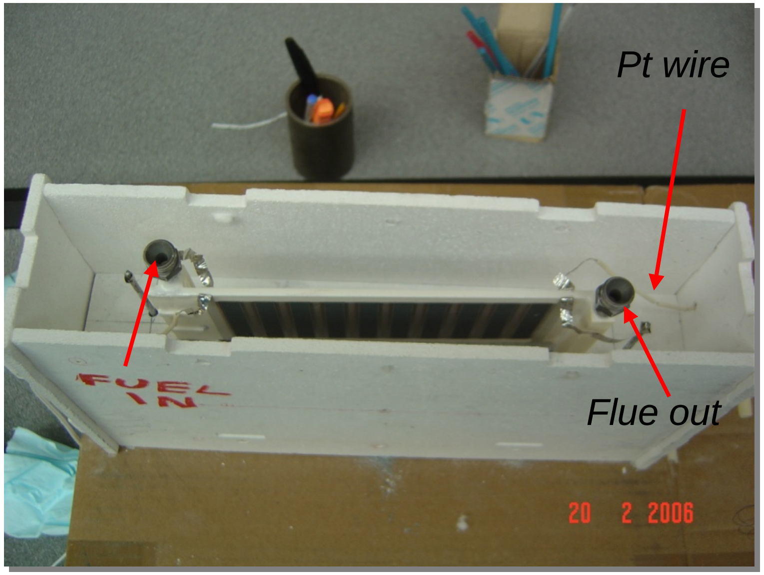
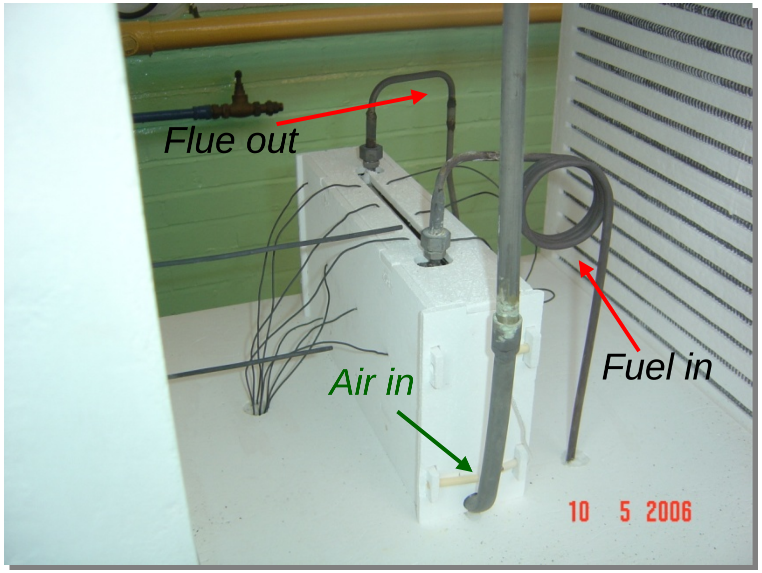
## Experimental set up:




10 cell tube in a box




Box inside the furnace

# Thermal cycling for 10 cell tube

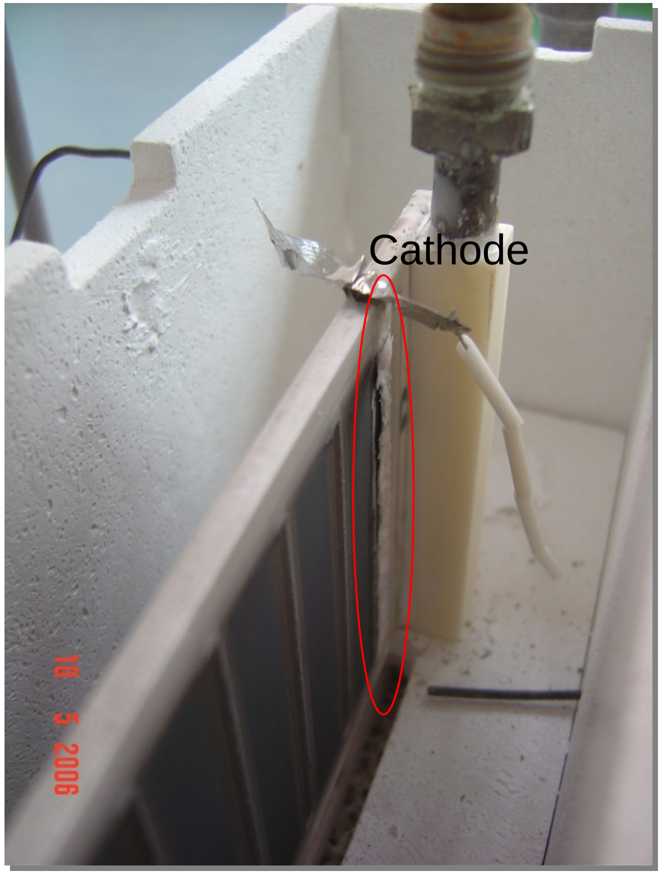
- **Cycling of RR IP SOFC tubes. Work performed within WT 1.4 for tube design consisting of 10 dual cells in series (4.5 $cm^2$ surface area of each single cell).**
- **The nominal temperature ranged from 800 to 950ºC with the actual difference from the target narrowed down to about 10ºC. The test temperatures were achieved at controlled level of change at 1ºC/min.**

## General results:

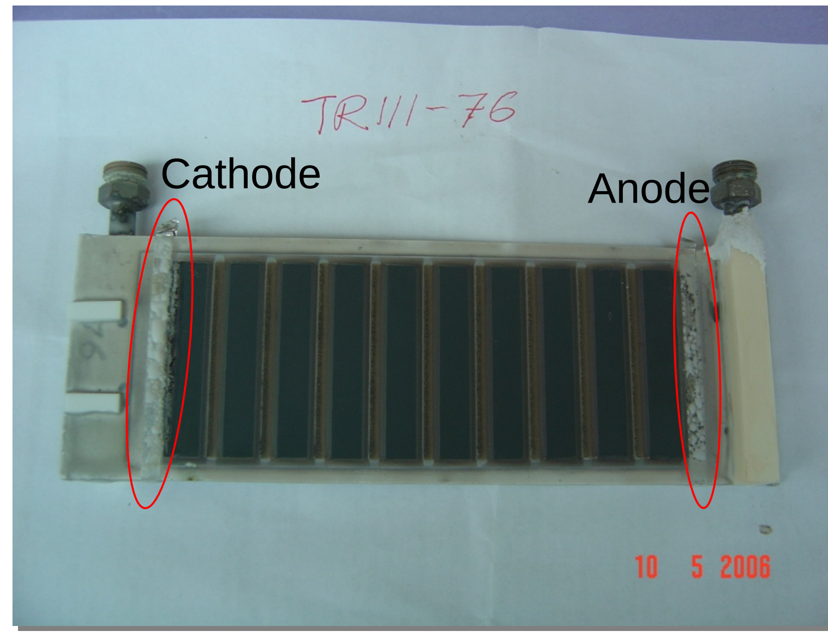
- Perfect results for the first 24 temperature cycles at each temperature used
  - Qualitative description:
    - Virtually the same OVC for all the runs with a little deterioration under load, too
    - Sudden change at $25^{th}$ cycle and since then progressive deterioration of the tube performance (judged by OCV and under full load voltage results)
    - Eventually from $31^{st}$ 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 $40^{th}$ cycle.

# 10 cell tube after 40 thermal cycles:

- 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 900ºC (but the actual value was different by up to 10ºC which was achieved due to repositioning of the thermocouples).




*Cathode-Pt* interphase




Deterioration at the *anode* and *cathode*



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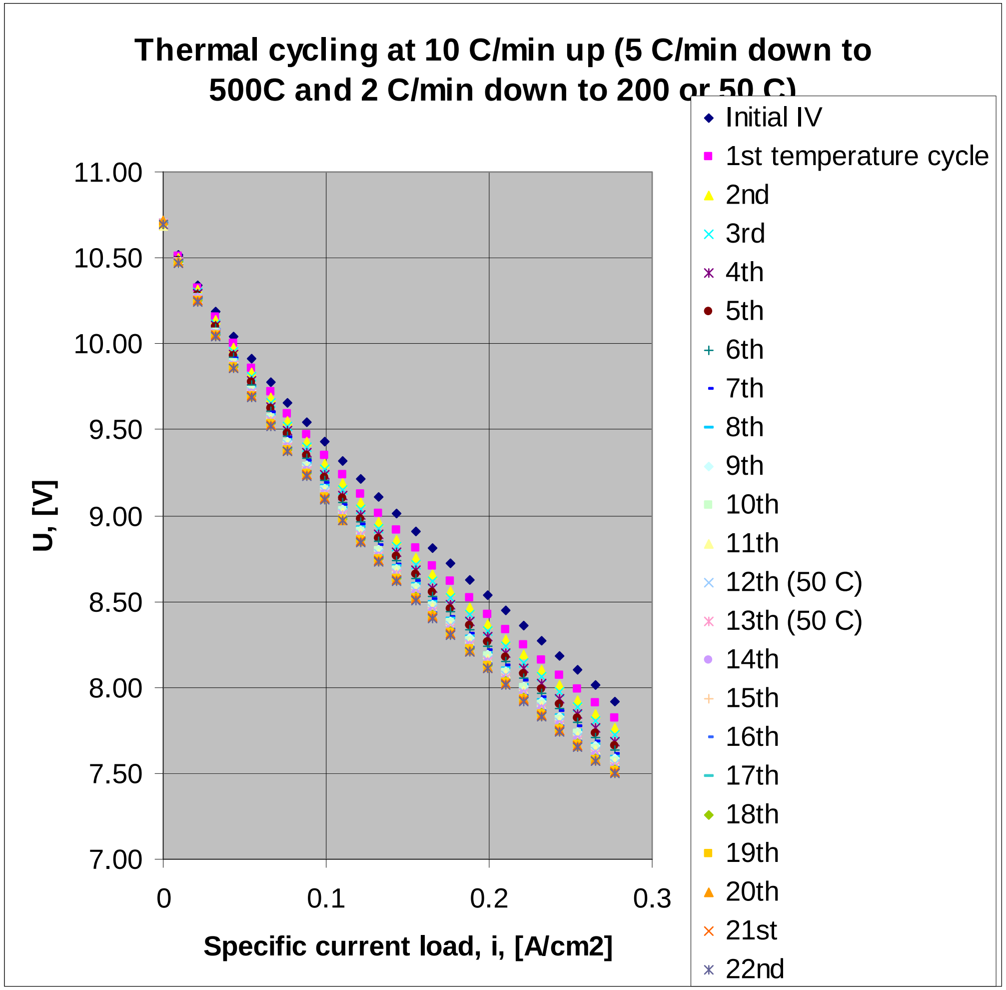
# 10 cell tube full thermal cycling:

- **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 900ºC and was very precisely controlled. Temperature cycling was carried out in the range of 900C to either 200C or 50C. IV curves were obtained for each cycle. Temperature rumps as shown in the graph header.**



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# Comments on the above results:

- 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).

## 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

REAL SOFC

# Comments on the above results:

- **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**

## 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).

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# Comments on 5 C/min thermal loading;

- **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). 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.



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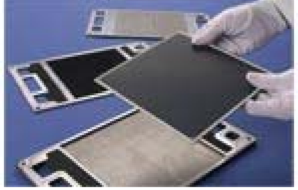

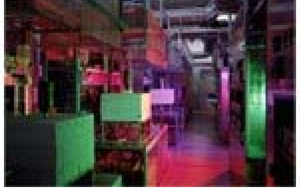


# Julich short stack experiments

- 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

- **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**

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 planed experimental work for the time being.



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# Closing remarks

- **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**

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.



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