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Degradation analysis of CFY-stacks MK35x – a guide for exact measurement

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Abstract

Degradation rates of $\Delta P/P_0<0.5$ %/1000 h are the target for SOC stacks in system operation. Planar stacks based on CFY interconnect especially in the design MK352 (see Fig. 1) are a good candidate for use in stationary applications. In this work an overview about testing of CFY SOFC stack assembly units for long-term stability and the influence of testing parameters concerning the degradation will be presented. Test parameters and conditions will be analysed regarding their impact of the stack behaviour. The constant current operation is the best option for long term testing and analysis of degradation. A guide for optimal conditions for stack tests is given to get an understanding for the values from rated power output and durability tests, which build the reference for accelerated testing. The used hardware for tests shows ageing effects and lead to an uncertainty of power output of 1.0 %. For a durability test of MK352 stacks for 3000 h a degradation rate of 1.0 to 1.4 %/1000 h were measured with an uncertainty up to +/-0.1 %/1000 h. To reach this accuracy exclusive hardware and maintenance are necessary. The most important factor for uncertainties is the temperature distribution, which is analysed in several stack tests and confirmed by simulation.

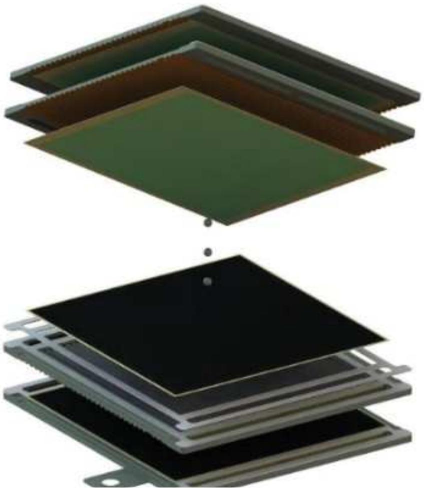

Figure 1: Exploded view of a MK352 stack assembly unit

Introduction

To measure the targeted degradation rate of <0.5%/1000 h stacks have to be tested within the conditions set by harmonized standards, but only few documents can be found in literature which address the description and specification of test procedures. A normative document (IEC 62282) developed by IEC [1] covers the basic principles for SOFC cell and stack testing. In this standard the general description of requirements on testing equipment, monitored values, eligible test procedures and experimental conditions in terms of limits for temperature deviations during the tests has been provided. SOCTESQA, which is the recent EU-project in this field of standardization, provides validated test modules and test programs for stack and cell testing as well as definitions of terms connected with such tests. The test modules from this project can be combined by sequential connection to test procedures and by defining the detailed operating conditions for every module to become test programs.

Based on available normative documents additional definitions for standardization contributing the special stack behavior have to be made and several uncertainties considered for stack durability tests. Furthermore, the test procedure, test conditions, and judging criteria for stable state shall be determined concerning test results and degradation. The MK35x technology gives a funded background of more than 17 years development for long term durability tests [2, 3, 4, 5, 6] and integration into several systems as a robust and efficient energy converter [7, 8]. Rated power measurements provide stack performance, which have a much better quality than current-voltage characteristics for stack assembly units. A special focus should be set to the instruments and the influence of their uncertainties to measured stack power and degradation rate. The goal of this work is to analyze the effect of uncertainties of instruments to the stack assembly unit and how they can lead to specific errors and deviations during long term durability tests. From obtained results a guide for precise measurement of stack assembly units will be derived.

2. Experimental

CFY-stack assembly units are tested in a hotbox inside an electrical furnace with comparable conditions concerning temperature distribution and relationship between thermal radiation and convection. This specific testing environment is very close to the real stack operation in a hotbox under system conditions. Ceramic air manifolds on two sides of the stack assembly unit and a passive insulation against heat loss by radiation and partly convection leads to a hotbox specific temperature profile inside the stack. High quality sealing between stack assembly unit and gas supply interface is required to avoid leakages, which has a directly influence on fuel utilization. Thin metallic adapter plate with low internal stresses and mica sheets fulfill this requirement.

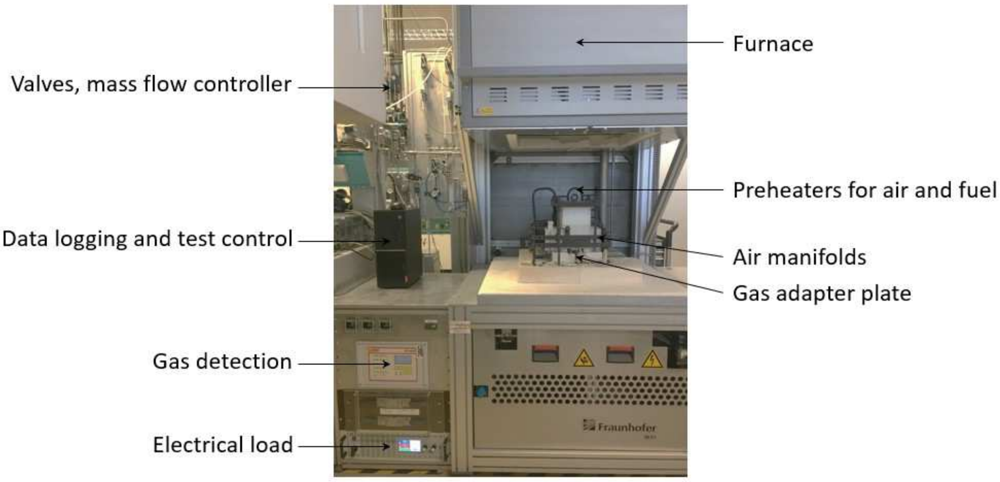

Figure 6: Test rig with electrical furnace and measurement devices (additional stack insulation around stack is removed for better visualization)

The key for a comparable and reliable characterization is rated power measurements (reference points), which are recorded regularly at constant temperature, flow rates, current, pressure and fuel utilization and used as rated operating conditions. After a short initialization phase of the stack, the rated power is measured. It is the basis for degradation rate calculation over testing time. After a defined time interval of at least 1000 h, rated power has to be recorded with necessary corrections of drifted conditions. Especially the raise of temperature when the stack assembly unit is degrading can be compensated by increased air flow or by air inlet temperature reduction. The importance of controlling temperature for a precise estimation of degradation rate will be explained afterwards.

The rated power is defined as the nominal power specified by the stack manufacturer. For description of the behavior of the stack assembly unit under different operating conditions a performance test at stationary rated power conditions is much more reliable than information obtained from current-voltage characteristics for stack assembly units, which are frequently used in literature. During current-voltage characteristics the absolute temperature, temperature distribution in the stack and the fuel utilization is not constant. Therefore, current-voltage characteristics for stacks give performance at non-stationary temperature distribution inside the stack. It is more precise to test each rated power point at constant parameters.

Based on the different principles for measurement of values for definition of stability of operating conditions, the uncertainties of single measured values during stack operation are considered below:

Air and fuel flow ($\dot{V}$)
The operating stack voltage depends on the gas distribution and uniformity of the flow. Low differences in flow rate supplied to singular cells lead to measurable deviations in the cell voltages and hence influence the stack power output. To generate a suitable flow of air and fuel precise mass flow meters are required. Due to thermal inline sensors, which are located directly in gas stream, a very fast response and even flow can be created. To guarantee this for stack testing time a maintenance and calibration of the mass flow meter in frequent intervals is important. The maintenance procedure depends on the environmental conditions and the total duty of the mass flow meter. For example, temperatures over 40°C, which can come from direct sun radiation, leads to deviations in the gas flow rate up to 10 % form the set value because of internal measurement and electronics. To avoid these issues, the manufacturer information should be considered, and mass flow meter calibrated each half a year. Clean and dry gases are also important for a stable operation. Inorganic impurities (Si, S, Cl...) and organics like carbon, which comes from oil polluted air, can poison electrodes over time and falsify measured values.

Pressure (p)
Two types of pressure can be considered simplified during stack tests, which have an influence of the stack performance: standard pressure (101325 Nm^{-2}) at rated conditions and dynamic pressure. Both pressures serve to determine the pressure loss over anode and cathode. High pressure loss can lead to leakages over stack sealing (mica), which can cause a non-effective fuel utilization. The standard pressure comes from outside of the stack assembly unit and is influenced by environmental conditions. Pressure loss results from specific density of the flow, the flow velocity and the cross-section area inside the stack assembly unit. Absolute pressure deviations can lead to slight power variations.

Measurement of cell voltages (U)
The single cell voltages on CFY-SOC stacks are measured by directly spot welded platinum wires to the CFY interconnect plate and measure one or more single cell units. Because the stack assembly unit operates as a voltage source only a pending potential can be measured between both wires. A precise rated power measurement is essential, whereby an isolating amplifier (MINI MCR-SL-U-U by Phoenix Contact) is used to get stable cell voltages. For the measurement itself suitable PLC-voltage devices are recommended because a logging over whole test time is necessary. Internal measurements show differences between Siemens PLC and Bachmann PLC (MX213 with voltage measurement module AIO288) which were used for stack testing in our laboratory. Disadvantage of this approach is the electronics inside the measuring section which can be influenced by temperatures and another factors, like electromagnetic emissions. Maintenance and continuous calibration of the devices are necessary.

Current (I)

Electric loads with high precision are used to measured CFY-stack assembly units in SOFC-mode. To avoid measuring inaccuracies a constant current (CC) operation is recommended because it has the lowest distortion of the temperature profile. In Figure 2 the temperature distribution is shown for a middle layer of a 30-cell stack in hotbox operation at start (left) with a temperature difference of ΔT=65 K over the active area (800-865°C). These simulated values are validated on stack level with an internal temperature measurement of 9 thermocouples at the green marked spots (Type N, in accordance with IEC 61515, IEC 60584-1, 576 IEC 60584-2 or IEC 60584-3). The operation temperature ($T_{\_Ref}$) is measured in the middle of the air outlet 10 mm away from the stack assembly unit with a rated thermocouple (Type N). By an absolute power degradation of $\Delta P/P_0$=3 % the stack assembly unit produces more heat (ΔT=70 K) and has to be cooled by lowering air inlet temperature till operating temperature at air outlet is $T_{\_Ref}$=835°C +/-1 K. If the stack assembly units are degrading in constant power (CP) operation the current density increases and lead to higher maximum temperature and higher temperature differences ΔT=73 K (see Figure 2 right).

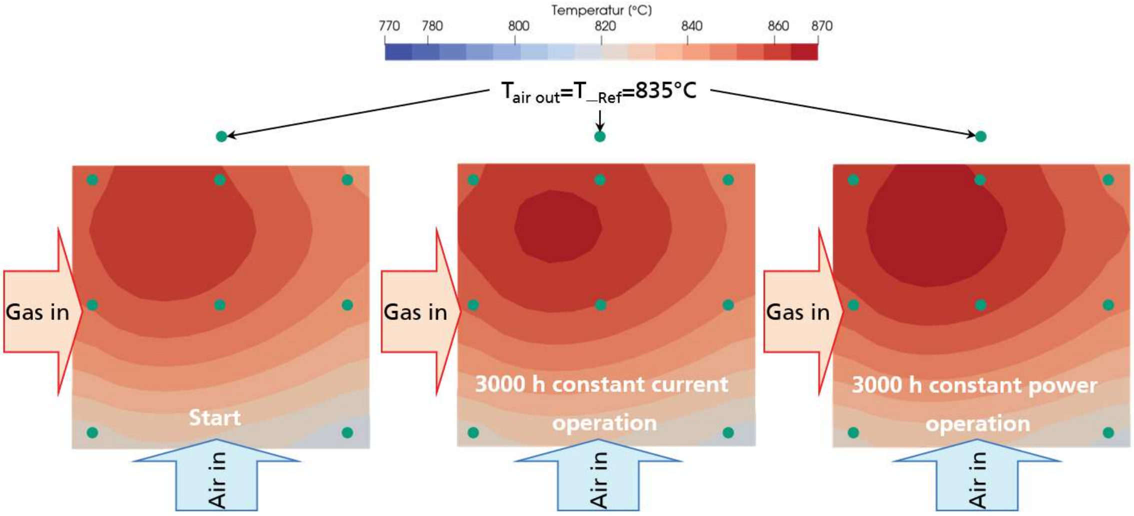

Figure 7: Simulated temperature distribution of a middle layer in MK352 stack assembly unit in hotbox operation at 35 A, 40 %H_2 in N_2 η_{FU}=75 %, air flow 60 Nl/min, $T_{\_Ref}$=835°C; at start with constant current CC (left), after 3000 hours with constant current CC (middle) and after 3000 hours with constant power CP (right), green points are positions of thermocouples

The increase of the temperature of the stack assembly unit with constant current operation is visible ($\Delta T_{3000h\ CC}$=5 K) but lower than in CP mode ($\Delta T_{3000h\ CP}$=8 K) because of constant fuel utilization of single cell. This special behavior can be transferred to other stack designs and can explain the difference in degradation rate in several publications i.e. [9]. The controlling of other parameters like fuel flow or air utilization is much easier in CC mode. Operating stack assembly units in constant voltage mode (CV) is not recommended because a decreasing current density leads to lower heat sources and very inhomogeneous temperature distributions.

Temperature (T)

From tests and literature, it is well known that the temperature is the most important parameter during stack tests [11, 12, 13]. To achieve a comparable and homogenous temperature distribution, a balancing between internal heating effects caused by ohmic resistances, fuel oxidation and external heat sources must be realized. Rated temperatures on defined positions of the stack assembly unit are recommended and are the key for comparable temperature distributions. The examples of temperature distribution in Figure 2 were given for a simulated CPOx reformate as a fuel and change significantly when methane containing fuel and internal reforming is used [10].

Furthermore, the measurement with a rated thermocouple is necessary and different stack tests are comparable because of rated measuring principle and reference temperature.

Deviations in power output and degradation between uniform stack assembly units can be explained mostly by differences of the single stack components and the temperature measurement itself. Even thermocouples can be sources of measurement deviations. On the one hand it is necessary to choose the right temperature range of the thermocouples, which must be suited to stack tests temperatures and on the other side it must be understood that thermocouples degrade differently depending on the type and diameter (see: IEC 61515, IEC 60584-1, 576 IEC 60584-2 or IEC 60584-3). For example, type S thermocouples are suitable for temperatures till 1400°C (Pt10%Rh) but they are prone for silicon (Si) which leads to an accelerated degradation and a faulty measurement of temperature. The measuring point inside the thermocouples shifts due to contamination through the protection case. This local shift of the measuring point can be detected in calibration furnace with an precise temperature distribution. The drift depends also on the thickness of the casing, thin thermocouples drift faster than thicker because the contamination diffusion through the material of the case takes longer [14]. Type N-thermocouples with high quality “class 1” are recommended because the Silicon is already inside the pair of filaments, they are much cheaper than type S and the working range up to +1200°C (specific casing: Pyrosil C) suits well for SOC-applications. Within several tests over the last years it was concluded that the deviation of one single batch thermocouples is less uncertain than data sheet specification. High-quality instruments for temperature measurement are delivered with a certification and producer measured rated values. Before thermocouples are used in tests, they should be calibrated, documented (ID-numbers) and compared with producer certifications. After the stack test, the thermocouple must be re-calibrated at the same initial temperature. It is recommended to use thermocouples only for one durability test. A temperature correction of measured data is necessary if differences between start and end of operation are recorded. For example, at start of the stack test a thermocouple positioned in the middle at air outlet recorded 830°C. After 3000 h the temperature at air outlet increased up to 840°C because of degradation of stack assembly unit and shifting of the thermocouple. A reference measurement of the thermocouple after test shows a $\Delta T=3$ K compared to start reference. The power output has to be linear interpolated by specific stack value for $\Delta P/\Delta T=0.025$ W/K/cell (see chapter 3) before the precise degradation rate can be calculated.

Instruments and expected uncertainties
In Table 9 the specific instruments used in 10-cell stack tests in hotbox are listed. To determine the temperature a thermocouple type N (Pyrosil C) with a diameter of 1.5 mm and quality-class 1 is used. The electric current is controlled by an electric load (PL912, Höcherl & Hackl GmbH, Germany) . The assembly of the cell voltage measurement consist of a PLC (AIO288, Bachmann electronic GmbH Germany), isolating amplifier (MINI MCR-SL-U-U, Phoenix Contact) and platinum wires as connection to the stack assembly unit. Mass flow meter (eFlow select, Wagner Mess- und Regeltechnik GmbH) are used for flow control. The pressure measuring occurs with a pressure sensor (DS1-010, Kalinsky Sensor Elektronik GmbH & Co.KG).

Table 9: Specification of test setup for CFY-SOC stacks at IKTS and the uncertainty based on data sheets

Physical value	Instrument	Range	Abs. deviation	Rel. deviation
Temperature	TC, Type-N, 1,5 mm, Class 1	<1250°C	±1.1°C	0.5°C/1000 h
Current	Electric load	0 …170 A	±0.5%	10 mA/1000 h
Voltage	Isolating amplifier and PLC	0 … 10 VDC	±10 mV	0.001 mV/1000 h
Flow	Mass flow meter	0 … 10 Sl/min	±0.5%	0.7%/year
Pressure	Pressure sensor	0.5 … 1.5 bara	<0.5%	2.5%/year

3. Results and Discussion

Performance tests is a good solution for right verification of stack assembly units and degradation. Figure 8 shows a performance test for different load cases over temperature. The key is to change only one parameter and reach a stable state under rated conditions. Figure 8 shows that the MK352 stack has a linear behavior inside the operation window from part load to full load. Temperature differences over the active area of a single cell increase with higher current density caused by the higher ohmic losses and therefore the cooling by lower temperature of air at the inlet is needed.

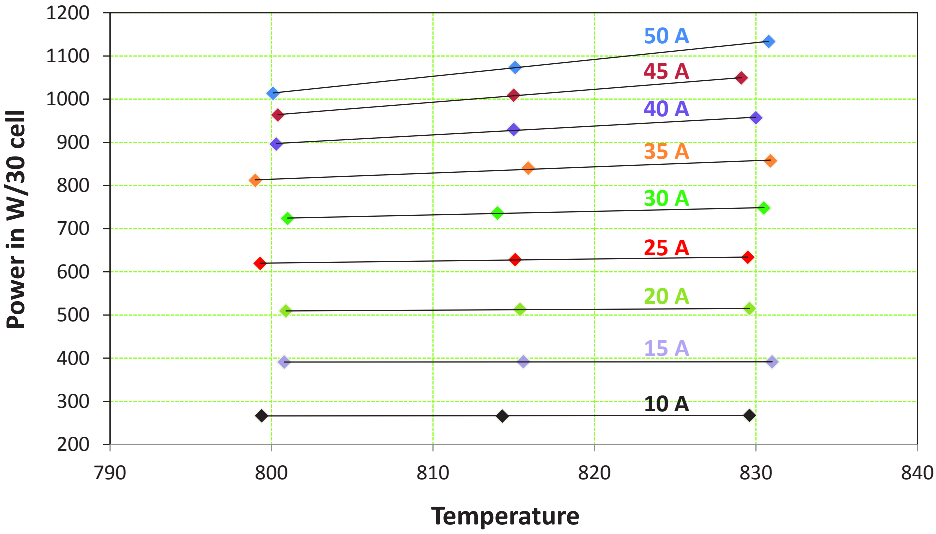

Figure 8: Performance test of a 30-cell MK352 stack assembly unit in a simulated hotbox, gas 40 % H_2 in 55 % N_2 and 5 % H_2O, η_{FU}=75 %, air 100 Nl/min, at different temperatures, charted over operation temperature ($T_{\_Ref}$)

For long term durability tests a rated power measurement at I=35 A, η_{FU}=75%, N_2/H_2=60/40, $T_{\_Ref}$=835°C was chosen from the upper part of the performance test and investigated with 10 cell-stack assembly units in hotbox relevant conditions. This rated power measurement is the initial value for the degradation rate estimation. After that, a stable operation for 1000 hours starts, with a continuous surveilling of test parameters and conditions, like current, voltage, temperature or gas quality.

A correction of the values is necessary if the deviation has been drifted for more than ±0.5 % compared to rated power measurement. Through that, a negative influence of the stack performance could be ruled. After 1000 hours the second rated power measurement with corrected parameter was determined. With both operation points the power loss and degradation can be calculated. Figure 4 shows initial power and power loss over time for similar stack assembly units operated for different periods.

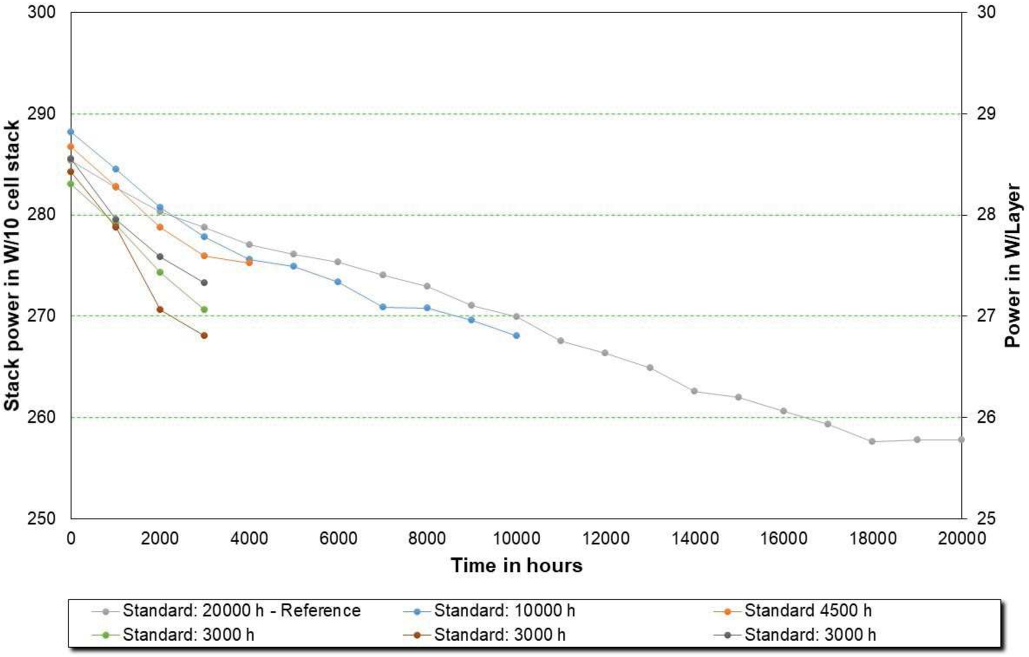

Figure 9: Long term durability test of MK352 CFY-stack assembly unit measured in rated power measurements @ I=35 A, ηFU=75%, 40% H_2 in N_2, $T_{\_air\_out}$=$T_{\_Ref}$=835°C

Starting from an average initial power of 28.5 W/cell (285 W/ 10 cell stack) a linear degradation of approx. 1.0 to 1.4 %/1000 h (@35 A, η_{FU}=75%, 40% H_2 in N_2, $T_{\_air\_out}$=$T_{\_Ref}$=835°C) was obtained. Obviously, the degradation rate of CFY stacks is higher in the first 2,000 h and will reduce with further test time. A linear degradation rate of 0.6 %/1000 h is recorded after 20,000 hours long term durability test (see curve “Standard: 20,000 h – Reference”) and shows that stack have a non-linear behavior till 3000 h. The specific temperature distribution inside an electrical furnace has an influence on the temperature distribution inside the stack assembly unit as well as the relation between heat radiation and convection over stack assembly unit outer faces.

Considering the differences of the stack power over time in Figure 9, it has to be checked which effect can be dedicated to the drift of the measuring devices. During stack tests cell voltage, current, fuel and air flow, pressure as well as temperature are recorded. Mostly less attention is paid to the instruments measured this parameter. For that reason, all instruments have been checked concerning measurement stability and drift over time. The specific deviations of the instruments, which are listed in Table 9, were calculated to the measured data's to show the impact of the average stack power of a 10 cell stack. For converting tolerances into power deviation, interpolation from rated power output measurements (Figure 3) were used. Inside the operation window of the stack the linear approach is sufficient and is exactly within the region of the measured points, i.e. rated power output 285 W/10 cell stack, comparable to 855 W/30 cell stack at 35 A and 835°C (Figure 3).

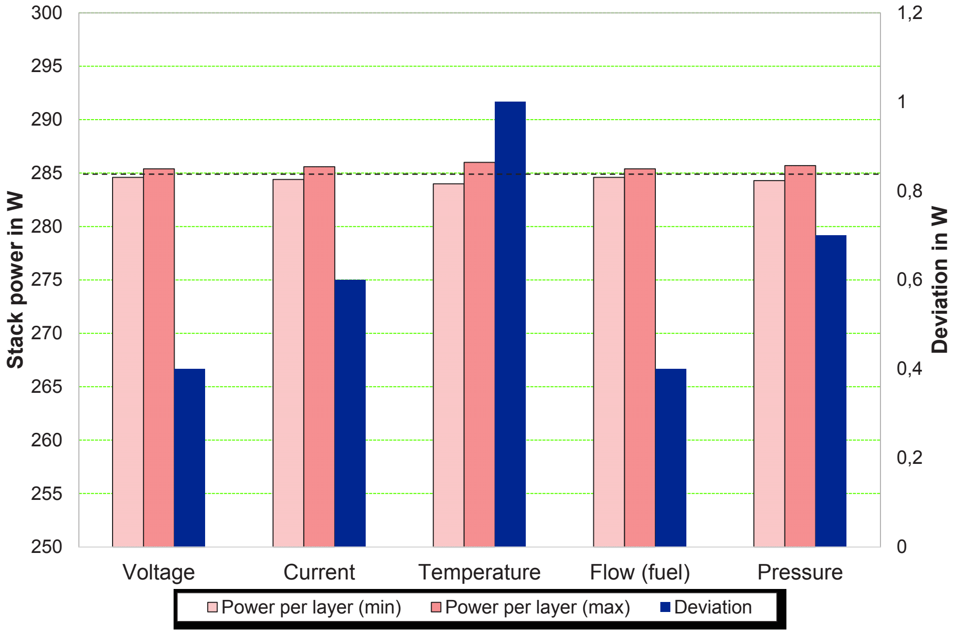

Figure 10: Considering the influence of instrument deviation to the electrical power output of a 10 cell stack at 35 A, η_{FU}=75%, $T_{\_Ref}$=835°C, 40 % H_2 in N_2, 1 bar_a, in furnace operation,

Figure 5 shows the deviations of the instruments determined from producer instructions while rated power measurements at initial state without degradation. The most important influence of the absolute power of the stack assembly unit has the temperature induced by deviations of thermocouples and their position during measurement. Especially the reference thermocouple, which defines the temperature of the stack assembly unit, must be positioned accurately and protected against radiation and environmental influences. Uncertain measurement of pressure will generate a high deviation but cannot be controlled by atmospheric operation. All absolute instrument deviations result in a power loss of a 10-cell stack assembly unit of about 3.0 W and a power difference of 1.0 %. These uncertainties must be considered by the evaluation of initial power and benchmarking of stacks.

After explaining the fluctuation of measuring signals to the initial state, the behavior while durability tests are discussed. While continuous calibration during maintenance after tests it can be reported that all values are ranging inside producer specification. Several events occur and lead to a malfunction of the device which will not be considered in this paper. The measurement of temperature will be focused for following investigations to show significance of influence to the stack power during long term durability tests.

From literature and stack tests it is known that instruments and thermocouples are drifting [14]. It could be shown that thermocouple (Type N, class 1, ∅1.5 mm) has a drift over 3000 h test time of approx. 0.5 K/1000 h, which leads to a difference of a MK352 10-cell stack assembly unit of 1.8 W (Figure 11). Based on single spiked thermocouples at rated power measurements, a drift with 1.5 K/1000 h is also illustrated and can lead to a 4.2 W higher measurement.

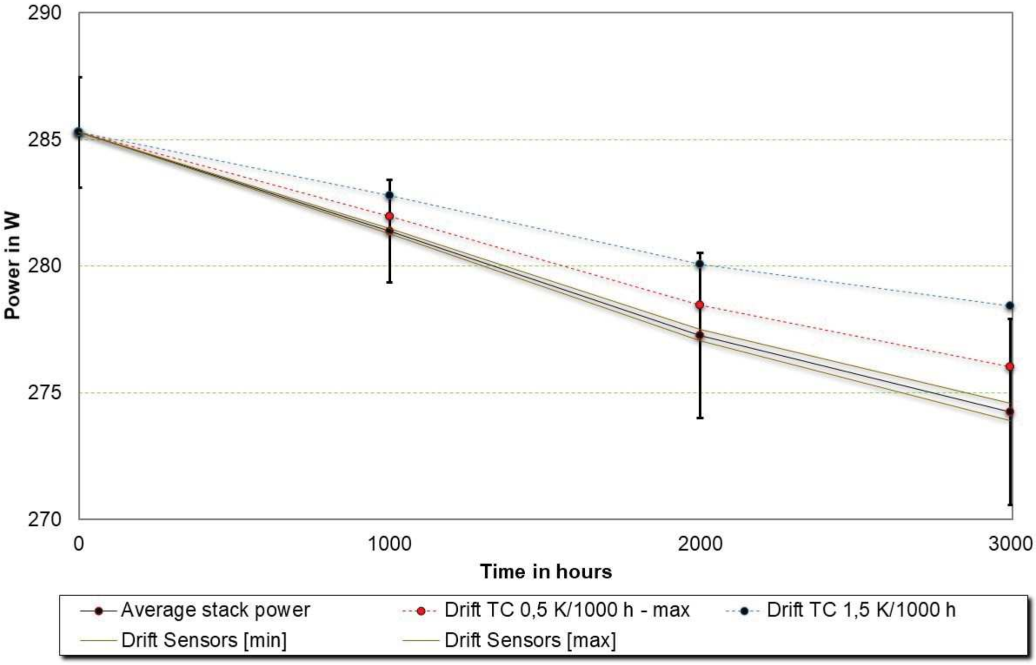

Figure 11: Average stack power and stack power during simulated drift of reference thermocouple and summarized stack power deviations of flow, pressure, current and voltage

Figure 6 shows the average stack power over time and the corrected power value by considering a temperature drift of the thermocouple. At temperature over 600°C the thermoelectric voltage of a thermocouple is decreasing over time [14], which leads to a continuous decreasing of the measured temperature. If the rated thermocouple is aging like this and a temperature correction will be executed within a rated power measurement, the stack power raises because the thermocouple will indicate a lower temperature. That will affect the calculation of the degradation because the stack assembly unit seems getting better and the power loss keeps low. The error bars in the chart show the deviations of stack power of all comparable durability tests with MK352 10-cell stack assembly units. The real measured value of 0.5 K/1000 h is over the whole time within the range, which could be one reason for power deviations but not the main reason. The theoretical curve with a drift of 1.5 K/1000 h is at the upper limit, which shows how measurements can be affected with only one instrument. The summarized drifts of remaining instruments (flow, pressure, current and voltage) with 0.1 %/1000 h show a low impact. If there is no surveilling of reference thermocouple and maintenance of other devices at test start and at the end, a valid calculation of stack performance and degradation will not be possible. Maintenance and calibration of lead instruments are essential.

For material investigations on stack level “rainbow stacks” were assembled with different changes at every repeating unit or stack layer. Caused by the different performance single units will have different conditions (mainly temperature distribution when geometry is comparable), which leads to a wrong assumption for rated power output as well as degradation rate. A precise comparison for variation of layers in one stack can only be made with comparable performance. Otherwise the specific behavior will be hidden by average stack characteristics.

The presented results show the influence of different parameters to the stack power over time in durability tests at higher load cases of the performance map. For widening the operation window the knowledge of stack assembly unit behavior, especially temperature distribution, is necessary.

The following example should show which local temperature exists inside the stack assembly unit if the current density will be increased. Figure 7 shows the temperature distribution of a middle layer of a 10-cell Mk352 stack assembly unit in a hotbox environment operated with different current densities at the same operation temperature at air outlet of $T_{\_Ref}$=835°C. Both cases have stable boundary conditions (ηFU=75%, N_2/H_2=60/40). While at current density of 275 mA/cm² (35 A) air with 60 Nl/min and 670°C are sufficient to reach the $T_{\_Ref}$=835°C a double air flow (120 Nl/min) and temperatures at air inlet of 650°C are necessary at 550 mA/cm² (70 A).

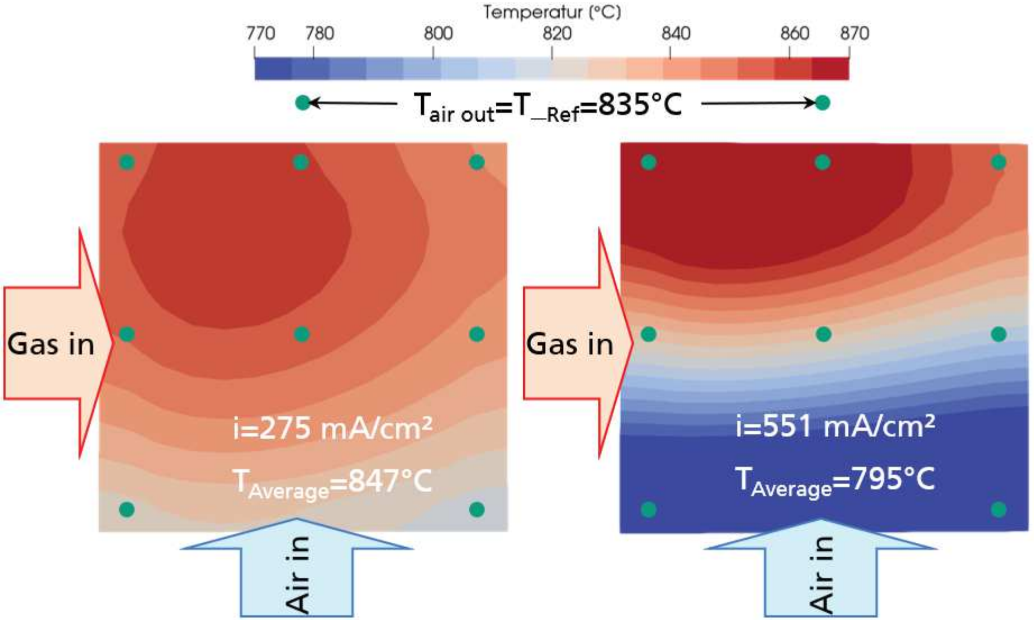

Figure 12: Simulated and measured temperature distribution over cell at 275 mA/cm² (left) and 551 mA/cm² (right) at 40 %H_2 in N_2 η_{FU}=75 %, $T_{\_Ref}$=835°C

The necessity of cooling at high current densities leads to the high temperature gradient between cathode inlet and cathode outlet: 60 K at 275 mA/cm² and 190 K at 551 mA/cm². The operation temperature at cathode outlet would raise intensely. To keep constant temperature, the air inlet preheater temperature was decreased to reach $T_{\_Ref}$=835°C at air outlet. Nearly the half of cell area at 550 mA/cm² is colder than 800°C and the average temperature is more than 50 K lower compared to the operation with 275 mA/cm². The local temperature can affect long term stability in a positive or negative way, which must be tested and simulated before it is a valid operation window of a performance map.

Compared to other stack designs with thin ferritic steel interconnects MK35x stack assembly units have a low thermal gradient over the active cell area caused mainly to the high thermal conductivity and thickness of the interconnect.

4. Conclusion and Outlook

The measurement of stack assembly units is influenced by specific stack specific as well as external test parameters like operation mode, thermal integration and accuracy of devices. MK35x stack assembly units have a low thermal gradient over the active cell area caused mainly by the high thermal conductivity of the interconnect. For rated power output evaluation and comparison of degradation rate the specific temperature profile of the stack assembly unit has to be considered. A clearly defined reference temperature at the air outlet measured with a high-quality class thermocouple and high accuracy digital computing devices provides repeatable values and guarantees a degradation rate calculation in the range of $\Delta P/P_0$=1.0 %/1000 h (@I=35 A, η_{FU}=75%, $T_{\_Ref}$=835°C). It includes a measured uncertainty of <0.5 %/1000 h mainly contributed to the temperature. For secured tests of low degradation rates, the measurement must be done under the mentioned accuracy, maintenance intervals of test equipment and operation mode with special surveillance of temperature. Otherwise falsified measurements can lead to wrong decisions in the development of components. Tests of different material combinations with different performance lead to a diverging operation and will be hidden by average conditions of the complete stack assembly unit. The examples given for MK352 stack assembly units show the actual status and open the path for reliable verification of higher power output with lower degradation rates and can help to understand degradation phenomena or surprising operation effects from other stack assembly unit concepts. Precise estimation of degradation rate is the basis for accelerated tests.

5. Acknowledgements

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