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Quality Assurance of Solid Oxide Fuel Cell (SOFC) and Electrolyser (SOEC) Stacks

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In the EU-funded project “Solid oxide cell and stack testing and quality assurance” (SOCTESQA), standardized and industry wide test modules and programs for high temperature solid oxide cells and stacks are being developed. These test procedures can be applied for the fuel cell (SOFC), the electrolysis (SOEC) and in the combined SOFC/SOEC mode. In order to optimize the test modules the project partners have tested identical SOC stacks with the same test programs in several testing campaigns. Altogether 10 pre-normative test modules were developed: Start-up, current-voltage characteristics, electrochemical impedance spectroscopy, reactant utilization, reactant gas composition, temperature sensitivity, operation at constant current, operation at varying current, thermal cycling and shut-down. The test modules were validated by comparing the results in terms of repeatability of the different testing campaigns and in terms of reproducibility among the different partners. Moreover, the results are discussed in context to the test input parameters.

Introduction

Only few documents can be found in the literature, which address the description and specification of test procedures for SOFCs and SOECs. The most relevant ones were developed in the frame of previous projects, e.g. “FCTESQA” for SOFC and the “RELHY” and “ADEL” projects for SOEC. Moreover, recently the Technical Committee 105 of the International Electrotechnical Committee (IEC) has developed a technical specification on SOFC testing (1). A survey of the existing test procedures for solid oxide cells is given in (2). However, none of these documents include detailed specifications of SOC cell/stack assembly units, e.g. for dynamic thermal cycling operation or for the combined fuel cell/electrolysis mode. Moreover established advanced characterization techniques, e.g. the electrochemical impedance spectroscopy (EIS) have not yet been integrated thoroughly in the test protocols. Additionally, several issues related to statistical data analysis and data presentation could not be resolved. Only few results were validated by round robin tests.

Therefore the EU-funded project “SOCTESQA” which started in May 2014 has the aim to develop standardized and industry wide test modules and programs for SOC cells/stacks (3, 4, 5). New application fields which are based on the operation of the SOC cell/stack assembly in the fuel cell (SOFC), in the electrolysis (SOEC) and in the combined SOFC/SOEC mode are addressed. This covers the wide range of energy conversion systems, e.g. stationary SOFC μ -CHP, mobile SOFC auxiliary power unit (APU) and SOFC/SOEC power-to-gas systems.

Altogether 18 test modules were identified in order to cover the above mentioned energy conversion systems (Table I). The most important 10 test modules (bold in Table I) were developed in the SOCTESQA project. Therefore, several test modules are connected in series to application specific test programs, which were applied by the project partners (DLR, CEA, DTU, ENEA, JRC, EIFER, NTU) on identical SOC stacks. In this project 5-cell short stacks with fuel gas electrode supported cells (ASC) were used, which were provided by an established stack supplier. For the optimization of the test modules several iterative testing campaigns have been performed. Moreover, the project addresses safety aspects, liaises with standardization organizations and establishes contact with industrial practice. As such, this collaborative project will essentially help to accelerate the development and the market penetration of hydrogen and fuel cell energy systems in Europe.

TABLE I. List of identified and developed (bold) test modules

Generic test modules (TM)			
TM01	Leakage test	TM10	Pressure sensitivity
TM02	Start-up	TM11	Mechanical load sensitivity
TM03	Current-voltage characteristics	TM12	Operation under constant current
TM04	Electrochemical impedance spectroscopy	TM13	Operation under varying current
TM05	Current interruption	TM14	Thermal cycling
TM06	Cyclic voltammetry	TM15	Redox cycling
TM07	Reactant utilisation	TM16	Shut-down
TM08	Reactant gas composition	TM17	Vibration test
TM09	Temperature sensitivity	TM18	Emergency stop

This paper compares the results of selected test modules (TM03, TM04 and TM12) in the SOFC and the SOEC mode of the different project partners. Important aspects are the evaluation of the results in terms of repeatability of the different testing campaigns and the reproducibility of the results among the partners. Electrochemical properties e.g. open circuit voltage (OCV), area specific resistance (ASR), power density, reactant utilization (FU) and impedance values of both stack and repeat units (RU) are presented. Moreover, the results are discussed in context to the test input parameters. Another important issue is the reproducibility of the different test methods, e.g. jV-characteristics and EIS-spectra. Finally, important aspects for the determination of reproducible degradation rates of SOC stacks will be presented and discussed.

Experimental

SOFC/SOEC short stacks in the light weight design were used for the validation of the test modules. The stack design is based on stamped metal sheet bipolar plates made of

ferritic steel “Crofer22APU” from ThyssenKrupp AG (6). The stacks were provided by an established industrial partner and were developed both for stationary and mobile applications, e.g. single-family household systems (μ -CHP) or auxiliary power units (APU). Fuel gas electrode supported cells (ASC) from CeramTec GmbH with an active area of about 100 cm^2 with Ni-YSZ and LSCF electrodes (7) are integrated in the stacks. Fig. 1 shows the corresponding setup of a short stack with 5 repeat units (RU) in the test station.

The interfaces between the short stack and the test station are classified in media, electric, thermal and mechanic interfaces, which are defined by different physical parameters, either of input nature (test input parameters, TIPs) or of output nature (test output parameters, TOPs). The gas inlets parameters are flow rate, composition, temperature and pressure. The gas inlet and outlet temperatures are measured with thermocouples which are located just below the stack (Fig. 1). The stack temperatures were measured with thermocouples attached at the top plate and the bottom plate of the stack. This enables to measure possible temperature gradients over the height of the stack. The voltage of each RU is monitored by a connected voltage probe. For recording of the electrochemical impedance spectra (EIS) the current probes are connected to the top and bottom plates of the stacks and the voltage probes are attached on the individual RUs of the stacks. The spectra were measured with a “Zahner, Type IM6” impedance analyzer. An AC-amplitude of 12 mA/cm^2 with a frequency range of 20 mHz to 500 kHz was applied to the stack.

Figure 1. SOFC/SOEC short stack with 5 repeat units mounted in the test station.

The stacks were operated between 720°C and 780°C with simulated reformat as fuel gas $50\%\text{H}_2+50\%\text{N}_2$ for SOFC and $80\%\text{H}_2\text{O}+20\%\text{H}_2$ for SOEC and air as oxidant gas. The gas inlet temperatures among the partners were in the range 720°C to 750°C . The current densities in the jV-curves increased up to 1 A/cm^2 . The long term operations were performed at 300 A/cm^2 and 500 A/cm^2 . The power densities (p), open circuit voltages (OCV) and area specific resistances (ASR) of the repeat units of the stack were determined from the current voltage curves. The electrochemical impedance spectra were

fitted with an equivalent circuit in order to determine the resistances of the stack components. More details of the electrochemical characterization of SOFC stacks are given in (8), (9) and (10).

Results

TM03: Current-voltage Characteristics

The results in SOFC mode show a high consistency and reproducibility among the different project partners. Moreover, the gas inlet temperatures and stack temperatures are the most influencing input parameters on stack performance. These results have been reported in a previous publication (11). Therefore, this section focuses on the quality issues of the jV-characteristics of the stacks in SOEC mode. Figure 2 shows the jV-curve with increasing current density and the measured top plate temperatures of the stacks of the different partners operated at 750°C with a fuel gas composition of 80% H_2O +20% H_2 and air.

Figure 2. Current-voltage curves of SOEC stacks at 750°C with 80 H_2O + 20% H_2 and air.

All stacks show the same open circuit voltage (OCV) of about 4.5 V which corresponds to an average OCV of the RUs of 0.90 V. This value is in good agreement with the theoretical value calculated with the Nernst equation based on a fuel gas composition of 80 H_2O + 20% H_2 . The stack voltages increase almost linearly with increasing current density. The best performance was achieved by stack#5 with an electrolysis voltage of about 6.3 V at a -700 mA/cm^2 . At this operating point the stack electrolysis power is -370 W or -880 mW/cm^2 at which a high steam conversion rate of 82 % is reached. However, especially at higher current densities differences among the stacks can be observed. Stacks #1, #2, and #3 have the same behavior whereas the performance of stack #6 is lower. At a current density of -500 mA/cm^2 the difference in

stack voltage (except stack #6) is about 0.2 V or 3.5 % relative to the mean value. This value is still below the performance quality threshold value of 5% given by the stack supplier.

The differences in stack performance can be explained by the temperatures measured at the stack and at the inlet gases. Fig. 2 shows that the top plate temperature of stack #5 is higher compared to the other stacks, which explains its better performance. Among the other stacks no clear correlation between performance and stack temperature can be observed. Therefore, in the next step the performance data were correlated to the measured fuel gas inlet temperatures. Stack #6 with the lowest fuel gas inlet temperature of 715°C exhibits the lowest electrolysis performance, whereas the higher performances of stack #1, #2, #3 and #5 are due to high fuel gas inlet temperatures of 750°C. Hence, high gas inlet temperatures result in high electrolysis performances. Therefore, accurate gas inlet temperature monitoring is obligatory for reliable and reproducible jV-characterization.

Moreover, the temperature of all stacks decrease with increasing current density. This behavior is caused by the endothermic reaction of the water electrolysis, whereas in SOFC a continuous increase in temperature is observed (11). In Fig. 2 all temperature curves show local minima at current densities around -550 mA/cm². At this point the voltage of the stacks is about 5.8 V which corresponds to 1.15 V of the RUs. According to the theory at thermo-neutral voltage the stack temperature should be the same as at OCV. Hence, the temperature minima should occur at the middle between OCV (0.9 V) and thermo-neutral voltage (1.28 V). This middle value of 1.09 V is lower than the observed 1.15 V. This can be explained by the thermal inertia of the stack which moves this point to a higher voltage during the jV measurement. A further increase of the current density higher -550 mA/cm² enhances the stack temperature by approaching the thermo-neutral voltage. In this context the possible hysteresis between increasing and decreasing jV-curves has to be mentioned. In SOFC usually the voltages with increasing current density are lower compared to the voltages with decreasing current density. This is caused by the exothermic H₂ oxidation reaction and the heat storage of the stack due to its specific heat capacity. Due to the endothermic H₂O splitting reaction in SOEC this behavior can be opposite.

In contrast to SOFC operation, almost all current voltage curves in Fig. 2 show voltage fluctuations which are caused by instable vaporization of water in the steam generator. This is a well-known issue in SOEC operation. The voltage fluctuations increase with increasing current density. Differences between the stacks can be explained by different humidification systems, e.g. controlled evaporation of water-gas stream mixtures or water bubbles, and different data logging rates. These fluctuations in stack voltage can reach maximum values of nearly 200 mV, which corresponds to maximum variations in the voltage of the repeat units of about 40 mV. During the project the voltage fluctuations could be minimized to below 10 mV for the repeat units by optimization of the humidifier operating parameters, e.g. the vaporization temperature, the water quality and the carrier gas for the steam. However, the voltage fluctuations have a strong impact on the ASR determination with the interval method. The ASRs of stacks #1, #2, and #3, which have the same performance, scatter quite strongly between 3.1 Ω cm² and 4.2 Ω cm². As an alternative it is recommended to determine the ASR by

linear or polynomial regression. In this case the ASR values of stacks #1, #2, and #3 are very close to $2.7 \Omega \text{ cm}^2$. More details of these methods are described in (12).

TM04: Electrochemical Impedance Spectroscopy

Figure 3 shows the impedance spectra of RU3 of different stacks at low DC current density of -12 mA/cm^2 , 750°C with process gases of $80\% \text{ H}_2\text{O} + 20\% \text{ H}_2$ and air. The spectra of stacks #1, #2 and #3 are quite uniform, whereas stack #4 differs from them. In the Nyquist diagram the high frequency impedances R_{HF} represent the ohmic resistances of the RUs whereas the overall impedances R_{LF} are measured at low frequencies. The ohmic resistance of stacks #1, #2 and #3 is approximately $0.20 \Omega \text{ cm}^2$ and therefore quite reproducible. Hence, the contact resistances of the cells in the stacks are very low and no detrimental isolating phases were formed during operation. This is in good agreement with results which were obtained in SOFC mode at OCV (11). In contrast to that, under electrical current load the spectra in SOEC show higher ohmic resistances compared to the spectra in SOFC. This can be easily explained by the lower temperature in SOEC due to the endothermic reaction compared to the exothermic reaction in SOFC. Stack #4 shows rather strong high frequency artefacts, which are caused by inductance interference of the connected current and voltage wires. This issue can be solved by twisting the corresponding wires to each other (13).

In contrast to SOFC operation nearly all spectra in Fig. 3 in SOEC mode show instabilities especially in the low frequency range. As mentioned above, these are caused by instabilities of the fuel gas humidification unit. In order to measure reliable and reproducible EIS spectra in SOEC mode it is therefore necessary to minimize voltage fluctuations by stabilizing the steam supply of the humidification unit as good as possible.

Figure 3. Impedance spectra of RU3 of different stacks at 750°C , -12 mA/cm^2 with $80\% \text{ H}_2\text{O} + 20\% \text{ H}_2$ and air.

Altogether three frequency dependent processes can be identified in the impedance spectra which are the steam reduction to H_2 at the fuel electrode at high frequencies, the oxidation of O^{2-} -ions to O_2 at the air electrode in the middle frequency range and the so-called gas concentration/diffusion process at the fuel electrode at low frequencies.

Because of the high flow rate usually gas concentration/transport impedances at the air electrode are negligible.

These impedances are similar to those observed in SOFC (10). In the Nyquist diagram in Figure 3 these three impedances can hardly be seen as depressed half-circles. Due to the strong overlapping it is difficult to separate the impedances of the fuel and air electrode processes from each other. The gas concentration impedance is caused by diffusive and convective gas phase transport processes in the fuel electrode support substrate and the fuel electrode itself (14, 15). This impedance strongly depends on the fuel gas composition and has a minimum at 50% steam in the fuel gas (16). Therefore, the small differences of the gas concentration impedance in Fig. 3 can be explained by small variations in the fuel gas composition supplied to the stacks. According to this the overall resistances of the repeat units at low frequencies vary between $0.46 \Omega\text{cm}^2$ and $0.56 \Omega\text{cm}^2$. These values are in good agreement with the ASR values of the jV-curves, which proves the reproducibility of both methods.

TM12: Operation under Constant Current

A very important issue for the solid oxide fuel cell technology is the reliable determination of the long term stability and the corresponding degradation rates. However, up to now no definition for the degradation rate exists. Therefore, in the SOCTESQA project following definition was determined: The absolute degradation ΔX of a quantity X (e.g. p , V , ASR...) within the time from t_1 to t_2 is calculated as the difference between the final value $X(t_2)$ and the initial value $X(t_1)$. This convention takes care that increasing values are positive and decreasing values are negative (17):

$$\Delta X = X(t_2) - X(t_1) \quad [1]$$

The relative degradation ΔX_{rel} is calculated by dividing ΔX by the initial value $X(t_1)$:

$$\Delta X_{rel} = \frac{X(t_2) - X(t_1)}{X(t_1)} \times 100\% \quad [2]$$

The absolute and relative degradation rates (rate of change) of quantity X during the time interval $(t_2 - t_1)$ is then calculated by:

$$\frac{\Delta X}{\Delta t} = \frac{\Delta X}{t_2 - t_1} \quad \text{with the unit (value/time unit)} \quad [3]$$

$$\frac{\Delta X_{rel}}{\Delta t} = \frac{\Delta X_{rel}}{t_2 - t_1} \quad \text{with the unit (\%/time unit)} \quad [4]$$

Degradation rates are typically expressed by the absolute or relative change per 1000 hours. It is thus advisable to normalize the results to 1000 h time interval.

Figure 4 shows the long term voltage behavior of two stacks operated in SOFC mode at 750°C , 300 mA/cm^2 with a fuel gas composition of $50\%\text{H}_2 + 50\%\text{N}_2$ and air. Additionally, the OCV values, which were taken from the jV-curves are plotted in the

diagram. Optically, Fig. 4 gives the impression that both stacks show nearly the same degradation rate. However, the calculated time dependent absolute degradation rates of the stack voltages are $-149 \mu\text{V/h}$ for stack #1 and $-121 \mu\text{V/h}$ for stack #2. The deviation between both stacks relative to the mean degradation rate is about 10%. In this context, it is very important that the reliable determination of degradation rates requires a very high stability and the accurate monitoring of all input parameters during long term test. Moreover, since the degradation rate is time dependent the evaluated operating time and operating duration has to be specified in the long term test.

The observed decrease in voltage at operation of the two stacks can be caused by two different mechanisms. Firstly, increase of the resistances of the RUs which are based on material and/or structural changes of the components. Secondly, the possible decrease in OCV of the RUs which is caused by decrease of the gas tightness of the sealing between fuel gas and oxidant gas compartment. Fig. 4 shows that the OCVs of both stacks slightly increase with time with values of $+43 \mu\text{V/h}$ for stack #1 and $+15 \mu\text{V/h}$ for stack #2. This behavior can be explained by the improvement of the sealing between stack and gas distribution plate (Fig.1). Since this is not a stack effect, the measured voltage degradation rates have to be corrected by the OCV increase rates. Hence, the real increase of the resistances of stack #1 are much higher than those of stack #2.

Figure 4. Long term behavior of two stacks operated in SOFC mode at 750°C , 300 mA/cm^2 with a fuel gas composition of $50\%\text{H}_2+50\%\text{N}_2$ and air.

In order to investigate the reproducibility of the different test modules jV-curves (TM03) and EIS spectra (TM04) were measured at different operating times. The evaluation of the jV-curves of stack #1 results in a stack voltage degradation rate of $-168 \mu\text{V/h}$ ($-3.9\%/1000\text{h}$) at 300 mA/cm^2 . This value is slightly higher compared to the degradation rate of Fig. 4 (TM12) because of different temperature evolution of the two methods. At constant operation (TM12) the stack temperature is slightly higher compared to the temperature of the jV-curve with increasing current. However, the increase of the

ASR values of the current voltage curves of stack #1 and stack #2 confirm the above mentioned higher resistance degradation rate of stack 1. This result was also confirmed by EIS spectra (TM04) measured at different operating times.

Summary

In the “SOCTESQA” project altogether 10 pre-normative test modules were developed and validated under SOFC, SOEC and combined SOFC/SOEC operation. These test modules were optimized in several testing campaigns. In the first step it is essential to properly define all interfaces between stack and test station. Moreover, the accurate monitoring of all test input parameters at these interfaces is obligatory for achieving reproducible results. For the quality of jV-curves (TM03), EIS spectra (TM04) and long term operation (TM12) the most influencing input parameters are the stack temperature, the gas inlet temperatures, and the stability of the fuel gas humidification unit. The first two ones have a strong influence on performance / resistances / degradation and the last one strongly influences voltage fluctuations especially in SOEC operation. Recommendations are the accurate control and monitoring of the most important temperatures and the stabilization of the steam supply of the humidification unit as good as possible.

Moreover, for jV-curves the electrical current change rate strongly influences the hysteresis between increasing and decreasing curves especially in SOFC mode. It is recommended to optimize the current change rate in terms of duration of jV-measurement and width of hysteresis. In case of voltage instabilities it is recommended to calculate the ASR not by interval but by linear or polynomial regression. For electrochemical impedance spectra (TM04) sometimes high frequency artefacts are caused by inductance interference of the connected current and voltage wires. This issue can be solved by twisting the corresponding wires to each other. For the reliable determination of degradation rates (TM12) a very high stability and the accurate monitoring of all input parameters during long term test is obligatory. Moreover, since the degradation rate is time dependent the evaluated operating time and operating duration has to be specified in the long term test.

Taking care of the above mentioned recommendations the results of the different stacks of the project partners are consistent, repeatable and reproducible. Moreover, the results of the different test methods are in good agreement which proves the reproducibility of the test modules. In the next step the results of the SOCTESQA project will be made available to the SOFC/SOEC community on the project homepage (3). Moreover, the results are already being transferred to standards developing organizations, e.g. the IEC (18). The usage of these documents will be very beneficial for the improvement of the quality assurance of the SOFC/SOEC technology, thus promoting its successful introduction and acceptance into the global energy market.

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