

# Differential Resistance Analysis – a New Tool for Evaluation of Solid Oxide Fuel Cells Degradation

Zdravko B. Stoynov<sup>1</sup>, Daria E. Vladikova<sup>1</sup>, Blagoy G. Burdin<sup>1</sup>, Jerome Laurencin<sup>2</sup>, Dario Montinaro<sup>3</sup>, Arata Nakajo<sup>4</sup>, Paolo Piccardo<sup>5</sup>, Alain Thorel<sup>6</sup>, Maxime Hubert<sup>2</sup>, Roberto Spotorno<sup>5</sup> and Anthony Chesnaud<sup>6</sup>

<sup>1</sup>*Institute of Electrochemistry and Energy Systems - BAS, 10 Acad. G. Bonchev St., Sofia 1113, Bulgaria,*

<sup>2</sup>*Universite Grenoble Alpes – CEA/LITEN, 17 rue des Martyrs, 38054, Grenoble, France*

<sup>3</sup>*SOLID power S.p.A, Viale Trento 117, 38017 Mezzolombardo, Italy*

<sup>4</sup>*Institut de Genie Mecanique, Ecole Polytechnique Federale de Lausanne, 1015 Lausanne, Switzerland*

<sup>5</sup>*University of Genoa, Department of Chemistry and Industrial Chemistry, I-16146 Genoa, Italy*

<sup>6</sup>*Centre des Matériaux, MINES-ParisTech, PSL, UMR CNRS 7633 BP 87, France*

## ABSTRACT

*Solid Oxide Fuel Cells (SOFCs) are a promising technology that can provide efficient and clean energy production. The general barriers hindering their market entry are durability, i.e. resistance to aging, and costs. In parallel to the deeper insight into the different degradation sources and improved understanding of ageing factors and their interactions, work towards higher accuracy for the assessment and monitoring of real-world fuel cell ageing is necessary. The requirements for operational stability formulate the parameter “degradation rate” (DR). Most often long term durability tests are performed at constant current load and the decrease of the voltage is used for its definition. In this work a new approach based on analysis of the volt-ampere characteristics, named Differential Resistance Analysis (DRA), is presented. It operates with the differential resistance, i.e. with the derivative of the voltage in respect to the current ( $dU/dI = R_d$ ) which is more sensitive to small deviations and thus increases the sensitivity of the analysis. Two performance indicators are derived ( $R_{d, \min}$  and  $\Delta U^*$ ) with differing selectivity:  $\Delta U^*$  is more sensitive to activation losses and  $R_{d, \min}$  - to transport hindrances. The application of the DRA is demonstrated on examples from measurements in fuel cell and in reverse (fuel cell/electrolyzer) mode, as well as on modeling data. The results show that the method is at least 10 times more sensitive to DR evaluation in comparison with the classical approach.*

Fuel Cells have an important role and hold great promise with their diverse and versatile portfolio in the transportation, energy and portable application sectors and in the integration of intermittent renewable energy sources into the overall energy system as part of a future low carbon economy. However, the scale and scope of the research and market entry agendas for their development and deployment across the spectrum of applications still meets some general barriers, summarized as durability, i.e. resistance to aging, and costs, which have to be overcome.

A challenging objective is to maintain the initial, or close to initial performance for as long as possible. However, the development of long-lasting systems remains still a goal since fuel cells degradation is influenced by the multiple factors of the operation conditions. The initial performance degrades over their lifetime due to the effect of use (electrochemical ageing), of time (calendar ageing), of different permanent and/or accidental stress conditions – thermal, current load, mechanical, conditioning, poisoning etc. Furthermore fuel cells performance and degradation tests are time consuming, e.g. several years required for extensive laboratory testing combined with field testing under actual application conditions, as well as costly.

Solid Oxide Fuel Cells (SOFCs) are a promising technology that can provide efficient and clean energy production. They have a number of advantages such as flexibility towards the type of fuel, ability to tolerate the presence of impurities, higher efficiency, application of non-noble metal catalysts. Considering the expected useful commercial maintenance-free lifetime of up to 80000 hours for stationary (combined heat and power) applications, there is a tangible need for the development of suitable accelerated life testing procedures and protocols to shorten test times, however, activating the same ageing mechanisms as in non-accelerated testing. Since SOFC durability needs sufficient improvement and reliable lifetime prediction, modeling at cell, stack and system level can serve as a powerful optimization tool. There is a sharp need for further development of sophisticated algorithms and tools for multi-scale assess of performance from cell to system level. In parallel to the deeper insight into the different degradation sources and improved understanding of ageing factors and their interactions, work towards higher accuracy for the assessment and monitoring of real-world fuel cell ageing in necessary, although this direction is often underestimated.

The requirements for operational stability formulate the parameter “degradation rate” (DR) [1, 2]. Usually it is defined as the change of a fuel cell performance indicator with time. Most often long term durability tests are performed at constant current load and the decrease of the voltage is used for definition of the DR [1]. It can be expressed as [mV kh<sup>-1</sup>], or with normalized value [% kh<sup>-1</sup>]. Obviously to calculate correctly the DR, measurements with high accuracy are needed in which a precise conditioning and high stability of the operating parameters is also necessary (temperature, current, gas flows, hydrogen humidity etc.). Currently SOFC degradation rate is about 1 % kh<sup>-1</sup>, while the near future target is 10 times less, i.e. 0.1% kh<sup>-1</sup> [1, 3, 4]. Since for the moment there are no realistic opportunities for further improvements in the measurement equipment and conditioning, new approaches for data analysis with higher sensitivity and accuracy are needed.

This work aims at presenting a new approach with increased information capability in respect to DR evaluation based on analysis of the volt-ampere characteristics, named Differential Resistance Analysis (DRA). It works with the derivative of the voltage in respect to the current, which is more sensitive to small deviations and thus ensures increased sensitivity. The method can give information about the origin of the degrading phenomena via combination with impedance measurements and numerical modeling.

The advantages of the DRA are demonstrated on examples from measurements

results show that the method is at least 10 times more sensitive to DR evaluation in comparison with the classical approach.

## Differential resistance analysis development

The most commonly applied methodology for calculation of the degradation rate from the voltage/time curves at constant current is the linearized average degradation rate, related to the change of the voltage at constant current during the full test, or divided on several segments, defining different degradation stages as initial and long term degradation. The degradation rate may be presented also as a change of the area specific resistance (ASR), i.e. as  $\text{m}\Omega\text{cm}^2\text{kh}^{-1}$  [1]. Another approach could be the calculation of the instantaneous degradation rate. However, it needs very high quality of the data, which is difficult to be realized, especially when impedance measurements are performed for deeper insight into the degradation in respect to the processes taking place in the fuel cell components. Thus experimentally linked set of performance indicators can be accumulated and applied for multiple evaluation of the degradation rate. However, they all reflect changes at a single working point of the fuel cell, most often determined by the external operating conditions and not by the internal behavior, i.e. by the state of health.

During long term durability tests periodically volt-ampere curves (VACs) are also taken. In principle they give the main fundamental and the simplest description of cells performance, ensuring an integral representation in a definite moment of the testing cycle. However the VACs behavior should be presented as a sensitive performance indicator that can be quantitatively described and evaluated.

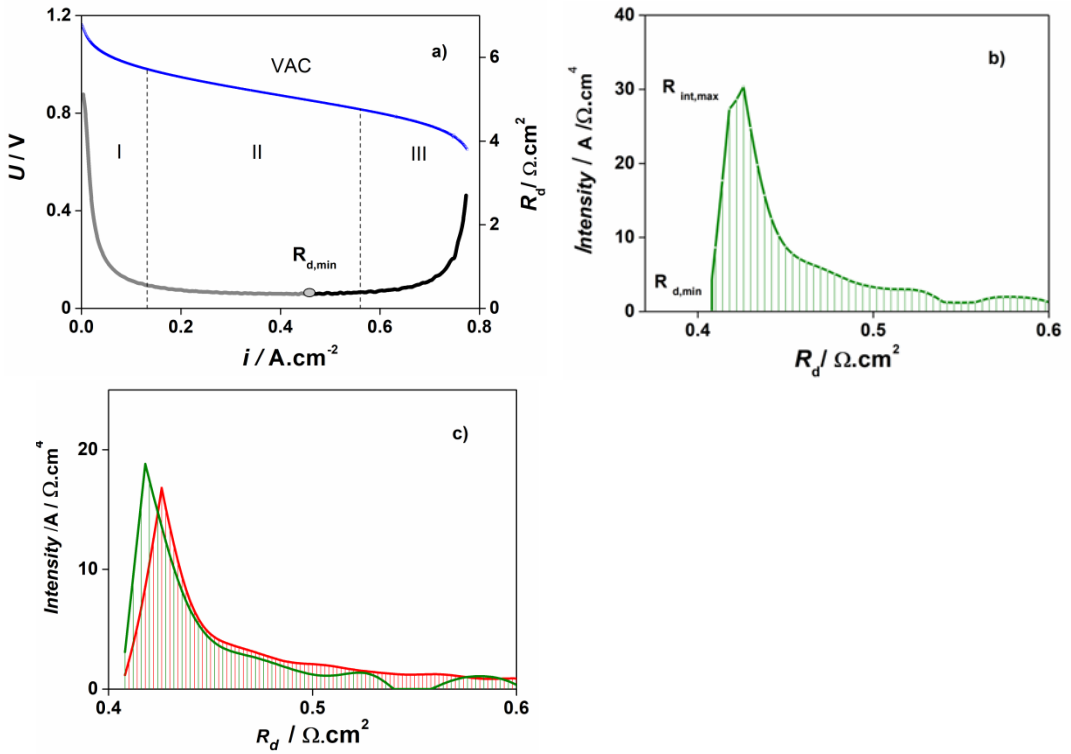
The most commonly applied parameters extracted from the VACs are: open circuit voltage  $U_0$ , maximal power density  $P_{\text{max}}$ , internal (secant) resistance  $R_i$  at  $P_{\text{max}}$  ( $R_{i, P_{\text{max}}} = U_0 - U_{P_{\text{max}}} / I_{P_{\text{max}}}$ ) and the corresponding area specific resistance  $\text{ASR}_{P_{\text{max}}}$ . However, for another working point,  $R_i$  will have another value. Its changes correlate with the VACs shape. The internal resistance can be calculated for every VAC point also as differential (tangential) resistance  $R_d$  defined as the derivative of the voltage  $U$  in respect to the corresponding current  $I$  [5]:

$$R_d = dU/dI \quad (1)$$

A detailed analysis of different VACs shows that their shape is sensitive to conditioning parameters and level of degradation. At constant operating conditions the deviations out of preliminary defined limits can serve as a measure for degradation. The periodic measurement of VACs during testing of cells and stacks life can ensure quantitative estimation of those deviations reflecting the degradation and its rate. Thus the change of VACs shape can be used as performance indicator. In this work an evaluation procedure, named Differential Resistance Analysis (DRA), is proposed. It is based on the analysis of  $R_d$  as a function of the current  $I$ . The procedure includes several steps (Fig. 1), starting with calculation of the differential resistance for every experimental point of the volt-ampere curve. The new two-dimensional data set  $D_2 [R_d, I]$  presents the DR as a function of the current. As seen in Fig. 1a, three well distinguished characteristic regions can be defined. They follow the VAC shape, reflecting the dominant types of polarization in the cell: Region I is connected with the activation losses, Region 2 concerns the transport losses and Region 3 - the gas diffusion limitations [6]. The minimum of the differential resistance  $R_{d, \text{min}}$  can be regarded as an

is determined by the intrinsic properties of the system and not by the external conditions (for instance load current).

The new functional dependence  $R_{d, \min}/I$  can be presented in the more illustrative spectral form (Fig. 1 b). The spectral peak describes Segment II, while segments I and III are introduced in the spectral tail. The spectrum has 2 characteristic points:  $R_{d, \min}$  and  $R_{int, \max}$ , which gives the most stable values of  $R_d$ , i.e. values which are observed in a wider current range. The intensity of the spectral peak is proportional to the current range with similar values of the differential resistance. The sharper the spectral line, the smaller the distribution of this parameter. The DR spectrum may have more complicated form (see Fig. 7c). Its deeper analysis is under development.



**Figure 1.** Differential Resistance Analysis: (a) VAC and  $R_d/I$  dependence ; (b) Spectral transform of the  $R_d/I$  dependence; (c) twin spectra. Data from experimental measurements of anode supported SOFC button cell at 850°C in pure hydrogen at 80% fuel utilization after 1200 hours testing [5].

The overlapping of Regions I and III in the spectral presentation can be eliminated by representing the single spectrum as “twin spectra” reflecting the behavior of the  $R_d/I$  dependence before and after  $R_{d, \min}$  (Fig. 1 c).

The DRA ensures the extraction of additional performance indicator.  $R_{d, \min}$  defines the value of the corresponding current on the VAC (Fig. 2). The tangent at this point ( $R_{d, \min}$ ) marks the voltage  $U_{00}$  at  $I = 0$  in case the system operates with constant resistance equal to  $R_{d, \min}$ . The difference  $\Delta U^* = U_0 - U_{00}$  can be introduced as a new performance indicator. Although, connected with the position of  $R_{d, \min}$  on the VAC, it is

related more closely to the non-linear losses which are dominated by the activation hindrances. Thus the shape analysis performed by the DRA derives two new performance indicators:  $R_{d, \min}$  and  $\Delta U^*$ . For degradation analysis based on VAC, a combined exploration of the indicators derived from the volt-ampere curves should be applied, since they are sensitive to different degradation processes and sources.

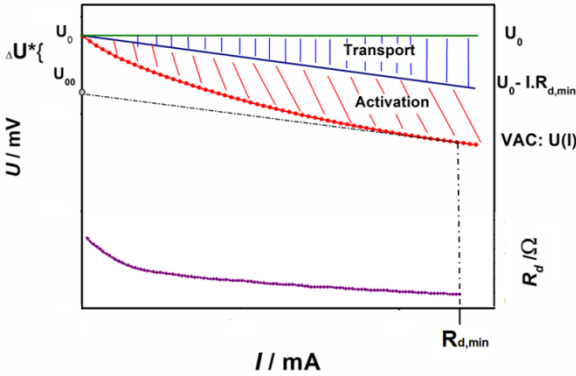


Figure 2. Schematic presentation of the procedure for definition of the performance indicator  $\Delta U^*$ .

As already marked, the working point (WP) defined by  $R_{d, \min}$  is supposed to be characteristic for the FC state of health which makes it important for impedance measurements. Fig. 3 presents impedance diagrams of SOFC button cell measured in different WPs. The diagram marked as “c” is measured close to the state corresponding to  $R_{d, \min}$ . As it can be seen, it differs in shape and represents richness of phenomena occurring at the electrodes. The negative loop is related to the water formation at the anode. The impedance analysis is out of the scope of this paper.

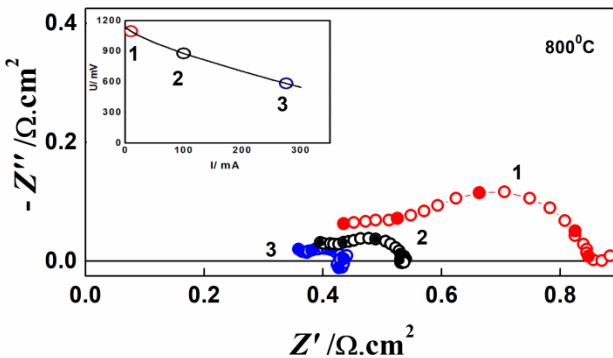


Figure 3. Impedance diagrams of SOFC button cell measured at 800°C in different WPs.

## Results and discussion

The practice of Warning Output Signals is largely used in different technical fields (machineries, buildings etc.). In fuel cells, however, the accepted up to now practice of life testing is based mainly on a simple measurement of the voltage at a constant current under constant conditioning. The produced voltage/time curves cannot give a lot of information applied for early diagnostic. The development of the Differential Resistance Analysis is a step forward in this direction. The Differential Resistance is a derivative, while the defined value of  $R_{d, \min}$  corresponds to the zero point of the  $R_d$  derivative, i.e. it can be regarded as a second derivative. As a result, the property of  $R_{d, \min}$  to change becomes observable much earlier than that of the other FC parameters. Thus the nature of this parameter, which is performance indicator, defines its ability to register early recognition of FC degradation performance and thus to support an adequate and early diagnostic. The introduction of the new performance indicators  $R_{d, \min}$  and  $\Delta U^*$  increases more than 10 times the sensitivity in respect to performance changes and degradation, which is demonstrated in several examples.

The applicability of the DRA is shown on two types of SOFC: (i) classical design of anode supported SOFC and (ii) innovative design of the so called “dual membrane fuel cell” (dmFC) [7-9].

The classical design applies: Ni/YSZ cermet as anode support; 8 mol.%  $Y_2O_3$  stabilized zirconia electrolyte; (La, Sr)(Co, Fe) $O_{3-\delta}$  (LSCF) cathode protected with 10 mol%  $Gd_2O_3$  doped- $CeO_2$  barrier layer (La, Sr) $CoO_{3-\delta}$  (LSC) current collector [10]. The technological procedure which is subject to proprietary information is developed by SOLID power. Data from button cells measured at 850°C in pure hydrogen at 80% fuel utilization on pristine sample and after 600 and 1200 hours operation at current load 0,5 A/cm<sup>2</sup> are used, as well as modeling data for long term degradation testing of the system.

The approbation of the DRA started on modeling data of the classical anode supported design, since the operation with derivatives increases the influence of the noise. Modeling ensures high quality of the “measured data”, combined with full control on the implemented degradation processes, thermal boundary conditions, operation history and reference conditions for characterization. A long term test (36000 h) of a cell in a large stack is modeled. The stack is in operation at constant power with the maximum cell temperature kept constant by manipulating the air flow. Every 1500 h VAC is produced under the same conditions. The selected degradation is based on experimentally observed alterations and the availability of a detailed calibrated model [11-19].

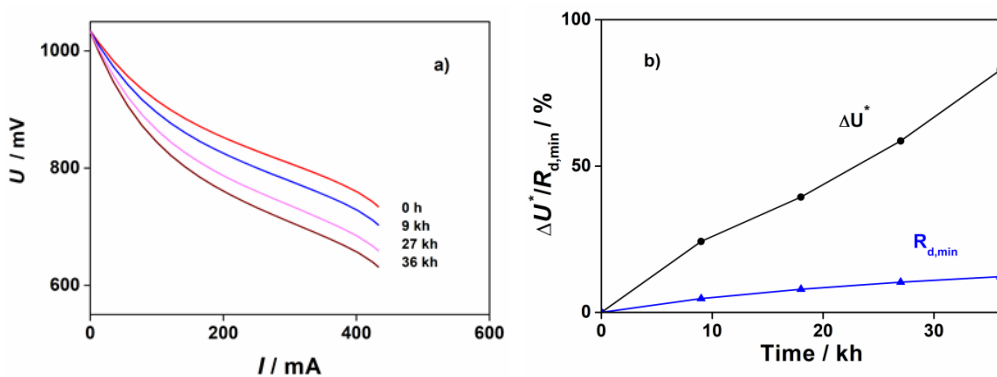
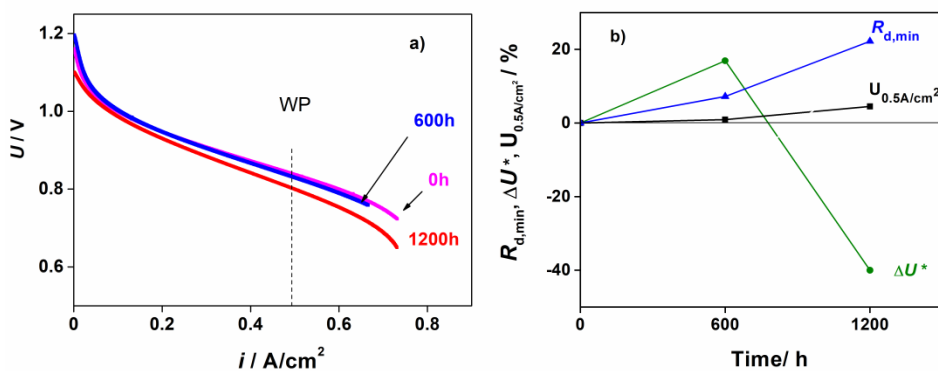
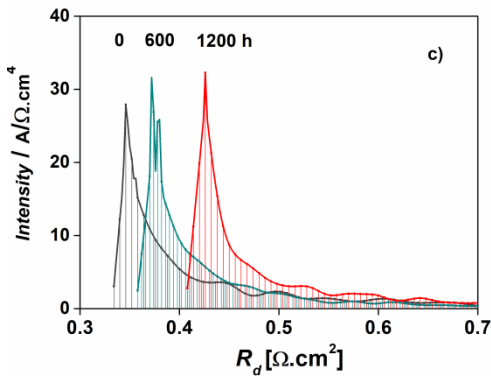


Figure 4. DRA of simulated data: (a) VACs at different operation time; (b) time dependence of  $R_{d, \min}$  and  $\Delta U^*$ .

The DRA of VACs from the 36000 h simulation is shown in Fig 4. The performed analysis is based on the change (expressed in % with accuracy  $\pm 0.4\%$ ) of the VAC performance indicators  $R_{d,\min}$  and  $\Delta U^*$  as a function of the testing time. Fig. 4a presents the development of the VACs and Fig. 4b - the corresponding DRA based on the selected indicators. The ageing reflects in increase of  $R_{d,\min}$  and  $\Delta U^*$ . It is more pronounced in  $\Delta U^*$ . The two performance indicators are connected respectively with dominating activation and transport hindrances. Thus it may be supposed that the long term performance affects both of them, however, with stronger influence of the activation losses. The smooth increase indicates similar degradation mechanism at constant operating conditions which is introduced in the model. There is not change in the regime of degradation during the simulations (i.e. the onset of acceleration is not yet reached), which is therefore correctly captured by the DRA. The presented results confirm the high sensitivity and selectivity of the developed DRA algorithm for the quantitative evaluation of SOFC degradation rate.

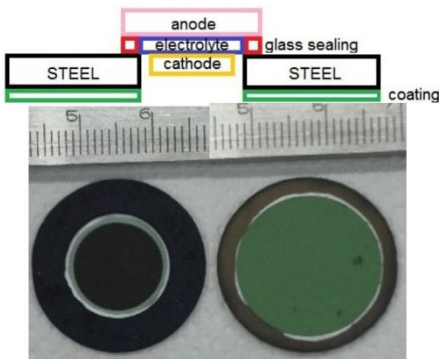
The increase of the DRA degradation assessment sensitivity is demonstrated on experimental VACs obtained on measurements of anode supported button cell with diameter 3 cm and thickness about 300  $\mu\text{m}$  at 850°C in pure hydrogen at 80% fuel utilization on pristine sample and after 600 and 1200 hours operation at current load 0,5  $\text{A}/\text{cm}^2$  (Fig. 5). As seen in Fig. 5a, there is no distinguishable difference in the VACs measured at 0 and at 600 hours, while the new performance indicator  $R_{d,\min}$  changes respectively 7 and 17% (Fig. 5b, c). Above 600 hours  $R_d$  continues to increase, whereas  $\Delta U^*$  decreases which marks some changes in the degradation mechanism connected with decrease of the activation losses influence in the total degradation. More detailed information can be obtained applying impedance measurements. For comparison the classical approach for calculation of the degradation rate gives a change of  $U$  at the measured WP ( $I = 0.5 \text{ A}/\text{cm}^2$ ) about 1% for 1200 hours, while the change of  $R_{d,\min}$  is 27% (Fig. 5b).





**Figure 5.** DRA of button cell tested 1200 hours: a) VACs at 0; 600 and 1200 h b) time dependence of  $R_{d, \min}$ ,  $\Delta U^*$  and  $U$  at  $I = 0.5 \text{ A/cm}^2$ ; c) corresponding DRA spectra.

Usually button cells are used for testing the electroactive part of the fuel cell (cathode/electrolyte/anode). For laboratory testing of a stack layer with all active and non-active elements, a special button-type cell (E-cell) was developed (Fig.6) [20]. The presence of the interconnect metal on which the cell is mounted using a glass-ceramic sealant introduces additional variables suitable to study the materials interaction and the real response to changes in the operating parameters.

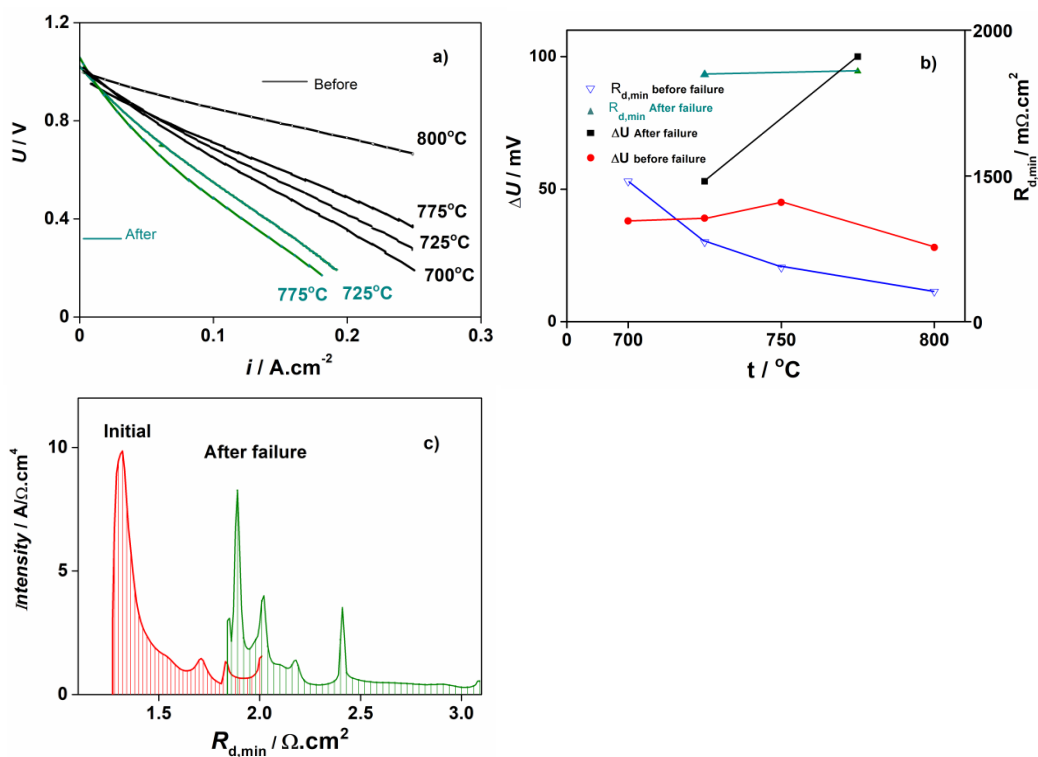


**Figure 6.** E-cell: design (top); cathode side (left); anode side (right).

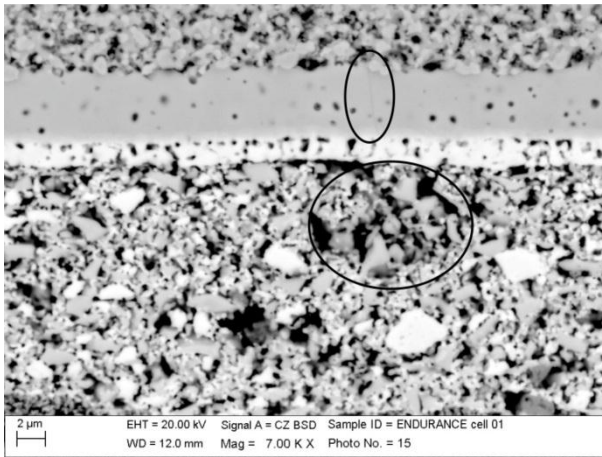
The E-cell is used for testing of classical anode supported button cells produced according to SOLID power technology which are mounted on a metal frame coated with cobalt manganese spinel on the side corresponding to the cathode of the cell [20]. A ferritic stainless steel of the family AISI441 commercial grade is used. The sealant is a commercial barium silicate glass-ceramic material in use for real stack manufacturing. It is applied to join the dense electrolyte with the metal frame on the bare side in order to separate the anode compartment from the cathode compartment. The testing is performed at  $750^\circ\text{C}$  for 100 hours under a current load of  $150 \text{ mA/cm}^2$ . Before and after the testing



maximum thermal gradient experienced in a stack. Fig.7a shows VACs before the testing and after an accidental testing during which the system has been in conditions of air starvation in the last 20 hours of the testing. It is interesting to note that after the failure the VACs taken at 725°C and 775°C change their position – the one measured at higher temperature goes below the lower temperature curve. The corresponding DRA (Fig. 7b) represents the curves quantitatively with the help of the performance indicators  $R_{d, \min}$  and  $\Delta U^*$ . At normal operation both parameters decrease with the increase of the temperature. Air starvation causes drastic increase of the two performance indicators. However, a change of their temperature behavior is observed:  $\Delta U^*$  sharply increases with the temperature, while  $R_{d, \min}$  keeps similar values. The combined qualitative and quantitative analysis based on the new VAC performance indicators elucidates the observed irreversible strong degradation which brings to sharp increase of  $R_{d, \min}$ . The activation polarization which influences stronger  $\Delta U^*$  increases with the temperature. This explains the position of the two measured VACs after the failure. Post-experimental studies demonstrate the formation of micro-cracks in the electrolyte leading to severe cathode degradation (Fig. 8) [20]. Modifications of the cathode at the interface LSCF-composite (LSCF-GDC) in the sealant at both interfaces respectively with the electrolyte and with the metal frame, as well as in the cobalt manganese spinel coating at the air side of the metal frame are also observed [20].



**Figure 7.** E-cell testing at different temperatures before and after the cell failure: a) VACs; b)  $R_{d, \min}$  and  $\Delta U^*$  changes; c) comparison of  $R_d$  spectra.



**Figure 8.** SEM-BSE image of the electrolyte-cathode interface of E-cell with circled microcrack and area affected by GDC decomposition.

The innovative dmFC design is under development for operation in a reversible mode [9, 21]. The new architecture eliminates the problems coming from the water formation and evacuation from the electrodes, introducing a separate water chamber [7-9]. The assembly combines the cathode compartment (cathode/electrolyte) of a SOFC and the anode compartment (anode/electrolyte) of pSOFC sandwiching a porous central membrane (CM) with mixed proton and oxide ion conducting ceramic structure, where water is produced and evacuated. This leads to numerous advantages (lower electrodes overpotential, easy pressurization, no dilution of fuel or oxidant). The separation of the water in a special compartment where it is formed (in FCFC mode), or injected (in SOEC mode), and thus the elimination of its damaging presence at the electrodes in both modes, combined with the reduced operating temperature from 800-950°C to 600-800°C, are strong advantages for the development of this concept and the application of the DRA for sensitive evaluation of the cell performance in reversible operation. The experiments were performed on button cells with Pt electrodes, thickness of the CM of about 200 μm and total thickness of the electrolytes and central membrane assembly (ECMA) about 1 mm.

The DRA of VACs measured on dual membrane fuel cell operating in reverse mode demonstrate in a very illustrative way the improved performance of the system in electrolyzer mode (Fig. 9). For the whole temperature range  $R_d$  is lower in electrolyzer mode. This behavior is not typical for the classical anode supported fuel cells. Since the volt-ampere curves have linear shape, the analysis is based on the change of  $R_{d, \min}$  as performance indicator (Fig. 9b). Logically it decreases with the temperature in both fuel cell and electrolyzer mode, with sharper temperature behavior in operation as fuel cell. The comparison of  $R_d$  spectra presented in Fig. 9c shows different shape which obviously reflects changes in the performance mechanisms and/or behaviour. The shape analysis of the spectra needs further studies.

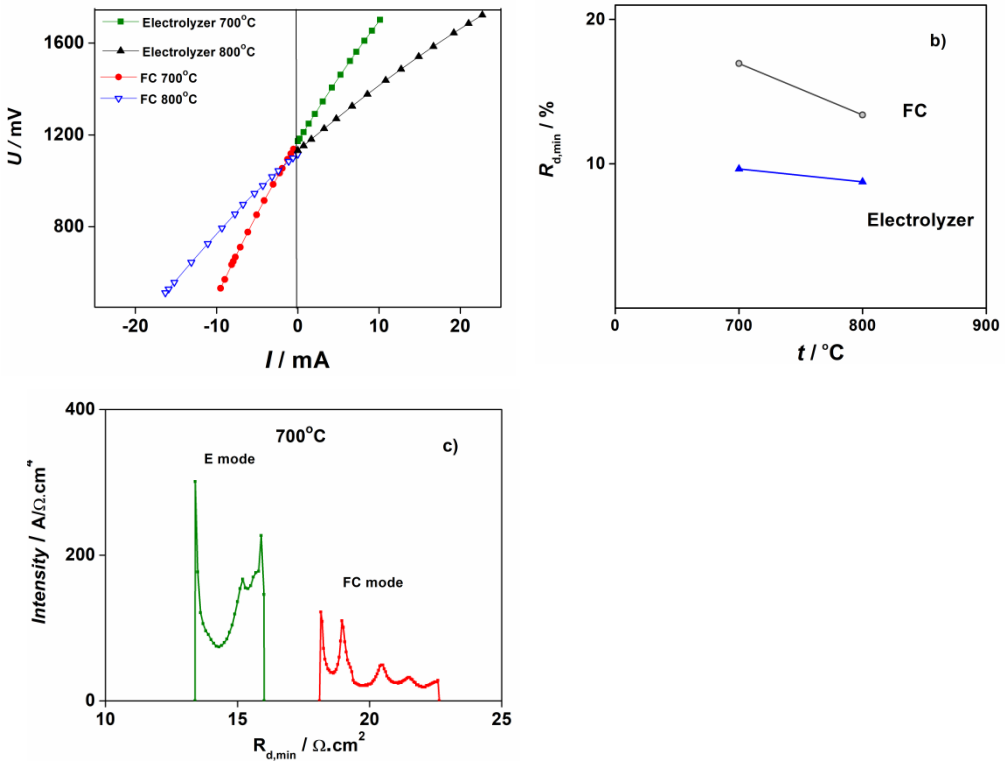


Figure 9. DRA of dmFC in fuel cell and in electrolyzer mode: a) VACs; b)  $R_{d,min}$  temperature dependence; c)  $R_d$  spectra.

## Conclusions

The DRA based on the measured VACs is useful tool with increased sensibility towards degradation, since it works with the derivatives of the measured parameters, which are in principle more sensitive to small deviations. It gives an opportunity for collection of reliable data from shorter tests avoiding for accelerating test conditions. The operation with derivatives, however, increases the influence of the noise. Thus the analysis needs higher quality of the measurements and reasonable volume of data. For one VAC 30-40 points are sufficient, but they should be measured after stabilization of the operating conditions (about 20-40 seconds). Special recommendations can be found in [22].

During durability testing the appearance of sharp deviations from the smooth time-dependence of the DRA indicators can serve as early warning signal for increased degradation. Every indicator has higher selectivity in respect to different degradation source ( $\Delta U^*$  is more sensitive to activation losses and  $R_{d,min}$  - to transport hindrances). In addition, impedance measurements can give more precise information about the origin of the degradation (ohmic losses, electrodes polarization etc.).

Although developed for SOFC degradation studies, the Differential Resistance Analysis can be applied for every system which is characterized with volt-ampere curves.

As every new diagnostic tool, the DRA needs further improvements and more practice for its introduction into the durability evaluation toolset.

## Acknowledgment

The research has received funding from FP7/2007-2013 Fuel Cells and Hydrogen Joint Undertaking (FCU-JU-2013-1) under grant agreement No 621207 and the Bulgarian National Science Fund under grant agreement E02/3/12.12.2014.

## REFERENCES

1. J. Hjelm in *Degradation testing – Quantification and Interpretation* (2nd International Workshop on degradation Issues of Fuel Cells, Thessaloniki, GR, 2011).
2. L.G.J. De Haart, J. Mougín, O. Posdziech, J. Kiviahó and N.H. Menzler, *Fuel Cells* 9 (6) 794–804 (2009).
3. R.S. Gemmen, M.C. Williams and K.Gerdes, *J. Power Sources* 184(1), 251-259 (2008).
4. A. Hagen, R. Barfod, P. V. Hendriksen, Y.-L. Liu and S. Ramousse, *J. Electrochem. Soc.* 153(6), A1165-A1171(2006).
5. Z. Stoynov, D. Vladikova and B. Burdin, *Bulg. Chem. Comm.No, XXX-OOO* (2017).
6. E.Ivers-Taffee and A. V. Virkar in *Electrode Polarizations: High-temperature Solid Oxide Fuel Cells: Fundamentals, Design and Applications 1st Edition*, edited by S.C. Singhal and K. Kendal, Elsevier, New York (2003).
7. A. Thorel, Z. Stoynov, D. Vladikova, A. Chesnaud, M. Viviani and S. Presto Patent No 20120156573 (21 June 2012).
8. D. Vladikova, Z. Stoynov, G. Raikova, A. Thorel, A. Chesnaud, J. Abreu, M. Viviani, A. Barbucci, S. Presto and P. Carpanese, *Electrochim. Acta* 56, 7955-7962 (2011).
9. D. Vladikova, Z. Stoynov, A. Chesnaud, A. Thorel, M. Vivianu, A. Barbucci, G. Raikova, P. Carpanese, M. Krapchanska and E. Mladenova, *Int. J. Hydrogen Energy*, 39(36), 21561-21568 (2014).
10. A.R. Contino, A. Dellai and M. Rolland, *Int. J. Hydrogen Energy* 39(36), 21638-21646 (2014).
11. A. Nakajo, A.P. Cocco, M.B. DeGostin, A.A. Peracchio, B.N. Cassenti, M. Cantoni, J. Van herle and W.K.S. Chiu, *J. Power Sources* 325, 786-800 (2016).
12. J. Laurencin, M. Hubert, D. Ferreira Sanchez, S. Pylypko, M. Morales, A. Morata, B. Morel, D. Montinaro, F. Lefebvre-Joud and E. Siebert, *Electrochimica acta* 241, 459-476 (2017).
13. M. Morales, V. Miguel-Pérez, A. Tarancón, A. Slodczyk, M. Torrell, B. Ballesteros, J.P. Ouweltjes, J.M. Bassat, D. Montinaro and A. Morata, *J. Power Sources* 344, 141–151 (2017).
14. J. Laurencin, M. Hubert, K. Couturier, T. Le Bihan, P. Cloetens, F. Lefebvre-Joud and E. Siebert, *Electrochimica Acta* 174, 1299-1316 (2015).
15. A. Nakajo, A.M. Kiss, A.P. Cocco, W.M. Harris, M.B. DeGostin, F. Greco, G.J. Nelson, A.A. Peracchio, B.N. Cassenti, A. Deriy, S. Wang, Y.-C.K. Chen-Wiegart, J. Wang, J. Van herle and W.K.S. Chiu, *ECS Transactions* 68 (1), 1069-1081 (2015).

16. F. Greco, A. Nakajo, Z. Wuillemín and J. Van herle, ECS Transactions 68 (1), 1921-1931 (2015).
17. V. Miguel-Pérez, J. P. Ouweltjes, A. Tarancón, D. Montinaro and A. Morata, ECS Transactions 68, 1803-1813 (2015).
18. A. Bertei, G. Arcolini, C. Nicolella and P. Piccardo, ECS Transactions, 68 (1), 2897-2905 (2015).
19. M. Hubert, J. Laurencin, P. Cloetens, J.C. Da Silva, F. Lefebvre-Joud, P. Bleuet, A. Nakajo and E. Siebert, Solid State Ionics 294, 90-107 (2016).
20. P. Piccardo, R. Spotorno, J.P. Ouweltjes, Z. Stoyanov and D. Vladikova, ECS Transactions 78(1), 2087-2098 (2017).
21. D. Vladikova, Z. Stoyanov, B. Burdin, G. Raikova, M. Krapchanska, A. Thorel, A. Chesnaud, Internat. journal for science, techniques and innovations for the industry 11 (4) 190-193 (2017)
22. Handbook of Test Procedures and Protocols (2017). Available at: <http://www.durablepower.eu/index.php/handbook> (accessed 29 September 2017).