

Testing of Electrodes, Cells, and Short Stacks

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Abstract The present contribution describes the electrochemical testing and characterization of electrodes, cells, and short stacks. To achieve the maximum insight and results from testing of electrodes and cells, it is obviously necessary to have a good understanding of the fundamental principles of electrochemistry, but it also requires proper test geometries and set up, well-chosen operating conditions for different test purposes, correct probing of voltages and temperatures, and solid knowledge on benefits and drawbacks of different characterization techniques to obtain reliable, accurate, and reproducible electrochemical measurements, and this will be the focus of this chapter. First, the important issue of understanding potential differences and measurements of potentials, which is linked to the choice of proper electrode geometries and test set up configurations for electrode and cell testing, is presented. Then probing of voltages and temperatures, choice of sealing and contacting, as well as considerations regarding the choice of operating conditions for different purposes mainly for single cell testing are outlined. Having considered optimization of test set up, geometries, and the selection of optimal operating conditions, the details of measurement of the electrochemical performance of the electrode, cell, or stack are explained. As part of this, the concept of area specific resistance (ASR) and how DC and AC methods can be used and optimized to provide not only the total ASR, but also the electrochemical characterization of specific parts (electrolyte, each electrode) in a full cell are described. Some experimental results are provided including illustrative examples of breakdown of losses in full cells and determination of their temperature and gas composition dependencies, and finally, challenging issues, such as the effects of impurities and the problem of leakage in cell testing, are discussed as well.

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1 Introduction

This chapter describes the electrochemical testing and characterization of electrodes and cells and provides a brief discussion of short stack testing as well. To achieve the maximum obtainable knowledge from testing of electrodes and cells, it is obviously necessary to have a good understanding of the fundamental principles of electrochemistry (Bockris 1970; Holze 2007; Hamann 1998; Greef 1985) and especially solid-state electrochemistry (Kharton 2009, 2011). Furthermore, a good and sound basic knowledge in materials science, especially in high-temperature materials (Ibach 2009; Smart and More 1996; Carter 2013), will inevitably increase the understanding of the results from electrode and single cell solid oxide cell (SOC) tests.

There are good reasons to pursue all three levels of tests: electrodes, cells, and short stacks. Testing of electrodes can provide detailed insight into the electrochemical performance and its correlation with parameters such as gas composition, exact chemical composition of electrode materials, and the effect of specific micro- or nano-structures on the electrochemical performance and durability; all electrode-specific information as elegantly shown in literature (Ramos et al. 2010; Nielsen et al. 2011; Kromp et al. 2012; Shearing et al. 2010a, b; Utz et al. 2011; Vels Jensen et al. 2001). Testing of full cells complicates the setup, test procedures, and analyses of the results, but on the other hand, testing of full cells provides insight into the effect of parameters such as high current density, i.e., overpotential gradients and current density gradient along the fuel flow direction due to fuel utilization as shown many times in recent years (Rasmussen and Hagen 2010; Nielsen et al. 2010; Hauch et al. 2014; Tietz et al. 2013). Testing of short stacks, sometimes named single repeating units (SRU), includes interconnects for current collection. Such test enables studies of, e.g., effects of corrosion of interconnect material, poisoning electrodes due to interconnects, and contacting issues. Detailed probing of voltages, gas compositions, temperature, and impedance measurements can still be obtained for SRU. If stacks are to be tested as a part of development of a commercial product, it is desirable to test a complete system including balance-of-plant components. Such tests are expensive and complex, and it is difficult to obtain detailed electrochemical information from such test and to interpret the results in detail. Stack testing will not be treated further in this chapter on electrochemical testing of SOC; however, recent work elegantly shows the possibilities of characterization of individual single cells in a stack (Mosbaek et al. 2013).

Thousands of reports can be found in literature on electrochemical testing of SOC electrodes, cells, and short stacks. As there is no general agreement on test protocols even inside regions such as USA and/or EU, it is particularly important that the testers carefully report the obtained fundamental parameters, such as area specific resistance (ASR), activation energy (E_a), and polarization losses, and provide the exact test conditions for the study to minimize misleading interpretations and inappropriate comparison of electrochemical test results.

This chapter will focus on the proper testing of these electrodes and cells—specifically the use and position of reference electrodes (Sect. 2), test set up and proper probing for cell tests and choice of test conditions and specific test sequences and conditions (Sect. 3). This section also describes the issue of impurities in gasses and raw materials which often complicates the interpretation of the results from electrochemical testing. As the majority of testing of electrodes and cells is performed to obtain a measure for the performance of the cell, e.g., given as the ASR of the cell, the concept of ASR, and a thorough description of its calculation is given here (Sect. 4) together with a review of key characterization methods for electrodes and cells (Sect. 5). This provides a more practical and test-related description of measurements to obtain results on contributions to the ohmic and polarization losses compared to descriptions found in literature (Boukamp 2004; Macdonald and Barsoukov 2005; Orazem 2008). Section 5 also provides examples of complementary non-electrochemical characterization techniques for SOC and finally describes the issue of gas leakage in cell testing (Sect. 6).

2 Electrodes

2.1 *Measurement of Potentials and Understanding of Potential Gradients*

The main problem with characterizing the individual electrodes in single cells and short stacks is the insertion of the reference electrodes, which are used to judge the performance of the individual components and interfaces in the cell. Since a single cell stack is made up of five components, an electrolyte, two electrodes, and two interconnects, there are four interfaces at which reference electrodes can be inserted. Unfortunately, such reference electrodes will not work in the geometries, which are normally employed.

Results of tests on cells with thin electrolyte layers using one or more reference electrodes have been reported on many occasions (Gödicke-meier et al. 1995; Erning et al. 1995; van Heuveln et al. 1993; Kleinlogel et al. 1997), but the electrodes under investigation appeared not to be polarized; the short explanation is that the reference electrodes were not working. The geometric requirements for the position of electrodes in three-electrode set-ups have been treated in detail by several researchers (Nagata et al. 1994; Jacobsen and Skou 1997; Winkler et al. 1998; Reinhardt and Göpel 1998; Adler 2002; Mogensen 2002), and there is a general agreement among these researchers. In spite of this, there still seems to be a great need in the SOC community for basic information on how to measure electrode potentials properly; some of these details are given below.

Figures 1 and 2 illustrate the problem. An electrode-supported cell with a “reference” electrode is often sketched as shown in Fig. 1a. However, such a sketch is very deceiving when it is used for an assessment of the current distribution. For this purpose, the sketch should be drawn to scale, i.e., the electrolyte thickness

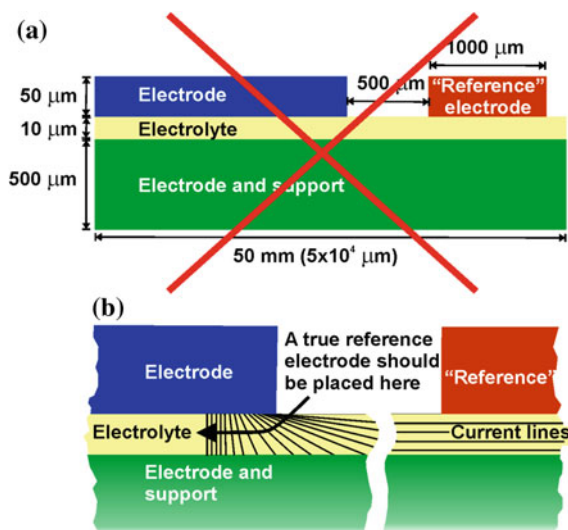


Fig. 1 **a** A typical sketch of an electrode-supported cell which is out of scale, because the gap between the top electrodes should be 50 times greater than the electrolyte thickness and **b** expanded view of electrode corners showing the current distribution indicated by schematic current lines. The current density, apart from being approximately parallel to the electrolyte plane, is very small at the position of the “reference” electrode, at least 50 ($500 \mu\text{m}/10 \mu\text{m}$) times smaller than the current density of the cell.

should be the relevant unit of length. When the correct length scale is used, as in Fig. 1b, it is evident that the gap between the upper working electrode and the “reference” electrode is huge. This means that the current distribution around the right-hand edge of the working electrode becomes very different from the even current distribution in the main part of the cell. Furthermore, the current in the vicinity of the “reference” electrode becomes parallel to the electrolyte plane, i.e., there is only a minute voltage difference across the electrolyte at the “reference” electrode position. The correct position of the reference electrode would be inside the electrolyte of the cell and some distance away from the corner, but this is difficult in a 10- μm -thick electrolyte.

Figure 2a illustrates the electrostatic potential across a cell at open circuit voltage (OCV) condition. Note that for a good electrolyte (ionic conduction only), there will be no potential gradient inside the electrolyte at zero current. The whole potential change across the cell is localized at the interfaces between the electrolyte and the electrodes. These regions are the so-called electrochemical double layers with thickness in the nano-meter range and with high space charge concentrations as a result of the very high potential gradients. Figure 2b and c show the potential across a cell when it is loaded, i.e., a current flows through it, in fuel cell, and electrolysis mode, respectively. Note that there is a potential loss across the electrolyte due to the electrolyte resistance. The potential steps at the interfaces are now smaller compared to the OCV condition in the fuel cell case and larger in the electrolysis case due to the

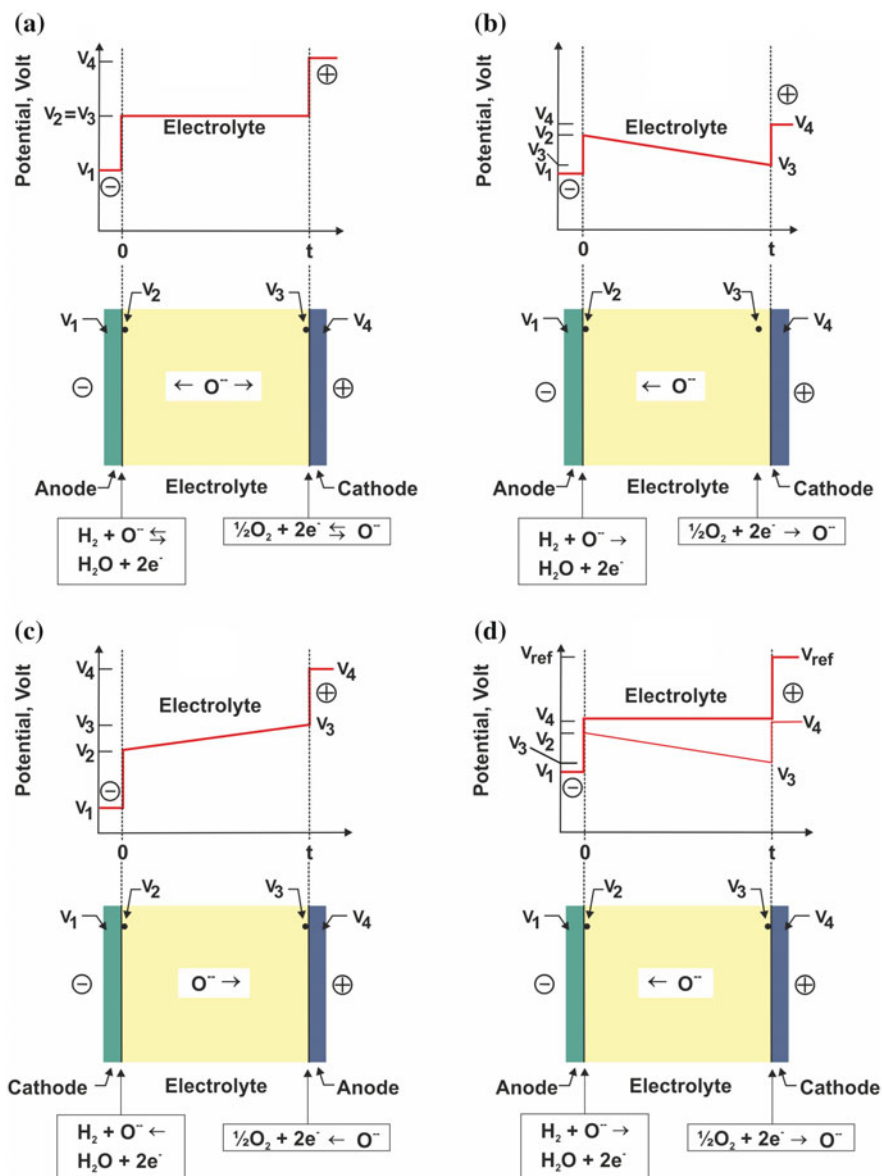


Fig. 2 Electrostatic potential through the electrode-supported cell with **a** no current, **b** current load in fuel cell mode, **c** current load in electrolysis mode, and **d** the potential across the electrolyte at the “reference” electrode position (*thick line*) and through the cell part with the current load (*thin line*). It is seen that: $(V_{\text{ref}} - V_4)/i = R_{\text{p,anode}} + R_{\text{p,cathode}} + R_{\text{elyt}}$ = the total polarization resistance of the cell apart from concentration polarization resistance. Note that the electrostatic potential single value cannot be measured with a voltmeter, but voltage (= potential difference between two points) can

losses originating from the polarization resistance of the electrode processes. Figure 2d illustrates the case of a fuel electrode—supported cell with an attempt of placing a reference electrode on the electrolyte surface next to the oxygen electrode. The potential is given for both the position of the “reference” electrode where no current flows across the electrolyte (i.e., the electrical potential is constant across the electrolyte) and a position far away from the electrode edges as in Fig. 2b. As the potential along the fuel electrode, which is a very good electronic conductor, is the same everywhere (the electrode constitutes an iso-potential plane), the potential of the fuel electrode at the “reference” electrode must be equal to the potential in the middle of the current bearing part of the fuel electrode. Thus, the two potential curves in Fig. 2d must start at the same point. Therefore, as seen in Fig. 2d, the potential difference between the “reference” electrode and the upper electrode in Fig. 1 is simply the total polarization of the full cell. Thus, it is clear that the “reference” electrode measures only the Emf of the cell with the actual gas compositions at the “reference” and inside the support at the lateral position opposite to the reference while the current is flowing. Thus, no information on what happens on any of the working electrodes can be derived from such measurements. If the concentrations of the reactants and products at the lateral position of the reference electrode were the same as in the active electrode/electrolyte interfaces, then it would be possible to deduce the total concentration overpotential. This is, however, in general not the case. This means that the voltage difference actually measured between the working and the “reference” electrodes cannot be assigned any clear meaning. In the present context, it is also helpful to remember that a single electrode potential cannot be measured directly; it is only possible to measure a potential difference between two electrodes. Furthermore, it may be useful to know that the Fermi potential of the electrons—also of mobile electrons inside an electrolyte—is not at all the same as the electrostatic potentials. The gradient of the Fermi potential will even have the opposite sign of the electrostatic potential gradient across the electrolyte in fuel cell mode (Jacobsen and Mogensen 2008; Mogensen and Jacobsen 2009). The various types of potentials relevant to an electrochemical cell are illustrated in Fig. 3.

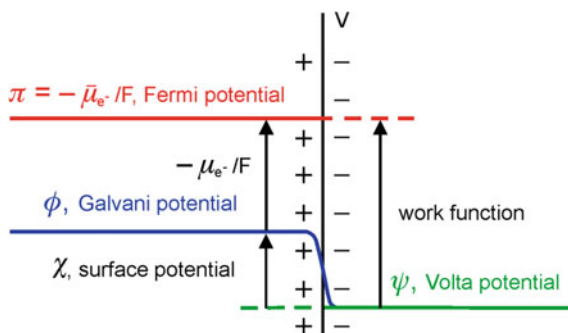


Fig. 3 Potentials in a solid conductor relative to the outer electric potential in vacuum. $\bar{\mu}_{e^-}$ is the electrochemical potential of the electrons and F is the Faraday’s number. The Galvani potential is the same as the electrostatic potential, and the Fermi potential, π , is also called the electromotive potential (Jacobsen et al. 2014)

Figure 4 shows the course of the Fermi potential (π) and the electrostatic potential (ϕ) across the electrolyte in a YSZ-based SOC for the cases of OCV, fuel cell mode (SOFC), and electrolysis mode (SOEC). It is worth noting that the S-shaped π -curve moves to the right when polarized in fuel cell mode and to the left in electrolysis mode. The ϕ -curves are linear, reflecting the simple ohmic loss in the electrolyte, and change the sign in its slope when going from fuel cell mode through OCV to electrolysis mode. The Fermi potential (π) and the electrostatic potential (ϕ) determine together the oxygen potential inside the electrolyte. If the oxygen potential is expressed as oxygen partial pressure (p_{O_2}), then in case of electrolyte materials with constant stoichiometry like YSZ, the electrode equilibrium potential, $(\pi - \phi)$, is related to p_{O_2} as:

$$(\pi - \phi) = (\pi - \phi)^\ominus + \frac{RT}{4F} \ln \frac{p_{O_2}}{p^\ominus} \quad (1)$$

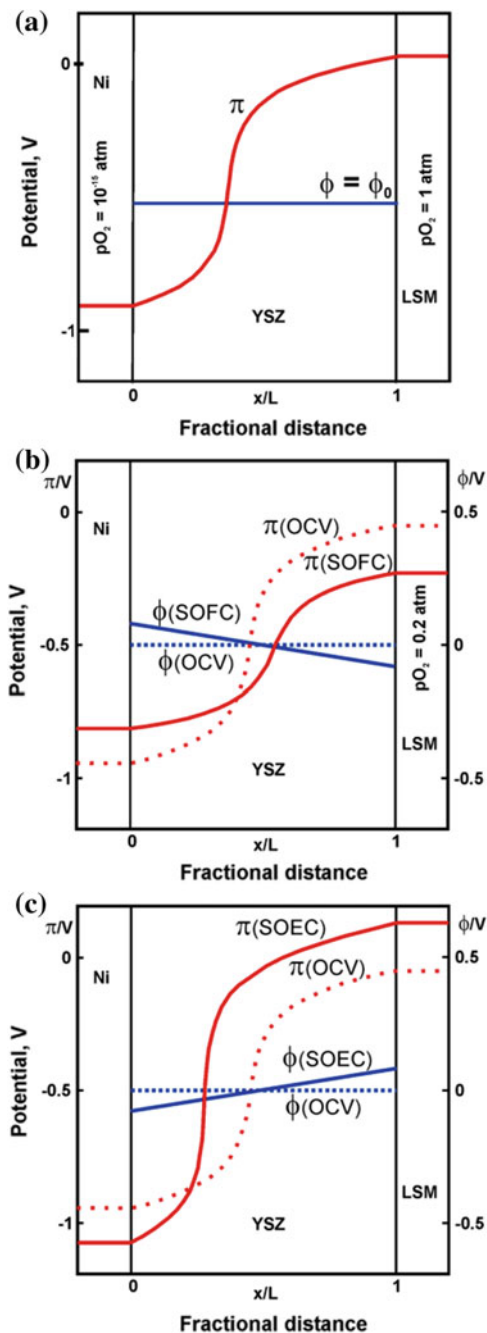
where $p^\ominus$ is taken at standard pressure of 1 bar (Jacobsen et al. 2014).

Apart from the demand that the reference electrode must sense a relevant electrostatic potential inside the electrolyte near the interface of the electrolyte–working-electrode at a position where the current density is uniform and representative of the electrode condition, the reference electrode must also have a well-defined electrode potential of its own. This means that it may be the electrode potential of the half-cell $\text{Pt}|\text{YSZ}|\text{O}^{2-}|\text{O}_2$ (at 1 atm. and given temperature), which is very suitable for SOC purposes. The general theory of reference electrodes in three-electrode set-ups may be found in most electrochemical textbooks. The three-electrode concept as such implies that it is not enough to imbed a wire of Pt or other metal inside the electrolyte. The “wire” inside the electrolyte must be an electrically insulated “wire” consisting of electrolyte in similarity to the Luggin capillary (in liquid electrochemistry). That provides the ionic contact to the half-cell that contains the reference electrode, which usually is placed outside the cell with the working and counter electrodes. As this is very difficult in solid-state electrochemistry, special attention has to be given to the geometry of three-electrode cells or to use the electrode test strategies, in which the electrode response can be measured or calculated without using a three-electrode set-up. This is the subject of the next section.

2.2 Electrode Testing

To avoid such problems, it is necessary to test the electrodes using a suitable three-electrode set-up or a symmetrical two-electrode cell, which gives interpretable results at OCV and in the overpotential region, where the current density is a linear function of overpotential (Jørgensen et al. 1999), even though all methods have their

Fig. 4 Calculated potentials versus fractional distance across the electrolyte of a Ni|YSZ|LSM SOFC. The symbols in Fig. 3 are used for the potentials. The three cases are: **a** OCV, **b** fuel cell mode, and **c** electrolysis cell mode. In **b** and **c**, the OCV potentials are also plotted as *dotted curves* (Jacobsen et al. 2014)



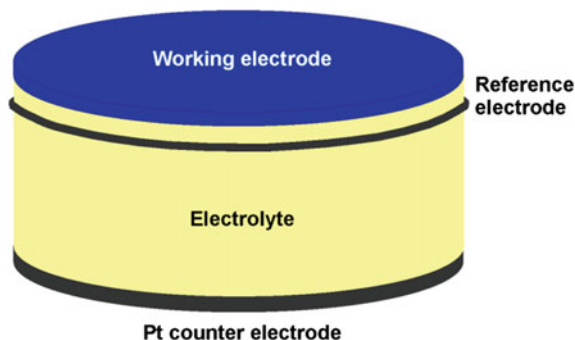


Fig. 5 Illustration of a simple three-electrode set-up. This can naturally only be used in a one atmosphere compartment, and therefore, it is important to continuously monitor the pO_2 in this compartment. The reference electrode may be a Pt-wire, which must be strictly parallel to the edge of the electrode

shortcomings. Some examples of useful set-ups for studying electrode performance are briefly presented here. The set-ups are based on zirconia pellets with an electrode arrangement suitable for three-electrode studies. The simplest type—“pellet with a belt”—is illustrated in Fig. 5. This can only be used in one atmosphere compartment set-up, and it is therefore absolutely important to monitor pO_2 continuously using a pO_2 sensor placed in the test compartment.

Another more sophisticated three-electrode set-up with two atmospheres is sketched in Fig. 6. If pure oxygen (1 atm.) is used in the counter and reference electrode compartment, then this set-up is a genuine three-electrode set-up. It is a correct geometry, and the gas composition is constant during testing. Such pellet-like geometries, where the reference electrode can be suitably placed (in a bore as in Fig. 6 or as a ring around the pellet in Fig. 5), are suitable for fundamental studies of electrode kinetics. The pellet-like test cell geometries suffer from two disadvantages; it is difficult to ensure that the fabrication process for the electrodes used is identical to the one used for the technological larger scale full cells, and the ohmic resistance between the working and the reference electrodes is quite substantial which may result in a “signal-to-noise” problem, when very high performing electrodes are studied.

To measure a particular electrode performance in detail, a symmetrical cell with identical electrodes on each side of the electrolyte can be used as shown in Fig. 7. This has a platinum mesh to ensure good contact with the electrodes and two platinum wires coming out from each side of the cell, one to measure current and the other for potential determination. Such a test cell is well suited for electrode development work, because there is no ambiguity about the source of electrode properties and performance in this case. However, its use is limited to the investigations close to OCV, where the electrode loss does not depend on whether it is anodic or cathodic polarized.

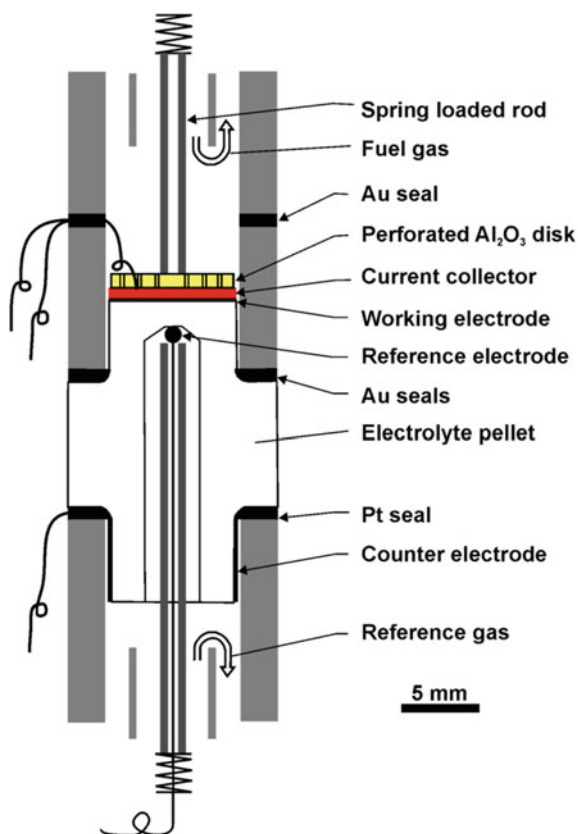


Fig. 6 Sketch of a three-electrode set-up based on a cylindrical YSZ electrolyte pellet, which is held between two alumina tubes that are supplying each their gas to the three-electrode cell. In order to make the composition of the reference and counter electrodes gas independent of the gas conversion on the counter electrode, it is recommended to use pure oxygen. In principle, other materials and gases may be used in such a set-up

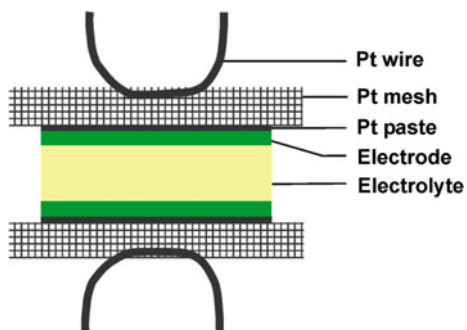


Fig. 7 A symmetrical two-electrode cell arrangement for measurements near OCV, i.e., inside the potential regime of polarization voltage in which the current density—overvoltage curve is linear for the tested electrode type

3 Cells and “Short” Stacks—Geometries, Test Setup, and Conditions

This section describes possible test set-up and cell assembly. Furthermore, focus will be on the probing of voltages and temperature, sealing and contacting of the cell; all important aspects of proper cell testing for obtaining reliable results; also including the aspect of impurities.

3.1 Test House and Cell Assembly for Full Cell Test

For testing of planar SOC, a test house and cell assemblies as illustrated in Fig. 8 can be used. As illustrated in Fig. 8a the cell will be sandwiched in between a fuel and air distributor (grooved) plates or meshes, and these are contacted with platinum or gold foil and Ni foil for the fuel side. The cell is sealed using glass seals (gas tightness) along the edges between two alumina blocks that constitute the cell house. This ensures that fuel and air are led to the electrodes and not simply mixed in the test house. The alumina blocks have built in gas channels for air (or oxygen) inlet and outlet and for fuel inlet and outlet. Different designs for test housing and cell assembly can be found in literature (Jensen et al. 2009a, b; Ebbesen et al. 2010; Kornely et al. 2011) but based on similar concept, i.e., the cell sandwiched between gas distributors and current collecting foils and applying sealing to avoid mixing of fuel and air. The whole assembly shown in Fig. 8 is subsequently enclosed in a furnace within a ventilated hood into which gases are fed from a manifold system. This allows a range of gasses to be led to the fuel side, e.g., H_2 , O_2 , N_2 , CO , CO_2 and CH_4 and to the oxygen electrode side, e.g., air, O_2 and N_2 . For humidification, H_2 can be led through a thermo-stated water bubbler, hydrogen and oxygen can be mixed in the fuel inlet tubing, or an external evaporator can be connected. For safety reasons, the ventilated hood should be equipped with sensors to monitor possible H_2 and CO leakages and monitor proper ventilation.

For stack testing, there are several examples on results—experimental and modeling—that illustrate the importance of awareness of the choice of flow design and geometries, e.g., cross-flow versus co-flow versus counter-flow, when considering temperature profiles (Lawlor 2013; Zhang 2012; Kromp et al. 2011). The effect of chosen flow geometry will be much smaller when considering single cell test compared to testing of large stacks. Besides considering air and fuel flow directions, it is also worthwhile to consider the applied flow field design given by the chosen gas distributor plates, meshes, or interconnects. As illustrated in Fig. 9 via experimental data and results from modeling (Kornely et al. 2011), the flow field design obtained by different design of gas distributor plates for single cell test can have a significant influence of obtained cell performance.

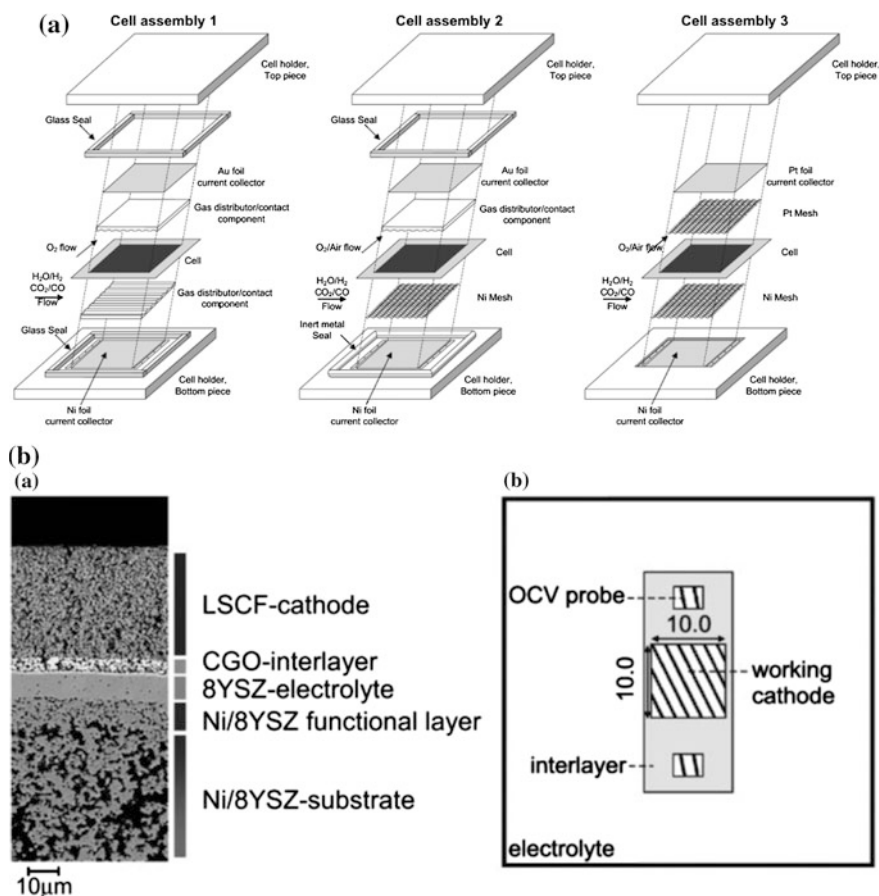


Fig. 8 **a** The assembly of cell and gas distribution plates in a cross-flow pattern with varying choice of gas distributors and solutions for sealing for 53 × 53 mm² single cells (Ebbesen et al. 2010) and **b** for a design-wise different test set-up/geometry for a 10 × 10 mm² single cell (Kromp et al. 2011)

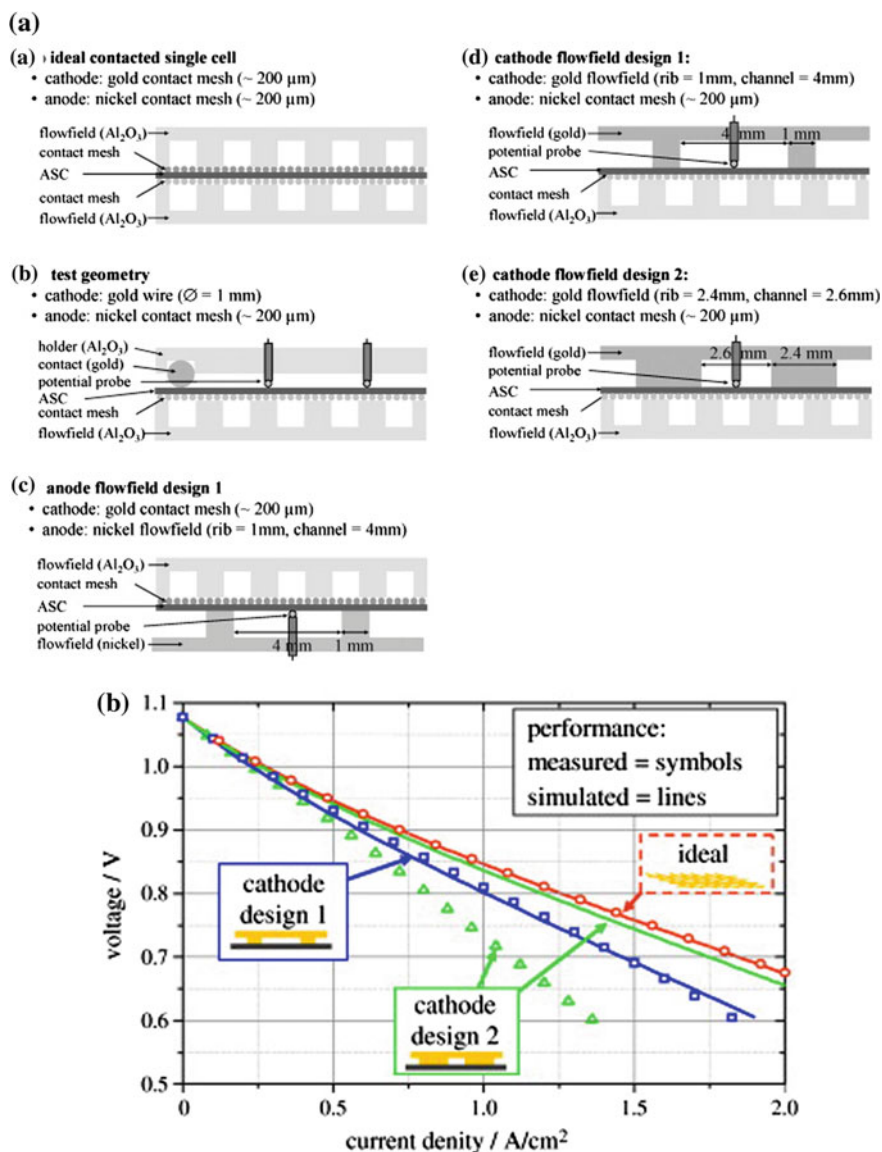


Fig. 9 Different flow field designs and their effect on cell performance (Kornely et al. 2011)

3.2 Voltage Probes

The drawing of alumina blocks in Fig. 10 shows the number and position of voltage probes, and probes for current pickup in the test house corresponding to the cell assembly shown in Fig. 8a. The voltage difference between voltage probe 1 and 3

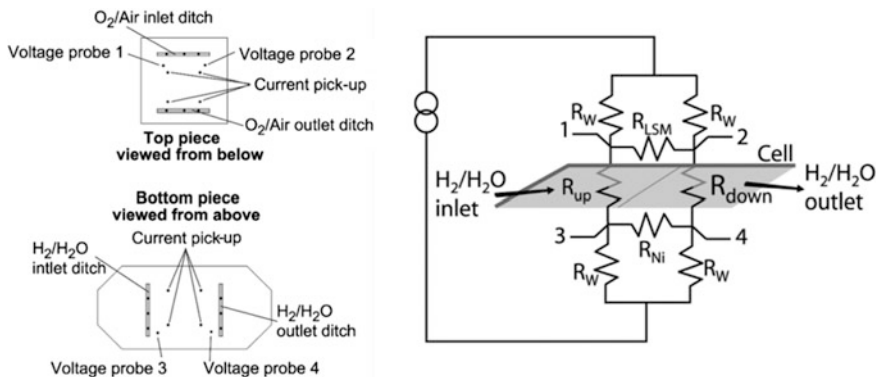


Fig. 10 Positions for voltage probes and current pickup for planar single cell test geometry given in Fig. 8a and sketch of corresponding electric circuit (Jensen et al. 2009a, b)

will monitor the voltage across the cell at the fuel inlet part of the cell while the voltage difference between probe 2 and 4 will monitor the voltage across the cell at the fuel outlet part of the cell.

Placing two or more voltage probes on the same side of the cell enables the measurements of the in-plane voltages, e.g., the voltage difference between probe 3 and 4 in Fig. 10 will measure the in-plane voltage difference between inlet and outlet for the fuel side of the cell. This voltage difference will of course be small compared to the cell voltage but in-plane voltage measurement will provide information on differences in current density from one region of the electrode compared to another region of the same electrode. Measuring, e.g., the fuel side in-plane voltage (probe 3 and 4 in Fig. 10) is advantageous in the analysis of differences in fuel electrode performance along the fuel flow, e.g., in cases where impurities in the gas phase are investigated as reported in literature (Jensen et al. 2009a, b; Ebbesen et al. 2010; Rasmussen and Hagen 2010; Hauch et al. 2014). Furthermore, such extended probing for single cell test as illustrated in Fig. 10 combined with the proper AC characterization of cell performance (see Sect. 5) enables detailed modeling of the cell performance and effects of the impurities when combined with models for the corresponding equivalent circuit as illustrated in Fig. 10 (Jensen et al. 2009a, b).

3.3 Temperature Measurements

Ideally, one would like to measure the temperature in the active electrodes upon cell testing; however, in practice, holes will typically be drilled in the alumina blocks for test housing and thermocouples will be placed similar to the voltage probes, i.e., in the cell assembly but not in the electrodes themselves. This enables measurements of the cell temperature during testing, e.g., during characterization via polarization curves as illustrated in bottom part of Fig. 11 (Ebbesen and Mogensen 2013).

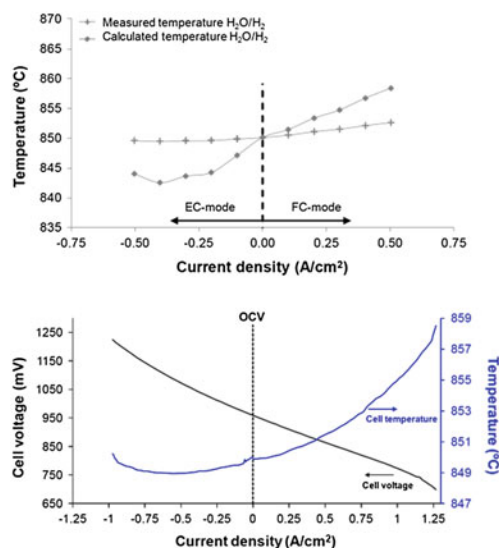


Fig. 11 Temperature measurements during IS at different currents (i.e. measurements of ohmic and polarization resistances at increasing current load) and temperatures calculated based on the changes in the ohmic resistance obtained from IS during step in current (*top*) and temperature measured during an IV-curve (Ebbesen and Mogensen 2013). Be aware of the different scaling for the two graphs

Notice how the cell temperature increases almost 10 $^{\circ}\text{C}$ during the fuel cell mode part of the polarization curve (exothermic reaction and joule heating) while slight decrease in cell temperature is observed during the majority of the electrolysis mode part of the polarization curve (cooling due to the endothermic reaction but partly counterbalanced by the increased joule heating upon increased current density). The left part of Fig. 11 clearly illustrates the issue of inaccurate temperature measurements due to the position of the thermocouples “away” from the spot of the electrochemical reactions. In Fig. 11, impedance spectra are recorded with intervals of 0.1 A/cm^2 between -0.5 A/cm^2 (electrolysis mode) and 0.5 A/cm^2 (fuel cell mode), and at each current density, the cell temperature was measured by thermocouples close to the cell via drilled holes in the alumina test house and plotted as “+” in Fig. 11 (top). Furthermore, the ohmic resistance was obtained from each of the recorded impedance spectra, and from the obtained ohmic resistance, the corresponding change in cell temperature was calculated (based on materials’ conductivity). Applying this alternative way of calculating the cell temperature, Ebbesen and Mogensen (2013) clearly illustrate the differences between temperatures measured by thermocouples “outside” of the cell and the actual temperature that can be expected in the active electrodes and electrolytes during DC characterization.

3.4 Sealing, Contacting, and Current Collection

Sealing of the cell assembly can be obtained by glass seals as illustrated in Fig. 8a by glass bars or glass felt that softens upon heating. Appropriate glass composition will typically depend on start-up temperature profile (including reduction temperature). A variety of glass compositions have been tested and reviewed for SOC sealing applications as described in literature (Schilm 2010; Chou et al. 2007; Reis et al. 2010; Nielsen et al. 2007). From a test point of view, the aim of the glass seal is of course gas tightness; however, from an electrode characterization point of view, one should pay attention to the fact that glass seals can contribute with undesirable impurities being led in the gas phase to the electrodes during testing especially during electrolysis operation as described in literature (Hauch et al. 2007; Wiedenmann et al. 2010). Minimizing the glass area exposed to the gas stream is recommendable and alternative sealing materials (e.g., gold) can be considered.

Proper current collection and contacting of the cell for single cell testing is necessary in order to avoid misinterpretation of cell performance results. Resistance due to the contacting of the cell will contribute to the overall resistance of the cell as described in literature (Barfod et al. 2006). This will affect the quantification of the ohmic resistance and the corresponding activation energy, $E_a(R_{\text{ohmic}})$. The ohmic resistance contribution from contacting the cell will of course depend on the exact set-up but typically be in the range of $20 \text{ m } \Omega \text{ cm}^2$ in the temperature range 700–850 °C (Barfod et al. 2006).

Furthermore, non-optimal contacting where, for instance, part of a larger cell is not contacted at all will lead to higher current density in the contacted regions of the cell compared to the nominal current density set by the cell tester, i.e., only smaller regions of the cell will be tested but at different test conditions than those set by the operator/cell tester, inevitably giving misleading results for the cell performance.

3.5 Galvanostatic Test Versus Potentiostatic Test

Here, the important concepts of overpotential and polarization have to be defined before discussing the pros and cons of galvanostatic test versus potentiostatic test. The term “polarization” is used for the whole cell and for any of the cell component that gives rise to a loss in voltage, when the cell is electrically loaded. The concept “overpotential” (the same as “overvoltage”) makes only sense for the electrodes that have an equilibrium potential at a given set of conditions. Thus, when the potential is moved away from this equilibrium, the electrode potential will change away from the equilibrium value, and the value of the change away from equilibrium is called the “overpotential.”

The electrochemical driving force of an electrode process is the overpotential, and the current density is reflecting the resulting reaction rate. Thus, the natural test method should be potentiostatic (constant voltage), but due to historical reasons and

the easiness of doing galvanostatic (constant current) tests, almost all reported cell tests have been galvanostatic. The galvanostatic test results may be significantly different from the potentiostatic, because a change in driving force (overpotential) may cause changes in electrode mechanisms during the test. Thus, at high current density, degradation over time may push the electrode overvoltages to a level where an electrode potential will exceed the stability limit of the electrode and/or the electrolyte material, and the electrode or the whole cell may be destroyed (Knibbe et al. 2010; Chen et al. 2013; Zhang et al. 2014; Hauch et al. 2016).

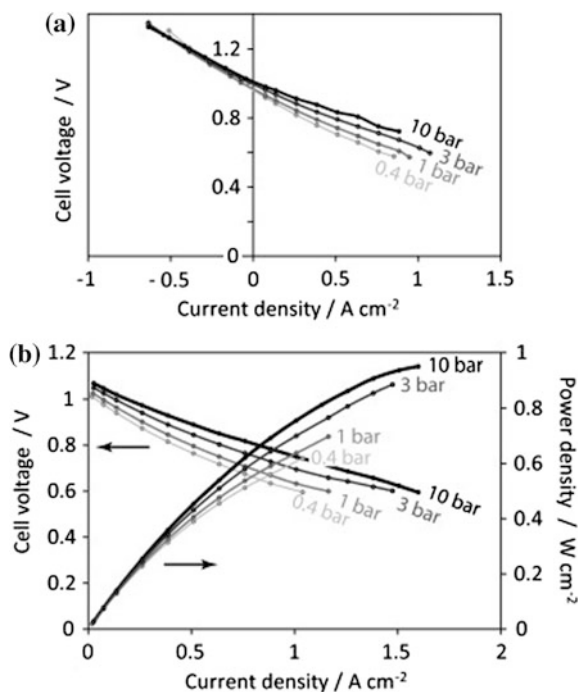
3.6 Other Types of Testing

Apart from simple performance tests and long-term durability tests, a number of other test variations may be performed using the same cell (and stack) test set-up. Some important types of tests are pressurized tests, redox tests, thermal cycling, electrical load cycling, and fuel-cell-electrolysis-cell-mode cycling, e.g., using a setup as shown in Fig. 8a.

Examples of pressurization tests have been published by Jensen et al. (2010) and Sun et al. (2015) and an illustration of polarization curves for pressurized tests is given in Fig. 12. The set-up is placed in a furnace inside an autoclave, which is approved to a pressure of 100 bar. The autoclave is filled with flowing pure nitrogen of the same pressure as the pressure in the electrode compartments, i.e., the pressure must be the same inside and outside the cell test house. Naturally, the testing with pressurized hydrogen, CO, and oxygen demands strong safety measures. A detailed description of this is comprehensive and too specialized to include in this text, but it should be noted that it is possible to satisfy even the very strict Danish safety rules for experiments inside a normal laboratory building. The strategy behind is that the flow rate and volume of the nitrogen are so that if all inflowing oxygen and hydrogen were mixed outside the cell house, then it would take an appreciable amount of time before dangerous concentration levels of CO, H₂, and O₂ would be reached. This is then combined with CO and H₂ sensors that stop the supply of H₂ and/or CO if a certain very low concentrations of these are detected. Furthermore, only so-called safety gas of N₂ with maximum 9 % H₂ is allowed in the setup until the temperature is above 600 °C. Over that temperature, H₂ plus O₂ will just burn with a simple flame, which will heat the test house. Then, a separate thermocouple with the purpose of surveillance only will trigger a valve that stops the H₂ and CO flow.

The same basic strategy of safety thinking is behind the testing with pure H₂ and CO in general in order to tolerate testing such as redox test and thermal cycling as these tests will, by nature, often end with a mechanical break down of the cell, which will allow oxygen or air to mix with the fuel gases. Examples of redox testing are given by e.g. Pihlatie et al. (2009) and of electrical load testing by e.g. Hagen et al. (2006), and examples of temperature cycling can be found in literature as well. Examples of fuel-cell-electrolysis-cell-mode cycling are more

Fig. 12 Polarization curves (IV-curves) at various pressure at 850 °C, H₂/H₂O:50/50, air to the oxygen electrode. Ni/YSZ|YSZ|LSM/YSZ-based and fuel electrode-supported planar SOC (Jensen et al. 2010)



rare, but a recent study has been published by Graves et al. (2015). In case of switching between fuel cell and electrolysis modes, extra gas supplies of steam and CO₂ are necessary. Otherwise, it is mainly the careful test planning, which will be different for the different types of testing.

3.7 Impurities

Both fuel electrodes and oxygen electrodes of SOC may be poisoned by many compounds coming from many sources, and even though the set-up and gases used are believed to be very clean, realities have often revealed the contrary. Unfortunately, there is seldom a simple procedure, which can clarify the reason and origin for an observed degradation over time. It is even difficult to figure out whether it is a poisoning initiated or another reason that is the cause of an observed degradation. One source of impurities is the cell itself. No material is totally pure. At least some trace elements will be present, and practically, “pure” materials will most often contain some impurities on the level of 100 ppm or more. Some impurities will be able to segregate from the surface and/or interface of electrodes and electrolyte. A relative simple set-up to test materials for any impurities, which in particular tend to segregate to surfaces and triple phase boundaries (TPB), is

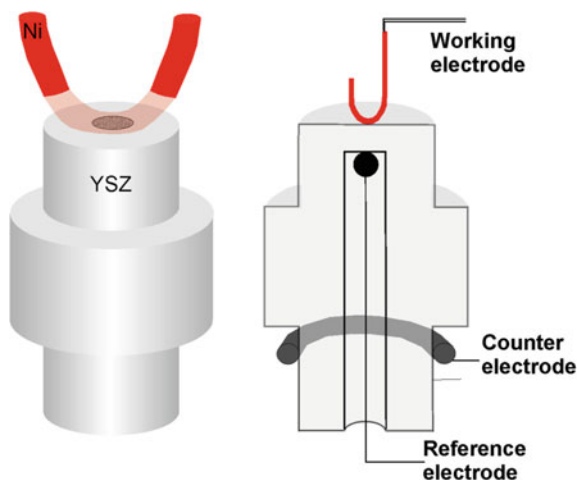


Fig. 13 An illustration of model electrode made by a bent Ni wire pressed onto an electrolyte pellet of YSZ as used at Technical University of Denmark. The YSZ have been a simple pellet shape instead of the slightly complicated pellet made for three-electrode setup. Modified from (Vels Jensen et al. 2001)

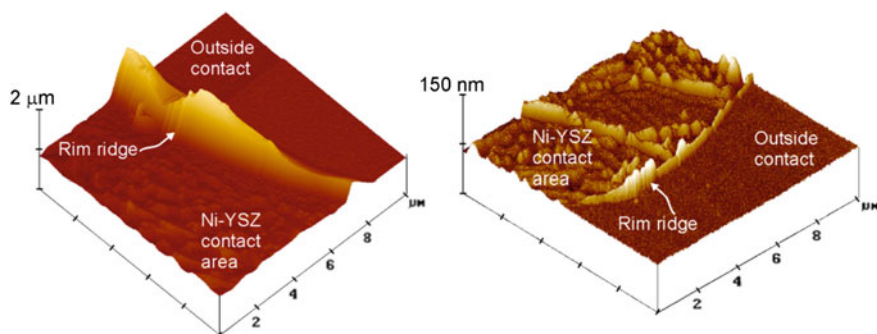


Fig. 14 AFM micrographs of the YSZ side of Ni-YSZ interfaces after few days of testing at 1000 °C in wet hydrogen (97 % $H_2/3$ % H_2O); *left* 99.8 % pure Ni, *right* 99.995 % pure Ni. The electrolyte contained <100 ppm impurities. The rim ridges consist only of impurities like SiO_2 , TiO_2 , and Na_2O . Notice the different scale in the two figures. From (Hansen et al. 2004; Vels Jensen et al. 2001)

sketched in Fig. 13, and results for two different impurity levels of Ni-wire electrodes on a YSZ electrolyte are shown in Fig. 14. The main part of the rim ridge in the left part of Fig. 14 originates from the 99.8 % pure (i.e., impure) Ni wire, while the much smaller rim ridge in the right part of Fig. 14 probably originates from the YSZ and is believed to consist mainly of SiO_2 (Hansen et al. 2004).

In spite of the differences, both TPB look fully blocked, but actually some current can pass even in such model electrodes. Fortunately, the situation is much less critical in a Ni-YSZ cermet electrode, because the ratio between the TPB

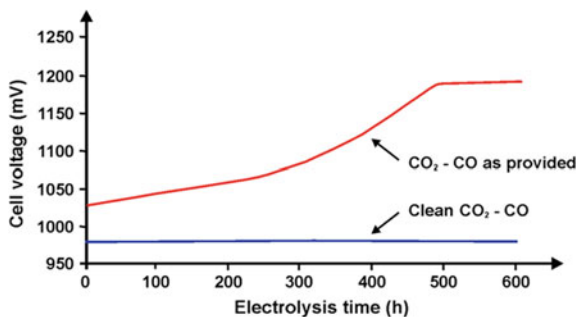


Fig. 15 A sketch of the influence of gas impurities in durability test of cells in electrolysis mode. The as provided gas contained a few ppb of sulfur (about the detection limit), and the other gas was cleaned using a scrubber of hot Ni-YSZ electrode powder. Modified from (Ebbesen and Mogensen 2010)

length and the volume of materials is much bigger for a cermet with submicron particles than in these model electrodes; yet, the TPB will probably not be really clean in any case, and the impurities seen using the model electrodes may also be found in Ni-YSZ electrodes using, e.g., TEM (Hauch et al. 2008).

An example of how to test for—and the effect of—impurities coming from a fuel electrode gas stream is presented in Fig. 15. The as-provided-gas was analyzed by gas chromatography, but only sulfur with a concentration just about the detection limit of ca. 20 ppb was measured. However, the test with clean CO + CO₂, which was cleaned using a filter consisting of fine electrode powder of Ni-YSZ in the “next experiment,” proved that the degradation was caused by impurities in the as-provided-gases, and the electrode was stable under same test conditions but applying cleaned gases (Ebbesen and Mogensen 2010).

4 Area Specific Resistance (ASR)¹

4.1 The Concept of Area Specific Resistance (ASR)

A fuel cell stack can be regarded as a “black box” into which hydrogen (gas) and oxygen (air) are inputs and electricity and exhaust gases are outputs. For such a stack, ASR is defined as:

$$\text{ASR} = \frac{\text{Emf} - U}{i} \quad (2)$$

¹ Based on previous work by Mogensen and Hendriksen (2003).

where E_{mf} is the electromotive force with the inlet fuel and air, and U is the cell voltage at the current density, i , at the design point. A possible design point, might be 0.6 V at 1000 °C, a fuel utilization of 85 %, and an air flow of 4 times the stoichiometric amount (“4 stoichs”).

The cell voltage, U , should be measured independently from the leads carrying the current, i.e., separate potential probes should be used. This ASR is in most cases not very sensitive to small variations in cell voltage and fuel utilization. By determining the ASR at a few different temperatures, apparent activation energy, E_A , may be derived. Thus, in a voltage interval (say from 0.5 to 0.7 V) and a temperature interval (say from 650 to 1050 °C), the cell may be characterized, with fair approximation, by only two characteristic numbers, namely ASR at one temperature and E_A . In case the I - V -curve is concave, it may be tempting to use a differential ASR (i.e., the tangent) at high current densities as this gives a nice low value. Such a number has the drawback that it does not reflect the cell performance over the full polarization range as does the quantity defined by Eq. (2).

Sometimes, cell tests are conducted with very low fuel and air utilizations, because these are easier to perform than the “realistic” tests with high fuel utilization. In case of insignificant fuel and oxygen utilizations, the relevant definition of ASR is again that of Eq. (2), but the insignificant utilization makes this value incomparable to ASR derived from experiments with high fuel utilization, because the concentration polarization resistance due to the fuel and air conversion can be a considerable fraction of the total cell resistance. Thus, it is, in general, necessary to specify also the fuel and oxygen utilization together with temperature and apparent activation energy. Furthermore, using almost dry hydrogen, as is the common practice for SOFC tests, it is not easy to conduct experiments with a real negligible fuel utilization, since even small current densities will create enough water to change significantly the E_{mf} of the hydrogen/water fuel gas versus air, e.g., if the inlet gas contains 0.1 % H_2O and the fuel utilization is 0.1 %, this changes the H_2O/H_2 ratio by a factor of 2, which in turn changes the E_{mf} by 34 mV at 850 °C. Therefore, in order to be able to compare results for different fuel utilizations, the ASR value should be corrected for the effect of fuel utilization. Before describing how this may be done, various contributions to the total ASR are examined below.

ASR may be divided into ohmic resistance, R_s , and electrode polarization resistance, R_p . The ohmic resistance originates from the electrolyte, the electrodes materials, and the current collection arrangement. This is very much dependent on geometric factors such as thickness of the cell components and the detailed geometry of the contact between the current collection and electrodes, and between electrodes and electrolyte as current constrictions may be important (Primdahl and Mogensen 1998). The electrode polarization resistance is further divided into contributions from the various rate-limiting steps. Thus, ASR can be broken down into five terms:

Table 1 Contributions to ASR for an anode-supported full cell having a multilayer tape cast anode half-cell sintered at 1295 °C and a screen printed LSCF/CGO cathode (Ni/YSZ|YSZ|CGO_{barrier}|LSCF/CGO)

Resistances ($\Omega \text{ cm}^2$)	850 °C OCV	750 °C OCV	650 °C OCV	750 °C 0.63 A/cm ²
$R_s (R_{\text{elyt}} + R_{\text{connect}})$	0.051	0.113	0.328	0.109
$R_{p,\text{electrochem; oxelectrode}}$	0.017	0.035	0.099	0.025
$R_{p,\text{electrochem; fuelectrode}}$	0.029	0.076	0.287	0.054
$R_{p,\text{diff}}$	0.029	0.025	0.030	0.010
$R_{p,\text{conv.}}$	0.062	0.059	0.062	0.043
ASR _{Total}	0.188	0.308	0.806	0.241

The cell was characterized at 850, 750, and 650 °C in a single cell test setup as illustrated in Figs. 8a and 10. Values for ASR contributions are based on CNLS equivalent circuit model fitting to experimental impedance spectra (Birkel 2012) (Master thesis by Christoph Birkel, supervised by Dr. Anne Hauch. This thesis is available via DTU Library, Technical Information Center of Denmark, Anker Engelundsvej 1, building 101, DK-2800 Kgs. Lyngby, Denmark). Results are obtained at a $p(\text{H}_2)/p(\text{H}_2\text{O})$ ratio of 80/20, air to the oxygen electrode, and a fuel utilization of ~23 % at 0.63 A/cm². Estimated error for the resistances ~ $\pm 0.004 \Omega \text{ cm}^2$

$$\text{ASR} = R_{\text{elyt}} + R_{\text{connect}} + R_{p,\text{elchem}} + R_{p,\text{diff}} + R_{p,\text{conv}} \quad (3)$$

where R_{elyt} is the electrolyte resistance calculated from the measured specific conductivity and the thickness; $R_{\text{connect}} = R_s - R_{\text{elyt}}$ is the resistance due to the non-optimized contact and current collection; $R_{p,\text{elchem}}$ is the electrode polarization originating from all the limiting chemical and electrochemical processes on the electrode surfaces, in the bulk electrode material and on the electrolyte–electrode interfaces, and $R_{p,\text{elchem}}$ contains contributions from the oxygen electrode ($R_{p,\text{electrochem; ox.electrode}}$) as well as the fuel electrode ($R_{p,\text{electrochem; fuelectrode}}$); $R_{p,\text{diff}}$ is the contribution from the gas phase diffusion; and $R_{p,\text{conv}}$ is the contribution due to the gas conversion, i.e., fuel oxidation and oxygen reduction. This division of ASR is based on what is possible to measure and calculate reliably rather than on any physical or electrochemical basis. Some terms in Eq. (3) can therefore be thought of as “equivalent resistances,” e.g., the Emf drop due to the changes in gas composition resulting from the fuel utilization is translated into an equivalent resistance. Depending on the exact type of electrode, different types of contributions are possible as derived from more basic electrochemical point of view. For example, current constriction may be important if the electrode has coarse porous structure but of less or no importance in case of a fine-structured electrode. In one type of oxygen electrode, the surface diffusion may be important, but in another the diffusion of oxide ions (and electrons) through the electrode particles may cause the main polarization loss. Values for “Eq. (3)”—ASR contributions for an fuel electrode-supported cell with a 300- μm -thick support fed with H_2 (with 20 % H_2O) are given in Table 1 for different temperatures at OCV and at a current density of

0.63 A/cm²; all numbers based on impedance spectroscopy data recorded on the same cell (Birkel 2012).

It is observed that the contributions from the concentration polarization, i.e., $R_{p,\text{diff}}$ and R_{conv} , is dominating at 850 °C but not at 750 and 650 °C. In an electrode-supported cell, the limitation of gas diffusion through the support is a cell relevant resistance, whereas R_{conv} results from operation demands and choice of test set-up geometries, and it is thus of special interest to be able to correct ASR for the effect of fuel conversion.

If a significant amount of fuel is consumed in the cell (or stack) under test, a resistance derived on the basis of Emf of the inlet gas [c.f. Eq. (2)] will be an overestimation of the “true” cell resistance. The larger the fuel utilization, the larger will be the overestimation. A comparison between cell test results obtained under different and non-negligible fuel utilizations must thus, to be meaningful, be based on a resistance measure, ASR_{cor} , where the effects of changes in gas composition over the cell area have been taken into account. How the correction is applied depends on how the gases are fed to the cell. Here, two idealized cases are considered, namely the case where the fuel compartment may be considered a continuously stirred tank reactor (CSTR) or a plug flow reactor.

If the fuel compartment can be considered CSTR-like due to effective mixing because of a turbulent gas stream and fast gas diffusion, ASR_{cor} can be calculated from the expression

$$\text{ASR}_{\text{cor}} = \frac{\text{Emf}_{\text{avg}} - U}{i} \quad (4)$$

where Emf_{avg} signifies the average Emf, which in this case is the same as the Emf of the outlet gas. An example of such conditions is reported elsewhere (Primdahl and Mogensen 1998).

The plug flow case is slightly more complex. Under the assumptions that the local ASR is independent of the position along the fuel and air flow channels, and the flow pattern is co-flow, ASR_{cor} may be calculated from the expression (Solheim et al. 1991):

$$\text{ASR}_{\text{cor}} = \left\{ \frac{i}{X_{\text{H}_2}^i - X_{\text{H}_2}^o} \int_{X_{\text{H}_2}^o}^{X_{\text{H}_2}^i} \frac{dX_{\text{H}_2}}{\text{Emf}(X_{\text{H}_2}) - U} \right\}^{-1} \quad (5)$$

where

$$\text{Emf}(X_{\text{H}_2}) = E^0 - \frac{RT}{2F} \ln \left\{ \frac{X_{\text{H}_2\text{O}}^i + X_{\text{H}_2}^i - X_{\text{H}_2}}{X_{\text{H}_2} \sqrt{X_{\text{O}_2}^i - \frac{N_f}{2N_a} (X_{\text{H}_2}^i - X_{\text{H}_2})}} \frac{1}{\sqrt{P_a/\text{atm}}} \right\} \quad (6)$$

here X is the molar fraction, P_a is the air pressure, and superscripts i and o signify inlet and outlet, respectively. N_f and N_a are the molar flows of fuel and air.

The assumption of a position-independent local ASR is an approximation, which is not always justifiable. Part of the fuel electrode polarization resistance is dependent on fuel composition. However, often this part is small. If the cell is not isothermal, the local resistance will vary with position due to its temperature dependence. Also, the actual flow pattern may be much more complex than just co-flow. Even so, if the fuel utilization is large, ASR_{cor} derived from Eqs. 4 to 5 will always be a better characteristic of a cell than a value derived neglecting the fuel utilization [Eq. (2)]. More precise evaluation of ASR_{cor} requires a rigorous 3-D modeling of the cell test.

For the purposes of evaluating Eq. (5), the integral may be approximated by a sum (Mogensen et al. 1999):

$$ASR_{cor} = \left\{ \frac{i}{N} \sum_{j=0}^{N-1} \frac{1}{Emf(X_{H_2}(j)) - U} \right\}^{-1} \quad (7)$$

where $Emf(X_{H_2}(j))$ is given by Eq. (5) with

$$X_{H_2}(j) = X_{H_2}^i + \frac{j + \frac{1}{2}}{N} (X_{H_2}^o - X_{H_2}^i) \quad (8)$$

The more terms are included in the sum, the better the approximation.

A “first-order” correction for the effects of finite fuel and air utilizations may be obtained by taking only one term in the sum in which case ASR_{cor} should be evaluated from Eq. (4) with

$$Emf_{avg} = E^0 - \frac{RT}{2F} \ln \left\{ \frac{\bar{P}_{H_2O}}{\bar{P}_{H_2} \sqrt{\bar{P}_{O_2}/atm}} \right\}, \quad (9)$$

where the bar indicates the “average,” i.e.,:

$$\bar{P}_{H_2O} = \frac{P_{H_2O}^i + P_{H_2O}^o}{2}, \quad \bar{P}_{H_2} = \frac{P_{H_2}^i + P_{H_2}^o}{2} \quad \text{and} \quad \bar{P}_{O_2} = \frac{P_{O_2}^i + P_{O_2}^o}{2} \quad (10)$$

If there are no significant leaks in the cell or the test equipment, then both fuel and air utilization, and from this the compositions, may be calculated from the flow rates and the current using Faraday’s law. Alternatively, the composition of the outlet fuel and air may be obtained by gas analysis. The conversion resistance, $R_{p,conver}$, may be calculated using the concept of Emf_{avg} :

$$R_{p,conver} = \frac{Emf_{inlet} - Emf_{avg}}{i} \quad (11)$$

However, the anisotropic nature (temperature, gas composition, and current flow) of a real cell under current flow often invalidates this simple approach. Gas

conversion under electric load causes an uneven distribution of the current density with decreasing current density in the downstream direction. Fuel composition gradients over the cell originating from leaks cause different driving potentials at different points. In extreme cases, this may result in high Emf areas driving low Emf areas in electrolysis mode. Thus, internal currents may flow in the cell even at OCV. Gas leaks in the cell also affect the current density distribution under *load* and cause localized heating by combustion. For tubular cell designs with high in-plane resistance, the current density distribution may be affected; furthermore,

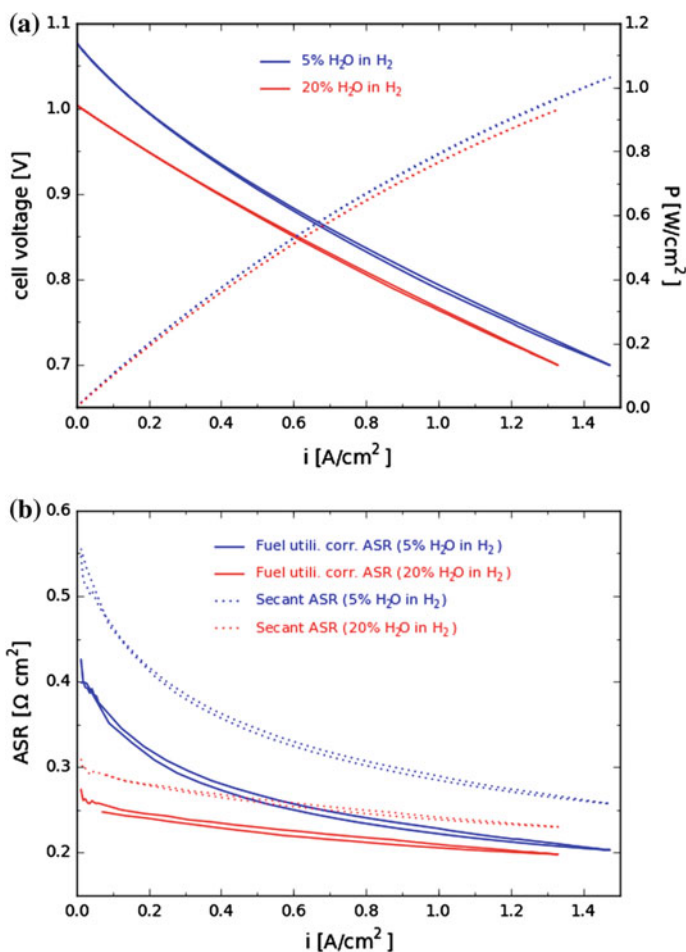


Fig. 16 IV-curve at 750 °C, air to the cathode and $p(\text{H}_2)/p(\text{H}_2\text{O}):80/20$ and $95/5$ to the fuel electrode for a planar Ni/YSZ|YSZ|CGO_{ba}|LSC/CGO-based cell. **a** IV-curve and the corresponding power density curves and **b** corresponding fuel utilization corrected ASR and ASR values based on the secant of the IV-curves (Data from DTU Energy, Technical University of Denmark)

the temperature may not be constant over the whole cell length with flowing gases adding to the inhomogeneity of the current density.

4.2 Fuel Utilization Corrected ASR

Modeling can simplify or reduce the extent of experimental tasks and predict likely behavior under a broad range of test conditions. However, subsequent validation by comparison with relevant cell and stack data is always important.

An example of the magnitude of $R_{\text{conv.}}$ is illustrated in Fig. 16, which shows I–V characteristics obtained in hydrogen with either 20 or 5 % water vapor for a fuel electrode-supported cell. Both the ASR deduced directly from the curves via secants of the IV-curves and ASR corrected for the fuel conversion [Eqs. (7) and (8)] are shown in Fig. 16. The correction for $R_{\text{conv.}}$ in the case of 20 % H_2O is in this specific case ca. $30\text{--}35 \text{ m } \Omega \text{ cm}^2$, reflecting the gas composition dependence of the Nernst voltage. After correcting for the effect of non-negligible fuel utilization, the cell resistance is still significantly smaller when measured with 20 % water in the feed than with 5 %. This reflects a gas composition dependence of some of the loss terms in Eq. (3). Further data and illustration of IV characteristic at different hydrogen steam ratios to illustrate this can be found in the work by Hendriksen et al. (2003), where it is argued that the observed composition dependence is primarily due to the composition dependence of the diffusive losses on the fuel electrode side (diffusion overvoltage), and it is shown how one may utilize characteristics obtained with different water vapor/hydrogen ratios to assess the magnitude of the diffusion loss (Hendriksen et al. 2003).

5 Methods for Characterization of Electrodes, Cells, and Short Stacks

This section will describe typical DC (polarization curves) and AC (impedance spectroscopy) characterization methods for electrodes and cells followed by a short section on complementary non-electrochemical characterization techniques. The focus will be on the practical aspects of obtaining proper and reliable impedance data and “intelligent” use of impedance spectroscopy for single cell characterization. For a comprehensive and more theoretical description of different polarization losses, which can be measured by impedance spectroscopy, the reader is referred to the literature (Nielsen and Hjelm 2014; Barfod et al. 2007; Kromp et al. 2011; Primdahl and Mogensen 1998; Primdahl and Mogensen 1999; Jacobsen et al. 2008; Jensen et al. 2009b; Huang et al. 2007).

5.1 DC Characterization/Polarization Curves

A typical result from a single cell test is a polarization curve also called current voltage curve (IV-curves). Figure 16 shows typical IV-curves for a planar Ni/YSZ|YSZ|CGO_{ba}|LSC/CGO-based SOC at 750 °C. The IV-curve will provide data for the performance of the SOC over a large polarization range; however, a complementary technique such as electrochemical impedance spectroscopy is necessary to analyze the different contributions to the total performance loss in detail. Figure 16 also shows the corresponding power density curve and the fuel utilization corrected ASR calculated from the IV-curve data. Often, the results of screening and performance tests are reported in terms of power density (W/cm²) figures, and corresponding values for the maximum power densities are extensively quoted. However, this measure can be confusing, because power density varies greatly with fuel composition and with electrode polarization. The IV-curves for SOC are often more or less linear, and therefore, allow an interpretation in terms of ASR. Even though ASR has no generally accepted definition, it is much less dependent on test conditions than power density, and it is preferable to use it to compare screening test results. The concept of ASR was discussed in the previous section. As stated in the previous section, the ASR value can be calculated in different ways, e.g., based on: (a) the chord from a given cell voltage, e.g., 700 mV to Emf using Eq. (2), (b) a differential ASR (i.e., the tangent) at, e.g., 700 mV, or (c) the fuel utilization corrected ASR at, e.g., 700 mV using Eq. (7).

From the IV-curves in Fig. 16, the fuel utilization can be calculated using Faradays law. The maximum current in this case of Fig. 16 only corresponds to approximately 56 % fuel conversion, and there is no notable sign of fuel starvation. The limit for maximum fuel utilization without notable loss due to the fuel starvation will of course depend on both the chosen design for the fuel flow and the cell microstructure. For cells as those applied for data for Figs. 16 and 22, the fuel starvation will typically only appear for fuel utilizations above 90 %. In the case illustrated in these two figures, the half-cell microstructure is comparable with previously reported, i.e., an approximately 300- μ m-thick anode support layer with a porosity of 25–30 % and a 10–15- μ m-active fuel electrode with a porosity down to 14 % (Hauch et al. 2012). Furthermore, IV-curves can be a useful tool in investigating leak problems in cell testing (Sect. 6).

5.2 AC Characterization/Electrochemical Impedance Spectroscopy

It is a non-trivial task to break down the total loss measured on a single cell into its components using the results from electrode testing and polarization curves of full cells. Impedance spectroscopy on full practical cells is, however, a technique by which a breakdown of losses can be made. General introduction to impedance

spectroscopy can be found in literature (Macdonald and Barsoukov 2005), and the following text will focus on the application for SOC characterization purposes. Impedance spectra obtained on a full SOC (or even a half-cell or symmetric cell) are difficult to interpret due to many processes involved and partly overlap in frequency range for these processes. Therefore, at least the following three aspects should be considered when applying electrochemical impedance spectroscopy for analysis of SOC: (1) proper impedance spectroscopy measurements, (2) intelligent use of different test conditions to analyze differences in contribution of losses, and (3) modeling the experimental impedance data to obtain the quantification of the breakdown of losses.

Regarding aspect 1, this includes measurements in the relevant frequency range, i.e., typically from 100 kHz to 0.1 Hz for SOC with an appropriate number of points recorded per frequency decade (i.e., 10–15 points per decade) if quantitative analysis is desired. Frequencies should be set to minimize effects of electronic noise from other electronic devices in the cell test rig. Furthermore, the signal amplitude of the AC signal for the impedance measurements should be adjusted to be small enough to provide a small perturbation in the linear region of the system and still be large enough for a reasonable signal-to-noise ratio (Huang et al. 2007) e.g. for a cell with the performance as given in Fig. 16 an AC signal amplitude of ~ 40 mV will be appropriate. The signal-to-noise ratio will of course also be optimized by running many cycles at the same frequency, e.g., 50–200 cycles depending on frequency range and time available for the measurement.

Regarding aspect 2, intelligent use of impedance spectroscopy at different test conditions provides valuable information on different processes constituting the total loss and gives necessary input to a subsequent quantitative modeling of the impedance response from an SOC. Impedance spectra at different temperatures but same gas compositions will enable the calculations of the activation energies and typically enable the separation of non-thermally activated processes (e.g., physical processes such as gas diffusion) from thermally activated processes (e.g., electrochemical processes in the electrodes). Impedance spectra at different gas compositions can be used to analyze the different polarization losses' dependency of gas composition and "one electrode sided" gas changes can be used to separate which impedance contributions originate from each of the electrodes for a full cell test. Analysis of differences in impedance spectra [ADIS, (Jensen et al. 2009b, 2007)] can, e.g., by use of impedance spectra at different gas compositions, be used qualitatively to analyze which part of a full cell that degrades. Figure 17 provides examples of using analysis of differences in impedance spectra (ADIS) upon changing gas composition at one electrode at the time, before and after long-term fuel cell test. It is evident from Fig. 17 (top) that when changing from air to oxygen for the LSM/YSZ-based oxygen electrode, a difference in impedance occur at a frequency around 200 Hz, and this response is basically identical before and after long-term test, indicating that the impedance from the oxygen electrode before and after test is similar. The ADIS for the fuel electrode gas shift in the top graph of Fig. 17 has several small peaks before long-term test [see also (Barfod et al. 2007)], and the ADIS for the fuel electrode gas shift has changed dramatically after test,

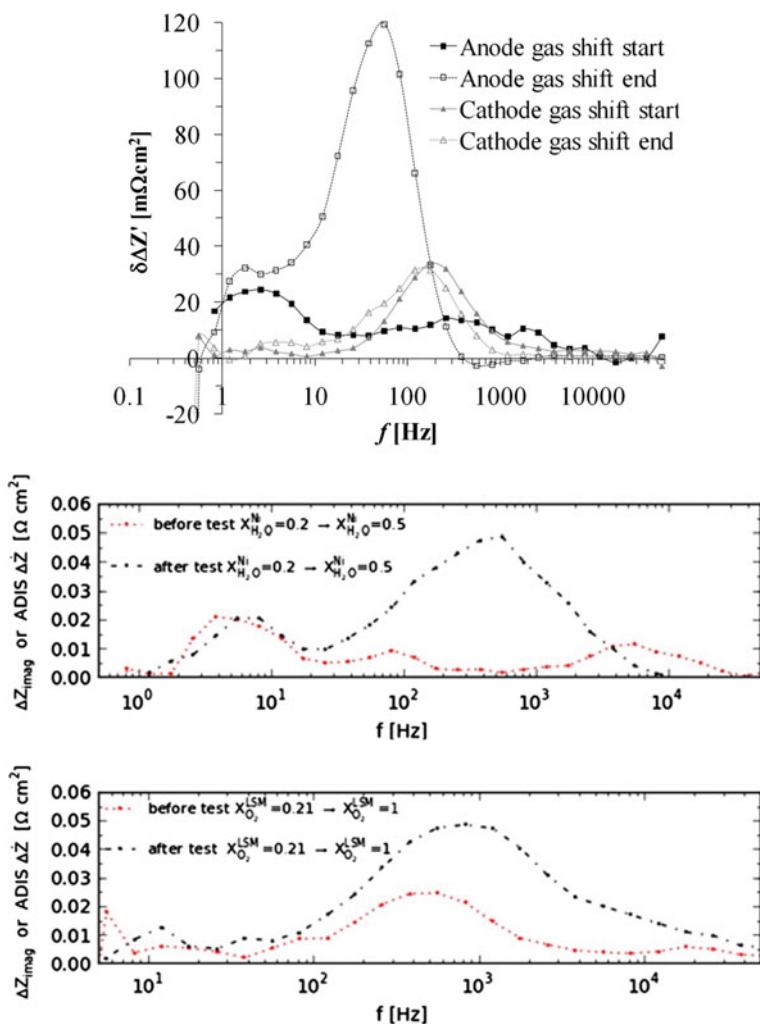


Fig. 17 *Top* Analysis of differences in impedance spectra (ADIS) before and after fuel cell test upon changing fuel gas composition between $p(\text{H}_2)/p(\text{H}_2\text{O}):96/4$ and $p(\text{H}_2)/p(\text{H}_2\text{O}):80/20$ and oxygen electrode gas between air and O_2 . Gas change impedance spectra were recorded at OCV and 750°C before and after fuel cell test (Hauch et al. [2011a, b](#)), and (*bottom*) ADIS before and after electrolysis testing (Hjalmarsson et al. [2013](#))

indicating that the fuel electrode has been severely affected by the long-term test, and the main peak has decreased from around 800 Hz before test to around 80 Hz after test. Figure 17 also provides an example of ADIS before and after long-term electrolysis testing using different gasses. In this example, significant changes for the impedance response for ADIS for both electrodes can be observed as a result of long-term electrolysis testing.

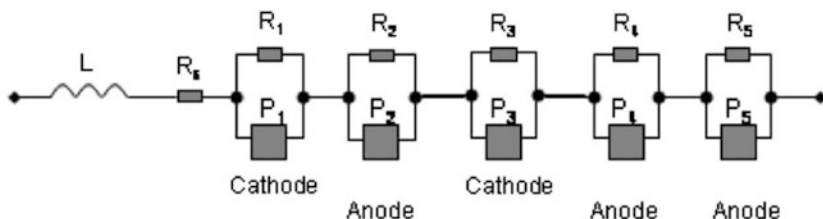


Table I. Arcs necessary to characterize losses of a full cell (approximate numbers).

	$f_{\text{sum}} 700^{\circ}\text{C}$ (Hz)	$f_{\text{sum}} 850^{\circ}\text{C}$ (Hz)	Characteristics
Arc 1	18,000	50,000	Small or no p_{O_2} dependence Small dependence on current
Arc 2	1000	8000	Dependent on fuel gas composition Strong p_{O_2} dependence
Arc 3	100	1100	Strong dependence on current Changes with $p_{\text{H}_2\text{O}}/p_{\text{H}_2}$
Arc 4	20	20	Changes with $p_{\text{H}_2\text{O}}/p_{\text{H}_2}$
Arc 5	3	3	Changes with $p_{\text{H}_2\text{O}}/p_{\text{H}_2}$

Fig. 18 Example of an applied equivalent circuit model for a full Ni/YSZ-YSZ-LSM/YSZ-based cell and the corresponding interpretation of processes and typical ranges for summit frequencies (Barfod et al. 2007)

The examples illustrated in Fig. 17 show how the use of impedance spectra at different gas compositions can provide qualitative but model-independent, electrode-specific information on different impedance contributions and assist in the analysis of which electrode—or both electrodes—that degraded during long-term testing, and provide information regarding the approximate summit frequencies for several of the different losses contributing to the total polarization loss for the full SOC.

Regarding aspect 3, to quantitatively characterize the different impedance contributions, the impedance spectra are typically analyzed in terms of equivalent circuit models applying a complex non-linear least squares (CNLS) fitting routine (Sonn et al. 2008; Nielsen and Hjelm 2014; Jacobsen et al. 2008; Orazem 2008). As an example, a resistor in parallel with a constant phase element (CPE) will typically be used as an approximation to model the charge transfer reaction process at the electrochemically active sites of the Ni/YSZ-based fuel electrode, and an example of an equivalent circuit model applied for a full SOC is given in Fig. 18 and many other examples can be found in literature. However, attention should be kept on the fact that such impedance data treatment can be highly affected by the “operator” and such impedance analysis should always be supported by complementary analyses such as ADIS using different gas compositions and temperatures. That being said, the results from the CNLS fitting of the impedance spectra applying equivalent circuit models will provide valuable quantitative information on, e.g., resistances and summit frequency for the different contributions to the total polarization loss.

Figure 19 illustrates a breakdown of losses for a fuel electrode-supported Ni/YSZ|YSZ|LSM/YSZ-based cell at 850 °C based on the equivalent model depicted in Fig. 18 in the current density interval from -0.5 to 0.5 A/cm². Notice

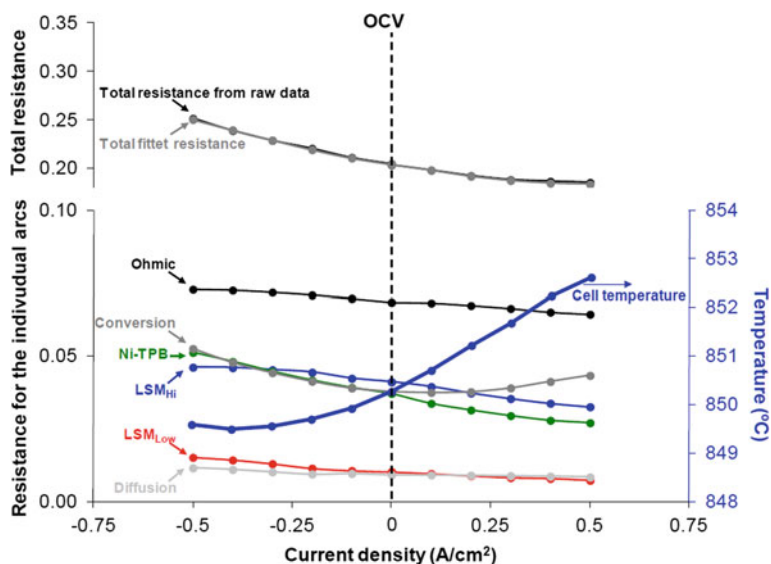


Fig. 19 Current density dependency of the different contributions to the total ASR of an fuel electrode-supported Ni/YSZ|YSZ|LSM/YSZ-based cell. The results are based on the analysis of impedance spectra at 850 °C, applying a H₂O/H₂:50/50 fuel (Ebbesen and Mogensen 2013)

the linearity of the IV-curve (DC characterization) also shown in Fig. 19 in the current density range, in which impedance spectra were recorded (Ebbesen and Mogensen 2013). The linearity of the cell response at the given conditions justify the comparison of results from DC and AC characterization of the cell, and the linearity of the IV-curve reveals that proper ASR can be calculated from the secant ASR from the IV-curve (DC characterization).

A complementary way of analyzing impedance spectra is via distribution of relaxation times (DRT) as described by Schichlein et al. (2002). This method can advantageously be combined with ADIS and equivalent circuit modeling; however, high quality impedance spectra are required for proper DRT analysis. Figure 20 shows an example of a Nyquist plot of impedance spectra and the corresponding DRT plot. As evident from Fig. 20, the DRT plot provides a high frequency resolution that enables clear distinction between the many processes contributing to the total polarization loss in the given full cell test. The area beneath the DRT curve represent the resistance of the cell.

There will be cases where it is not possible to perform a breakdown of the losses of a full SOC based on the measured impedance spectra, e.g., because of overlapping impedance contributions or quality of the spectra. However, there can still be useful information to obtain from the impedance spectra in terms of the ohmic resistance and the total polarization resistance of the SOC.

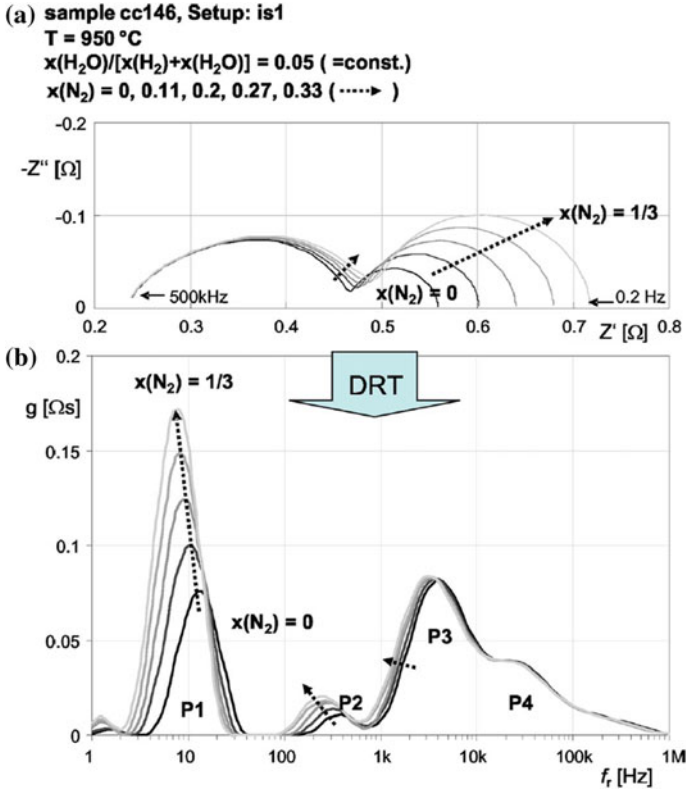


Fig. 20 Nyquist plot of impedance spectra and corresponding distribution of relaxation times (DRT) plot upon dilution of fuel electrode gas with nitrogen for a fuel electrode half-cell at 950 °C (Sonn et al. 2008)

5.3 Concentration Polarization Contributions to the Total Loss

Performance losses from processes like gas conversion and gas diffusion and resistance contributions with a metallic-type dependency (e.g., from metallic connections) are not thermally activated, i.e., they do not follow Arrhenius equation type of behavior. These contributions to the total cell resistance will be the most significant at higher temperature where contributions from the thermally activated processes are the smallest (see e.g., in Table 1). This fact can be used to separate and thereby also quantify, e.g., resistance contributions due to the gas diffusion (non-thermally activated) from resistance contributions due to the electrochemical processes in the oxygen electrode based on MIEC (thermally activated process) which frequency wise can be overlapping at certain test conditions (Leonide et al. 2008; Nielsen and Hjelm 2014).

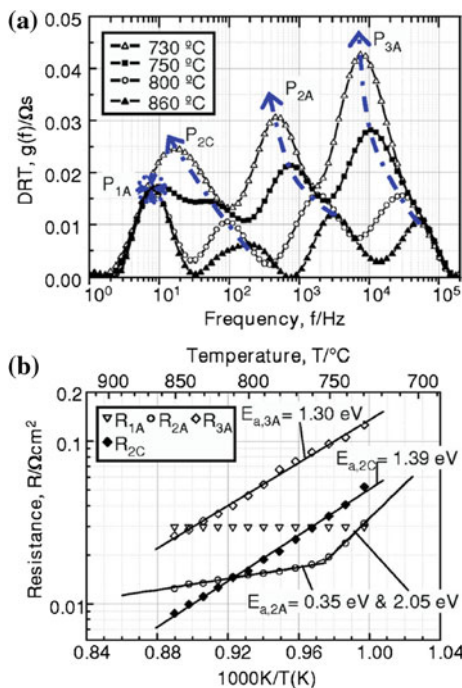


Fig. 21 Temperature dependency of the different contributions to the total ASR of an anode-supported Ni/YSZ|YSZ|LSCF/CGO-based cell (Leonide et al. 2008)

Losses due to gas diffusion (typically observed in the impedance spectrum in the frequency range 10–100 Hz) will of course depend on several microstructural parameters such as porosities, tortuosity, and layer thicknesses as described and modeled by several authors (Jacobsen et al. 2008; Bessler 2005) as well as gas composition (Kromp et al. 2011). The gas diffusion impedance will typically be small, i.e., in the range of 10–40 $\text{m}\Omega\text{ cm}^2$ (Birkel 2012; Barfod et al. 2007) at relevant test conditions for cells with fuel electrode-supported layer thicknesses of $\sim 300\text{ }\mu\text{m}$ and larger for thicker anode support layers.

5.4 Thermally Activated Contributions to the Total Loss

The thermally activated processes include resistance contributions originating from the electrolyte and the two electrodes. The resistance due to the electrolyte (conduction of ions) is often denoted ohmic resistance or series resistance, and from a cell testing and characterization point of view, it is worthwhile to notice that the activation energy for this electrolyte resistance can easily be affected by

non-thermally activated resistance contributions from, e.g., contacting of the cell which will also contribute to the ohmic resistance. Figure 21 shows a breakdown of losses for a fuel electrode-supported Ni/YSZ|YSZ|LSCF/CGO-based cell at OCV (Leonide et al. 2008) in the temperature interval from 760 to 860 °C. The results shown in Fig. 21 are based on the analysis of impedance spectra and illustrate the thermal activation of the electrochemical processes in the fuel and oxygen electrode.

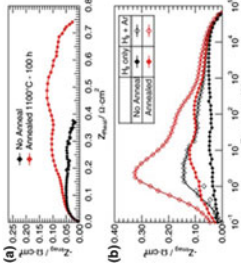
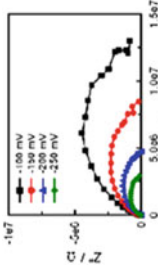
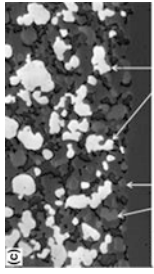
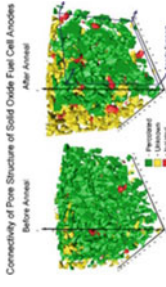
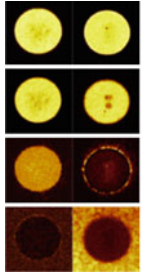
5.5 Complementary Characterization Techniques

The characterization techniques described above are in situ electrochemical characterization of the performance and characteristics of an SOC. It is desirable to supplement the in situ electrochemical characterization with non-electrochemical, and mainly ex situ, characterization of the cells which can provide complementary information, e.g., on cell degradation mechanisms, typically microscopic and spectroscopic methods (Leng 2008).

Electron microscopy is widely used for studying SOC on a nano- and micrometer scale both as feedback for processing optimization and start-up profile optimization (Hauch et al. 2012; Jiao and Shikazono 2015; Jørgensen et al. 2015) and for investigation of aging and degradation of cells (Chen et al. 2013; Liu and Jiao 2005; Hauch et al. 2008; Tietz et al. 2013; Knibbe et al. 2010; Shimura et al. 2014). Besides more traditional, SEM investigation of electrodes which can also include quantitative image analysis (Holzer et al. 2011; Iwanschitz et al. 2012; Faes et al. 2009), more specialized SEM imaging techniques can also be used to gain information on the percolation of, e.g., the Ni network as described by Thydén et al. (2008), and illustrated in Table 2.

In recent years, combined focused ion beam (FIB) milling and SEM imaging have also been used to make 3D reconstructions of electrode structures (Wilson et al. 2006; Shearing et al. 2010a, b; Jørgensen et al. 2015; Shimura et al. 2014; Jørgensen et al. 2010) leading to very detailed and quantitative information on the active TPB lengths, particle size distributions, tortuosity etc. in a given electrode structure. Such data may advantageously be used to investigate an electrode microstructure before and after long-term test and be correlated to the electrochemical characterization obtained during long-term testing. An example of the use of 3D reconstructions of electrodes is given in Table 2. Over the last decade, there have also been several examples of applying transmission electron microscopy (TEM) for the analysis of SOC which enables, e.g., investigation of nano-sized impurities in the electrode structures, oxygen “bubbles” formation in the electrolyte, and formation of undesirable secondary phases, (Knibbe et al. 2010; Hauch et al. 2008; Liu and Jiao 2005) and even examples of in situ studies of NiO reduction using TEM (Jeangros et al. 2012). Besides electron microscopy, techniques such as scanning tunneling microscopy (STM) (Hansen et al. 2013; Khabibulakh et al. 2009;

Table 2 Example of combination of electrochemical characterization techniques combined with complementary non-electrochemical characterization techniques such as SEM, 3D reconstructions of electrodes and ToF-SIMS analysis

Characterization/cell type	Technologically relevant fuel electrode-supported cell	Full cell	Model electrode/LSM microelectrode on YSZ
Illustration of electrochemical performance characterization	p^o		
Description of electrochemical performance characterization	Simple cell voltage measurement over time under current load (technological relevance)	Impedance spectroscopy—for cells annealed differently—provides data for “overall”/average electrode	Impedance spectroscopy for a well-known electrode geometry (enables studies of electrode kinetics)
Illustration of complementary characterization			
Description of complementary characterization	Fast/non-time-consuming microscopy for the analysis of Ni percolation (of test B)	Time-consuming 3D reconstruction of porous structure and its percolation via FIB milling and imaging	ToF-SIMS measurements for a reference (upper) and tested sample
References	Hauch et al. (2011a, b)	Cronin et al. (2011)	Wu et al. (2013)

Cai et al. 2011) and scanning probe microscopy (Wu et al. 2013) have also been used to investigate impurities and secondary phases at the electrode surface structures.

Furthermore, a variety of analysis techniques based on spectroscopy can be and have been applied for SOC characterization such as secondary ion mass spectroscopy, SIMS (Horita et al. 2012; Hansen et al. 2006; Schmidt et al. 2008; Kiebach et al. 2014) and Raman spectroscopy (Li et al. 2012; McIntyre et al. 2015; Kishimoto et al. 2012; Shimazu et al. 2011), and spectroscopy based on X-rays such as energy dispersive spectroscopy, EDS, wavelength dispersive spectroscopy, WDS (Wiedenmann et al. 2010), and X-ray photoelectron spectroscopy, XPS (Backhaus-Ricoult 2008; Utz et al. 2011). To illustrate and highlight how electrochemical measurements have recently been combined with complementary techniques for SOC characterization, a couple of examples are given in Table 2.

Table 2 illustrates the strength in applying both electrochemical characterization (DC and AC methods) and complementary characterization techniques, as well as illustrating the different levels of complexity both regarding the type of cells to test and characterize (model electrodes versus technological relevant full cells) but also with respect to choice of methods for characterization.

The data shown in the second column illustrate a rather extended and complex test matrix in the sense that the tests are performed on full cells—sister cells with identical microstructure but different test conditions. The corresponding analyses of impedance spectra (not shown here, reported elsewhere) during the fuel cell test, i.e., under current load strongly suggest that it is for all 3 tests the Ni/YSZ electrode that degrades. As the impedance data was obtained on full cells including oxygen electrodes, it is valuable to support the impedance analysis by postmortem microscopy investigations from which it was evident that the fuel electrodes degraded and also that the degradation was related to a decreased level of percolation for the Ni network, possibly due to impurities (Hauch et al. 2011a, b).

The data in the third column illustrate more complex electrochemical test in form of impedance, however, measured at OCV, and the impedance data is supplemented by 3D reconstruction of the electrode structure.

The data in the right most columns provide an example with a very simple test geometry (model electrode), and this enables a more direct comparison of the electrochemical characterization results with precise microstructural measurements of changes in the structure/composition upon polarization.

Comparing the results reported in Table 2 in horizontal direction, it also illustrates variety in electrochemical characterization techniques (in situ characterization) and microstructural characterization techniques (ex situ). Cell voltage curves (and polarization curves) represent simple but valuable overall information on the performance of a cell, whereas the AC characterization (impedance spectroscopy) provides more detailed information (3rd and 4th column); however, the analysis of the different contribution to the overall resistance of the cell can be complex when applied for full cells (3rd column). The examples on microstructure characterization in Table 2 illustrate different techniques having different pros and cons. The ToF-SIMS analysis (4th column) provides high accuracy measurement on a simple geometry, where as the 3D reconstruction provides very accurate information on a

more complex structure; however, this technique can be somewhat time-consuming and only a tiny part of the electrode structure is investigated (which is hopefully representative for the entire electrode structures). The 2D SEM analysis as illustrated in the 2nd column provides a simple and fast investigation of the electrode structure and can be done for several hundred micrometer within less than a day, which provides the possibility of investigating, e.g., gradients in coarsening or impurity level along, e.g., a flow direction in a technologically relevant large scale cell.

6 The Problem of Gas Leakage in Cell Testing

A significant source of error in cell testing can be due to leakage. This section first describes possible origins and observed effects of leakage followed by a paragraph on assessment of the size of leakage.

6.1 *Origin and Effects of Leakage*

Gas leakage of air or oxygen from the oxygen electrode compartment to the fuel electrode compartment can happen either along the edges of the cells due to improper sealing or from the oxygen electrode through the cell via cracks or holes in the electrolyte (Rasmussen et al. 2008). Normally, the air flow is much higher than the fuel flow, and thus, the pressure is slightly higher in the oxygen electrode compartment than in the fuel electrode compartment leading to gas leakage from the oxygen electrode compartment to the fuel electrode compartment rather than vice versa. A gas leakage of air to the fuel electrode compartment will inevitably lead to lowering of the OCV, and attention should be paid to the measured OCV compared to the theoretically calculated Emf from the Nernst equation. Furthermore, one should notice that a large localized leak of air to the fuel electrode compartment, e.g., at the rim/edge of the cell causes a local increase in temperature due to the hydrogen combustion, and such temperature increase may not necessarily be registered if the thermocouple is positioned away from the leak (e.g., at the center of the cell). However, the increased temperature will decrease the cell resistance locally. Large leaks in the cell or in seals cause inhomogeneous gas composition over the cell, even at OCV. Another possible explanation for differences in OCV compared to the calculated Emf is in principle electronic leak through the electrolyte. If the electronic conductivity in such a leak decreases with cell voltage, a flat IV-curve may be obtained and care should be taken in the interpretation of such IV-curve measurements.

6.2 Assessment of the Size of Gas Leakage

The effect of gas leaks on cell tests may be separated into two components: (1) a loss of driving force (Emf) caused by oxygen entering to convert hydrogen into steam, and (2) a volumetric loss of fuel affecting the fuel utilization caused by a pressure gradient versus ambient. A gas leak may be before, in, or after the cell in a test set-up fuel gas line (see e.g., Fig. 10). Leaks before the cell affect the intended fuel composition and quantity; however, a true average composition of the gas may be obtained from the measured OCV for the cell. Leaks after the cell are seldom important to the cell test, unless of course analysis of the outlet gas composition is carried out. A cell test set-up including measurement of the pO_2 in the inlet gas stream, measurement of the cell's OCV, the pO_2 in the outlet gas stream, and appropriate temperature measurements provides reasonable monitoring of inlet gas composition (in relation to expected composition based on set gas flows via mass

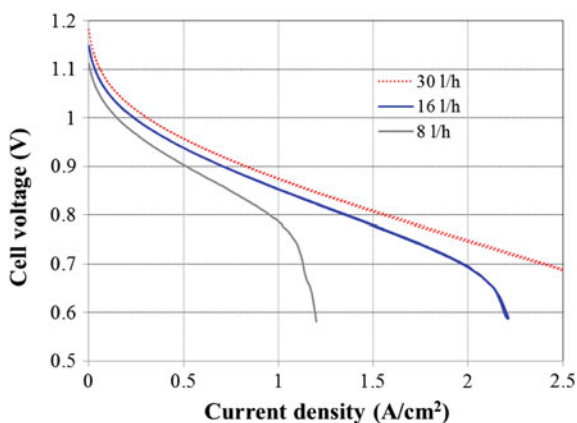


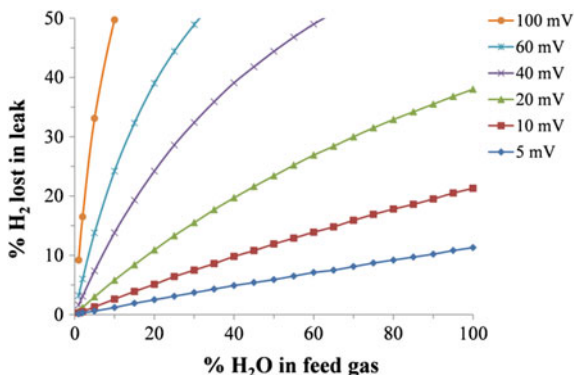
Fig. 22 IV-curves at 850 °C, 140 l/h of air to the oxygen electrode and dry H_2 to the fuel electrode for a Ni/YSZ|YSZ|CGO/LSC/CGO-based cell. H_2 flows, theoretical Emf, measured pO_2 at fuel gas inlet and outlet, and OCV are given in Table 3 together with the calculated percentage of H_2O in the gas stream at the gas inlet and over the cell. (Data from DTU Energy; Technical University of Denmark)

Table 3 H_2 flows, theoretical Emf, measured pO_2 at anode gas inlet and outlet, and OCV together with the calculated percentage of H_2O in the gas stream at the gas inlet and over the cell for the two IV-curves depicted in Fig. 22

H_2 flow (l/h)	Emf (theo)	pO_2 -inlet (mV)	OCV (mV)	pO_2 -outlet (mV)	% H_2O in inlet gas	% H_2O in gas in electrode	FU at max. current
8	1218	1163	1131	1116	0.7	1.4	97
16	1218	1163	1163	1150	0.7	0.7	96

The pO_2 values are given as voltage against air, and FU is the fuel utilization

Fig. 23 Deviation of 5, 10, 20, 40, 60, and 100 mV between measured OCV and theoretical Emf translated into percent loss of feed hydrogen as a function of H_2O concentration in fuel gas, $\text{H}_2/\text{H}_2\text{O}$ mixtures at 700 °C



flow controllers), and leaks affecting the cell's OCV (deviation from Emf) and total gas leaks from inlet to outlet and enables the quantification of possible leaks. Figure 22 shows IV-curves measured on a planar fuel electrode-supported Ni/YSZ|YSZ|CGO_{ba}|LSC/CGO-based cell with different H_2 flows to the fuel electrode, and Table 3 lists the corresponding values for measured pO_2 in inlet and outlet gasses and corresponding percentage of H_2O in the fuel electrode gas stream at the inlet and over the cell. From such simple characterization, one can assess the size of the gas leak and a measure of the maximum fuel utilization obtainable for the tested SOC. A clear effect of the fuel starvation is observed at current densities of around 2.1 and 1.05 A/cm^2 , respectively. Extended characterization should include similar flow variations for the gas to the oxygen electrode.

In more general terms, assuming oxygen leakage into the fuel compartment, one can estimate the loss of fuel based on observed OCV deviation from the theoretically expected Emf as calculated from the Nernst equation. Figure 23 shows a number of such curves at 700 °C for a $\text{H}_2/\text{H}_2\text{O}$ gas feed. From a given feed percentage of H_2O , the loss of H_2 can be estimated from the Emf–OCV difference. On the other hand, reading the curve in another way, a given loss of H_2 , e.g., 6 % of the feed causes a loss of driving force of 60 mV if the feed contains 2 % H_2O , whereas for a feed gas with 50 % H_2O the loss is only 5 mV. Such calculations illustrate the effects of gas leakage, and the effects are closely related to the chosen cell test conditions and highlight the importance of thorough measurements of gas compositions. There will rarely be leak from the “low-flow” fuel electrode side of the cell to the “high-flow” oxygen electrode side of the cell, and on top of that the potential of the oxygen electrode gas is far less sensitive to reaction of O_2 with H_2 and production of H_2O , e.g., an injection of 5 % H_2 in the air stream will only reduce the potential by less than 7 mV at 700 °C.

7 Summary

This chapter has considered the main types of electrochemical tests which have been applied to SOC and has outlined the main issues which require detailed attention for obtaining meaningful test results for electrodes and cells. One important aspect in the electrode testing is to assure a correct geometry in three-electrode set-ups. This is very difficult in practice in case of electrode-supported cells with thin electrolytes. Unfortunately, even testing the individual electrodes in sound geometry set-ups is not a perfect procedure either, because the sum of the contributions from individual cell components to the cell resistance does not add up to the actually measured total cell resistance. This is probably due to the differences in the fabrication of the special cells for electrode characterization and the practical full cells.

In the description of recommendable test geometries and set-up, such issues as proper probing of voltages and temperatures was also treated along with the complex matter of impurities, and their—often undesirable—effects on SOC cell test results.

As described in this chapter, it is recommended that cell test results be reported in a way that makes it easy to derive ASR from the IV-curves. Sufficient information should be provided so that the ASR values can be corrected for the effects of finite fuel utilization. Also, the choice of fuel composition should preferably reflect real cell operation conditions. The ASR should be derived using the Emf and a cell voltage in the range of 0.5–0.7 V and its corresponding current density. In case of a grossly non-linear IV-curve, a differential ASR value is of little practical use.

Furthermore, the description of electrochemical characterization methods for electrodes and cells was provided including characterization via DC methods (polarization curves) and AC methods (impedance spectroscopy), as well as an overview of complementary non-electrochemical characterization techniques relevant to SOC characterization. In the description of impedance spectroscopy for characterization of full SOC, introduction to and examples of break down of polarization losses were also given. Finally, the issue of leakage which can cause significant errors in performance data have been treated. Especially in the case of gas leakage, cells can easily be at a higher temperature than their surrounding environment, causing cells to give better apparent performance than individual electrodes tested under better controlled conditions. Also, the gas composition at the electrodes (usually at the fuel electrode) may be different from the intended composition in case of gas leakage. A method for estimating the size of the gas leakage has been presented here.

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