PAPER

Effect of direct-current operation on the electrochemical performance and structural evolution of Ni-YSZ electrodes

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Abstract

The effect of electrolysis operations on Ni-YSZ fuel electrode stability was studied at different current densities and fuel mixtures during 1000 h life tests. For a typical electrolysis mixture of 50% H₂/50% H₂O and 0.6 A cm⁻² current density, cell ohmic resistance values were reasonably stable and no structural changes occurred. However, for more reducing conditions (97% H₂/3% H₂O), increasing the current density above 0.4 A cm⁻² increased the ohmic resistance accompanied by significant electrolyte degradation including fracture and void formation at grain boundaries. Numerical analysis was carried out to determine the effective oxygen partial pressure across the electrolyte. The results show that the oxygen partial pressure values at high current density and low steam content may be low enough to reduce zirconia to form a Ni-Zr alloy product, initiating the observed electrolyte structural degradation.

Introduction

Solid oxide fuel cells (SOFCs) have been extensively developed for efficiently converting fuels into electricity [1], but recent interest has also focused on solid oxide electrolysis cells (SOECs) [2–4]. This is largely driven by the increasing utilization of intermittent renewable electricity production; SOECs can be used for storing renewable excess electricity by converting to various chemical fuels (so-called ‘Power-to-X’), thereby avoiding the need to curtail renewable resources [5–7]. Among different energy storage methods, SOECs have the potential for relatively high efficiency, low energy storage cost, and scalable energy capacity [8].

Long-term durability is a crucial challenge for the commercialization of SOECs, because a number of new degradation mechanisms have been observed that are not normally seen in SOFC mode [9]. Many SOEC studies have focused on oxygen electrode degradation [10–12], where the high oxygen activity present during electrolysis leads to fracture, delamination, or oxygen bubble formation. In addition, degradation has been associated with structural changes observed in the Ni-YSZ fuel electrode and the yttria-stabilized zirconia (YSZ) electrolyte. Depletion of Ni from the Ni-YSZ near the interface with YSZ has been observed [13]. Void formation on YSZ electrolyte grain boundaries has been observed after electrolysis life tests [14–16], along with an increase in ohmic resistance [16]. Another report showed significant fuel-electrode polarization resistance increases for current densities > 1 A cm⁻² at 800 °C or 850 °C, associated with the formation of ZrO₂ nanoparticles on Ni particles; this was explained by zirconia reduction at the very low oxygen partial pressures p_O₂ that can arise during electrolysis [17]. Most tests of steam electrolysis have been carried out in relatively high steam contents ranging from 40% to 90%. It is desirable, from a system perspective, to achieve high steam utilization, and hence low steam conditions near the fuel outlet, but few life tests have been done under such conditions.

Here we report results from life tests carried out with 97% H₂/3% H₂O at different constant current values. This very low steam content is unlikely to be present in a steam electrolyzer, but it is used here as a way to accelerate degradation effects resulting from highly reducing conditions. A comparison test at 50% H₂/50% H₂O is also reported. Symmetric Ni-YSZ/YSZ/Ni-YSZ cells were utilized for these fundamental studies of degradation for a few reasons: (1) effects arising from the oxygen electrode, present in full cell tests, are
eliminated, simplifying the interpretation of the results; (2) effects arising from interconnectors and gas seals are not present; and (3) one electrode in the symmetric cell operates in electrolysis mode, while the other is in fuel cell mode, allowing a direct comparison of these operating modes. The cells were fabricated by tape casting and co-sintering processing methods similar to the widely-used Ni-YSZ-supported full cells, with similar microstructures and layer thicknesses; thus, the results should be relevant to these full cells. Cell performance changes and electrochemical impedance spectroscopy (EIS) results are discussed with input from cell microstructural observations. Numerical analysis of the effective oxygen partial pressure across the cell was carried out and used to help explain observed degradation effects.

**Experimental**

The symmetric cells were processed as described elsewhere [18]. A ~ 25–30 μm thick YSZ electrolyte was sandwiched between identical Ni-YSZ electrodes, each with an ~20 μm thick functional layer with composition 50:50 wt.% NiO:YSZ and a ~ 150 μm thick support layer. The support layer had the same composition as the functional layer, but an additional pore former was added to the support layer to improve gas transport. These structures were prepared by tape casting, laminating, and co-sintering in air at 1400 °C for 4 h. Note all the thicknesses given above were measured after sintering. All cells were then heated in a 5% H2/3% H2O/92% Ar gas mixture at 800 °C for 3 h to fully reduce NiO.

Life tests were performed at 800 °C in a 97% H2/3% H2O or 50% H2/50% H2O gas mixture. In both cases, hydrogen gas was passed through a bubbler containing pure deionized H2O. For 97% H2/3% H2O, the bubbler was kept at 28 °C, whereas for 50% H2/50% H2O the bubbler was maintained at 82 °C. Direct current was applied using a Keithley source meter controlled by Labview. The current density j was maintained constant in each long-term test, at 0.4, 0.6 or 0.8 A cm −2. The current was paused at ~24 h intervals for EIS measurements from 106 to 10−1 Hz at open circuit voltage using a Zahner IM6 electrochemical testing station. Half-cell area-specific Ohmic resistance (Rohm) values are estimated from the Nyquist plots as the first intercept with the Zreal axis, while polarization resistance (Rpol) is taken as the difference between the high and low frequency intercepts, and dividing the measured impedances by 2 and normalizing to the electrode area. These intercept values, used for expedience due to the large amount of EIS data, agree within ±2.5% error of those obtained from the EIS fitting (mostly due to inductance effects at high frequency), and the error is consistent throughout the tests. Note that it was not practical to use a potential probe in these thin electrolyte cells, and the division by two effectively assumes that both electrodes have the same impedance; while this is presumably true for the identical electrodes at the beginning of the life test, if the two electrodes degrade differently, this assumption may be flawed. In particular, since no degradation was expected or observed on the fuel cell side of the cells, it is assumed that observed increases in cell operating voltage and resistance arose solely from the electrolysis–side electrode.

Fracture cross-sections of selected cells were imaged by a scanning electron microscope (SEM; Hitachi SU8030) at 20.0 kV accelerating voltage using a secondary electron detector. 3D tomography and electron channeling contrast imaging (ECCI) were acquired, from cells that were prepared by cross sectioning, epoxy infiltrating, and mechanically polishing, in an FEI Helios Focused ion beam—scanning electron microscope (FIB-SEM). 3D tomography was done with an accelerating voltage 5.0 kV and the backscattered electron detector. Image segmentation and 3D reconstruction of serial sectioning images were done as described elsewhere [19]. ECCI across the YSZ electrolyte was first started with surface cleaning under focused ion beam, with accelerating voltage of 30 kV and high beam current of 2.8 nA. After that, imaging was also accomplished under focused ion beam with a 30 kV accelerating voltage, low beam current at 28 pA and in secondary electron mode. This ECCI imaging provides good grain orientation contrast [20].

**Results**

**Electrochemical characterization**

Figure 1 shows the cell voltage versus time measured during the life tests done at different current densities and gas compositions. The cell operated in 50% H2/50% H2O and j = 0.6 A cm −2 showed an initial voltage that decreased with time early in the life test and then stabilized (aside from the test interruption at ~350 h). The tests done in 97% H2/3% H2O showed higher voltages that increased rapidly in the first 50 h but then more slowly. For the j = 0.4 A cm −2 case, the voltage stabilized after ~500 h, but the degradation continued throughout the tests for the higher j. Note that extended break-in periods of a few hundred hours have been observed for cells with Ni-YSZ fuel electrodes [17], and the early-stage voltage changes observed here are likely such an effect. However, the continuing voltage increase, seen in the low steam and higher j conditions, appears to indicate cell degradation.
Figure 2 shows an example of EIS data measured at zero current during brief interruptions of the life test carried out at 0.8 A cm\(^{-2}\) in 97\% H\(_2\)/3\% H\(_2\)O. The Nyquist plot shows a slight increase in the low-frequency intercept with time, consistent with the cell voltage increase seen in figure 1. The high frequency intercept increases more rapidly with time, indicating that the ohmic resistance \(R_\Omega\) increases with time while the polarization resistance \(R_P\) decreases with time. The EIS data were fitted with an equivalent circuit model containing an inductance and a resistor in series with two R-CPE elements (presented in figure 2) as previously.
used to model Ni-YSZ electrodes [18], [21, 22] The fit agrees very well with the EIS data as shown in figure 2. A breakdown of the fit is illustrated for the case of the cell tested for 963 h. The RQ response at ∼10 Hz can be associated with the gas diffusion process, and the ∼100 Hz RQ response is related to an electrochemical process in the Ni-YSZ electrode. From the Bode plot in figure 2, the response centered at ∼10 Hz decreases with time, mostly after ∼400 h, and shifts to a lower frequency. The response at ∼100 Hz also decreases with time, but shifts to higher frequency.

Figure 3 plots $R_Ω$ (a) and $R_P$ (b) versus time for all four life tests, taken from EIS data such as that shown in figure 2. The initial $R_Ω$ values (figure 3(a)) are similar, since the electrolyte thicknesses were all 25–30 μm. For the cell operated at 0.4 A cm$^{-2}$ in 97% H$_2$/3% H$_2$O, $R_Ω$ increased in the first 400 h and then became relatively stable. On the other hand, for the tests in 97% H$_2$/3% H$_2$O at 0.6 and 0.8 A cm$^{-2}$, $R_Ω$ increased steadily during the entire test, yielding a factor of 3–4 increase over 1000 h. However, during the test in 50% H$_2$/50% H$_2$O and 0.6 A cm$^{-2}$, $R_Ω$ was fairly stable from 50 to 350 h, abruptly increased at 350 h due to a power outage, and then remained relatively stable throughout the rest of the test. $R_P$ (figure 3(b)) decreased gradually and continuously during all the tests, aside from the artifact introduced by the interruption of the higher-steam test.

**Microstructural observations**

Figure 4 shows fracture cross sectional SEM images of the electrolyte of various cells after life testing. Figures 4(a)–(c) show the low steam tests at increasing $j$, with an un-tested as-reduced electrolyte shown for comparison in figure 4(d). The relatively featureless YSZ fracture surface, almost dense with a few isolated pores in figure 4(d), gives way to an increasing tendency for intergranular fracture with increasing $j$, and finally an opening or crack across the electrolyte near the SOEC electrode for the highest $j$. Figure 4(e) shows that the electrolyte after the higher-steam test is featureless similar to the un-tested electrolyte. Figure 4(f) shows a higher-magnification view of a region near the center of the electrolyte from figure 4(c) (0.8 A cm$^{-2}$) where voids are clearly present along the grain boundaries produced during sample fracture for SEM imaging. These pores presumably weaken the grain boundaries, resulting in the characteristic intergranular fracture.

![Figure 3. Ohmic resistance (a) and polarization resistance (b) measured during life tests performed with 0.4, 0.6, 0.8 A cm$^{-2}$ in 97% H$_2$/3% H$_2$O, and with 0.6 A cm$^{-2}$ in 50% H$_2$/50% H$_2$O (a disturbance from power outage at around 350 h was marked by red arrow).](image-url)
Figure 5 shows cross sectional ECCI images of polished YSZ electrolyte surfaces from the cells. Figures 5(a)–(c) show the low steam tests at increasing \( j \), with an un-tested as-reduced electrolyte shown for comparison in figure 5(d). The ECCI imaging gives good grain contrast but the electrolyte is otherwise featureless except for a few isolated pores in figure 5(d). The intergranular fracture seen in figure 4 is of course not observed in these polished cross-sections, and the grain-boundary pores are not so evident because the grain boundaries are not exposed. Figure 5(a) shows small extended pores on the electrolysis-side of the electrolyte that become more extensive at higher \( j \) (figure 5(b)) and then extend to produce a nearly complete separation in the electrolyte at \( j = 0.8 \text{ A cm}^{-2} \) (figure 5(c)), along with secondary lines of extended pores. Greater than 90% of the electrolyte
Figure 5. Representative polished cross sectional electron channeling contrast (ECCI) SEM images of the YSZ electrolyte after long-term tests in 97% H₂/3% H₂O gas condition at 0.4 (a), 0.6 (b) and 0.8 (c) A cm⁻², in the as-reduced state (d), and after the long-term test in 50% H₂/50% H₂O at 0.6 (e) A cm⁻². For all the images, the SOEC electrode is at the top and the SOFC electrode is at the bottom.
length observed by SEM was separated in this case. Figure 5(e) shows that the electrolyte after the higher-steam test is similar to the un-tested electrolyte, i.e., there are no observable structural changes.

Figure 6 summarizes SEM-EDS data from the cell after the 0.8 A cm$^{-2}$ life test, but taken at a different region. Similar to the image shown in figure 5(c), figure 6(a) shows an extended crack along with two minor crack lines on the SOEC side. The EDS elemental intensity map reveals Ni enrichment along large crack; Ni enhancement is also detected along the smaller crack lines, although it is barely visible in figure 6(b).

Figure 7 presents representative FIB-SEM 2D images from SOEC-side (a) and SOFC-side (b) after the 0.8 A cm$^{-2}$ life test compared with an as-reduced electrode (c). Ni, YSZ, and pore volume fractions versus distance from electrolyte for the SOEC (d), SOFC (e) and as-reduced (f) electrodes, calculated from the FIB-SEM 3D datasets in the four regions indicated in (a), (b) and (c).
YSZ electrolyte (orange box in solid line in a or b). The values were obtained in four equally-partitioned volumes (indicated by the blue dashed lines in a, b and c) in the functional layer. In both the SOEC and SOFC electrodes, there appears to be a slightly lower Ni volume fraction and slightly higher pore volume fraction near the electrolyte, compared with the as-reduced electrode. Given that the present changes are barely beyond measurement errors and are not expected in Ni-YSZ operated as a SOFC electrode, it is unclear whether the Ni depletion is real or is a measurement artifact. Based on prior reports \[13\], one might expect Ni loss near the electrolyte in the SOEC electrode. The lack of an obvious change in the present experiments may be the result of the much shorter life test times of 1000 h, compared to \(\sim\)10 000 h in the prior report.

Figure 8 shows fracture cross sectional SEM images of the Ni-YSZ taken near the electrode/electrolyte interfaces, of both electrodes after test in 3% H\(_2\)O/97% H\(_2\) with 0.6 A cm\(^{-2}\). Nanoparticles are observed on both sides, but their density is higher on the SOEC side than on the SOFC side. Nanoparticles can often be observed in Ni-YSZ after anode-supported SOFC operation; this was explained by the small solubility of NiO in YSZ during high-temperature firing; upon reduction of the electrode, Ni exsolves from the YSZ, forming Ni metal nanoparticles on the surface \[23\]. However, the higher nanoparticle density in the SOEC electrode indicates that some additional process is present.

**Discussion**

Three different microstructural changes were observed here—voids formed primarily on grain boundaries in the YSZ electrolyte, extended separation in the electrolyte near the SOEC-side electrode, and nanoparticle formation within the Ni-YSZ electrode. In the following, each of these changes are discussed, along with their anticipated effect on long-term SOEC degradation. Ni depletion from the Ni-YSZ functional layer, often observed after SOEC life tests, was too small to be definitively confirmed here. This may be explained by the low H\(_2\)O pressure used in these experiments, whereas Ni depletion is typically explained by the formation of volatile Ni-containing compounds in high-steam conditions \[24–26\]. Electrochemical degradation is observed at a current density of \(\geq 0.6\) A cm\(^{-2}\) for 3% H\(_2\)O, but not for 50% H\(_2\)O, suggesting that the low steam conditions accelerate degradation.

Since SOEC fuel electrode evolution has been associated with an extremely low oxygen partial pressure \(P_{O_2}\), a model calculation is used here to determine the \(P_{O_2}\). The model used here has been described in detail elsewhere \[27, 28\]. Briefly, it accounts for oxygen vacancy, electron, and hole transport across the electrolyte in order to calculate the electric potential and \(P_{O_2}\) as a function of position across the electrolyte. Boundary conditions are given by the electrode overpotentials, which are estimated based on the measured polarization resistance values \[27\]. Figure 9 shows \(P_{O_2}\) versus position across the YSZ electrolyte. Electrolysis operation drives \(P_{O_2}\) to lower values on the SOEC side with increasing \(j\), due to higher overpotentials. Shifting from 3% to 50% H\(_2\)O moderates the \(P_{O_2}\) value, both because of the higher effective \(P_{O_2}\) value in the gas phase and reduced electrode overpotentials.

Figure 9 indicates the critical \(P_{O_2}\) where zirconia reduction to form Ni-Zr phases is expected \[17, 29\]; this is shown as a range of values due to the uncertainty in the thermodynamic data. Nonetheless, it can be seen qualitatively that increasing/ or decreasing the H\(_2\)O content can decrease \(P_{O_2}\) into the range where zirconia
reduction can be expected. This zirconia reduction effect was previously used to explain nanoparticle formation in the Ni-YSZ functional layer during electrolysis \cite{17, 29}, and may also explain the present observation of nanoparticles (figure 8). Furthermore, the steady decrease in $R_p$ with time during the life tests (figure 3(b)) may be explained by an increase in TPB density associated with the nanoparticle formation. However, in longer-term SOEC operation the continued re-structuring of the Ni-YSZ electrode could lead to damage near the electrode/electrolyte interface, similar to that observed during reversing current operation where nanoparticle formation is more extensive \cite{18, 29}.

The formation of electrolyte grain-boundary voids is detected primarily because it causes intergranular fractures during sample preparation for SEM observation (figure 4(e)); they are not evident in the polished SEM images (figure 5). Note that these voids should not be confused with the grain-boundary voids observed near the oxygen electrode after the electrolysis operation; these are explained by high $P_O$, values that are not present under fuel-electrode conditions. The mechanism for void formation is unclear, but presumably cation transport is involved. A possible driving force for cation transport is the relatively large electric field present during electrolysis. Such transport would result in cation vacancies that could agglomerate on grain boundaries. Alternatively, cation transport may occur preferentially on grain boundaries due to a higher mobility, resulting in void formation. The grain-boundary void density appears to be too low to play a direct role in the increases in electrolyte resistance observed at higher $P_O$ values (figure 3(a)). However, the continued build-up of voids may weaken the grain boundaries sufficiently to allow mechanical failure after longer-term operation. A similar phenomenon was observed after a SOFC life test, with the ultimate intergranular fracture in the YSZ electrolyte apparently related to Mn enrichment (from the oxygen electrode) at grain boundaries \cite{30}.

The partial or complete separation observed on the SOEC side of the YSZ electrolyte is the likely cause of the observed $R_O$ increase during life tests (figure 3(a)). Similar increases in ohmic resistance are observed to result from oxygen-electrode delamination \cite{14, 15, 31}. However, the mechanism causing the electrolyte separation is unclear.

We explored the possibility that elastic strains, arising from YSZ oxygen loss and associated lattice expansion in the highly reducing conditions near the SOEC electrode (left side in figure 9), could cause fractures. The lattice parameter at vacancy concentration $C_V$ is estimated using a Taylor series expansion about the lattice parameter value at the reference state with concentration $C_V^r$ \cite{32}. The expansion is done using the first and second order coefficients that are estimated by fitting with experimental data for Gd-doped Ceria (GDC) \cite{33}, because the experimental data for YSZ is lacking. Using a relationship between oxygen partial pressure and oxygen vacancy concentration, obtained via a mathematical model described elsewhere \cite{34}, the oxygen partial pressure versus lattice expansion of YSZ is obtained. For a current density of 0.8 A cm$^{-2}$ and 3% H$_2$O, $P_O$, at the SOEC side is $1.45 \times 10^{-29}$ atm (figure 9) and the oxygen loss is 0.74%. The associated lattice expansion of 0.08% relative to the higher partial pressure part of the electrolyte produces a stress of $1.5 \times 10^5$ N m$^{-2}$, given that the Young’s modulus of YSZ is $1.9 \times 10^{11}$ N m$^{-2}$ \cite{34}. According to the discussion in \cite{35, 36}, the critical stress for crack propagation with a flaw size of 1 $\mu$m in YSZ is $\sim 2 \times 10^9$ N m$^{-2}$. Thus, the predicted stresses in our experiments may be too low to cause fracture in YSZ. In order to reach stress levels sufficient to cause fracture by this mechanism, an oxygen partial pressure $<10^{-33}$ atm would be required, a value that could only be reached at very high electrolysis overpotentials.

Figure 9. $P_O$, versus position across the YSZ electrolyte from the SOEC side (left) to the SOFC side (right), for the different current densities and gas compositions in the present life tests.
Other possible explanations arise from the observation of Ni at the electrolyte separation (figure 6(b)). NiO is dissolved in YSZ at ~1–5 mol% levels during high-temperature YSZ/Ni-YSZ co-firing [23]. The reducing conditions during the solid oxide electrolyzer cell operation are known to reduce NiO to metallic Ni [29, 36]; this occurs preferentially at free surfaces such as the pre-existing pores or voids formed during the electrolysis operation, where the energy barrier for nucleation is low. One possible explanation is as follows: for sufficiently low \( P_{O_2} \) conditions, (near the SOEC electrode in figure 9), zirconia can be reduced to form Ni-Zr [17], thereby consuming some of the YSZ electrolyte leading to growth of the voids. With continued growth, the voids may join to form a crack-like object. Note that the electrolyte separations (figures 4–6), occur at the location where the \( P_{O_2} \) value becomes low enough for zirconia reduction (figure 9), providing circumstantial evidence for this proposed mechanism. Another possible mechanism involves tensile and radial elastic stresses at voids that are filled with Ni. In this case, the stress can become large enough to induce crack propagation. A possible source of the stress is the lattice parameter mismatch coherency strain among the different materials (Ni, YSZ, and Ni-doped YSZ) at the void. Note that this crack generation and propagation can be coupled with NiO compositional gradients in YSZ near in newly created crack structures where metallic Ni precipitates [37].

**Summary and conclusions**

Solid oxide electrolysis cell life tests were carried out in different gas mixtures at various current densities on Ni-doped YSZ/Ni-YSZ electrode-supported symmetric cells. The following conclusions can be made:

1. Increases in ohmic resistance during life tests in 97% \( \text{H}_2 \)/3% \( \text{H}_2\text{O} \) at current density \( \geq 0.6 \text{ A cm}^{-2} \) were explained by growth of electrolyte voids leading, in the most extreme case, to extended separations in the electrolyte;

2. The formation of grain-boundary voids in YSZ and nanoparticles in Ni-YSZ were also observed after life tests under 3% \( \text{H}_2\text{O} \) and high current density conditions. While the former apparently had little effect on degradation, and the latter appeared to actually reduce polarization resistance, these effects may lead to degradation during longer-term operation;

3. Model calculations suggest that the microstructural changes are driven by the strong electric potential gradient and the very low oxygen pressure that develops across the electrolyte during electrolysis;

4. Electrolysis operation under low-steam conditions was found to strongly accelerate degradation compared to the high steam electrolysis life test where no degradation was observed. While this can be useful for accelerated testing, it also illustrates the importance of avoiding high steam utilization in practical SOECs.

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