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B0504 (with revisions after EFCF)**Review of Ni migration in SOC electrodes**

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Abstract

Several kinds of Ni migration in Ni-YSZ (yttria stabilized zirconia) electrodes of solid oxide cells (SOCs) have been reported in the literature, which is briefly reviewed in this extended abstract. Emphasis is put onto the migration of Ni away from the YSZ electrolyte in solid oxide electrolysis cells (SOECs) as this is today seen as an important obstacle to the commercialization of SOC systems.

The characteristics of the various migration types are apparently of different nature varying from: (i) Ni-particle migration on top of the YSZ electrolyte in a model electrode to (ii) long distance Ni migration out of porous Ni-YSZ composite cermet electrode in solid oxide fuel cells (SOFCs), and (iii) migration of Ni towards the YSZ electrolyte at 950 °C, but away from the electrolyte at temperatures below 875 °C under otherwise similar conditions in Ni-YSZ cermet electrodes in SOECs.

Apart from temperature, degradation of Ni-YSZ electrodes in SOCs is related to overpotential and partial pressure of steam ($p_{\text{H}_2\text{O}}$), hydrogen (p_{H_2}), carbon dioxide (p_{CO_2}), carbon monoxide (p_{CO}) and oxygen (p_{O_2}) through the influence of these parameters on the mobility of Ni. Actually, the local partial pressure values are most important. Furthermore, impurities from cell materials and reactants may increase mobility of Ni and in general highly affect the long-term durability of SOECs negatively.

Possible Ni migration mechanisms and methods of mitigating the degradation associated with Ni migration are discussed. It is concluded that a lot of further work is necessary in order to describe this Ni migration properly, and the type of work is listed.

Introduction

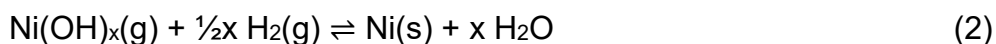
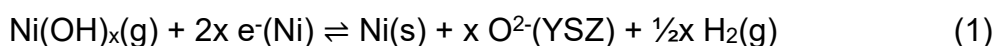
Several kinds of Ni migration in Ni-YSZ (yttria stabilized zirconia) electrodes of solid oxide cells (SOCs) have been reported in the literature. The characteristics of the various Ni migration types seem apparently to be of different nature. One example is fast migration of solid Ni (up to about 5 μm during the first hour) on top of a planar YSZ electrolyte away from a 0.5 V anodic polarized YSZ-Ni-H₂ three-phase-boundary (3PB) in a model electrode at ca. 850 °C [1]. Another one is long distance Ni migration out of Ni-YSZ composite cermet electrodes in solid oxide fuel cells (SOFCs) [2], and a third is depletion of Ni from the active Ni-YSZ fuel electrode layer of solid oxide electrolysis cells (SOECs) next to the YSZ electrolyte and Ni enrichment next to the depleted zone [3].

This paper gives a brief review of selected reports on migration of Ni in and on YSZ. It is not possible to cover all available literature on this subject, but we have endeavored to cover most important aspects of this issue, which in particular is related to the degradation of SOECs. Many researchers think that this SOEC problem is the most important to solve in order to boost SOEC commercialization.

1. Literature data

Ni has a high mobility in the typical operation temperature range of SOCs from 650 – 900 °C. This is most easily observed in the relatively high rate of Ni particle coarsening in Ni-YSZ cermet electrodes. The particle coarsening was previously assumed to be a main reason for the degradation of Ni-cermet electrodes in SOFCs as Ni particle coarsening correlates well with degradation of the electrical conductivity of cermets [4,5]. Pihlatie et al. [5] studied the Ni particle growth in two atmospheres: 1) p(Ar) = 0.70, p(H₂) = 0.15, p(H₂O) = 0.15 bar, and 2) Ar exchange with He, but with same H₂ and H₂O partial pressures as in 1). The temperature was 850 °C and the electric conductivity of the cermets decreased by more than 50 % within less than 50 h. It was found that the grain particle growth was dependent on type of inert gas applied, i.e. the Ni-particles grew faster in the gas with He than in the one with Ar. A change of a majority of inert gas from a heavy atomic weight to a low one will increase the diffusion rate of minority species in the gas. Thus, this result strongly indicates that at least part of the observed Ni particle growth and Ni-Ni contact loss originates from gas phase Ni transport.

This high mobility of Ni has also been observed in migration of Ni away from its original and intended position in the Ni-YSZ cermet electrode. Figure 1 summarizes the main types of Ni migration behavior observed in SOC cells produced by DTU [6,7]. At current density lower than $\pm 0.25 \text{ A cm}^{-2}$ (low overpotential) Ni does not seem to migrate over periods up to a few thousand hours. At high current density more than $\pm 1.0 \text{ A cm}^{-2}$ various migration processes take place. In SOFC mode and in SOEC mode at 950 °C, Ni migrates down the pH₂O gradient, which in this context is the same as Ni migration down the electrical potential gradient in YSZ. This behavior can be rationalized by the formation of surface or gas phase species of Ni(OH)_x, x = 1 or 2.



Under relatively reducing conditions x may be 1 and under more oxidizing conditions x may be 2, i.e. $x = 1$ is dominating in SOEC mode and $x = 2$ in SOFC mode.

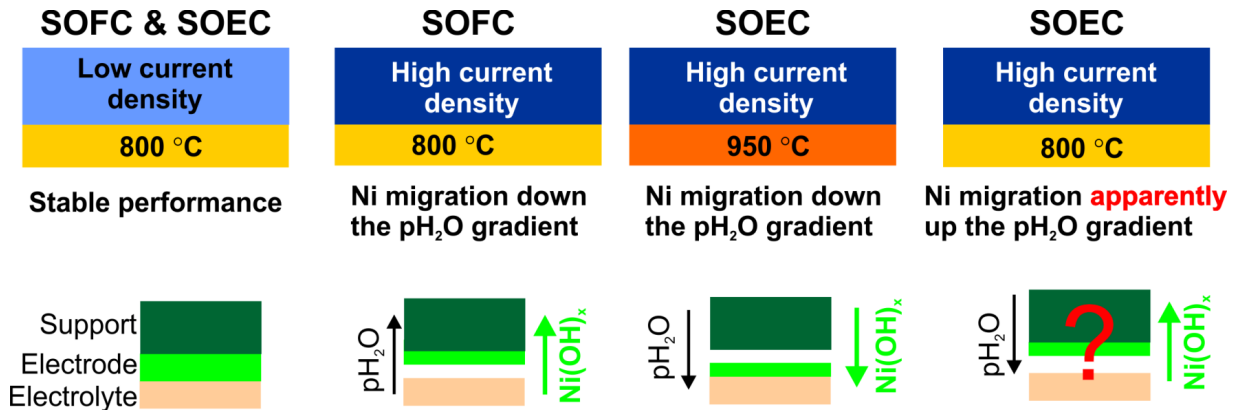


Figure 1: Summary illustration of observed migration of Ni in Ni-YSZ cermets in SOEC and SOFC based on [6,7]. “Low current density” means the range between -0.25 and $+0.25$ A cm^{-2} , “high current density” means current density with numeric value above 1 A cm^{-2} .

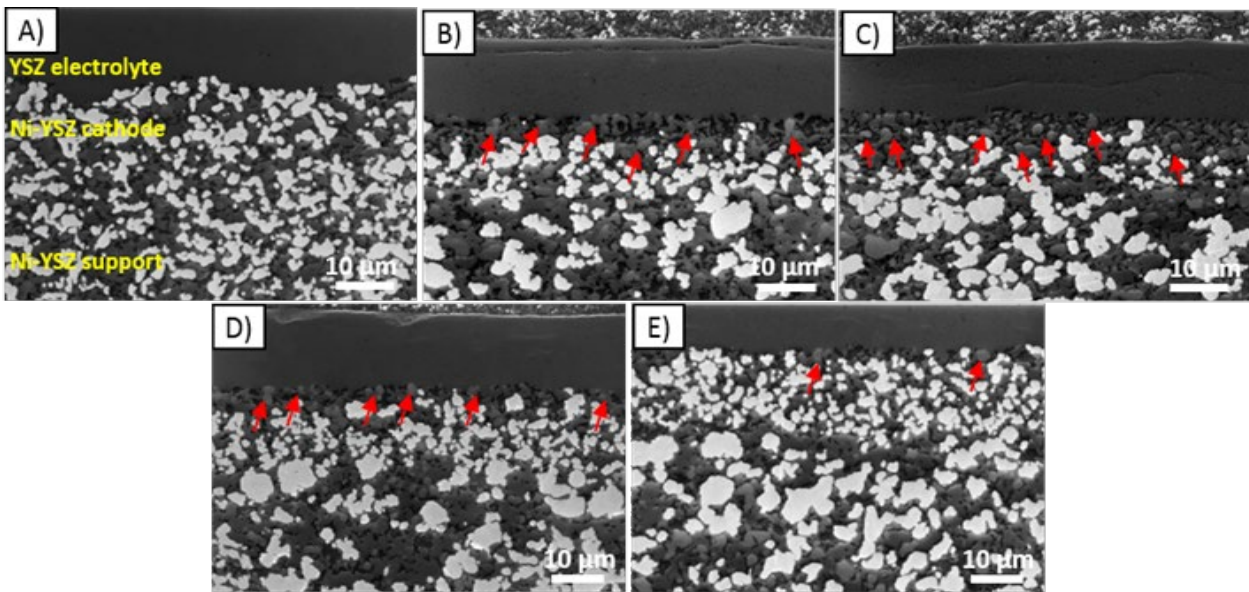


Figure 2. Low-voltage SEM micrographs of fine-structured Ni-8YSZ active electrode between the coarse Ni/YSZ support and the 8YSZ electrolyte. A) not tested, and B) to E) after galvanostatic test at -2.0 A cm^{-2} , inlet gas: 45 % H_2O + 45 % CO_2 + 10 % H_2 , 60 % (H_2O + CO_2) conversion, temperature: 865 °C increasing to 875 °C. B) H_2O - CO_2 - H_2 inlet and C) outlet tested 678 h, D) inlet and E) outlet tested 138 h. Bright colored particles: interconnected Ni; light grey: uncontacted Ni; dark grey: YSZ; black: pores. Red arrows point to uncontacted Ni. The cell voltage of the 678 h test was below 2.0 V and the 138 h test below 1.8 V. From Tao et al. [9] with permission from Elsevier.

At temperatures below 900 °C in SOEC mode, Ni is observed to migrate away from the electrolyte, which apparently is up the $p(H_2O)$ gradient and the electrical potential gradient in the YSZ phase of the cermet. Figure 2 shows a typical scanning electron micrograph (SEM) of the region of the Ni-YSZ electrode next to the YSZ electrolyte.

The overpotential of the fuel electrode (cathode in SOEC) and the current density are by nature significantly higher at the steam/CO₂ inlet than at the outlet in case of high reactant utilization. Thus, Figure 2 reveals that the migration of Ni is driven by overpotential and possibly also by pH₂O.

This SOEC degradation phenomenon has been studied by many research groups [3,8-21] as this seems to be a serious problem for SOEC durability. In many cases, the degradation related to Ni migration continues with an unacceptably high rate over several hundreds of hours, and the overall conclusion from all these studies is that the Ni migration may continue over several thousands of hours, fortunately with a decreasing rate, and in many cases it almost stops after a few thousand hours. In a paper that reports a 9000 h SOEC test at 775 - 782 °C and -1 A/cm², D. The et al. [3] state: "The most severe performance loss of the cell is caused by depletion of nickel in the cathode followed by an agglomeration in the inner part. Consequently, the electrolyte grows with nickel depletion and the electrochemical active 3PB area is reduced. Calculations for a depleted zone of 10 μm yields a high ohmic overpotential of $\eta_{ohm} = 0.17$ V for a current density of -1 A/cm²". This opinion seems to be shared by many SOEC researchers.

Loss of contact between Ni and YSZ particles and between Ni-particles are observed together with Ni migration out of the active SOEC fuel electrode layer at electrolysis current densities higher than 1 A cm⁻² [21]. If loss of contact between Ni and YSZ particles happens before the start of the Ni migration then the Ni migration will also in this case be a migration down to the most negatively polarized Ni particles [7].

A more direct way of measuring the Ni migration was applied by Jiao and Shikazono [1,22]. Using model electrodes with microns thin planar Ni patterns on planar YSZ surfaces they studied the Ni migration as function of potential and found that in case of cathodic potential (SOEC mode) the Ni did not migrate at all, whereas in case of anodic polarization, the Ni migrated down the electrical potential in similarity with the behavior of the anodic polarized Ni-YSZ cermet in SOFC mode and cathodic polarized Ni-YSZ at 950 °C. This behavior was previously reported by other researchers, who used point Ni electrodes on YSZ pellets, e.g. [23,24]. Furthermore, Jiao and Shikazono [22] found that the interface free energy between Ni and YSZ was positive under cathodic polarization, i.e. Ni detached spontaneously, whereas the interface energy became more negative the more the interface was anodically polarized, i.e. stronger attachment with increasing anodic polarization.

An overlooked aspect in almost all articles about Ni migration on YSZ and in Ni-YSZ cermets is that YSZ surfaces and grain boundaries as well as Ni-YSZ interfaces have thin monolayers of impurities. This is extremely difficult to avoid since the trace impurities tend to segregate to the surfaces, and it takes dedicated surface science techniques such as XPS or TOF-SIMS in order to observe such thin films [25]. YSZ raw materials that are regarded as pure may contain relative high levels of S and Cl. In a batch of 8YSZ a content of 200 ± 15 ppm S and 5700 ± 200 ppm Cl was measured by glow discharge mass spectrometry [26].

Likewise, trace impurities in the gas stream can, within a few hundred hours of SOEC testing, lead to detrimental damage of the network properties for the Ni-YSZ cermet electrodes due to Ni migration, as reported in [27] where this subsequently led to carbon deposition. Studies following this work showed that it was gas stream sulfur-based impurities

in quantities of 15-20 ppb in the CO₂ feed for the CO₂ electrolysis that initiated the severe degradation.

2. Mechanism considerations

Previously, we have hypothesized that the migration of Ni away from the YSZ|Ni-YSZ electrolyte|electrode interface was caused by loss of contact between Ni- and YSZ-particles in the volume of the Ni-YSZ cermet next to the electrolyte, followed by NiOH migration towards the new 3PBs with lowest electrical potential formed just outside the layer with loss of Ni-YSZ particle contact [7].

This hypothesis needs to explain 1) why the loss of Ni-YSZ contact happens, and 2) how it is possible for Ni to migrate so fast? We calculated very low partial pressures of Ni species at relevant test conditions using available thermodynamic data [5,6]. If this is to explain the observed Ni migration rates, it would require exorbitantly high diffusion rates.

Regarding 1), the loss of Ni-YSZ contact, this seems to be well explained by the spontaneous Ni detachment observed by Jiao and Shikazono [22]. The apparent Ni-YSZ interface energy may vary a lot depending on the impurities present at the interface. Often silica containing interface films are observed on YSZ. Such surface or interface films may be electrochemically reduced under SOEC conditions and thus separate Ni and YSZ particles, in similarity with the proposal by Tong et al. [17] (but the Ni-YSZ electrode potential will generally not be low enough to reduce zirconia).

Regarding 2), the migration rate of Ni is faster than we can explain by the calculated very low partial pressure of gaseous Ni(OH)_x species. Other explanations are surface diffusion of Ni species, or solid Ni migration driven by electrical potential gradients along YSZ [20,22,28]. These latter explanations cannot, however, explain the migration over very long distance out of the SOFC cell [2], or the effect of type of inert gas [5]. This raises the questions: can we think a) of reasons for why we calculate too low partial pressures of Ni-OH species, or b) of other gaseous Ni species that may have a higher partial pressure at SOEC operation temperature?

Regarding a) the available thermodynamic data for Ni - H₂O at temperatures above 650 °C seems not very well explored, so unknown species or unknown properties cannot be ruled out. Regarding b) combinations of Ni with impurities like sulfur or phosphor resulting in more volatile compounds, such as NiSH or NiPH₂, can be imagined and should be explored. Based on this we made a slightly modified picture compared to our previous one [7] to explain the migration away from the bulk YSZ electrolyte. This is illustrated in Figure 3, in which we have denoted the migrating Ni-species NiX, because impurity containing species totally different from NiSH or NiPH₂ may be responsible. We do not know yet what X can be, and e.g. what will the NiX be in case of a CO₂ electrolysis test with no H₂O/H₂ present? We imagine that NiX will migrate away from the non-polarized Ni particles in the "lost contact" layer and reduced again at the 3PBs of the strongly negative polarized Ni-particles in the "intact layer" that are in contact with YSZ, which is in ionic contact with the electrolyte, see Figure 3 [29].

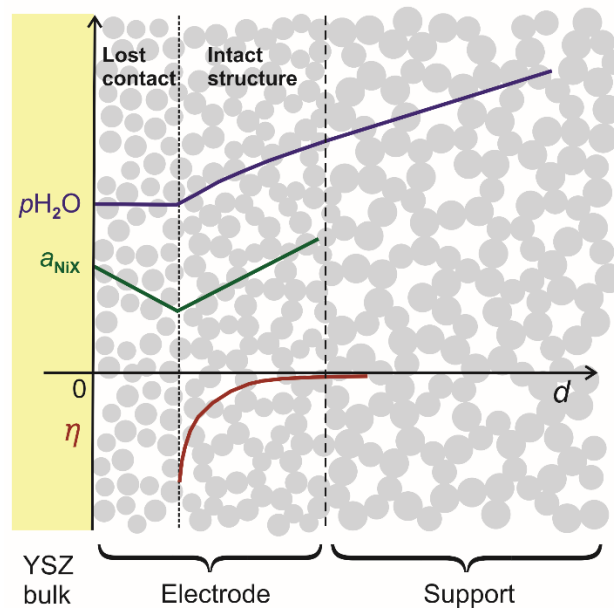


Figure 3. Sketch of qualitative variation of three important parameters away from the surface of the bulk electrolyte ($d = 0$) through the two zones of the as-fabricated SOEC active cathode (Electrode), which has been separated into a zone with partially lost contact between Ni and YSZ particles and a zone with intact structure. d is the distance from the bulk YSZ electrolyte, η is the local overvoltage of Ni particles, a_{NiX} is the activity of NiX species, which has its minimum at the lowest potential at the inner points of active 3PB, and p_{H_2O} is steam partial pressure, which is indicative of the redox potential. From [29] with permission from Electrochemical Society.

3. Alleviation of the Ni migration problems

Fortunately, several methods to mitigate the Ni migration problems have been reported. Below a number of proven and hypothetical methods are listed. Also a list of research that should be performed in order to be able to describe the Ni migration quantitatively is given. According to our hypothesis, the Ni migration related degradation of SOECs is caused by the loss of contact between Ni and YSZ particles as well as between Ni particles in the Ni-YSZ cermet electrode.

Hauch et al. [27] observed that Ni migration in the active Ni-YSZ electrode layer is caused by gas impurities in case of electrolysis of dry CO_2 . This indicates that proper gas cleaning can hinder or at least mitigate the observed degradation and be an important factor in maintaining the desired Ni-YSZ and Ni-Ni network properties. Possibly, cleaning of the cell raw materials will also help.

Infiltration or other ways of adding a mixed ion and electron conductor like Gd_2O_3 doped CeO_2 (CGO) seems to alleviate the Ni migration related SOEC degradation as nanoparticles to the Ni-SZ-composite [17,30]. CGO is a mixed ionic and electronic conductor in reducing atmosphere and decreases the polarization resistance of the Ni-YSZ 3PBs, which in turn decreases the overvoltage of the electrode. By infiltrating CGO nanoparticles into the Ni-YSZ electrode, Ovtar et al. [30] reduced the cell voltage degradation rate from 699 mV kh^{-1} for the bare Ni-YSZ electrode to 66 mV kh^{-1} for the infiltrated electrode. Even after significant degradation, it is possible to reactivate cells by infiltrating Ni-YSZ cermet of the cell with

CGO [31], and re-activation of degraded Ni-YSZ cermet anodes is possible by subjecting it to reverse current pulses that form zirconia nano-particles [32].

Instead of infiltration of CGO nanoparticles, YSZ may be substituted with CGO in the active fuel electrode, i.e. use a Ni-CGO composite, in order to broaden the 3PBs as much as possible. An SOEC with such a fuel electrode was operated during 23,000 h in the steam electrolysis mode, of which 20,000 h were with -0.9 A cm^{-2} . The Ni-CGO electrode had no visible damage of the H_2 electrode after testing [33]. However, during operation this approach may have disadvantages of a mechanically weaker and a less sintering stable active electrode layer. A better way may be use a dopant that turns YSZ into a good mixed conductor.

Addition of a wetting agent that makes the interface free energy more negative is also a possibility. This is probably what happens by addition of Al and Ti in the form of Al_2TiO_5 , which Law and Sofie [34] and Driscoll et al. [35] used to “anchor” Ni-particles to the YSZ to prevent coarsening and migration of Ni particles. Infiltration with aluminum titanate increased Ni catalyst stability in Ni-YSZ electrode such that “the time required for degradation to 90% of initial current output was increased by a factor of 115” [35].

The ultimate way to circumvent the degradation problems related to Ni migration is to replace the Ni with another electron conducting catalytic material. Such materials have been researched for many years [36] This may be a single phase mixed ionic and electronic conductor (MIEC) oxide materials, which provide electrochemical reaction sites on the entire surface/gas two-phase boundary (2PB), and making them less susceptible to blockage by impurities than 3PB-based electrodes. Alternatively, it may be a mixture of MIEC and an electron conductor. CGO is a MIEC and very promising as an electrocatalytic component of a composite electrode where another material provides sufficient electronic conductivity [37,38].

4. Conclusion

It seems that we now qualitatively have a rough picture of processes that may contribute to Ni migration. Significant evidence for Ni migration via gasborne compounds is available, but it is not necessarily the only migration mechanism. Maybe migration of solid Ni on the YSZ surface is also taking place at the same time as the evaporation-condensation of Ni compounds. Furthermore, there are indications that trace impurities in reactant gases may be very important. The Ni migration away from the electrolyte in case of SOEC is not the cause, but rather the consequence of loss of contact between Ni and YSZ in the active layer of the fuel electrode.

In order to be able to describe quantitatively the Ni migration processes to an extent that will make us able to predict the associated degradation rate of the SOC fuel electrode under given operation conditions, we need a lot of quantitative knowledge about Ni migration on YSZ and in Ni-YSZ cermets. Also, the distributed local values and gradients of temperature and gas composition, $p_{\text{H}_2\text{O}}$, p_{H_2} , p_{CO_2} , p_{CO} and p_{O_2} at the 3PBs are needed. First of all, we need to be able to reproduce the degradation, i.e. show that we can control the parameters. Next, we have to measure vapour-pressures of gaseous Ni containing species over Ni nanoparticles as function of reactant gas composition, pressure, temperature and electrochemical polarisation. Also surface coverage of Ni-species on Ni and on SZ (stabilized zirconia in general) should be measured. Further, determination of free energy of Ni-SZ interfaces, of Ni and SZ surfaces and their dependence on the above mentioned basic

parameters must be carried out. It is in particular important to include tests with selected and known impurities, including trace impurities in all work mentioned above.

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