

# Durability of Solid Oxide Electrolysis Cells for Hydrogen Production

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## Abstract

In the perspective of the increasing interest in renewable energy and hydrogen economy, the reversible solid oxide cells (SOCs) is a promising technology as it has the potential of providing efficient and cost effective hydrogen production by high temperature electrolysis of steam (HTES). Furthermore development of such electrolysis cells can gain from the results obtained within the R&D of SOFCs. For solid oxide electrolysis cells (SOEC) to become interesting from a technological point of view, cells that are reproducible, high performing and long-term stable need to be developed. In this paper we address some of the perspectives of the SOEC technology i.e. issues such as a potential H<sub>2</sub> production price as low as 0.71 US\$/kg H<sub>2</sub> using SOECs for HTES; is there a possible market for the electrolyzers? and what R&D steps are needed for the realisation of the SOEC technology?

In the experimental part we present electrolysis tests results on SOCs that have been optimized for fuel cell operation but applied for HTES. The SOCs are produced on a pre-pilot scale at Risø National Laboratory. These cells have been shown to have excellent initial electrolysis performance, but the durability of such electrolysis cells are not optimal and examples of results from SOEC tests over several hundreds of hours are given here. The long-term tests have been run at current densities of  $-0.5 \text{ A/cm}^2$  and  $-1 \text{ A/cm}^2$ , temperatures of 850°C and 950°C and  $p(\text{H}_2\text{O})/p(\text{H}_2)$  of 0.5/0.5 and 0.9/0.1. Long-term degradation rates are shown to be up to 5 times higher for SOECs compared to similar SOFC testing. Furthermore, hydrogen and synthetic fuel production prices are calculated using the experimental results from long-term electrolysis test as input and a short outlook for the future work on SOECs will be given as well.

# 1 Perspectives

Whether or not there is a market for efficient electrolyzers for production of hydrogen and/or synthesis gas (a mixture of CO and H<sub>2</sub> which is a “precursor” for synthetic fuels such as methane and methanol) is closely related to the following two conditions: 1) restriction of fossil fuel consumption by political means and thereby an increase in energy supply from renewable energy sources and increased interest in hydrogen related energy technologies and 2) if the energy price for fossil fuels is significantly higher than the price for energy from alternative energy sources such as renewable energy from wind, solar and hydropower. Regarding condition 2), economic estimates of production prices for hydrogen and synthetic fuel by high temperature electrolysis is important in order to map the potential of SOECs for hydrogen and synthesis gas production. Furthermore, the economic estimates are closely related to 1) the electricity price and 2) the characteristics of the state-of-the-art (SoA) SOEC especially the following 3 parameters: A) cost of the cells/stacks, B) performance of the SOECs and C) durability of the SOECs.

First, we address the economic assessment of the SOEC technology. Given the assumptions in Table 1 and the initial cell performance shown in Figure 1, the H<sub>2</sub> production cost was found to be 71 US¢/kg H<sub>2</sub>, equivalent to 30 \$/barrel crude oil using the higher heating value (HHV) [1]. The CO production cost was found to be 5.6 US¢/kg equivalent to 34 \$/barrel crude oil using the HHV.

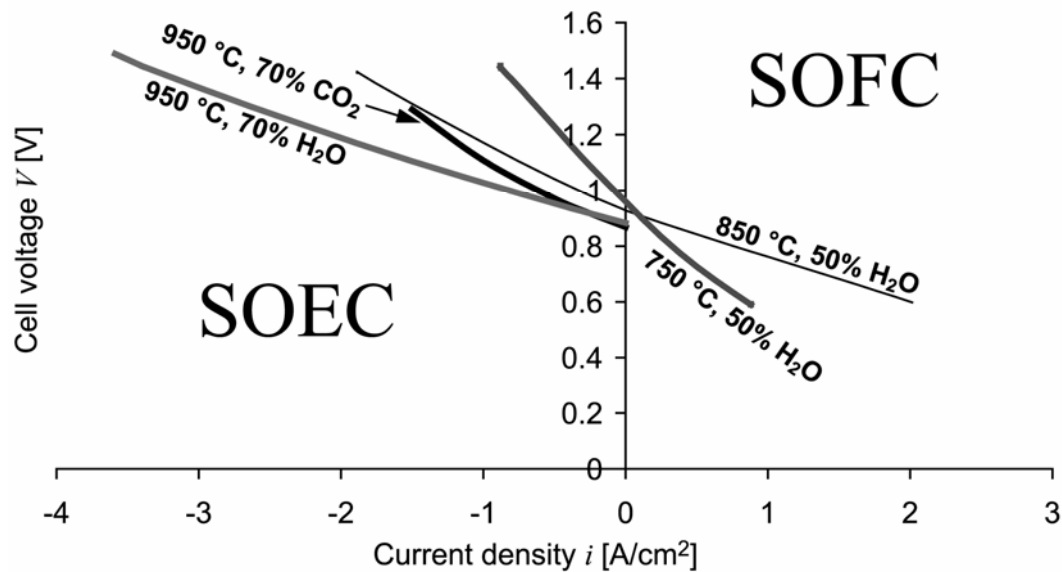


Figure 1: Kinetics of a Risoe SOC working as an electrolyser cell (negative current densities  $i$ ) and as a fuel cell (positive current densities  $i$ ) at different temperatures and steam or CO<sub>2</sub> partial pressures in the inlet gas to the cell [1].

Table 1: Cost estimation input parameters

Electricity price	1.3US¢/kWh (3.6US\$/GJ)
Heat price	0.3US¢/kWh
Investment cost	4000 US\$/m <sup>2</sup> cell area
Demineralised Water cost	2.3 US\$/m <sup>3</sup>
CO <sub>2</sub> cost	2.3 US\$/ton
Interest rate	5%
Life time	10 years.
Operating activity	50%
Cell temperature	850 °C
Heat reservoir temperature	110 °C
Pressure	0.1 MPa
Cell voltage (H <sub>2</sub> O electrolysis)	1.29 V
Cell voltage (CO <sub>2</sub> electrolysis)	1.47 V
Energy loss in heat exchanger	5%
H <sub>2</sub> O or CO <sub>2</sub> concentration in inlet gas	95%
H <sub>2</sub> O or CO <sub>2</sub> utilization	95%

If heat for steam generation can be provided from a waste heat source, the production price can be lowered even further. For synthetic fuel production, some reduction in the production price may be achieved by utilizing the heat from the catalysis reaction for steam generation. The main part of the production cost for both H<sub>2</sub> and CO is the electricity cost [1]. In Figure 2 is shown the estimated H<sub>2</sub> production cost vs. electricity price at various investment costs.

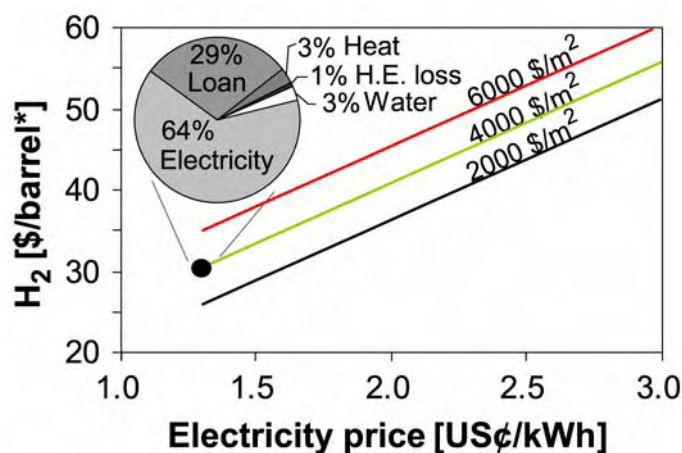


Figure 2: H<sub>2</sub> production cost vs. electricity price at various investment costs. Details on the assumptions for the calculation are specified in Table 1 and Figure 1. The pie diagram shows the parts of the production price. \*Production price in equivalent crude oil price per barrel, using the HHV.

In Figure 3 is shown the estimated CO production cost vs. electricity price at various investment costs.

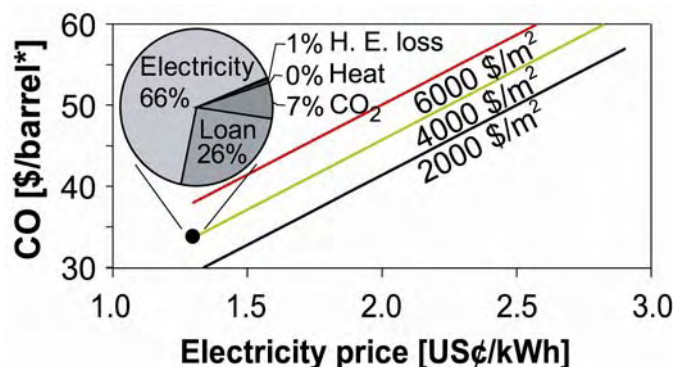


Figure 3: CO cost vs. electricity price at various investment costs. Details on the assumptions for the calculation are specified in Table 1 and Figure 1. The pie diagram show the parts of the production price. \*Production price in equivalent crude oil price per barrel, using the HHV.

The H<sub>2</sub>O and CO<sub>2</sub> electrolysis reactions becomes increasingly endothermic with temperature, and at 850 °C, 25% and 34% of the energy demand is heat for the H<sub>2</sub>O and CO<sub>2</sub> electrolysis reaction, respectively. For this reason, the efficiency from electricity to fuel is more than 90% in the presented calculation, since the Joule heat produced in the SOEC is utilized in the endothermic electrolysis reaction. The calculation does not include heat loss to the surroundings. However, the loss is expected to be limited if properly insulated by cheap materials, such as mineral wool, is used

Secondly, within recent years a huge rise in the number of abnormal weather events has occurred. Meteorologists agree that these exceptional conditions are signs of a Global Climate Change. Scientists agree that the most likely cause of the changes are man-made emissions of the so-called greenhouse gases that trap heat in the earth's atmosphere. Although there are six major groups of gases that contribute to the global climate change, the most common is carbon dioxide (CO<sub>2</sub>). For this reason there are much research in sequestration of CO<sub>2</sub> from power plants and other point sources for storage and removal of CO<sub>2</sub>. Using SOECs for recycling or reuse of CO<sub>2</sub> from energy systems (or CO<sub>2</sub> capture from air) would therefore be an attractive alternative to storage of CO<sub>2</sub> and would provide CO<sub>2</sub> neutral synthetic hydrocarbon fuels.

Capture of CO<sub>2</sub> for recycling can be achieved by absorption processes employing amines or carbonates as absorbents. The regeneration includes heating of the absorbent; therefore reduction of the energy requirement becomes a determining factor for realizing CO<sub>2</sub> recycling. From the viewpoint of energy saving in regeneration of the absorbent, carbonates are preferable to amine solutions, since the energy requirement for CO<sub>2</sub> removal in the carbonate process is about half of that of the amine process [2]. However the rate for CO<sub>2</sub> absorption and desorption with carbonates is slow, but for CO<sub>2</sub> capture/recycling from air, the absorption and desorption rate may not be a determining factor.

Mineral carbonation has been recognized as a potentially promising route for permanent and safe storage of carbon dioxide, and thereby also a promising route for recycling of CO<sub>2</sub>. Both the potentially large CO<sub>2</sub> sequestration capacity and the exothermic nature of the carbonation reactions

involved have contributed to an increasing amount of research on mineral carbonation in recent years [3; 4]. A number of different carbonation process has been reported, of which aqueous mineral carbonation route was selected as the most promising in a recent review [3]. Calcium carbonate is a well known CO<sub>2</sub> absorbent [3]. Also the less known magnesium carbonate or calcium magnesium carbonate can be employed. The required energy for regeneration is a determining factor. Thermodynamic equilibrium calculations (calculated using Factsage 5.5 Software [5] at 1 atm) show that CO<sub>2</sub> capture/recycling using magnesium carbonate can be operated at approximately 400 °C lower than the case for calcium carbonate. A carbon neutral energy cycle utilizing CO<sub>2</sub> capture from air with magnesium carbonate in combination with a fuel cell is sketched in Figure 4. Magnesium carbonate is abundant in nature as calcium magnesium carbonate. A carbonate cycle for CO<sub>2</sub> capture with calcium magnesium carbonate can be operated between 250 °C and 400 °C utilizing magnesium carbonate only. Using only magnesium carbonate from calcium magnesium carbonate, higher amount of minerals would have to be mined and transported.

A carbonate cycle for CO<sub>2</sub> capture/recycling is definitively technically feasible, but the practical and economic aspects regarding calcium carbonate, magnesium carbonate or calcium magnesium carbonate have to be assessed to determine the most suitable absorbent for CO<sub>2</sub> capture/recycling.

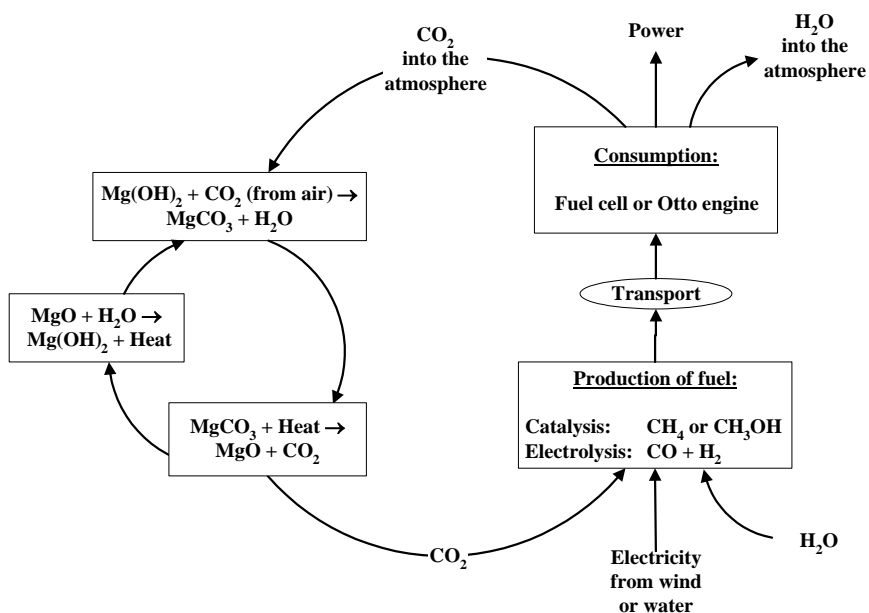


Figure 4: Carbon dioxide neutral energy cycle utilizing CO<sub>2</sub> capture from air with calcium carbonate in combination with a Solid Oxide Electrolysis Cells (SOEC).

Thirdly, the realization of the SOEC technology will depend greatly on the price and performance of SOECs as this in turn influences the hydrogen/synthetic fuel production price. The SOCs produced on a pre-pilot scale at Risoe National Laboratory can be used both as fuel cells and electrolysis cells and can be considered inexpensive due to 1) inexpensive production methods such as tape casting and robot spraying are use [6] and 2) no expensive materials such as noble metals are used for the

cells. Furthermore, the Risoe SOECs have been shown to have an excellent initial performance [1; 7] i.e. a low internal resistance in the cells which in turn contribute to minimize hydrogen/synthetic fuel production prices. The high initial performance of the SOECs clearly shows the potential of the cells. Nevertheless, high initial performance of the cells is a necessary but not sufficient characteristic of the SOECs. The cells need to be long-term stable i.e. keep the high performance (low cell resistance) over thousands of hours of testing. At the time being the durability issue is the critical point for the Risoe SOECs and the topic for further R&D. In the “experimental/result/discussion” sections of this paper results from long-term testing of Risoe SOECs will be given.

## 2 Theoretical background

The basic operational principle for a SOFC is shown in part A and for a SOEC in part B of Figure 5. In SOFC mode the cell is used to produce electricity by converting the chemical energy in the fuel ( $H_2$ ) directly to electrical energy. In SOEC mode the input to the cell is steam and electrical energy from an external power supply in order to split water into hydrogen and oxygen. The SOEC also has the capability to electrolyse a mixture of steam and carbon dioxide i.e. it can split  $CO_2$  into CO and  $O_2$ .

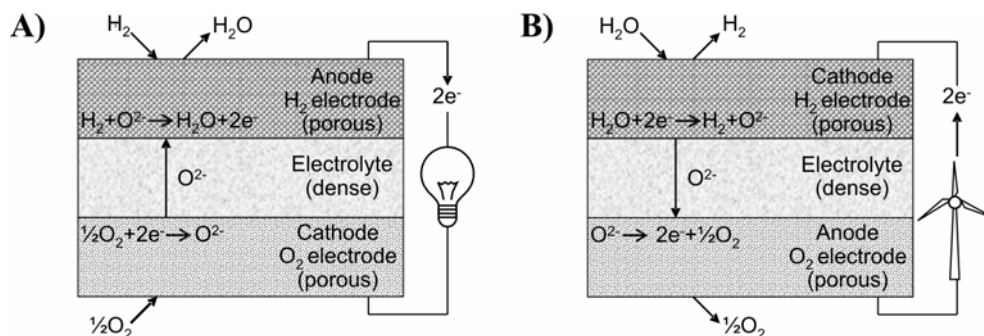


Figure 5: Sketch of the basic operational principle for the SOFC (part A) and the SOEC (part B).

For endothermic reactions such as  $H_2O$  and/or  $CO_2$  electrolysis it is, from a thermodynamic point of view advantageous to operate at high temperature as a part of the energy required for water splitting is obtained in the form of high temperature heat e.g. heat from solar concentrators or waste heat from nuclear power plants [8-10]. The high temperature electrolysis using SOEC can therefore be performed with a lower electricity consumption compared to low temperature electrolysis cells [11; 12]. Furthermore, the reaction kinetics is speeded up at high temperature and this in turn lead to a decreased internal resistance of the cell and thereby increased efficiency i.e. lowering the necessary electrical energy consumption to produce a certain quantity of hydrogen or synthetic fuel. Even though it is, from a thermodynamic and electrode kinetic point of view, advantageous to operate the SOECs at high temperature, material durability issues makes an upper limit for the operation temperature. SOEC tests are typically performed in the temperature range from  $750^\circ C$  to  $950^\circ C$ .

### 3 Experimental

Ni/YSZ supported DK-SOFC cells were used for the electrolysis tests. The cells are full cells produced at Risoe National Laboratory [6; 13]. The SOCs are planar  $5 \times 5 \text{ cm}^2$  cells with an active electrode area of  $16 \text{ cm}^2$ . Detailed description of the setup and the start-up procedure i.e. heating up and reduction of NiO is given elsewhere [7; 14; 15]. The steam is produced by burning of  $\text{O}_2$  and  $\text{H}_2$  in the inlet tubing to the cell. The results presented here originate from two electrolysis durability tests that were run galvanostatic. The test conditions are given in Table 2. DC characterization was performed by recording polarization curves (iV-curves) for each of the cells before and after the long-term electrolysis tests. AC characterization was performed by recording electrochemical impedance spectra (EIS) using a Solartron 1260 frequency analyzer [16].

Table 2: Test conditions for long-term galvanostatic electrolysis tests using SOECs.

Test no	Test time (h)	Temp. ( $^{\circ}\text{C}$ )	$p(\text{H}_2\text{O})/p(\text{H}_2)$	$i$ ( $\text{A}/\text{cm}^2$ )	Steam utiliza.
A	620	950	0.9/0.1	-1.0	53%
B*	1510	850	0.5/0.5	-0.5	27%

\*) The glass sealing was pre-treated by leaving test B for 12 days at 90% steam at  $950^{\circ}\text{C}$  prior to reduction of the cell and electrolysis testing.

### 4 Results

It has been shown previously that the hydrogen electrode supported SOC produced at Risoe National Laboratory have excellent and reproducible initial performance [1; 7]. Two examples of cell voltage curves recorded during electrolysis durability testing of such Risoe cells are shown in Figure 6. The both test an increase in the cell voltage during testing was observed. For SOECs an increase in the cell voltage correspond to an increase in the internal resistance of the cell i.e. a decrease in performance of the SOEC.

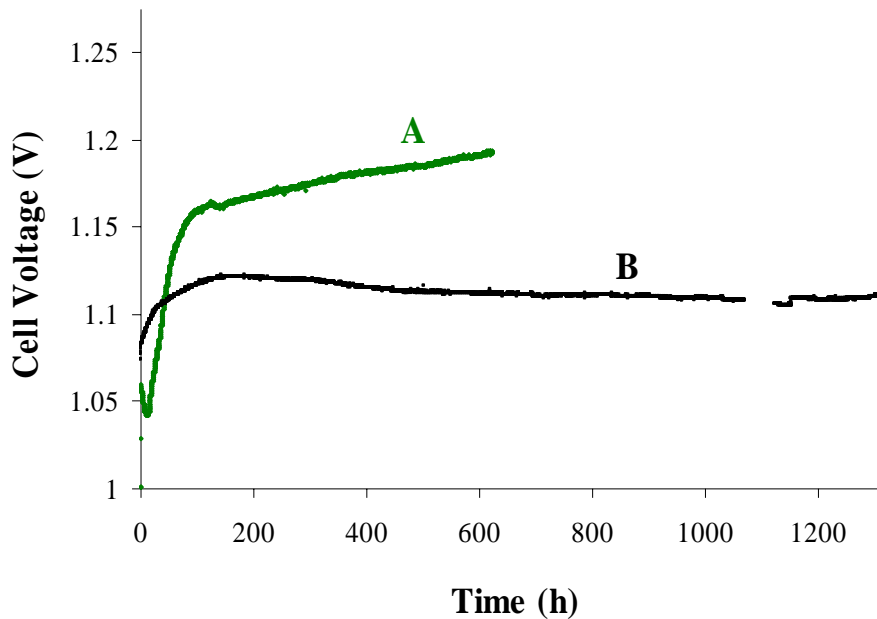


Figure 6: Development of cell voltage during two long-term galvanostatic electrolysis tests. Operation conditions are given in Table 2 and development of the internal cell resistances during electrolysis testing are in Table 3.

For test A the cell voltage increased 134 mV over 620 h of testing and for test B an increase of 35 mV over 1510 h of testing was observed. For test A the internal resistance of the cell increased from  $0.22 \Omega\text{cm}^2$  to  $0.45 \Omega\text{cm}^2$  but only from  $0.26 \Omega\text{cm}^2$  to  $0.36 \Omega\text{cm}^2$  for test B (Table 3). The passivation history for test A consists of three parts: 1) a relatively fast initial passivation of more than 100 mV within approx. 100 h, 2) a few mV of activation of the cell i.e. a decrease in cell voltage and 3) a long-term degradation of 65 mV/1000 h. The glass sealing used for test B was pre-treated with 90%  $\text{H}_2\text{O}$  at  $950^\circ\text{C}$  for 12 days prior to electrolysis testing and this significantly changed the cell voltage curve of test B compared to test A. An electrolysis test applying the same test conditions as for test B but without pre-treating the glass sealing showed the same general trends as for the cell voltage curve of test A, that is: 1) an initial passivation of cell within the first few hundred hours, 2) a reactivation of the cell and 3) subsequently a long-term degradation (20 mV/1000 h), see [12] for details. For the last 300 hours of test B a degradation of  $\sim 30$  mV/1000 h was observed.



Table 3: Development of cell resistances during long-term galvanostatic electrolysis testing. Resistances were obtained from EIS during electrolysis operation of the cells.

Test no	Time (h)	$i$ (A/cm <sup>2</sup> )	Resistance ( $\Omega\text{cm}^2$ )*
A	1	-1.0	0.22
A	610	-1.0	0.45
B	1	-0.5	0.26
B	1510	-0.5	0.36

\* The resistances are the sum of the ohmic and polarization resistance obtained by impedance spectroscopy during electrolysis operation of the cells. For both tests only the polarization resistance increased during electrolysis testing.

Examples of polarization curves (iV-curves) before and after the 1510 hours of electrolysis testing for test B is shown in Figure 7. This illustrates the development of the area specific resistance (ASR) of the cell that has occurred as an effect of the 1510 hours of electrolysis testing. Fuel utilization corrected ASR [17] at 0.7 V was 0.17  $\Omega\text{cm}^2$  before testing and 0.22  $\Omega\text{cm}^2$  after testing for the fuel cell iV-curve recorded with 5% H<sub>2</sub>O and 95% H<sub>2</sub> to the hydrogen electrode.

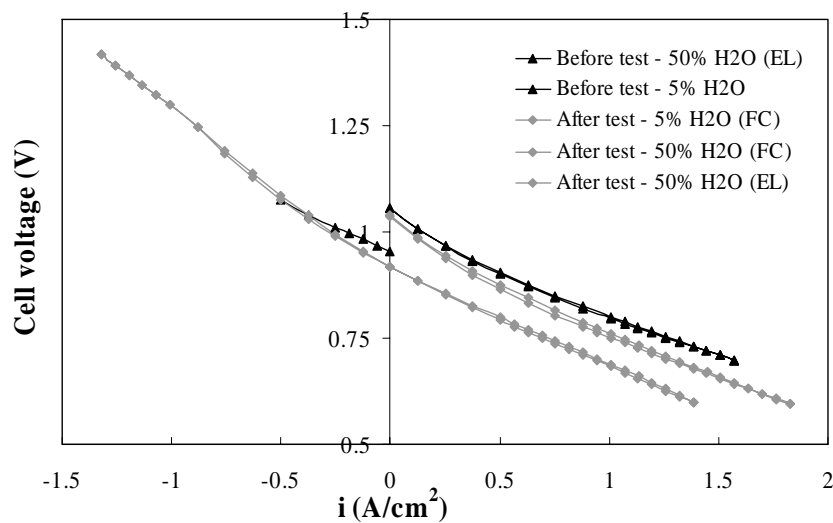


Figure 7: Polarization curves (iV curves) before and after test B at different  $p(\text{H}_2\text{O})/p(\text{H}_2)$  ratios. Negative current densities correspond to electrolysis operation of the cell and positive current densities to fuel cell operation of the cell.

The cell performance results obtained during the long-term test B are relevant input data for calculation of the H<sub>2</sub> production price using state-of-the-art SOEC at conditions at which the SOEC has been operated for more than 1500 h. Using Table 1, but with a cell voltage of 1.15 V and 50% H<sub>2</sub>O inlet concentration (corresponding to 0.5 A/cm<sup>2</sup> in the calculation) the H<sub>2</sub> production cost is estimated to be 108 US¢/kg equivalent to 46 \$/barrel crude oil using the HHV.

## 5 Discussion

In general increasing the  $p(\text{H}_2\text{O})$  will speed up the time constant for the “initial” passivation process for the electrolysis tests and increasing the current density – i.e. a higher overpotential - seems to increase the degree of the long-term degradation. The effect of increasing the  $p(\text{H}_2\text{O})$  has also been reported in [18] upon changing from 30% to 70% steam as inlet gas and is confirmed by comparison with test A. Seen from a technological point of view it is relevant to notice that tests applying up to 98% of steam has been performed successfully for these SOECs [8; 19]. It has been argued that the significant passivation within the first ~100-200 h of electrolysis testing could be related to silica impurities in the hydrogen electrode and that these impurities can originate from the glass sealing used in the cell test set-up [18]. This was the reason for the pre-treatment of the glass sealing for test B. There is no doubt that the changes for the glass sealing significantly changed the passivation course of the SOEC though a full understanding of the effect of the glass sealing on the passivation history is still a topic for ongoing investigations. Even if we consider the initial “fast” passivation of the SOEC as a sealing related problem and view this as a practicable problem, the long-term degradation of 65 mV/1000 h (~5%/1000 h) and 30 mV/1000 h (~3%/1000 h) for test A and B respectively, obtained from the cell voltage curves for test A and B in Figure 6, are still significantly higher than the degradation obtained for similar cells used for SOFC testing. In SOFC mode the cell degradation at 850°C was below 1%/1000 h measured over 1500 h of test at current densities up to 1 A/cm<sup>2</sup> [13]. The overall decrease in the cell performance of the SOEC after test is also seen from the ~30% increase in the ASR calculated from the iV-curves recorded before and after test. The long-term degradation mechanism (Figure 6) is not yet understood and a substantial R&D effort is needed for the investigation of this phenomenon to obtain the goal: SOECs that are not only initially high performing – we already have such SOECs – but also high performing in the long-term i.e. for thousands of hours of electrolysis operation!

The durability issue for the SOECs is closely related to the production prices for H<sub>2</sub> and synthetic fuel. For lifetimes above 3-4 years the H<sub>2</sub> production price starts to become relatively independent of the life time [12]. For the CO production price this is about 6 years. The Risoe cells do not yet meet these lifetimes, but reasonable stability for more than 1000 h has been achieved (see Figure 6) and our ongoing research is in this field. Generally, durability seems to be decreasing with increasing current densities, but a quantitative measure is still to come.

## 6 Conclusion

Economic estimates of production prices for hydrogen and synthetic fuel by high temperature electrolysis of steam and/or carbon dioxide using SOECs give very promising results. Prices down to 4.8 US\$/GJ or 71 UScent/kg H<sub>2</sub> using the HHV of H<sub>2</sub> and the excellent initial electrolysis performance at 950°C for Risoe SOECs as experimental input for the calculations. The long-term

testing of SOECs presented here show a degradation for the SOECs that is approximately 5 times larger than the one for similar cells tested as SOFC at the same temperature and current density. The long-term degradation of the SOECs is ~2-3 times larger for test A (950°C, 90% H<sub>2</sub>O, -1 A/cm<sup>2</sup>) compared to test B (850°C, 50% H<sub>2</sub>O, -1/2 A/cm<sup>2</sup> with and without pre-treatment of the glass sealing prior to test). Using the long-term test results, the H<sub>2</sub> production cost is estimated to be 108 US¢/kg equivalent to 46 \$/barrel crude oil using the HHV.

## 7 Outlook

Comparing the present world market oil prices with the estimated hydrogen production prices based on our experimental SOEC results, it is clear that the SOEC technology has the potential for price competitive hydrogen and synthetic fuel production; especially where inexpensive electricity from for instance renewable energy sources such as wind energy (e.g. Denmark) or hydro power systems (e.g. Iceland and Norway) is available or in combination with nuclear power plant where excess high temperature heat is available (e.g. France, Belgium, USA).

To realize the obvious potential of the SOEC technology a substantial R&D effort is necessary. The SoA Risoe SOECs are initially high performing and inexpensive production methods are used; however the durability of the cells needs to be increased significantly and this is the focus in the present SOEC work. Furthermore, work on SOEC stacks has been initiated and in a longer perspective, if the interest of today in hydrogen and renewable energy technologies is maintained for the next ~10 years, R&D work will be needed to integrate the SOEC stacks into the existing energy grid for instance in order to optimize the efficiency of and energy supply from wind energy.

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