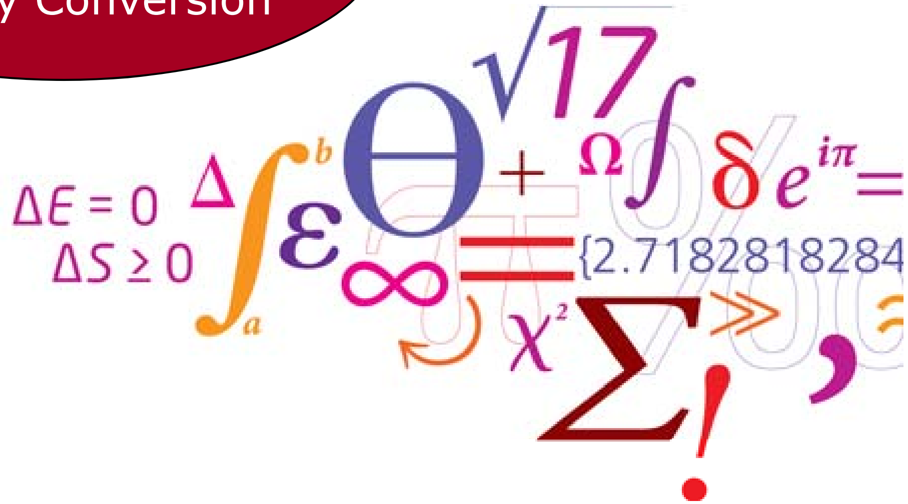
SOEC: Materials, properties & challenges

Acknowledgements to colleagues at DTU Energy Conversion

Anne Hauch
DTU Energy Conversion

hauc@dtu.dk



Outline

1. Intro – who am I and who are you? + learning objectives
2. Repetition of basic working principle – *small exercise*
3. SOE from a CO_2 cycle point of view
4. SOE from a economy / fuel production prices point of view

5. SOE cells – Materials & initial performance (H_2O / CO_2 / Co-electrolysis)
6. SOE cells – Materials & durability
7. *Assignment* – Examples of impurity related degradation of SOE cells

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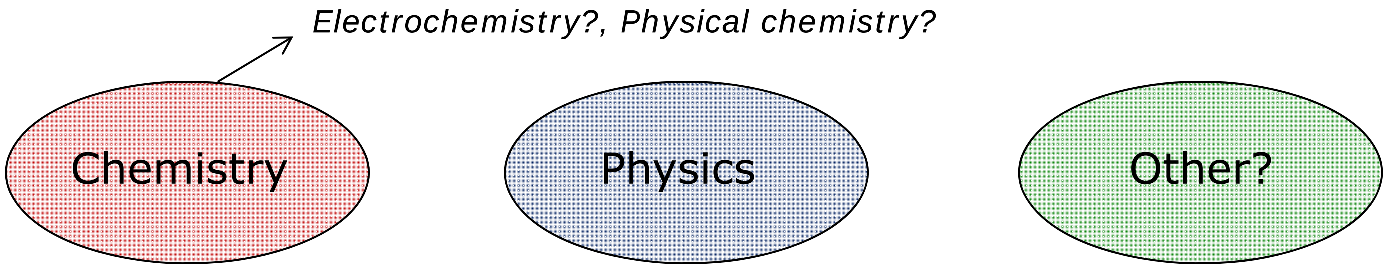
10. SOE cells – where are we heading?

11. Summing up / leaning objectives

1. Intro – who are you? + learning objectives

Intro – who am I and who are you/your background?



Learning objectives (2 x SOE lectures):

1. Explain/sketch qualitatively how electrolysis (potentially) can become an important player in a future energy grid?
2. Explain the basic operation principle for steam electrolysis, CO_2 electrolysis, co-electrolysis
3. List typical electrode materials incl. requirements and operation conditions
4. Give examples of SOEC degradation issues and explain why some of these can be somewhat different from what can be observed for similar SOFCs (based on material properties, test conditions etc.)

2. Basic working principle – small exercise

Only pen & paper “allowed” – no Google or Wikepedia help ☺

1. Make a sketch of an SOEC including names for typical electrode and electrolyte materials

2. Note on your sketch which electrode is cathode, anode, steam/hydrogen electrode and oxygen electrode

3. Write the electrode reactions and the total reaction for:

   a) Electrolysis of steam
   b) Electrolysis of carbon dioxide

4. *Electrolysis of steam and carbon dioxide – why is this more complex than just adding reaction a) and b) ?*

2. Basic working principle - answers

1. Make a sketch of an SOEC including names for typical electrode and electrolyte materials
2. Note on your sketch which electrode is cathode, anode, fuel electrode and oxygen electrode

...or with barrier layer and a MIEC electrode

2. Basic working principle - answers

Write the electrode reactions and the total reaction for:

a) Electrolysis of steam

b) Electrolysis of carbon dioxide

Ref. Ebbesen, Jensen, Hauch & Mogensen, "High Temperature Electrolysis", submitted, InTech, 2012
Ref. Haber, Zeitchr. Physik. Chem., 68, 731 (1909)

2. Basic working principle – Operating conditions

<u>Typical temp.:</u> ~ 800-900 °C

<u>Gas compositions:</u>

H_2O electrolysis – avoid Ni oxidation (inlet of $p(H_2O)$ = 0.99 atm possible)

CO_2 electrolysis – avoid catalytic coke formation, T>800 °C and p(CO)<0.9 ⇒ Boudouard reaction shifted towards CO

<u>Current density / cell voltage:</u>

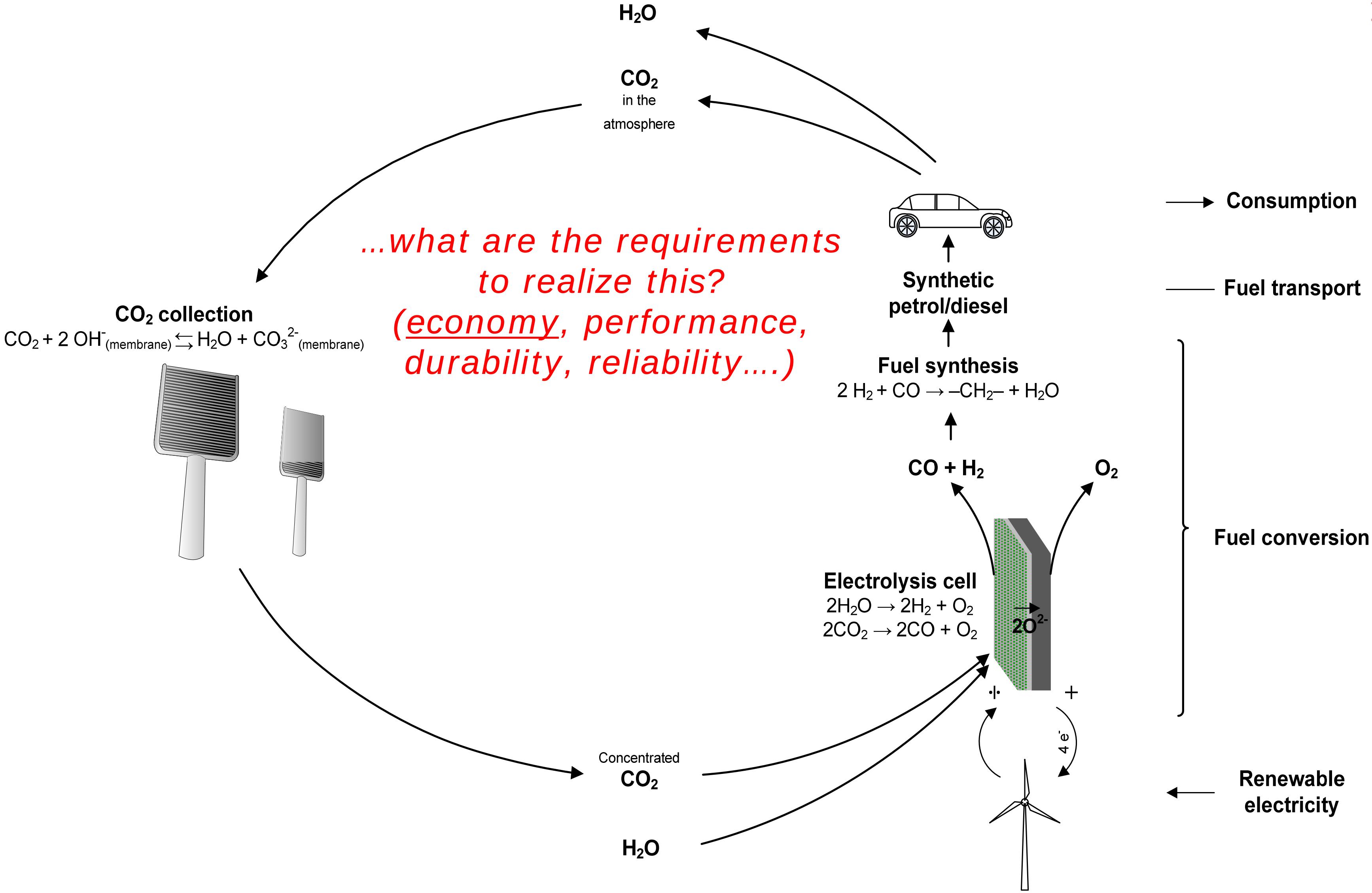
Lab scale — Up to 2 A/cm^2 durability testing

Techn. relevant — Potentiostatic / thermo-neutral potential
The possibility for load cycling

Ref. Hauch, "Solid Oxide Electrolysis Cells – Performance and Durability", PhD Thesis, DTU (2010)
Ref. Knibbe, Traulsen, Hauch, Ebbesen and Mogensen, J. Electrochem. Soc., 157, B1209 (2010)

3. SOE from a CO_2 cycle point of view

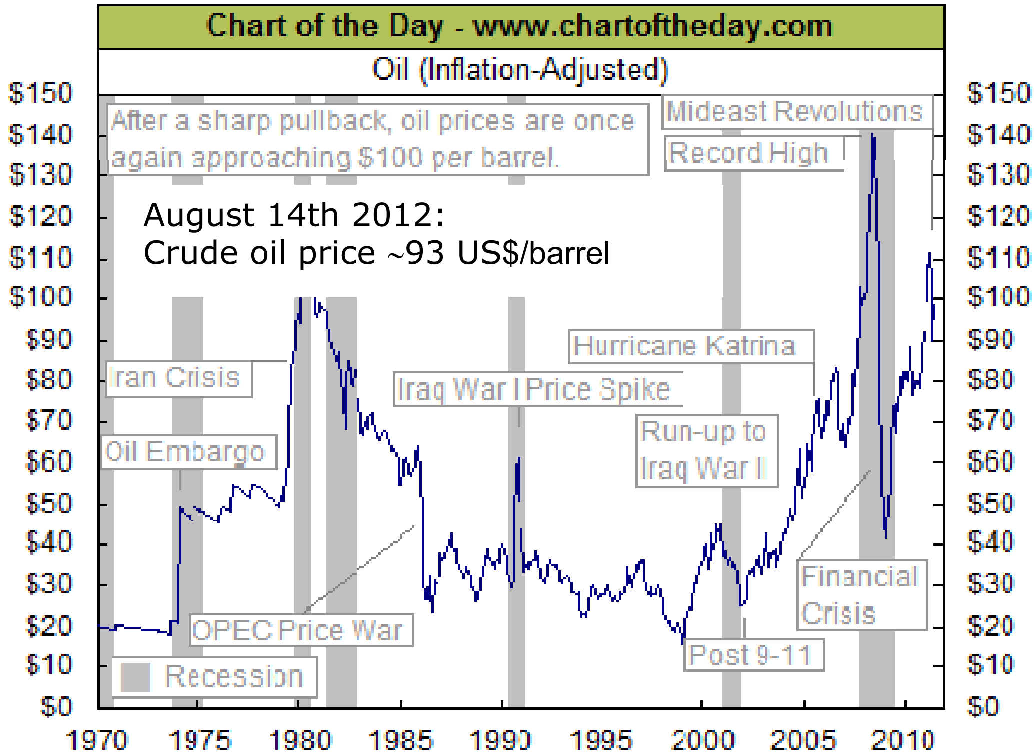
…what are the requirements to realize this? (economy, performance, durability, reliability….)



Ref. Ebbesen, Jensen, Hauch & Mogensen, "High Temperature Electrolysis", submitted, InTech, 2012

4. SOE from a economy / fuel prod. prices point of view

Our ”competitor” crude oil



4. SOE from a economy / fuel prod. prices point of view

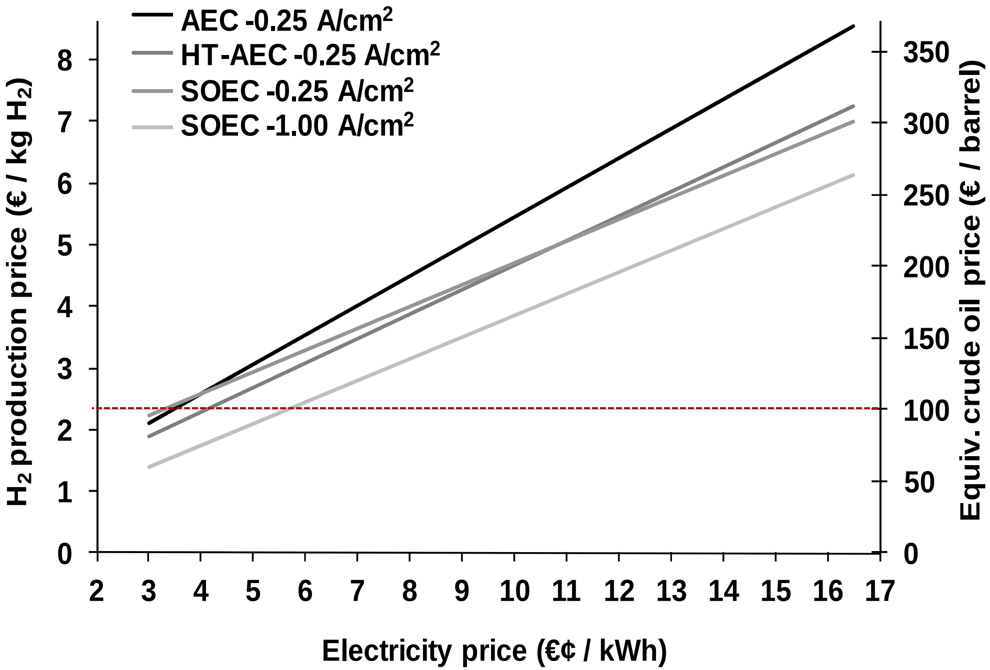
	Commercial low temperature alkaline electrolyser	High temperature alkaline electrolyser	High temperature Solid oxide electrolyser
Operation temperature	80 ºC	264 ºC	850 ºC
Investment cost	4420 €/m^2 cell area	4420 €/m^2 cell area	3620 €/m^2 cell area
Fuel composition	—	—	$H_2O/H_2 = 90/10$
Cell voltage at -0.25 A/cm^2	1.77 volts	1.46 volts	0.92 volts
Cell voltage at -0.50 A/cm^2	—	1.55 volts	0.95 volts
Cell voltage at -1.00 A/cm^2	—	—	1.01 volts
Life time	35 years	35 years	10 years
Operation time	50 %	50 %	50 %
Purified Water cost	1.6 €/m^3	1.6 €/m^3	1.6 €/m^3
Electricity price	7.65 €¢/kWh	7.65 €¢/kWh	7.65 €¢/kWh
Interest rate	8 %	8 %	8 %
Energy loss in heat exchanger	5 %	5 %	10 %

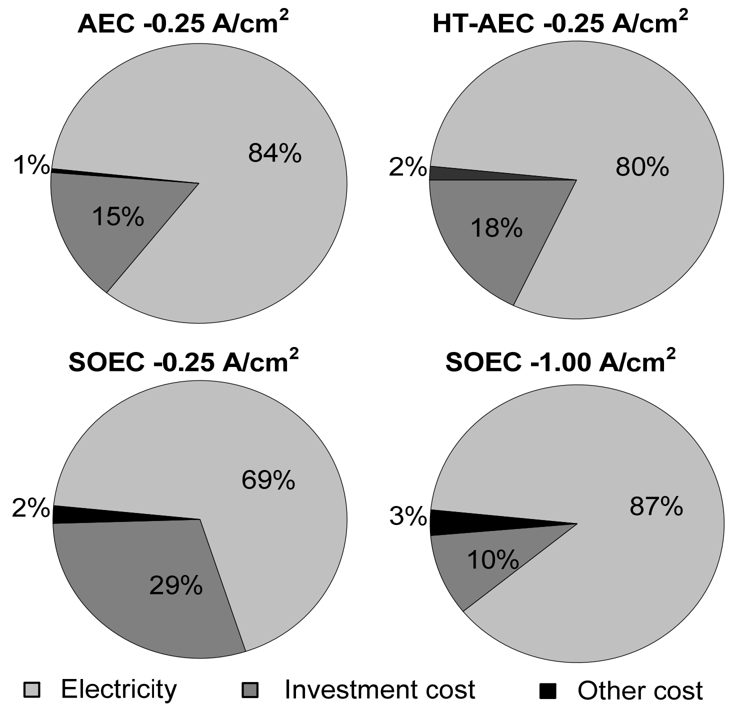
Ave. EU price
for retail/end-users
in 2007

Electricity prices 2012:
http://www.energy.eu/

Ref. Atmospheric electrolysers (Norsk Hydro), http://www.hydro.com/electrolysers/ (2008)
Ref. M. H. Miles, G. Kissel, P. W. T. Lu, S. Srinivasan, *J. Electrochem. Soc.*, **123**, 332 (1976).
Ref. A. Hauch, S. H. Jensen, S. Ramousse, M. Mogensen, *J. Electrochem. Soc.*, **153**, A1741 (2006)
Ref. S. H. Jensen, P. H. Larsen, M. Mogensen, *Int. J. Hydrogen Energy*, **32**, 3253 (2007).

4. SOE from a economy / fuel prod. prices point of view



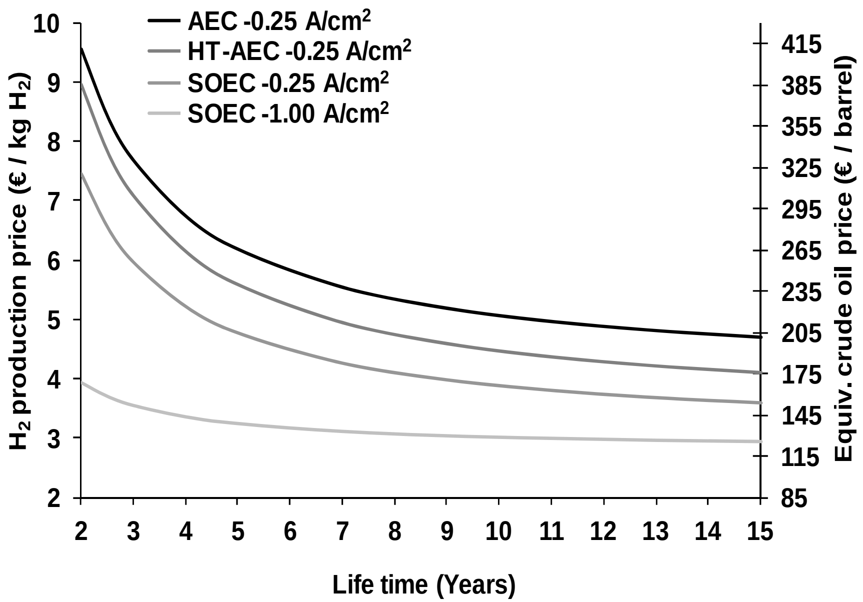


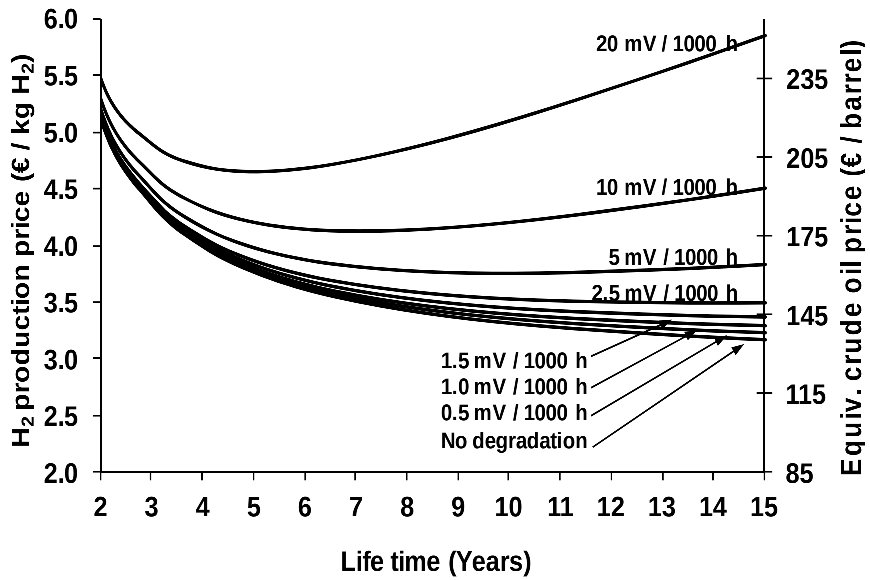
The pie chart shows the production price parts given the assumptions on previous slide.

Ref. Ebbesen, Jensen, Hauch & Mogensen, "High Temperature Electrolysis", submitted, InTech, 2012

4. SOE from a economy / fuel prod. prices point of view

Based on SOEC at 850 °C, -0.5 A/cm²





H_2 production price vs. lifetime

(Assuming an electricity price of 7.65 €¢/kWh - 2007 electricity price).

H_2 production price for SOECs depending on lifetime and cell degradation

(Assuming an electricity price of 7.65 €¢/kWh - 2007 electricity price).

For an estimate of syn-gas prodruction prices / SOE economic estimates:
Syngas production via high-temperature steam/CO2 co-electrolysis,
Fu, Mabilat, Zahid, Brisse & Gautier, Energy & Enviro. Sci.,3, 1382 (2010)

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Many different reports in lit. I just show a few selected ones!

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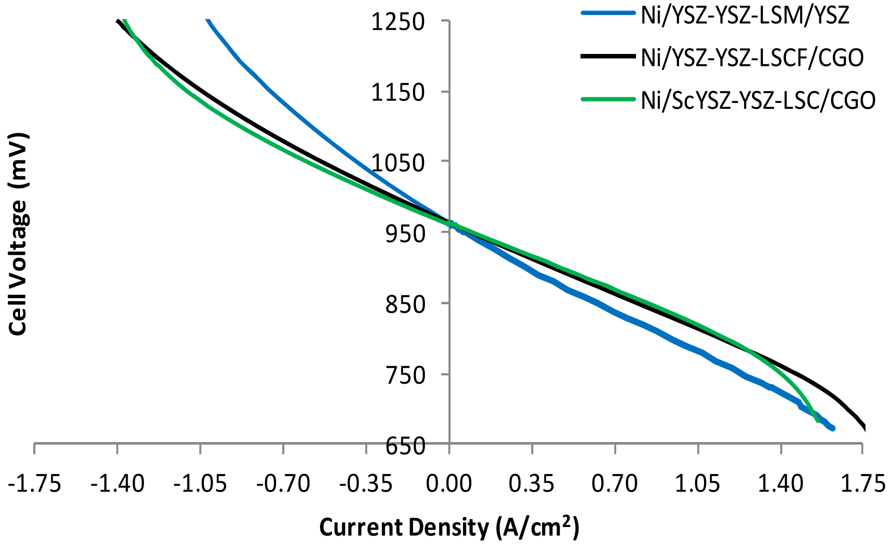
5. Materials & initial performance - H_2O electrolysis

Different oxygen electrode materials

Some of the highest electrolysis performance to date
(850 ºC, H_2O/H_2:50/50; O_2 to the oxygen electrode).
Ref. S. Ebbesen, Dept. of Energy Conv. and Storage, DTU (former Risø DTU)



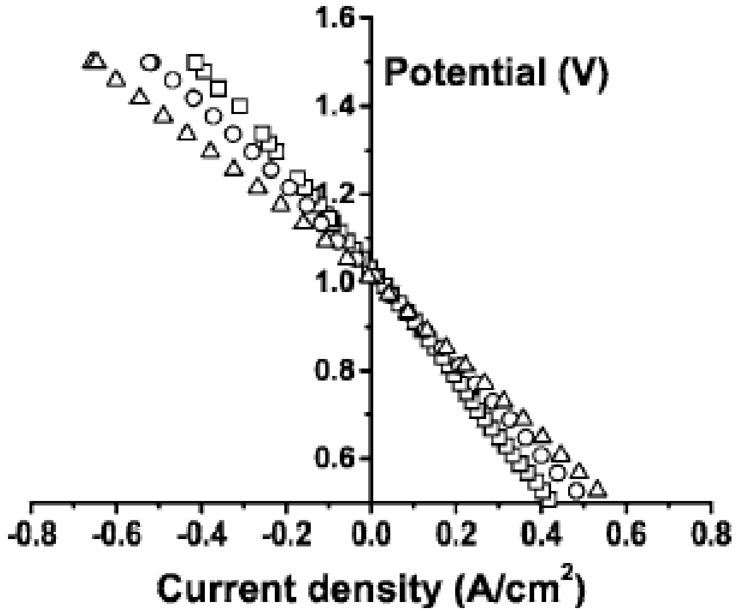


Figure 1. (a) V-i curves measured at 973 K, with 15% H_2O in H_2 at the Co-ceria-YSZ counter electrode and a P_{O_2} = 1 atm at the perovskite-based electrode. Data are for YSZ composites with LSM (□), LSF (○), and LSCo

Ref. Wang et al., *JECS*, **153**, p.A2066 (2006)

5. Materials & initial performance - H_2O electrolysis

Different fuel electrode materials

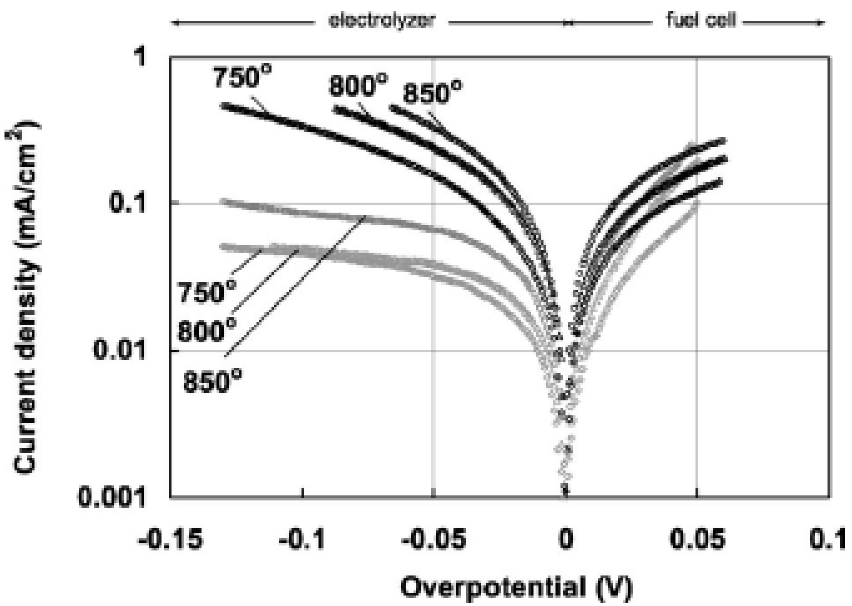
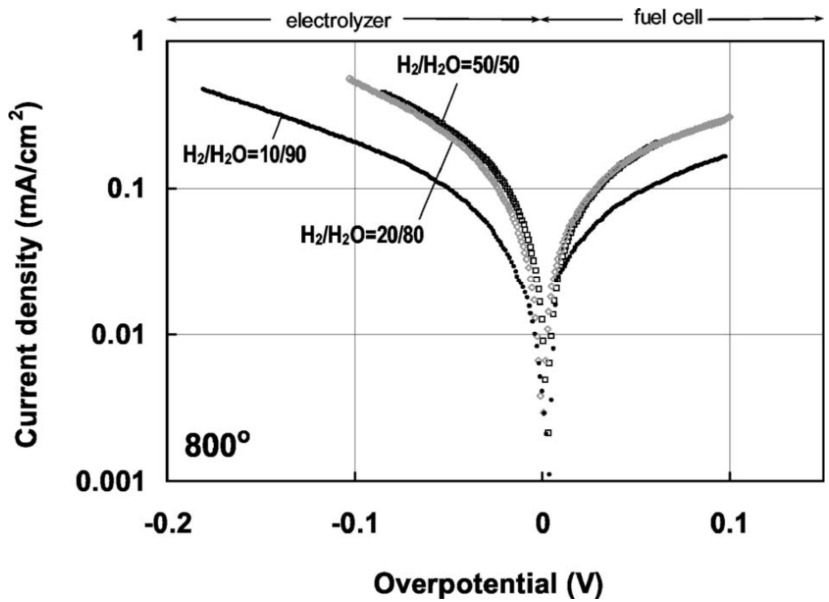


Figure 3. Current-overpotential dependencies obtained on Ni-YSZ (gray symbols) and ceramic composite $La_{0.35}Sr_{0.65}TiO_{3-\delta}$-$Ce_{0.5}La_{0.5}O_{1.75-\delta}$ (black symbols) electrodes at 750–850°C and H_2/H_2O = 50/50.

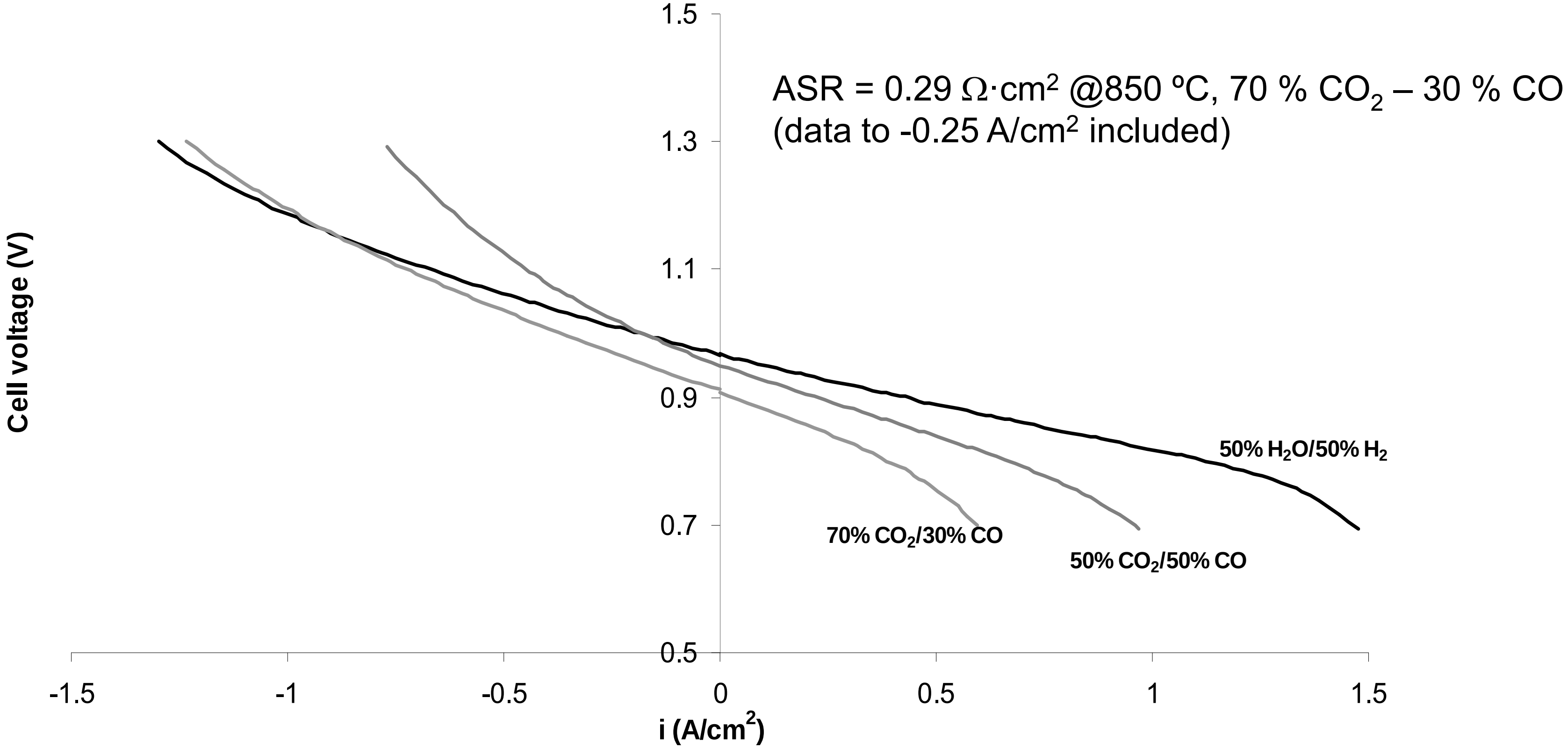


$La_{0.35}Sr_{0.65}TiO_3-Ce_{0.5}La_{0.5}O_{1.75}$ electrode at 800C at H_2/H_2O = 50/50, 20/80, and 10/90.

Ni/YSZ performance results SOFC><SOEC not necessarily a "universal" performance result! (can be structure dependent)

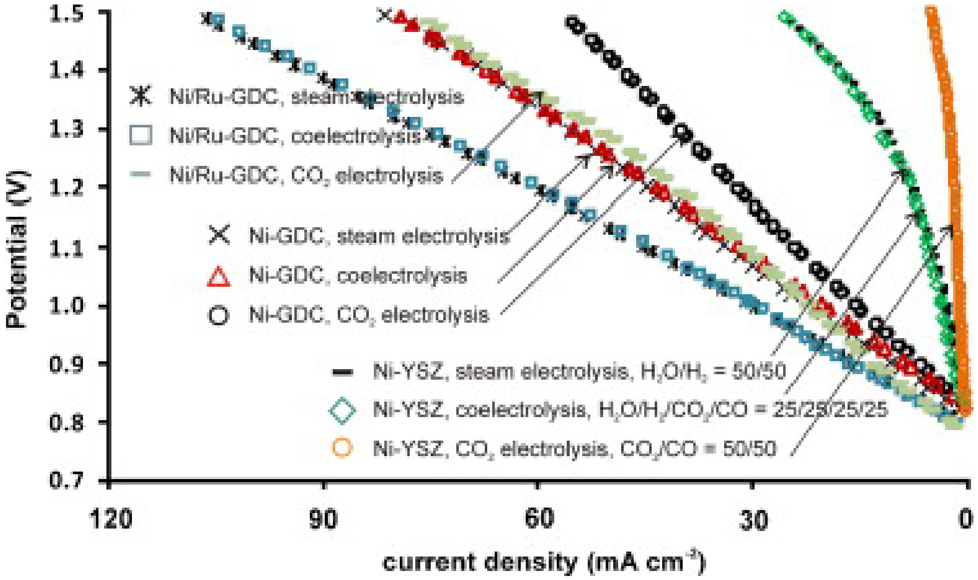
(Ref. Marina et al., *JECS*, **153**, p.A2066, 2006 - Results obtained on half cells model set-up)

5. Materials & initial performance – CO_2 electrolysis



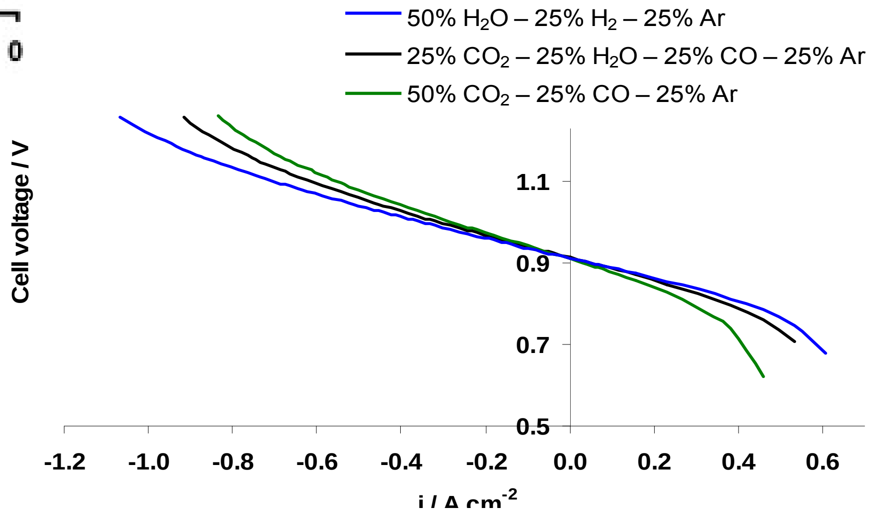
(Ref. S. D. Ebbesen and M. Mogensen, in *Proceedings of the 32nd International Conference & Exposition on Advanced Ceramics and Composites (ICACC)*, p. 271, The American Ceramic Society (2008).

5. Materials & initial performance – CO_2 / H_2O electrolysis



Electrolyte supported cells (H_2 electrode/YSZ electrolyte/Pt reference electrode, 800 ° C)

Planar Ni-YSZ|YSZ|LSM-YSZ at 850 ° C



H_2O/H_2: $ASR_{EL} = 0.24\ \Omega cm^2$	$ASR_{FC} = 0.25\ \Omega cm^2$
CO_2/CO: $ASR_{EL} = 0.34\ \Omega cm^2$	$ASR_{FC} = 0.32\ \Omega cm^2$
$H_2O/CO_2/CO$: $ASR_{EL} = 0.28\ \Omega\ cm^2$	$ASR_{FC} = 0.27\ \Omega cm^2$

Ref. P.Kim-Lohsoontorn, J. Bae, J. Power Sources, 196(1), 7161 (2011)
Ref. S. D. Ebbesen, R. Knibbe, M. Mogensen, *J. Electrochem. Soc.*, **159**, F482 (2012)

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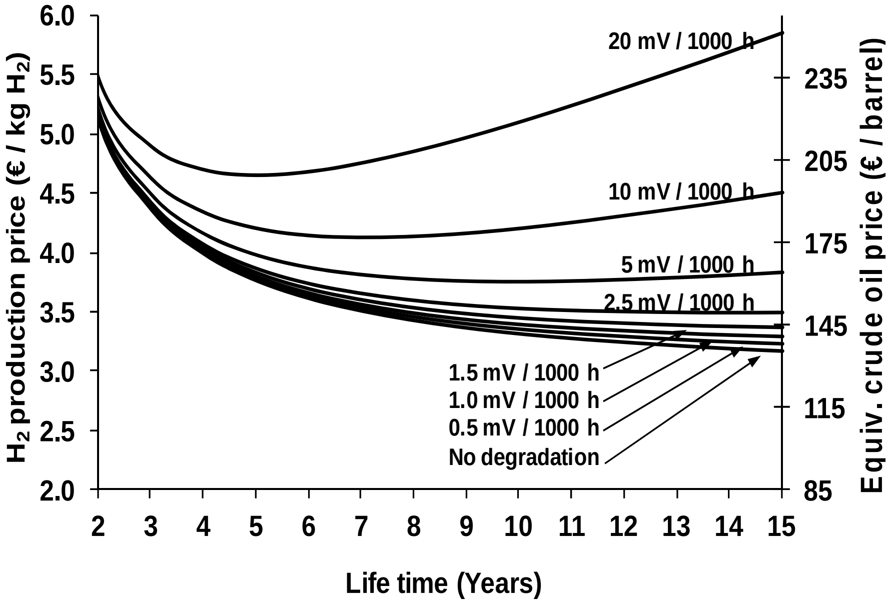
- Let us review the “demand” on life-time based on economic estimates on fuel production prices…



- We’ll have a look at two SOEC degradation case stories today
  + incl. a small assignment for you.

6. Materials & durability – gas purity/impurity

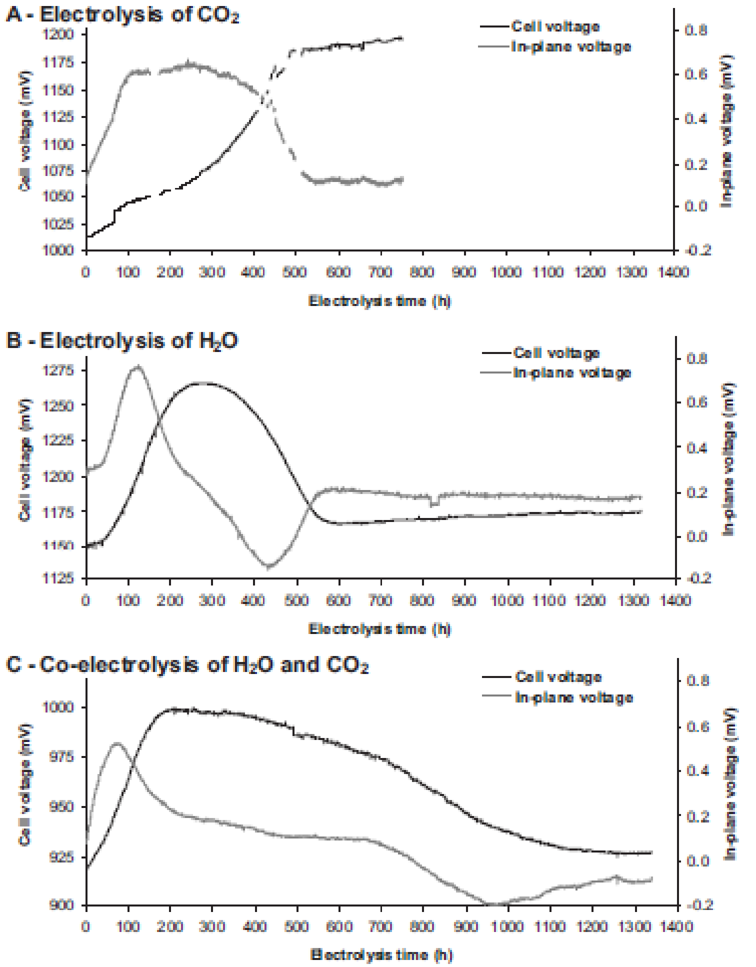
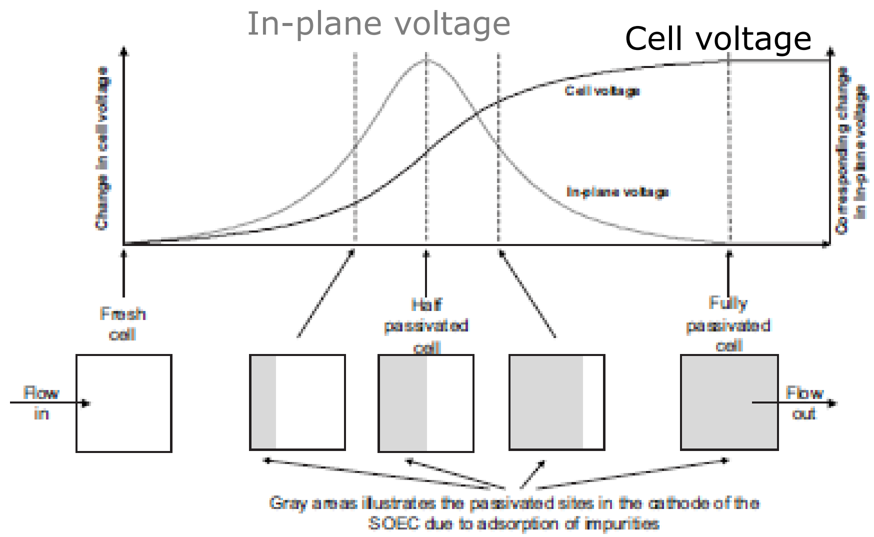


Figure 2. Cell voltage and corresponding in-plane voltage at the Ni/YSZ electrode measured during (A) CO_2 (850°C, −0.25 A/cm^2, 70% CO_2–30% CO), (B) H_2O (850°C, −0.50 A/cm^2, 50% H_2O–50% H_2), and (C) co-electrolysis (850°C, −0.25 A/cm^2, 45% H_2O–45% CO_2–10% H_2).



Observed change in in-plane voltage measurements made us believe that some sort of impurity contamination took place…

Ref. Ebbesen, Graves, Hauch, Jensen & Mogensen, J. Electrochem. Soc., 157(10), B1419 (2010)

6. Materials & durability – gas purity/impurity

Assignments – impurities from different "sources" - estimates only (1000 h test)

A) Sulphur impurities in the gas stream:

In the experiments (see graphs) a total flow of 25 h/l was used for each electrode. How much sulphur did we lead to the tested cells if there is sulphur impurity of e.g. 100 ppb in the inlet gas? (is 100 ppb a realistic number?)

B) Si-impurities e.g. from the sealing material:

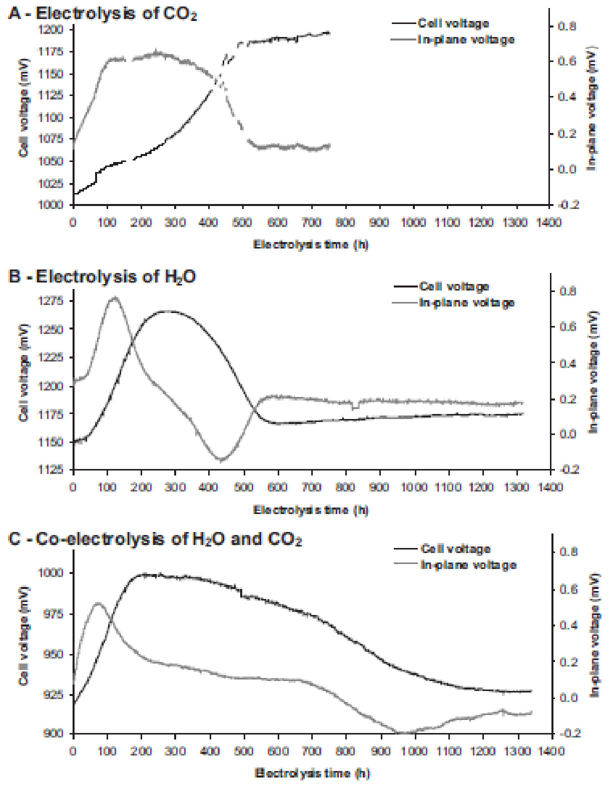
At the test conditions given the $p(Si(OH)_4)$ originating from the relevant glass sealing is app. $2{\cdot}10^{-8}$ atm (still a flow of 25 h/l was used for each electrode).

C) Impurities in raw materials:

Calculate the total amount (mass) of SiO_2 in the raw materials in the half cell (i.e. electrolyte, H_2 electrode and support layer) based on:

"The cells had a 10–15 μ m thick hydrogen electrode of Ni/YSZ cermet and were supported by a 300 μm thick Ni/YSZ layer, a 10–15 μm thick YSZ electrolyte, and a 15–20 μm thick LSM-YSZ composite oxygen electrode. The ratio between Ni and YSZ (ZrO_2 stabilized with 8 mol % Y_2O_3) was 40/60 vol % both for the support layer and the active electrode layer. The active electrode had an area of 16 cm^2. The porosity is app. 30%. The Si content was app. 12 ppm for both Ni and YSZ raw materials"

(ρ(Ni)=8.9 g/cm^3, ρ(YSZ)=5.9 g/cm^3, M(Ni)=58,7 g/mol, M(YSZ)= 123 g/mol and M(SiO_2)=60 g/mol)



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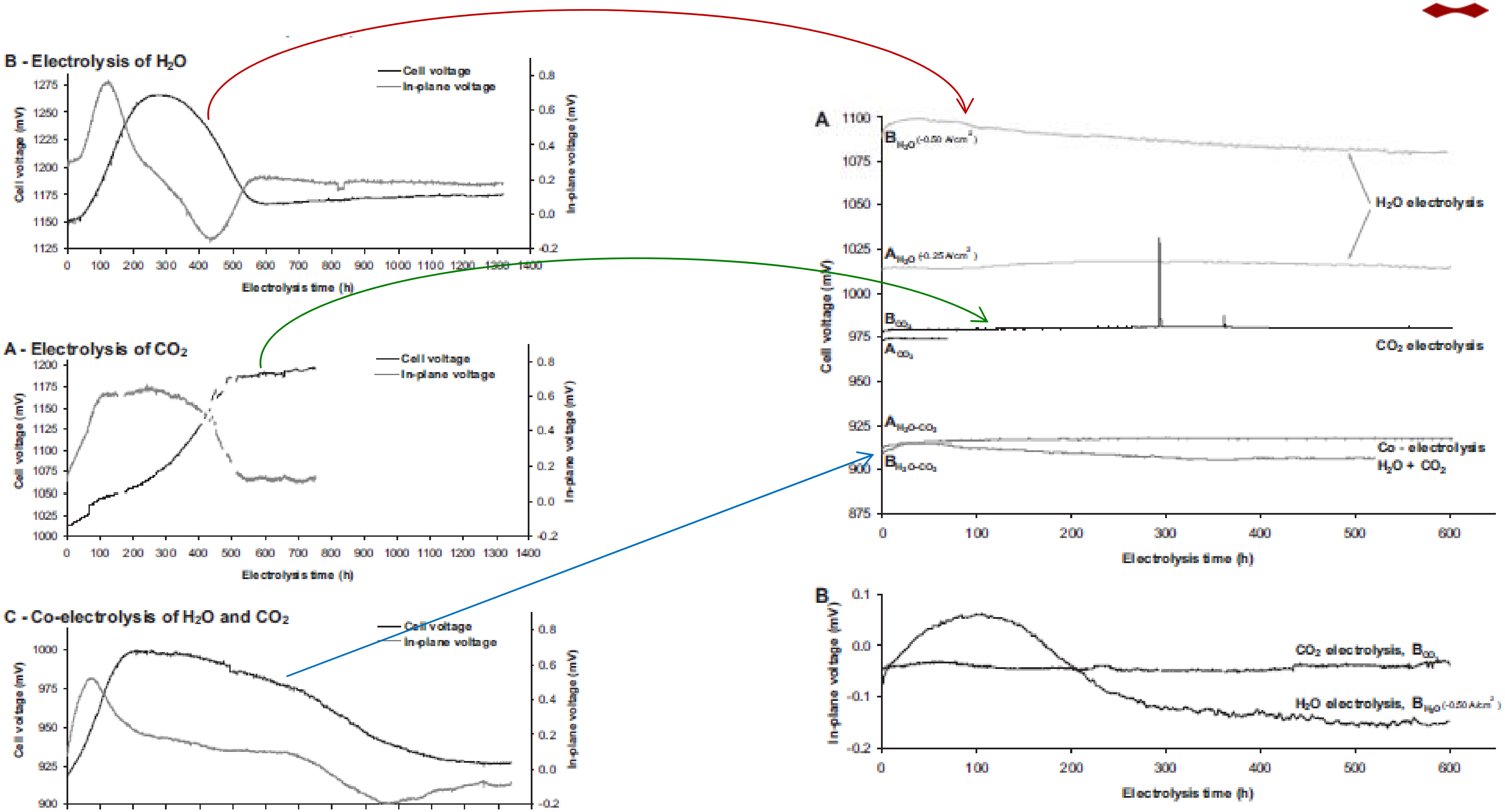
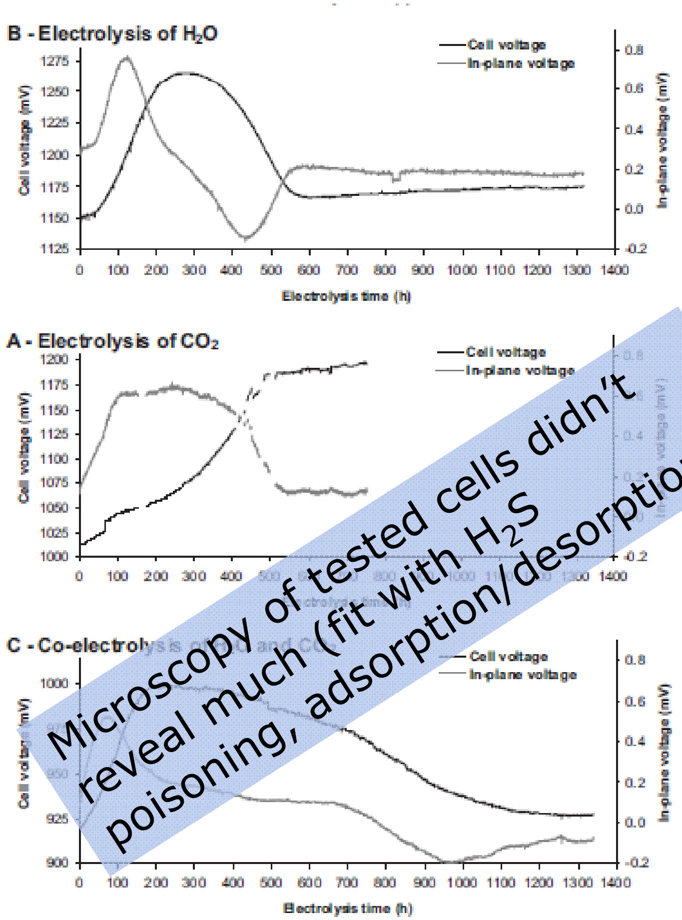


Figure 7. (A) Cell voltage measured during CO_2 electrolysis (850°C, −0.25 A/cm^2, 70% CO_2–30% CO), H_2O electrolysis (850°C, −0.25 A/cm^2 and −0.50 A/cm^2, 50% H_2O–50% H_2), and during co-electrolysis of CO_2 and H_2O (850°C, −0.25 A/cm^2, 45% CO_2–45% H_2O–10% H_2) with cleaned inlet gases. The increase in cell voltage during CO_2 electrolysis after 295 and 363 h of operation was caused by a sensor break in the oven temperature control causing a lowering of the cell temperature to 795 and 835°C, respectively. (B) Corresponding in-plane voltage at the Ni/YSZ electrode measured during either H_2O electrolysis (B_{H_2O}) or CO_2 electrolysis B_{CO_2}.

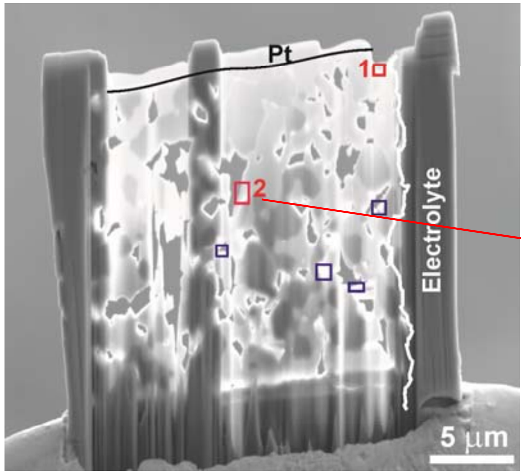
Ref. Ebbesen, Graves, Hauch, Jensen & Mogensen, J. Electrochem. Soc., 157(10), B1419 (2010)

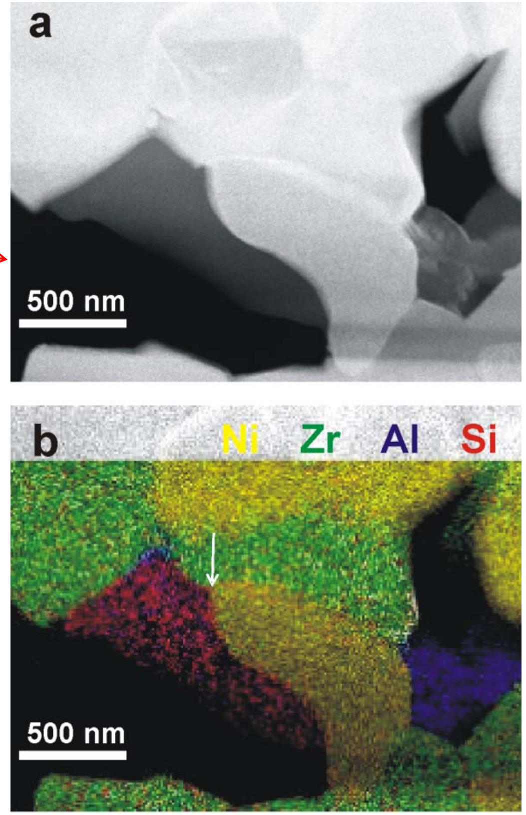
6. Materials & durability – gas purity/impurity

Postmortem microscopy…



Microscopy of tested cells didn't reveal much (fit with H_2S poisoning, adsorption/desorption)





Element	Na	Al	Si	Ni	Cu	Zr
Metal atom%	13±2	36±2	41±3	2±1	7±1	1±1

Conclusion:

Impurities can play a significant role in SOE durability
– and to some extend different compared to SOFC

Ref. Ebbesen, Graves, Hauch, Jensen & Mogensen, J. Electrochem. Soc., 157(10), B1419 (2010)

Ref. Hauch, Bowen, Kuhn & Mogensen, Electrochem. & Solid State Lett., 11(3), B38 (2008)

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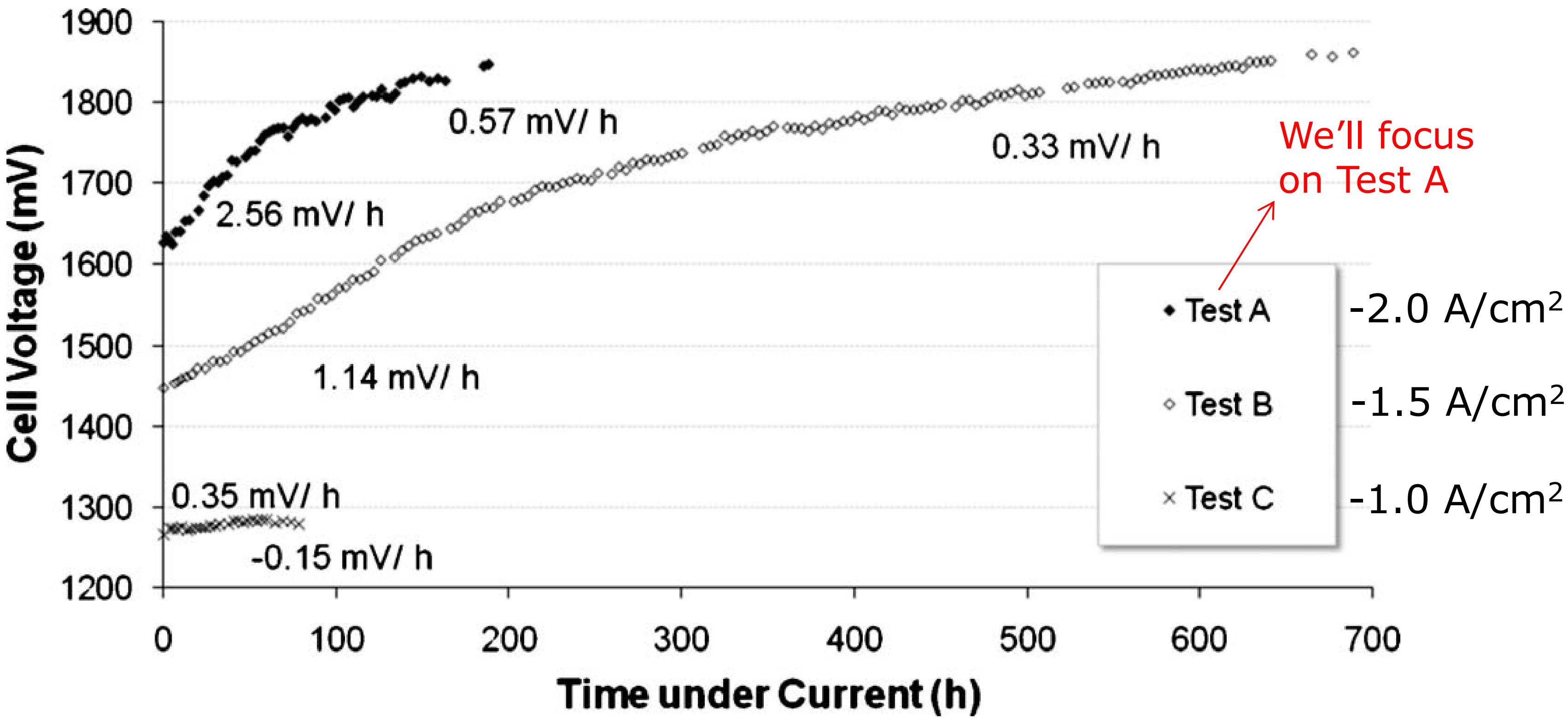
Electrolyte/O_2 electrode interface degr. at high current densities

850° C ,50:50 $H_2O:H_2$ to the Ni/YSZ electrode and O_2 to the oxygen electrode



Ref. Knibbe, Traulsen, Hauch, Ebbesen & Mogensen, *J. Electrochem. Soc.*, **157**(8), B1209, 2010

9. SOE – Other durability and degradation issues

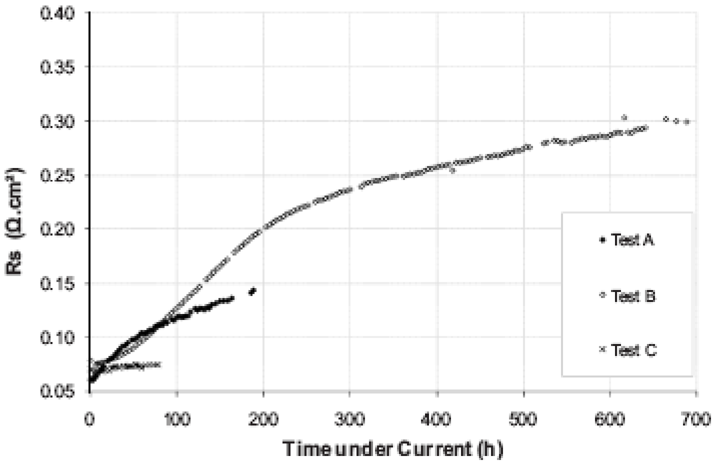


Figure 4. Ohmic resistance degradation with time for tests A (-2.0 A cm^{-2}), B (-1.5 A cm^{-2}), and C (-1.0 A cm^{-2}).

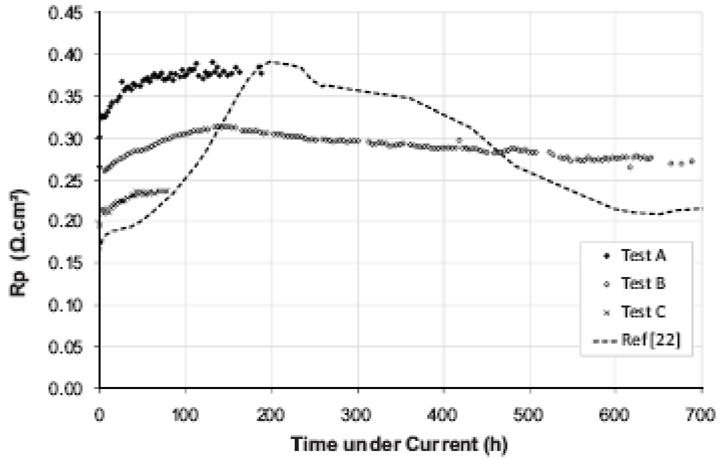


Figure 5. Electrode polarization resistance degradation with time for tests A (-2.0 A cm^{-2}), B (-1.5 A cm^{-2}), and C (-1.0 A cm^{-2}). R_p degradation from Ref. 22 included for reference point [850°C, 50:50 (H_2O:H_2), -0.5 A cm^{-2} with Si seals].

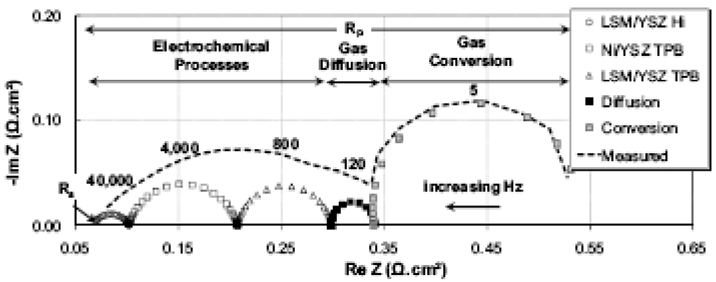


Figure 2. Impedance spectra obtained at 850°C with 50:50 (H_2O:H_2) supplied to the Ni/YSZ hydrogen electrode, oxygen supplied to the LSM/YSZ oxygen electrode, and current density of -1.5 A cm^2. Characteristic process frequency of above given process arc.

Main conclusion:

High current densities
$\Rightarrow R_s$ increase (also some R_p increase)

Low current
$\Rightarrow R_s$ constant

What can we learn from post-mortem/microscopy?

Electrolyte/O_2 electrode interface degr. at high current densities

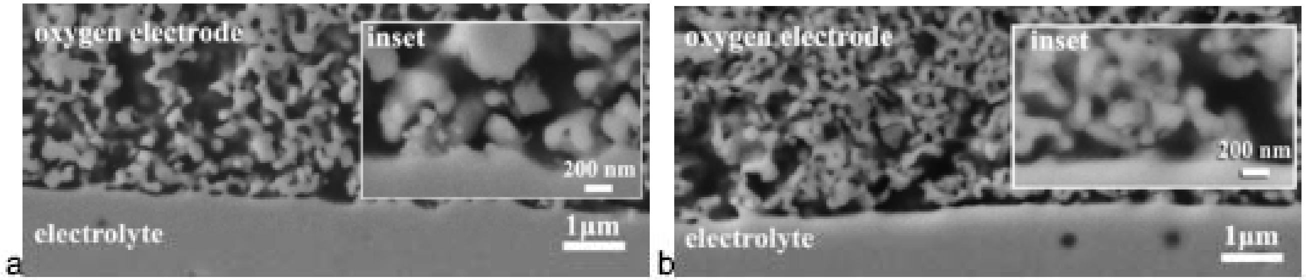


Figure 8. Oxygen electrode overview: (a) Test A (-2.0 A cm^{-2}) and (b) reference. Low magnification SE2 image with higher magnification inset.

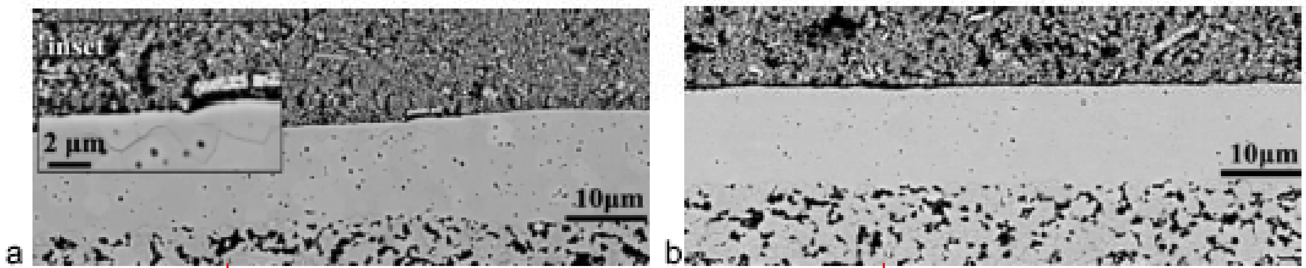


Figure 9. Electrolyte overview: (a) Test A (-2.0 A cm^{-2}) inset of intergranular fracture running along grain boundary and (b) test C (-1.0 A cm^{-2}).

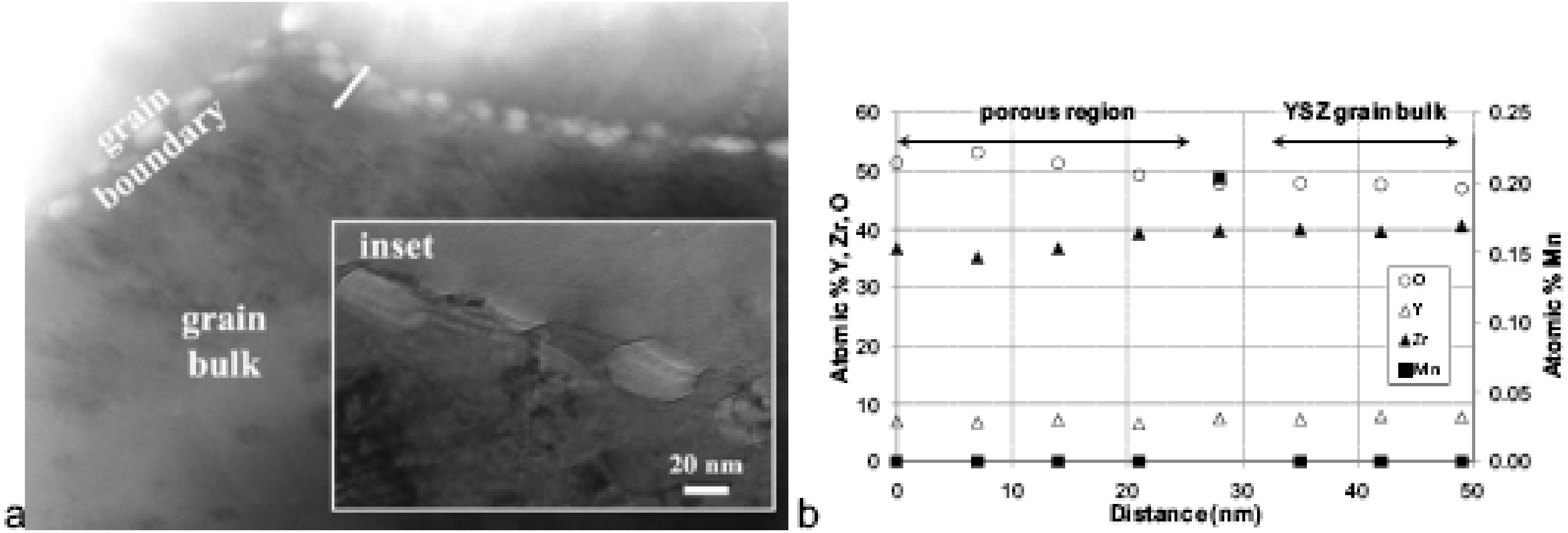
Cell with R_s increase (-2 A/cm²)

Cell with R_s constant (-1 A/cm²)

TEM study of the YSZ grain boundaries…. →

Ref. Knibbe, Traulsen, Hauch, Ebbesen & Mogensen, *J. Electrochem. Soc.*, **157**(8), B1209, 2010

9. SOE – Other durability and degradation issues



TEM of YSZ grain boundary near oxygen electrode from cell tested at -2 A/cm^2 (R_s increase)

Testing of similar cells at 1.9 A/cm^2 (FC) do not show similar Rs degr. & YSZ grain boundary changes (A. Hagen et al., *J. Electrochem. Soc*)...⟶

9. SOE cells – Other durability and degradation issues

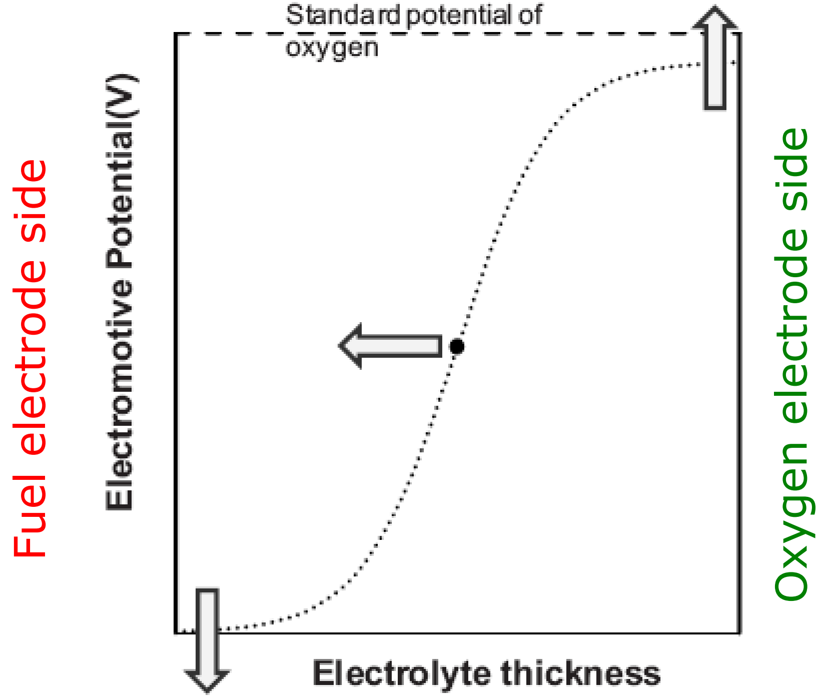
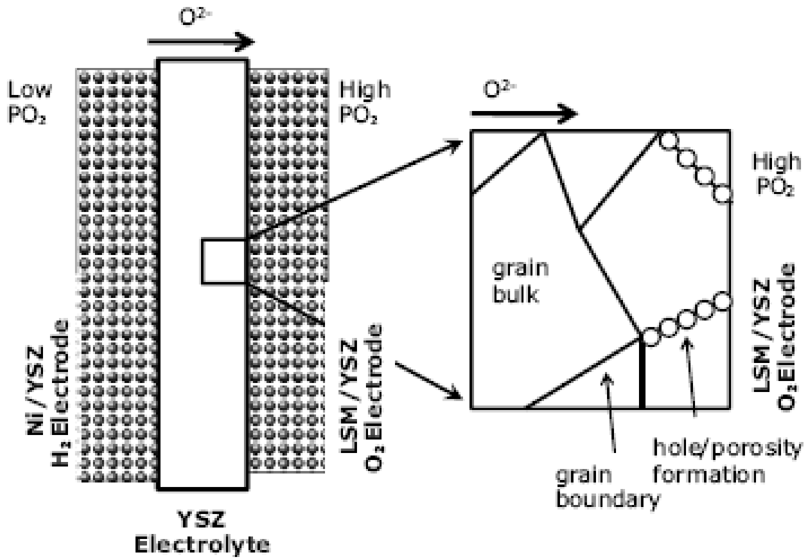


Figure 11. Sketch of electromotive potential distribution through electrolyte thickness with the hydrogen electrode to the left and the oxygen electrode to the right. The arrows indicate the electromotive potential distribution changes with increase in SOEC current density.

Arrows indicate changes from OCV to electrolysis testing.



Nucleation and growth of oxygen in the YSZ grain boundaries (near O_2 electrode)

Formation of pores in the grain boundaries ⇒ very high grain boundary resistivity, which increases the YSZ ohmic resistance.

Ref. Knibbe, Traulsen, Hauch, Ebbesen & Mogensen, *J. Electrochem. Soc.*, **157**(8), B1209, 2010

9. SOE cells – Summary: Durability & degradation issues

For the hydrogen/steam electrode:

Ni/YSZ advantages:

Well-known, inexpensive electrode material

Ni highly catalytic active for H_2O reduction (and for CO_2 red. as well)

Ni/YSZ disadvantages:

Prone to impurities (Sc-doping could be an alternative option)

Ni evaporation (only relevant at high temp. and high $p(H_2O)$)

Ni coarsening (all ceramic based electrode could be an alternative)

For the oxygen electrode and electrolyte/oxygen electrode:

Delamination of the oxygen electrode (dependent on actual electrode/microstructure)

Oxygen formation in YSZ grain boundaries

A couple of review papers:

Sohal et al., *J. Fuel Cell Science and Technology*, **9**(1), 11017 (2012)

Hauch et al., *J. Mater. Chem.*, **18**, 2331 (2008)

Knibbe et al., *Green*, 1(2), **141** (2011)

Laguna-Bercero, *J. Power Soruces*, **203**, 4 (2012)

Outline

1. Intro – who am I and who are you? + learning objectives
2. Repetition of basic working principle – *small exercise*
3. SOE from a CO_2 cycle point of view
4. SOE from a economy / fuel production prices point of view

5. SOE cells – Materials & initial performance (H_2O / CO_2 / Co-electrolysis)
6. SOE cells – Materials & durability
7. *Assignment* – Examples of impurity related degradation of SOE cells

Thursday Sept. 27th 2012

8. Feedback on assignment on impurity related degradation of SOE cells
9. SOE cells – Other durability and degradation issues
10. SOE cells – Where are we heading?

11. Summing up / leaning objectives

10. SOE cells – Where are we heading?

I. Increased life time under realistic/technological relevant conditions

II. Alternative electrode materials and cell design

– all ceramic fuel electrodes, Sc-doped electrode, Sc-doped electrolyte, MIEC oxygen electrode (demands for barrier layer!)

III. Cycling between SOFC and SOEC

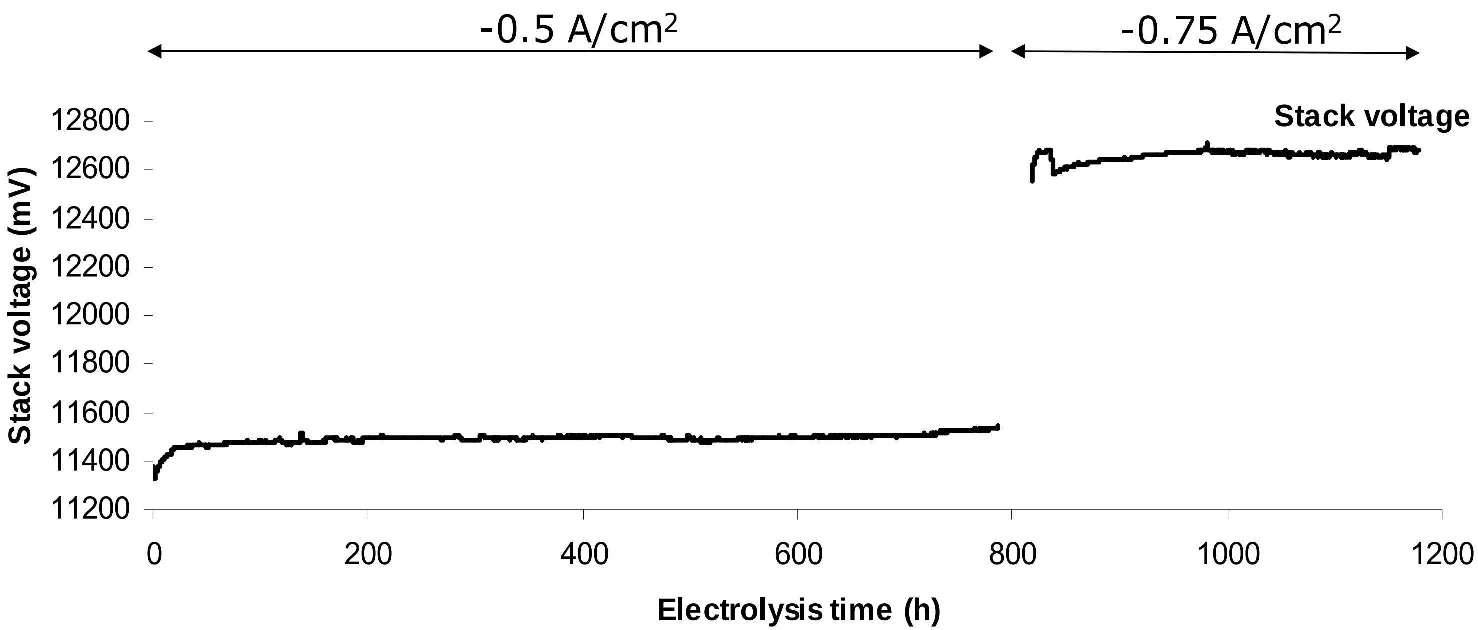
– interesting from at technological point of view

IV. Pressurized test

V. Stack testing

10. SOE cells – Where are we heading?

Stack testing – a couple of examples



Durability during co-electrolysis; 10-cell stack at 850 ºC with 45 % H_2O – 45 % CO_2 – 10 % H_2 supplied to the Ni-YSZ electrode, dry O_2 was supplied to the LSM-YSZ electrode

Ref. Ebbesen, Høgh, Agersted, Nielsen & Mogensen, *Int. J. Hydrogen Energy*, **36**, 7363 (2011)

10. SOE cells – Where are we heading?

Stack testing – a couple of examples

Also other groups working with SOEC stack testing e.g. Brisse/Schefold at CEA, and O'Brien/Stoots at INL (see graph below from "Status of the INL High-Temperature Electrolysis Research Program – Experimental and Modeling", 4th Information Exchange Meeting on the Nuclear Production of Hydrogen, 2009)

800 °C, steam electrolysis, -0.15 A/cm^2 (as far as I can read…)

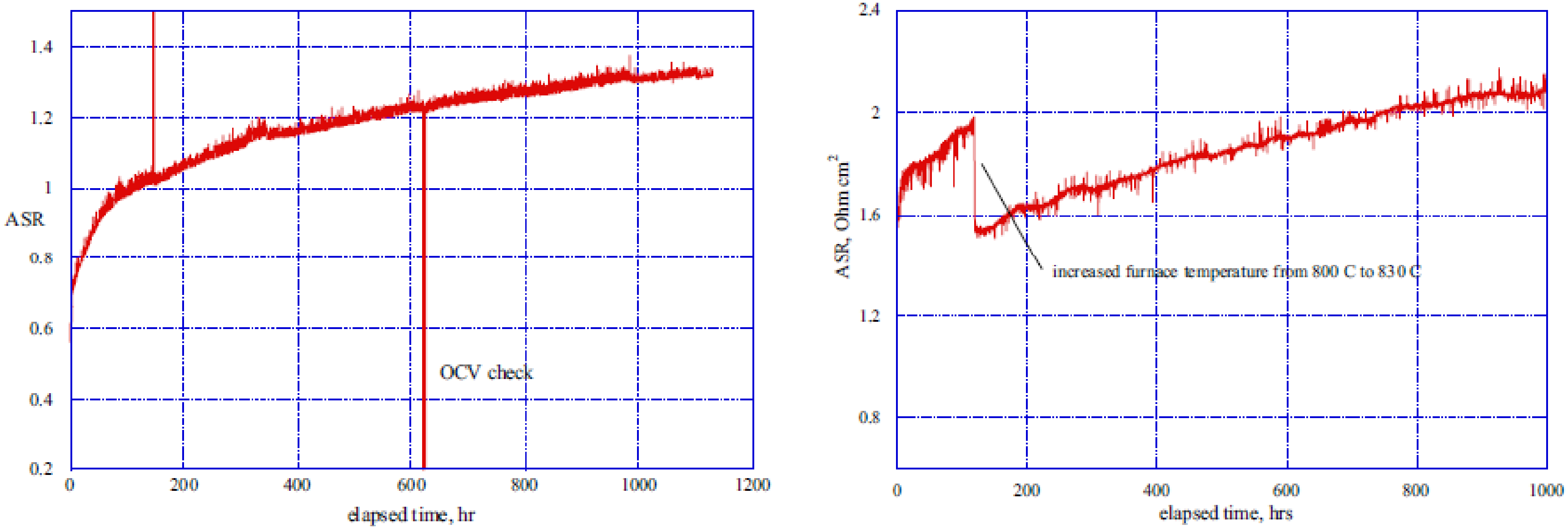
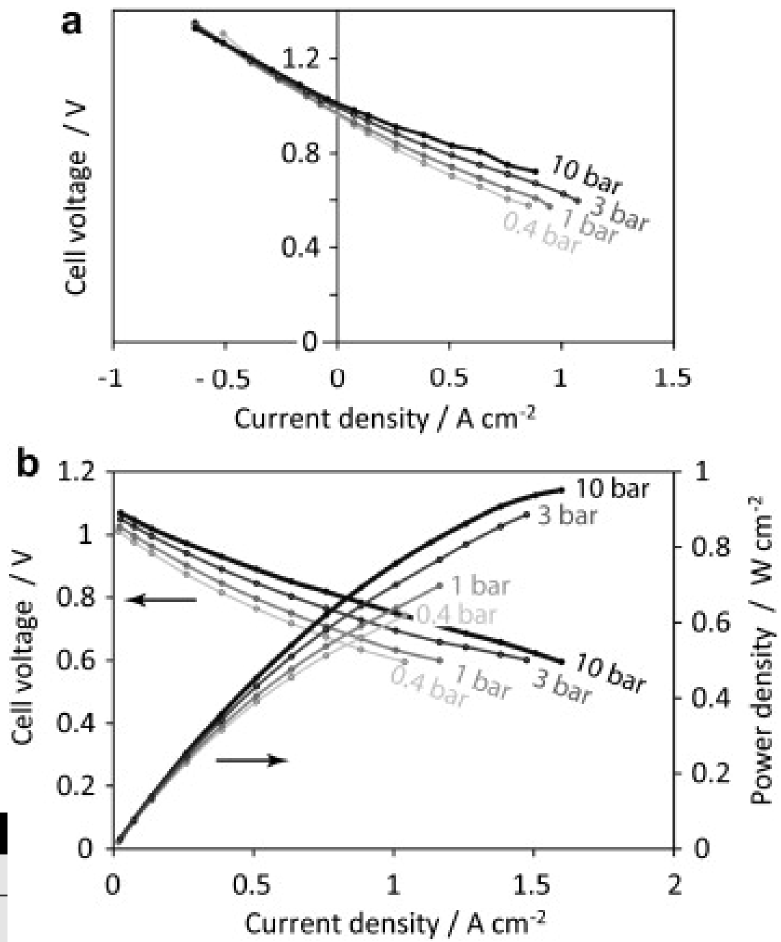


Figure 9. (a) Area-specific resistance of a button cell as a function of time for 1100-hour test; (b) Area-specific resistance of a 25-cell stack as a function of time for a 1000-hour test.

Pressurized test

Production of synthetic hydrocarbon fuels are normally operated at pressures exceeding 40 bars!



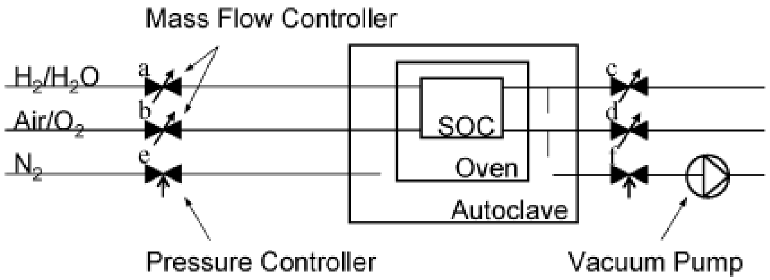


Table 1 – OCV and ASR measured at 750 °C at various pressures and Ni/YSZ-electrode gas compositions.

Pressure (bar)	50% H_2 + 50% H_2O			80% H_2 + 20% H_2O			
	OCV (V)		ASR (Ω cm^2)	OCV (V)		ASR (Ω cm^2)	
	Theory	Measured	i–V	Theory	Measured	i–V	Impedance
0.4	0.972	0.961	0.59	1.033	1.021	0.62	0.64
1	0.992	0.969	0.52	1.053	1.037	0.55	0.56
3	1.016	0.996	0.47	1.077	1.062	0.49	0.53
10	1.043	1.011	0.42	1.104	1.084	0.40	

Ref. Jensen, Sun, Ebbesen, Knibbe & Mogensen, *Int. J. Hydrogen Energy*, **35**(18), 9544 (2010) + SOC references in this.

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11. Summing up using Learning objectives

Learning objectives – 5 minutes – only pen and paper:

1. Explain/sketch qualitatively how electrolysis (potentially) can become an important player in a future energy grid?

2. Explain the basic operation principle for steam electrolysis, CO_2 electrolysis, co-electrolysis

3. List typical electrode materials incl. requirements and operation conditions

4. Give examples of SOEC degradation issues and explain why some of these can be somewhat different from what can be observed for similar SOFCs (based on material properties, test conditions etc.)

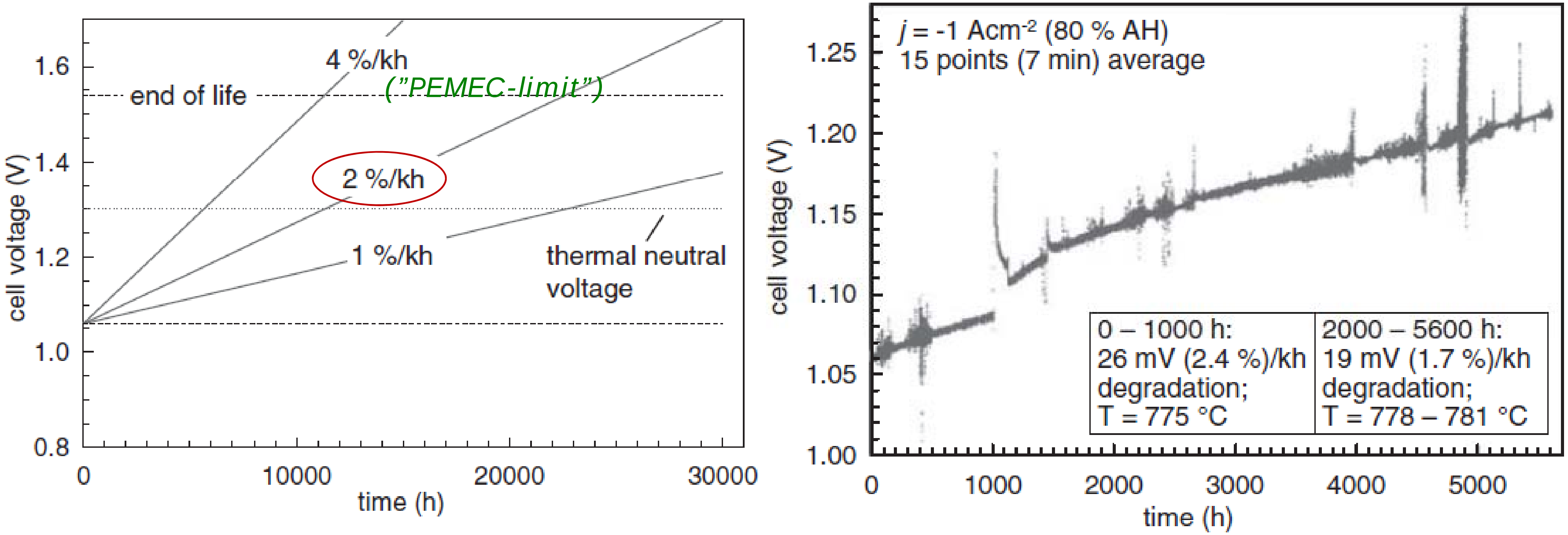
Evaluation – SOE part / 2 lectures

Maintain	Improve	Muddy point	New insight

EXTRA SLIDES

10. SOE cells – Durability

Durability



Single cell, steam electrolysis, Ni/YSZ-YSZ-CGO-LSCF/CGO cell.

Ref. Shefold, Brisse & Tietz, J. Electrochem. Soc., 159 (2), A137 (2012)