

# **Program & Abstracts**

*International Conference on Electrolysis*

June 9-13, 2019

**Hotel Alexandra**

Loen, Norway

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## Welcome

Dear Participant,

We continue the success of the first international electrolyser conference (ICE2017) in Copenhagen where a whole conference was devoted to the topic of electrolysis for energy conversion. ICE2019 will be the second instalment of what we hope will be a thriving conference series on electrolysis for energy conversion.

The research in all aspects of electrolysis for energy conversion and storage is growing rapidly. Demonstration and commercial installation of electrolysers coupled with renewable energy sources have grown from sub-MW installations a few years ago to tens of MW today. There is an increased recognition and awareness that the conversion of electrical energy to storable chemicals and fuels is an inevitable part of the green transition from a fossil based to a sustainable energy system.

Now we are gathered here at the ICE2019. We will do our utmost to host you all, but only you – the participants – can ensure the quality and success of the conference.

On behalf of the scientific and organising committees,

Welcome to ICE2019 in Loen, Norway!

## Organisation

### International Scientific Committee

**Dmitri Bessarabov**, HySA Infrastructure at North-West University (ZA)

**Marcelo Carmo**, Forschungszentrum Jülich (DE)

**EunAe Cho**, Korea Advanced Institute of Science and Technology (KR)

**John T. S. Irvine**, University of St Andrews (UK)

**Pierre Millet**, Université Paris Sud (FR)

**Bryan Pivovar**, National Renewable Energy Laboratory (US)

**Yang Shao-Horn**, Massachusetts Institute of Technology (US)

**Tom Smolinka**, Fraunhofer ISE (DE)

**Mogens Bjerg Mogensen**, DTU Energy (DK)

**James O'Brien**, Idaho National Laboratory (US)

**Jens Oluf Jensen**, DTU Energy (DK)

**Magnus Thomassen**, SINTEF (NO)

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**Anita Reksten**, SINTEF (NO)

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**Anders Ødegård**, SINTEF (NO)

**Thulilie Kouza**, SINTEF (NO)

**Alejandro Oyarce Barnett**, SINTEF (NO)

The conference is organised by



## Sponsors

The organisers acknowledge financial support from



Forskningsrådet  
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## Invited speakers

**Peter Veenstra** - Shell Global Solutions (NL)

**Gregory Jerkiewicz** - Queens University (CA)

**Annabelle Brisse** - Eifer (FR)

**Peter Strasser** - TU Berlin (DE)

**R.J. (Cobus) Kriek** - North West University (SA)

**Chang-Hee Kim** - Korea Institute of Energy Research (KR)

**Truls Norby** - University of Oslo (NO)

**Elena R Savinova** - University of Strasbourg, CNRS (FR)

**Marcelo Carmo** - Forschungszentrum Jülich (DE)

**Peter Blennow** - Haldor Topsøe A/S (DK)

**Nikolaos Lymperopoulos** - Fuel Cell and Hydrogen Joint Undertaking (BE)

**Kathy Ayers** - NEL Hydrogen / Proton Onsite (US)

**Yu Morimoto** - Toyota Central R&D Labs (JP)

**Oliver Posdziech** - Sunfire (DE)

## Programme overview

	Sun 09	Mon 10	Tue 11	Wed 12	Thu 13
08:20		Opening - Welcome		Session 9 SOEC	
08:40		Session 1 PEMEL	Session 5 SOEC		Session 11 PEMEL
09:00					
09:20					
09:40					
10:00					
10:20			Coffee		
10:40		Coffee	Session 6 AEL	Coffee	Coffee
11:00		Session 2 AEL		Session 10 AEL	Session 12 AEL
11:20					
11:40					
12:00					
12:20					
12:40		Lunch	Lunch	Lunch	Closing of conference
13:00					
13:20					
13:40		Session 3 SOEC	Session 7 PEMEL	Social programme	
14:00					
14:20					
14:40					
15:00					
15:20					
15:40		Coffee	Coffee		
16:00	Registration	Tutorials	Session 4 Other	Session 8 PEMEL	
16:20					
16:40					
17:00					
17:20					
17:40					
18:00			Welcome reception & Poster 1	Poster 2	
18:20					
18:40					
19:00	Dinner				
19:30		Dinner	Dinner		
20:00				Conference dinner	
20:30					
21:00					
21:30					
22:00					
22:30					
23:00					

## Detailed programme

### Monday June 10<sup>th</sup>

08:20	Welcome, opening remarks	<b>Magnus S. Thomassen</b> SINTEF Norway	
08:40-10:40	Session 1. PEMEL	Chair: Marcelo Carmo	No.
08:40	Energy for a changing world, Reactor Engineering aspects related to <i>GREEN CHEMISTRY</i>	<b>Peter Veenstra (INVITED)</b> Shell Global Solutions International B.V. Netherlands	1
09:00	Understanding electric current reversal phenomena in PEM water electrolysis cells	<b>Christoph Immerz</b> Leibniz Universität Hannover Germany	2
09:20	Current density distribution as a function of PEM electrolyser flow-field design by in-situ neutron imaging	<b>Dmitri Bessarabov</b> HySA Infrastructure South Africa	3
09:40	Local measurement of anode current collector potential in a PEM water electrolyser	<b>Hans Becker</b> National Physical Laboratory United Kingdom	4
10:00	Progress in the development of Pt-free cathodes for PEM water electrolysis	<b>Pierre Millet</b> Paris-Sud University France	5
10:20	Minimizing the Differential Cell Resistance of PEM Electrolysis Cells – A Hypothesis based on EIS Calculations	<b>Katrine Elspø</b> IRD Fuel Cells A/S Denmark	6
10:40	Coffee Break		
11:00-12:40	Session 2. AEL	Chair: EunAe Cho	No.
11:00	Surface and Materials Science, and Electrochemical Analysis of Nickel Materials	<b>Gregory Jerkiewicz (INVITED)</b> Queen's University, Canada	7
11:20	Highly efficient anion exchange membrane water electrolysis and the role of KOH concentration	<b>Alejandro Barnett</b> SINTEF Norway	8
11:40	Microstructural optimization of gas diffusion electrodes for high temperature and pressure alkaline electrolysis	<b>Christodoulos Chatzichristodoulou</b> Technical University of Denmark Denmark	9
12:00	Porous Electrodes as Efficient Catalysts for the Oxygen Evolution Reaction	<b>Thomas Rauscher</b> Fraunhofer Institute for Manufacturing Technology and Advanced Materials IFAM Germany	10
12:20	Intensification of alkaline water electrolysis using 3-D electrodes, forced electrolyte flow and pulsed voltage	<b>Grégoire Thunis</b> Université catholique de Louvain Belgium	11
12:40	Lunch		



## Monday June 10<sup>th</sup> (Continued)

13:40-15:40	Session 3. SOEC	Chair: Annabelle Brisse	No.
13:40	Status of Sunfire's Large-Scale High-Temperature Electrolysis	<b>Oliver Posdziech (INVITED)</b> Sunfire GmbH Germany	12
14:00	Power-to-X activities at Haldor Topsoe: a stepping-stone approach towards commercialization	<b>Bengt P. G. Blennow (INVITED)</b> Haldor Topsoe A/S Denmark	13
14:20	Development of metallic interconnects manufactured by powder metallurgy for solid oxide electrolyser systems	<b>Mari Carmen Monterde</b> AMES PM Tech Center Spain	14
14:40	A 25 kW High Temperature Electrolysis Facility for Flexible Hydrogen Production and System Integration Studies	<b>James E. O'Brien</b> Idaho National Laboratory USA	15
15:00	Power-to-X with high temperature Solid Oxide Cells	<b>Remi Costa</b> German Aerospace Center Germany	16
15:20	Enhanced Value of Renewable Energy via High Temperature Electrolysis	<b>Olga A. Marina</b> Pacific Northwest National Laboratory USA	17
15:40	Coffee Break		
16:00-18:00	Session 4. Other	Chair: Bryan Pivovar	No.
16:00	11 years of FCH JU support to electrolyser development and demonstration	<b>Nikolaos Lymperopoulos (INVITED)</b> Fuel Cells and Hydrogen Joint Undertaking	18
16:20	PERIC's development on Power to Gas	<b>Chen Tianshan</b> Purification Equipment Research Institute of CSIC China	19
16:40	Effect of power quality on the specific energy consumption of water electrolyzers	<b>Joonas Koponen</b> LUT University Finland	20
17:00	OxEon Energy Developments Targeting Synthetic Liquid Fuels Production Using Non-Fossil CO <sub>2</sub> as a Store of Renewable Energy	<b>Joseph Hartvigsen</b> OxEon Energy, LLC USA	21
17:20	Hydrogen from Molten Carbonate electrolysis for green steel production	<b>Andries Krüger</b> KTH Royal Institute of Technology Sweden	22
17:40	Towards an atomistic understanding of electrocatalytic partial hydrocarbon oxidation: theory and experiments synergies	<b>Luca Silvioli</b> University of Copenhagen Denmark	23
18:00-19:30	Welcome reception & Poster 1		
19:30	Dinner		

## Tuesday June 11<sup>th</sup>

08:40-10:20	Session 5. SOEC	Chair: James O'Brien	No.
08:40	Performance and long-term stability of electrolyte supported Solid Oxide Electrolyser Cells	<b>Annabelle Brisse (INVITED)</b> European Institute for Energy Research Germany	24
09:00	Advancement of reversible proton-conducting solid oxide cells at Idaho National Laboratory (INL)	<b>Dong Ding</b> Idaho National Laboratory USA	25
09:20	On the development of electrodes for tubular proton ceramic electrolyzers for pressurized hydrogen production	<b>Marie-Laure Fontaine</b> SINTEF Norway	26
09:40	Scale up and integration of proton-conducting ceramics into multi-cell stacks	<b>Neal Sullivan</b> Colorado Fuel Cell Center USA	27
10:00	An Evaluation of High Temperature Water Splitting Systems using Protonic Ceramic Electrolyzers	<b>Robert Braun</b> Colorado School of Mines USA	28
10:20	Coffee Break		
10:40-12:40	Session 6. AWE	Chair: Jens Oluf Jensen	No.
10:40	Catalyst Development for PEM and AEM Water Electrolyzer Anodes	<b>Peter Strasser (INVITED)</b> Technical University Berlin Germany	29
11:20	Operando X-ray absorption investigations into the role of Fe in the electrochemical stability and oxygen evolution activity of Ni <sub>1-x</sub> Fe <sub>x</sub> O <sub>y</sub> nanoparticles	<b>Daniel Abbott</b> Paul Scherrer Institut Switzerland	30
11:40	Oxygen evolution at porous Ni electrodes	<b>Daniel Guay</b> INRS – EMT Canada	31
12:00	High performing and economic platinum group metal free anode catalysts for AEM and PEM electrolyzers – Opportunities and Challenges	<b>Li Wang</b> German Aerospace Center (DLR) Germany	32
12:20	Electrolysis market opportunities	<b>Marius Bornstein</b> NEL Hydrogen Norway	33
12:40	Lunch		

## Tuesday June 11<sup>th</sup> (Continued)

13:40-15:40	Session 7. PEMEL	Chair: Dmitri Bessarabov	No.
13:40	Low Temperature Water Electrolysis at Large Scale: A Comparison of Technology Benefits and Challenges	<b>Katherine Ayers (INVITED)</b> Nel Hydrogen US USA	34
14:00	REFHYNE – 10 MW PEM electrolyser for refinery	<b>Anders Ødegård</b> SINTEF Norway	35
14:20	PEM electrolysis development for enhancing renewable energy integration and advancing Power-to-X technologies	<b>Wouter Schutyser</b> Hydrogenics Europe NV Belgium	36
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15:00	Degradation analysis at increased stressor level in PEM water electrolysis single cells	<b>Tom Smolinka</b> Fraunhofer Institute for Solar Energy Systems Germany	38
15:20	System relevant Observation of Gas Crossover – Necessity of Mitigation Strategies	<b>Patrick Trinke</b> Leibniz Universität Hannover Germany	39
15:40	Coffee Break		
16:00-18:00	Session8. PEMEL	Chair: Pierre Millet	No.
16:00	Cobalt Platinum Bronze for an Active and Durable OER Electrocatalyst of PEM Electrolysis without Ir or Ru	<b>Yu Morimoto (INVITED)</b> Toyota Central R&D Labs Japan	40
16:20	Improving the performance of low loaded PEMWE electrodes	<b>Friedemann Hegge</b> Forschungszentrum Jülich GmbH Germany	41
16:40	High performing PEMEC MEAs with (ultra)-low PGM-loading	<b>Laila Grahl-Madsen</b> IRD Fuel Cells A/S Denmark	42
17:00	Low Temperature Electrolysis Advances at NREL	<b>Bryan Pivovar</b> National Renewable Energy Lab (NREL) USA	43
17:20	Investigation on the Effect of Ionomer Loading and Catalyst Loading on Tantalum Carbide Support on Polymer Electrolyte Membrane Electrolyser Performance	<b>Rutendo Mutambanengwe</b> Queen's University Canada	44
17:40	Direct membrane deposition – a novel membrane electrode assembly for proton exchange membrane water electrolysis	<b>Peter Holzapfel</b> Forschungszentrum Jülich GmbH Germany	45
18:00-19:30	Poster 2		
19:30	Dinner		

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08:20-10:40	Session9. SOEC	Chair: Mogens Mogensen	No.
08:20	Proton ceramic electrochemical cells for efficient hydrogen production	<b>Truls Norby (INVITED)</b> University of Oslo Norway	46
08:40	Cobalt substituted Lanthanide Nickelates (Ln <sub>2</sub> Ni <sub>1-x</sub> CoxO <sub>4+δ</sub> , Ln = La, Pr; x=0, 0.1, 0.2): Impact on Electrochemical Performance and Stability as SOECs Oxygen Electrode	<b>Vaibhav Vibhu</b> Forschungszentrum Jülich GmbH Germany	47
09:00	Degradation Phenomena in Solid Oxide Electrolysis Cell Fuel Electrodes	<b>Scott A. Barnett</b> Northwestern University USA	48
09:20	Experimental analysis of SOE stacks under pressurized operation	<b>Marc Riedel</b> German Aerospace Center (DLR) Germany	49
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10:20	Demonstration of reversible Solid Oxide Cell technology: results at cell, stack and system level	<b>Olivier Thomann</b> VTT Technical Research Centre of Finland Finland	52
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11:40	For When Materials Matter	<b>Nick van Dijk</b> PV3 Technologies Ltd United Kingdom	55
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13:40-19:00	Social program		
20:00	Conference Dinner		

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## Poster session 1

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## Poster Session 2

**Tuesday June 11<sup>th</sup>, 18:00-19:30**

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## Abstracts

Abstract No 1 (INVITED)

### **Energy for a changing world, Reactor Engineering aspects related to *GREEN* *CHEMISTRY***

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An overview will be given of some of the reactor engineering aspects in the field of green chemistry. Several interfaces and language barriers between disciplines will be highlighted, including the interface between material science, electro-chemistry and fluid flow, and the interface between scaling-up and the integration with the process engineering discipline. Several projects in high temperature electrolysis and alkaline electrolysis will be used as examples. Furthermore, the opportunities with the fast-evolving world of modelling and digitalisation will be discussed.

## Understanding electric current reversal phenomena in PEM water electrolysis cells

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The integration of power-to-gas systems into the energy infrastructure of the future will be characterised by an increasing degree of flexibility in operating strategies [1]. A proton exchange membrane water electrolysis (PEMWE) system, which is able both to operate at continuous loads and to absorb or quickly reduce dynamic generation peaks, has significant competitive advantages in order to operate power-to-gas plants economically [2].

The basic prerequisite for these operating strategies is a highly dynamic response behavior of various electrical, electronic and electrochemical system components. In the context of this work, the electrical response of a single cell to load steps of constant current (galvanostatic), constant voltage (potentiostatic) and constant power is investigated.

Potentiostatic power steps from high (1.76 V) to low voltages (1.45 V) result in a change of the current density flow direction in the initial step response (cross-symbols in Fig. 1a). The experimental results are further analyzed using a simple mathematical model in order to provide a generic understanding of the current reversal phenomena (s. Fig. 1 b).

The model considers the internal processes as interactions between capacitive and Faradaic currents. Therefore, downwards voltage steps lead to a discharge of the double layers in both half-cells, which causes instantly high capacitive current flows. While the Faradaic current flows remain unchanged, the discharge capacitive currents have the opposite direction. Depending on how large the capacitive currents are, the integral current direction is reversed as it is a sum of both, the faradaic and the capacitive current.

PEMWE cells are able to follow load changes without affecting the electrolysis operation. Additionally, the results indicate that the PEMWE cells meet the electro-technical requirements for a dynamic operation mode. With this analysis a first insight in the overall cell behaviour is given, which can be extended to a more detailed analysis of the processes in the cell during load changes in future works.

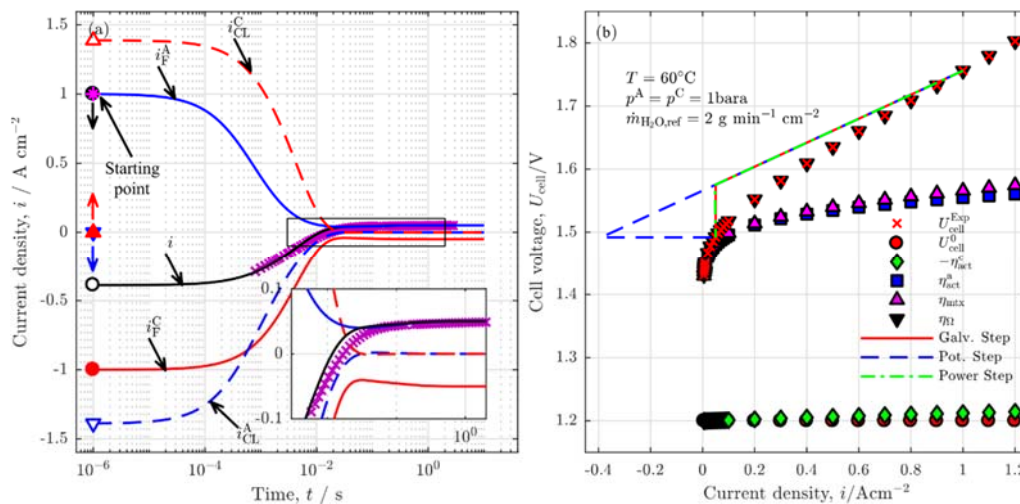


Figure 1: (a) Transient current flows for a potentiostatic load step from 1.76 V (1.0 A cm<sup>-2</sup>) to 1.45 V (0.05 A cm<sup>-2</sup>) experimentally measured (cross symbols) and modelled. (b) Initial response curves of the same step galvanostatic, potentiostatic and with constant power in polarisation curve, similar to fuel cells [3].

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Abstract No 3

## **Current density distribution as a function of PEM electrolyser flow-field design by in-situ neutron imaging**

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PEM electrolyser plates with various flow-field designs were fabricated by HySA Infrastructure and tested in a proton exchange membrane (PEM) electrolyser single cell with an active area of 5cm × 5cm. The anode flow-field in a PEM electrolyser cell is used for the water feed which could offer an additional benefit as eliminating separate flow fields for reactant and coolant transport<sup>1</sup>. Therefore, to ensure an even workload distribution across the surface of the cell, the flow field design should be optimized to ensure sufficient flow rate through all the channels, proper thermal management, proper contact surface with the gas diffusion layer (GDL) for optimal electrical conductivity and effective transfer of the reactants to the PEMEC outlet<sup>2</sup>. To investigate which designs meet these criteria, the various designs were tested in terms of current density distribution, polarization curves and electrochemical impedance spectroscopy (EIS) to ensure sufficient and equal utilization of all areas within the PEMEC by using S++ hardware and also in-situ neutron imaging. It is well known that higher operating temperatures may lead to improvement in efficiency<sup>3</sup> due to lower ohmic losses. However insufficient water supply through the anode channels may lead to hot spot formation which in turn will degrade certain areas of the membrane. Neutron imaging was also done on two of the designs to validate the measured current density distribution profiles<sup>4</sup>. It was observed that current density tends to be higher at the anode inlet due to less gas bubbles being present, which can also be observed in the temperature mapping as a lower temperature at the inlet of the cell. It was observed that using square shaped pin-type flow fields for the landing areas results in a better and equally distributed current and temperature density distribution profiles as compared with other designs.

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## Local measurement of anode current collector potential in a PEM water electrolyser

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Reduction in the cost of polymer electrolyte membrane water electrolyser (PEMWE) technology is urgently required for the realization of commercially viable green hydrogen production. At present, as much as two thirds of the manufacturing cost of a PEMWE stack is associated with the bipolar plates and current collectors [1]. Selection of appropriate materials for these components is dominated by the requirement for (i) stability in the PEMWE environment and (ii) maintenance of low contact resistance during operation. Current manufacturing practice is typically to use titanium coated with a precious metal such as platinum, which adds significant cost.

A common assumption in the electrolyser community is that the anode current collector needs to withstand the relatively high potentials encountered at the anode electrode. However, it is unlikely that the potential of the anode current collector is equal to that of the anode electrode due to the very low ionic conductivity of the aqueous phase between the two components, which is Type I deionized water.

In this work, an innovative *in situ* reference electrode consisting of a Nafion®/H<sub>2</sub>SO<sub>4</sub> salt bridge connected to a reversible hydrogen electrode is used to measure the local potential of the anode current collector during PEMWE cell operation. Using this technique, we demonstrate for the first time that the potential of the anode current collector is indeed completely decoupled from that of the anode electrode. For example, we show that the potential of a platinum-coated anode current collector remains at ~ 1 V vs RHE (its open circuit potential in oxygenated water) even when the anode potential is increased to ~ 1.8 V vs RHE.

This new observation opens up the possibility to relax the constraints on materials and coatings used for anode bipolar plates and current collectors in PEMWE stacks, with significant implications for reduction in the cost of this technology.

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## Progress in the development of Pt-free cathodes for PEM water electrolysis

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Platinum group metals (PGMs) are commonly used as electrocatalysts in PEM water electrolysis cells. The most popular PGM electrocatalysts found in the electrocatalytic layers of such cells are carbon-supported platinum particles at the cathode for the hydrogen evolution reaction (HER), and unsupported iridium dioxide particles at the anode for the oxygen evolution reaction (OER). The need to use PGMs is dictated by the high acidity of perfluorosulfonated (PFSA) polymer materials, used as cell electrolyte and separator. Despite the possibility to replace platinum by palladium [1,2], to reduce PGM loadings at constant electrochemical activity [3,4], it is generally admitted that the development of large scale (> 10 MW) PEM water electrolyzers and the large scale deployment of the technology will require alternative and less expensive solutions. In addition, the high activity of platinum for the HER is somewhat counter-balanced by a high sensitivity to surface contamination (especially when nano-particles are used) that requires highly deionized water and a continuous monitoring of process-water conductivity during operation.

Less expensive transition group metals and their oxides are used in alkaline water electrolysis technology. The implementation of such compounds in PEM water electrolysis cells would contribute to make the technology less expensive and less sensitive to electrode poisoning. The purpose of this communication (the discussion is restricted to the HER for which it is easier to find viable alternatives) is to report on the interest of using cobalt, iron or nickel clathrochelates functionalized onto appropriate substrates. Instead of using particles, our approach is to implement monolayer-thick molecular mats, chemically bounded to electron-conducting carbonaceous substrates. We started to investigate the interest of using organometallic complexes of transition metals in PEM water electrolysis cells in 2007 [5] and since then, we have improved the selection of complexes and the procedures for surface functionalization. First, the structure and chemistry of these compounds will be presented. Then, their electrochemical properties in solution (which were investigated by cyclic voltammetry in aprotic media and after addition of controlled amounts of protons), will be described. It will be shown that the appropriate selection of peripheral substituents can be used to tune the redox properties of such molecular catalytic sites, and favour the half-cell reaction of interest. Then, different technique used for surface functionalization will be described and compared (the microstructures of such interfaces have been investigated by AFM, SEM, XRD and XPS analysis). Experimental I-V curves have been measured over an extended range of operating temperature and modelled to determine the value of microscopic rate parameters such as exchange current density and roughness factors. Performances will be compared to those obtained with conventional Pt-cathodes. Results obtained after implementation in PEM water electrolysis cells, including performances, specific energy consumption, thermal and pressure stability, durability and sensitivity to poisoning, will also be reported. Limitations of such innovative systems and future perspectives will be discussed.

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## Minimizing the Differential Cell Resistance of PEM Electrolysis Cells – A Hypothesis based on EIS Calculations

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An optimized polymer electrolyte membrane electrolysis cell (PEMEC) design minimizing the differential cell resistance of PEMECs is suggested in this presentation.

A variety of PEMECs with different anode configurations have been examined with EIS at various current densities and temperatures, with cyclic voltammetry (CV) and current density – voltage (iV) curves. Capacitances and charges of the various processes contributing to the total differential cell resistance were determined through fitting of the measured EIS to a simple equivalent circuit consisting of a serial resistance and three (RQ) elements and for some of the EIS additionally an inductor as seen in figure 1. The calculated capacitances and charges were normalized to the geometrical cell area and the electrochemically accessible area determined from CV measurements. These calculations supported the hypothesis previously presented that the total differential resistance of PEMECs originate from the Nafion electrolyte resistance, resistances originating from current constrictions at the electrode/ Nafion electrolyte interface, and from the resistance of the Nafion binder in the anode catalyst layer. [1]

iV curves measured on the various PEMECs were fitted to a model derived from the hypothesis. The modeled iV curves fitted well with the measured iV curves, which may support the presented hypothesis. [2] Finally, based on the hypothesis, an optimized PEMEC design, which should lower the total differential cell resistance of PEMECs, is suggested. [3]

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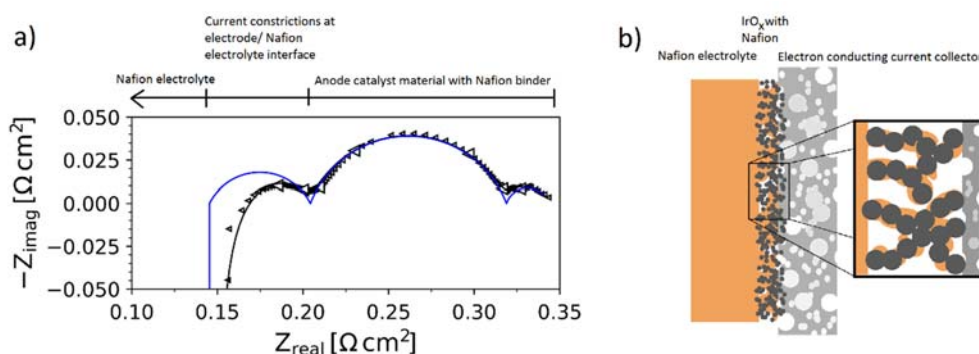


Figure 2 a) EIS spectrum of a PEMEC operated at 63 °C and 0.35 A cm<sup>-2</sup> shown together with the total impedance fit ( $LR_s(R_hQ_h)(R_mQ_m)(R_lQ_l)$  - black line) and shown together with each of the serial elements of the fit (blue lines). b) Schematic drawing of the proposed optimized PEMEC design.

Abstract No 7 (INVITED)

## Surface and Materials Science, and Electrochemical Analysis of Nickel Materials

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Nickel is an important transition metal that finds numerous applications in industrial (stainless steels, non-ferrous alloys, magnets) and consumer (coinage, guitar strings) products, catalysis (Raney nickel), and energy storage (batteries). In relation to electrochemistry and electrocatalysis, nickel is used in rechargeable batteries (NiCd, Ni-M(H)), electrode materials in alkaline water electrolysis, electroplated coatings, just to mention a few. The application of Ni in rechargeable batteries and water electrolysis stems from its remarkable stability and durability in aqueous alkaline solutions, and catalytic properties that are related to electronic characteristics. Nickel is also used as an electrode material in alkaline fuel cells, which were used in the Apollo and Gemini space programs. The application of Ni in electrochemical energy conversion is closely related to its interfacial properties, which define its electrocatalytic behaviour. This contribution reviews our research that focuses on the interfacial behaviour of Ni materials and discusses the following aspects: (i) preparation of metallic Ni surfaces; (ii) reversible formation of  $\alpha$ -Ni(OH)<sub>2</sub>; (iii) irreversible formation of  $\beta$ -Ni(OH)<sub>2</sub>; and (iv) reversible formation of  $\beta$ -NiO(OH) [1-3]. Analysis of the interfacial behaviour and surface chemical composition of Ni surface oxide/hydroxide species is accomplished using cyclic-voltammetry (CV) and X-ray photoelectron spectroscopy (XPS). The contribution describes a novel experimental approach that is applied to determine the electrochemically active surface area ( $A_{\text{ecsa}}$ ) of Ni materials. It explains that in the case of metallic Ni materials the determination of  $A_{\text{ecsa}}$  can be accomplished using CV alone, while in the case of partially oxidized materials CV measurements need to be accompanied by XPS measurements [4]. The presentation also introduces porous Ni materials, such as foams, and discusses their analysis using materials science, surface science, and electrochemical techniques. Scanning electron microscopy (SEM) data reveal that Ni foams consist of interconnected struts; their thickness and separation determine the material's density. Combined focused ion beam (FIB), transmission electron microscopy (TEM), and electron tomography (ET) measurements reveal that the struts are hollow, and their inner wall possesses a nano-structure making the Ni foams dual-porosity materials [4,5]. The contribution also discusses how the surface chemical composition and morphology of Ni materials can be controlled or fine-tuned through mechanical polishing, micro sandblasting, chemical etching, electro-oxidation, or thermal treatment in H<sub>2</sub>(g) atmosphere [4].

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### Acknowledgements.

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## Highly efficient anion exchange membrane water electrolysis and the role of KOH concentration

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Alkaline membrane water electrolyzers (AMWE) promise to use anion exchange membranes (AEM) to combine the benefits of alkaline and PEM electrolyzers to produce hydrogen at high current densities, high efficiency using low cost catalysts. Unlike fuel cells, electrolyzers stay completely humidified and are not exposed to ambient air eliminating some of the drawbacks associated with AEM in fuel cells. In reality dilute KOH (ca. 1 M) must be added to the water loop to ensure good cell performance. We disclose the latest results from the Norwegian HAPEEL project, which is searching for suitable AEMWE catalysts, as well as studying the influence of the KOH concentration on the performance of these promising electrochemical devices.

The study will show that AEM electrolyser using commercially available materials shows excellent performance as long as the KOH concentration is >1M. The EIS characterisation of these electrolyser cells show an increase in HFR at lower KOH concentrations. However, this increase does not account for the entire loss in performance of the AEMWE. Instead, a process with relatively slow time constant (< 10 Hz), attributed to the kinetics of the hydrogen evolution cathode and/or the oxygen evolution anode seems to be limiting the performance of the AEMWE at low KOH concentrations.

As part of the HAPEEL project, HER catalysts are also being developed (NiFe, Ni-NiO-CuO and NiMo). The amorphous NiMo supported on carbon (60wt%) is showing promising performance<sup>1</sup>. Although higher activation overpotentials compared to Pt/C, the catalyst allows for zero noble metal loading AEMWE operation. On the other hand, low KOH concentrations (< 1M) has a more negative effect on the NiMo cathode compared to the PGM catalyst.

In addition, a series of OER catalysts for AEMWE have been screened in AEM operation. Ni and NiFe nanoparticles (~ 5 nm) show excellent performance in AEMWE and comparable to the performance to PGM catalysts. The OER anode also seems less affected by changes in KOH concentration compared to HER cathode. In addition, a few of the Ni-based catalysts even seem to be less affected by changes in KOH concentration compared to the Ir-black anode.

In summary, the AEMWE technologies are showing promising performance even using Non-PGM catalysts for the OER and HER at relatively low KOH concentrations ( $\approx$  1M KOH). On the other hand, the stability of these materials needs further investigation.

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Abstract No 9

## Microstructural optimization of gas diffusion electrodes for high temperature and pressure alkaline electrolysis

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In a world with constantly increasing energy demand and increasing environmental concerns, more focus is placed on renewable energy sources to decrease carbon dioxide emissions and the use of fossil fuels. However, the production of renewable energy is fluctuating by nature, and as such needs to be supported by appropriate energy storage possibilities. In this aspect, hydrogen represents a great candidate, both as an energy vector and as a key component for the catalytic production of hydrocarbons.

Alkaline electrolysis represents a cheap, robust and reliable technology for the production of hydrogen from electrical energy, but is characterized by a low efficiency and production rate. Raising the operating temperature and pressure of the process is known to increase cell efficiency and production rate [1], but commercially available units operate at 60-100°C due to materials' corrosion concerns; especially so for the separator [2]. Allebrod et al. [3–5] have recently demonstrated a novel alkaline cell concept, relying on a porous ceramic separator and metal foam based gas diffusion electrodes, able to operate at temperatures up to 250 °C. A current density of up to 3.75 A·cm<sup>-2</sup> at a cell voltage of 1.75 V at 200 °C and 20 bar has been demonstrated for such cells, bringing them closer to as the performance of proton exchange membrane electrolysis cells [6].

In order to further improve the performance of these cells, Ni-based porous high surface area electrodes were fabricated by screen printing on tape casted and pre-sintered ceramic separators. Electrodes with varying thickness, porosity and particle size were produced, characterized by SEM, and tested in a high temperature and pressure electrochemical test station [7]. A Pd/PdH reference electrode was implemented in order to measure individual electrode overpotentials. The correlation between electrode performance and microstructure will be presented in this talk.

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## Porous Electrodes as Efficient Catalysts for the Oxygen Evolution Reaction

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In view of the CO<sub>2</sub>-free and at the same time cost-competitive production of hydrogen by renewable energy sources alkaline electrolysis (AEL) is considered as a promising technology. Nevertheless, it is mandatory to improve the performance of the electrolyzer. A higher efficiency can be achieved with modern cell designs, improved membrane or separator structures as well as electrode materials. The cell voltage is mainly limited by sluggish reaction kinetics of the oxygen evolution reaction (OER). Therefore, advanced electrodes with large surface area and a high catalytic activity are needed. High surface area catalysts can be produced by laser processing technologies [1], galvanic deposition or by selective chemical leaching of specific phases, e.g. Raney-Ni electrodes [2].

In this contribution, porous electrodes were produced by a space holder method using the short-time sintering technology. This approach allows tailoring of the porosity, the thickness as well as the pore size distribution of the catalytic layer (cf. Fig. 1a). Consequently, the real surface area of the electrode can be enhanced in a defined manner up to a roughness factor of 1,200 determined by double layer charging. Furthermore, nanocrystalline (nc) catalysts are efficient catalyst [3] which can be solid bonded onto a substrate via a short-time sintering technology.

The OER activity of porous, nc Ni-Fe layers was characterized by potential-time curves, steady-state polarization curves, cyclic voltammograms and impedance spectroscopy in 29.9 wt.-% KOH at 333 K in comparison to Ni. Thereby, it turns out that the efficiency of the porous Ni-Fe electrodes is related to the enlarged surface area and the high intrinsic activity resulting in a low Tafel slope of 23 mV dec<sup>-1</sup>. An overpotential of 230 mV at 0.3 A cm<sup>-2</sup> was achieved. Moreover, the stability and activity under operating conditions of intermittent electrolysis (up to 1 A cm<sup>-2</sup>) for 100 h was evaluated and will be discussed (cf. Fig. 1b).

In summary, novel catalytically active and stable layers onto a substrate were produced using a short-time sintering technology. The manufacturing process is fast, scalable and environmentally friendly which should be beneficial for another application of electrolysis.

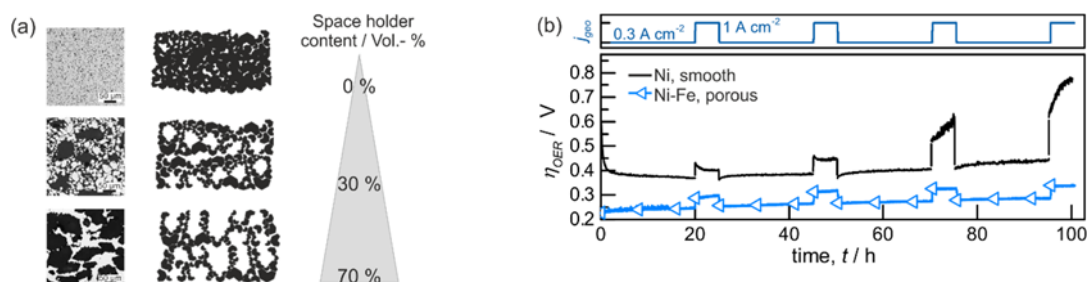


Fig. 1: (a) Different porous catalytic layers. (a) Stability measurements for 100 h in 29.9 wt.-% KOH at 333 K.

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Abstract No 11

**Intensification of alkaline water electrolysis using 3-D electrodes, forced electrolyte flow and pulsed voltage**

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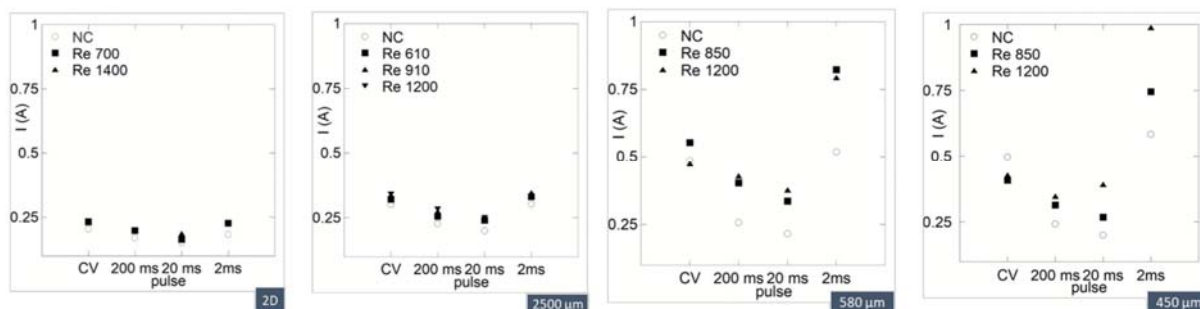
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For hydrogen to be used as an energy vector in the transition to renewable energy, the water electrolysis process must first become more efficient in order to be competitive with the SMR. This current work focuses on intensifying the electrolysis of alkaline water using 3-D electrodes, forced electrolyte flow and pulsed electrical power.

3-D electrodes, having a much larger active surface area than 2-D electrodes for the same projected surface, reduce the overpotential of the electrochemical reaction. But under natural convection, gas bubbles may remain trapped inside the macro-porous 3-D structure, which counterbalances this intrinsic advantage. Implementing a forced electrolyte flow through the electrodes is therefore necessary in order to take full advantage of their benefits. A third way to intensify the process is to use pulsed electrical power.

The synergetic influence of these three effects is illustrated in Figure 1. It shows the measured current for an applied cell voltage of 2 V for both 2-D and 3-D electrodes, the latter with pore sizes of 2500  $\mu\text{m}$ , 580  $\mu\text{m}$  and 450  $\mu\text{m}$ , for different electrolyte flow rates and both conventional and pulsed electrical signals. Comparing the current obtained with the conventional DC current (marked as CV on the x-axis of Figure 1), a current increase is observed when porosity decreases. According to the Butler-Volmer equation, the overpotential of a chemical reaction increases with the density of the current. Since lower porosity means a larger specific electrode surface, i.e. a lower current density, this explains why a 3-D electrode with a pore size of 450  $\mu\text{m}$  is more efficient than a conventional 2-D electrode. Comparing the influence of the electrolyte in Figure 1, it appears to have little influence on a 2-D electrode, but has a strong impact on the results of 3-D electrodes with lower porosity. Smaller pores tend to hinder the evacuation of the hydrogen produced and thus reduce the electrically active surface area. Forced flow is then useful to evacuate gas bubbles out of the structure of the 3-D electrode.

To even further increase the efficiency of the electrolysis cell, we applied a pulsed voltage. The pulses were square pulses from 1.2 V to 3 V and applied in such a way that the polarity of the electrodes remains constant. First of all, we can observe that the impact of pulsed voltage increases as porosity decreases. Secondly, since a significant negative current was measured during the time interval for the 200 ms and 20 ms pulses, there is an overall decrease in the measured current. This is not the case for 2 ms pulses for which an overall current increase is measured. Finally, when analyzing the current obtained with the 2 ms pulse at low porosity (580  $\mu\text{m}$  and 450  $\mu\text{m}$ ), we can see that this increase is the largest for high forced flows, whereas the impact of the flow is hardly visible for the two other electrodes. We can therefore conclude that there is a combined influence of the three effects.



**Fig. 1:** Influence of a pulsed electrical signal on the measured current at 2 V in 1M KOH and 20°C. The same cathodic and anodic flow rate was applied; CV = conventional CycloVoltammetry, i.e. no pulse; NC = Natural Convection.



## Status of Sunfire's Large-Scale High-Temperature Electrolysis

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With its roadmap for moving to a competitive low-carbon economy in 2050, the European Commission sets greenhouse gas emissions targets for different economic sectors. To achieve international energy and climate policy goals, more is needed than an electricity supply based on renewable energies: Using a high-temperature electrolysis (HTE) process, Sunfire offers the opportunity to use green electricity as a renewable feedstock for industry and mobility. Linking the energy sectors thus turns the electricity transition into a more general energy transition.

The iron and steel works of Salzgitter Flachstahl GmbH (Germany) is the location where Sunfire tests the robustness and scalability of its HTE technology. From 2015 to 2019 the GrInHy (Green Industrial Hydrogen) project was dedicated the industrial integration and validation of an HTE with a nominal electrolyser power of 150 kW<sub>AC</sub>. Based on steam, the project showed the achievability of an overall electrical efficiency of more than 80%<sub>LHV</sub>. Furthermore, the system could be operated reversibility in SOFC mode, where electrical efficiencies of more than 50%<sub>LHV</sub> were achieved. Performance data and results of long-term operations of more than 8000 h will be presented.

The successful testing of the first large-scale prototype resulted in the development of an optimized SOEC module generation. Here, the volumetric power density could be increased together with a strong reduction of complexity and costs. The new module was tested under variation of loads in SOFC and SOEC mode and with very fast rSOC cycles. Here, a switching time from 100% load SOFC to 100% load SOEC and back below 10 min was achieved at full system level. This new SOEC module will be the basis for the first 1 MW class HTE container (HyLink HL200) that will be operated from 2020 in Salzgitter in the framework of GrInHy2.0 project. Here, the main focus is on durability as well as the paving of the paths to competitive costs.

One unique feature of the HTE technology is its co-electrolysis capability: the conversion of steam and CO<sub>2</sub> to syngas (H<sub>2</sub> and CO). Only recently, Sunfire achieved a technological breakthrough with the successful start-up and test run of a complete co-electrolysis system with a power input of 10 kW<sub>el,AC</sub>. This technology, called SUNFIRE-SynLink, enables highly efficient production (in future approx. 80 % efficiency on an industrial scale) of synthesis gas in a single step using water, CO<sub>2</sub> and green electricity. This significantly reduces investment and operating costs for Power-to-X projects (e-Crude, e-fuels). In previous power-to-liquids processes, two separate process steps were used to break water vapour down into its components, hydrogen and oxygen (electrolysis), and to turn carbon dioxide into carbon monoxide (reverse water-gas-shift reaction). With Sunfire's co-electrolysis, hydrogen and carbon monoxide can now be recovered in a single process step, significantly improving the efficiency of the overall process and lowering investment (CAPEX) and operating costs (OPEX).

Abstract No 13 (INVITED)

## **Power-to-X activities at Haldor Topsoe: a stepping-stone approach towards commercialization**

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Haldor Topsoe considers high temperature electrolysis (SOEC) as an enabling technology for the electrification of the chemical industry. Compared to other electrolysis technologies, SOEC allows for the conversion of CO<sub>2</sub> and/or H<sub>2</sub>O at high efficiencies for downstream use into chemicals. Haldor Topsoe is commercializing the CO<sub>2</sub> electrolysis technology as eCOs™: a platform for on-site on-demand CO generation from CO<sub>2</sub> feedstock for customers requiring a reliable and safe feed of carbon monoxide at a scale of up to thousands of Nm<sup>3</sup> CO/h. The use of SOEC for the production of H<sub>2</sub> is also interesting, not merely due to the inherently high conversion efficiencies that can be achieved, but also due to system-level synergies found in integrating the endothermal electrolysis process with an exothermal chemical synthesis process. The pilot plant for upgrading CO<sub>2</sub> in biogas into pipeline quality synthetic natural gas (SNG), located at Foulum, Denmark, provides an example of such integration. Here we combine a 16 Nm<sup>3</sup> H<sub>2</sub>/h SOEC module (50 kW) with a methanation unit.

Rapidly dropping renewable energy (RE) prices have created a unique opportunity space for introducing new technology based on electricity and RE over the more conventional fossil-based processes. Electrolytic hydrogen production is a key technology for the transition towards a 100 % sustainable energy system, as it can both facilitate large-scale storage of renewable electricity (RE) and provide the necessary supply of CO<sub>2</sub>-free H<sub>2</sub> for industrial use and for the production of sustainable fuel and chemicals. One of the most essential of the latter in our modern society is ammonia (NH<sub>3</sub>), which is used for fertilizer production and thus vital for feeding the world's population.

Haldor Topsoe has a stepping-stone approach of integrating renewable electricity (RE) into fossil-based NH<sub>3</sub> plants using a hybrid plant design. Compared to a conventional NH<sub>3</sub> plant the hybrid design offers up to 30% reduction of natural gas usage by H<sub>2</sub> from electrolysis using RE, up to 70% reduction of CO<sub>2</sub> in the flue gas, and on-par production cost. In such design, H<sub>2</sub> is produced by combining autothermal reforming (ATR) with electrolysis. The ultimate vision is to eliminate the fossil feedstock completely at a competitive price. One scenario in that vision is to utilize the ability of SOEC stacks to operate in endothermal mode and the inherent property of SOEC cells as oxygen ion membranes. A novel process has been developed where the nitrogen for the ammonia synthesis is provided by burning air between the SOEC stacks and utilizing steam generated in the Haber-Bosch loop as feedstock. This results in very efficient plant (71 % LHV efficiency) and eliminates investment in an air separation unit.

Common for the applications mentioned above is that the SOEC stacks must have high robustness to handle dynamic operation, as well as long lifetimes, to lower the overall cost of stacks and systems. The technical features of Topsoe's SOEC stack will be presented, along with lifetime test results in different electrolysis operations.

## Development of metallic interconnects manufactured by powder metallurgy for solid oxide electrolyser systems

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The need for the replacement of fossil fuels by renewable energy sources has gained importance in recent years for economic and environmental reasons. Hydrogen has arisen as the energy vector of this future scenario, as an alternative fuel to be consumed and stored. In this context electrolyser systems will be key to link renewable energy sources with energy consumption profiles. Among other technologies Solid Oxide Electrolysis Cells (SOEC) offer a solution for a highly efficient hydrogen supply allowing not only the reduction of H<sub>2</sub>O to produce H<sub>2</sub>, but also the transformation of H<sub>2</sub>O and CO<sub>2</sub> into syngas (H<sub>2</sub> and CO) in the so-called coelectrolysis mode. The high operating temperature of SOEC systems, which brings significant thermodynamic advantages, requires the use of ferritic stainless steel (FSS) interconnects [1]. In this work the production of metallic FSS interconnects produced by a powder metallurgy (PM) compaction and sintering process at AMES is presented. The FSS composition, interconnect design, and parameters of the PM process such as compaction pressure, sintering temperature and annealing atmosphere have been systematically optimized to ensure high electrical conductivity, thermal compatibility and good mechanical properties at the working temperature (700-850°C). At these temperatures FSS used alone can give problems due to the premature degradation of the fuel cell stack particularly on the oxygen side. These problems include rapid increase of the contact resistance and volatilization of Cr from the oxide scales, resulting in oxygen electrode poisoning by chromium. Hence protective coatings based on MnCo<sub>2</sub>O<sub>3</sub> spinel must be applied to prevent the diffusion of harmful CrO<sub>3</sub> (g) and CrO<sub>2</sub> (OH)<sub>2</sub> (g) compounds to the SOEC electrodes [2]. These coatings must present a dense structure, good adhesion with the FSS surface, good electrical conductivity and similar thermal expansion coefficients (TEC) to the rest of the components, as well as good chemical stability at the working temperatures under oxidizing atmospheres. In this work, the protective coating of manganese cobalt spinel has been deposited by roll painting and by direct inkjet printing [3] since they are easily reproducible and scalable systems. Both methods have been optimized in terms of composition, rheology of the inks, number and application rate of the coating as well as the annealing conditions.

Microstructural, compositional and chemical studies of the FSS and the Cr barrier layer as well as interconnect conductivity measurements and area specific resistance (ASR) show that interconnects produced by PM coated with manganese cobalt spinel offer great promise for use in SOEC systems



Fig 1: Image of a) pressed, b) sintered, c) machined and d) coated interconnect

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Abstract No 15

## A 25 kW High Temperature Electrolysis Facility for Flexible Hydrogen Production and System Integration Studies

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A 25 kW high-temperature electrolysis (HTE) flexible test facility has been developed at Idaho National Laboratory (INL) for performance evaluation of solid-oxide electrolysis stacks operating independently or in thermal integration with co-located systems. This facility is aimed at advancing the state of the art of HTE technology while demonstrating dynamic grid and thermal energy integration and operational characteristics. The 25 kW HTE flexible test station will provide a test bed for state-of-the-art HTE stack technologies from multiple industry partners. The test station will ultimately be integrated with co-located thermal energy systems including a high-temperature, high-pressure water flow loop and a thermal energy distribution and storage system within the new INL Dynamic Energy Transport and Integration Laboratory (DETAIL) which is currently under development under the DOE NE-EERE Hybrid Energy System research program. The HTE test station will also be designed to communicate with co-located digital real-time simulators for dynamic performance evaluation and hardware-in-the-loop simulations. Establishment of the 25 kW HTE system will be followed by deployment of a test skid with infrastructure support for up to 250 kW HTE turnkey systems.

A detailed description of the 25 kW HTE system will be provided along with results of initial testing performed at the 5 kW scale. SOEC stacks for the initial testing were provided by OxEon Energy based on their ruggedized hermetic stack technology. This stack technology was originally developed for NASA/JPL for the Mars Oxygen In Situ Resource Utilization (MOXIE) experiment, for demonstration of oxygen production by solid oxide electrolysis from Mars atmospheric CO<sub>2</sub> [1]. The stacks are designed to withstand multiple full thermal cycles without loss of performance.



*Figure 3. Overview of INL 25 kW HTE test station.*

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## Power-to-X with high temperature Solid Oxide Cells

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In the context of energy transition, the penetration of renewables to lower CO<sub>2</sub> emissions requires large energy storage capacity to balance the energy system. Ceramic based Solid Oxide Cells, present the unique feature to be capable of operation in Fuel Cell mode (i.e. Power Generation) or Electrolysis operation (i.e. Energy Storage). Not only it allows conversion of steam into hydrogen but also single step conversion of CO<sub>2</sub> / H<sub>2</sub>O mixture into a valuable synthetic gas made of CO and H<sub>2</sub> that can be processed downstream into valuable chemicals. The high operating temperature allows conversion at high efficiency which makes Solid Oxide Cells particularly attractive for sector coupling and the valorization of electricity surplus into valuable molecules and chemicals to be used in the industry or in the transport sector. This efficiency can be further improved if steam can be generated by external heat sources, for instance from Concentrated Solar Plant.

In this contribution, we will report on the electrochemical behavior of Solid Oxide Cells in different configuration ranging from the Steam Electrolysis, to the Co-electrolysis in order to define an operating window and identify potentially critical regimes. Cells were operated in electrolysis and co-electrolysis mode in long term runs of more than 1000 hours of operation with various gas conditions, operating temperature and current density. Electrochemical behavior was investigated by Electrochemical Impedance Spectroscopy while the degradation was monitored by Chrono-potentiometry. Post-test analysis with Scanning Electron Microscope was systematically performed in order to correlate possible microstructural changes with degradation. The main degradation features will be first presented and discussed as well as prospective innovative materials solutions.

Moreover, we will report about the first experimental coupling of a commercial high-temperature steam electrolysis unit and a solar thermal steam generator (Figure 1).

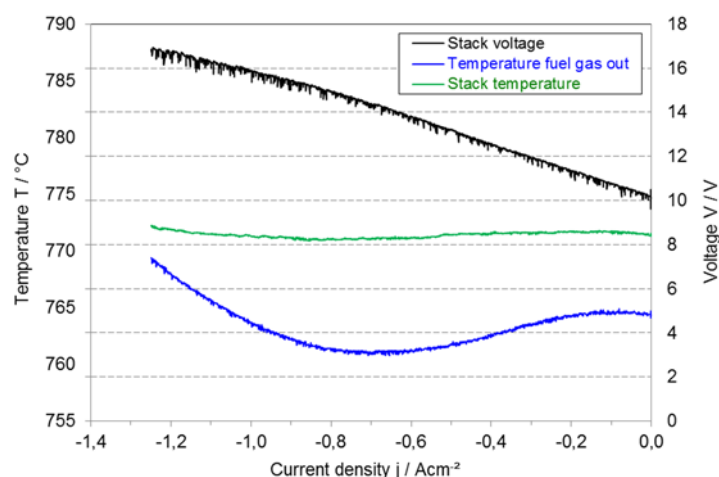


Figure 1: Current-voltage characteristics of the electrolyser stack operated at 770 °C and 12.0 slpm steam flow rate

Abstract No 17

## Enhanced Value of Renewable Energy via High Temperature Electrolysis

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Renewable electric energy has become a significant source of the nation's electricity. However, wind and solar power technologies are intermittent and fluctuating, and have to be balanced for electric grid stability purposes. Relieving the grid of excess renewable electricity can be accomplished by powering a water electrolyzer to produce renewable hydrogen. Solid oxide electrolysis cells (SOEC) offer the highest overall efficiency among other water splitting technologies, because the high operating temperature of SOEC significantly reduces the amount of required electrical energy.

This paper provides an overview of the development of the SOEC materials, cells and stacks at the Pacific Northwest National Laboratory (PNNL). Long-term electrochemical performance and degradation processes in the SOEC electrodes were investigated. Multiple nominally identical button cells with customary SOEC materials – yttria-stabilized zirconia (YSZ) electrolyte, Ni/YSZ negative electrode, and (La,Sr)(Co,Fe)<sub>3</sub> positive electrodes – were tested simultaneously. Electrochemical performance was recorded at 0.9 A/cm<sup>2</sup> near or below thermoneutral voltage for over 1000 hours. Open-circuit voltage was periodically recorded to verify the quality of seals in a high steam environment. For each cell, losses associated with ohmic and electrodic processes were separated using electrochemical impedance spectrometry. Cell performance somewhat decreased during the initial 200 hours because of an increase in both ohmic and polarization resistances. During the following 1000 hours, a rather stable performance (degradation rate < 0.4%/1000 hours) was observed with only a minimal increase in ohmic resistance and no changes in the electrode polarization resistances. Following termination of electrochemical tests, cross-sections of individual cells were analyzed using SEM/EDS, XPS, and ToF-SIMS.

PNNL's 2D and 3D SOFC modeling tools were modified to simulate SOEC operation as functions of operating conditions, cell materials, and stack structures including flow configurations. Button cell model was developed to accurately represent the cell performance and serve as stepping stone to stack model creation. Stack model is being constructed to predict expected thermal and electrical performance of SOEC stack. This presentation will also provide an insight into SOEC stack manufacturing at PNNL and developing new approaches to mitigate factors limiting state-of-art SOEC commercialization.

Abstract No 18 (INVITED)

## 11 years of FCH JU support to electrolyser development and demonstration

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<sup>a</sup>Fuel Cells and Hydrogen Joint Undertaking

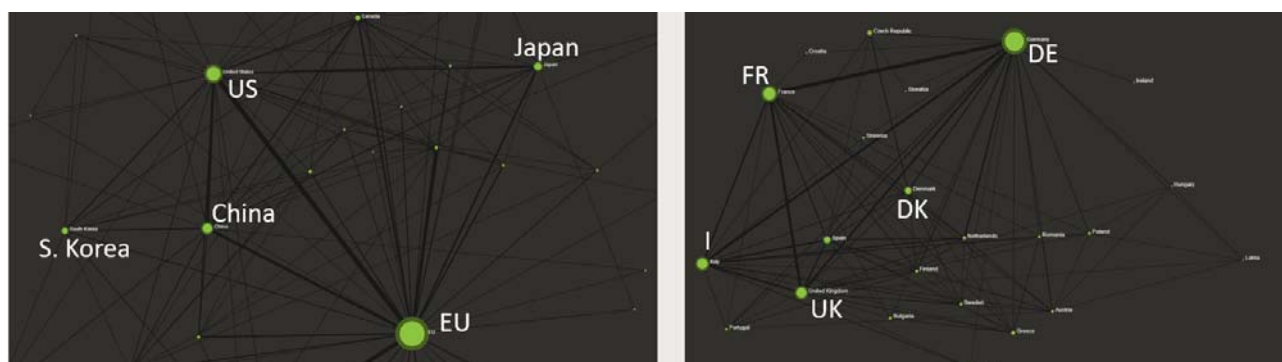
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The Fuel Cells and Hydrogen Joint Undertaking is a Public Private partnership between the European Commission and European Industry and Researchers, its aim being to help hydrogen energy technologies reach market readiness by 2020. Out of the 893MEuro of support provided by the FCH JU to 244 projects since its launch in 2008, 154 MEuro (17%) were provided to 57 projects on Hydrogen Production&Storage topics and out of those 103 MEuro (11%) to 31 projects on electrolysers.

PEM electrolysers have received more than half of FCH JU electrolysis support (57.3 MEuro) with demonstration projects (Hybalance, H2Future and REFHYNE) accounting for 40MEuro and capacities ranging from 1 to 10MW. Solid Oxide electrolysers have received 25MEuro mostly for research projects as this technology is at lower TRL levels but recently project GrInHy2 received 4MEuro to demonstrate a 3MW SOE. Alkaline electrolysers being the most mature technology have received 9MEuro for research and demonstration (4MW in Demo4Grid).

Further Improvements in efficiency, degradation, current density, operating pressure are aimed for in the most recent “game changer” research projects supported by the FCH JU aiming at targets like energy consumption of <50kWh/kg H<sub>2</sub>, current densities of up to 8 A/cm<sup>2</sup> and operating pressures of 100bar, non-precious metal coatings. Also supported is development of innovative types of electrolysers like tubular proton ceramic or anion exchange electrolysers.

This support has led to a vibrant, world leading European electrolyser community of researchers and industry. The schematic below by the TIM software (adapted by the JRC for the FCH JU) shows the total number of articles, publications, conference participations, books, patents and projects for PEM electrolysers from 2008 to 2017. On the left figures for the world (EU 823, US 430, China 270, Japan 193, S. Korea 143) and on the right for the EU member states (Germany 224, France 136, Italy 116, UK 111, Denmark 62).



Besides support to technology development and demonstration, the FCH JU is supporting the sector through studies like the “Commercialisation of Energy Storage in Europe” study that identified a long term (2050) potential of 170GW of electrolysers in Germany and the “Early business cases for Hydrogen in Energy Storage” study that identified a 2.8 GW of electrolysers in the EU by 2025 with a value of 4.2bEuro. Similarly, the JRC has a mandate from the FCH JU to harmonise testing protocols for electrolysers, the effort to be finalised by the end of 2019. Finally, the FCH JU is supporting the development of an EU-wide Guarantees of Origin scheme that has come up with definitions of green and low-carbon hydrogen (70,000 Guarantees of Origin issued already) and is aiming to establish a centralised Certificates scheme.



## PERIC's development on Power to Gas

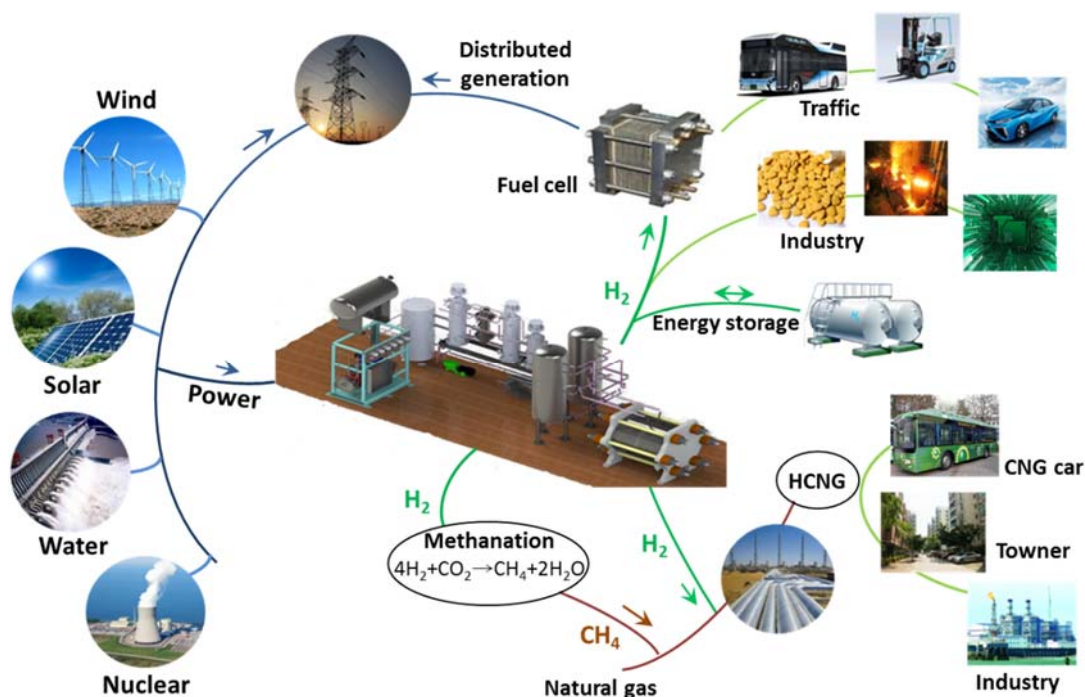
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**Abstract.** As one of the most advanced technologies owned by PERIC, Power to Gas (P2G) technologies and projects have been well developed and researched, including feasibility study, technology consulting, innovative solutions, and construction and operation of P2G projects. Up to now, a number of P2G projects have been completed, such as the hydrogen generation project by Alkaline-type electrolysis with wind power supply in Zhangbei county, Hebei province (Wind-P2G), and hydrogen generation project by alkaline-type electrolysis with wind/solar power supply in Hebei University of Engineering (Wind-solar-P2G). In addition, the ongoing projects includes hydrogen generation and application project by PEM-type electrolysis with wind power supply supported by the Sino-Danish International Science and Technology Cooperation, and hydrogen generation project by alkaline-type electrolysis with wind power supply, supported by the Key Research and Development Plan of Shanxi Province, China.

Hydrogen generation system by water electrolysis (alkaline-type and PEM-type) which have been developed by PERIC, can satisfy hydrogen production from renewable energy with high efficiency and great fluctuation. The system can adapt to such a great fluctuation from 20 – 140% of renewable energy power supply, and more than 80% of energy can be easily converted. The maximum hydrogen production by a single stack (alkaline-type) is 1000Nm<sup>3</sup>/h with purity of hydrogen gas better than 99.9%, which can be directly used in hydrogen fuel cells.





## Effect of power quality on the specific energy consumption of water electrolyzers

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**Summary.** Current harmonics excited by the industrial rectifiers have a significant effect on the specific energy consumption of the water electrolyzers. Simulations and experiments indicate that the specific energy consumption of the electrolysis can be reduced up to 14% by improving the power quality. The effect of current harmonics is emphasized at partial loads.

**Abstract.** The rectifiers of the industrial-scale electrolyzers are typically based on grid frequency commuting thyristors because of the high DC current values. The grid frequency commutation excites high amplitude AC harmonics to the DC current. The relative proportion of harmonics is emphasized at partial loads.

The electrical parameters of a commercial BabyPIEL 2.8 kW (35 A<sub>DC</sub>, 65 V<sub>DC</sub>, 30 cells in series) alkaline water electrolyzer (McPhy) were characterized to be used in simulation of a 1 MW alkaline water electrolyzer (5000 A<sub>DC</sub>, 200 V<sub>DC</sub>, 90 cells in series). In electrical characterization, no phase shift between the current and voltage was seen in the measured waveforms of the alkaline stack with 1 kHz AC frequency. Therefore, simple linear *UI*-characteristics were used to define the stack current based on the supply voltage waveform.

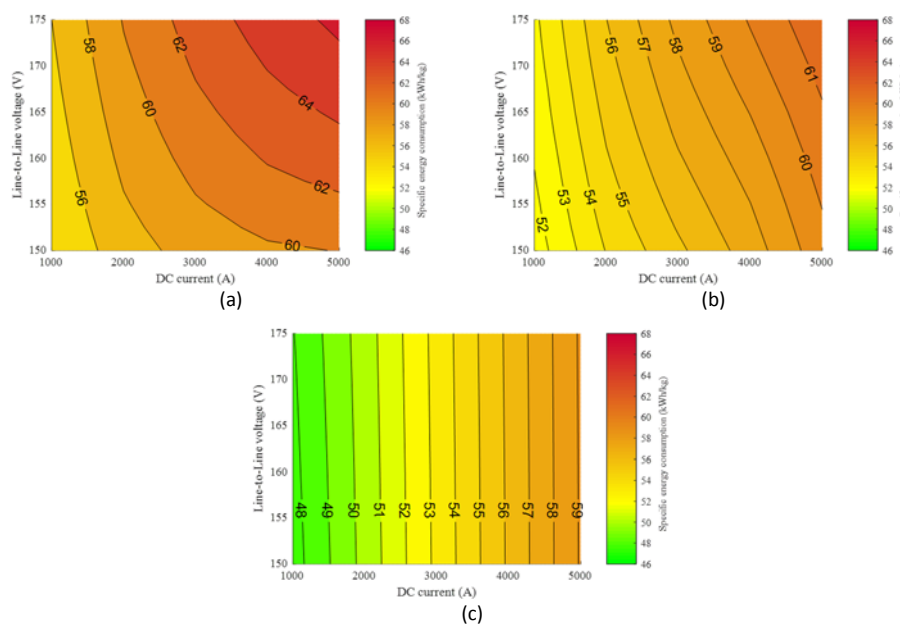


Fig. 4. Simulated specific energy consumption of the 1 MW alkaline water electrolyzer as a function of DC current level and line-to-line AC voltage. (a) 6-pulse thyristor bridge rectifier. (b) 12-pulse thyristor bridge rectifier. (c) Diode bridge followed by a IGBT buck converter [1].

consumption than the 6-pulse thyristor rectifier and up to 9.2% lower electrolyzer stack specific energy consumption than the 12-pulse thyristor rectifier as the current varies between 5000 A and 1000 A. It was also noted that the AC voltage level selection has a significant effect on the power quality and the stack energy consumption in case of thyristor bridge supplies. The simulated change in the stack specific energy consumption of the MW-scale alkaline water electrolyzer outweighs the losses occurring in the rectifiers as the converter losses contribute to specific energy consumption by only up to 1.5 kWh/kg<sub>H2</sub>.

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## **OxEon Energy Developments Targeting Synthetic Liquid Fuels Production Using Non-Fossil CO<sub>2</sub> as a Store of Renewable Energy**

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OxEon Energy was formed in 2017, the key technical staff having direct participation in 30+ years of continuous development effort in SOFC and SOEC. The advances made over three decades by the OxEon team while at Ceramtec culminated in the mid-2017 delivery of a production run of flight qualified CO<sub>2</sub> electrolysis stacks for the Mars2020 mission. When the owners of Ceramtec decided to close the company, the authors as the management team, started OxEon as a new independent company and have setup a SOEC development and pilot production facility. Near the conclusion of the Mars2020 effort, members of the OxEon team worked with Plansee (and US affiliate GTP) to capture lessons learned in the design of a full scale commercial SOEC stack. This new stack design, designated as the Eon 7130 electrolyzer (Figure 1 left), has been put into prototype production by OxEon and GTP. Initial stack builds have all demonstrated the hermetic seals developed at the smaller scale for the Mars project.

Initial Eon 7130 electrolyzer short stacks have been tested in both steam electrolysis and CO<sub>2</sub> co-electrolysis mode and subjected to multiple thermal cycles and accumulated multi-thousand hours of operation. The first 5kW Eon 7130 electrolyzer stack is being produced for testing at the Idaho National Laboratory in Q2 of 2019.

In addition, the OxEon team has setup its small modular synthetic fuel (Fischer Tropsch and related variants) reactor to validate reactor design and catalyst performance (Figure 1 right). The target application for this reactor design is production of non-fossil synthetic fuels from bio-derived CO<sub>2</sub> (by-products of fermentation and anaerobic digester gas). The energy content in this fuel would be 100% renewable and the embodied carbon fossil-free. Since bio-carbon sources are smaller and distributed, reactor designs that scale down, are modular and transportable are required. There are already CO<sub>2</sub> priced markets that would make such fuels competitive with petroleum, given a commercial-scale implementation of the technology.

There is synergy with this vision of renewable synfuels, and the needs for electrically driven fuel and oxygen production to support long endurance space flight. OxEon is applying these same technologies, CO<sub>2</sub>-steam co-electrolysis and methanation to the challenge of In Situ-Resource Utilization (ISRU) and spacecraft habitat atmosphere revitalization. An overview of OxEon's activities in these areas with early results will be presented.



*Figure 5. Eon 7130 Electrolysis stack (left) and OxEon Synfuel Pre-Pilot Plant (right)*

## Hydrogen from Molten Carbonate electrolysis for green steel production.

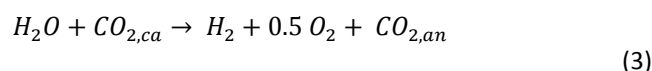
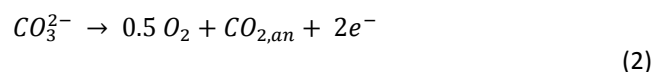
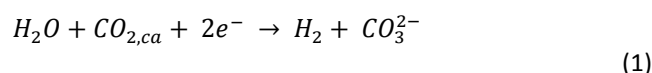
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The Hydrogen Breakthrough Ironmaking Technology (HYBRIT) concept, a joint venture between LKAB, SSAB and Vattenfall, is focused on developing a process that use hydrogen as reducing agent instead of coal within the steel industry. Replacing coal with hydrogen would produce water vapour instead of CO<sub>2</sub> as by-product. Apart from developing an effective process for hydrogen based steel production a challenge is producing fossil-free energy and cost efficient hydrogen. A full scale implementation of the HYBRIT concept in Sweden would require several hundreds of MWs of electrolyzers and would offer opportunities of utilizing the strengths of various electrolyzer technologies for an optimized mix.

High temperature electrolyzer technology could potentially offer interesting opportunities for future HYBRIT plants. Both from an energy efficiency point of view through integration with the rest of the steel process, but also as potential source for supplying the carbon required to achieve the final steel product quality through means of co-electrolysis.

Solid oxide electrolysis is one such HTE option and is considered to be in demonstration phase and an electrical consumption of 42 kWh/kg H<sub>2</sub> has been reported [1 –2]. Recent work has also shown that molten carbonate fuel cells can also be operated in electrolysis mode (MCEC) [3 – 5]. In molten carbonate electrolysis, water vapour is electrolysed over a nickel cathode in the presence of CO<sub>2</sub> to produce carbonate ions which are transferred to a nickel oxide cathode to produce oxygen and CO<sub>2</sub> according to equation 1 – 3.



Apart from producing hydrogen, MCEC also transports CO<sub>2</sub> from the cathode to the anode, which serves as a purification step. This talk will discuss the application of MCEC for high temperature hydrogen production and its integration into the HYBRIT concept. An Aspen model is proposed to determine both the electrolyser and system energy requirements to further our understanding of how an MCEC compares to SOEC technology. The model shows that the MCEC has the capability to produce low energy intensive hydrogen and could be considered for both a hydrogen and CO source.

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Abstract No 23

## **Towards an atomistic understanding of electrocatalytic partial hydrocarbon oxidation: theory and experiments synergies**

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Electrochemistry has great potential for replacing outdated energy-intensive industrial chemical processes, for it is inherently safer and allow fine selectivity control in optimized systems. Electrochemical processes are also versatile, thus suited for decentralization and embedding in national energy grids, to help compensate the intermittance of renewable sources energy output. A great opportunity lies in fuel cell systems, incorporating oxygen from water into valuable chemicals easier to store, transport and use for further industrial transformations. Our recent work provides critical fundamental insights into the mechanisms governing hydrocarbon electro-oxidation, through the study of model, yet industrially relevant, reactions. Our work is critical because to understand the factors controlling electrocatalytic systems at the atomic level is the first step towards efficient large scale electrochemical processes. During my oral presentation, I will outline: 1) the benefits of implementing an iterative, multi-angle approach consisting of computational modelling, experimental observations and advanced characterization techniques. We aim this approach to become the benchmark in conducting high quality research; 2) novel fundamental insights for hydrocarbon partial oxidations, starting from our ready to publish study in Energy and Environmental Science on propene electro-oxidation. I will introduce the audience to important dynamics occurring at the catalyst surface, where organic bound species formed in-situ at low surface coverage steer the reaction selectivity towards allylic oxidation at high coverage. Starting from this model, I will discuss findings relevant for the production of other oxygenates too; 3) possible catalyst design strategies for hydrocarbon partial electro-oxidation reactions, to tune selectivity and activity by achieving greater surface composition control through different techniques.

Abstract No 24 (INVITED)

## **Performance and long-term stability of electrolyte supported Solid Oxide Electrolyser Cells**

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In recent years, steam electrolysis with solid oxide cells (SOC) has evolved from the lab to a technology with prototypes in the > 100 kW range. The main application driver for this fast development is the growing need for decarbonized hydrogen production based on renewable electricity, the SOC has the advantages of higher electrical energy-conversion efficiency, in particular if heat for steam generation is available from surrounding processes. EIFER performs long-term electrolysis cell and stack testing in laboratories. In addition, operation of a 7.5 kW system is performed at the hydrogen refuelling station of Karlsruhe with the support of the German federal state of Baden Württemberg.

In several tests, operation times of 10 000 hours to more than 23 000 hours were achieved with electrolyte-supported cells. This provides reliable lifetime extrapolations for industrial systems for which lifetimes over 50 000 are foreseen. The cells used show a high degree of SOFC/SOEC reversibility with low degradation ( $U_{\text{cell}}$  degradation < 0.5 %/1000 h). Other issues like the capability of operation with power variation and either an increase in the current density to > 1 Acm<sup>-2</sup>, or a decrease of the operation temperature are treated. In all tests, impedance spectroscopy is used as in-situ tool for performance and degradation analysis and for longest experiments ex-situ post-test analysis were performed at the nanoscale. An update of the mentioned testing activities will be given.

Abstract No 25

## **Advancement of reversible proton-conducting solid oxide cells at Idaho National Laboratory (INL)**

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Reversible protonic ceramic electrochemical cell (PCEC) is an emerging and attractive technology which converts energy between power and hydrogen using solid oxide proton conductors at intermediate temperatures [1, 2]. Economically competitive PCEC systems have distinct advantages over conventional oxygen-ion conducting ceramic electrochemical counterpart, but further technology development and widespread market acceptance will require continuous innovation of materials and structures in order to improve cell performance, enhance system lifetime and reduce cost. In this work, we report the advancement of reversible proton-conducting solid oxide cells with novel triple conducting electrode materials [3,4] and 3D self-architected electrode structure [5] below 600°C in INL.

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## On the development of electrodes for tubular proton ceramic electrolyzers for pressurized hydrogen production

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High temperature electrolysis (HTE) of steam offers high efficiency of conversion of renewable and peak electricity to H<sub>2</sub> and may increase efficiency further by utilising available sources of heat and steam from solar, geothermal, or nuclear power plants. They operate therefore ideally best at temperatures above those of the supplied steam (e.g. 200-600 °C). Technologies developed to date comprise solid oxide electrolyser cells (SOECs) utilising oxide ion conducting electrolytes operating by virtue of necessity around 800 °C. They produce hydrogen on the steam feed side. Hence, separation and drying of H<sub>2</sub> cost energy and add plant complexity and footprint. In comparison, a high temperature proton conducting electrolyte will instead pump protons (H<sup>+</sup>) and form dry H<sub>2</sub>, leaving O<sub>2</sub> on the steam side. Such proton ceramic electrolyser cells (PCECs) thus require less separation process stages and can produce pressurised dry H<sub>2</sub> directly. Protons exhibit lower activation energies than oxide ions, and ceramic proton conductors will be able to operate at lower temperatures – 500-700 °C – i.e., closer to the ideal range for integration with solar and geothermal plants. Incentives for developing high temperature electrolyzers (HTEs) using proton conducting electrolytes stem from the fact that a proton ceramic electrolysis cell (PCE) pumps out and pressurises dry H<sub>2</sub> directly. Existing HTEs design utilises the high packing density of planar stacks, but the hot seal and vulnerability to single cell breakdown give high stack rejection rate and questionable durability. In the GAMER FCH JU project started in January 2018, we develop a novel cost-effective tubular Proton Ceramic Electrolyser (PCE) stack technology integrated in a steam electrolyser system to produce pure dry pressurized hydrogen. The cells consist of a porous Ni-BZCY negatrod for the H<sub>2</sub> side, a thin dense BZCY-based electrolyte, a porous positrod for the H<sub>2</sub>O+O<sub>2</sub> side, and a current collector system. Extensive work is carried out to develop stable, efficient and well adhered positrodes using the double perovskite Ba<sub>1-x</sub>Gd<sub>0.8</sub>La<sub>0.2+x</sub>Co<sub>2</sub>O<sub>6-δ</sub> (x = 0-0.5). The stability of the functional materials of the cell was firstly validated after testing at 600°C at 28 barg (75% steam/25%O<sub>2</sub>) for 100 hours. Various electrode architectures have been prepared, including single phase, composites and infiltrated backbones, using either printing or sol-gel impregnation methods. The microstructure of the resulting tubular cells was thoroughly characterized by various microscopic techniques. The electrochemical performance of the produced tubular cells was evaluated in steam electrolysis mode. We report on the results of this work, highlighting how the electrode architectures and preparation methods strongly impact on the stability and faradaic efficiency of the cells.

### Acknowledgment

This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking under grant agreement (number 779486). This Joint Undertaking receives support from the European Union's Horizon 2020 research and innovation programme, Hydrogen Europe and Hydrogen Europe research.



Abstract No 27

## Scale up and integration of proton-conducting ceramics into multi-cell stacks

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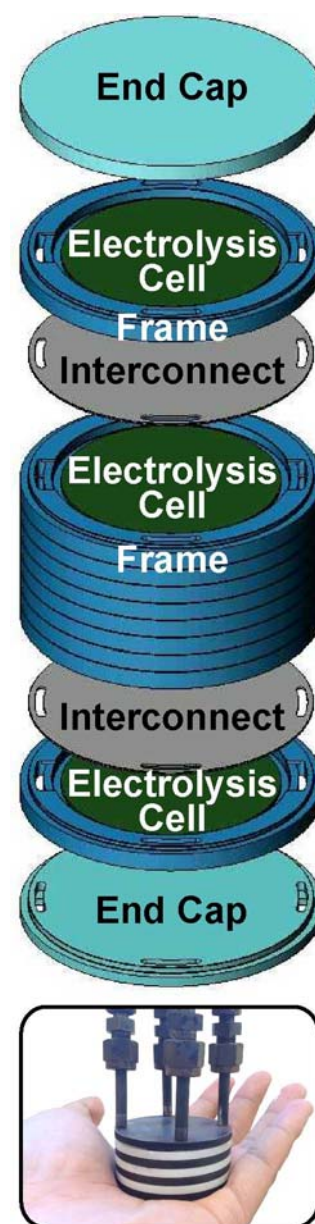
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We present our current efforts to scale up proton-conducting ceramic electrochemical devices for integration into multi-cell stacks. Proton-conducting ceramics are an exciting new class of materials that are now emerging from research laboratories to address societal challenges in electricity generation, energy storage, and fuels synthesis. The high ionic conductivity of these materials in comparison to their oxygen-ion-conducting counterparts enables efficient electrochemical performance at  $\sim 500$  °C. Such operating temperatures may be up to 300 °C lower than state-of-the-art electrolyzers based on yttria-stabilized zirconia. This enables use of lower-quality waste heat to provide thermal energy in driving electrolyzer processes, simplifying integration of protonic-ceramic electrolyzers into existing technologies. Additionally, lower-temperature operation should enable longer device life through reduced degradation rates, and potentially lower stack and system costs by reducing materials demands. That said, scale up of protonic-ceramic electrochemical cells (PCECs) is in its infancy, with no stack demonstrations reported to date in the archival literature. In this presentation, we will review our efforts at the Colorado School of Mines to increase the size of PCECs beyond the button-cell level, and to integrate these cells into multi-cell stack assemblies.

An illustration of our stack design is shown in Figure 1, and includes a photograph of a fully assembled three-cell stack after performance testing. The design is centered on three repeating components: the protonic-ceramic electrolysis cells, the composite-ceramic frames in which the cells are bonded, and the thin metallic interconnect / bipolar plates that conduct electricity between adjacent cells. The cells feature a dense, 20 $\mu$ m-thick  $\text{BaCe}_{0.2}\text{Zr}_{0.6}\text{Y}_{0.2}\text{O}_{3-\delta}$  (BCZY26) electrolyte on a porous Ni-BCZY26 anode fabricated through reactive-sintering methods. The cathode is a novel  $\text{BaCo}_{0.4}\text{Fe}_{0.4}\text{Zr}_{0.1}\text{Y}_{0.1}\text{O}_{3-\delta}$  (BCFZY) atop of the BCZY26 electrolyte with an active area of  $\sim 4\text{cm}^2$ . Reactive gases are fed through fuel and oxidizer ports machined into the frame. The metallic interconnect is a ferritic steel developed for intermediate-temperature solid-oxide fuel cells and electrolyzers, and is compression sealed to the frame. Metallic meshes (not shown) electrically connect the electrodes to the interconnects.

While the low operating temperature of PCECs should present benefits for the long-term stack stability, many questions remain regarding the compatibility of the cell and stack materials. High protonic-ceramic stack performance has been previously demonstrated at the Colorado School of Mines, with fuel-cell power densities reaching  $258\text{ mW cm}^{-2}$  under direct methane-steam fuel at 550 °C. In an effort to better understand degradation in fuel-cell and electrolysis stacks, we have integrated numerous voltage taps throughout the stack shown. Such diagnostics help us to identify the sources and root causes behind performance degradation. In this presentation, we will review our results for longer-term protonic-ceramic stack operation, discuss mechanisms behind stack-performance degradation, and present strategies for reducing degradation rates and extending stack life.



**Figure 1:** Stack schematic with photograph.



## An Evaluation of High Temperature Water Splitting Systems using Protonic Ceramic Electrolyzers

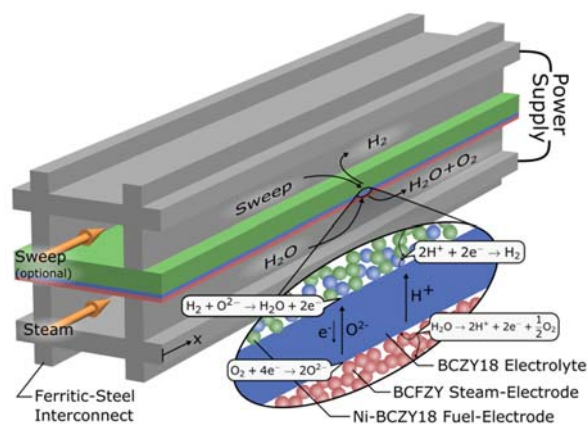
Amogh Thatte and Robert J. Braun

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High temperature water splitting (HTWS) via electrochemical processes are of growing interest due to both their potential for achieving high thermodynamic efficiency and their ability to produce high quality hydrogen with minimal product gas processing. In particular, protonic ceramic electrolysis cells (PCECs) operating between 500°-600°C have the attractive feature that only pure, dry hydrogen is produced at the hydrogen electrode (see Figure 1) and, unlike more conventional solid oxide electrochemical cells (SOECs) operating at 800°C, no further gas separation is needed. These features allow much simpler and elegant hydrogen production system concepts that have the potential to be significantly less costly and more efficient. For example, the dry H<sub>2</sub> gas production at the fuel electrode allows for a much simpler balance-of-plant. The lower operating temperature also has numerous benefits, including lower plant heat losses, the ability to find more options for integrating various process heat sources by virtue of the lower grade heat requirements, and reduced capital cost due to a reduction in both gas process heat exchanger temperature and surface area requirements. A simple thermodynamic evaluation indicates that balance-of-plant steam generation specific energy (kJ/kg) requirements are some 17% lower when operating at 500°C versus 800°C.

The present work focuses on scale-up of BZY-based PCECs, developed by collaborating faculty at the Colorado School of Mines, and their design/integration into kW/MW-scale hydrogen production systems. Realizing high efficiency HTWS systems based on novel BZY-based protonic ceramics requires understanding of numerous system-level considerations. One of our efforts is largely concerned with developing viable system designs that enable >75% system efficiency at centralized hydrogen production costs of < \$2/kg (without compression, dispensing, and storage). This requires evaluation of plant operating conditions, especially in the PCEC stack periphery, where operating temperature, pressure, reactant utilization, sweep gas (if any) on the fuel electrode, thermal management, gas compression, and balance-of-plant integration all play critical roles in establishing cost effective, high performance HTWS systems.

This presentation will provide the broader PCEC community of systems-level considerations that can inform materials and cell development efforts, while offering an early glimpse into the potential techno-economic-environmental benefits via life cycle costing and life cycle assessment (LCA) of the technology. LCA is used as the tool to evaluate cumulative environmental impacts resulting from all stages in the technology life cycle. Preliminary techno-economic analysis of system costs and results from the initial LCA studies are presented.



**Figure 1.** Illustration of planar, co-flow configured protonic ceramic electrolyzer with ionization reactions.

Abstract No 29 (INVITED)

## Catalyst Development for PEM and AEM Water Electrolyzer Anodes

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In this talk, I will highlight some of our recent work<sup>1-10</sup> on the design and the molecular understanding of PGM and PGM-free metal oxide catalysts and of their liquid-solid interfaces for the evolution of molecular oxygen from water. I will outline the preparation, characterization, and catalytic performance of oxide model catalysts and discuss mechanistic insights derived from structure-activity relationships.

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## Operando X-ray absorption investigations into the role of Fe in the electrochemical stability and oxygen evolution activity of $\text{Ni}_{1-x}\text{Fe}_x\text{O}_y$ nanoparticles

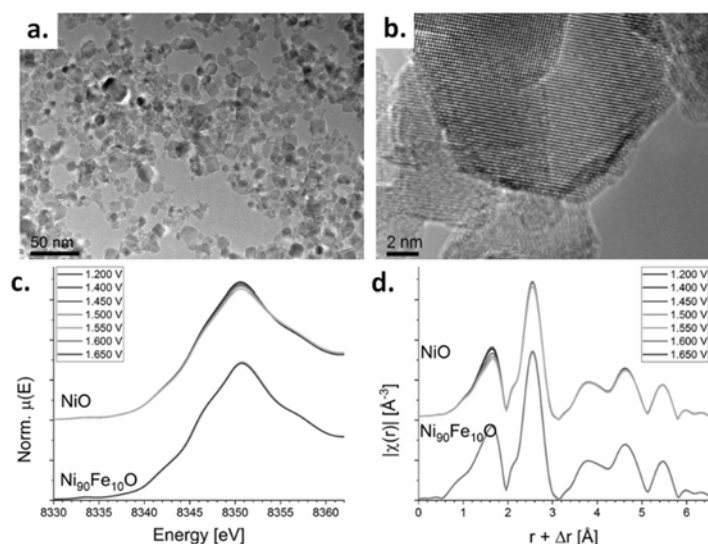
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Nickel oxide currently represents one of the most attractive anode catalysts for alkaline water electrolyzers (AWEs) due to its high activity and stability for the oxygen evolution reaction (OER) in alkaline media. More recently, however, the effect of Fe doping on the OER has been the subject of many intensive investigations since the incorporation of small amounts of Fe into NiO can greatly enhance its OER activity.[1] Here we demonstrate a practical flame-spray pyrolysis synthesis capable of producing highly crystalline Ni-Fe oxide nano-powders ( $\text{Ni}_{1-x}\text{Fe}_x\text{O}_y$ ) with high surface areas ( $\text{SA}_{\text{BET}} \approx 20 - 75 \text{ m}^2/\text{g}$ ). By using different Ni-to-Fe ratios, we aim to investigate how Fe incorporation influences the surface electronic properties and local coordination structures of Ni catalysts and how this impacts the electrochemical stability and OER activity via *operando* X-ray absorption spectroscopy investigations.[2] The resulting *operando* XANES and EXAFS analyses of the Ni and Fe K-edges permit useful insight into the nature of the valence states and rearrangements in local structure that occur under real operating conditions. Combined with a broad range of *ex-situ* physical characterization techniques, including high-resolution transmission electron microscopy (HRTEM), annular dark field (ADF) imaging, and energy dispersive X-ray (EDX) mapping, we then relate the structural, electronic, and morphological changes to the observed electrochemical OER activity. Ultimately, we find that the incorporation of Fe leads to an overall stabilization of the initially compact and crystalline rock salt structure of  $\text{Ni}_{1-x}\text{Fe}_x\text{O}_y$  and thereby inhibits the transformation to more layered and disordered polymorphs.[2]



**Figure 1.** TEM (a) and high resolution (HR) TEM (b) micro-graphs of  $\text{Ni}_{0.90}\text{Fe}_{0.10}\text{O}_y$  catalysts. Normalized XANES spectra (c) and Fourier transformed Ni EXAFS spectra (d) recorded in *operando* for  $\text{Ni}_{1-x}\text{Fe}_x\text{O}_y$  over a range of applied potentials. All Ni  $\chi(k)$  functions were Fourier transformed over a  $k$ -range of 2.6–12.5 Å.

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## Oxygen evolution at porous Ni electrodes

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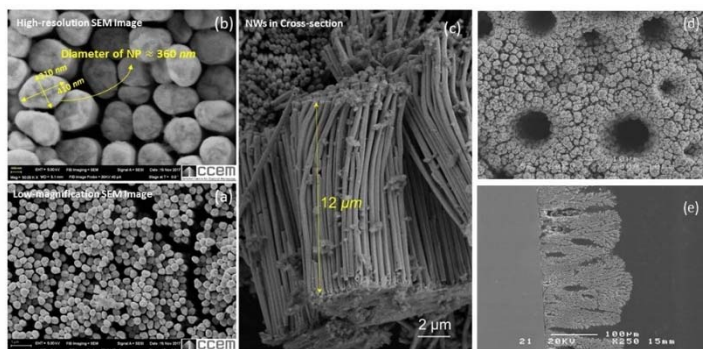
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The promise of hydrogen as a sustainable, secure, and clean alternative energy carrier to satisfy the growing global energy demand requires efficient, low-cost, and environmentally friendly production. Water electrolysis to generate hydrogen fuel - preferably driven by renewable energy - is a highly attractive means to produce hydrogen, but advanced electrolyzer systems are required for electrolytic H<sub>2</sub> production to become a commercially viable technology. Lately, new classes of have emerged with improved performance over their predecessors, improving the future prospect of commercialized (A)PEMWEs. The prospect of new anion exchange membranes for anionic polymer electrolyte membrane water electrolyzers that could match the performances of cationic exchange membranes in terms of ionic conductivity and longevity has spurred a renewed interest in research to improve catalyst performance for the oxygen evolution reaction (OER) in alkaline electrolyte.

In this presentation we will focus on the electrochemical characterization of arrays of micrometer long Ni nanowires (NWs) (Fig. 1a, 1b and 1c) and mesoporous Ni electrodes synthesized by the dynamic hydrogen bubble templating (DHBT) method (Fig. 1d and 1e). Detailed structural and electrochemical analyses were conducted including HR-TEM and EELS analysis providing information on the chemical state of atoms from quantitative analysis of the signals and electrochemical impedance spectroscopy providing information on the charge transfer resistance and capacitance in the potential region where the oxygen evolution reaction (OER) is occurring. The effect of Fe on the electrocatalytic activity for the OER will also be investigated. By fine-tuning the Fe content of the electrolyte, the OER overpotential under galvanostatic conditions (250 mA cm<sup>-2</sup>) was reduced to  $\eta_{250} = 260$  mV and remained stable over the polarization duration.

Figure 1 High-resolution SEM images of Ni Nanowires (a, b and c) and mesoporous Ni (d and e) revealing their cross-sectional (c and e) and top views (a, b and d).



Acknowledgement: This research was conducted as part of the Engineered Nickel Catalysts for Electrochemical Clean Energy project administered from Queen's University and supported by Grant No. RGPNM 477963-2015 under the Natural Sciences and Engineering Research Council of Canada (NSERC) Discovery Frontiers Program.

## High performing and economic platinum group metal free anode catalysts for AEM and PEM electrolyzers – Opportunities and Challenges

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Global warming associated with anthropogenic emissions of CO<sub>2</sub> caused by the use of fossil fuels as energy carriers has become a severe problem for the modern society. To mitigate this issue, alternative sources of energy carrier have to be evaluated and implemented into our everyday life. One promising and ubiquitous energy carrier is hydrogen, which can be generated by splitting water via water electrolysis. This technology is not only an excellent way to create hydrogen but can also be modified to create other fuels or valuable chemicals such as ammonia and methanol from nitrogen and carbon dioxide, respectively. Yet although electrolysis has a great potential to be the technology for a greener future the challenges to be overcome certainly dampen the optimism and prevent its breakthrough as the leading technology for a more sustainable and green energy economy. One of the major drawbacks of the electrolysis technology is the high energy consumption to split the water at the anode side for the oxygen evolution reaction (OER), for which platinum group metals (PGM) are employed to mitigate this circumstance [2]. The use of PGMs as OER enhancing catalysts in electrolysis however, especially in the polymer exchange membrane (PEM) electrolysis is, if not the major but one of the flaws of the technology. This can be mitigated by employing a slightly other approach in the upcoming sibling technology, the anion exchange membrane (AEM) electrolysis. Yet, the stability and performance [3] of the catalysts to drive the OER in the AEM electrolysis are as much as a drawback as using PGMs in the PEM electrolysis. The present work is devoted to developing PGM free and high performing OER catalysts for both PEM and AEM electrolysis.

For the AEM electrolysis, a new OER catalyst NiFeOOH was prepared through electrochemical dissolution-precipitation method and modified by phosphorus intrusion to synthesize NiFeOOH-P. These materials and a group of layered double hydroxide NiFe, NiCo, and NiMn, were evaluated via various electrochemical characterization techniques in a lab scale AEM electrolyser under realistic conditions to evaluate their performances.

For PEM electrolysis the PGM free catalyst AgCeCrO<sub>3</sub> was prepared via a co-precipitation method and subsequent calcination, which was evaluated as well in a lab scale PEM electrolyser under real world conditions.

In this talk we will discuss electrochemical characterisation results of all materials and their performances as OER catalyst and discuss the difference between realistic testing conditions and techniques which are less suited for evaluating the performance of catalysts in real electrolysis systems.

In addition various physical analysis methods such as SEM, EDX, BET, XPS, TEM, and ICP-MS were employed to deepen our understanding of the reaction and aging mechanism of the OER catalysts, which will be also discussed in this talk.

### Acknowledgement.

This project has received funding from the European Research Council (ERC) under the European Union's Horizon 2020 research and innovation programme (grant agreement n° 761093).

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Abstract No 33

## **Electrolysis market opportunities**

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Abstract No 34 (INVITED)

## Low Temperature Water Electrolysis at Large Scale: A Comparison of Technology Benefits and Challenges

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The two primary commercial pathways today for water electrolysis were originally developed for very different applications. Liquid alkaline systems have a long history in large scale installations, where electricity cost was a primary driver. Systems were therefore designed to optimize efficiency, operating at low current densities. Proton exchange membrane-based electrolyzers, in contrast, were developed later, and were introduced commercially in small scale, distributed markets, where the cost of electricity was not a concern to the customer. Systems were therefore developed to operate at the lowest capital cost, driving the operating current as high as possible to enable fewer and smaller stacks. Based on this legacy, the two technologies require different cost reduction approaches to serve the emerging energy markets today. This talk will discuss the opportunities for each technology to reduce total cost of ownership (operating and capital costs), and the technical challenges that need to be solved.

Alkaline technology can benefit from pressurized operation to enable higher current densities, and improvements in catalyst and electrode structure to maintain high operating efficiency. New research in hydrogen and oxygen evolution catalysts has shown the potential for lower cell voltages at higher current densities, and improvements in chlor alkali electrode technology can be applied to alkaline electrolysis. Most likely, the operating point will move towards higher current density while maintaining efficiency. In addition, there are still incremental improvements possible for large scale systems, despite the long history of alkaline electrolyzers. For PEM technology, designing membrane materials specific to the electrolyzer application (vs. adapting PEM fuel cell materials) should enable much thinner materials, driving up efficiency. Component manufacturing is also still highly manual and limits the degree of catalyst loading reductions. Leveraging the manufacturing development from fuel cells will allow thinner, more uniform catalyst layers as well as reducing labor significantly. The PEM systems are therefore likely to remain at high operating current density, while increasing efficiency and reducing operating cost. These shifts are shown conceptually in Figure 1.

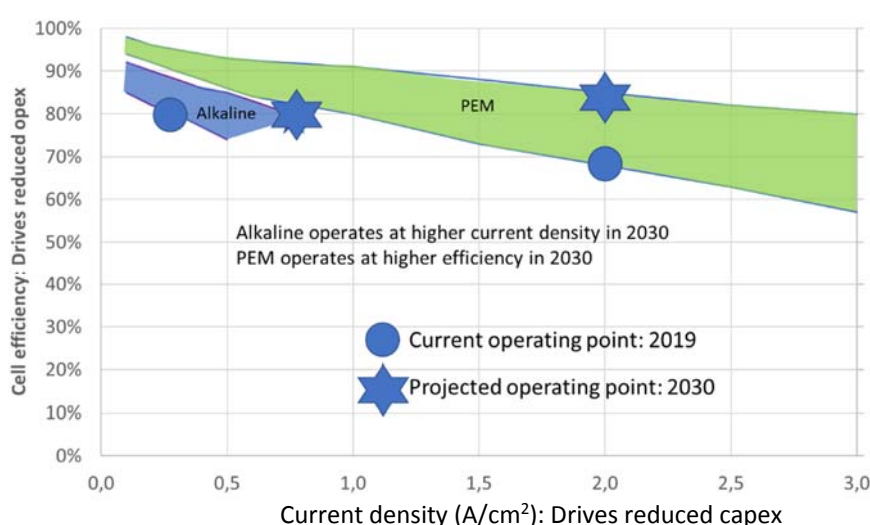


Figure 1: Operating windows for alkaline and PEM electrolysis (efficiency based on higher heating value)



Abstract No 35

## **REFHYNE – 10 MW PEM electrolyser for refinery**

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The overall objective of the REFHYNE project (Clean Refinery Hydrogen for Europe) is to deploy and operate a 10MW electrolyser in a Power to Refinery setting [1]. In doing this, REFHYNE will validate the business model for using electrolytic hydrogen as an input to refineries, prove the revenues available from primary and secondary grid balancing in today's markets and create an evidence base for the policy/regulatory changes needed to underpin this market.

The REFHYNE project is funded by the FCHJU/H2020 programme and will install and operate a 10MW electrolyser from ITM Power at a large refinery in Rhineland, Germany, which is operated by Shell Deutschland Oils. The electrolyser will provide bulk quantities of hydrogen to the refinery's hydrogen pipeline system (currently supplied by two steam methane reformers). The electrolyser will be operated in a highly responsive mode, helping to balance the refinery's internal electricity grid and also selling Primary Control Reserve service to the German Transmission System Operators. The combination of hydrogen sales to the refinery and balancing payments create a business case which justifies this installation. This business case will be evaluated in detail, in a 2 year campaign of techno-economic and environmental analysis. The REFHYNE business model is replicable in markets with a similar regulatory structure to Germany. However, to expand this market to a GW scale, new business models will be needed. These will include valuing green hydrogen as an input to industrial processes (to meet carbon policy targets) and also on sales to H2 mobility markets. The REFHYNE project will gather real world data on these models and will use this to simulate the bulk electrolyser model in a range of market conditions. This will be used to produce reports on the conditions under which the electrolyser business models become viable, in order to provide the evidence base required to justify changes in existing policies. A campaign of targeted dissemination will ensure the results of these studies reach decision makers in large industrial sites, financiers, utilities and policy makers. The REFHYNE electrolyser will be the largest in the world and has been designed as the building block for future electrolysers up to 100MW and beyond. REFHYNE includes a design study into the options for a 100MW electrolyser at the Rhineland refinery, which will help prepare the market for deployments at this scale.

This presentation will give an overview of the project and an update on the progress and plans for the demonstration.

[1] [www.refhyne.eu](http://www.refhyne.eu)



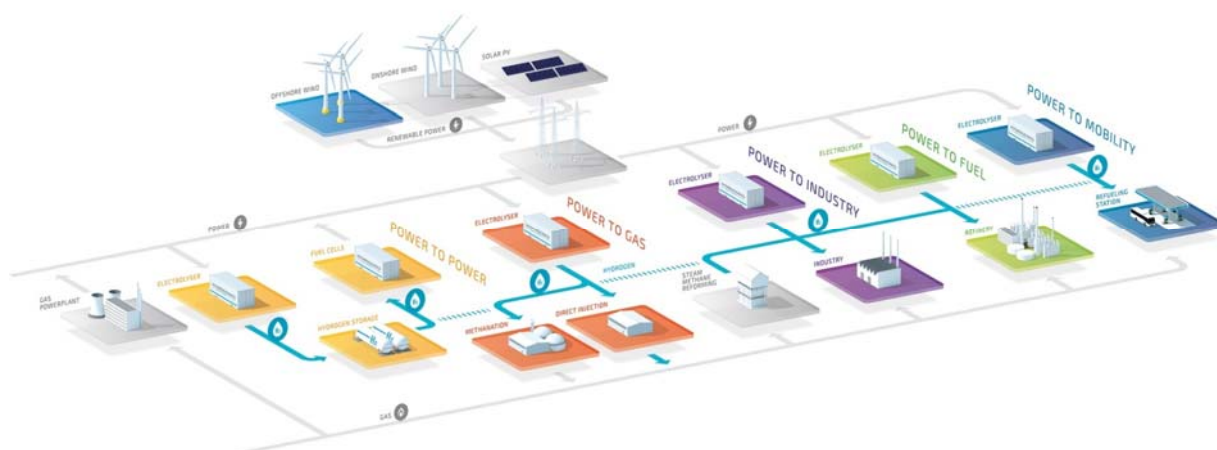
## PEM electrolysis development for enhancing renewable energy integration and advancing Power-to-X technologies

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Hydrogen has a long-standing history as a feedstock gas or process medium in industrial applications. Electrolytic hydrogen generation is a good option whenever small to medium flows are required, and is also often the technology of choice when high purity streams are needed. Currently there is an increased demand for electrochemical hydrogen because it gives an answer to the growing practical issues related to the integration of renewable energies in today's energy mix. In the power-to-gas, power-to-fuel and power-to-chemicals concept, which provide routes for a carbon-neutral/free economic future, water electrolysis is the key technology to store renewable energy surpluses, allowing the crossover between different energy vectors; electricity on the grid, gas or liquid as chemical energy storage and fuel for transport applications (see Figure below). This contribution presents the recent PEM based electrolyser developments of Hydrogenics, with a focus on the developments in two EU-funded projects, HyBalance and MefCO<sub>2</sub>. HyBalance, an FCH-JU and Energynet.dk co-funded project, illustrates how PEM water electrolysis, complimented with additional hydrogen compression and transportation technologies, demonstrates different hydrogen applications in real life when a number of important boundary conditions are met. Electricity is converted by pressurized water electrolysis into hydrogen and further compressed and transported at high pressure for Hydrogen delivery to the fueling stations operated by CHN in Denmark. MefCO<sub>2</sub>, an EU funded project (H2020), aims to demonstrate the economic feasibility of valorising captured CO<sub>2</sub> by turning it into a versatile platform chemical and renewable fuel such as methanol using hydrogen produced from renewable energy surplus. The CO<sub>2</sub> for the process is provided from the CO<sub>2</sub> capture pilot plant at RWE's lignite-fired power plant at Niederaussem. These projects enabled Hydrogenics to further improve and expand their PEM design, which will be highlighted in this contribution.



*Power-to-X: Hydrogen produced from renewable power via water electrolysis can be used to reduce carbon emissions further in the power, gas, industry, fuel and mobility sectors, for a cleaner and moresustainable future.*

Abstract No 37

## Low temperature electrolysis, yet at higher temperature

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Electrolyzers are finally gaining the attention they long deserved, partly pulled by the emerging need for energy conversion in the green transition. The three parameters, performance, durability and cost are still the key drivers for improvement of the technologies. A good way to reduce cost is to improve performance, either to enable higher current density (CAPEX) or lower cell voltage (OPEX).

One direct way to increase performance is to increase the working temperature of the cell. This reduces the free energy demand, which directly allows for utilization of more of the heat generated by losses in the cell or even of additional heat from an external source. Additionally, it improves kinetics and conductivity (anything else unchanged). All this has already been demonstrated with solid oxide cells. If the starting point is the low temperature electrolyzers, i.e. alkaline and PEM, significant modifications are necessary to enable a higher working temperature. Especially the electrolyte in which conductivity is normally based on water in a condensed form is critical at temperatures above 100 °C.

The paper will review attempts at the department with alkaline electrolyzers at 200-250 °C [1-3], PEM electrolyzers at 120-130 °C [4,5] and phosphate based electrolyzers at 200-300 °C [6,7]. In the alkaline cells, the humidity of the electrolyte was maintained by a moderate pressurization, in the PEM cells, water was replaced by phosphoric acid and in the phosphate-based cells, the electrolyte was either a molten phosphate or solid CsH<sub>2</sub>PO<sub>4</sub>. The advantages and challenges will be assessed followed by some considerations on the suitability of such cells in the energy system.

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## Degradation analysis at increased stressor level in PEM water electrolysis single cells

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To reduce hydrogen generation costs from PEM electrolysis systems to competitive levels, novel cell materials with improved properties (lower gas permeation rate, higher stability or lower costs) are needed. To reduce the measurement time to an economically and scientifically acceptable level of few 100 hours maximum, accelerated stress tests (AST) are necessary. As also defined by the harmonisation group of the Joint Research Centre (JRC) [1] by the European Commission, stressors were defined, like temperature, current density, water flow rate, ionic contamination level etc. at high and moderate level. In this work long term measurement at high stress levels with a temperature of  $T = 80\text{ }^{\circ}\text{C}$ , a current density of  $i = 4\text{ A/cm}^2$  and a water flow rate of  $Q = 50\text{ ml/min}$  is presented. As reference, tests at moderate stressor level were performed. Therefore stressors more close to current applications level were chosen ( $T = 60\text{ }^{\circ}\text{C}$ ,  $i = 2\text{ A/cm}^2$  and  $Q = 300\text{ ml/min}$ ). All tests were performed on single cell laboratory test equipment with a built in reference electrode (developed in house at Fraunhofer ISE) to distinguish between anodic and cathodic contribution to the observed degradation. Electrochemical impedance spectroscopy (EIS) was done at full cell and half-cell level to have more insight on the degradation phenomena. The cells were equipped with a commercial membrane electrode assembly (MEA) based on a Nafion 117 membrane. Strong degradation was observed for the degradation test at high stressor level,  $221\text{ }\mu\text{V/h}$  for a measurement time of 1,412 h and basically no degradation was observed for the reference measurement for a measurement time of 1,169 h (see Figure 6). Based on transient signals,  $V_i$ -curves, analysis of gas quality and Nyquist plots from the impedance analysis the conclusion was made, that the observed degradation at high stressor level originate mainly from the anodic side (Figure 6 & 2). Here, kinetics changes are almost not quantifiable and ohmic losses change strongly (seen by an HFR change over the measurement time). Due to an analysis of the high frequency resistances (HFR) after a steady-state operation for 1,420 h, degradation is mainly ohmic in nature (protonic conductivity of the membrane and/or interfacial contact resistances (ICR)). Other publications point to the same direction [2]. Further ex-situ analysis of the porous transport layer (PTL) and the membrane will provide deeper information on the underlying effects. In order to gain general validity of the observed degradation, the tests under harsh condition are currently done on other test benches and at other institutions.

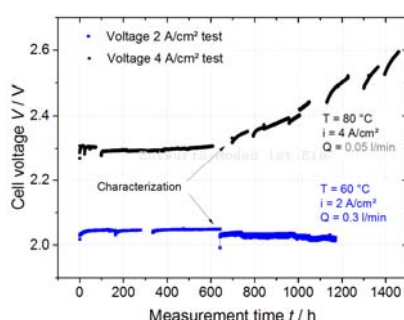


Figure 6: Voltage transients for both the degradation test at high (black) and reference test at low (blue) stressor levels

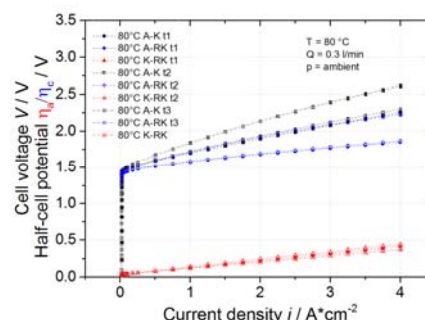


Figure 7:  $V_i$  curves at full-cell and half-cell configuration for the degradation test at high stressor level

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## System relevant Observation of Gas Crossover – Necessity of Mitigation Strategies

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Gas crossover is a big issue for PEM water electrolysis. Especially, the hydrogen crossover through the thin polymer membranes can cause Faraday losses and safety issues that lead to an unfavourable part load behaviour. In the framework of this contribution experimental data in combination with model results will be presented that observe the effects of gas crossover in context of the system relevance.

Fig. 1a) shows the current density range for three different hydrogen pressures as a function of the membrane thickness. The upper limit represents the maximal current density at a selected cell voltage of 2 V [1]. The lower limit is set to a H<sub>2</sub> in O<sub>2</sub> content of 2 vol.% [1]. So, the range between the upper and lower current density can be also called as the operating window. It can be clearly seen that this operating window significantly decreases with increasing hydrogen pressure, because of the increasing hydrogen crossover and H<sub>2</sub> in O<sub>2</sub> content. This highlights the necessity of mitigation strategies to reduce the H<sub>2</sub> in O<sub>2</sub> content or directly the hydrogen crossover. Fig. 1b) shows exemplary the effect of a Pt recombination interlayer within the membrane, which strongly reduces the H<sub>2</sub> in O<sub>2</sub> content [2, 3]. This and other mitigation strategies are important to solve the safety issue at part load to extend the operating window for higher pressures and thin membranes.

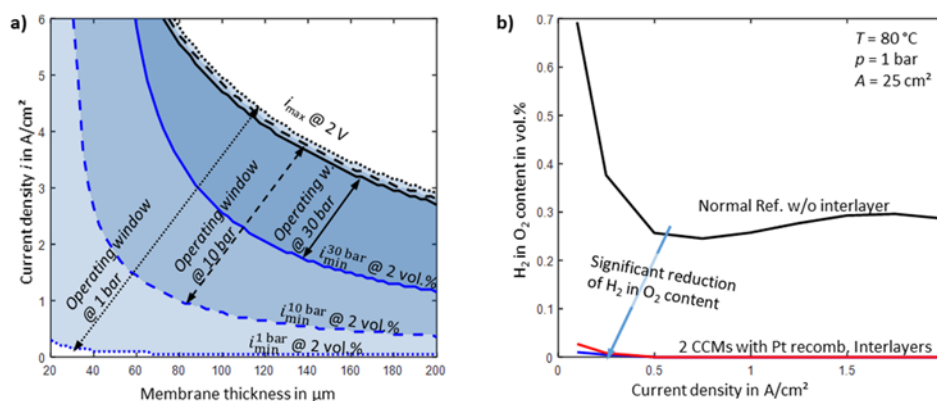


Figure 8. a) Operating window for different cathode pressures for a N115 membrane:  $i_{min}$  @ 2vol.% and  $i_{max}$  @ 2 V. b) H<sub>2</sub> in O<sub>2</sub> reduction via recombination interlayer (data are taken from [2]).

Faraday losses gain importance at high pressures and thin membranes. Thus, the overall cell efficiency for thin membranes can be lower as for thicker membranes especially at low to moderate current densities, if the beneficial reduction of ohmic losses of thinner membranes is smaller than the increased Faraday losses due to crossover. Consequently, the optimal membrane thickness depends on the operating conditions and load profiles. In the framework of this contribution a model was developed to determine the optimal membrane thickness for certain operating conditions and load profiles.

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## Cobalt Platinum Bronze for an Active and Durable OER Electrocatalyst of PEM Electrolysis without Ir or Ru

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Water electrolysis is an essential technology for realizing the renewable energy society. PEM electrolysis, in particular, is promising because of its high power density despite its high cost per unit active area. Its OER catalyst often contains Ir or Ru (or both) because of their high OER activities and/or durabilities. Although the current prices of these two metals are not extremely high compared with other precious metals because of their relatively low demand, Ir and Ru are, however, ones of the scarcest elements on earth and this extreme scarcity could be a major obstacle for wide distribution of PEM electrolysis technology.

Recently we have started fundamental studies [1] on mixed oxides of platinum so called "platinum bronze" (Bronze in this context means mixed oxides with metallic sheen) consisting of platinum, oxygen and other metal ions (such as Li, Na, Mg, Ca, Zn, Cd, Co, Ni) as a versatile electrocatalyst for ORR, OER and HOR.

Co-Pt bronze was prepared according to the literature [1] with some modifications. Platinum oxide and  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  powders were mixed and heat-treated under air conditions at  $650^\circ\text{C}$  for 5 hours. The obtained powder was washed with heated ( $80^\circ\text{C}$ ) aqua regia for 3 times and the remaining powder was then thoroughly rinsed with pure water.

The electronic conductivity of the bronze measured with a compression cell was compared in Fig. 1 with those of Vulcan XC-72 (a typical conductive carbon black),  $\text{IrO}_2$  (a typical OER catalyst) and  $\text{PtO}_2$ . The Co-Pt bronze showed a comparable conductivity with XC-72 and  $\text{IrO}_2$ , which is much higher than that of  $\text{PtO}_2$ , an insulator.

OER activities were measured by potential sweep and potential hold experiments. The electrodes were prepared by depositing the catalysts on a glassy carbon disk electrode. Fig. 2 show that the Co-Pt bronze has a similarly active and stable OER activity with  $\text{IrO}_2$  not like Pt black and Pt/Vulcan, which show rapid decay of OER current due to formation of  $\text{PtO}_2$ .

This study is financially support by the New Energy and Industrial Technology Development Organization.

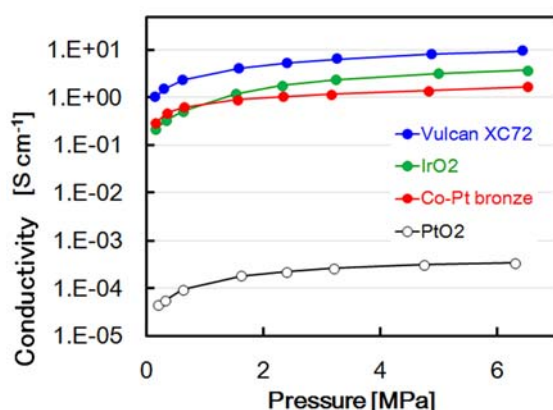


Fig. 1 Electronic conductivities under pressure

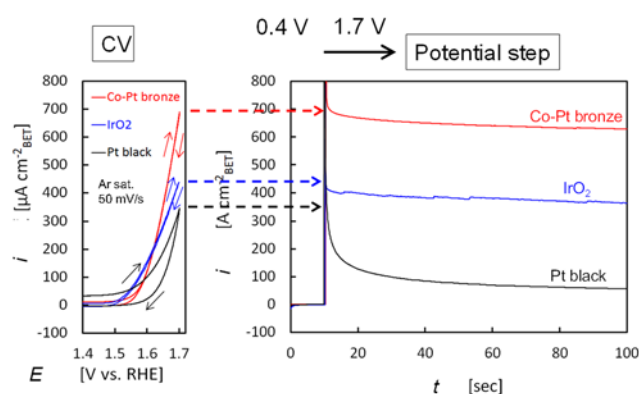


Fig. 2 OER activity comparison. Left: Potential cycle, Right: Potential step

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## Improving the performance of low loaded PEMWE electrodes

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In polymer electrolyte membrane water electrolysis (PEMWE) the reduction of the catalyst loading is one of the key challenges with respect to commercialization. As catalyst support materials are either not stable in the corrosive anode or difficult to fabricate and process, pure catalyst anodes are very common. Therefore, high loadings are used in order to provide a sufficiently large active surface area and to achieve high performance. When reducing the loading, insufficient electric contact of catalyst material becomes a major contributor to the overvoltage. This is due to poor in-plane electron conductivity in the catalyst layer (CL) between the contact points with the porous transport layer (Figure 9) [1,2]. A microporous layer (MPL) between the CL and the PTL can improve the in-plane electron conduction and the ohmic interface resistance. MPLs, produced e.g. by vacuum plasma spraying [3] show a significant performance increase, but are still expensive. We present a novel fabrication method, which reduces costs and improves performance of PEMWEs with low loading.

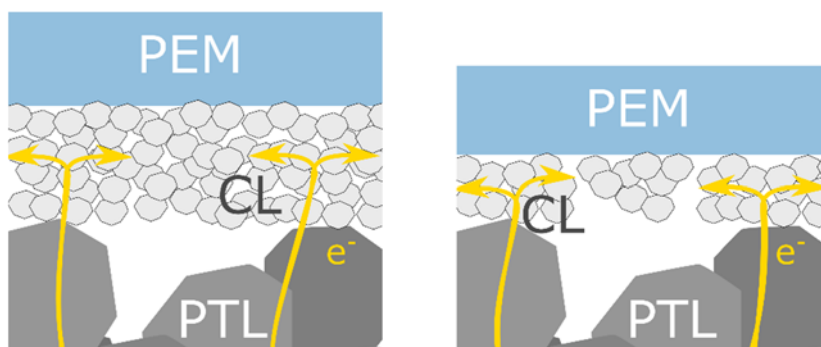


Figure 9 Electron path ways from porous transport layer to catalyst layer in a PEMWE. At high catalyst loading in plane transport is enabled by the comparably thick CL (left). At low loading the in plane electric resistance is increased and some parts of the CL loose the electric connection leading to a reduced active surface area (right).

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## High performing PEMEC MEAs with (ultra)-low PGM-loading

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Hydrogen generation by polymer electrolyte membrane water electrolysis (PEMWE) was investigated with regard to the development and characterization of PEM-electrolysis MEAs (membrane electrode assembly) with (ultra)-low Platinum Group Metal (PGM) loading, high performance and low degradation. A screening analysis of different MEAs based on various precursors and combinations was made. High performance was achieved for MEAs based on the Solvay membrane Aquivion® E98-09S, stabilised Solvay ionomer (D98-06ASX), and optimised catalysts developed by CNR-ITAE (cathode 40% Pt on C and anode: Ir<sub>0.7</sub>Ru<sub>0.3</sub>O<sub>x</sub>). Small, medium and full sized MEAs (<10, 130 and 415 cm<sup>2</sup> active area, respectively) were manufactured by IRD and investigated at ITM in a short stack for hydrogen production. Long-term test have illustrated that the decay rate declines with time. The performance decay recorded for optimised MEAs was 8 μV/h @ 3 A/cm<sup>2</sup> after 4,000 h operation using a total catalyst loading of 0.44 mg cm<sup>-2</sup><sub>MEA</sub> (Fig. 1). Current and thermal cycling of single cells proved to have limited impact on degradation. Pronounced reversible degradation has been observed at low temperature (55°C) and in a much lower extent at high temperature (80°C).

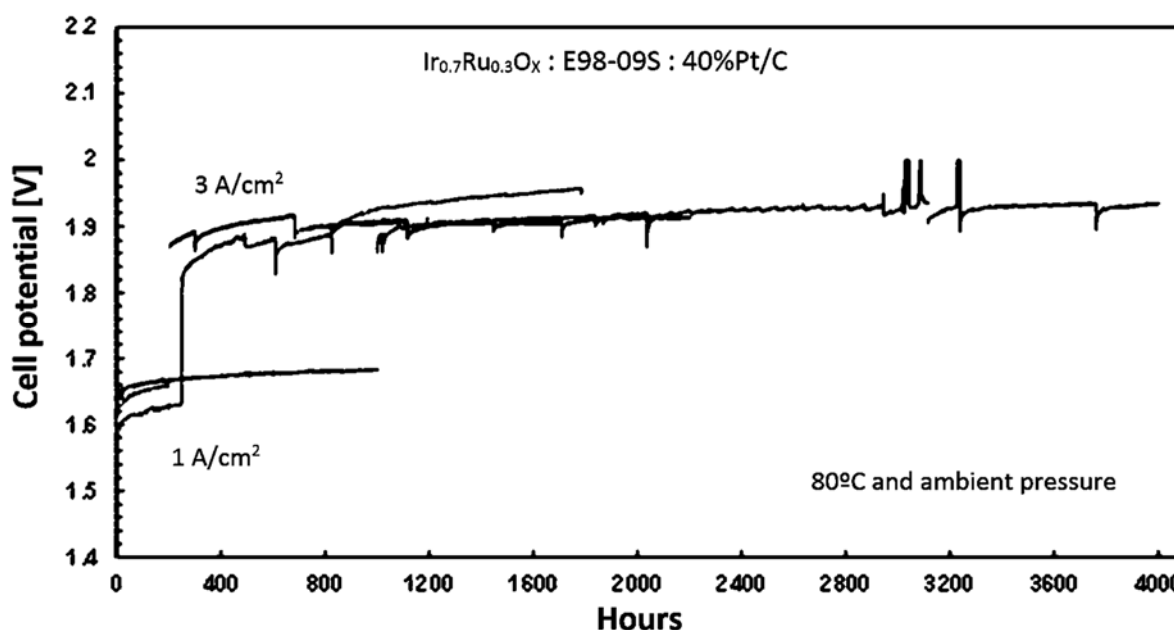


Fig. 1 Durability studies at CNR-ITAE for different MEAs with a total catalyst loading of 0.44 mg cm<sup>-2</sup><sub>MEA</sub> at 80°C.

The research leading to these results has received funding from the European Community's Horizon 2020 Programme (H2020-JTI-FCH-2015-1) for the Fuel Cells and Hydrogen Joint Technology Initiative under grant agreement HPEM2GAS n° 700008.

## Low Temperature Electrolysis Advances at NREL

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In commercial electrolysis, the cost of electricity input drives the cost of hydrogen production. Therefore, electrolysis is typically run at high catalyst loading and constant power input over long periods of time. Lowering water-splitting hydrogen production costs to a level comparable to steam methane reformation requires: coupling electrolysis with low-cost power input (wind, solar) to reduce feedstock costs; and dropping catalyst loading to reduce the capital cost at lower capacity.[1,2] While minimal durability loss is seen in commercial electrolyzers, catalyst losses can be masked by high loading (several mg cm<sup>-2</sup>). At low loading, however, these losses become more apparent.[3]

In this study, electrolyzer durability was evaluated at low iridium-anode loading (0.1–0.5 mg cm<sup>-2</sup>) and with different power inputs (potentials, intermittency). Higher loading tended to delay the onset of durability losses; in contrast, a loading of 0.1 mg cm<sup>-2</sup> resulted in incremental but immediate loss when exposed to high potential. Increasing the upper potential limit appeared to increase iridium dissolution and migration and resulted in higher performance losses. This trend was expected and iridium dissolution is anticipated to be a primary factor in electrolyzer loss at low loading.[4] Introducing cycling (square/triangle wave), however, significantly increased the rate of performance decay and was less expected from half-cell tests and even though less time was spent at elevated potential.[3] Changing the rate of potential increase (saw tooth profiles) confirmed that rapid input increases accelerated loss and may be due to localized potential spikes occurring within the catalyst layer. A variety of system control-based mitigation strategies have been evaluated for lessening durability losses when handling intermittent power sources.

Several iridium catalyst types (oxides, surface areas) have been tested in half- and single-cells. Catalyst development efforts often focus on metallic- or hydroxide-based iridium structures, due to higher activity in ex-situ tests. These performance advantages, however, largely disappear in single-cell tests and may be due to surface/near-surface oxidation during conditioning protocols. Metallic/hydroxide durability losses further tend to be larger in both half- and single-cells and may be related to the kinetics of iridium metal/hydroxide/oxide dissolution.

In single-cell testing, we have focused on how loading, test parameters, and catalyst type affect proton exchange membrane-based electrolyzer durability. These tests have significant implications on lowering the cost of electrolysis-based hydrogen production and on coupling electrolysis with renewable power inputs.

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## Investigation on the Effect of Ionomer Loading and Catalyst Loading on Tantalum Carbide Support on Polymer Electrolyte Membrane Electrolyser Performance

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The use of hydrogen to incorporate renewable energy technologies into the mainstream energy mix has been identified as a viable pathway for the replacement of fossil fuels. Polymer electrolyte membrane water electrolysis can be used to generate hydrogen from surplus energy generated from renewables. One of the main obstacles to the widespread use of PEM electrolysis is the high cost of the systems. Strategies to reduce costs include the use of supports for the catalysts to reduce the loading of the noble metals in the catalysts. It is also known that the materials used to fabricate the electrode individually and synergistically contribute to the performance of the electrolyser cell. There exists an optimal composition of each material in the electrode that will result in the best performance for the cell. Tantalum carbide has been identified as a suitable support for iridium based catalysts [1-3]. However, not many studies have been reported in literature on the optimisation of electrodes made from these catalysts. In this study, the effect of ionomer content, and iridium oxide ( $\text{IrO}_x$ ) catalyst loading on tantalum carbide (TaC) support on the performance of a polymer electrolyte membrane electrolyser cell were investigated. The  $\text{IrO}_x/\text{TaC}$  catalyst was prepared using a surfactant mediated method. The anode was prepared using the decal method. The ionomer content in the anode was varied between targets of 10%, 15% and 22% while the iridium oxide loading on the support was varied between target values of 7.5%, 15% and 30%. The cell was operated potentiostatically at 60°C, at atmospheric pressure and a flowrate of 6 ml/min. An in-situ reference electrode was used to determine the contributions of the anode and cathode to the cell voltage. The results indicate that there is an optimum combination of ionomer content and catalyst loading that will result in optimal electrolyser performance.

**Keywords** polymer electrolyte membrane water electrolysis; iridium oxide, tantalum carbide support

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## Direct membrane deposition – a novel membrane electrode assembly for proton exchange membrane water electrolysis

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The improvement of manufacturing methods for membrane electrode assemblies (MEAs) is not only a crucial part of industrial processes towards lower cost and high throughput, but also has the potential to decrease voltage losses significantly. In this work we show results from a novel MEA manufacturing approach for proton exchange membrane (PEM) water electrolysis, in which the catalyst and membrane material is subsequently deposited onto porous transport layers (PTLs). This was previously used for PEM fuel cells and called ‘direct membrane deposition’ (DMD) [1]. Fabricating fuel cell MEAs via DMD, rather than the conventional catalyst coated membrane (CCM) approach, resulted in performance improvements and enables a higher design freedom.

Previously reported DMD fuel cell MEAs were fabricated by depositing membrane layers onto both, anode and cathode gas diffusion electrodes. Anode and cathode were then sandwiched together with a PTFE subgasket in between to seal the edges. Due to the typical, macroscopic porosity of the anodic titanium-based PTE, the optimal DMD process for PEM water electrolysis was found by depositing the membrane solely on the cathode side and using membrane material to seal the edges instead of a subgasket (Figure 1A).

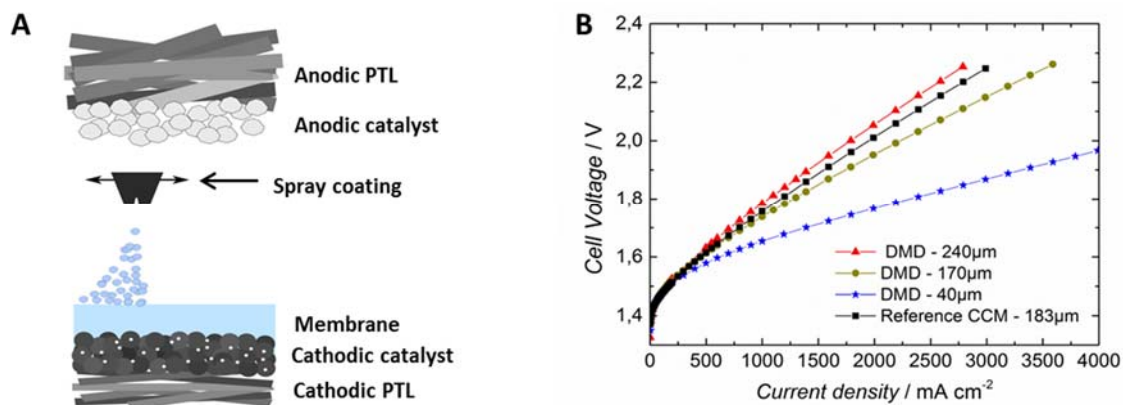


Figure 10: (A) Structure of a PEM water electrolysis DMD MEA with catalyst coated porous titanium PTL at the anode, a catalyst coated carbon PTL at the cathode and the membrane coated onto the cathode. (B) Polarization curves of DMD MEAs with different membrane thicknesses, anodic catalyst loading:  $1.5 \text{ mg IrO}_2 \text{ cm}^{-2}$ , cathodic catalyst loading:  $0.5 \text{ mg Pt cm}^{-2}$

Polarization experiments of DMD MEAs (Figure 1B) show promising results. In comparison to a CCM reference (identical catalyst loading, N117 membrane) we found lower voltage losses in spite of a comparable membrane thickness of  $170 \mu\text{m}$ . Highest performance was achieved with an only  $40 \mu\text{m}$  thin membrane due to reduced ohmic resistance of the thinner membrane. A remarkable current density of  $\sim 4 \text{ A/cm}^2$  was reached at  $1.97 \text{ V}$ .

In addition to cell tests, also methodologies to mitigate cross-over in thin, directly deposited membranes are presented in this work. Finally we demonstrate the application of DMD on non-noble metal cathode catalyst systems.

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## Proton ceramic electrochemical cells for efficient hydrogen production

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### Abstract.

Proton ceramic electrochemical cells (PCECs) comprise fuel cells (PCFCs) and electrolyzers (PCEs) with proton conducting oxide electrolytes, notably Y-doped BaZrO<sub>3</sub> (BZY) based formulations. They operate at temperatures in principle a bit lower than oxide ion conducting solid oxide electrochemical cells (SOECs) – due to the lower activation energy of mobility for protons than for oxygen vacancies – typically 400-700°C. The transport of protons allows PCFCs to run on hydrogen with 100% fuel utilisation (no fuel dilution) and PCEs to directly produce pure dry electrochemically compressed hydrogen, making them in principle efficient, safe, and simple in terms of BoP and footprint. The operating temperature of PCEs allows integration with available sources of heat (e.g. solar-thermal or industrial waste heat) or steam for superior overall efficiency. Uniquely, proton transport allows very efficient electrochemical hydrogen production from natural gas in combination with direct dehydrogenation or steam reforming with CCS. Co-ionic transport of protons and oxide ions can be achieved by partial dehydration at increased operating temperature or by a secondary phase oxide ion conductor, and yields the possibility for co-electrolysis of H<sub>2</sub>O and CO<sub>2</sub> to synthesis gas, CO + 2H<sub>2</sub>.

Challenges for proton ceramic electrolytes comprise high sintering temperatures causing evaporative Ba loss, high grain boundary resistance due to proton-depleted space charge regions, partial p-type electronic conductivity causing reduced faradaic efficiency in steam electrolyzers, and issues related to a relatively low thermal expansion coefficient and chemical expansion during hydration. Ongoing developments to understand and solve these issues will be discussed briefly.

Negatrodes for PCECs are mostly Ni-BZY cermets, which perform well due to the catalytic activity of Ni and seemingly precipitation of Ni nanoparticles on BZY surfaces under reducing conditions from Ni<sup>2+</sup> ions dissolved in the BZY during sintering under oxidising conditions.

Positrodes on the other hand pose bigger challenges in terms of processing (e.g. adherence) and the lack of good mixed proton electron conductors (MPECs). Highly performing positrodes are essential for PCEs with high electrical and faradaic efficiency, since the overpotential here increases p-type electronic conductivity in the electrolyte. Recent progress in performance and in understanding the rate-limiting electrode mechanistic steps of proton ceramic positrodes will be discussed briefly.

Abstract No 47

## Cobalt substituted Lanthanide Nickelates ( $\text{Ln}_2\text{Ni}_{1-x}\text{Co}_x\text{O}_{4+\delta}$ , Ln = La, Pr; x=0, 0.1, 0.2): Impact on Electrochemical Performance and Stability as SOECs Oxygen Electrode

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The  $\text{K}_2\text{NiF}_4$ -type layered perovskites *e.g.* nickelates  $\text{Ln}_2\text{NiO}_{4+\delta}$  (Ln = La, Pr) are promising mixed ionic and electronic conducting (MIEC) oxygen electrode materials due to their high anionic bulk diffusion as well as surface exchange coefficients, combined with good electrical conductivity and thermal expansion properties matching with those of yttria-stabilized zirconia (YSZ) or  $\text{Ce}_{0.8}\text{Gd}_{0.2}\text{O}_{2-\delta}$  (GDC) used as electrolytes. To further enhance the physico-chemical properties, electrochemical performance as oxygen electrode and durability of Solid Oxide Electrolyzer Cells (SOECs), in this work, we have performed the substitution of nickel with cobalt in these nickelates. Two compositions (x=0.1 and 0.2) were mainly considered (as higher cobalt containing nickelates are difficult to synthesize under air atmosphere) and completely characterized using several techniques. The symmetrical half-cells (8YSZ/GDC/electrode) as well as single cells (Ni-YSZ/8YSZ/GDC/electrode) were then prepared and characterized using electrochemical impedance spectroscopy in the temperature range 700 - 900 °C.

The *i*-V characteristics of the single cells show an increase in the performance with increasing cobalt content for  $\text{Ln}_2\text{Ni}_{1-x}\text{Co}_x\text{O}_{4+\delta}$  electrodes (Fig. 1). For instance, the cell current densities obtained at 900 °C with an applied voltage of 1.5 V are 2.06, 2.19, 2.10 and 3.0  $\text{A}\cdot\text{cm}^{-2}$  respectively for  $\text{La}_2\text{NiO}_{4+\delta}$  (LNO),  $\text{La}_2\text{Ni}_{0.8}\text{Co}_{0.2}\text{O}_{4+\delta}$  (LNCO20),  $\text{Pr}_2\text{NiO}_{4+\delta}$  (PNO) and  $\text{Pr}_2\text{Ni}_{0.8}\text{Co}_{0.2}\text{O}_{4+\delta}$  (PNCO20) single cells. The outstanding performance is obtained with the single cell containing PNCO20 electrode, most importantly, far better than that of commercial single cells containing conventional LSCF electrodes. Moreover the durability test up to 250 h were also investigated at 1  $\text{A}\cdot\text{cm}^{-2}$  current density at 800 °C with 50%  $\text{H}_2$  and 50%  $\text{H}_2\text{O}$  feed gas mixture indicating different degradation behaviour (Fig. 1b). The largest degradation is observed for LNO cell (113 mV/kh) and the most stable performance obtained with PNCO20 single cell with an estimated degradation rate of 22 mV/kh. The post-test analysis performed on the single cells after 250 h of operation at electrolysis conditions revealed a large reactivity between the LNO electrode and the GDC interlayer most likely leading to high degradation rate observed. Results including physico-chemical and electrochemical properties of these materials, electrochemical performance as oxygen electrode, long term stability under electrolysis conditions and post-test analysis (using SEM-EDX, XRD) will be presented and discussed in detail.

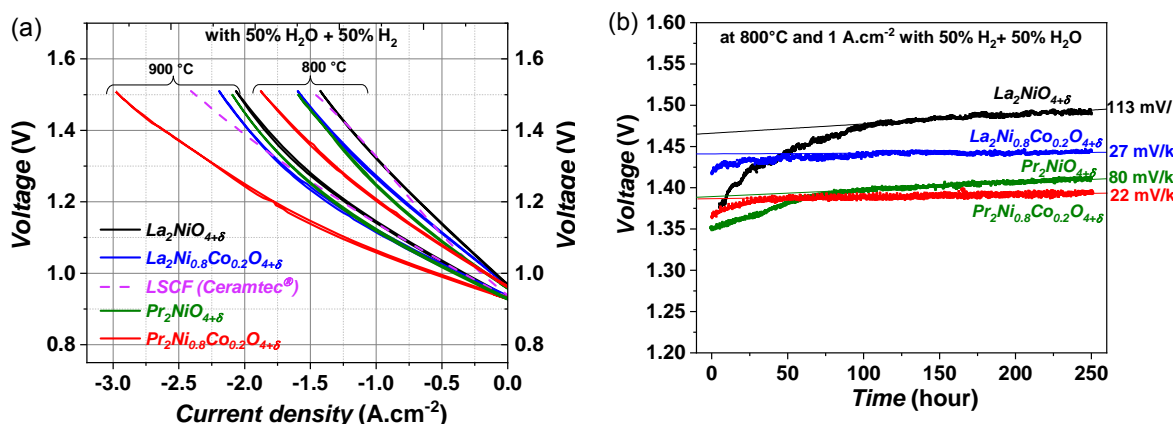


Fig. 1: (a) Voltage vs. current density curves at 800 and 900 °C, (b) Durability up to 250 h at 800 °C at 1  $\text{A}\cdot\text{cm}^{-2}$  current density; for LNO, LNCO20, PNO and PNCO20 single cells with 50 %  $\text{H}_2$  + 50 %  $\text{H}_2\text{O}$  gas mixture.

\* The results reported here were obtained within the frame of an European project SElySOs. This project has received funding from the Fuel Cells and Hydrogen 2 Joint Undertaking under grant agreement No 671481.

## Degradation Phenomena in Solid Oxide Electrolysis Cell Oxygen Electrodes

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In order to ensure a viable lifetime of solid oxide electrolysis cells (SOECs), it is important to understand degradation mechanisms. Mechanisms such as delamination and oxygen bubble/pore formation have been observed at the oxygen electrode during operation of SOECs. Although the degradation has been shown to be strongly dependent on current density, the effects of other operating parameters are generally not known. Here we report results on life tests done on both symmetric cells and full cells with Yttria-Stabilized Zirconia (YSZ) electrolytes with Gd-doped Ceria (GDC) barrier layers, with various oxygen electrode materials including  $(\text{La}_{0.6}\text{Sr}_{0.4})(\text{Co}_{0.2}\text{Fe}_{0.8})\text{O}_3$  (LSCF) and two materials recently shown to provide superior performance and stability:  $\text{Sr}(\text{Ti}_{0.3}\text{Fe}_{0.7})\text{O}_3$  (STF) and  $\text{Sr}(\text{Ti}_{0.3}\text{Fe}_{0.63}\text{Co}_{0.07})\text{O}_3$  (STFC). The effects of operating temperature, current density, and electrode material are described. For example, cells with LSCF or STF electrodes operated at 1 A/cm<sup>2</sup> are stable at high temperature, but for temperature < 650 °C, the cells fail due to delamination at the GDC/electrode interface. Delamination is generally observed for conditions (temperature, current density, and electrode type) that give rise to an oxygen electrode overpotential  $\eta \gtrsim 0.2$  V.

A quantitative model for charge carrier transport across the electrolyte, based on that reported by Chatzichristodoulou *et al* [1], is used to predict the electrical and oxygen potentials across the YSZ/GDC electrolyte and thereby provide insights into degradation. The distribution of oxygen partial pressure is determined for different electrode materials and different operating modes. The oxygen pressure at the electrolyte/electrode interface is largely determined by the electrode overpotential. The oxygen pressures reached at the interface under a given condition are identical for symmetrical and full cells with identical oxygen electrodes. Thus, to the extent that failure occurs at the electrolyte/electrode interface, symmetrical and full cells should give the same failure condition, as observed experimentally. The maximum oxygen pressure is observed at the interface between YSZ and the GDC barrier layer; nonetheless, the model predicts failure at the GDC/electrode interface because of the relatively low fracture toughness of the perovskite electrode material. The model-predicted failure conditions, where the oxygen pressure exceeds the perovskite fracture toughness, are in quantitative agreement with the experimentally observed current density and temperature (and critical overpotential value) where failure is observed.

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## **Experimental analysis of SOE stacks under pressurized operation**

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The major part of the prospective electricity system will be based on solar and wind energy. The associated intermittency of this energy supply requires flexible storage and usage options. Converting electrical into chemical energy could be an essential constituent in this challenge. One promising path is the solid oxide electrolysis (SOE) technology which can largely contribute to hydrogen and hydrocarbon production as well as electric energy storage.

SOEs offer a pronounced potential for a highly efficient energy conversion due to their high operating temperature. The fast kinetics of electrochemical reactions lead to reduced electrochemical losses. Additionally, previous studies have shown that the performance of solid oxide cells can be significantly improved by operating at elevated pressure [1 - 3]. A further reason for pressurization is the use of pressurized hydrogen or syngas in downstream processes for storage or fuel synthesis.

Experimental results of SOE stacks operated under pressurized conditions in steam- and co-electrolysis mode are presented. For the studies, 10-layer stacks with fuel electrode and electrolyte supported cells were used and compared in terms of their performance in a pressure range between 1.4 and 8 bar. Pressure influence on the reachable power density and the overall performance was examined on the basis of current-voltage curves. The ASR values were found to decrease due to reduced activation and diffusion resistances under pressurized conditions for both cell concepts. However, for stacks with fuel electrode supported cells the pressure influence was found to be more significant and an overall performance gain was already observed during endothermic operation. Gas analysis was further used to determine the produced syngas composition in pressurized co-electrolysis tests. Impedance spectroscopy was carried out to quantify and distinguish between different pressure influenced electrochemical phenomena. Furthermore, degradation tests up to 2000 hours performed on stacks with electrolyte supported cells showed increased degradation rates with higher operating pressures. EIS measurements identified the ohmic resistance as major contributor to the stack degradation whereas the polarization resistance was found to play a subordinate role.

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## Recent Solid Oxide Electrolysis Research Highlights at DTU Energy

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At DTU Energy a range of research activities on the solid oxide cell technology is carried out in parallel projects. In this presentation highlights from these projects are presented.

In the development of new electrodes, working with nano-sized powders is appealing, as surface area is increased, and new properties may appear. In the ProEco project a continuous hydrothermal flow synthesis reactor has been developed. By using a flow-type setup, the oxide nanoparticles can be synthesized and harvested in a continuous manner, starting from the corresponding metal nitrates and super-critical H<sub>2</sub>O. Results on improved cell performance using nano-sized powders as compared to conventional micron-sized powders will be shown.

Using the hydrothermal reactor several new types of nano-sized powders were also used to tailor the doping of zirconia in the fuel electrode support to achieve a significant strength enhancement over state-of-the-art (3YSZ) in the SYNFOEL project. It was shown that lowering the yttria doping will increase the strength but decrease the chemical stability. However, by doping with both yttria and ceria ~30 % higher strength than that of 3YSZ can be achieved together with an improvement of the chemical stability.

The nano-sized powders also open the opportunity for well dispersed inks, which can be used for various printing processes. In the Cell3Ditor project deposition of functional thin layers using nanomaterials by inkjet printing and thick supports by robocasting have been demonstrated. Here it is shown how nanodispersions of NiO, CGO and YSZ have been converted into inks for inkjet deposition, achieving thin (< 1 μm) dense electrolytes and porous nanocomposites.

Through the sintering, nano-powdered materials are however prone to necking and coalescence, minimizing surface area resulting in poorer performance. In the EP2Gas project infiltration of a solution in the fuel electrode has been used to decorate it with nano-catalysts of CGO20. By impedance analysis it is revealed that this lowers the polarization resistance, and at -1 A/cm<sup>2</sup> water electrolysis it decreases the degradation rate by a factor of ~20.

A significant depressing of the degradation rate in CO<sub>2</sub>/CO electrolysis was also found in the Maturing SOEC project. Here it was found by Raman spectroscopy that it is not only the purity of CO for CO<sub>2</sub>/CO electrolysis that is critical, but even ppb levels of impurities in the CO<sub>2</sub> stream can lead increased fuel electrode overpotential that subsequently leads to detrimental carbon deposition. Subsequently a CO<sub>2</sub> gas cleaning system was employed to improve the stability.

Carbon deposition is also promoted by the nickel in the fuel electrode, why a novel cell design has been developed with a stainless steel mesh as structural component integrated into a porous nickel-free fuel electrode. Thin electrolytes (1-2 μm) are employed enabling operation at 500-600 °C. This approach has the potential to significantly lower manufacturing and operational costs.

CO<sub>2</sub> electrolysis opens together with H<sub>2</sub> electrolysis for the opportunity to synthesize a range of chemicals and fuel gasses (e.g. synthetic natural gas). In the European Eco project, the compatibility with fluctuating electricity input reflecting a wind profile on cell and stack level was studied, and the durability compared to operation at constant conditions. Results of the detailed electrochemical test and microstructural analysis will be presented.

To avoid failures during operation cell and stack level models can be applied to investigate the local overpotentials, partial pressures, mechanical stresses etc. To investigate a range operational conditions or long-term operation, the models must be relatively fast. Here a multi-scale stack modelling approach is presented including mass, charge and heat transport, electro-chemistry, and mechanical stresses. The multi-scale modelling approach allows for computation ~100 faster than state-of-the-art models.



Abstract No 51

## **Boosting the performance of reversible solid oxide cells by nano-sized electro-catalysts**

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Globally the amount of electricity generated from renewable energy sources is increasing. To integrate high amount of fluctuating energy into the existing energy grid, efficient and cost competitive conversion of electricity into other kinds of energy carriers is needed. The Solid Oxide Cell (SOC) technology has the potential to become a key technology in enabling this integration. SOC's can be operated both as fuel cell and electrolysis cell and are hence often called as Reversible Solid Oxide Cell (RSOC). When operated as a solid oxide electrolysis cell (SOEC), electrical energy can be converted to chemical energy and stored as H<sub>2</sub> or synthesis gas (syngas, CO+H<sub>2</sub>) via high temperature electrolysis of steam or co-electrolysis of steam and CO<sub>2</sub>. H<sub>2</sub> and syngas can be further processed to a variety of synthetic fuels, which may be stored and later reconverted into electricity using the same RSOC, but operated as a solid oxide fuel cell (SOFC).

This work focuses on the development of RSOC cell technology. The solid oxide cells developed at Technical University of Denmark are evaluated under various reversible operation scenarios with regard to performance and long-term durability. These cells are fuel-electrode supported planar solid oxide cells, originally developed for SOFC application with further optimization for electrolysis. The cells are tested in reversible mode at 750 – 800°C, with current densities varying from 0.25 to 1.5 A/cm<sup>2</sup>. The degradation of RSOC is dominated by the degradation in SOEC mode. At 750°C and 0.5 A/cm<sup>2</sup>, the cells show a degradation rate of 88 mV/1000 h in SOEC mode, but only 9 mV/1000 h in SOFC mode. Reversible operation does not introduce additional degradation. Under certain conditions, operating the cell in SOFC mode actually helps reducing the SOEC degradation. Impedance analysis indicates that a large part of the degradation is originated from the Ni/yttria stabilized zirconia (YSZ) electrode, associated with an increase in the Ni/YSZ electrode polarization resistance. By decorating the Ni/YSZ electrode with Ce<sub>0.8</sub>Gd<sub>0.2</sub>O<sub>2-δ</sub> (CGO) nano-catalysts derived from solution infiltration, a significant enhancement on the cell durability in SOEC mode is achieved. The CGO nano-catalysts presumably provide more three-phase boundaries (TPBs) for the steam reduction reaction but also act directly as an electro-catalyst leading to reduced polarization resistance and strongly enhanced long-term durability.

### **Acknowledgement.**

This work is supported by the project “BALANCE - Increasing penetration of renewable power, alternative fuels and grid flexibility by cross-vector electrochemical processes” under the European Union’s Horizon 2020 research and innovation programme (grant agreement No. 731224).



## Demonstration of reversible Solid Oxide Cell technology: results at cell, stack and system level

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The BALANCE project aims at accelerating the development of the reversible Solid Oxide Cell technology (rSOC). rSOCs can perform both the water electrolysis reaction (electrolysis mode) and the electrochemical oxidation of hydrogen (fuel cell mode) with the same device and at high efficiency. Therefore, such power-to-gas-to-power plants have a significant advantage over purely power-to-gas plants because their operation hours are not limited to the hours with availability of renewable power. When the power demand is high, they can switch to fuel cell mode and produce power based on renewable hydrogen.

One of the objectives of the BALANCE project is the demonstration of rSOC operation at system level at VTT. The cells are developed, manufactured and characterised at DTU. The stacks are assembled by CEA using their own stack design adapted for DTU cells. Two stacks will be integrated into a system at VTT for a total power of 6 kW in electrolysis mode (stack input DC power), corresponding to about 2 kW in fuel cell mode (DC power stack output). The aim of the demonstration is to show the concept feasibility, validate the achievable efficiency, obtaining experience with the Balance-of-Plant components and assess the durability behaviour. This presentation consists of results obtained from cell and stack electrochemical characterisation and system modelling.

The cells developed at DTU are fuel-electrode supported. The Area Specific Resistance (ASR) was measured to be 0.3 ohm cm<sup>2</sup> at -0.25 A/cm<sup>2</sup> in SOEC and 0.41 ohm cm<sup>2</sup> at 0.25 A/cm<sup>2</sup> in SOFC at 750°C. The degradation is dominated by SOEC operation with degradation of 88 mV/kh at 750°C at -0.5 A/cm<sup>2</sup>, while only 9 mV/kh was measured during SOFC operation at the same temperature at 0.5 A/cm<sup>2</sup> in steady-state operation. The cells were integrated in a 5-cell stack at CEA, which exhibits performance close to the unit cell (0.43 ohm cm<sup>2</sup> in SOEC and 0.46 ohm cm<sup>2</sup> in SOFC at 750°C on average), indicating excellent physical contact between cells and interconnect and good gas distribution. The good performance of the cell and the stack enables operation at -0.63 A/cm<sup>2</sup> at the thermoneutral point of 1.29 V at 700°C. Degradation rates of the stack were in line with the unit cell test with 6 mV/kh on average in SOFC mode and 97 mV/kh (excluding the worst cell at 200 mV/kh) in SOEC mode at +/- 0.5 A/cm<sup>2</sup> at 700°C.

The system model includes characteristics of stack, heat exchangers, electric steam generators, air blower and electric gas heaters. In addition, thermal losses are also taken into account. Two nominal operation points at 700°C were defined corresponding to an efficiency of 54 % in fuel cell mode (from LHV hydrogen to DC power) at 0.4 A/cm<sup>2</sup> and an efficiency of 71 % in electrolysis mode (from DC power to LHV hydrogen) at 1 A/cm<sup>2</sup>. This latter value rises up to 86 % if waste steam above 100°C is available. It is expected that higher efficiency can be achieved for a larger system because of better thermal integration (up to 9% point efficiency for better insulation and water pre-heating).

The next steps consist of the manufacturing of two large 25-cell stacks at CEA, their integration into VTT rSOC demonstration system and the completion of the test campaign.

**Acknowledgements** This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 731224.

Abstract No 53

## **Green Hydrogen Production by Alkaline Water Electrolysis**

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The renewable energy sources, i.e. wind power and photovoltaic systems, are characterized by a fluctuated and intermitted energy production. The power profile shows transient and dynamic shapes with peaks and frequent on-off. The electricity grid from renewable energy copes with management problem resulting in huge amount of curtailment. The economic efficiency of green hydrogen production from electrolysis can be achieved by using dynamic curtailed renewable electricity.

We selected alkaline electrolysis as a green hydrogen electrolyzer because it exhibits great advantages over other electrolysis technologies in terms of maturity, large capacity, and cost-effective features. The alkaline electrolysis uses simple iron and nickel electrode rather than noble metal electrode to produce hydrogen. In this presentation, the challenges in material developments including an electrode and a separator to improve the cell performance and durability of alkaline electrolysis as advance designs will be introduced. Researchers can be guided by knowledge of the interaction of economics, performance, and durability as the new materials are developed. Furthermore, this presentation outlines opportunities for water electrolysis integration with renewable energies

## **Electrode improvements for zero gap alkaline polymer electrolyte membrane electrolysis cells.**

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The green transition to a sustainable energy system requires large-scale conversion of electrical energy to storable and portable fuels. A key technology for this is water electrolysis. For large-scale implementation, energy efficient, cost efficiency, and TW scalability are crucial parameters.

One approach to achieve this is to develop an alkaline water electrolyzer (AWE) based on an alkaline ion conducting membrane. This has the potential to combine the electrical efficiency of the PEM electrolyzer and the cost efficiency of the alkaline electrolyzer in an alkaline PEM electrolyzer equivalent.

The key component in an alkaline PEM electrolyzer is the membrane, but equally important are the porous electrodes optimized for a zero-gap configuration. One way to produce electrodes for this application is the casting of catalytically active powders into a catalyst layer on a support serving as a porous transport layer (PTL).

Catalyst layers have been produced through a spray-gun method using nanopowders identified to be electrochemically active for the oxygen evolution reaction (nickel and nickel-iron alloys) and the hydrogen evolution reaction (nickel and nickel-molybdenum alloys). For cathodes, carbon paper has been used as a PTL, whereas nickel felt paper has been used as anode PTL. The electrodes have been evaluated in single cell electrolyzer tests in 20 wt% KOH at 60°C, and are further characterized by electrochemical impedance spectroscopy. Variations in catalyst loading, binder content, and preparation details have been investigated to identify the optimal preparation strategy.

Abstract No 55

## For When Materials Matter

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### Abstract.

PV3 Technologies is a business that focuses on electrochemical materials for a wide range of electrochemical technologies. Products for water electrolysis, fuel cells, corrosion protection and water treatment are currently commercially available, and products for Li-ion batteries are under development. The product development philosophy adopted by PV3 Technologies is one to ensure customers have products that have high performance, economic prices and high durability. Only when all three elements are built into the material do we call it a product.

Water electrolysis has been an important sector for our company from its establishment in 2011. Working in partnership with customers, suppliers and collaborators we have developed a range of industry-critical materials, specifically:

- Coatings for PEM water electrolyser cell components providing high conductivity and long-life protection against corrosion and embrittlement
- High performance anode and cathode catalysts for PEM water electrolyzers
- High performance cathode catalyst coatings for alkaline water electrolyzers

To facilitate the development of these products we have developed a range of key tests, including accelerated stress tests, that have been demonstrated to translate well to real-life operation. This paper will describe the tests and test philosophy that has allowed PV3 Technologies to develop commercially-relevant materials.

As a commercial organisation, we understand the importance of price and how price reductions can lead to greater market penetration for our and our customers' products. In 2016 PV3 Technologies began a development programme to establish a modular and scalable volume-production process for coatings PEM water electrolyser cell components. This highly successful development project resulted in an investment in new production capability, with industry-leading production velocity, capacity and quality. This paper will further describe these developments.

## Intensification of alkaline electrolysis

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Alkaline electrolysis is typically regarded as a mature technology, since the technology has been operated on large scale for almost 100 years. However, due to the limited market for water electrolysis equipment relatively little technology development has taken place over the past decades.

Classic alkaline electrolysis is still the most competitive water electrolysis technology as evidenced by the figure, which shows two current-voltage graphs: one with the current density expressed in A/m<sup>2</sup> and one with the current density expressed in A/€. The latter is obtained by dividing the current density by the stack costs per m<sup>2</sup> (given in the left graph). These stack costs were deduced from quotations received from suppliers for a 20 MW facility in 2017-2018. From the right graph it can be concluded that the much lower stack costs of classic alkaline technology make it more attractive than PEM technology, even though it operates at a lower current density.

To make green hydrogen competitive with fossil hydrogen stack costs of <100 €/kW at an energy consumption of <4.5 kWh/Nm<sup>3</sup> are needed. This can potentially be achieved by increasing the current density in alkaline electrolysis by using new separators, improved electrode coatings, increased temperature and new cell designs. Promising research on all these topics has been carried out over the past decades. In this presentation a review of this research will be given and the future potential of intensified alkaline electrolysis will be discussed in light of possible GW-scale factories in 2030.

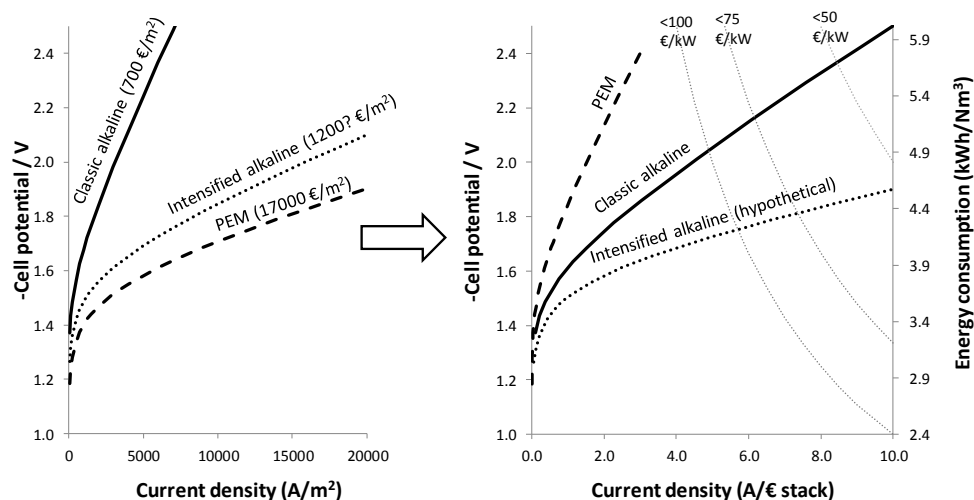


Figure: Current-Voltage curves for classic alkaline, PEM and intensified alkaline technology. The curves in the left graph were obtained from the literature [1,2] and the stack costs from quotations of alkaline and PEM suppliers. The current density in the right graph was obtained by dividing the current density (A/m<sup>2</sup>) by the stack costs (€/m<sup>2</sup>).

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Abstract No 57

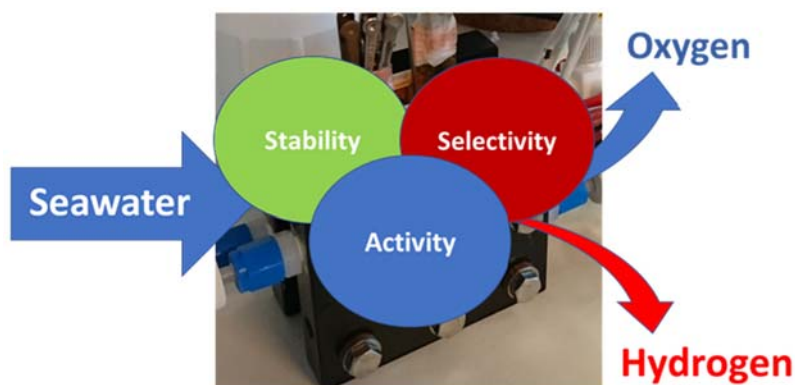
## Analysis of a direct alkaline seawater electrolyzer - material science, catalysis and efficiency

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Unlike conventional water electrolysis of ultrapure water, seawater electrolysis faces fundamental chemical challenges, such as the suppression of highly detrimental halogen chemistries, which has to be ensured by selective catalyst and suitable operating conditions.<sup>[1]</sup> Especially the competing anodic reactions of the chlorine evolution reaction (CER) and the oxygen evolution reaction (OER) are the greatest difficulty for this technology.

In the present study, we analyze a working alkaline seawater electrolyzer from the molecular materials chemistry level, to the electrochemical single cell performance, to membrane and cell degradation patterns, all the way to cell-level power performance recovery effects.<sup>[2]</sup> Nanostructured NiFe layered double hydroxide and Pt nanoparticles were selected as catalysts for the anode and cathode, respectively. The seawater electrolyzer was tested successfully for 100 h at maximum current densities of 200 mA cm<sup>-2</sup> at 1.6 V employing surrogate seawater and compared to fresh water feeds. In-situ X-ray absorption spectroscopy (XAS) revealed high structural stability of the anodic NiFe-LDH catalyst material at all conditions. Rotating disc electrode measurements towards the hydrogen evolution reaction (HER) and OER, scanning electrode microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) verified the catalytic stability and indicate the anion exchange membrane as bottleneck for future devices. Detected anodic oxygen faradaic efficiencies of up to 88 % and calculated voltage efficiencies of 79 % compete with established electrolyzer technologies and confirm the practical viability of full seawater electrolyzer systems towards large-scale seawater electrolysis installations. Finally, we address the challenges for a future direct seawater electrolyzer device and estimate in combination with a fuel cell the suitability as freshwater production technology.



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Abstract No 58 (INVITED)

## **Water electrolysis for hydrogen production – Repairing Breaches to achieve High Efficiency, High Durability and Low Cost**

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Hydrogen is largely considered the best means by which to store energy coming from renewable and intermittent power sources such as wind and solar. With the growing capacity of out-spread renewable energy sources surpassing the gigawatt range, a storage system of equal magnitude is required, such as the production of electrolytic hydrogen by water electrolysis. Despite of more than 100 years of experience in constructing electrolyzer cells, stacks, and innumerable electrolyzer-plants installed all over the world; today only a few demonstration systems are used for energy storage. And the cost of electrical energy has always remained as an uncomfortable barrier, with electrolytic hydrogen costs not being able to compete with the costs for the production of hydrogen by conventional steam reforming of fossil fuels. Nevertheless, today, increased interest can be observed to further develop alkaline, PEM, and solid oxide water electrolysis technology. Over the past 10 years, new companies and projects have appeared, with new leaders being consequently established in this growing “niche”. In this presentation I will show our latest R&D approaches; aiming to increase efficiency, improve durability, and reduce the investment/operating costs of electrolyzers to allow large market penetration of these systems. We believe that only through cutting-edge R&D and significant advancements; we will be able to ultimately establish hydrogen as a commodity in the energy storage market, and contribute to our so much desired environmentally friendly hydrogen economy.

Abstract No 59 (INVITED)

## **The oxygen evolution on perovskites in alkaline media: with or without carbon?**

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Transition metal (Co, Ni, Mn, etc) oxides with a perovskite structure have attracted considerable attention as promising oxygen evolution reaction (OER) catalysts for applications in anion exchange membrane and alkaline electrolyte water electrolyzers. Perovskite structure accommodates various cation substitutions thus allowing to widely vary physico-chemical properties and electrocatalytic activity of the ensuing oxides. Although electrochemical instability of carbon materials in the potential interval of the OER is widely recognized, they are systematically applied as conductive binders when perovskite oxides are used as OER catalysts in alkaline media [1]. Indeed, several studies have evidenced that the OER on perovskite oxides can be significantly improved by either mixing them with or supporting them on carbon materials.

In this presentation we will address several issues related to the carbon utilization during water electrolysis in alkaline media. We will first show how to apply the rotating ring-disc electrode as a quantitative tool for separating the OER and corrosion currents [2]. We will then discuss how the OER activity of Co/Mn perovskite oxides depends on the addition and on the type of carbon materials and whether carbon conductive binders could be replaced by metal nanoparticles [3]. We will then discuss potential and time dependence of the concurrent OER and carbon corrosion on carbon materials and oxide/carbon compositions and speculate on their possible interplay.

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## Contamination Effects in Polymer Electrolyte Water Electrolyzers

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If polymer electrolyte water electrolysis (PEWE) is to be deployed to produce hydrogen for energy storage purposes in the grid and to serve as a key element in power-to-X scenarios, the potential challenges related to the component durability need to be understood and addressed. Maintaining a high stack voltage efficiency is key to a competitive end-cost of H<sub>2</sub>, as the main contribution is projected to come from the cost of electricity<sup>1</sup>.

Since water impurities are an important factor determining electrolyzer reliability<sup>3</sup>, we have designed *in-situ* Fe<sup>3+</sup> doping experiments to simulate the accelerated reversible contamination of the CCM. A detailed overpotential analysis was done to determine the effect on the ohmic, kinetic and mass-transport overpotential. In addition, electrochemical impedance spectroscopy (EIS) coupled with sub-second *operando* neutron imaging was done to determine the relative cross-sectional position of the contaminant in the CCM during the different operating conditions, and its effect on the overpotentials. The cationic contaminant was found to reside in the cathode catalyst layer at elevated cell potential, thereby increasing its ionic resistance and pH. The area resistance of the membrane and the catalyst layers was calculated from the EIS measurements and used to estimate the fraction of the ionomer occupied by Fe-ions. Subsequently, the contribution of pH change in the catalyst layer ionomer to the kinetic overpotential was calculated. Finally, a novel *operando* regeneration method is proposed to extract the ionic contaminant from the CCM and prolong the reliability of a PEWE cell<sup>4</sup>.

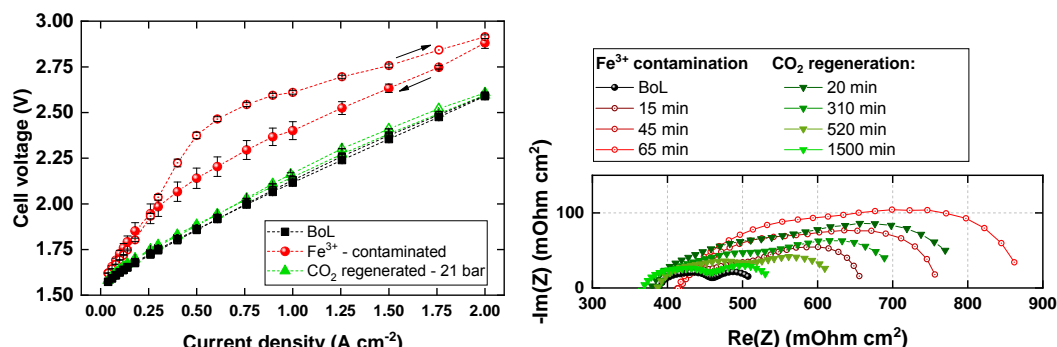


Figure 1. Left: Polarization curves of a N117-based PEWE cell at 40°C, at the beginning of life (■), fed with Fe<sup>3+</sup> solution (●), and post-regeneration with 21 bar CO<sub>2</sub> in the cathode recirculation loop (▲). Right: EIS at 1 A cm<sup>-2</sup> from 10 kHz → 300 mHz during the contamination and regeneration process.

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## Porous transport electrodes for PEM water electrolysis: improved performance via studying materials interfaces

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The use of several mg/cm<sup>2</sup> platinum group metals to catalyse the oxygen evolution reaction (OER) in proton-exchange-membrane water electrolysis (PEMWE) is still one obstacle for its large scale industrial implementation.<sup>1</sup> A promising approach to reduce the noble metal loading are alternative designs of the membrane-electrode assembly (MEA). For PEM fuel cells tailoring the interface between membrane and electrode for a better adhesion and improved water management has been widely investigated.<sup>2</sup> The symmetric design of PEM fuel cells with carbon substrates and Pt/C catalyst at both electrodes allows a flexible assembly of all components and therefore a high degree of design freedom e. g. for integrating additional interlayers. Either catalyst-coated-membranes (CCM) pressed against carbon gas diffusion layers (GDL) can be used or the PEM can be sandwiched between two gas diffusion electrodes (GDE) where the Pt/C catalyst is directly deposited on the GDL. In PEMWE however carbon would corrode at the anode and is therefore substituted by titanium.<sup>3</sup> So far no free standing porous transport electrodes (PTE) for the OER in PEMWE as pendant to GDEs are commercially available which basically limits the PEMWE cell design to the CCM setup. In this work PTEs prepared via spray-coating IrO<sub>2</sub> on porous Titanium substrates will be presented. A study for a detailed understanding of the influence of changing the electrode interfaces on the polarization behaviour (Fig. 1) is shown and general challenges when manufacturing PTEs are elucidated. Performances better than commercially available CCMs motivate further development efforts to optimize PTEs as a promising alternative to standard CCMs.

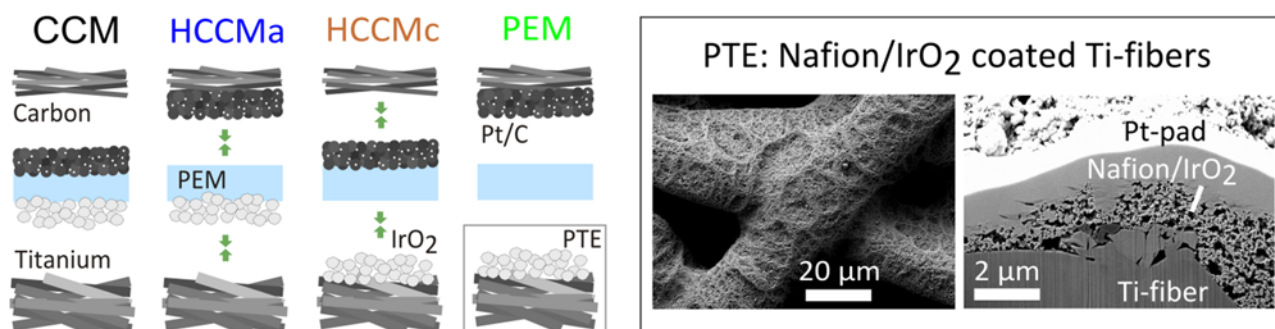


Figure 11: Porous transport electrodes (PTE) allow to vary the electric and protonic interfaces of a state-of-the-art catalyst coated membrane (CCM) via half CCMs with only the anode (HCCMa) or the cathode (HCCMc) deposited on the proton exchange membrane (PEM) towards a free standing PEM. Focused-ion-beam SEM images help to study the microstructure of e.g. fiber PTEs.

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## The Role of Interface Properties and Polymer Electrolyte Water Electrolysis Performance

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Efficiency is a key parameter for techno-economical relevance of polymer electrolyte water electrolysis (PEWE) in energy applications. Primarily the three governing overpotentials associated to kinetics, electrical transport and mass transport determine the efficiency of PEWEs. Porous transport layers simultaneously providing electrical, thermal and fluid transport between bipolar plates and catalyst layers, are one of the key components. A wide spectrum of different PTL classes based on foams, particles and fibres PTLs have been characterized in literature<sup>1, 2</sup>. Differences in performances and in particular mass transport losses were attributed to PTL bulk properties whereas the interface characteristics of PTL surface and catalyst coated membrane have not been considered due to difficult accessibility and missing methodology of interface property analysis.

In this work, the missing link between PTL surface properties and its impact on the PEWE performance is investigated. PTL surface and PLT/CL interface properties are quantified using 3D X-ray tomographic microscopy of *post operando* and *ex-situ* analysis of PTLs and catalyst coated membranes (CCM). Figure 1 gives an example of catalyst layer deformation obtained from this method. Surface properties were determined for a systematic matrix of Ti fibre based PTLs and compared to a state of the art particle sinter material Sika T10 and structural parameters are correlated to overpotential losses. Polarization curves with detailed overpotential analysis and electrochemical impedance spectroscopy were performed. It was found that the PTL surface properties rather than PTL bulk properties have an impact on all overpotentials and the correlations were quantified. Kinetic and ohmic losses are correlated to the microscopic interface contact area (ICA) between CL and PTL. Analysing the differences in kinetic activation showed that the mass transport overpotential is not affected by PTL bulk structures for pore sizes above a certain threshold value. More than two third of the mass transport overpotential occur in the catalyst layer, acting as a so-called local resistance<sup>3</sup> as previously reported in in the field of polymer electrolyte fuel cells.

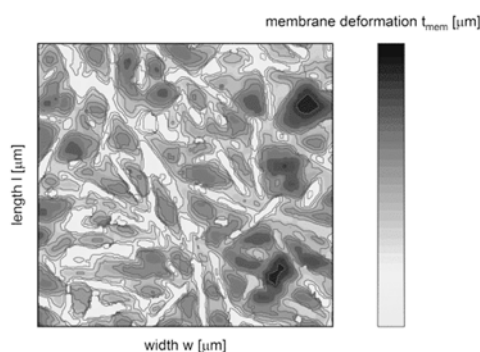


Figure 12. 2D surface plot of post operando catalyst layer deformation. Grey scale value represents membrane deformation.

[1] M. Suermann, K. Takanohashi, A. Lamibrac, T. J. Schmidt and F. N. Büchi, *Journal of The Electrochemical Society*, 2017, **164**, F973.

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Abstract No 63

## High resolution and sub-second Neutron imaging of porous transport layers of proton exchange membrane water electrolyser

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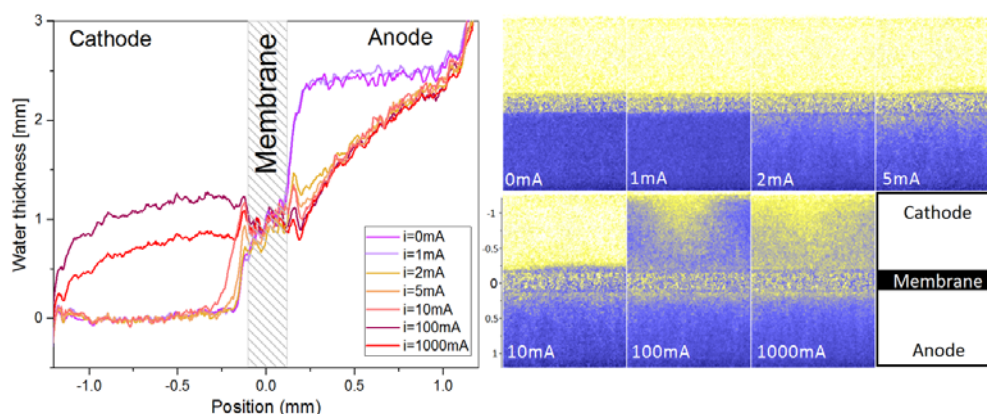
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Proton exchange membrane water electrolysis (PEMWE) is a relatively mature technology, which until recently was used exclusively in areas where the benefit of high gas quality is outweighing the investment and operation costs, like in research or space applications. Due to advances in material sciences, the technology becomes more and more attractive as a mean of storing energy on a wide scale. Especially when coupled with intermittently generating sources. Before the market penetration will be possible however, it is still necessary to improve the efficiency and to reduce the capex and opex. Innovative porous transport layers (PTLs) are made out of sintered titanium, which design optimization and the potential cost cuts are yet to be determined. The present study focuses on fluid dynamics within porous transport layers and the PTL relevance in the overall design of PEMWE.

Characterising mass transport loss origins in PEMWE, is one of the challenges that have to be overcome in order to make another step towards a fundamental understanding and efficiency improvement of PEMWE. Suermann et al. [1] determined the influence of pressure and current density on mass transport losses by employing electrochemical impedance spectroscopy (EIS) and potential-current curve breakdown. Neutron radiography enabled us to take a closer look at the water saturation of in porous transport layers (PTL) during the cell operation in the configuration that separates the anode and the cathode signals from each other and compare the results to previously performed electrochemical experiments [1]. Measurements were done in two modes: one prioritizing high resolution for steady-state condition measurements. Another mode focused on transient measurements to determine the dynamics of gas pathway establishment and re-imbibition with water upon start up and shutdown due to which exposure time was reduced at the cost of spatial resolution.



**Figure 13.** GKN T10 porous transport layer water saturation profiles at current densities ranging from tafel to mass transport governed regions at ambient conditions.

[1] M. Suermann, K. Takanohashi, A. Almibrac, T.J. Schmidt and F. Buchi, *J. Electrochem. Soc.* **164** (2017), F973-F980.

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## Physical vapour deposited electrocatalysts for electrolysis – an overview

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Electrolysis reactions have ‘witnessed’ numerous electrocatalyst developmental iterations, with nanoparticles making up the brunt of researched and developed electrocatalysts. These electrolysis processes include, amongst others, water electrolysis (acidic and alkaline), sulphur dioxide (SO<sub>2</sub>) depolarised electrolysis, and carbon dioxide (CO<sub>2</sub>) electrolysis. A common drawback of these electrolysis processes, however, is the kinetic and cost limitations associated with the employed electrocatalysts that impact negatively on the large-scale performance viability of these technologies. Contrary to conventional electrocatalyst preparation methods, physical vapour deposition (PVD), in conjunction with combinatorial high-throughput screening, offers (i) accurate synthesis of mono, bi- or poly-metallic and/or oxide thin films on varying substrates, and (ii) accelerated characterisation of multiple electrocatalyst compositions with the aim of reaction dependent optimisation of electrocatalyst structure-property relationships. Ultimately, active electrocatalyst compositions of interest can be identified in a fraction of the time compared to when a time-consuming serial approach is followed. Of late thin metal films, produced by means of PVD, have been investigated for their activity (and durability) towards the above-mentioned electrolysis reactions.

An overview of PVD for electrocatalyst research and development for different electrolysis reactions will be discussed. This will include, amongst others, investigating Pt<sub>x</sub>Pd<sub>y</sub>Al<sub>z</sub> for aqueous SO<sub>2</sub> electro-oxidation (Figure 1A)<sup>1</sup>, Pt<sub>x</sub>Ni<sub>y</sub>Al<sub>z</sub> for alkaline oxygen evolution (Figure 1B), and Au<sub>x</sub>Ag<sub>y</sub>Zn<sub>z</sub> for CO<sub>2</sub> electro-reduction (Figure 1C)<sup>2</sup>. For SO<sub>2</sub> electro-oxidation it was ascertained that Pt<sub>40</sub>Pd<sub>57</sub>Al<sub>3</sub> exhibited a much higher mass-specific current density (mA.mg Pt<sup>-1</sup>) of 396.73 compared to 108.11 for pure Pt. For the alkaline oxygen evolution reaction some ternary combinations, for example Pt<sub>9</sub>Ni<sub>56</sub>Al<sub>35</sub> and Pt<sub>15</sub>Ni<sub>55</sub>Al<sub>30</sub>, were found to outperform the single and binary combinations. In the case of CO<sub>2</sub> electro-reduction, two ternary electrocatalysts (Au<sub>16</sub>Ag<sub>10</sub>Zn<sub>74</sub> and Au<sub>32</sub>Ag<sub>51</sub>Zn<sub>17</sub>) exhibited higher selectivity and lower overpotentials compared to the respective pure metals. For these three electrolytic reactions, it was found that ternary co-deposited combinations outperform their individual deposited counterparts pointing to an interesting synergistic effect between the co-deposited metals.

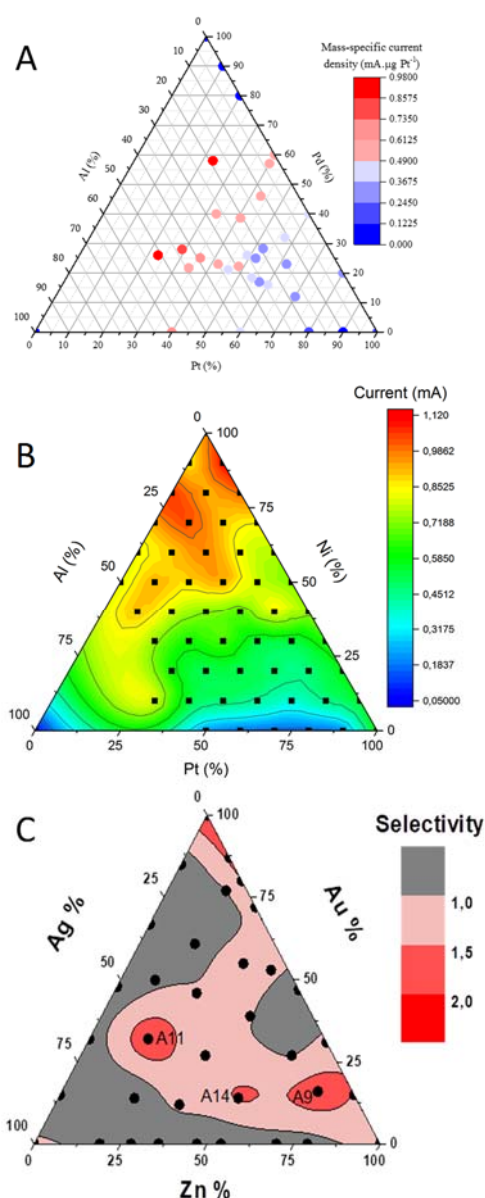


Figure 1 A: Mass-specific current of Pt<sub>x</sub>Pd<sub>y</sub>Al<sub>z</sub> for SO<sub>2</sub> electro-oxidation<sup>1</sup>, B: current of Pt<sub>x</sub>Ni<sub>y</sub>Al<sub>z</sub> for the OER, and C: selectivity of Au<sub>x</sub>Ag<sub>y</sub>Zn<sub>z</sub> for CO<sub>2</sub> electrolysis<sup>2</sup>

- [1] A. Falch, V. A. Badets, C. Labrugère and R. J. Kriek, *Electrocatalysis*, 2016, **7**, 376–390.
- [2] V. Lates, A. Falch and R. J. Kriek, *Electrocatalysis*, 2015, **6**, 308–314.

Abstract No 65

## **Zirfon Perl: Advancing the H2 industry with superior electrolysis membranes**

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'Zirfon Perl' is Agfa's separator membrane for alkaline water electrolysis and is rapidly growing to become the preferred choice for system developers and research institutes as a replacement for PPS cloth or asbestos-based membranes. While the latter have long been considered the standard separator in alkaline electrolyzers, their strong association with health hazards drove already over 55 countries worldwide to ban them from usage. Instead, 'Zirfon Perl' consists of an open mesh polyphenylene sulphide fabric which is symmetrically coated with a mixture of a polymer and zirconium oxide.

ZIRFON is also selected by many because it boosts the overall performance of electrolysis systems in terms of output and operational efficiencies. Thanks to the reinforcement with fabric and the material's low shrinkage property ZIRFON PERL can post durability of 10+ years of real-life operation.

The reinforcement in combination with a relaxation process step during its production, results in a thermally stabilized membrane, even up to temperatures of 110deg. The addition of ZrO<sub>2</sub> to the matrix makes it a highly wettable-hydrophilic membrane, which delivers a unique ionic conductivity. A precise control of the gas pressure resistance (bubble point) at both sides of the membrane gives low HTO and OTH - even at high current densities.

All these synergistic properties deliver a unique membrane that allows systems to perform at exceptional low voltages – high purities, even at high current densities and result to large benefits on the OPEX of electrolyser systems.



## **A New Class of *Bubble-Free* Water Electrolyzer that is Intrinsically Highly Efficient**

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Highly efficient electrochemical splitting of water into hydrogen and oxygen constitutes the most critical capability needed for the development of a future hydrogen economy based on renewable energy. In this work we demonstrate that direct conversion of water into hydrogen and oxygen gas, without the intermediacy of bubble formation, may notably decrease the energy required. We report the performance of “bubble-free” alkaline electrolyzers with electrodes comprising of a Gortex gas diffusion layer coated with high-performing water-splitting catalysts. At 80 °C ( $E^{\circ}_{\text{cell}}$  1.18 V), the best electrolyzer exhibited an onset potential of only 1.28 V, with a total activation overpotential of 0.09 V. It needed a mere 1.31 V to endothermically generate 10 mA/cm<sup>2</sup> over 1 h at 80 °C. A capillary action (6.3 bar) by the hydrophobic, porous Gortex appears to extract newly-formed gases that coalesce on the PTFE surfaces of the electrode before they nucleate bubbles. In so doing, the energy penalties arising from bubble formation appear to be avoided. A comparison with the best conventional and commercial “bubbled” water electrolyzers, suggests that bubble formation and release decreases the energy efficiency of those systems by  $\geq 11.9\%$  (LHV). To the best of our knowledge, the above cell constitutes the most intrinsically efficient water electrolyzer yet reported when the effect of impedance is stripped out.

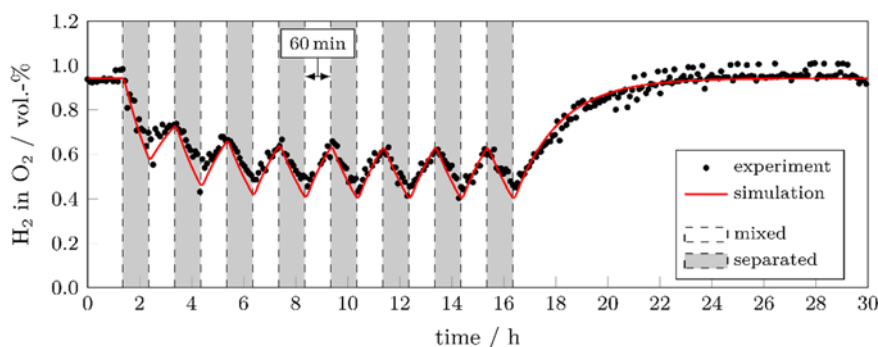
## Dynamic operation strategies and design criteria for alkaline water electrolyzers powered by renewable energies

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To utilize higher amounts of fluctuating and intermittent renewable energies for hydrogen production, precise knowledge of the dynamic behavior of alkaline water electrolyzers is essential. Conventional systems are usually limited to 10 – 40 % of their nominal load. Below this operating range, the gas quality is significantly reduced by contamination with the respective by-product [1]. Two aspects generally cause this effect. Both the diffusion of dissolved product gases through the separator into the other half-cell and the combination of the electrolyte cycles saturated with hydrogen and oxygen result in a reduction of gas quality in the partial load range. An increasing system pressure leads to a higher solubility of the product gases in the electrolyte. Thus, the gas quality decreases also [2]. As a consequence, the fluctuating operation of the process required by regenerative energy sources can cause a safety-related shutdown of the plant at about 2 vol.-% hydrogen in oxygen [1].

In this contribution a dynamic process model of a pressurized alkaline water electrolyzer is utilized, which extends an existing approach [3]. For model validation, experimentally determined gas purities are used. With an adjustment of process variables, the product gas quality can be enhanced. Within a simulation study, dynamic operation concepts are developed and design criteria are identified to handle fluctuating energy profiles more efficiently. Since the product gas contamination is much higher with combined electrolyte cycles, application of a separated mode is favorable. On the other hand, this leads to drifting electrolyte concentrations due to the production and consumption of water in the half-cell reactions. When leaving the optimal conductivity range of the used lye, the process efficiency decreases. Therefore, an optimization problem has to be solved. Selected results with a constant switching time between modes are shown in Figure 14.



**Figure 14:** Anodic gas impurity during switching between flow modes at 5 bar, 60 °C, 0.35 L min<sup>-1</sup> and 30 wt.-% KOH.

Starting with an anodic gas contamination of slightly below 1 vol.-% in mixed mode at the given conditions, the gas impurity can be almost halved by alternated switching. Since the dynamic behavior of an electrolyzer depends on the overall system design, various criteria can be identified to prevent problems under critical operation conditions. Thus, dynamic process simulation offers many possibilities to develop and operate efficient alkaline water electrolyzers powered by renewable energies.

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## Polysulfone-polyvinylpyrrolidone blend membranes in alkaline electrolysis

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Homogeneous and dense polymer blend membranes based on poly(arylene ether sulfone) (PSU) and poly(vinylpyrrolidone) (PVP), as illustrated in Figure 1, have been explored as an ion-solvating polymer matrix for alkaline electrolysis. The prepared membranes are free from vulnerable ion-exchange functionalities, but mediate ionic conductivity by means of substantial electrolyte uptake. Transparent and homogeneous membranes were prepared in the entire composition range from 0-90 wt% PVP. The mechanical properties of the membranes deteriorate with increasing PVP content, but remain sufficient for processing and handling, and thin (50-250  $\mu\text{m}$ ) mechanically robust membranes are achieved at 75 wt% PVP. Compositions with 75 and 90 wt% PVP show electrolyte uptakes above 45 wt% and ionic conductivities above  $70 \text{ mS cm}^{-1}$  in 20 wt% KOH (aq) at room temperature. One week *ex-situ* stability tests in 20 wt% KOH (aq) at  $60^\circ\text{C}$  indicate only minor PVP washout, showing that the water soluble PVP is successfully immobilized by the inert PSU matrix. Single cell electrolyser tests in 20 wt% KOH (aq) at  $60^\circ\text{C}$  show no changes in ohmic resistance over the course of one week at  $500 \text{ mA cm}^{-2}$ , and the membranes display no signs of degradation upon disassembly. Measurements of the PVP content after cell tests found a minor decrease in PVP content, most notable for the thinnest membranes, indicating a possible partial washout near the surface. The cells using PSU-PVP blend membranes showed superior performance compared to Zirfon.

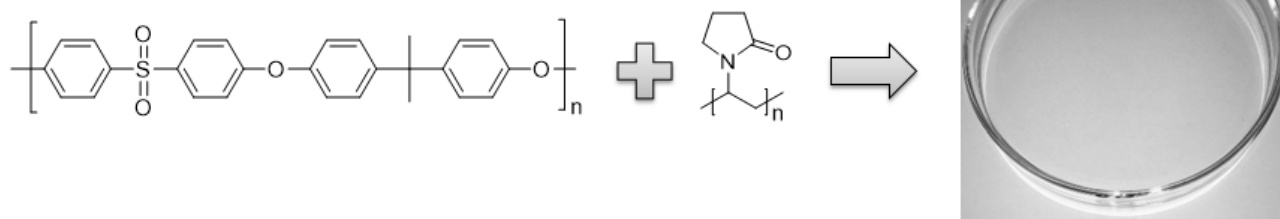


Figure 15 Homogeneous and transparent membrane from blends of poly(arylene ether sulfone) (PSU) and poly(vinylpyrrolidone) (PVP)

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## **Performance of alkaline water electrolyzers following dynamic operation patterns: validation of grid services testing protocols**

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The Low temperature water electrolyzers are able to work under highly dynamic conditions which allow to participate in Electricity Grid Services. To fulfil DSO (Distribution System Operator) and TSO (Transmission System Operator)'s requirements the technical capabilities at stack and system level must be evaluated for each case of study. The grid service requirements may be different depending on the country as legislation and electricity system structure and configuration differ. The lack of standardized testing protocols for provision of grid services is a challenge.

Within QualyGridS project (FCH JU No Grant 735485) a set of standardized testing protocols to qualify alkaline (AWE) and proton exchange membrane (PEM) technology electrolyzers to participate in Electricity Grid Services have been defined. A variety of European regions and countries have been considered. In this work state of the art alkaline water electrolyzers are tested under several standardized testing protocols. The AWE technology at system level is validated for its application to grid services provision.

Results are expected to give valuable information about system and system components behaviour under dynamic conditions, how and to what extent BoP components influence the stack performance under such situations and contribute to review and update existing relevant KPIs (Key Performance Indicators).

## Operating results of PEMEL, AEL and SOEC systems

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Three commercially available stand-alone electrolyser systems are tested, covering the significant technologies alkaline water electrolysis, proton exchange membrane water electrolysis, and high temperature steam electrolysis using solid oxide electrolysis cells. The hydrogen outputs of the systems are in the range between 5 to 10 Nm<sup>3</sup>/h hydrogen at delivery pressures between 10 and 35 bar. Each system comprises its own water purification, hydrogen purification, cooling units and safety systems.

Goal of the comparative study is to evaluate the systems regarding reaction time, stack stability, durability of BoP and maintenance effort under dynamic operation and grid supporting operation. Therefore different load profiles have been defined, simulating the shift between stationary and dynamic application. In this paper the results of the on-going test procedure up to this date are presented and discussed.

The following diagram Fig. 2 shows the behaviour of different temperatures, cell voltage and current during a load change from 100% to 25% of the SOEC system. The depicted development of the load serves as an example of the test procedure for the dynamic operation. Multiple cycles of this load change are conducted over the course of several days to simulate high dynamic demand of hydrogen.

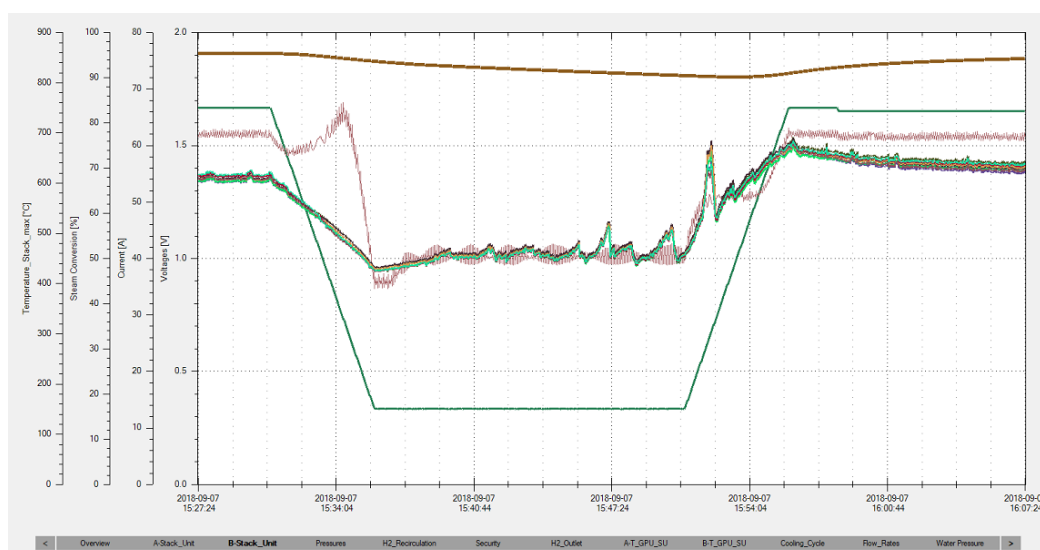


Fig. 2: Dynamic profile from minimum load to maximum load of the SOEC system

The most recent results show that there are problems concerning the stability of the plants. The weak spots are mostly malfunctioning periphery, such as sensors, water pumps, solenoid-valves and cooling aggregates. As relevant parts of the hydrogen-production or the safety measures these errors caused several weeks of downtime. Additionally, signs of degradation were found in one system after around 1000 h of operation, while another electrolysis module needed to be exchanged due to failure of the cells and release of the electrolyte.

The work has been funded by the Federal Ministry of Education and Research (03EK3038B).

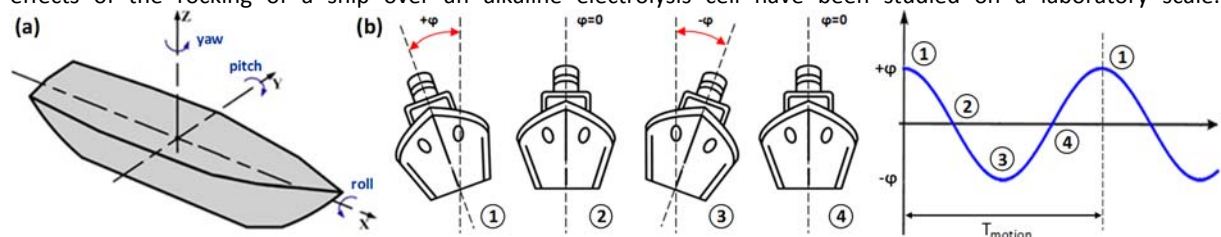
## The effect of the ship motion on the performance of alkaline water electrolysis

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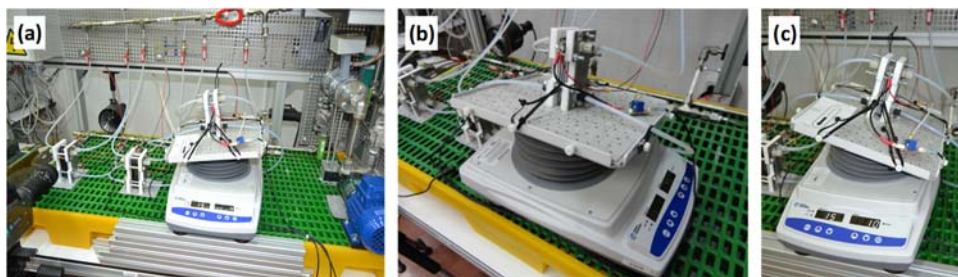
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The use of off-shore renewable energies, such as ocean waves and wind, can be an adequate strategy to produce H<sub>2</sub> using water electrolysis, particularly on ships and marine platforms. However, the marine environment is very harsh in regard to dynamic loads, due to the constant movement of a ship in all directions (Fig. 1). In the present work [1], the effects of the rocking of a ship over an alkaline electrolysis cell have been studied on a laboratory scale.



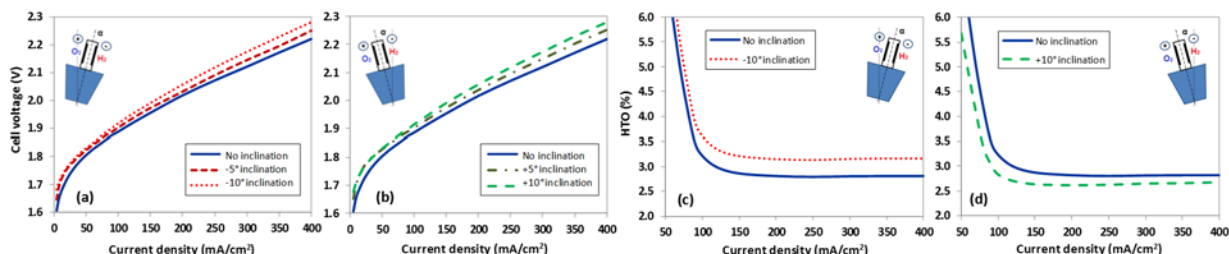
**Fig. 1.** Ship motions [2]: a)degrees of freedom of a vessel; b)tilting rotation of a ship about its longitudinal axis (port-starboard)

In order to evaluate the influence of the movements applied to the electrolysis cell, a rotator waver has been used (Fig. 2a) to emulate the different angles of inclination ( $\varphi$ ) and speeds of oscillation ( $T_{\text{motion}}$ ) of the ship. In addition, with the aim of determining the most suitable orientation on board the ship, the tests have been carried out with different orientations for the cell: bow-stern (Fig. 2b) and port-starboard (Fig. 2c). The polarization curve and the purity of the gases produced (hydrogen-to-oxygen, HTO) have been measured for each movement shown in Fig. 1b.



**Figure 2.** Experimental setup: a) rotator waver; b) pitch motion (bow-stern cell position); c) roll motion (port-starboard cell position)

The results show that the cell works with greater efficiency if it is kept completely horizontal, since any inclination increases the potential required to produce hydrogen (Fig. 3a and Fig. 3b). Regarding the purity of the gases, when the cathode is located below the anode during the tilt of electrolysis cell (Fig. 3c), the impurities increase considerably. So, in order to minimize these effects, the electrolysis system should be oriented with the electrodes towards bow-stern, since the pitch movements of a ship are less pronounced and frequent than the rolling movements.



**Figure 3.** Experimental results in the roll motion at different inclinations: a) polarization curve (cathode below the anode); b) polarization curve (anode below the cathode); c) HTO (cathode below the anode); d) HTO (anode below the anode)

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## Improved mesoporous oxygen electrode for Solid Oxide Electrolyser Cells application

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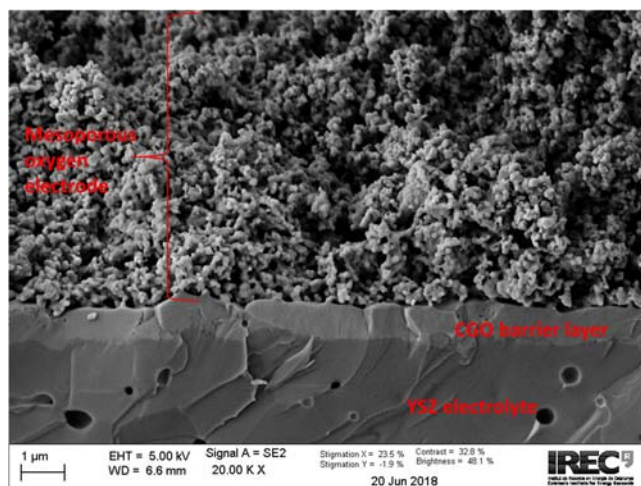
The use of nanocomposite oxygen electrodes in Solid Oxide Electrolyser Cell (SOEC) ensures both electrical and ionic conductivity while maximize the active area and the density of triple phase boundaries (TPB) where the oxygen evolution reaction takes place.

Mesoporous materials based on gadolinium doped ceria (CGO) have demonstrated to be good candidates as backbone materials for oxygen electrode functional layers. This type of backbone provides ultrahigh surface areas (BET area up to 120 m<sup>2</sup>/g), large pore volumes and a very homogeneous phase distribution within the ionic and electronic infiltrated active phase, which is, in the case of the present study, the La<sub>0.6</sub>Sr<sub>0.4</sub>Co<sub>0.2</sub>Fe<sub>0.8</sub>O<sub>3-δ</sub> (LSCF) perovskite.<sup>1</sup>

One of the particular features of described ceramic mesoporous is their thermal stability up to 1000°C, that offers optimum conditions for SOEC operation<sup>2</sup>. However this thermal stability makes difficult the attachment between the electrolyte and oxygen electrode, forcing the use of sintering aids to lower the processing temperature during the fabrication of the device.<sup>2,3</sup>

The mesoporous CGO powders are produced following a nano-casting method based on the infiltration of the commercial silica hard template (KIT6) by the CGO precursors.

The present work presents an optimised route that allows the selective removal of the remaining SiO<sub>2</sub> from the mesoporous template and ensures the electrode attachment by the use of Co as sintering aid. Combining described treatments the proposed functional layer of the oxygen electrode presents high stability and high activity on electrolysis and coelectrolysis mode. These devices were electrochemically characterized with EIS and IV measurements and compared with cells with regular mesoporous oxygen electrode, revealing improved performances.



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Abstract No 105

## HieffPEM – a game changing High efficiency PEM electrolyser for hydrogen production

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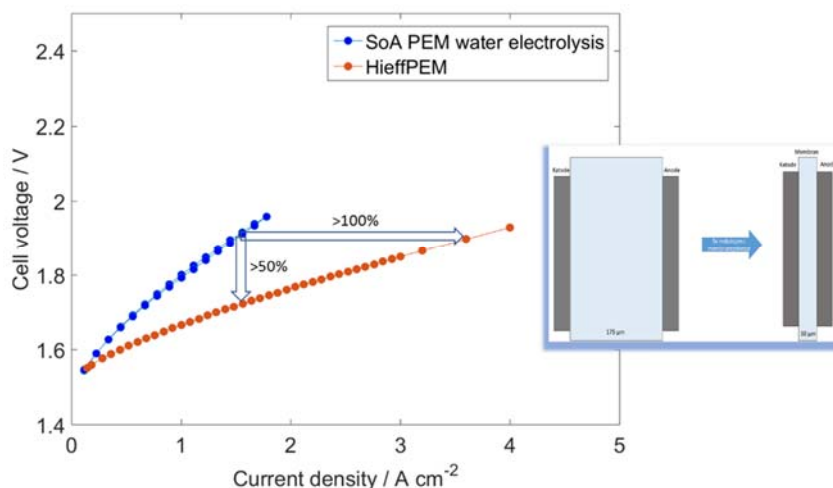
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The yearly production of hydrogen is over 70 million ton with a market price of around \$118 billion. However, most of this hydrogen is still produced through natural gas reforming which is later used in many established industry processes, e.g. refineries and synthetic fertilizers. On the other hand, emission free hydrogen may also be produced from renewable energy sources, e.g. wind and solar. Therefore, renewable hydrogen is emerging as one of the best alternatives for emission free transport, industry, and as an excess renewable energy storage alternative. Unfortunately, the cost of electrolytic hydrogen is still higher (almost double) compared to produce hydrogen from fossil fuels.

Researchers from SINTEF have recently demonstrated a technical breakthrough at lab scale in order to decrease the PEM electrolyser efficiency losses by more than 50% compared to state-of-the-art electrolyser technology. The SINTEF HieffPEM technology results in more than 100% increase in hydrogen production capacity, or 50% cost reduction. This breakthrough may potentially make hydrogen production from electrolysis competitive against large scale hydrogen production from fossil fuels.

The HieffPEM concept consists of reducing all ohmic losses of the electrolyser stack, including membrane ionic resistance and electrical resistances of the porous transport layers (PTLs) and bipolar plates (BPPs). In order to achieve a low membrane resistance, a thin PEM membrane is employed. Normally, employing thin PEM membranes in electrolyzers may result in unsafe electrolyser operation, as a thin membrane may have hydrogen permeation rates from cathode to anode close to the explosion limit. In contrast to state-of-the-art PEM electrolyzers, operating with DI water at the anode, the HieffPEM electrolyser operates with humidified air and therefore diluting any hydrogen that may have crossed over to a safe operating level.

In this paper, the HieffPEM electrolyser will be electrochemically characterized at different operating conditions, cathode back pressure, temperature, humidified air flow rates, including the used electrochemical impedance spectroscopy. In addition, the hydrogen quality has been analysed and preliminary durability data will be reported. Finally, other benefits of using thin membranes in PEM electrolyser technologies will be discussed, e.g. cost reduction potentials.





## Degradation Phenomena in Solid Oxide Electrolysis Cell Oxygen Electrodes

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In order to ensure a viable lifetime of solid oxide electrolysis cells (SOECs), it is important to understand degradation mechanisms. Mechanisms such as delamination and oxygen bubble/pore formation have been observed at the oxygen electrode during operation of SOECs. Although the degradation has been shown to be strongly dependent on current density, the effects of other operating parameters are generally not known. Here we report results on life tests done on both symmetric cells and full cells with Yttria-Stabilized Zirconia (YSZ) electrolytes with Gd-doped Ceria (GDC) barrier layers, with various oxygen electrode materials including  $(\text{La}_{0.6}\text{Sr}_{0.4})(\text{Co}_{0.2}\text{Fe}_{0.8})\text{O}_3$  (LSCF) and two materials recently shown to provide superior performance and stability:  $\text{Sr}(\text{Ti}_{0.3}\text{Fe}_{0.7})\text{O}_3$  (STF) and  $\text{Sr}(\text{Ti}_{0.3}\text{Fe}_{0.63}\text{Co}_{0.07})\text{O}_3$  (STFC). The effects of operating temperature, current density, and electrode material are described. For example, cells with LSCF or STF electrodes operated at  $1 \text{ A/cm}^2$  are stable at high temperature, but for temperature  $< 650 \text{ }^\circ\text{C}$ , the cells fail due to delamination at the GDC/electrode interface. Delamination is generally observed for conditions (temperature, current density, and electrode type) that give rise to an oxygen electrode overpotential  $\eta \gtrsim 0.2 \text{ V}$ .

A quantitative model for charge carrier transport across the electrolyte, based on that reported by Chatzichristodoulou *et al* [1], is used to predict the electrical and oxygen potentials across the YSZ/GDC electrolyte and thereby provide insights into degradation. The distribution of oxygen partial pressure is determined for different electrode materials and different operating modes. The oxygen pressure at the electrolyte/electrode interface is largely determined by the electrode overpotential. The oxygen pressures reached at the interface under a given condition are identical for symmetrical and full cells with identical oxygen electrodes. Thus, to the extent that failure occurs at the electrolyte/electrode interface, symmetrical and full cells should give the same failure condition, as observed experimentally. The maximum oxygen pressure is observed at the interface between YSZ and the GDC barrier layer; nonetheless, the model predicts failure at the GDC/electrode interface because of the relatively low fracture toughness of the perovskite electrode material. The model-predicted failure conditions, where the oxygen pressure exceeds the perovskite fracture toughness, are in quantitative agreement with the experimentally observed current density and temperature (and critical overpotential value) where failure is observed.

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## In Search of the best Catalyst Layer – the Necessity of Application based Designs

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Optimizing the catalyst layer design is a very important topic for PEM water electrolysis to improve cell performance and reliability as well as to reduce costs and degradation phenomena. It is obvious that it is almost impossible to optimize all aspects, since there exist several trade-offs. The priority of the different aspects (performance, costs, ...) is changing with the application and consequently the design of catalyst layers has to match to the specific application.

Development of catalyst layers for specific applications make it necessary to understand the complex correlations between the catalyst layer design and different mechanisms e.g. electrochemical activity, mass and charge transport. To understand these interplays in more detail a steady state, one dimensional model (sandwich coordinate – through plane) was developed. The fundamental model includes the description of electric/protonic potentials, temperature, gas/liquid pressures and dissolved gas concentrations. The left hand side of Fig. 1 shows schematically important local profiles of state values as proton and electron potential a), temperature b) and dissolved gas concentrations c).

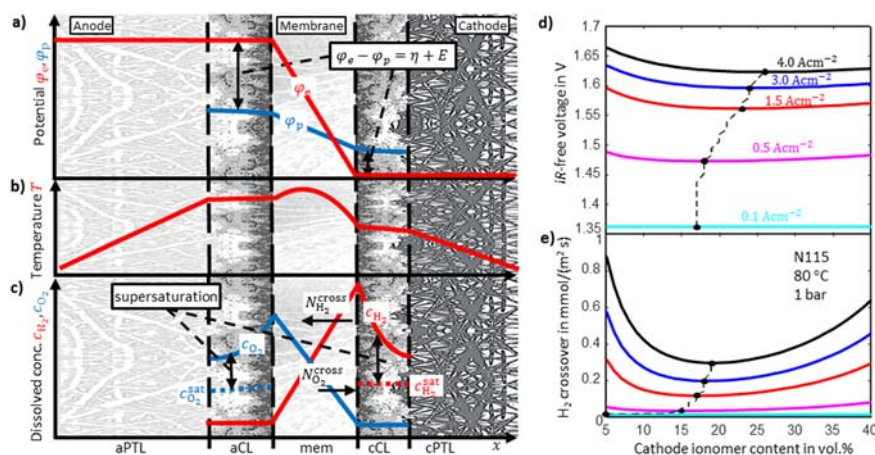


Figure 16. Simulation results: a-c) local profiles and d-e) cathode ionomer variation.

The model results show that there are different optima in e.g. ionomer content, catalyst loading and pore diameter. This is caused by the trade-offs between ohmic losses, mass transport losses and kinetic losses. The right hand side of Fig. 1 shows the  $iR$ -free voltage d) and hydrogen crossover flux e) as a function of the cathode ionomer content for five current densities. At low ionomer contents the proton transport in the catalyst layer is hindered and at high ionomer contents the mass transport is negatively affected, which consequently increases the mass transport losses. The optimal ionomer content is represented by the dashed lines. The analysis suggests to use small ionomer contents at lower current densities but to increase the ionomer content at higher current densities. Which ionomer content should be applied? It corresponds to the current density range at which the electrolyzer should be operated. Hence, as previously stated, the optimal catalyst layer design is a function of the application.

This contribution highlights the necessity of application based catalyst design for different catalyst structure parameters as a function of different operating conditions, e.g. current density, pressure and temperature. The model results are validated by use of published experimental results [1,2,3].

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Abstract No 108

## Comparative study of anion exchange membranes for low cost water electrolysis

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Currently, the majority of hydrogen is produced from the steam reforming of natural gas, hydrocarbons and alcohols [1]. But water electrolysis has gained much attention, since it allows the relatively quick and convenient production of pure hydrogen only from water and electricity directly “on site” [2].

Over the past few years, anion exchange membranes (AEMs) have been developed for electrochemical system applications. They have been mainly used for alkaline fuel cells and in the significant lesser degree have been tested for water electrolysis [3]. AEM water electrolysis owns all traditional alkaline water electrolysis advantages such as the usage of inexpensive non-noble metal-based catalysts and stack materials (in contrast to the acidic polymer electrolyte membrane electrolysis) and easy handling due to the relatively low temperatures. Moreover, AEM electrolysis allows to overcome some considerable disadvantages of traditional alkaline electrolysis like the usage of high concentrated and still low conductive alkaline solutions as it could operate at relatively low KOH concentration (1-2 mole L<sup>-1</sup> KOH solution or even pure water) without the electrolyte carbonization process taking place.

Although AEM-based water electrolysis technology is promising, not much research has been carried out to date and the information on the electrolysis performance, hydrogen yield and purity is currently not enough [4] especially in the case of thin membranes.

At presented work several commercially available membranes were studied in AEM electrolysis process using the nickel-based electrolysis cell with 5 cm<sup>2</sup> working surface area and non-noble metal catalysts. The different membranes resistance and its dependence on the KOH concentration and operating temperature were investigated. Great attention was paid to the purity of generated hydrogen (intrinsic purity, without any auxiliary used) and the hydrogen yield. Also, the capability of tested membranes to operate only with pure water feeding without KOH was demonstrated and evaluated.

### Acknowledgements.

Authors thankfully acknowledge HySA Infrastructure at NWU for providing laboratory facilities as well.

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Abstract No 109

## Magneli phase $\text{Ti}_4\text{O}_7$ supported Ir-based catalysts for polymer electrolyte membrane electrolysis

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Among the various methods developed for hydrogen production from renewable energy sources, water electrolysis is considered as one of the most practical and flexible technology. In particular, polymer electrolyte membrane (PEM) electrolysis offers a sustainable solution to produce hydrogen, which may be coupled with intermittent renewable energy sources (e.g. wind and solar) or nuclear. However, the penetration of PEM water electrolysis technology into large-scale industrial applications is slow due to the high costs of its components including noble metals-based electrocatalysts (for instance up to 2 – 4 mg cm<sup>-2</sup> of Ir loading is used currently at anode) [1].

Ir (IrO<sub>2</sub>) is the most commonly used oxygen evolution reaction (OER) catalyst employed in PEM water electrolyzers, but it is an expensive material and depositing it on a support material with high surface area could be an effective way to increase its utilization. High anodic potential and acidic environment tends to corrode carbon material during oxygen evolution process [2] and the common carbon-based supports cannot be used, as well as the choice of materials that can be used as supports are limited.

Magneli phase titanium oxides own high electrical conductivity combined with excellent corrosion resistance and, thus, are promising candidates for OER catalyst support. Moreover, its hypo-d-electron character implies realization of electronic interactions with the hyper-d-electron Ir, leading to synergy and improved efficiency [3].

In presented study Ir-based electrocatalysts supported on conductive titanium oxides were synthesized by different methods and evaluated. Their activity and stability in oxygen evolution reaction were evaluated by electrochemical approaches like cyclic voltammetry, rotating disk electrode voltammetry etc. Moreover, titanium oxides supported catalysts were tested as anodes in PEM water electrolyzer cell. The data related to the structure and morphology of the catalysts obtained using TEM, SEM, EDS etc. are provided as well.

### Acknowledgements.

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## Tomographic Study of Electrodes of PEM Water Electrolysers - Gaining Information about Performance, Degradation and Process Control

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One of the biggest challenges of renewable energies are their spatial and temporal fluctuations. Therefore, their success is inevitably depending on energy storage solutions. PEM water electrolysis (PEMWE) could be a promising candidate for an electrochemical energy storage solution, especially due to its flexibility and rapid system response [1]. Performance, durability and costs have to be improved for this technology to allow itself to prevail in the market [2].

Knowing the microstructure of a PEMWE, particularly the anode, is essential to get a deeper understanding of the performance and degradation behaviour. The electrode morphology has a strong influence on the cell efficiency by co-determining the transport properties of the species and the accessibility of the catalysts [3]. There are only a handful of publications on tomographic studies of PEMWE, e.g. by using X-ray [4] or focussed ion beam - secondary electron microscopy tomography (FIB-SEMT) [5].

In this study, the microstructure of different catalyst layers was determined via FIB-SEMT. The ionomer loading of the catalyst layers varied between samples. The data was acquired with serial sections. The cutting and imaging was accomplished by a Zeiss Crossbeam 540 with GEMINI II. The investigated volume was of the order of 100  $\mu\text{m}^3$ . After reconstruction, the segmentation was performed with an advanced image processing algorithm. Analysing this data yielded information about structural parameters, such as pore size distribution, porosity, tortuosity, permeability and diffusion coefficients [5].

Additionally, the microstructural analysis is combined with electrochemical measurements (polarization measurements and electrochemical impedance spectroscopy). Thereby microstructure is related to the performance of the cell.

The medium- or long-term aims are: (i) to get a better understanding of degradation behaviour of PEMWE by comparison of the microstructure of pristine and aged electrodes, (ii) to use the obtained information about the structure properties for the manufacturing process of the PEMWE. This should create a loop between the investigation of the structure, the determined properties and the overall process of manufacturing.

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## Creep and stress relaxation in PEM electrolysis cells

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Polymer Electrolyte Membrane (PEM) water electrolysis is a technology that makes use of renewably-produced electricity to generate hydrogen without incurring CO<sub>2</sub> emissions in the process. An important factor that influences the performance of the PEM electrolysis cell is the clamping pressure. As the performance is reduced by up to 30% when operating in the unfavorable pressure range, strenuous effort should be made to always keep the pressure at an optimum (2-3 MPa) [1].

However, several studies show that the Nafion® membrane exhibits viscoelastic behavior [2-4]. This behavior is especially pronounced under electrolysis conditions, where the membrane is completely saturated with water and the temperature is 80 °C. The viscoelastic behavior could lead to stress relaxation or creep in the cell, depending on whether the pressure or displacement is kept constant. The influence of creep and stress relaxation on cell performance remains unclear. However, it is expected that stress relaxation in the Nafion® membrane and thus the catalyst-coated membrane (CCM) leads to a loss of clamping pressure, and thus to poorer performance due to higher contact resistances. The aim of this work is to address this issue by investigating the influence of creep and stress relaxation on the performance of PEM electrolysis cells.

The experiments were performed with a special test cell, in which the pressure was directly applied by a stamp on the active cell area. In the creep tests, the pressure in the cell was kept constant over time and the change in displacement detected. The creep of the CCM was found for cells with porous transport layers (PTLs) having a pore size of >200 µm. Through creep, the pores of the PTL became blocked, increasing mass transport losses.

In the stress relaxation tests, the cold and dry cell was loaded with an initial pressure and the displacement was kept constant. In order to operate in the optimum pressure range, the initial pressure may only be 0.5 MPa. Additional pressure was introduced through temperature expansion and membrane swelling. For cell designs with a poor water supply, the pressure also depends on the current density, as the water content of the CCM is reduced at high current densities. Higher current densities thus lead to a reduced thickness of the CCM. It was also found that in an electrolysis cell, pressure over time is reduced as a result of stress relaxation. Stress relaxation in the cell is primarily attributed to the CCM. Creep and the reduction of pressure due to stress relaxation were found to be part of the degradation effects of the cell over time. The proportion of mechanical degradation due to stress relaxation to total degradation across 75 hours was over 17.4%. Thus, for the first time a mechanical effect is described as being part of the cell's degradation.

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## High Temperature Electro-oxidation of Glycerol and Product Characterization

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This work involves the long term electrooxidation of glycerol in a custom autoclave setup. Experiments are usually performed over approximately 48 hours. The custom autoclave setup was adapted from work done by Thomas Holm [1,2]. Within the autoclave a typical three electrode cell is built, wherein all glass pieces have been replaced with alkaline stable counterparts. This is largely done with Teflon; however a PDMBI membrane provided by the Holdcroft group is used to separate the working and reference electrode volumes from the bulk volume at the counter electrode to avoid contamination at the reference, and to keep the oxidation volume small.

Analysis of the oxidized products is performed via HPLC using an Aminex HPX-87H column. Using these techniques we have been able to show that the temperature at which electrooxidation occurs significantly alters the selectivity of products, as well as causing some new products to form. Calibration curves have been made up for each of these products using known standards, and so we are able to quantitatively determine how much of each product is formed.

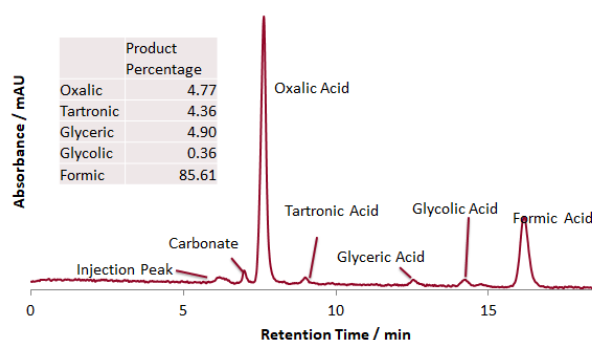


Figure 1: HPLC Spectrum of 48 hr Oxidation of Glycerol on Nickel at 20 °C

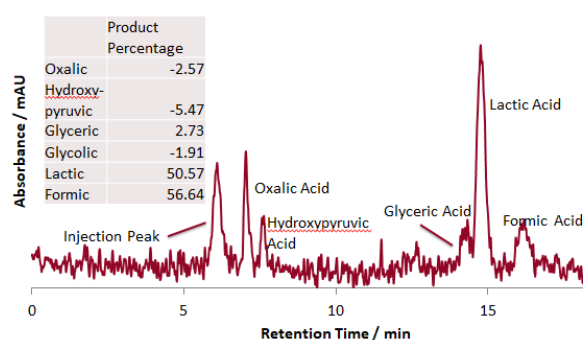


Figure 2: HPLC Spectrum of 48 hr Oxidation of Glycerol on Nickel at 140 °C

The formation of a polymer product has been discovered when performing oxidation at 140°C. The results of this are shown in figure 2, where little enough of some of our other products remain that their calibration curves are extrapolated to the point of being useless. This polymer product is easily reproducible; however it is difficult to obtain in large quantities so far. It also doesn't appear to dissolve in many of the common solvents typical to polymer characterization processes. Ongoing work involves the optimization of the polymer synthesis, collection of the polymer in bulk, and polymer characterization.

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## High Temperature Co-electrolysis Systems for Power-to-Gas Applications

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High temperature co-electrolysis for power-to-gas applications is a promising concept for enabling greater penetration of intermittent renewables and improving grid flexibility and energy management capabilities. Solid oxide electrolysis cells benefit from the potential for high efficiency and the ability to co-electrolyze water and carbon dioxide simultaneously to produce syngas. While conventional SOECs operate at high temperatures (>750°C), operating at intermediate temperature (600°C) and elevated pressure (10-20 bar) with both water and carbon dioxide promotes the exothermic methanation reaction, which can provide the necessary heat for steam reduction. This simplifies balance-of-plant design and facilitates easier integration with natural gas pipeline infrastructure relative to steam/hydrogen electrolytic processes. Natural gas is a desirable product due to the extensive existing infrastructure for its transport and utilization. Recent studies have shown high performance for intermediate temperature solid oxide cells fabricated with strontium- and magnesium-doped lanthanum gallate (LSGM) electrolytes.

This study proposes an SOEC system integrated with a natural gas pipeline and carbon capture and storage (CCS) infrastructure for carbon utilization and synthetic natural gas production as shown in Figure 1. Carbon dioxide and water are co-electrolyzed in the stack to produce methane and hydrogen, which can be injected directly into a natural gas pipeline or further refined into a purer stream of methane. We explore performance and cost of a 50 MWe SOEC system for four system design concepts for methane purification: (1) with no methane purification, (2) with a post-stack methanation reactor, (3) with a post-stack hydrogen permeable membrane, and (4) with both a methanation reactor and a hydrogen membrane. Results indicate that without external methanation, the system can produce a synthetic gas mixture of roughly 42% hydrogen and 55% methane at a cost of 72 \$/MWh (20 \$/GJ or 21.1 \$/MMBTU) and a lower-heating value efficiency of 82.7%. Adding both a methanation reactor and a membrane increases methane purity to 92%, while only increasing cost by about 5% to 77 \$/MWh (21.4 \$/GJ or 22.6 \$/MMBTU). Over half of this cost is associated with the cost of carbon dioxide and electricity. The proposed system is compared with low-temperature electrolysis pathways to synthetic natural gas of similar plant power capacity.

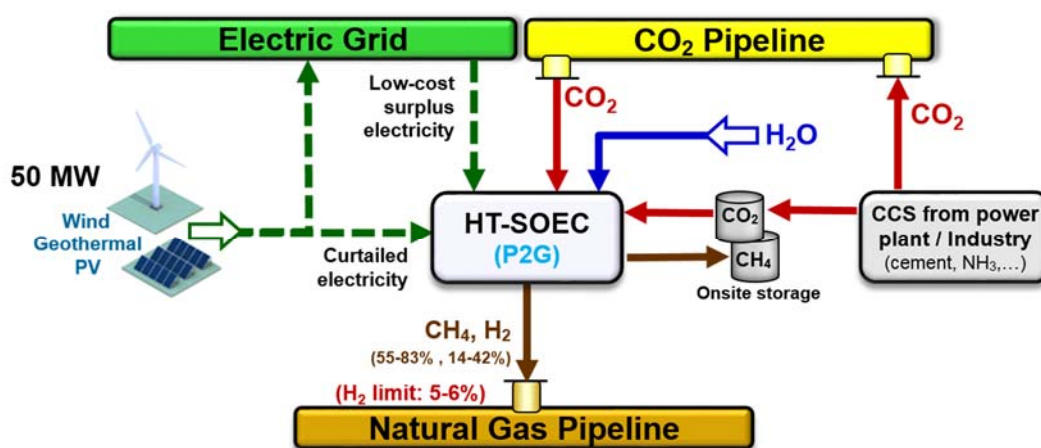


Figure 1. HT-SOEC System for SNG Production

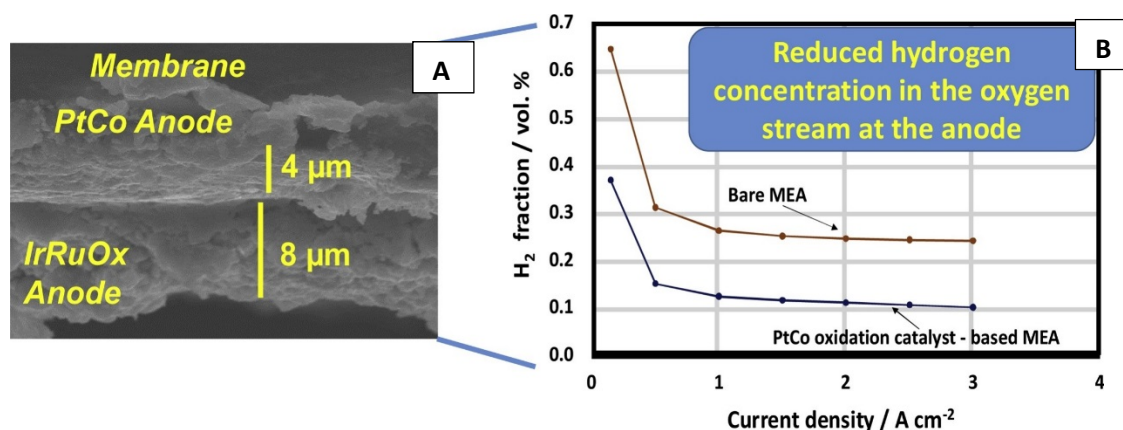
## Reduction of H<sub>2</sub> concentration in the anode stream of a pressurised water electrolyser based on a thin polymer electrolyte membrane

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Various Pt-based materials (unsupported Pt, PtRu, PtCo) were investigated as catalysts for recombining hydrogen and oxygen back into water. The recombination performance correlated well with the surface Pt metallic state. Alloying cobalt to platinum was observed to produce an electron transfer favouring the occurrence of a large fraction of the Pt metallic state on the catalyst surface. Unsupported PtCo showed both excellent recombination performance and dynamic behaviour. In a packed bed catalytic reactor, when hydrogen was fed at 4% vol. in the oxygen stream (flammability limit), 99.5% of the total H content was immediately converted to water in the presence of PtCo thus avoiding safety issues. The PtCo catalyst was thus integrated in the anode of the membrane-electrode assembly of a polymer electrolyte membrane electrolysis cell. This catalyst showed good capability to reduce the concentration of hydrogen in the oxygen stream under differential pressure operation (1–20 bar), in the presence of a thin (90 μm) Aquivion® membrane. The modified system showed lower hydrogen concentration in the oxygen flow than electrolysis cells based on state-of-the-art thick polymer electrolyte membranes and allowed to expand the minimum current density load down to 0.15 A cm<sup>2</sup>. This was mainly due to the electrochemical oxidation of permeated H to protons that were transported back to the cathode. The electrolysis cell equipped with a dual layer PtCo/IrRuOx oxidation catalyst achieved a high operating current density (3 A cm<sup>-2</sup>) as requested to decrease the system capital costs, under high efficiency conditions (about 77% efficiency at 55 °C and 20 bar). Moreover, the electrolysis system showed reduced probability to reach the flammability limit under both high differential pressure (20 bar) and partial load operation (5%), as needed to properly address grid-balancing service.



**Figure 1.** Scanning electron micrographs of the MEAs (a) with the PtCo hydrogen electro-oxidation catalysts (b) Hydrogen fraction in the outlet anode stream at various current densities for the bare MEA and the MEA containing the PtCo oxidation catalyst at 1 bar at a constant water inlet temperature of 55 °C.

### Acknowledgements

CNR-ITAE authors acknowledge the financial support from the NEPTUNE project. This project has received funding from Fuel Cells and Hydrogen 2 Joint Undertaking under grant agreement N 779540. This Joint Undertaking receives support from the European Union's Horizon 2020 research and innovation programme and Hydrogen Europe and Hydrogen Europe Research. The authors also thank Solvay Specialty Polymers for supplying the membrane and the ionomer, ITM Power (UK) for supplying the high-pressure single cell test fixture and Mr. Giuseppe Monforte for the XPS analysis.



Abstract No 115

## **Solid Oxide Cell Performance Improvement Using Auxiliary Non-Thermal Plasma and Fluidic Oscillation**

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The reduction in carbon emissions required to meet the 2°C scenario (2DS) and the resultant necessary decarbonisation of energy generation require an increased focus on renewable energy sources. Solid Oxide Cells (SOCs) are widely seen as a leading technology for future clean power generation and chemicals production, whether operated in fuel cell (SOFC) or electrolysis (SOEC) mode. Renewable electricity can be utilised directly in electrolysis mode to reduce CO<sub>2</sub> and/or H<sub>2</sub>O which can then be further reacted to produce a myriad of hydrocarbon related products. Electrical efficiencies of around 50% can be reached, and this, combined with their high operating temperature, can achieve total system efficiencies of around 80% for power generation [1]. However, SOCs have a number of performance limitations, including activation and concentration polarisation, which lead to over 80% of the cost of co-electrolysis of CO<sub>2</sub> and H<sub>2</sub>O being attributed to electrical input [2].

A number of improvements to cell geometry and composition have been used to improve performance, however increasing efficiency through the use of auxiliary technologies such as plasma and fluidic oscillation has the potential to dramatically reduce the operating cost of the system. Reactions in non-thermal plasmas (NTPs) enable very high temperature chemistry to occur at low bulk gas temperatures [3] and improve the kinetics and efficiency of CO<sub>2</sub> dissociation to form O<sup>2-</sup> ions in a SOC, thereby avoiding activation polarization. Additional increases in performance are seen by using a rapidly oscillating gas flow which minimises concentration polarisation resistance by disrupting boundary layer formation. As part of this work, a bespoke rig was used to combine electrocatalysis, plasma catalysis and fluidic oscillation to create a highly efficient energy conversion device, providing a path for a paradigm shift in the ability to store renewable energy in chemical form.

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## Carbon Based Cathode Materials for Li-S Batteries

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The increased demand for portable electronic devices requires batteries with high energy density, low cost, long lifecycle and safety. Practical energy density of Li-ion battery approaches its theoretical energy density limits. Lithium-sulfur batteries are considered the most promising charging system. Capacity of Li-S battery is five times higher compared to commercial lithium-ion battery. Theoretical capacity of sulfur is 1675 mAh/g and energy density is 2600 Wh/kg. These batteries are nature friendly, nontoxic and inexpensive. There are several problems with practical application due to sulfur behaviour in battery: the insulating characteristic of elementary sulfur, inadequate power supply and large volumetric expansion or shrinkage during charge. Sulfur reacts with lithium during cycling and the results of reaction are lower ( $\text{Li}_2\text{S}$ ,  $\text{Li}_2\text{S}_2$ ) and higher ( $\text{Li}_2\text{S}_4$ ,  $\text{Li}_2\text{S}_6$ ,  $\text{Li}_2\text{S}_8$ ) polysulfides. The higher polysulfides soluble in the organic electrolyte migrate freely between electrodes leading to the “shuttle effect” during the cycle process. “Shuttle effect” causes an irreversible loss of capacity, corrosive process on the negative electrode and a discharge resulting in instability of electrode structure [1], [1].

The solution for insulated characteristic of sulfur is addition of carbon into the cathode material. Sulfur-carbon composites can be produced with carbon black, multi-walled carbon nanotubes, nanostructured carbon or metal-organic frameworks. Conductive carbon polymers are promising materials in lithium-sulfur batteries thanks to their high conductivity and strong absorbing properties to the polysulfides [3].

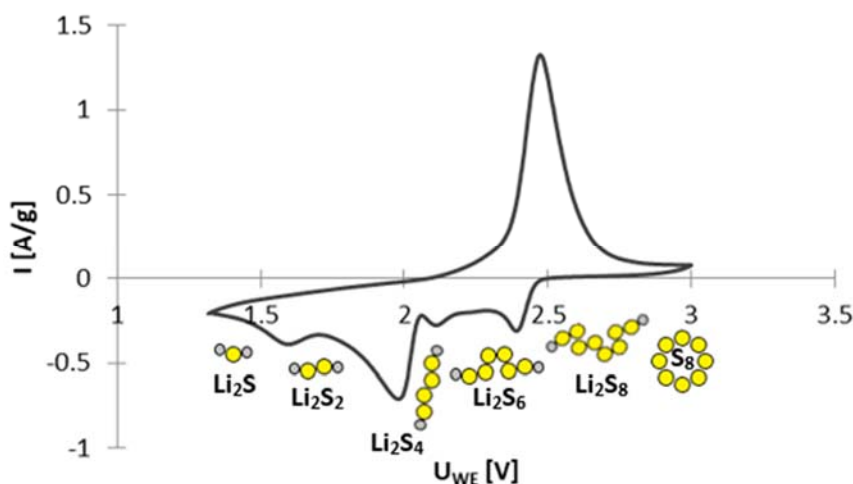


Fig. 3 Cyclic voltammogram of Li-S battery at scan rate  $0.1 \text{ mV s}^{-1}$  between 1.3 V to 3.0 V

### Acknowledgment

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Abstract No 117

## Advancing alkaline electrolysis cell performance by electrode microstructural optimization and high temperature operation

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Electrolytic hydrogen production is gaining commercial ground as a technology that can efficiently and economically link the electricity with the fuels and chemicals branch of the energy system, while offering a means for large-scale and long-term energy storage. Amongst the different electrolysis technologies, alkaline electrolysis (AE) stands out as the most well established for large-scale electrolytic hydrogen production, with commercially available multi-MW units combined in plants of 100s of MW and operated for decades. Besides proven reliability and availability, a key advantage of AE over alternative technologies when it comes to large-scale deployment is the relatively abundant and inexpensive materials it relies on. Nevertheless, AE suffers from relatively poor performance in terms of production rate and efficiency when compared to proton exchange membrane electrolysis (PEME) and solid oxide electrolysis (SOE). This contribution discusses the main issues leading to the inferior AE performance and approaches to overcome them.

One of the main reasons is associated with the sluggish hydrogen evolution reaction (HER) kinetics in alkaline environment [1]. Improvements in HER catalysts, promoting the rate of H<sub>2</sub>O dissociation, have reduced the HER kinetics difference between alkaline and acidic environment. Furthermore, the far lower price of these catalysts (e.g. Ni, Ni<sub>1-x</sub>Mo<sub>x</sub>) compared to Pt, allow for much higher catalyst loadings, which can circumvent this challenge in conjunction with the much higher ionic conductivity of concentrated aqueous KOH as compared to PEME and SOE electrolytes. Taking full advantage of this opportunity requires a careful optimization of the AE electrode microstructure to achieve both a high electrochemically active surface area in close proximity to the separator as well as macro-porosity to enable gas evolution with minimal blocking of the active area. This was attempted here by applying high surface area catalytic coatings of Ni and Ni<sub>1-x</sub>Mo<sub>x</sub> on porous conducting supports with varying macro-pore structure. Electrodes of ca. 12 cm<sup>2</sup> size were tested at 80 °C and ambient pressure under flow of 20 wt.% KOH. Furthermore, a finite element multi-physics simulation model was employed to provide further insight and guidance to the microstructural optimization effort.

Raising the operating temperature offers an additional means to drastically improve performance, as both ionic transport and reaction kinetics are exponentially activated with temperature [2]. The development of a corrosion resistant ceramic separator [3] has enabled a novel concept of alkaline electrolysis cells operating at 200-250 °C and 20-50 bar [4,5], showing pronounced thermal activation, and achieving a current density of up to 3.75 A cm<sup>-2</sup> at a cell voltage of 1.75 V at 200 °C and 20 bar [6]. The feasibility and promise of this concept, as well as the challenges that lie ahead are also discussed.

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## Defining Nafion ionomer roles in alkaline oxygen evolution reaction (OER)

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Electrocatalysing oxygen evolution reactions (OER) are critical for clean-energy storage and conversion techniques, such as water electrolysis. [1-2] OER is not strictly a surface reaction, because it takes place in hydrous electronic double layer (EDL). Currently, the study of EDL microstructure and behavior poses a challenge due to the limitation of in-site characterization tools. Moreover, it is a standard practice for catalyst layer (CL) fabrication to incorporating a small amount of Nafion ionomer as a binder, which also forms steady reaction interfaces. As shown in Figure 1, the co-existence of catalyst, polymer, and liquid electrolyte further complicates the electro-catalysis study in EDL. In this work, we perform an in-depth study of Nafion ionomer functions in both catalyst inks and catalyst layers (CL) for high-efficiency OER in alkaline media. Based on cyclic voltammetry results, an analysis method is developed to characterize pseudocapacitance and EDL charging capacitance independently. This enables accurate quantification of interfacial charge transport behavior and active sites for OER. Zeta potential measurements confirm that Nafion ionomer serves as a stabilizing and binding agent in catalyst-solvent inks. The collective results from standard thin-film rotating disk/ring-disk electrode methods reveal that increasing ionomer content impedes interfacial mass transport and reduces active site utilization. However, as compared with the ionomer-free sample, the addition of Nafion shows improved OER performance, which indicates that Nafion is necessary for continuous OER and efficient catalyst utilization. Therefore, there exists an optimal loading of Nafion to catalyst mass ratio. Our study shows that the optimized ionomer content in the CL is 10-30 wt. % of catalyst loading. Within this range, Nafion, catalyst particles, and electrolyte solution form efficient interaction, resulting in good connectivity of the charge conduction paths without inhibiting the gas diffusion.

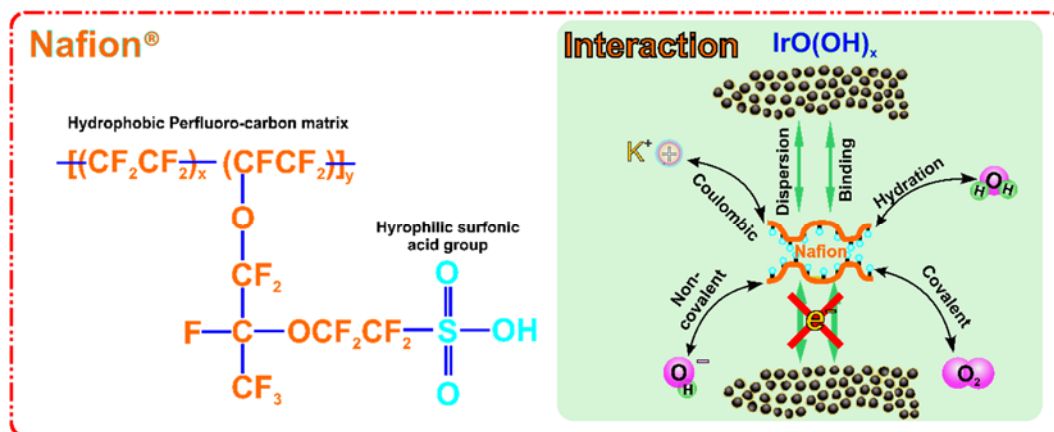


Figure 1. Schematic illustration of Nafion structure and interfacial interactions in alkaline OER

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Abstract No 119

## **Hierarchical nanostructures of NiWO<sub>4</sub> on Ni foam for high-performance urea electro-oxidation and urea assisted low energy electrolytic hydrogen production**

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Urea oxidation reaction (UOR) has been considered as a promising electrochemical reaction for hydrogen production since the theoretical reversible potential of urea electrolysis (0.084 V) is much lower than that of the water electrolysis (1.23 V) [1]. Moreover, urea is solid, non-toxic, non-flammable and stable compound, and hence easy to store and transport. In addition, urea is the largest constituent of urine, which is one of the most abundant wastes on earth. Among the extensively studied fuels in fuel cell systems, urea is the only non-flammable compound. Thus, using UOR for hydrogen generation not only reduces the energy consumption but also has the potential to mitigate the environmental pollution from urea rich wastewater. Cost-effective Ni-based transition metal oxide catalysts remain to be a great challenge for UOR due to their poor electronic conductivity. Tungstate materials possess many advantages such as simple synthesis processes, low cost, low toxicity and stable multifunctional properties [2]. Most importantly tungstate materials have high electronic conductivity than most binary and some ternary metal oxides [3]. In this study, nanostructures of NiWO<sub>4</sub> on Ni foam as a freestanding hierarchical porous electrode is synthesized by hydrothermal process. Ni foam is used as a substrate to grow porous electrode structure for enhancing electrocatalytic surface area and enabling efficient mass transport. The morphology of the electrode structure is investigated by X-ray diffractometer (XRD), Field Emission-Scanning Electron Microscope (FE-SEM) and Transmission electron Microscope (TEM). The hierarchical nanostructures of NiWO<sub>4</sub> on Ni foam is shown to be a highly active, stable, and low cost electrocatalyst for UOR. In addition, the NiWO<sub>4</sub> on Ni foam is also investigated for hydrogen evolution reaction (HER) in alkaline medium to study its potential as a bifunctional catalyst for urea assisted low energy electrolytic hydrogen production. Detail experimental results will be presented at the conference.

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## Ni-based Anodes for Improved Alkaline Membrane Water Electrolysis

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Renewable energy sources such as wind, tidal and solar, are green alternatives to fossil fuels, however they introduce a new problem; an imbalance between energy supply and demand. Water electrolysis is a promising means to store renewable energy as chemical energy in hydrogen. Particularly, anion exchange membrane water electrolysis (AEMWE) has shown promising progress due to its compact cell design and potential to operate using non-noble metal catalysts, thus providing an efficient and cost-effective solution to energy storage. <sup>[1]</sup> A known challenge to water electrolysis is the kinetically unfavorable oxygen evolution half-cell reaction (OER). Many studies have shown that nickel (Ni) in combination with iron (Fe) significantly enhances activity towards OER. <sup>[2]</sup> Furthermore, it has been shown that cerium oxide (CeO<sub>2</sub>) support can have positive effects on catalytic performance. <sup>[3]</sup> This study covers the preliminary evaluation of Ni, Ni<sub>90</sub>Fe<sub>10</sub> (at%) and Ni<sub>90</sub>Fe<sub>10</sub>/CeO<sub>2</sub> (50wt%) spherical nanoparticles (NPs), synthesized by chemical reduction with sodium borohydride, as OER catalysts in AEMWE using commercial membranes. A transmission electron microscopy (TEM) image of the Ni NPs is shown in the accompanying figure. For an applied current density of 2 A cm<sup>-2</sup>, results show that the non-noble metal catalysts achieved overpotentials of 1.85-1.90 V in 1 M KOH at 50°C, which is comparable to the selected iridium-black reference catalyst. In 0.1 M KOH, the Ni<sub>90</sub>Fe<sub>10</sub> catalyst was the only one capable of accommodating the applied current of 2 A cm<sup>-2</sup> without inducing too high of an overpotential. Although the Ni<sub>90</sub>Fe<sub>10</sub>/CeO<sub>2</sub> catalyst shows good kinetics, electrochemical impedance spectroscopy (EIS) results show that further optimization of the support content in the material must be done to minimize cell resistance.

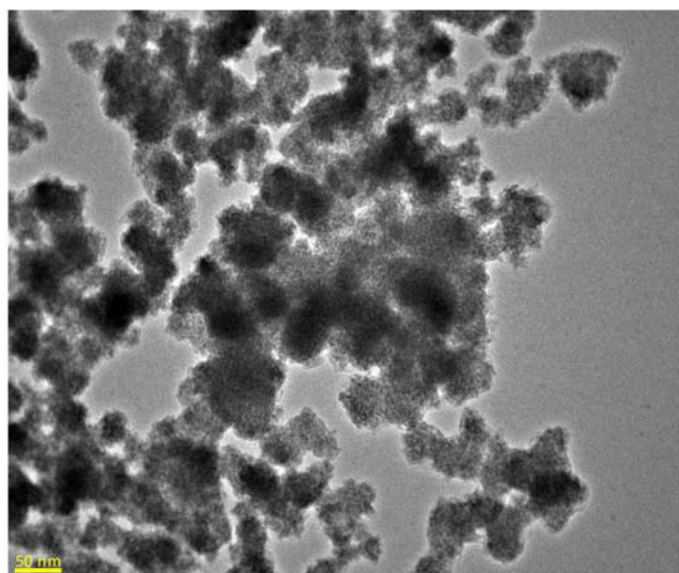


Figure 17: Transmission Electron Microscopy of Spherical Ni Nanoparticles

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Abstract No 121

## Scale-up of a DEFT™ alkaline electrolyser through intensive flow reduction

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Conventional electrolysis systems utilise the flow of electrolytic solution to overcome the overpotentials related to differences in electrolytic concentrations and gas residence times within the cell. Electrolytic flow additionally facilitates the even distribution of heat within an electrolysis system, particularly important during initial start-up [1]. The physical action of electrolytic flow, coupled with low viscosity properties, can have a significant influence on mass transfer (ionic), bubble liberation dynamics, and consequently, the enhancement of the electrolyser performance. The majority of research related to electrolysis systems focuses on alleviating mass transfer limitations for the proton exchange or anion exchange membrane. Improved ionic mass transfer, however, does not always translate into enhanced gas production as the heightened rate of gas formation can hinder effective interface contact between the surrounding electrolytic solution and the electrode surface.

Divergent-Electrode-Flow-Through (DEFT™) membraneless alkaline electrolysis utilises the divergent flow to maintain separation of the produced gaseous constituents, along with providing a mechanism to accelerate the liberation of bubbles from the electrode surface. Sufficient flow must be maintained per unit area of the porous electrode (determined to be  $0.075 \text{ m.s}^{-1}$ ) in order to generate high gas purities (> 99.5 vol%) at high operating current densities (>  $3.5 \text{ A.cm}^{-2}$ ). This was observed to be a consequence of a gaseous meniscus that develops within the electrode gap [2]. In scaling DEFT™ electrolysis from a laboratory concept to a 5 kg H<sub>2</sub> / day demonstration prototype, methods were developed and employed to drastically reduce the required flow velocity, while still maintaining effective gaseous separation to boost overall plant efficiencies. By inhibiting gas formation on the frontal electrode face, gas production can be localised to the interstitial and rear regions of the porous electrode. This resulted in a 75-fold reduction in the required flow velocity, yielding a solution that generates high purity gases (> 99.5 vol%) and estimated system efficiencies greater than 60 HHV%. Effective scale-up of DEFT™ electrolysis is now feasible due to the increase of the previously allowable maximum flow path (< 15 mm). Larger circular electrodes (> 230mm) and flow paths (> 65 mm) with a centralised electrolytic injection configuration produced a practical, simplistic and inexpensive design that allows for elevated production rates utilising DEFT™.

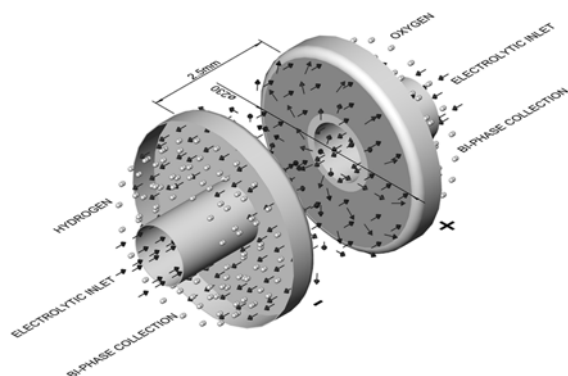


Figure 18: DEFT™ electrolyser ring-shaped electrode pair

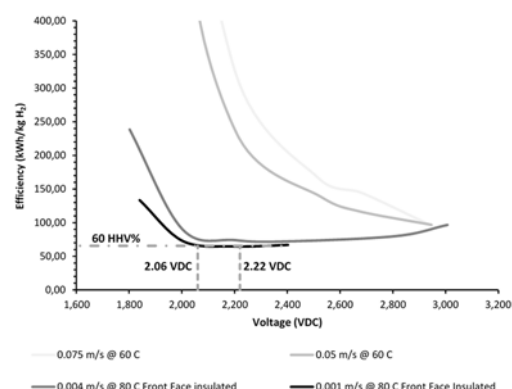


Figure 2: DEFT™ system power consumption versus selected operating cell potential as a function of flow velocity

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## Development and Understanding of Oxygen Evolution Reaction Catalysts and Catalyst Layers for Proton Exchange Membrane Water Electrolyzers

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Electrolysis is rapidly gaining interest in the context of increasing penetration levels of renewable energy sources on electrical grids and the need to consume stranded or curtailed electrons. The resulting renewable hydrogen can then be integrated into ancillary chemical, thermal, transportation or electrical industries in order to substantially decrease CO<sub>2</sub> emissions and increase energy security and grid resilience. Proton exchange membranes water electrolysis (PEMWE) offers several advantages vs. traditional liquid electrolyte systems that use concentrated potassium hydroxide. In water electrolyzers, the overvoltage of the oxygen evolution catalyst is one key efficiency loss, typically contributing over 300 mV of overpotential in PEMWE's. In addition, the catalyst loading is very high, in order to maintain activity throughout operating lifetimes surpassing 50,000 hrs, due to the lack of stability of most catalyst supports in acidic environments at electrolysis potentials. Thus in order for PEMWE technology to make serious inroads in the renewable energy markets the system cost must be decreased and the efficiency increased. One of the key ways to achieve this is decreasing the precious metal loading of the anode (oxygen evolution side of the cell) while simultaneously improving efficiency and maintaining durability. However, as catalyst loading is decreased the performance and durability suffer. In this talk we dissect the driving factors behind this performance and durability loss using component level characterization techniques, model systems, cell level transport modeling, in situ tomography and radiography. Finally, we will present several catalyst and catalyst layer approaches to alleviate these issues.

This talk will focus on the efforts at LBNL through the DOE HydroGEN research consortium on resolving the activity stability drivers on state of the art, iridium oxide based, PEMWE anodes performed in partnership with NEL/Proton OnSite, Tufts University/UC Irvine.

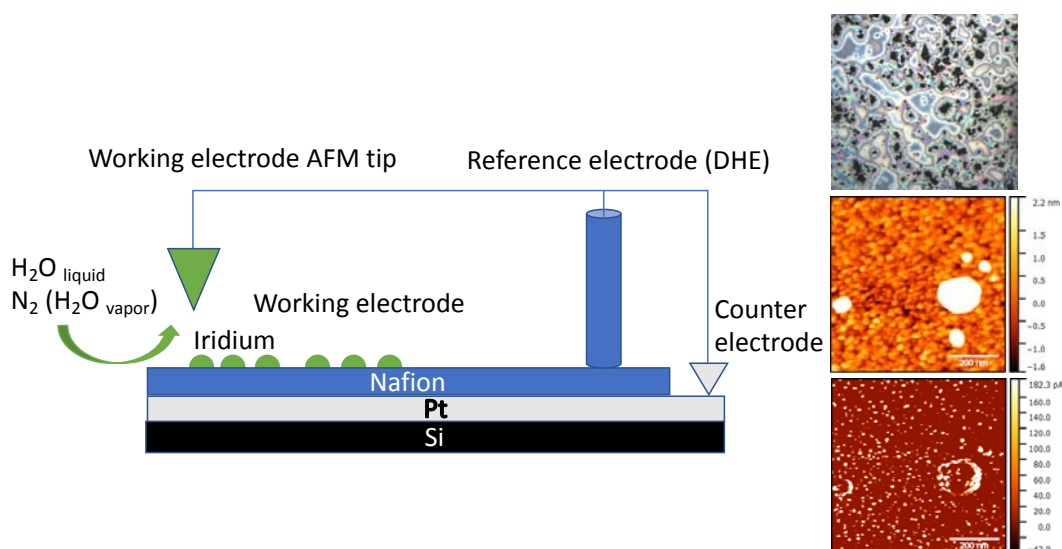


Figure 1. Conductive AFM schematic of model anode catalyst layer half cell (left), optical and conductive images of the ionomer and catalyst phases (right)



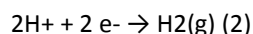
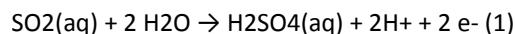
## Advanced PBI based membranes for the SO<sub>2</sub> depolarized electrolysis at high temperature

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To supply of clean and sustainable energy is one of the most important scientific challenges [1]. Reducing the total emissions to the atmosphere and providing the society with green energy is the main goal of renewable energies which are the alternative to the existing carbon-based energy generation. Hydrogen also plays an important role in the green energy economy, in fact the term “Green Hydrogen” [2] refers to a hydrogen produced via renewable sources. Nowadays, 90% of total hydrogen is produced by means of non-renewable sources as coal. As consequence, hydrogen and renewable energies will have a great impact in the future of energy production and storage. The way to combine them is to use the renewable energy sources to produce the previously mentioned “Green Hydrogen” which will act as energy carrier for a later use in fuel cells for example. Water molecule dissociation is the obvious approach for green hydrogen generation. However, direct thermal water splitting needs extremely high temperatures which does not make this option suitable for large hydrogen production [3]. Water electrolysis also needs considerable high electric power due to the high theoretical voltage ( $E^0=1.23$  V). In this scenario, technologies combining a chemical step and a thermal step are becoming a promising alternative. One of the leading thermochemical cycles is the hybrid Westinghouse [4] cycle which combines a thermal step to decompose sulfuric acid and an electrochemical step in which oxidation of SO<sub>2</sub> with water ( $E^0 = 0.158$  V vs SHE) occurs to produce hydrogen. The protons are conducted across a proton exchange membrane (PEM) that acts as a separator to the cathode where they recombine with the electrons to form hydrogen according to equations 1 and 2 [5].



This work is focused on the study of the SO<sub>2</sub> depolarized electrolysis at high temperature (100-200°C) using acid doped PBI membranes as PEM. Three different membranes will be studied for the electrolysis, a commercial PBI membrane, a lab-made PBI membrane and a composite membrane modified with TiO<sub>2</sub>, this method is explained elsewhere [6], as inorganic filler. The tests are performed in a 25 cm<sup>2</sup> SO<sub>2</sub> depolarized electrolysis cell. A novel operating procedure for SO<sub>2</sub> depolarized electrolysis will be employed for this study. SO<sub>2</sub> and H<sub>2</sub>O will be fed both in the gas phase to the anode. Steam will be generated and mixed with the SO<sub>2</sub> stream before being introduced in the anode. Electrodes with a platinum loading of 1 mgPt/cm<sup>2</sup> will be employed in the anode and in the cathode. The method to prepare the electrodes is to spray an ink which is obtained by mixing a solvent with the platinum used as catalyst (40% Pt/Vulcan XC-72). Firstly, the membranes will be characterized according to their acid uptake, acid retention and ion conductivity. Secondly, the effect of different parameters such as temperature, SO<sub>2</sub> flow and the ratio SO<sub>2</sub>/H<sub>2</sub>O molar ratio will be studied. Polarization curves and impedance spectroscopy analysis are carried out to evaluate the performance of the cell and the ohmic and charge transfer resistances. A preliminary stability test will be performed to assess the stability of the different components of the cell (membrane and electrodes).

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Financial support from the Junta de Comunidades de Castilla-La Mancha and the FEDER –EU Program, Project ASEPHAM. Grant number “SBPLY/17/180501/000330” is gratefully acknowledged.



## On activity and stability of NiSn, LaSrCrMnO<sub>3</sub> and GdCeO towards rWGS and carburization for H<sub>2</sub>O/CO<sub>2</sub> co-electrolysis to syngas

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Ni-cermets are the state-of-the-art cathode material for H<sub>2</sub>O/CO<sub>2</sub> co-electrolysis where electrochemical water reduction and heterogeneous reversible Water-Gas-Shift reaction (rWGS) occur whereas electrochemical CO<sub>2</sub> reduction cannot be excluded. Main challenges are related to activity and more especially to stability of Ni-based catalysts [1] in presence of large amount of water vapor and to carbon formation in case of lower steam/carbon ratio, low temperature and high pressure that shifts Boudouard reaction product to solid one. The latter can lead to irreversible destruction of the cathode material (metal dusting).

In this work, alternative catalyst materials like NiSn and LaSrCrMn perovskite were synthesized and tested regarding their activity for reverse water-gas shift reaction (rWGS) in a glass tube reactor under different H<sub>2</sub>:CO<sub>2</sub> mole ratios at 700 and 800 °C and atmospheric pressure, and compared to that of pure Ni. Since Gadolinium-doped Ceria (GDC) is commonly used as ionic conductor in the reaction layer and Cerium is active for reverse Water-Gas-Shift reaction, activity of GDC20 material for rWGS was instigated as well. Gas composition was evaluated at reactor outlet by gas chromatography. First results are shown in Figure1. As expected, highest CO:H<sub>2</sub> ratios for all catalysts were yielded with 1:1 H<sub>2</sub>:CO<sub>2</sub> mixture. Interestingly, LSCM and CGO exhibited comparable activity to that of pure Ni. Influence of temperature on structural changes and sintering behavior of powder material was studied by XRD and SEM/EDX. Since probability of carbon formation increases with increasing pressure values, influence of feed pressure, H<sub>2</sub>:CO<sub>2</sub> gas composition and temperature on catalyst activity for carbon formation was investigated in a pressurized stainless steel tube reactor up to 10 bar. Post-mortem analysis of powders was performed by XRD, SEM/EDX and Raman. While strong carbon formation was observed at Ni in mixed H<sub>2</sub>:CH<sub>4</sub>:CO<sub>2</sub>:CO atmosphere according to thermodynamics calculation shown above, no carbon was detected at other catalysts. First results on co-electrolysis experiments in a button cell will be presented as well.

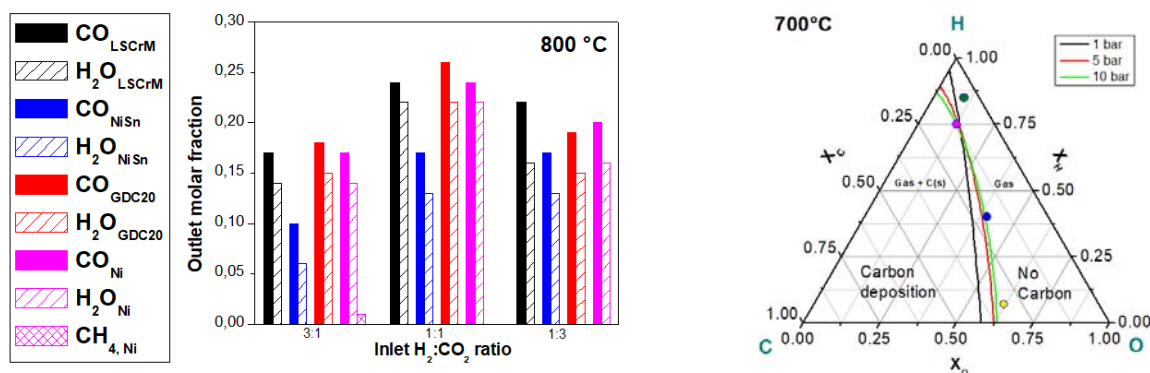


Figure 1: (Left): product composition of rWGS at reactor outlet in function of catalyst nature and H<sub>2</sub>:CO<sub>2</sub> feed ratio at 800°C. (Right) thermodynamic probability of carbon formation at 700°C in function of H<sub>2</sub>O:H<sub>2</sub>:C ratios and for four selected atmospheres represented by dots: black H<sub>2</sub>:CO<sub>2</sub>=1:9, pink H<sub>2</sub>:CO<sub>2</sub>:CO:CH<sub>4</sub>=7:1:1:1, blue H<sub>2</sub>:CO<sub>2</sub>=1:1, yellow H<sub>2</sub>:CO<sub>2</sub>=9:1.

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Abstract No 125

## **TiO<sub>2</sub>-MoO<sub>x</sub> supported Iridium catalyst for the Oxygen Evolution Reaction in acidic electrolyte**

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For the oxygen evolution reaction (OER) in polymer exchange membrane electrolytic cells (PEMEC), large amounts of precious metal group catalysts are required for good cell efficiency [1]. Thus, many researchers have been reported to reduce the catalyst usage amounts without performance degradation [2]. Introduction of supporting materials into the catalyst can solve the problem. Titanium dioxide (TiO<sub>2</sub>) is well-known supporting material which is stable under high electrical potential and low pH conditions [3]. However, its low electronic conductivity interrupts the electron transfer, which makes catalytic reaction be suppressed.

In this presentation, titanium oxide-molybdenum oxide (TiO<sub>2</sub>-MoO<sub>x</sub>) will be reported for supporting materials. The structural morphology of TiO<sub>2</sub>-MoO<sub>x</sub> was characterized by SEM, TEM and XRD. Also those chemical properties was characterized with XPS. To evaluate the catalytic performance and its stability, Iridium particles were loaded on TiO<sub>2</sub>-MoO<sub>x</sub>. Ir/TiO<sub>2</sub>-MoO<sub>x</sub> catalyst shows higher performance and better stability than commercial Iridium black, Ir/C and Ir/TiO<sub>2</sub>. These results would be originated from introduction of Mo into Ir/TiO<sub>2</sub>-MoO<sub>x</sub>. In the presentation, the Mo effects on catalytic activity and stability will be reported. Also, the cell performance will be reported.

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## Copper Phosphide/N-Doped Carbon based Alkaline Water Electrolysis Catalyst for Hydrogen Evolution Reaction

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Water electrolysis technology is one of the attracting technologies with environment friendly power production. In particular, this technology is becoming more and more important as it attempts to combine power-to-gas (P2G) technology with power generation in the form of hydrogen-fuel cells. In the electrolytic water, an oxidation reaction occurs at the anode and a reduction reaction occurs at the cathode, generating oxygen and hydrogen, respectively. Since this reaction requires an overpotential of 1.23 V or more, which is the theoretical water decomposition potential, a catalyst study is being continuously carried out to reduce it. In general, noble metal materials exhibit the highest activity, but non-noble metal catalyst materials are required considering economic efficiency and reserves. Transition metal phosphide is one of the materials attracting attention due to their low price and abundant reserves. Among them, copper phosphide catalysts have excellent HER activity under alkaline conditions, which can lead to additional performance improvements by utilizing N-doped carbon.<sup>1-3</sup> However, previous researches have been complicated synthesis process and toxic gas release in phosphorization process, making industrial application difficult. In order to overcome this problem, this study proposes a simple and one-pot synthesis method of phosphorization and N-doped carbon synthesis. Copper phosphide/N-doped carbon catalyst exhibited better activity (210 mV at 10 mA/cm<sup>2</sup> in 1 M KOH) than the previously studied copper phosphide catalysts. This result is due to the synergy effect with the carbon structure, which appears in the results of XPS and raman spectra.

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Abstract No 127

## **A template-free porous Ni-P for oxygen and hydrogen evolution reaction in an alkaline electrolysis**

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Water electrolysis is an electrolysis reaction of water, and it is a technology that can produce a large amount of hydrogen environmentally friendly. The purposes of water electrolysis are responding to current hydrogen demand and storing electric energy produced from renewable energy such as solar or wind power. In the anode, an oxidation reaction occurs, oxygen is generated, and a reduction reaction occurs in the cathode, generating hydrogen. The theoretical equilibrium potential of the water electrolysis is 1.23 V, but the electrolysis reaction of the actual water requires much higher voltage due to the slow kinetics of oxygen and hydrogen reaction. Therefore, in order to increase hydrogen production efficiency, it is necessary to develop a catalyst having high activity and low cost for OER and HER.

At present, transition metal-based materials such as Ni, Fe, Co are being studied as high efficiency low cost catalysts. Among various type of compounds, transition metal phosphide has some advantages of good stability and high electrical conductivity, and can be synthesized using non-toxic precursors. However, since most nickel phosphide catalysts require several hours to days for synthesis, including high temperature heat treatment processes, the efficiency problem is not solved yet.

In this study, we have developed a method for producing nickel-phosphorus electrodes for high efficiency oxygen and hydrogen evolution reaction in a few seconds by electrodeposition method. In addition, it was confirmed that Ni-P electrode has higher catalytic performance for OER and HER than the precious metal based catalysts (IrO<sub>2</sub>, Pt/C) in the 1M KOH solution, and the durability was also superior to the precious metal based catalysts and Ni electrode in the long-term chronopotentiometry test.

**Keywords:** alkaline water electrolysis, oxygen evolution reaction, hydrogen evolution reaction, nickel, phosphorous, electrocatalyst

## Optimized NiCu Catalyst for Enhanced Hydrogen Evolution Activity in Anion Exchange Membrane Water Electrolysis

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Anion-exchange membranes (AEM) water electrolysis has the potential for combining the advantage of non-precious catalysts of the alkaline process, with the fast power-up/power-down rates and low energy consumption advantages of PEM water electrolysis.<sup>1</sup> We report a new transition metal-based hydrogen evolution electrocatalyst with superior performance as cathodes in AEM water electrolysis. A carbon-supported bimetallic nickel-copper catalyst was prepared by a simple chemical reduction method on the surface of a (KetjenBlack 600 J) carbon support. Detailed structural characterization of catalyst was done using scanning electron microscopy, energy dispersive X-ray spectroscopy, X-ray diffraction, Raman spectroscopy, and X-ray photoelectron spectroscopy. Electrochemical investigation studied the activity of NiCu/ KetjenBlack as a function of the loading of electrocatalyst, type of binder, KOH concentration, and ionomer to carbon ratio. NiCu/ KetjenBlack is optimized to deliver current density of 10 mA-cm<sup>2</sup> at 180 mV overpotential under AEM conditions. The use of an anion-exchange polymer (Fumion FAA-3) as the binder instead of Nafion led to a lower kinetic current density. Single cell testing of AEM water electrolysis has been performed in different concentration of KOH electrolyte. Polarization curves and Electrochemical impedance spectroscopy were measured at different current densities.

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## Electrochemical hydrogen compression for decentralized district applications

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In a future energy system hydrogen plays an important role to store volatile renewable energies. Hydrogen can be used in mobile and stationary power applications as well as in the chemical industry. Especially in fuel cell vehicles a high pressure level of 350 or 700 bar is needed, which cannot be achieved by common PEM electrolyzers which are nowadays limited to a pressure of 30 bar. Thus the hydrogen has to be further compressed which can either be realized by conventional mechanical compressors, ionic compression or by an electrochemical hydrogen compressor.

To investigate the electrochemical compression, a variable EHC test cell with an active area of 50 cm<sup>2</sup> was used. The EHC test cell tested in a self-constructed test bench by which the membrane can be humidified externally at the low pressured anode via a bubbler (AH = anodic humidification) or internally at the high pressured and flooded cathode (CH = cathodic humidification). In Figure 6 it is shown that a Nafion 117 based catalyst coated membrane shows significant lower cell voltages with CH at 40 °C and 100 bar than with AH. A high saturated CCM is needed to achieve high ionic conductivities and low ohmic resistances. It is also shown that a higher compression (HC = high compression) leads to a further reduction of the cell voltage, which is due to reduced interfacial contact resistance which occurs especially at high pressure.

The setup CH-HC was also investigated in a long term measurement of more than 150 h. In the first 90 h the current density was kept constant at 0.5 A/cm<sup>2</sup> at a cathode pressure of 100 bar and a temperature of 40°C whereas in the last 60 h the cathode pressure was reduced to 50 bar and the temperature was increased to 60°C. The voltage is constantly increasing over time for both pressures temperature pairs as can be seen in Figure 20. This is mainly due to the temperature which is decreasing more than 10 °C during the measurement. This is an issue of the temperature control in the system which can be improved.

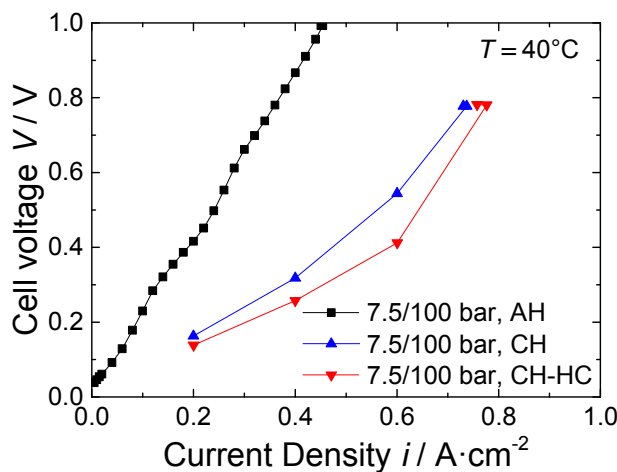


Figure 19: Vi-characteristic for different humidification concepts (AH = anodic humidification, CH = cathodic humidification, HC = high compression)

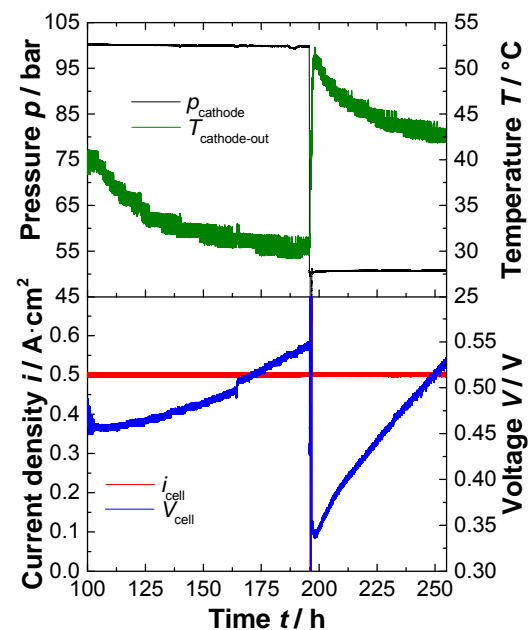


Figure 20: Current density, voltage, cathodic pressure and temperature during long-term measurement over 150 h

## SOC technology development at ECN part of TNO

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Mid 2018, ECN part of TNO restarted its activities on the development of Solid Oxid Cell technologies, with the objective to develop a next generation of SOC technologies for large scale hydrogen production in existing industrial infrastructure like refineries or ammonia plants, for grid balancing by renewable energy storage and industrial CO<sub>2</sub> re-use processes aiming towards generation of fuels or upgrading of biomass streams. The R&D lines followed by ECN part of TNO are the followings:

- Develop high performance and robust SO cells, able to operate in hydrogen electrolysis (SOE), reversible SOFC/SOE (r-SOC) and co-electrolysis of H<sub>2</sub>O and CO<sub>2</sub> (co-SOE)
- Realize a test platform based on electrochemical testing and accelerated test methods to assess SOC life-time and evaluate cell degradation issues intrinsic to the three SOE operating modes.
- Upscale the SOC manufacturing to both larger cell dimensions and quantities through low cost and environmental-friendly (additive) manufacturing processes, to deliver a competitive technology for large scale SOE systems.

Based on these objective, a lab-scale manufacturing line based on tape casting and screen-printing operations has been settled within the Faraday lab of ECN part of TNO, to produce high performance SOE cells based on SOFC technology developed by ECN in 2010. ECN part of TNO is also in the process of setting up electrochemical testing facilities for single cell testing with an outlook to stack testing, to assess cell/stack performance and degradation in SOE, r-SOC modes and co-SOE. The first electrochemical tests highlighted high performance of the ECN part of TNO cells in both fuel cell and electrolysis modes (figure 1). Most recent development regarding SOC manufacturing (infrastructure, cell upscaling) and testing, as well as an overview of the current projects of ECN part of TNO will be presented during the conference.

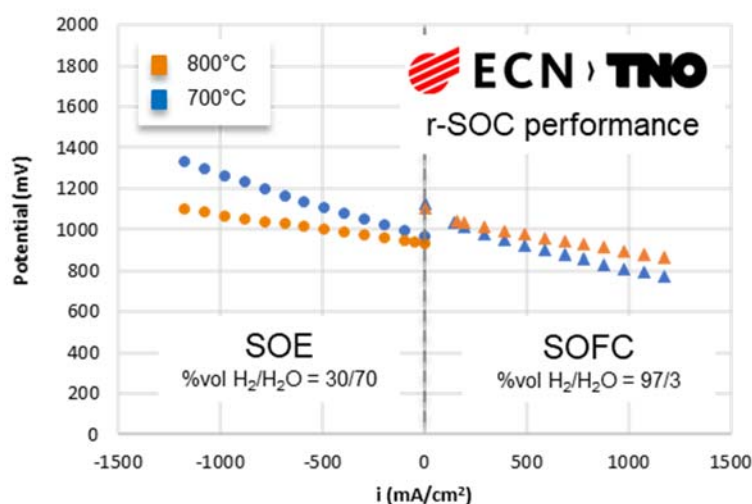


Figure 21: Performance of the TNO cells in r-SOC mode

## Co-Electrolysis in Power-to-X scenarios for renewable Industrial Chemistry

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The broad sector of applied industrial chemistry is relying on basic chemicals dominantly generated from fossil resources on enormously large scales. One of the essential petrochemical foundations is syngas, the mixture of carbon monoxide and hydrogen. Chemical conversions based on fossil resources to produce syngas have been optimized up to very narrow optimal steady-state points of operation and thus create a generic reluctance for changes in the methods of supply of these most fundamental components. With the on-going change towards renewable energy sources, electrochemical conversion of carbon dioxide to syngas induces a possibility for the defossilization process in conventional petrochemistry by Power-to-X scenarios<sup>[1]</sup>. The direct high-temperature co-electrolysis of water and carbon dioxide offers a suitable technology to provide white syngas. We discuss the proper technological framework in the scopes of supply of CO<sub>2</sub>, electrochemical performance of Solid Oxide Electrolysis Cells (SOEC) and products of white syngas. We divide into energy-related and process-related carbon dioxide, whereas process-related CO<sub>2</sub> originates from chemical reactions in certain process chains, like production of aluminium, steel, cement, etc. By this we show, that these emissions add up to 243 MMt·a<sup>-1</sup> for Europe in 2017 and will still be present as a carbon source in future scenarios even with 100 % renewable energy supply. The performance of co-electrolysis is compared to different electrolysis technologies and describes why high-temperature co-electrolysis has the ideal potential for the production of white syngas. In contrast to low-temperature routes, HT co-electrolysis experiments show current densities of up to 2.5 A·cm<sup>-2</sup> at 1.4 V compared to 300 mA·cm<sup>-2</sup> at 4 – 5 V for the LT-processes<sup>[2, 3, 4]</sup>. Furthermore, it is suitable for the production of all relevant syngas ratios used in industrial chemistry (1:1 to 3:1 H<sub>2</sub>:CO) by product composition tailoring at different operation parameters in one setup, even with constant performance (Figure 1). Also, the Fossil Carbon Equivalent (FCE) is introduced, with which we are able to show the impact of white syngas on industrial chemistry by matching energy demand, need of installed electrolysis capacities, consumption of carbon dioxide and substitutable amount of fossil resources to the market volumes of products of syngas chemistry (Figure 2).

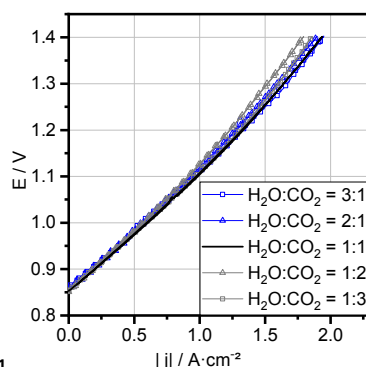


Fig.1

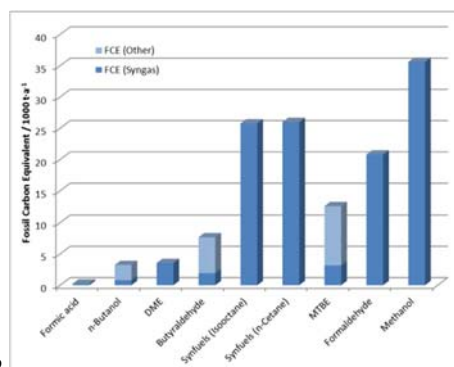


Fig. 2

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## Tubular proton ceramic electrolyzers for pressurized hydrogen production

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Incentives for developing high temperature electrolyzers (HTEs) using proton conducting electrolytes stem from the fact that a proton ceramic electrolysis cell (PCE) pumps out and pressurises dry H<sub>2</sub> directly. Existing HTEs design utilises the high packing density of planar stacks, but the hot seal and vulnerability to single cell breakdown give high stack rejection rate and questionable durability. In the GAMER FCH JU project started in January 2018, we develop a novel cost-effective tubular Proton Ceramic Electrolyser (PCE) stack technology integrated in a steam electrolyser system to produce pure dry pressurized hydrogen. The electrolyser system will be thermally coupled to renewable or waste heat sources in industrial plants to achieve higher AC electric efficiency and efficient heat valorisation by the integrated processes. The project aims at establishing a high volume production of novel tubular proton conducting ceramic cells. The cells are qualified for pressurized steam electrolysis operation at intermediate temperature (500-700°C) and designed as innovative single engineering units (SEU) encased in tubular steel shells. This modular technology is amenable to various industrial scales. GAMER focuses on designing both system and balance of plant components with the support of advanced modelling and simulation work, flowsheets of integrated processes, combined with robust engineering routes for demonstrating efficient thermal and electrical integration in a 10 kW electrolyser system delivering pure hydrogen at minimum 30 bars outlet pressure. The consortium covers the full value chain of the hydrogen economy, from cell and SEU manufacturer (CMS), system integrators (MC2, CRI), through researchers (SINTEF, UiO, CSIC), to end users in refineries, oil and gas, chemical industry (CRI, SGSI, with advisory board members YARA and AirLiquide). This presentation gives an overview of the project's main goals and current progress.

### Acknowledgment

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Abstract No 133

## **Development and implementation of innovative Electrolysis-PEM components in the frame of METHYCENTRE project**

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### ***METHYCENTRE project***

METHYCENTRE is a 5 years power to gas demonstration project started in 2018 and supported by ADEME (France) and funded by PIA (Programme d'Investissements d'Avenir) and FEDER (région centre Val de Loire). It is the first large scale Power to Gas project in France combining Methanization from agricultural wastes and catalytic Methanation.

Two other French industrial scale projects address Power to Gas : JUPITER 1000, leaded by GRT-gaz launched in 2017 which is based on a combination of gaseous catalytic Methanation step green H<sub>2</sub> and an industrial CO<sub>2</sub> source and HYCAUNAS which is based on a combination of Methanization (landfill gas) and biological Methanation step.

Both METHYCENTRE and HYCAUNAS are leaded by STORENGY (ENGIE). METHYCENTRE demonstrator will be commissioned close to the STORENGY natural gas geological storage facility in Céré le Ronde (100 km eastern from Tours)

The project includes a 250 kW PEM electrolyzer supplied by AREVA H2 GEN and powered by electricity produced from renewables energies. The aim of the demonstrator is to analyse what are the components that limit flexibility to adapt them.

Beside this commercial electrolyzer an additional 20 kW electrolysis facility will be implemented by 2021. The two electrolyzers will run in parallel. The aim of the experimental 20 kW facility is to assess the performance of new components and to consolidate the supply chain of these components.

### ***E-PEM innovation***

The 250 kW PEM electrolyzer is based on commercial membranes (Nafion type) and titanium bipolar plates. Nafion type membranes are expensive and their performances can be enhanced.

CEA developed new hybrid membranes based on a PVDF substrate and grafted with silicon based copolymer (SiO<sub>2</sub>-g-PSSA) to ensure the ionic transport of the proton. At the laboratory scale these membranes exhibit higher current density (+40% vs NAFION) a reduced H<sub>2</sub> permeability (-50% vs NAFION) and a good lifetime. The supply chain involves two steps : membrane formulation and roll to roll film production. The expected cost reduction compared to standard NAFION is about a factor 2.

Before the start of the demonstrator in 2021, there are two issues to overcome :

- Membrane Electrode Assembly needs further modification to fit optimally to the new membrane.
- The swelling of the membrane due to water should be reduced.

The MEA modification will be based on a CCM (catalyst coating membrane) technology.

To reduce the swelling an improved grafting procedure will be set up.

CEA has also developed composite bipolar plates for PEM applications. In the case of E-PEM, a composite cathodic bipolar plate will be developed to replace the titanium cathodic plate. A significant cost reduction is expected (-30%) as well as a higher life time with composite bipolar plate. The composite bipolar plate contains about 80% of graphite to ensure a high conductivity (planar conductivity about 95 S.cm<sup>-1</sup>).

Compared to PEM bipolar plates, the main issue is to design a porosity gradient across the plate thickness in order to facilitate water vapor evacuation.

## Pt-doped thin membranes for hydrogen crossover suppression in Proton electrolyte water electrolysis

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A key concern for the safe operation of polymer electrolyte water electrolysis (PEWE) cells is the high hydrogen gas crossover that could lead to explosive hydrogen-oxygen gas mixtures. The safety aspect is especially important with thin membranes and high differential pressures. So far different approaches have been taken to suppress the hydrogen gas crossover or to reduce the content of hydrogen in the anode compartment<sup>1</sup>. The concept of Pt doping of the proton exchange membrane (PEM) by Pt-salt impregnation followed by chemical reduction is well known from polymer electrolyte fuel cells (PEFCs), where it was used for electrode preparation and self-humidification<sup>2,3</sup>. The Pt particles in such impregnated membranes serve as recombination catalyst and can be applied to reduce hydrogen crossover in PEWE<sup>4</sup>. In this study, we present a new reduction method yielding thin membranes with high crossover suppression as well as good performance for an increase of the operational range of the PEWE cell.

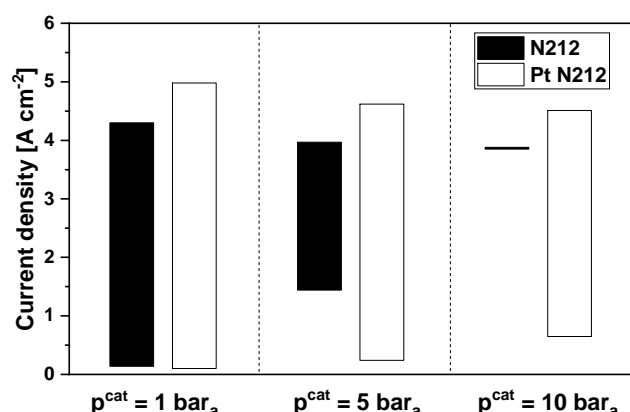


Figure 1: The turndown ratio displaying the operational range of PEWE at different cathodic pressures. The data shows cells using a commercial Nafion membrane of 50  $\mu\text{m}$  (N212, black) and a suchlike platinum impregnated membrane (Pt N212, white). The upper bound  $i_{\text{max}}$  is given by the maximum cell voltage of 2.0 V, the lower bound  $i_{\text{crit}}$  is given by the limit of 2% of hydrogen in oxygen.

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Abstract No 135

## Activity and stability of high performance electrocatalysts for hydrogen and oxygen evolution in Alkaline Water Electrolysis

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Electrolytically produced hydrogen is a core technique of the emerging green technologies. Besides the high performing Solid Oxide Electrolysis Cells (SOEC) and Proton Exchange Membrane Electrolysis Cells (PEMEC), the mature and well-established Alkaline Electrolysis Cells (AEC) ensure a reliable long-term production of hydrogen, but at lower current densities and lower efficiency compared to the other techniques [1]. Overcoming these disadvantages of the AEC is a promising way to combine high efficiency, high current densities and long-term performance for electrolytically produced hydrogen.

A key component to achieve this are catalytically active, stable and low-cost, non-noble metal electrocatalysts. Highly performing low-cost electrocatalysts for AEC have been identified and intensively investigated over the last decades, e.g.  $\text{Ni}_{1-x}\text{Mo}_x$  and  $\text{Ni}_{1-x}\text{Fe}_x\text{OOH}$  for hydrogen and oxygen evolution, respectively [2][3]. These electrocatalysts show outstanding performance, exceeding the Iridium Dioxide used for oxygen evolution in PEMEC.

Normalizing the performance of such novel electrocatalysts to the electrochemically active surface area (ECSA) remains challenging, but is necessary for a fair comparison between different materials. Furthermore, the long-term stability of such electrocatalysts, under technologically relevant conditions, has not been sufficiently addressed, despite its importance in ensuring the reliability of AEC.

This work focuses on the activity and stability of the  $\text{Ni}_{1-x}\text{Mo}_x$  and  $\text{Ni}_{1-x}\text{Fe}_x\text{OOH}$  electrocatalysts as a function of temperature from 25 - 100 °C in 1.0 M KOH and 45.0 % KOH. Different approaches in assessing ECSA are employed and compared, in an attempt to deduce the intrinsic (surface area normalized) activity of the electrocatalysts. We find that both electrocatalysts are far superior to state-of-the-art Ni at 25 °C and 1.0 M KOH, in line with the existing literature. Thermal activation is also observed, but  $\text{Ni}_{1-x}\text{Fe}_x\text{OOH}$  suffers from severe stability issues under technologically relevant conditions of 100 °C in 45.0 % KOH.

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## Progressive scale-up of a DEFT™ membraneless alkaline electrolyser

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Hydrox Holdings Ltd.'s alkaline Divergent-Electrode-Flow-Through (DEFT™) electrolyser has demonstrated that, through the development of small-scale prototypes, high purity hydrogen and oxygen gas (>99.5 vol%) can be attained by utilising a membraneless operating principle relying on diverging electrolytic flow through porous electrodes to achieve gaseous separation [1]. A Current density of 560 mA.cm<sup>-2</sup> (at 2 VDC, 60 °C), with the use of pure nickel circular electrodes, has been achieved with prior test plants. This has allowed for the design of units with high stack efficiencies, however, high parasitic loads as a consequence of high fluid velocities (0.075 m.s<sup>-1</sup>) and challenges associated with scale-up, have prevented the adoption of membraneless DEFT™ electrolysers as a viable means of decentralised hydrogen production.

Circular electrodes suffered from pressure/velocity gradients across the face of the electrode. This limited early membraneless prototypes to short electrolytic flow paths to maintain adequate gas purities. This proved problematic for effective scale-up. The flow path was found to be proportional to the required flow velocity for effective gaseous separation. At a flow path of 15 mm, a flow velocity of 0.075 m.s<sup>-1</sup> was required to maintain gas purities of > 99.5 vol%. This equated to high parasitic loads which resulted in reduced system efficiencies in the range of 30–40 HHV%.

Hydrox Holdings has addressed these challenges of high parasitic load and limited scale-up through optimisation of the design. Progressive scale-up and accompanying flow reductions were realised by centrally introduced flow, and by targeting preferential gas formation on the electrodes' inner surfaces. Two further prototypes were commissioned; a 110 mm mono-polar DEFT™ electrolyser, and a larger 230 mm bi-polar DEFT™ electrolyser. A 75-fold reduction in the necessary flow velocity, from 0.075 to 0.001 m.s<sup>-1</sup>, was realised. This reduced the estimated system power consumption (for a 5 kg/day plant) to 64.7 kWh/kg H<sub>2</sub> (equating to a system efficiency of 60.84 HHV%) @ 2.2 VDC, 80 °C, utilising pure nickel electrodes. The circulation pump accounting for 6.2 kWh/kg H<sub>2</sub> (9.6% of the total demand). A Current density of 293 mA.cm<sup>-2</sup> and H<sub>2</sub> purities of > 99.5 vol% were achieved. Using improved catalysts, further flow reductions, and higher temperature and pressure regimes, DEFT™ will achieve HHV% efficiencies in the 70 - 80 percentile range.

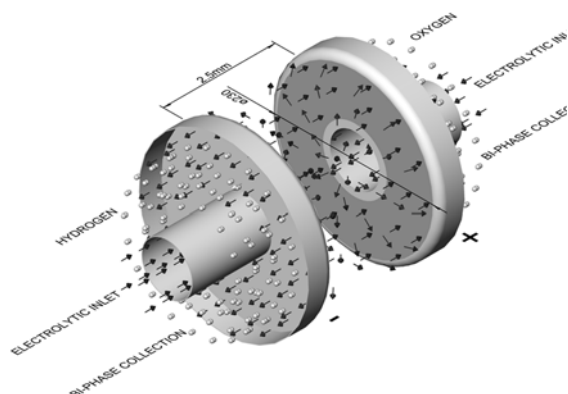


Figure 22: Bi-polar DEFT™ electrolyser ring-shaped electrode design

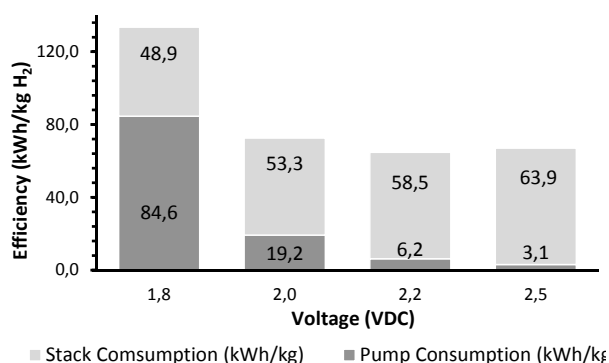


Figure 2: Power consumption and stack size @ 80 °C for an electrolytic velocity of 0.001 m.s<sup>-1</sup>

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Abstract No 137

## High dynamic testing of large active area PEMWE stacks and comparison with a simulation including automated parameter calibration and degradation analysis

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Initially first results of high dynamic testing of large area PEMWE stacks are presented here. This dynamic testing is part of the ongoing BMWI-funded project Carbon2Chem<sup>®</sup>. In order to proceed these measurements a test bench was developed and built up which allows automatic testing of short stacks up to 5 cells with active areas of about 900 cm<sup>2</sup> and a maximum current of 2000 A (depending on the number of cells). The pressure on both electrode sides can be adjusted up to 35 bar. For further analysis electrochemical-impedance spectroscopy (EIS) and single cell voltage tap are implemented.

In this work PEMWE stacks with 5 cells and active areas of 300 cm<sup>2</sup> and 450 cm<sup>2</sup> were examined regarding the degradation at operation with high dynamic load cycles. The following Fig. 4 shows an example for such a dynamic operation which realised with a 5 cell stack and 50 cm<sup>2</sup> active area. The depicted curves show the applied current (diamonds) and the measured stack voltage (circles). The main part in the middle shows a cycling operation with current jumps between 15 A and 60 A keeping up the value for 10 s.

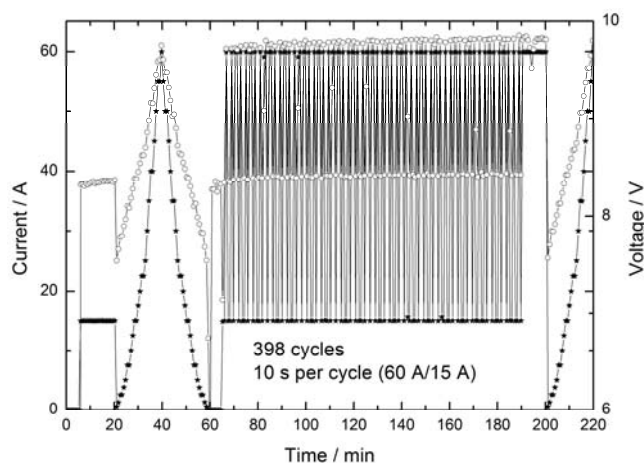


Fig. 4: Example for testing protocol measured with 5 cell stack and 50 cm<sup>2</sup> active area

The developed MATLAB model for Proton Exchange Membrane Electrolysis describes the current-voltage dependency in operation. It is designed to calculate static conditions and is used for parameter calibration. Besides physical constants and parameters characterizing material properties it also contains parameters that cannot be measured ex situ, like e.g. the charge transfer coefficients and exchange current densities. To determine those parameters, the model includes an automated parameter calibration procedure that is able to find the best parameter combinations. Due to the low computational capacity requirements, the model can be integrated into a system simulation. Since the MATLAB model takes no dynamic and time-variant behaviour into account, it is implemented in MATLAB/Simulink that has advantages for dynamic simulations. Furthermore, it can be easily combined with other Simulink models for system simulation. The new model can now be expanded with various dynamic influences to simulate and reflect the degradation of the electrolysis cell. An exemplary integration in MATLAB/Simulink is presented in this work, including a validation with dynamic measurement data. Further work will comprise the investigation of parameter changes due to dynamic and degradation effects. This work has been funded by the Federal Ministry of Education and Research (03EK3038B).

## An Investigation of the Sputtered Nickel Electrodes for Alkaline Water Electrolysis

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In this study, we investigated the kinetic efficiency of various sputtered Ni electrodes for hydrogen evolution reaction (HER) in low-temperature alkaline water electrolysis (AWE). A porous sponge Ni is usually used as the active HER electrode due to the high electrochemical active surface area (i.e., roughness factor). Here we prepared and compared three different sputtered Ni electrodes with controlling the roughness factor. First, a Raney Ni-Al electrode was prepared by physical vapor deposition (PVD) of Al on Ni substrates to form the Ni-Al intermetallic phase along with the heat treatment at 883 K followed by selective leaching of Al from Ni-Al intermetallic phases in 30wt.% KOH. Second, a porous co-sputtered Ni-Al electrode was prepared followed by selective leaching of Al from Ni-Al alloy in 30wt.% KOH. Last, an RF-sputtered Ni electrode was prepared by applying RF-magnetron sputtering on Ni substrates. Physical analyses such as XRD and SEM-EDS were conducted to confirm the existence of Ni phase and porous morphology. Ex-situ half-cell HER test and in-situ single cell test of AWEs were also conducted. It was found that the RF-sputtered Ni electrode exhibited the best-performance among the Ni electrodes as 90% efficiency [HHV] at 400 mA/cm<sup>2</sup>. The RF sputtering of Ni-induced well-controlled surface morphology around 10-fold roughness enhancement compared to the Ni substrate. Also, the RF-sputtered Ni electrode improved the on/off durability by enhancing adhesion property of the electrode interface, which may provide a promise for use AWE as a cost-effective energy conversion system for intermittent renewable energies.

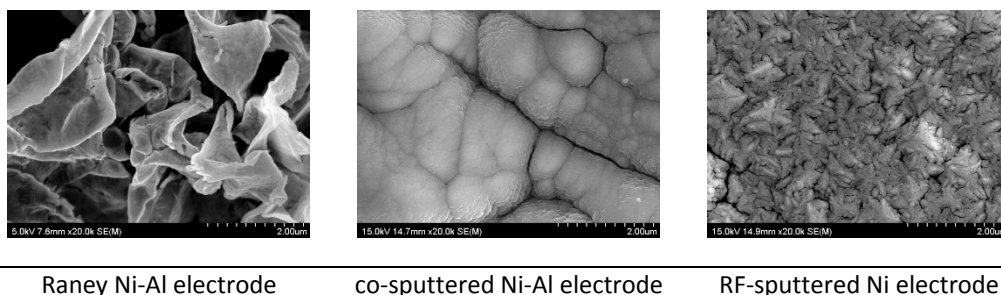


Figure 1. SEM images of various sputtered Ni electrodes

## Synchrotron studies of Pt oxidation and restructuring

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Pt catalysts remain the practical choice for PEM electrolyzers and fuel cells; more active catalysts than Pt are typically less durable. Catalyst degradation through dissolution is promoted by potential excursions that promote oxidation and reduction of the oxide film [1]. The surface oxidation/oxide film can also mediate slow restructuring of the catalyst, and is key to the activity for the oxygen evolution reaction, which occurs on the oxidized surface. Despite its importance, the nature of the oxidized surface at the atomic level has remained elusive, both in terms of structure and composition.

The locations and movements of Pt atoms during in situ electrooxidation of Pt(111) surfaces in perchloric acid were investigated at the atomic level by Surface X-ray Diffraction (SXRD) and at the nanoscale level by Grazing Incidence Small Angle X-ray Scattering (GISAXS). The locations of the first Pt atoms that move out of their lattice sites in a place-exchange process were determined more precisely than in earlier SXRD measurements, with determination of the coverages dynamically and simultaneously with potential steps or cycles. After less than about 0.15 monolayers of Pt oxidation, reduction is reversible, but further oxidation leads to irreversible restructuring of the surface [2]. Oxidation at constant potential at higher potentials up to 1.62 V leads to a disordered oxide film of about 0.5 Å thickness.

GISAXS measurements [3,4] determined the average nanoscale island spacing and the distribution of islands during potential cycling. The extent of nanoscale island production (surface roughening) increases with both the upper reversal potential and the number of cycles. The average spacing of islands increases slightly, but is mainly determined after the first cycle. The island size distribution sharpens with cycling, i.e., the short range order increases. Although the electrochemistry appears unchanged after about 15 cycles, the GISAXS shows continued changes to the surface to beyond 60 cycles. The structural changes are similar to those in UHV Pt growth and ion erosion, implying the same basic processes. A qualitative model of the growth in terms of adatom production and vacancy coalescence is proposed.

The interaction of the oxide with oxygen and vice versa has been controversial. Here it was investigated by SXRD while flowing oxygen-saturated electrolyte [5]. The presence of oxygen does not alter the initial stages of oxidation. Furthermore the oxygen reduction current decreases at potentials more negative than surface oxidation, showing that the activity of the reduction reaction is limited by the adsorbed intermediates in the reaction and not the presence of the oxide.

The implications of these measurements for understanding catalyst activity and degradation will be discussed.

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## Ion Exchange Membrane Based On Poly(norbornene)s Derivatives for Fuel Cell

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Ion exchange membrane, as the core component of fuel cell and liquid-flow cell, plays a key role in battery performance. Because of the double bond in the main chain of the norbornene polymer with ring opening metathesis polymerization, it has excellent thermal stability and good processability, and it has gradually become a research hotspot for ion exchange membrane materials. In our group, a series of ion exchange membranes were designed and developed based on polymer skeletons of polynorbornene structural derivatives: A class of ionic functionalized crosslinking agent was designed and synthesized by means of theoretical calculation<sup>1</sup> and experiment<sup>2</sup>. It was co-polymerized with norbornene cationic monomer to increase the ion exchange capacity of polymer film and improve the ionic conductivity. It also improves the mechanical stability and thermal stability of the polymer film, and makes up for the disadvantages such as the swelling and deformation of the polymer caused by hydrophilic ionic groups. And a new type of cationic exchange membrane polymer material with a dual-function ion exchange element, which are different from conventional membrane materials with a single ionic functional group, the performance of the ion exchange membrane were significantly improved compared with those of commercial perfluoronic acid films<sup>3</sup>. It is expected that the series of ion exchange membranes have a good application value in fuel cells<sup>4</sup>.

### Acknowledgments :

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Abstract No 141

## Reinforced hydrocarbon membrane for proton exchange membrane water electrolysis

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Proton exchange membranes (PEMs) are currently under wide investigation for membrane water electrolysis to deliver efficient production of high purity hydrogen. The state-of-the-art PEM for water electrolysis is Dupont's perfluorosulfonic acid membrane, Nafion. However, the drawbacks of Nafion, including high gas permeability, poor mechanical stability at high temperature, and high cost resulted in increased attention toward developing alternative membrane, i.e., hydrocarbon-based membranes. In this study, reinforced hydrocarbon membranes composed of porous nonwoven substrate and sulfonated multiblock copolymer ionomer were prepared as proton exchange membranes for water electrolyzers. The influence of the reinforcement on dimensional and mechanical stability, and electrochemical properties of the membranes were investigated and compared to those of the bare membrane and Nafion. The new reinforced membrane shows high proton conductivity ( $160 \text{ mS cm}^{-1}$  at  $80^\circ\text{C}$  and 100% RH), while retaining dimensional stability under high temperature, hydrated conditions. Testing in electrolysis has shown good energy efficiency ( $1.62 \text{ V}$  at  $1 \text{ A cm}^{-2}$  and  $80^\circ\text{C}$ ), making this membrane a potential candidate for commercial application in PEM water electrolysis.

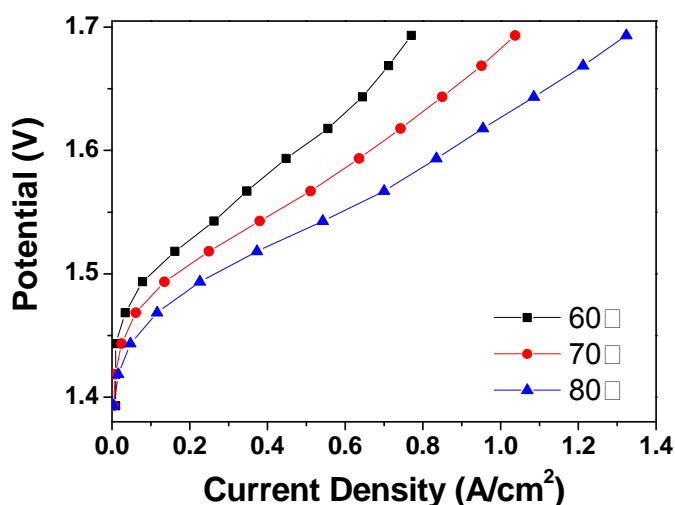


Figure 1. Single cell electrolysis using the reinforced hydrocarbon membrane at 60, 70, and  $80^\circ\text{C}$

## A Proven, Low Cost Coating Technology to Protect PEM Electrolyzer Separators

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### Abstract

Hydrogen is an important industrial feedstock having many different uses including its use as an energy carrier. Today, the majority (>90%) of the hydrogen produced uses a low cost, fossil based process called steam methane reforming (SMR). The disadvantage of SMR process is that it produces and releases carbon (as CO<sub>2</sub>). Water electrolysis, also a mature technology, converts water into hydrogen (and oxygen) and is carbon free. One type of electrolyzer, a proton exchange membrane electrolyzer (PEMEC), offers advantages including higher purity and differential pressures. The disadvantage of PEMEC is its high capital cost and the price for electricity.

With the advent of renewable energy sources (solar and wind), the price of the electricity is starting to come down. In addition, solar and wind farms are turned down depending on the supply and demand needs. Running these farms at peak performance can put further downward pressure on the price, where the extra electricity can be pumped into the PEMEC to produce hydrogen.

To address the capital cost, electrolyzer integrators are investigating innovative technologies that will reduce the capital cost and enhance the performance and durability of the electrolyzer. Contributing to the high the capital cost, the largest portion (48%) of the stack cost is the flow field and separators (Figure 1). To protect the separators from corrosion measured by high through put resistance (TPR)<sup>3</sup> and H<sub>2</sub> impregnation, system integrators use expensive platinum (Pt) based coatings.

This paper presents a low cost coating technology (DOT technology) that reduces the overall capital cost by lowering the expensive coatings used today. The DOT coating demonstrates superb performance and durability and as shown in Figure 2, stable TPR at various compression pressures. In addition and as shown in Figure 3, the DOT technology demonstrates much lower H<sub>2</sub> uptake into the titanium separator.

The DOT coating technology significantly reduces the amount of materials used (lowering costs) and does not use plating baths, a source of coating contamination that leads to separator failure. The process is well established for dimensions as large as 1 m<sup>2</sup>.

Figure 1: Stack Cost Breakdown<sup>2</sup>

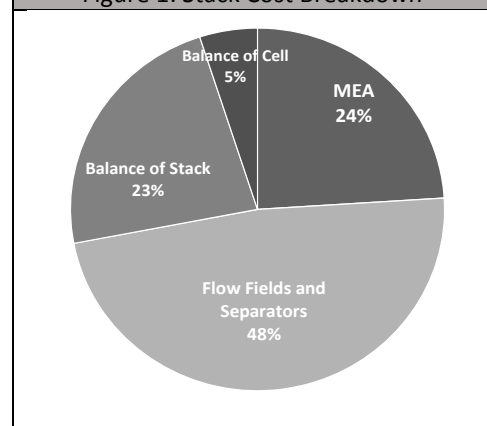


Figure 2: Through-Put Resistance<sup>2</sup>

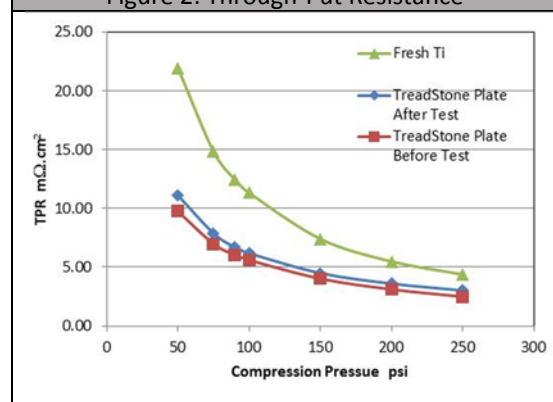


Figure 3: Accelerated Testing (1,000 hr. voltages and pressure cycles)

Bipolar Separators	H <sub>2</sub> Pres. (psi)	Time (hours)	H <sub>2</sub> uptake (ppm)
DOT Coated Ti separators	2,400	1,000	51
DOT Coated Ti separators	230	5,365	55
Zr Coated Ti separators	230	500	140
Dual Layer Pt coated Ti separators	230	500	1,105

<sup>[1]</sup> Proton Onsite, DOE AMR Presentation, Project ID: PD094, June 2014

<sup>[2]</sup> Through Put Resistance (TPR) is the sum of the contact resistance of the cathode and anode sides

## Physical Modeling of Co-Electrolysis in Solid Oxide Electrolysis Cells

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Solid oxide electrolysis cells (SOECs) are a promising technology to convert CO<sub>2</sub> and steam via co-electrolysis into syngas, i.e., a mixture of hydrogen and CO, which can be used downstream for producing synthetic fuels or chemicals. This technology becomes particularly attractive if the energy required for the co-electrolysis is obtained from renewable sources. The tailoring of the syngas composition obtained from co-electrolysis – based on the requirements of the subsequent processes – is key for the successful implementation of this process chain. Therefore, a detailed understating of the underlying mechanisms is important in order to optimize the SOEC design and operating strategies.

Here, we present a physical model for SOEC co-electrolysis which spatially resolves all relevant processes in the cell in 2D or 3D. The model includes gas transport in the channels and porous electrodes, electronic and ionic transport as well as heat transport. Water and CO<sub>2</sub> electrolysis as well as the reverse water gas shift (RWGS) are described by a thermodynamically consistent elementary kinetic model. The model has been implemented in our numerical framework NEOPARD-X [1] and allows for steady state and transient simulations. Experimental polarization curves and impedances at various temperatures and gas compositions are used for model validation. The model provides a physical interpretation of the impedance spectra. In particular it explains the occurrence of the inductive loop which is observed under certain operating conditions. Furthermore, we discuss the fuel utilization of H<sub>2</sub>O and CO<sub>2</sub> respectively and how to tailor the syngas composition based on the operating conditions.

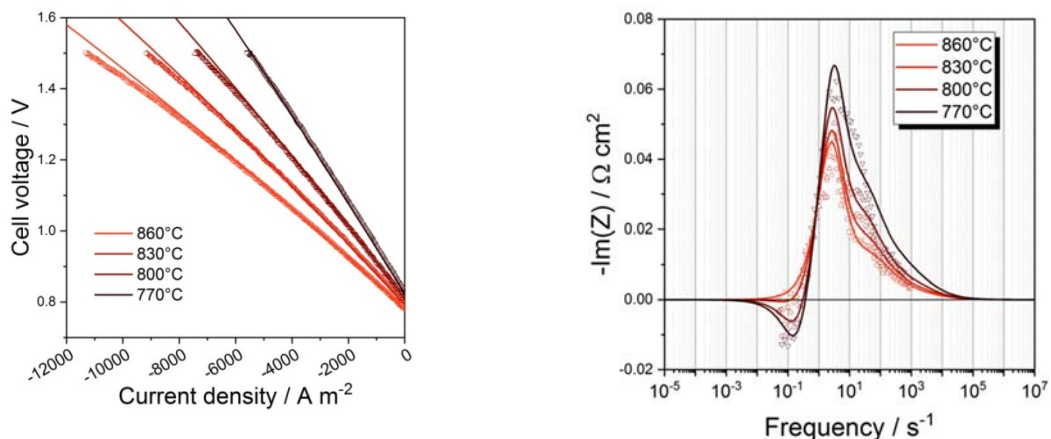


Figure: a) Comparison of measured (symbols) and simulated (lines) polarization curves at various temperatures under co-electrolysis with gas composition 5% H<sub>2</sub>, 63.7% H<sub>2</sub>O, 31.3% CO<sub>2</sub>; b) Comparison of respective impedance spectra at 0.6 A/cm<sup>2</sup>.

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- [1] G. A. Futter, P. Gazdzicki, K. A. Friedrich, A. Latz, T. Jahnke, *Journal of Power Sources*, 2018, **391**, 148–161.

## Electrochemical performance and durability of Ir-Ni nanostructures in PEM water electrolyzers

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Polymer electrolyte membrane water electrolysis (PEMWE) technology is currently emerging as a feasible alternative for large scale hydrogen production using excess electricity generated from renewable energy sources. However, insufficient catalyst stability, low electrochemical activity and efficiency due to sluggish oxygen evolution reaction (OER) kinetics on the anode, still remains a challenge for widespread application of PEMWE. Therefore, major advancements in catalyst design are required to overcome these challenges.

Alloying and nano-structuring are some of the most effective methods used to improve catalytic activity<sup>1</sup>. Alloying increases the intrinsic activity by tuning the d-band structure. Whereas nano-structuring increases the number of active sites available for electrochemical reactions to occur, and thus improving catalyst utilisation and efficiency. Therefore, this research focuses on development of highly structured and alloyed Ir-based nanoparticles, in efforts to improve OER kinetics and possibly durability.

In this work, Ir-Ni nanostructures were prepared via solvothermal technique and physically characterised using transmission electron microscopy, X-ray diffraction, and energy dispersive X-ray spectrometry. Synthesised nanostructures initially showed poor electrochemical performance, both ex-situ and in-situ. However, after successive cycling the cell performance progressively increased from 2.5 to 4.8 A cm<sup>-2</sup> at 2V, 60 °C. The increase in performance was mainly attributed to structural evolution induced by Ni dissolution, resulting in porous Ir-rich nanostructures with 81.5 % catalyst utilisation. The polarisation curves at 60, 70 and 80° C showed superior electrochemical performance at high current densities for Ir-rich nanostructures, due to improved mass transport properties compared to commercial Ir-black. In addition, these structures were found to be more electrochemically stable under dynamic accelerated stability test (AST). During AST, the current density at 1.9 V decreased by ca. 54.0 % for Ir-black, and increased by ca. 3.08% for Ir-rich nanostructures over the period of 160 h. The performance gain on Ir-rich nanostructures was due to improved kinetics as the capacitive semi-circle decreased in size over time. Post-mortem analysis showed no signs of Ir dissolution and precipitation on the membrane for Ir-rich nanostructures, whereas a significant amount of nanoparticles was observed for Ir-black.

Through nano-structuring, alloying and dealloying, both the catalytic activity and stability could be improved simultaneously. Furthermore, this approach opens up new opportunities for MEA designs with improved properties and low Ir-loading.

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Abstract No 145

## **Synthesis and Modification of Non-precious Multi Component Bi-functional Electro-catalyst for the Oxygen Electrodes in Unitized Regenerative Fuel Cells**

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Due to dwindling fossil raw materials and the ongoing climate change, it is necessary to develop concepts to provide clean and renewable energy sources and storage systems, that can compete with conventional fossil fuel based technologies<sup>[1]</sup>.

Unitized regenerative fuel cells (URFC) combine several advantage. Bi-functional oxygen and hydrogen electrodes are used in URFCs, which allow the supply of hydrogen by water electrolysis and the conversion of hydrogen in electrical energy with a single independent device. The resulting compact design connected with low amounts of used materials is reflected in low costs, lightweight and small space requirements. Such a device is of great interest for applications in remote areas or mobile devices<sup>[2]</sup>. However, since the used catalyst materials are based on expensive platinum group metals (PGMs) which limit broader applications, research is focussed on the development of highly efficient catalysts consisting of low-cost materials<sup>[3]</sup>.

In this study, we describe the synthesis as well as the modification of a bi-functional oxygen reduction (ORR) and oxygen evolution (OER) electro-catalyst, based on abundant transition metal oxides (TMO) and carbon, which exhibits outstanding electrochemical activity and stability. Aiming at a catalytic system, which is able to catalyse both oxygen reactions, two electro-catalysts were separately synthesised and physically mixed. The combined over potential to provide -3 mA cm<sup>-2</sup> and (ORR) 10 mA cm<sup>-2</sup> (OER) is 0.701 V. Modifications of the separated materials improve the activity and stability further. The resulting system exhibits a total over potential of 0.681 V and therefore clearly outperforms the activity and stability of all literature known non-noble based ORR/OER electro-catalysts as well as commercial noble metal based reference catalysts.

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- [2] Sadhasivarn, T., *et al.*, International Journal of Hydrogen Energy, 2017, **42** (7), 4415-4433.
- [3] Dutta, K., *et al.*, Fuel Cells, 2017, **17** (6), 736-751.

## Solid oxide electrolyzers for sustainable fuels production

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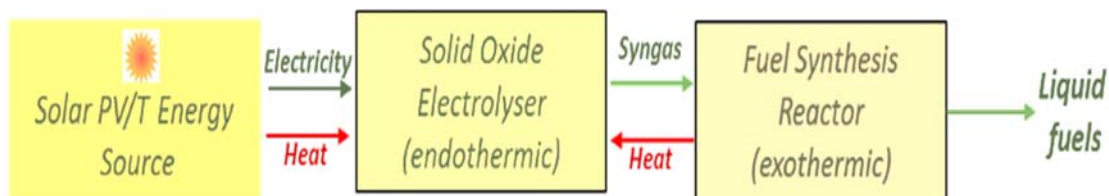
<sup>a</sup>Affiliation: Commonwealth Scientific and Industrial Research Organisation (CSIRO)

<sup>b</sup>affiliation: Ben Gurion University at Negev (BGUN)

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Solid oxide electrolyzers (SOEs) powered by renewable energy provide an efficient and economically feasible route for the production of sustainable fuels. Running SOE in an endothermic mode with external heat source allows up to 40% reduction in the electrical energy requirements as compared to low temperature electrolysis process. As the cost of solar thermal input is substantially lower compared to the electric input (Solar PV) to the process, this way of operating the electrolyser leads to significant process-cost savings per Kg of hydrogen produced. In addition to the steam electrolysis, SOEs can be used for the co-electrolysis of CO<sub>2</sub> and steam to produce a mixture of H<sub>2</sub> and CO (synthetic gas). The synthetic gas thus produced can be converted into transportable liquid fuels and commodity chemicals like lubricants using thermally integrated downstream catalytic reactor. The thermal integration of the electrolyser with exothermic downstream fuel synthesis process further boosts process efficiency. In this presentation a brief overview of SOE development being carried out by CSIRO led consortium will be presented with an emphasis on the electrochemical performance of the tubular solid oxide electrolyzers with newly developed cathodes that show lower overpotential losses compared to the conventional cathode materials. Also, the theoretical energy balance for different fuels (hydrogen, gasoline and methanol) will be discussed along with preliminary capital cost estimates.



**Figure 1:** Schematic of the process for liquid fuel production using solid oxide electrolysis



**Figure 2:** Tubular reactors with different zirconia electrolytes

## Semi-empirical steady-state model for a PEM electrolyser at varying operating conditions

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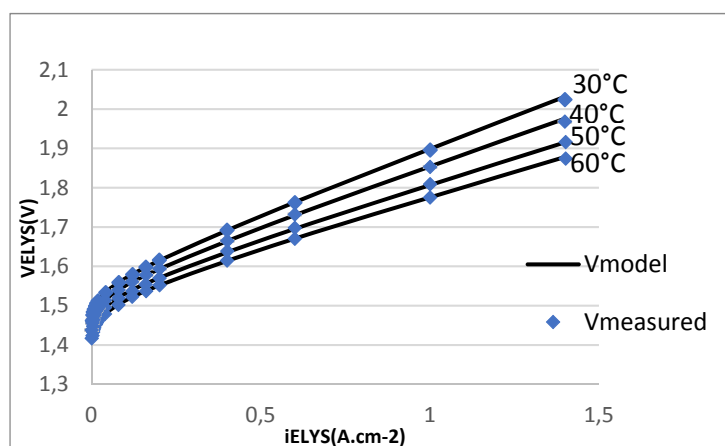
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The need to reduce greenhouse gas emissions is significantly changing the electrical power generation across the world. The increasing deployment of renewable energy systems, for instance wind and solar, requires new energy storage solutions in order to overcome their fluctuating and intermittent nature. PEM electrolyser associated with a hydrogen tank seems to be a promising solution for a long-term electricity storage as chemical energy. [1]

The interactions of an electrolyser with other elements of a power-to-gas system might have an impact on its performance and its durability. Many physico-chemical phenomena occur in an electrolyser and they can affect its operation. Understanding and quantifying the impact of main phenomena on the overvoltage of an electrolyser is crucial. Therefore, we developed an electrical equivalent steady-state model for a proton exchange membrane (PEM) electrolyser at varying operating conditions.

The aim of the model is to distinguish the main overvoltage components: activation overvoltage, ohmic potential and diffusion over-potential, each of which has a specific sensitivity to operating conditions. The model is semi-empirical and is based on a set of 9 experimental V-I curves measured by current steps at different pressure and temperature conditions. At the end of each steps, an electrochemical impedance spectroscopy is made to extract the ohmic resistance.

With all these data we can fit the experimental V-I curves with a V-I equation containing 6 parameters, 5 of which have a physical significance. This steady-state model allows us to predict the performance of the electrolyser at various operating conditions and to quantify the different overvoltage components. It also gives us information about its ageing: we compared the parameters obtained by curve fitting in 2016 with the parameters obtained in this work. They were similar which means that the electrolyser's performance did not degrade in time while it was not in operation. This approach was developed few years ago for a PEM Fuel Cell model [2] and was adapted for a PEM electrolyser.



T	30°C	40°C	50°C	60°C
%error min	0.001 %	0.01 %	0.002 %	0.001 %
%error max	0.38 %	0.39 %	0.34 %	0.542 %

Table 1: Relative error of different curves

Figure 23: V-I curves measured and V-I curves modelled at different temperature conditions

[1] Gerda Gahleitner, *Hydrogen from renewable electricity: An international review of power-to-gas pilot plants for stationary applications*, 2012, International Journal of Hydrogen energy 38, 2039-2061

[2] Labach, O. Rallières, C. Turpin, *Steady-state Semi-empirical Model of a Single Proton Exchange Membrane Fuel Cell (PEMFC) at Varying Operating Conditions*, 2017, Fuel Cells DOI: 10.1002/fuce.20160007



## Large Scale PEM Electrolysis for Industrial Applications

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Polymer electrolyte membrane (PEM) based water electrolysis for hydrogen production is widely considered as a key technology to decarbonize our fossil-based energy system. Due to its highly dynamic behavior PEM electrolyzer systems accomplish the requirements to be operated with renewable energy-based electricity such as wind or PV power. The production of chemical energy carrier such as hydrogen enables the integration of renewable energy into industrial processes and mobility and therefore sector coupling.

Changes need to be made in energy production to achieve the long-term goal of the Paris agreement, to keep global warming below 2°C [1], and to reach the CO<sub>2</sub> target by 2050 whereas hydrogen could make up to 20% of the abatement [2]. Reaching this target, a transition of the fossil-based hydrogen production towards green hydrogen produced by electrolysis is an essential factor. Therefore, PEM electrolyzer systems have to reach industrial relevant sizes and significant cost reduction.

Siemens Hydrogen Solution has released its next generation of PEM electrolyzer system Silyzer 300 in 2018. By choosing a highly modular concept, Siemens AG is offering hydrogen generation plants from 10 to 100 MW based on PEM technology. It allows the full utilization of economy of scale effects also for customized solutions.

Together with our partners in H2Future, an FCH JU funded project, a demonstration plant is currently being built and will be operated at the largest steel production site of voestalpine Group in Linz, Austria. In a nutshell, the fundamental goal of H2Future is to demonstrate that an industrially integrated PEM electrolyzer system is able to produce green hydrogen and provides grid services at the same time.

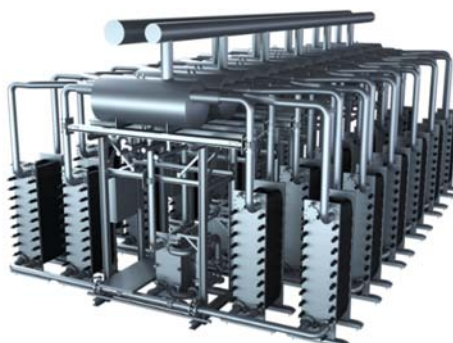


Figure 1: Siemens AG's latest electrolyzer generation SILYZER300 in the preferred configuration of a 24-module array (~340 kgH<sub>2</sub>/h)

[1] [https://ec.europa.eu/clima/policies/international/negotiations/paris\\_de](https://ec.europa.eu/clima/policies/international/negotiations/paris_de)

[2] [Hydrogen Council, Hydrogen Scaling up: A sustainable pathway for the global energy transmission, Nov. 2017](#)

Abstract No 149

## **The influence of ferric ion impurities on a proton exchange membrane electrolyzer operated at varying temperature and current density conditions**

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Hydrogen is a clean and flexible energy vector, and will act as an important energy source in the future. The proton exchange membrane (PEM) water electrolysis, which separates water to produce hydrogen and oxygen from renewable energy source through an environmentally friendly electrochemical process, is considered to be a promising hydrogen generator.<sup>1</sup> A water purification system is typically used in PEM water electrolysis systems, but over time the quality of the circulating water may deteriorates for example due to the production of metallic impurities (such as cations of Fe<sup>3+</sup>, Ca<sup>2+</sup>, Na<sup>+</sup> etc.). These impurities can be originated from feed water, the fabricating process of MEA, the corrosion of components materials such as water pipes, membranes, bipolar plates, the dissolution of electrocatalysts and other sources.<sup>2</sup> The impurities were proved to have higher affinity than protons for the sulfonic acid group sites and could substitute protons in normal ion exchange processes. The cationic impurities could markedly reduce the ion conductivity of Nafion membrane and lower water content of membranes by displacement of protons, which will lead to a progressive increase of cell voltage. Impurities for instance Fe<sup>3+</sup>, Cu<sup>2+</sup>, Na<sup>+</sup>, etc. migrate into and deposit on the membrane and catalyst layer, occupying ion exchange sites and covering the electrochemical active surface of catalysts, leading to sluggish reaction kinetics and increased cell overpotential both at the cathode and anode. Also, the external impurities may agglomerate electrochemically on the cathode side, hindering the hydrogen evolution reaction.<sup>2,3</sup> Furthermore, some metallic impurities may promote the generation of hydroxide radicals from hydrogen peroxide and hence degrade the membrane severely by the caused Fenton mechanism.<sup>4</sup> In this work, the influence of 5 ppm Fe<sup>3+</sup> contamination on cell performance of PEM water electrolysis has been investigated, and the temperature and current density effects on cell performance in the presence of Fe<sup>3+</sup> were also studied. The Electrochemical impedance spectroscopy (EIS) and polarization results showed that cell performance decreased severely when 5 ppm Fe<sup>3+</sup> was introduced into feed water. Higher temperature (80 °C) could reduce ohmic resistance and higher current density (2 A/cm<sup>2</sup>) can contribute to reduce charge transfer resistance and mass transfer resistance at 5 ppm Fe<sup>3+</sup> concentration.

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4. F. Fouda-Onana, M. Chandesris, V. Médeau, S. Chelghoum, D. Thoby and N. Guillet, *Int. J. Hydrogen Energy*, 2016, **41**, 16627-16636.

## Detection of Electrooxidation Products in Microfluidic Devices Using Raman Spectroscopy

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Microfluidics combined with Raman Spectroscopy has great promise for monitoring reaction processes with very little volume of liquid provided. This advanced method for in-situ and online detection allows for flexible manipulation of fluids, micro/nano-particles, and biological samples. Lab-on-a-chip applications are often used for chemical analysis, but Raman microscopy has also been used to monitor reactions within microreactors. In electrochemistry, the use of SERS to analyze surface species is common. However, the combination of the controlled mass transport in microfluidics with detection of soluble intermediate and product species by Raman offers the possibility of mechanistic kinetic studies of electrocatalytic reactions. We here show the feasibility of this method for studying electrocatalysis of oxidation of alcohols.

This research makes use of a channel where the laser focuses downstream of the Pt wire/mesh working electrode. The channel is connected to a reservoir with a reference electrode and a counter electrode. A Teflon tube connected to a pump flows the liquid through the channel at a pre-set flow rate to oxidize alcohol for online measurements. Highly concentrated KOH is used as the electrolyte, and 5 M methanol is used for a high S/N ratio in Raman spectra. Time sequences of Raman spectra show production of formate and carbonate, and its dependence on potential and flow rate were investigated. Lower flow rates and higher overpotentials yielded higher ratio of complete oxidation products, i.e. CO<sub>2</sub> or carbonate, while high flow rates and low overpotentials yielded higher ratio of partial oxidation products, i.e. formate.

Recent experiments using carbon paper with electrodeposited Ni nanoparticles as working electrode in the above-mentioned microfluidic device show a very high conversion rate on ethanol and ethylene glycol with highly concentrated KOH. Acetate (major), formate (major) and oxalate (minor) are identified to be the oxidation product of ethanol. Oxalate and formate are produced in the oxidation of ethylene glycol.

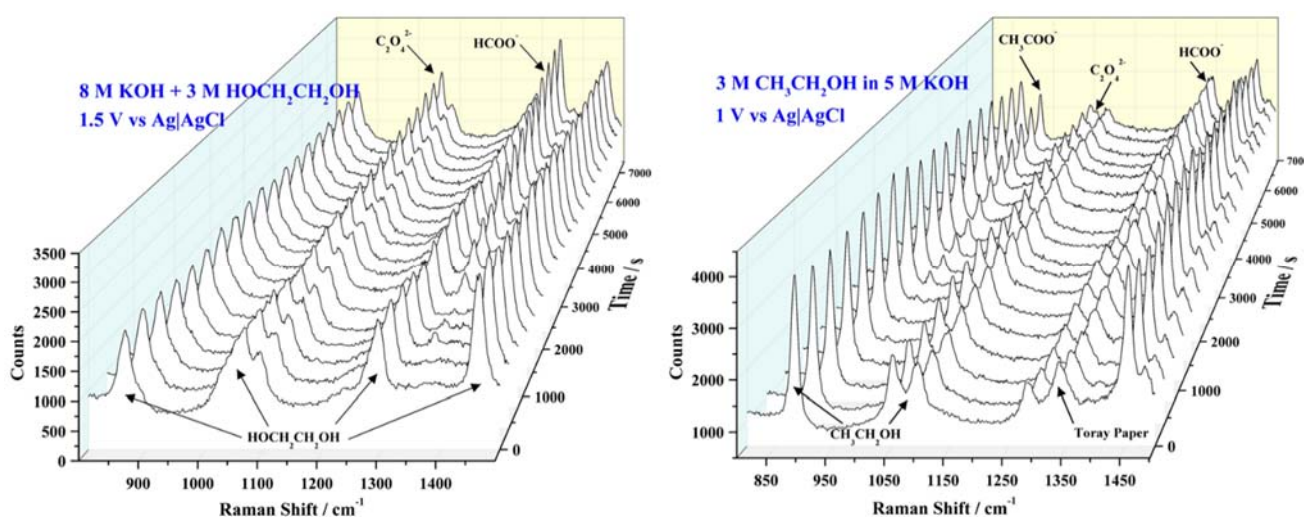


Figure 1. Time-dependent Raman Spectra of Ethanol / Ethylene Glycol electrooxidation in alkaline condition.

Abstract No 151

## **Proton Exchange Membranes of Low Hydrogen and Methanol Permeability for Elevated Temperature Electrolysers**

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In comparison with liquid alkaline electrolytes, a proton exchange membrane (PEM) is characterized by low gas permeability. This, in combination with high conductivity and mechanical robustness, makes it possible to utilize membrane electrolytes in small thickness (less than 200 microns) and therefore lower ohmic resistance. As a result, higher current densities and voltage/energy efficiencies are achieved for the PEM electrolyzers.<sup>[1]</sup> The low hydrogen and oxygen permeability is also critical in order to yield high purity hydrogen and allow for a wide load range of operation, an issue in association with the use of fluctuating renewable energies.<sup>[2]</sup> An extended application of the technology is the co-electrolysis of carbon dioxide and water, generating hydrocarbons or alcohols instead of hydrogen.<sup>[3]</sup> For this purpose, slightly elevated temperatures above 100°C are desired, which imparts challenges for the proton exchange membranes in term of proton conductivity, mechanical stability and permeability of products e.g. methanol. These challenges are addressed by selection and development of PEM materials i.e. composites of the short side chain perfluorosulfonic acid polymers e.g. Aquivion® with inorganic fillers. In this paper, characterization of the membranes, particularly the measurement of hydrogen and methanol permeability, is reported in a temperature range of up to 140°C under varied humidity. A significant reduction of the permeability of hydrogen (by 10 times) and methanol (by 100 times) is observed as soon as the membrane is switched from saturation with liquid water to water vapour. Further discussions will be made including in-plane and through-plane conductivity and tensile behaviours of both the pristine and composite membranes.

### **Acknowledgement**

Financial support from the EU H2020 LOTERCO2M project “CRM-free Low Temperature Electrochemical Reduction of CO<sub>2</sub> to Methanol” (Grant number 761093) and Innovation Fund Denmark project Wind2H, “Robust and Durable Electrolyzers for Hydrogen Based Energy Storage of Wind Power” (project number 5185-00025B) is acknowledged.

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Abstract No 152

## Design and operation of a hydrogen refilling station with on-site alkaline electrolysis

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The Laboratory of Physical and Analytical Electrochemistry has developed and built a hydrogen refuelling station with on-site alkaline electrolysis. The balance of plant of a 50 kW alkaline electrolyser was redesigned and reprogrammed to monitor 43 process variables and power consumption data per second. A hydrogen processing unit based on pressure swing adsorption and compressed air gas boosters was designed to allow 350 and 700 bar refills.

The design and the control system of the electrolyser shall be presented as well as a complete characterisation of the energy consumptions from grid to mobility [1]. Detailed measurements for various current set points will be provided together with the data from the synchronised compression system between the outlet of the electrolyser at 10 bar and the supply storage at 200 bar.

Finally, refilling strategies using a supply storage at 200 bar, a high pressure cascade at 500 bar and gas boosters will be presented, for up to 210 L of H<sub>2</sub> at 700 bar.



Figure 24 - Pressure swing adsorption system and alkaline electrolyser (left) - Hydrogen refilling station being used for a 350 bar refill (right)

This work was supported by a grant from the Swiss Federal Office for Energy (grant Nr: SI/501286-01).

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Abstract No 153

## Performance enhancement of PEM electrolyzers through iridium-coated titanium porous transport layers

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Hydrogen can be generated from renewable energy sources by means of PEM electrolysis. Due to the harsh conditions such as high overpotential, oxidation and low pH in anode, the components of the anode demand very high corrosion resistance for PTL and bipolar plate. Therefore, titanium is generally employed to construct cell and stack components, such as flow fields and porous transport layers (PTLs) which today accounts for 50% of overall stack costs [1,2]. However, the passivation of the titanium based porous transport layer (PTL) cannot be ignored, since titanium (Ti<sup>0</sup>) changes its oxidation state over time, which can increase surface electrical resistivity and detrimentally impact cell performance [3,4]. To circumvent this issue, PTLs are typically coated with considerable amounts of platinum or gold to ensure reasonable performance profiles over time [5-7]. Moreover, it is well known that the oxide forms of platinum and gold are not stable under electrolysis conditions.

This presentation will introduce an easy and scalable method to protect the titanium PTL from passivation by sputtering very thin layers of iridium onto commercially-available titanium PTLs [8]. The iridium layer reduces the overall ohmic resistance of the PTL/catalyst layer interface and significantly improves the cell performance.

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## Reversible Hydrogen-Chlorine PEM fuel cell development

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Reversible Hydrogen-Chlorine cells are a promising type of devices for the regulation of the energy produced by solar PV panels and wind turbines. In the electrolytic mode, this type of cell transforms hydrochloric acid into hydrogen and chlorine, which can be stored helping to regulate the exceeding energy. In fuel cell mode, the reversible reactions occur and hydrochloric acid is regenerated [1]. Thus, the compounds which have been produced during the electrolysis of hydrochloric acid are fed to the same cell operating as a fuel cell to produce energy [2]. Integration of the both modes of operation into the same cell is an important handicap which has been faced in this work, using different Mixed Metal Oxides anodes in order to determine the most efficient formulation, which are characterized by XRD and by EDX-SEM (physical characterization), and by cyclic voltammetry and by electrochemical impedance spectroscopy (electrochemical characterization), different proton exchange membranes and operation conditions [3] [4]. This work reports some of the most interesting results obtained in this promising technology.

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### Acknowledgements.

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Abstract No 155

## Novel catalyst supports based on the mixture of SiCTiC-C for the SO<sub>2</sub> electrolysis

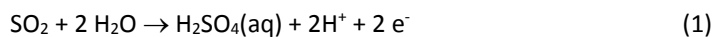
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The aim of this work is the assessment of a novel support composed of carbon black (Vulcan XC 72R) and a composite titanium silicon carbide (SiC-TiC) as catalyst support for the SO<sub>2</sub> depolarized electrolysis at high temperature (100-150 °C). Firstly, mixtures with different weight ratios of Vulcan and SiC-TiC are prepared and characterized. Thus, the evaluation of in-plane conductivity of the different prepared mixtures is developed at different temperatures in the range of 100-200 °C by using a potentiostat/galvanostat AUTOLAB PGSTAT 302N equipped with an FRA module. To obtain comparable results, a commercial carbon cloth electrode provided by Freudenberg is used as a base surface to deposit the mixture of catalyst support by spreading the ink suspension on it to get 0.4 mg<sub>solid</sub>/cm<sup>2</sup>, as reported elsewhere [1]. Moreover, cyclic voltammetry (CV) analyses were also performed to assess the electrochemical degradation of the different prepared supports. The best novel catalyst supports are selected to be catalysed with a 40 % of Pt nanoparticles following a standard method [2]. The samples are physicochemically (XRD, TEM, ICP) and electrochemically characterized by means of cyclic voltammetry analyses to evaluate the Electrochemical Active Surface Area (ECSA) and their electrochemical stability in sulphuric acid media.

Finally, the Pt based catalysts on the novel supports and standard one (Vulcan carbon) are tested in a bench scale electrolysis cell of 25 cm<sup>2</sup> operating at the temperature range of 100-150 °C and using PBI (Polybenzimidazole) based membranes. In this case, the anodic stream consists of SO<sub>2</sub> and water in gas phase where it is oxidized to form H<sub>2</sub>SO<sub>4</sub>, electrons and protons that emigrate across a proton exchange membrane (PEM) to the cathode where they are reduced to form hydrogen according to equations 1 and 2 [3].



This work contributes to the advance of knowledge for the improvement of electrodes for the SO<sub>2</sub> electrolysis. A process that is part of the hybrid Sulphur Cycle (also known as Westinghouse cycle) to produce “green” hydrogen [4]. Moreover, the electrolysis of SO<sub>2</sub> can be used for the sulphur dioxide removal from flue gases of sulphuric acid [5].

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## **Production Techniques of Making a Truly Intimate Alloy of Iridium Ruthenium Oxide and Important Considerations as an End-Product in PEM Electrolysers.**

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As a PGM catalyst manufacturing company, Ceimig has many considerations which impinge on research, development, production, quality control and end-use; all under the umbrella of cost. We present here a journey showing the timeline and outcome of developing one such specific product: namely Iridium Ruthenium Oxide and how academia, end-users government and even the public can affect and influence inter-company R&D direction. The iridium-ruthenium has been developed as a unique material at production scale and has been confirmed to be a true alloy as was previously suspected<sup>1</sup> and not just an intimate particle mix. The advantages of this uniqueness will also be presented.

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Abstract No 157

## **1 kW solid oxide prototype system coupled to a water treatment plant for synthetic gas production**

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The CoSin project aims to validate the production of renewable synthetic natural gas at a water treatment plant based in Barcelona area. The sewage plant is currently operating two anaerobic digesters to treat primary and secondary slurry for environmental purposes. The obtained biogas, which previously was only used to maintain the temperature of the digesters, is now redirected to an alternative route to valorise this biogas: first, a biogas upgrading with membranes and then coupled with a carbon dioxide hydrogenation, to produce a biogas richer in synthetic methane.

The first step consists in drying, cleaning and separate the methane from the carbon dioxide already contained in the produced biogas. In addition, the second step aims to valorise the separated carbon dioxide by producing methane and inject it into the natural gas network. The hydrogen necessary for the catalytic methanation unit will be produced by water electrolysis.

Two types of electrolysis technologies are considered in this project. To ensure a low cost hydrogen production from the beginning of the project, a commercial alkaline electrolyser of 37 kW working under pressure (12 bar) was integrated in the water treatment plant together with the biogas cleaning and the catalytic methanation unit. In parallel, a 1 kW SOEC prototype with higher efficiency is being installed to be operated under electrolysis and co-electrolysis mode [1].

As mentioned, apart from a higher efficiency considering a cheap heat source, high temperature electrolysis presents the advantage of being able to work in co-electrolysis mode. A carbon dioxide mixture with water can directly be injected into the stack to produce hydrogen and carbon monoxide (syngas). The syngas formed by the reduction of those two species is the precursor to synthesize methane but also other synthetic fuels as diesel, ethanol, dimethyl ether, etc [2]. Thereby, the high temperature electrolyser is planned to be operated in electrolysis mode and in co-electrolysis mode with a methanator coupled to the system.

The high temperature electrolyser prototype is completely designed and developed in Spain through the collaboration of research centres (IREC, UPC) and companies (FAE, AMES). The stack is composed by thirty 8x8 cm<sup>2</sup> cathode supported cells made with Ni-YSZ, YSZ electrolyte, CGO barrier layer and LSCF-CGO oxygen electrodes. Interconnectors made with ferritic stainless steel by powder metallurgy and recovered by MnCO<sub>3</sub> are used to ensure a good current collection and gas diffusion along the cell. All the prototype elements have been already validated at laboratory scale obtaining promising results in electrolysis and co-electrolysis mode.

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## Investigation of feed water impurities on life-time of PEMWE

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With the introduction of fuel cell electric vehicles (FCEV), hydrogen gas produced without fossil fuels is required to reduce the CO<sub>2</sub> emissions. At the same time, the production of renewable energy is increasing. Water electrolysis to produce hydrogen with the use of electricity from renewable sources allows for storage of the energy in the form of hydrogen. The gas can be utilized either back to the electric net or as fuel for FCEVs.

However, the cost of water electrolysis systems needs to be reduced while the lifetime must be increased. One of the main limitations of the proton exchange membrane water electrolyser (PEMWE) system is the degradation of the membrane<sup>1</sup>. This limits the lifetime of the system and is expensive to replace. It has been shown that impurities from feed water and the degradation products from other component poison the membrane, lowering the proton conductivity. Furthermore, metal ion impurities catalyse the formation of hydrogen peroxide at the cathode further contributing to irreversible membrane thinning<sup>2</sup>. In industrial systems, the water circulated to the cells is purified to minimize the degradation. However, the purification limits the operating temperature of the systems and increases the total system cost<sup>2</sup>.

The water quality used in most electrolysis cells today utilises ASTM type II deionized water. However, little research is done on the limitations, and quantifying the reduction in efficiency dependent on the water quality. Dedigama et al.<sup>3</sup> calculated the minimum flow needed, and further state that in industry, 5 times the necessary flow of water is circulated to ensure proper wetting of the membrane. However, in research, an excess of water is often used, up to 100 times higher flow than required, to exclude mass transport restrictions on the reactions<sup>3,4</sup>.

Increasing temperature decreases the kinetic overpotential and increases the membrane conductivity<sup>4</sup>. However, also dissolution of the catalyst and degradation of the cell components increase with temperature. Furthermore, in industrial applications the maximum temperature of the water into the purification system is 60°C<sup>5</sup>. Dependent on the aim of the research, experiments at temperatures as low as 25°C are performed to fit with the industry, while others run at 80 or 90°C to probe the upper limits of current density and efficiency<sup>2</sup>.

In this project we aim to analyse the effect of varying water purity on the membrane degradation in a single PEM electrolysis cell test setup. Furthermore, the effect of changing temperature from 60 to 80°C on the impurity tolerance will be studied. The circulating feed water will be analysed with respect to conductivity, metal ion and fluorine concentration. A parallel "blank" system with only tubings, fittings etc will be assembled and compared to the data measured from the electrolyser. Contaminating species will be added to the feed water to study their impact.

The project is in its early stage and in this poster only preliminary results will be presented.

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## Bipolar Plates for PEM electrolyzers

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Even though the alkaline water electrolyzer is a much more mature technology compared to the Proton Exchange Membrane water electrolyzer (PEMWE), the latter is considered a more promising alternative, as it can be operated under differential pressure, high current densities and a wide range of power inputs compared to the alkaline electrolyzer [1]. When combining several single electrolyzer cells in a stack, Bipolar Plate (BPP) separator plates are often used to conduct heat and current between each cell in the stack, as well as to collect product gases within the stack without mixing [1]. The BPP should possess high mechanical strength, high shock durability, have high corrosion resistance and be easy to manufacture [2]. Titanium has until now been the most common substrate material for BPPs PEM electrolyzers [1, 2], However, the oxide formed on the titanium surface during anodic polarization causes a high and increasing ohmic resistance. Several studies on BPP materials have been conducted, with the main focus on coated or alloyed metals [3-5]. However, there are few studies with the objective of testing and



Figure1: Bipolar Plate inn a PEMWE. comparing a selection of metals and alloys under conditions similar to those inside a PEM electrolyzer.

SINTEF and NTNU have tested various substrate materials under simulated PEMWE conditions [6]. The objective of the study was to investigate the corrosion properties of the materials as well as study the oxide formation and contact resistance development. The materials were polarized up to 2 V<sub>SHE</sub>, which, to the authors knowledge, had not been done before for several of the materials. In addition to the material study, a Ta-ITO bi-layer coating was investigated for use on BPPs with promising results. The dual effect of Tantalum and Indium Thin Oxide kept the contact resistance much lower than for bare titanium.

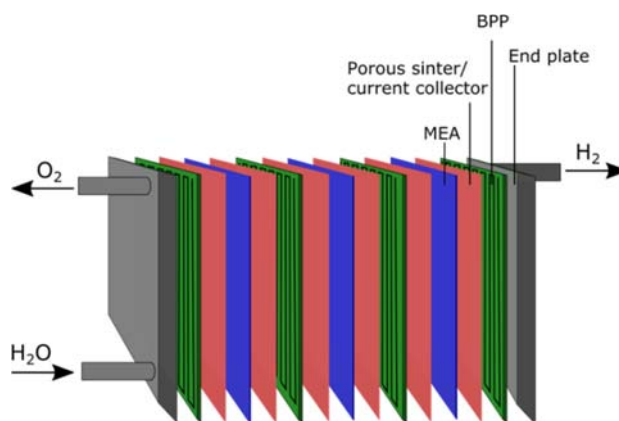


Figure 2: PEMWE stack

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## Critical Raw Materials Free Low Temperature Electrochemical Reduction of CO<sub>2</sub> to Methanol (LOTTER.CO<sub>2</sub>M)

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Low temperature electrochemical utilization of CO<sub>2</sub> has been strived for more than 60 years. Through funding from the Horizon 2020 programme, the project LOTTER.CO<sub>2</sub>M will develop advanced, low-cost electro-catalysts and membranes for the direct electrochemical reduction of CO<sub>2</sub> to methanol by low temperature CO<sub>2</sub>-H<sub>2</sub>O co-electrolysis. The produced methanol is intended as chemical feedstock or for effective chemical storage of renewable energy. The project aims at demonstrating high-efficiency CO<sub>2</sub> utilization at a real-life industrial environment.

The project consortium consists of a strong collaboration of partners (see bottom of page) covering the entire supply-chain from material development and upscaling, component production, system design and manufacturing as well as a demonstration at TRL 5. Thus the LOTTER.CO<sub>2</sub>M project brings together university researchers, various industries, research institutes and end users to ensure the success of the undertaking.

In order to realize this ambitious promise, the performance of the new materials will need to meet the highest standards. For the critical raw material free catalyst at the anode side a target of >50 A/g at 1.5 V/cell must be met, while the cathode catalyst need to exceed a CO<sub>2</sub> conversion rate of >60%, and a selectivity of >90% towards methanol production. A significant increase in durability under intermittent operation is also targeted in the project through several stabilization strategies to achieve a stack degradation rate of <1%/1,000 h.

The developed low-temperature CO<sub>2</sub> conversion reactor will offer fast response (frequency >2-5 Hz) to electrical current fluctuations typical of intermittent power sources and a wide operating range in terms of input power, i.e. from 10% to full power in less than a second. Such aspects are indicative of an excellent dynamic behaviour as necessary to operate with renewable power sources.

Field-testing of the co-electrolysis system in an industrial relevant environment will enable evaluation of the commercial competitiveness and the development of a forward exploitation plan.

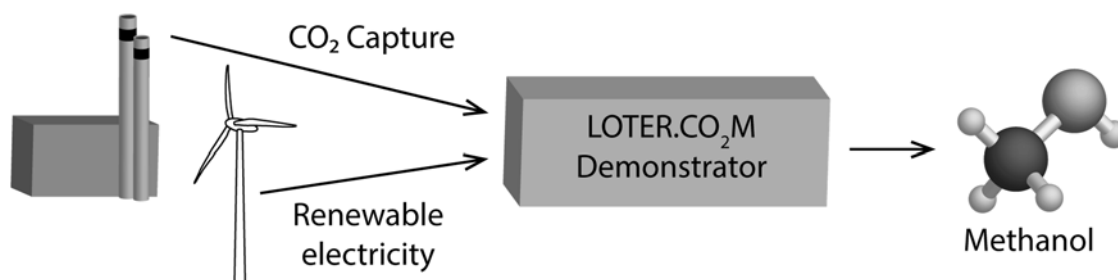


Figure 25: Schematic of the concept of methanol production from CO<sub>2</sub> by-products and excess renewable electricity through a high-efficiency electrolyser stack.

### Acknowledgement

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### List of partners:

Deutsches Zentrum fuer Luft- und Raumfahrt EV, Consiglio Nazionale delle Ricerrche, Hydrogenics Europe NV, IRD fuel cells A/S, EWE-Forschungszentrum für Energietechnologie e.V., Universitat Politecnica de Valencia, Johnson Matthey PLC, Danmarks Tekniske Universitet, RWE Power AG and NV Bekaert SA

Abstract No 161

## **Best Practices Benchmarking Framework Development for Low and High Temperature Electrolysis**

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The urgent and growing demand for large scale, sustainable hydrogen production drives the need for more detailed technical roadmaps, in order to focus on critical path R&D across technology options and build a progression from near term to long term technology deployment. The US DOE's Benchmarking Advanced Water Splitting Technologies project aims to contribute to this strategic roadmap development through the creation of a balanced portfolio of standards and protocols for materials testing, from screening of fundamental material characteristics, to device or system level benchmarking in controlled and real world operating conditions. Engaging a broader community, we aim to create a comprehensive Best Practices framework, so comparisons between devices from different research groups can be made in future. This presentation provides the status of the current materials standards, advanced characterization techniques, best practices and testing protocols development for both low and high temperature electrolysis technologies.

## Optimal sizing of H<sub>2</sub>-based hybrid EES in remote areas: the case study of Ginostra, Italy

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The exploitation of local Renewable Energy Sources (RES) can reduce, or even replace, diesel engines that are currently used to cover the electric load of isolated remote areas. Electrical Energy Storage (EES) technologies are key system components of remote micro-grids to match load and supply. Intermittent RES coupled to a H<sub>2</sub>-based storage system can thus represent a reliable, cost-effective and low carbon alternative to fossil fuel usage. In the framework of the EU project REMOTE, four different H<sub>2</sub>-based Power-to-Power (P2P) systems will be installed in isolated areas across three different countries (Italy, Greece and Norway). We focus in this work on the demo that will be installed in Ginostra, located on the island of Stromboli (IT). PV panels will be integrated with an innovative storage system comprising of Li-ion batteries and hydrogen. An alkaline electrolyzer will be used for H<sub>2</sub> production from surplus RES energy, while a PEM fuel cell will generate electricity back during RES shortage. The battery bank acts as complementary shorter term buffer.

Based on meteorological and electrical load data from the Ginostra site, a techno-economic analysis of various system configurations has been performed. PV panels and diesel generators were considered as electric power sources; whereas batteries and hydrogen were adopted as means of energy storage. Different technology combinations of these components were analysed to identify the most economically feasible option in terms of Levelized Cost of Electricity (LCOE). The heuristic genetic algorithm (GA) technique was used for the optimal sizing of the various components of the power system with the aim of minimizing the LCOE. The complete (i.e., 100%) satisfaction of the electrical load was set as constraint within the optimization routine. The main objective is to prove the competitiveness of the H<sub>2</sub> P2P system.

Results show that energy storage is required for a better usage of local RES sources, thus reducing the final cost of electricity. As shown in Figure 1, when no diesel generator is available for technology mix, energy storage hybridization (i.e., battery plus hydrogen) results in the lowest LCOE of 0.78 €/kWh, compared to 0.94 and 1.02 €/kWh for the system with only battery and hydrogen, respectively. In particular, the hydrogen technology was found to be useful as a means of seasonal energy storage, thus providing power in the summer period during which a higher electrical load demand occurs because of tourism.

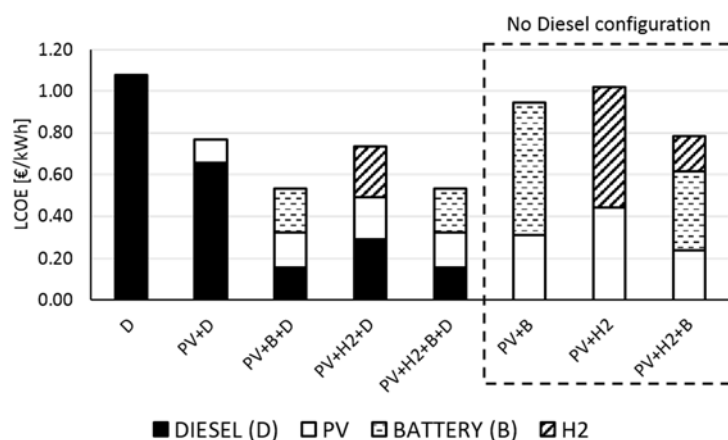


Figure 26. LCOE for the different configurations of the stand-alone system



## Direct Membrane Deposition in Anion Exchange Membrane Water Electrolysis

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Water electrolysis based on anion conducting polymers is a very promising concept for hydrogen production without the demand for expensive platinum group metals or titanium. Novel membrane materials exhibit improved stability towards elevated temperatures and alkaline media.<sup>1</sup> These could allow for high-performance water electrolyzers with the ability to compete against the established acidic PEM systems as a low-cost alternative.

The nature of the material is not the only factor. It has been shown that the MEA performance is strongly influenced by the fabrication method and the resulting interface structures.<sup>2</sup>

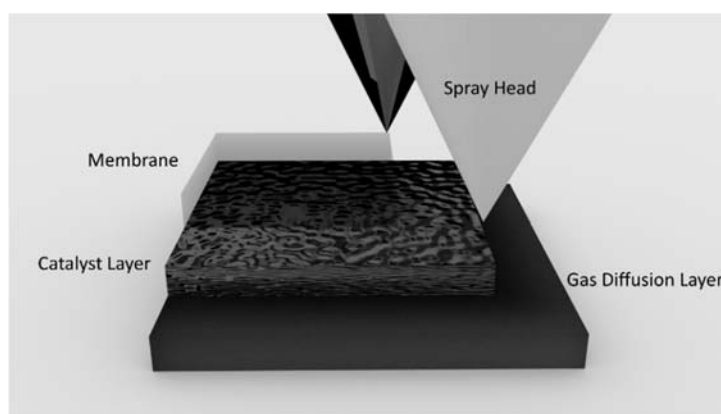


Figure 27: DMD process: Catalyst layer and membrane are deposited subsequently onto a gas diffusion layer in a spray-coating process.

The direct membrane deposition (DMD) process (Figure 27) is an industrially scalable additive manufacturing technique. In two simple spray-coating steps, which can be performed with a single device, catalyst layer and membrane are deposited sequentially onto a gas diffusion layer. It is thus possible to circumvent cumbersome decal transfer steps as used e.g. in the catalyst coated membrane (CCM) process.<sup>3</sup>

DMD has already proved to be beneficial for fuel cells<sup>4</sup> and PEM electrolyzers<sup>5</sup> due to an improvement in mass transport or the reduction of contact resistances within the assembly. Moreover, it is possible to fabricate stable membranes with reduced thickness.

In this work, the DMD process is applied to anion exchange membrane water electrolysis and the benefit of this fabrication technique with respect to cell performance is investigated.

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## A new generation of bipolar plates for PEM fuel cells

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Bipolar plates are a key component within PEM fuel cell and electrolyser stacks, with roles such as electron transfer, fuel and waste transport, and stack support. However, stainless steel as a bipolar plate material suffers from poor corrosion properties, with increasing interfacial ohmic resistances across the cell and stack due to the formation of non-conductive oxide layers reducing voltage efficiency over time. These increases in resistance can be mitigated by coating the steel with a conductive and corrosion resistant material.

Previous work has shown that electrodeposited Sn is a viable coating material for bipolar plates (BPPs)<sup>1</sup>. A thin layer of Sn is electrodeposited onto a stainless steel bipolar plate (BPP) and joined (soldered) with a gas diffusion layer (GDL) material through hot-pressing at a temperature just below the melting point of tin. This process solders the GDL to the BPP through the Sn, providing an uninhibited conduction pathway and drastically reducing interfacial contact resistance. In addition, the Sn layer forms a passive oxide upon exposure to a PEM environment, preventing further corrosion whilst maintaining conductivity through GDL fibres that have infiltrated the Sn layer during hot-pressing (see figure 1)<sup>1</sup>. Very low ICR and good longevity after 200 hours of operation in a working fuel cell are obtained; however, accelerated ex-situ testing in dilute sulfuric acid solution (pH 5.5) revealed instability of the SnO<sub>2</sub> layer.

We propose that introducing an alloying element, in this case Bismuth, will increase the stability of the oxide formed and thus impede the degradation of the Sn coating. This work investigates that claim, by using a one-step deposition process to deposit Sn alloys containing up to 10 wt % Bi onto a stainless steel bipolar plate. Conventional electrochemical measurements such as chronoamperometry and cyclic voltammetry, as well as interfacial contact resistance and long term in-situ testing were performed to investigate the performance of this system.

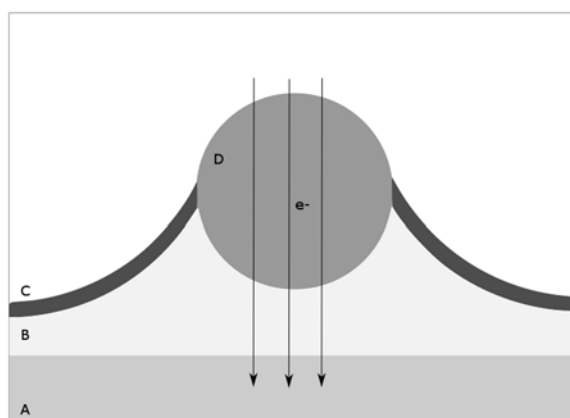


Figure 28. The combined Sn/GDL concept. A is the steel bipolar plate, B the deposited Sn, C the SnO<sub>2</sub> layer and D the carbon fibre. Electrons can move through the system without obstruction whilst the SnO<sub>2</sub> prevents further oxidation of Sn.<sup>1</sup>

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Abstract No 165

## Nickel-iron Hexacyanoferrate as a Novel Oxygen Evolution Catalyst for Alkaline Electrolysers

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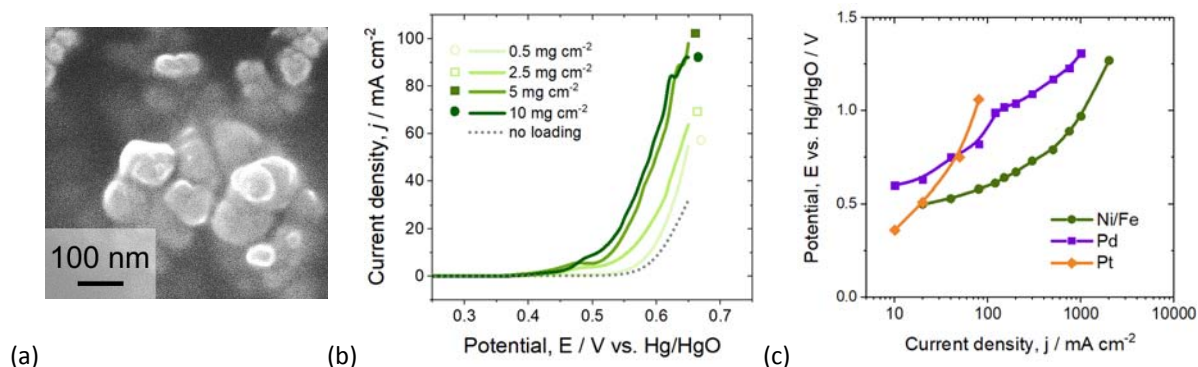
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For alkaline electrolysis, nickel meshes or powders have often been used as the primary component of the oxygen evolution catalysis, replacing the highly expensive but more efficient IrO<sub>2</sub> or Pt. <sup>[1]</sup> Nickel is often alloyed with iron to decrease oxygen evolution overpotentials, however, iron is inherently unstable at oxidising potentials at pH 14, dissolving to form Fe(VI) soluble species. <sup>[2]</sup> In this abstract, we present a low-cost stable combined nickel and iron oxygen catalyst that can be synthesised in a single step and has excellent oxygen evolution properties. <sup>[3]</sup>

The catalyst is synthesised by mixing NiSO<sub>4</sub>·6H<sub>2</sub>O and FeSO<sub>4</sub>·7H<sub>2</sub>O salts in a 4:1 molar ratio, and combining this solution with a K<sub>3</sub>[Fe(CN)<sub>6</sub>] solution under stirring at 1200 rpm at room temperature. Afterwards, the solution is centrifuged to separate the Ni/Fe catalyst, and oven dried at 60°C. A green nanopowder results (Figure 1a). It was found by FTIR and XRD that the chemical composition of the catalyst is Ni<sub>5</sub>Fe[Fe(CN)<sub>6</sub>]<sub>4</sub>. <sup>[3]</sup>

The Ni-Fe hexacyanoferrate catalyst can be subjected to highly oxidising currents with no change in structure. It shows superior performance to many other catalysts including precious metals. To make electrodes catalysed with Ni<sub>5</sub>Fe[Fe(CN)<sub>6</sub>]<sub>4</sub>, the catalyst powder was mixed with a corrosion-resistant carbon powder, and hot-pressed between a carbon cloth and a nickel mesh. To be certain that the catalytic performance was due to the catalyst, a blank electrode was produced that only contained the nickel mesh and carbon. There was a clear difference, with the Ni-Fe hexacyanoferrate producing a much greater oxygen evolution current at up to 0.65 V vs. Hg/HgO for the blank electrode (Figure 1b). Also, the Ni-Fe catalysed electrode outperformed a commercial oxygen evolution electrode composed of Pt/C, and an in-house Pd/C catalysed electrode, with lower overpotentials at current densities > 20 mA cm<sup>-2</sup> when subjected to half-hour runs at increasing current densities (Figure 1c).



**Figure 1.** (a) SEM image of Ni<sub>5</sub>Fe[Fe(CN)<sub>6</sub>]<sub>4</sub> catalyst particles, (b) cyclic voltammetry showing oxygen evolution at hot-pressed electrodes with different catalyst loadings (c) oxygen evolution potential at different current densities compared to Pt (commercial) and Pd (in-house) catalysed electrodes.

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## **Coupling a renewable energy source to a PEM electrolyser using a resonant converter**

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Renewable energy sources are receiving much attention due to their clean and sustainable properties. One of the challenges with these renewable energy sources is that they do not provide stable and constant power. Instead, they are only able to provide intermittent power when certain conditions are met. High efficiency DC/DC converters, capable of operating at a wide range of input voltages, are required to maximise the energy storage from these renewable sources. An LLC resonant converter, capable of delivering up to 1-kW at an average efficiency of 94.3%, was implemented and tested by using photovoltaic panels as the renewable energy source and described in this communication. Maximum Power Point Tracking (MPPT) was implemented to maximize the amount of energy extracted from the solar panels. This configuration is simple to implement, cost effective, very environmentally friendly and will also ensure a more secure source of energy as there is less risk of depletion and price volatility that can result in economic pressures<sup>1</sup>. Direct conversion to hydrogen from a renewable energy source provides both a method of energy storage that produces no harmful by-products or gasses<sup>2,3</sup> while achieving a high total system efficiency. A microprocessor was used to monitor the input and output voltage as well as the current and can then use this information to calculate the total amount of energy extracted from the renewable source while also providing real time short-circuit protection. The system also calculates the amount of hydrogen produced in normal litres as well as the total weight of hydrogen produced (in grams). This information, which is logged to a computer system and can then be used to estimate the amount of energy (in kWh) stored in the form of hydrogen.

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Abstract No 167

## Electrochemical Behaviour of the IrO<sub>x</sub> Electrode at High Overvoltage in PEMEC

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Practical polymer electrolyte membrane electrolysis cells (PEMEC) using IrO<sub>2</sub> as the oxygen evolution reaction (OER) electrocatalyst usually have linear iV curves in the cell voltage region where current densities of practical interest are obtained, i.e. above ca. 0.4 A cm<sup>-2</sup> (cell voltage > ca. 1.55 V). This is commonly taken as evidence of a totally dominating ohmic resistance in this region [1]. Therefore, the fact that electrochemical impedance spectra (EIS) measured in this potential region show at least two significant arcs, one at middle frequencies around 100 Hz and another at low frequencies around 1 Hz, is very surprising [1]. This fact combined with low resistance of PEMECs means that the middle and low frequency arcs reflect very high capacitances in parallel with small resistances. It also follows that the total sum of resistances under the arcs in the EIS is constant, equal to the slope of the iV-curve, i.e. the differential resistance of the cell in the linear iV region. Next surprise is that the ratio between the resistances associated with the middle and low frequency arcs changes significantly while their sum is constant.

A hypothesis that is able to explain the observed behaviour will be presented. Important parts are: 1) the dominating contributions to the differential cell resistance above ca. 0.4 A cm<sup>-2</sup> originates from the Nafion electrolyte; 2) only the oxygen electrode and the Nafion electrolyte contribute significantly to the impedance spectrum of the cell; 3) the capacitances reflected by the middle and low frequency arcs originate from the oxidation/reduction during the ac-cycles of IrO<sub>2</sub> + H<sub>2</sub>O ⇌ IrO<sub>2</sub>OH + H<sup>+</sup> + e<sup>-</sup> and IrO<sub>2</sub>OH + H<sub>2</sub>O ⇌ IrO<sub>2</sub>(OH)<sub>2</sub> + H<sup>+</sup> + e<sup>-</sup>, respectively. In other words, the capacitances originate from the equilibria Ir<sup>4+</sup>/Ir<sup>5+</sup> and Ir<sup>5+</sup>/Ir<sup>6+</sup>. This means that the IrO<sub>x</sub> electrocatalyst, the electrochemically active oxygen electrode material, significantly changes composition (oxygen content) with change in the electrode overvoltage. The interplay between the changing OER catalyst chemistry and the OER kinetics will be discussed.

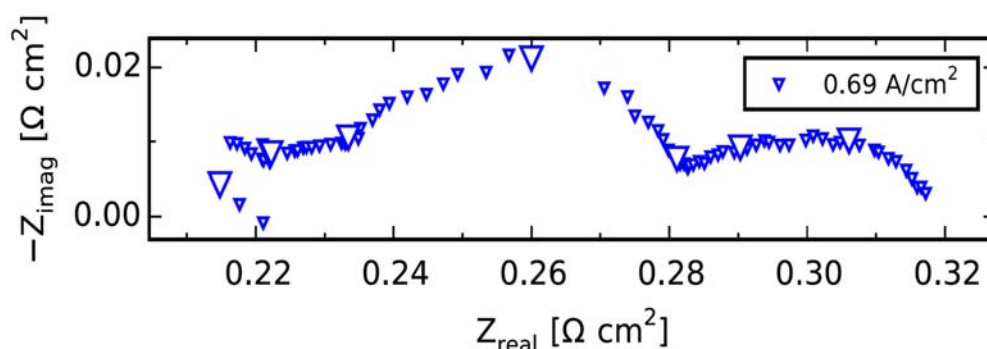


Fig. 1. Nyquist representations of EIS of a PEMEC operated 0.69 A cm<sup>-2</sup> (ca. 1.7 V) at 61 °C and ambient pressure. Frequencies of 100 kHz, 10 kHz, 1 kHz, 100 Hz, 10 Hz, 1 Hz and 0.1 Hz are marked with larger markers [1].

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## Homogeneous electron mediator and packed-bed scrubber assisted electrolytic reduction of CO<sub>2</sub>

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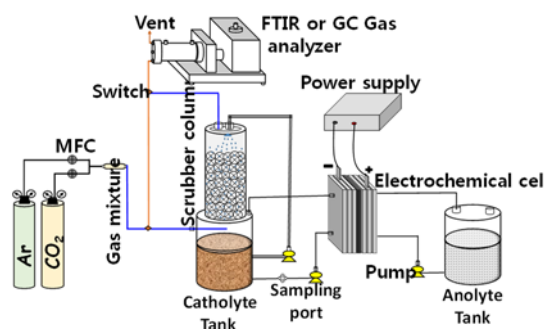
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Electrochemical reduction of carbon dioxide (CO<sub>2</sub>) into value-added chemicals as a feedstock and provides a pathway for the utilization and (temporary) storage of electrochemical energy is a promising strategy. In order to produce value added product with high efficiency, many strategies were approached such as over potential reduction by selecting a suitable electrode as electrocatalysts [1], solubility of CO<sub>2</sub> medium[2], and gaseous product formation [3]. In order to form gaseous product generation, homogeneous electrocatalysts showed considerable advantageous [4]. Moreover, except few publications, all the CO<sub>2</sub> reduction experiments use H-type divided cell configuration instead of prototype flow through electrolytic cell [5].

We have vast experience in generation of homogenous electron mediators using membrane divided flow through electrolytic cell either oxidative or reductive electron mediators [6] in air pollutants removal process. Very recently, we found a cobalt based complex that has high solubility in alkaline medium and stabilized its active Co(I) state, which was produced at Ag electrode with less over potential during Nafion324 membrane divided electrolysis. At the same time, the oxidation/reduction potential (ORP) value of the produced Co(I) electron mediator in solution found to be -1.270 V (vs Ag/AgCl). In order to assist the solubility of gas and electron mediator containing liquid, a packed-wet scrubber column was attached on the top of the reductive electron mediator generation tank (Figure1), where the gas and liquid electron mediators react by counter flow pattern under optimized applied current density, gas and solution flow rate,

We also found combined reaction products in gaseous form such as formaldehyde, methanol, and acetonitrile that are identified using online FTIR gas analyzer during CO<sub>2</sub> reduction. Also, hydrogen formation was identified by GC analysis that support the formation of hydrocarbons from carbon source. The electrolytic regeneration of Co(I) electron mediator was confirmed by consecutive and long term electrolysis experiments. The presentation will be paid more attention on the found product mole ratio with feed CO<sub>2</sub> concentration and discussion on its application in energy storage field.

Figure 1 Schematic representation of Nafion324 membrane divided electrolytic cell with packed-wet scrubber to facile CO<sub>2</sub> reaction to form useful products



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## Highly proton conducting hydrocarbon membranes for PEM Electrolysis

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Here, we present a family of membranes making use of the amazing properties of highly sulfonated poly (phenylene sulfone)s as their key constituent. It is the particular electronic structure of this polyelectrolyte and its high density of ionic groups which leads to a combination of high chemical stability [1,2], superior proton conductivity [3-5] and low electroosmotic water drag [3]. Since pure sulfonated poly (phenylene sulfone)s show salt-like behaviour, i.e. brittleness in the dry state and exaggerated swelling or even dissolution in water [2-5], we have used these polyelectrolytes as constituents of polymer blends for forming robust membranes.[6] These membranes combine the advantageous properties of pure poly (phenylene sulfone)s with mechanical robustness needed for their application in PEM electrolyzers. As compared to PFSA membranes (e.g. Nafion), intrinsically they don't show any HF release, while offering a low gas-crossover even at high temperature and mechanical properties which are almost independent of temperature ( $T < 180^{\circ}\text{C}$ ).

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## A 3D-printed Parallel Plate Membrane-less Electrolyser Cell for H<sub>2</sub> Production

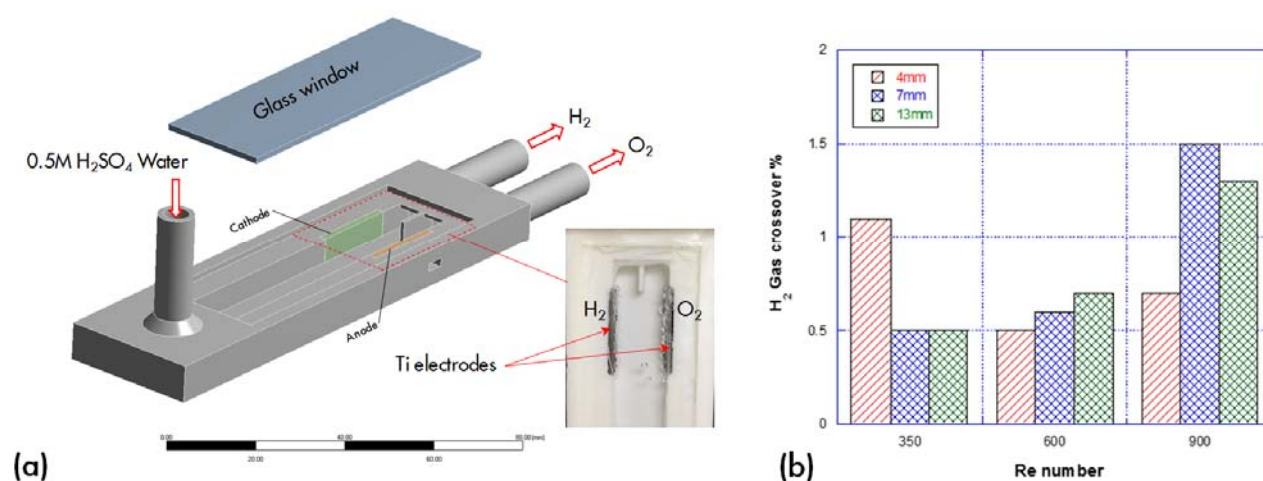
V. Narasaiah<sup>a</sup>, S. Das<sup>a</sup>, S. Govindarajan<sup>a</sup>, J.T. Davis<sup>b</sup>, X. Pang<sup>b</sup>, D.V. Esposito<sup>b</sup>, A.D. Harvey<sup>a</sup>

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Electrolytic hydrogen production is a promising process with zero emission of CO<sub>2</sub> to the environment; if the power required for the electrolytic process comes from the renewable energy source like solar energy, wind energy etc. The commercialization of the water-splitting devices is obstructed by the availability of low-cost electricity and low-cost stable ion conducting membranes typically used in fuel cells. The main focus of the present work is to develop and study parallel plate membrane-less electrolyser cell where the products (H<sub>2</sub> and O<sub>2</sub>) separated by the hydrodynamics.



**Figure 1:** (a) CAD geometry for 3D printed electrolyser body, glass viewing window, and two electrodes. Inset shows the actual electrolyser cell with H<sub>2</sub> and O<sub>2</sub> bubble formation. (b) H<sub>2</sub> purity for different flow Reynolds number and channel width.

In the present electrolyser cell two parallel electrodes are embedded in a 3D printed cell-body (Fig. 1-a). A small separator is used to separate the formed H<sub>2</sub> and O<sub>2</sub> bubbles. Pt deposited Ti plate is used as both cathode and anode for electrolysis experiment whereas 0.5 M H<sub>2</sub>SO<sub>4</sub> solution is used as an electrolyte. The distance between the electrodes is varied from 4 mm to 7 mm and the Reynolds number of the electrolyte flow is varied from 350 to 900. A constant voltage difference is applied. We report that the current density is also not very sensitive with flow Reynolds number, however varies significantly with the channel width (60 mA/cm<sup>2</sup> to 160 mA/cm<sup>2</sup>) mainly due to the ohmic resistance of the electrolyte. Product purity was investigated using gas chromatography (GC) of the collected product. We report the maximum H<sub>2</sub> crossover of 1.5% (Fig. 1b) which is not very sensitive with the channel width and Reynolds number. The present experimentations provide a valuable understanding of bubble dynamics in fluidic electrolysis cells for further process and design optimization.



Abstract No 171

## Systematic Study on Electrodeposition of NiMo alloys as Electrocatalysts for Hydrogen Evolution Reaction (HER) in Alkaline Medium

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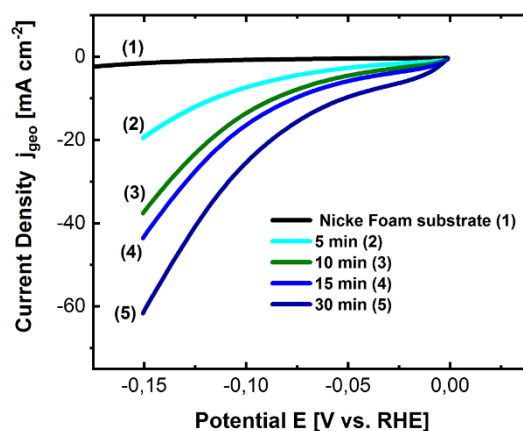
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In this work, the electrochemical deposition of NiMo alloys from aqueous deposition bath at constant potential onto the Nickel foam as a substrate will be presented. Variant parameters of deposition have been studied such as electrochemical treatment of the substrate before the deposition process, the dimension of the substrate, time of the deposition, temperature of the deposition and stirring during the deposition. Moreover, the post-treatment of the deposit after the deposition in terms of heat treatment in argon atmosphere and oxidation either chemically by leaving it in open atmosphere or electrochemically by treatment in a high positive potential window have been studied. The aim is to study the effect of all these parameters on the catalytic activity of the prepared alloys towards the hydrogen evolution reaction (HER) in alkaline medium and therefore optimizing the deposition process.

The catalysts were characterized electrochemically towards their catalytic activity for HER in 5 M KOH by electrochemical methods and structurally by Raman Spectroscopy to get information about the structural features of the materials. The electrochemical measurements showed a dependence of the activity of catalysts (expressed in reduction current ( $j_{\text{geo}}$  mA cm<sup>-2</sup>) obtained at -0.1 V vs. RHE and the charge transfer resistant  $R_{\text{ct}}$  obtained from EIS ) on the temperature of deposition in the studied range of 15-35 °C. Increasing the temperature could enhance the diffusion of metal ions and therefore fast transport of the ions into the substrate resulting in high loading and more homogenous deposit. The same trend has been observed by increasing the deposition time from 5 min to 30 min. The obtained higher activity could be attributed to the higher electrochemical surface area as established from the double layer capacitance values for the different electrodes. Furthermore, it has been also observed that the substrate dimension plays an important role in the homogeneity of the deposit. The substrate with small dimension showed extremely good homogeneity of the deposit on the substrate which consequently affected the activity of the catalysts. Better homogeneity on the large dimension substrate is limited by longer deposition time and higher deposition temperature. Moreover, the post-treatment of the deposit electrochemically at a higher positive potential (1.3 V vs. RHE) and/or at higher temperature or by exposing to air atmosphere at room temperature reduce the activity of the catalysts to a higher extent as a result of oxide formation under all these conditions. The study could provide a guide for optimization the preparation of high catalytic active NiMo alloys for HER to be used as cathode catalysts in alkaline water electrolyzers.

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LSV of NiMo alloys prepared at different deposition time on small dimension substrate (effective deposition area 1.0 cm x 1.0 cm) in 5M KOH at scan rate 5 mVs<sup>-1</sup>. Deposition Temperature 25 °C.



Abstract No 172

## Making fuel on Mars: Methane synthesis from Martian-derived CO<sub>2</sub> and H<sub>2</sub>O using a Sabatier Electrolyzer based on proton-conducting ceramics

Duc Nguyen, Long Q. Le, and Neal P. Sullivan

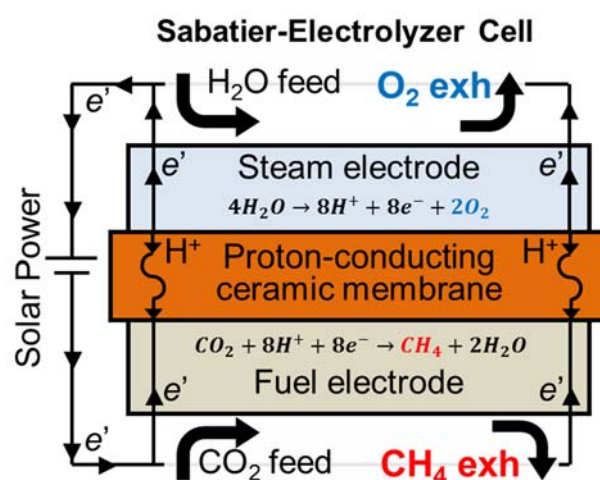
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We present our development of proton-conducting ceramic materials to synthesize methane from CO<sub>2</sub> and H<sub>2</sub>O feed streams. The National Aeronautics and Space Administration (NASA) seeks such technology to enable manned missions to Mars. The CO<sub>2</sub> and H<sub>2</sub>O are in great supply on The Red Planet; solar power could be harnessed to convert these feedstocks into fuel to sustain human life and enable permanent bases.

NASA's current approach features a two-stage process that starts with hydrogen synthesis through solar-driven water electrolysis with PEM technology. Following H<sub>2</sub> synthesis, the product hydrogen is then mixed with CO<sub>2</sub> drawn from the Martian atmosphere, and then the two are converted to CH<sub>4</sub> in a downstream Sabatier reactor featuring a ruthenium catalyst. Our work seeks to combine the two processes into a single reactor based around proton-conducting ceramics.

As shown in Figure 1, H<sub>2</sub>O and CO<sub>2</sub> are fed to the opposing electrodes of a protonic-ceramic electrolysis cell. The steam is electrolyzed, and as the product O<sub>2</sub> is exhausted from the cell, the product protons are driven across the protonic-ceramic membrane. These protons arrive at the methane electrode, where they react with CO<sub>2</sub> to form methane and H<sub>2</sub>O. Such process intensification can match the exothermicity of CO<sub>2</sub> hydrogenation with the endothermicity of H<sub>2</sub>O electrolysis, promoting thermal balance and high efficiency. Additionally, process intensification can reduce complexity and mass of fuel-generating equipment, and lessen the challenge of interplanetary travel.

NASA researchers term this use of Martian-derived materials to sustain life as "In-situ Resource Utilization" (ISRU). CSM researchers have previously demonstrated encouraging results with proton-conducting ceramics for a number of applications, including electric-power generation and fuels synthesis. In this presentation, we will review our efforts to extend development of protonic-ceramic cells to the Sabatier Electrolyzer shown in Figure 1. The cells feature a BaCe<sub>0.2</sub>Zr<sub>0.6</sub>Y<sub>0.2</sub>O<sub>3-d</sub> (BCZY26) electrolyte, a Ni-BCZY26 fuel electrode support, and a BaCo<sub>0.4</sub>Fe<sub>0.4</sub>Zr<sub>0.1</sub>Y<sub>0.1</sub>O<sub>3-d</sub> (BCFZY) steam electrode. Results will be presented for CO<sub>2</sub> conversion and CH<sub>4</sub> selectivity under electrolysis operation.



**Figure 29:** Concept of the Sabatier Electrolyzer based on proton-conducting ceramics. The device converts H<sub>2</sub>O and CO<sub>2</sub> feed streams into CH<sub>4</sub> and O<sub>2</sub> products to help sustain life on Mars.

## Study of design parameters in MEA for AEM electrolysis

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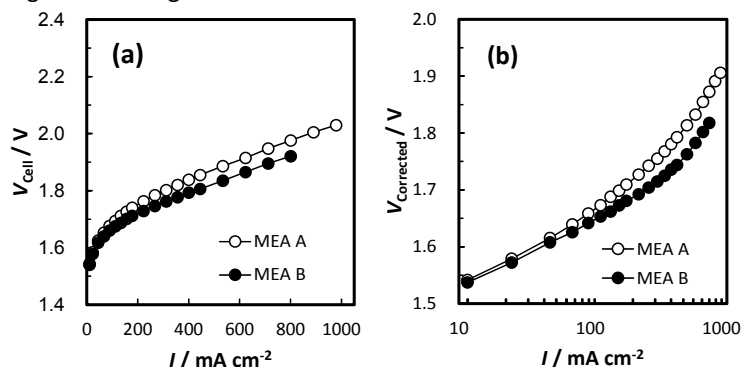
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With usage of mild alkaline water, anion exchange membranes (AEM) have become much attractive for the application of water splitting system [1, 2]. Its durability is being proved as the long term performance has been reported from several institutes [1-8]. Although AEM electrolyser is highly cost competitive with the other systems such as PEM and alkaline electrolyzers [8, 9], its operational cost should be further decreased and so should its electric consumption.

Reported data have demonstrated that the ohmic resistance of AEM is sufficiently low for the electrolysis; however, the other overpotential is not as low as it of PEM [10]. Figure 1 shows one of our primitive results. Anode catalyst was ACTA 3030 (Acta SpA) [1] and it was splayed on a Ni foam with loading of about 4 mg/cm<sup>2</sup>. An aqueous solution of 10 % potassium carbonate was circulated in the anode side. After subtraction of the ohmic overpotential ( $iR$ ), the corrected cell voltage is plotted as a function of logarithm of current (Figure 1.b). Two slopes in each curve are observed. We speculate the first moderate slope less than 50 mA cm<sup>-2</sup> represents the activation overpotential though the second steep slope is quite obscure. It could be a part of the activation overpotential but using less active area. Or it could be local dilution or a veiled concentration overpotential.

Nevertheless, the second slope of MEA A starts from smaller current than the one of MEA B. During its preparation, the anode catalyst of MEA B was splayed on a Ni foam in longer time than MEA A's was. By this way, in each splaying step of MEA B, more anode ink might dry before the next ink was splayed on, and more amount of catalyst might distribute closely on the splayed surface of the Ni foam. Considering these data sets, structure of MEA may affect the overpotential especially at large current region.

In the presentation, we will present the performance of various MEAs obtained with different techniques. We have been intentionally developing tractable parameters to change the structure of MEA. Performance of several anode catalysts will also be presented, which may or may not change the first slope only. At present, we attempt to elucidate the nature of the second slope thereby and ultimately to reduce both first and second slopes.



**Figure 1:** I-V curves of MEA A and MEA B (a) and their  $iR$  corrected plots of voltage vs. logarithm of current (b).

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## Stable Reference Electrode in Polymer Electrolyte Membrane Water Electrolyzer for Three-electrode Measurements

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Polymer electrolyte membrane water electrolyzer (PEMWE) is by default a two-electrode device. One electrode is attached to the anode and another to the cathode. This means that the result of any measurement in a PEMWE is the combination of both electrodes. For more accurate electrode material studies, individual study of electrodes would be a great benefit. In order to study electrodes individually, a reference electrode, acting as the third electrode, is required. In fuel cells, various three-electrode setups have been developed employing conventional reference electrodes, dynamic hydrogen electrodes (DHEs) and pseudo-reference electrodes [1]. For PEMWEs, only few such designs have been developed [2]. Conventional reference electrodes are usually connected by *i.a.* a salt bridge to the cell and are therefore considered external type electrodes. However, DHEs and pseudo-reference electrodes are commonly configured internally. Both setups often require special cell designs and therefore cannot be used in a common test station.

We have developed a stable reference electrode that can be implemented in a regular single cell PEMWE testing station. The stabilisation of the potentials is shown in Fig. 1 when  $500 \text{ mA cm}^{-2}$  is applied to the cell. Full cell stabilises after one minute and is very stable, as expected. Anode stabilises also fast, under five minutes and the cathode in 15 minutes. We use the three-electrode configuration to record electrochemical impedance spectra (EIS) of the cathode and the anode, separately. The cell is subjected to a start stop cycling procedure and the aging of each electrode is studied by EIS. As a result, we see which features of the electrodes change during a long-term aging test.

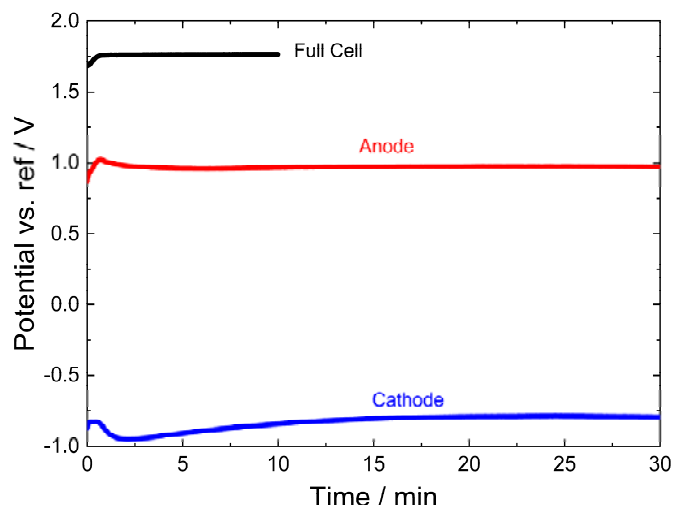


Figure 1. The stabilisation of the voltage of the full cell and the potentials of the anode and the cathode after applying  $500 \text{ mA cm}^{-2}$ .

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Abstract No 175

## **Boosting the green transition through electrolysis – The Hydrogen Valley perspective**

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HyBalance, an FCH-JU and [Energynet.dk](https://www.energynet.dk) co-funded project, illustrates how electrolysis can enable the storage of cheap renewable electricity from wind turbines, help balance the grid and downstream renewables into fossil dependent sectors such as transportation. The HyBalance project will develop a model in which the operation of the hydrogen plant is simulated on hourly basis in relation to hourly power prices, the need for balancing the electricity grid and the hydrogen demand. The HyBalance plant is located in Denmark, a global frontrunner in integrating wind into the energy production and was inaugurated in September 2018. It is the forerunner of large electrolyser demonstration projects and the first to reach Megawatt-scale.

Green hydrogen production provides multiple opportunities, which will be presented in this presentation:

- **Grid balancing**

A main perspective of the HyBalance project is to showcase the production of hydrogen, using fluctuating energy sources. This presentation will present a use case of hydrogen in the Danish Energy system and will also present a possible utilisation of the Energy Reserve in Denmark.

- **Production of green methanol and green ammonia**

The successful inauguration of the megawatt-scale electrolyzers is the main enabler of production of green synthetic fuels for the transport industry. BEV's and FCEV's will provide a large amount of this transition, but not all transportation can be electrified – e.g. bunker ships and airplanes. In these transportation areas, green methanol and ammonia are seen as the most likely fuels to boost the green transition. Hydrogen Valley Denmark have just been awarded a large grant from the Danish authorities for building the first green methanol reactor.

- **Energy storage**

The area around Mariager Fjord in Denmark is one of the most suitable areas for underground gas storage. The perspectives of cavern storage of hydrogen is enormous – a single cavern can provide the same capacity as million of large-scale batteries. The potential of the Hydrogen Storage will be utilised as electrolyzers reach large scales of +100MW

## Functional ceramic 3D printing of enhanced energy devices: High surface Solid Oxide Cells working as electrolyzers

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In the last decades the additive manufacturing technologies arise in the technological field due to their capability of design and produce pieces with high degree of freedom, minimising the wasted material and consequently lowering the cost of the final devices. The technology is well established and diffused on the market for polymeric materials and starts to be ready for metallic materials, while is still under development for ceramic materials [1-2]. The development of ceramic 3D printing technology is constrained by the high fusion temperature and low machinability of this kind of material. Nevertheless, ceramic materials arise high interest in the energy sector and particularly in the future scenario based on hydrogen technologies [3]. Chemical energy storage systems, such as electrolyzers, will be crucial for the market penetration of the H<sub>2</sub> based technologies.

Solid Oxide electrolyser cells (SOEC), completely based on ceramic materials, present high efficiency (up to 75%) in H<sub>2</sub> yield compared to other type of electrolyzers and the capability of store not only hydrogen but also a wide variety of synthetic fuels by co-electrolysis plus the catalytic processes [4]. In this type of cells the electrolyte is made of a ceramic material with a high ionic conductivity at high temperature, the low machinability of this kind of material has constricted the design of the final devices into mainly two shapes: the planar and the tubular one. The use of additive manufacturing to produce functional ceramic devices such as SOEC allows to produce freeform devices with a reduction of manufacturing cost and wasted material. The first works published regard the application in Solid Oxide Fuel Cells, [5] while in this work we propose their use on electrolysis mode. Micro structuration of the active surfaces, which cannot be obtained with traditional manufacturing techniques, increases the active area of the electrolytes and consequentially the performance of the electrolyser. Moreover, the freedom of design achieved by 3D printing technologies can be applied to improve other features of the SOEC like the sealing or stacking of the cells.

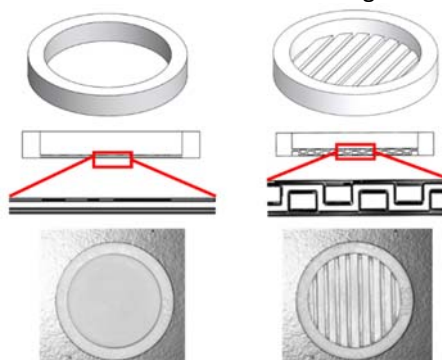


Figure 30: 3D printed electrolytes, reference on the left, increased area with micro structuration on the right.

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Abstract No 177

## Nanostructured Iron Nickel as an Efficient Electrocatalyst for the Oxygen Evolution Reaction

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Designing cost-efficient and durable electrocatalysts toward oxygen evolution reaction (OER) have been of vital significance for the commercial development of various renewable energy systems. Numerous electrocatalysts have been developed in the past decades to accelerate the OER process. Up to now, the first-row transition metal-based compounds are in pole position under alkaline conditions, which have become subjects of extensive studies. Among them, the most accepted catalytic activity trend is reported as Ni > Co > Fe > Mn [1]. Mixed Ni and Fe oxide electrocatalysts are among the most active binary metal oxide OER catalysts in alkaline electrolyte. The role of Fe in these catalysts is still debated; however, Fe is clearly a critical component for high activity [1].

In this regard, Ni and NiFe nanoparticles as an efficient oxygen evolution reaction (OER) electrocatalyst for electrochemical water splitting application have been synthesized by the thermal reduction procedure consists, the reaction of [Ni(acac)<sub>2</sub>] with oleylamine (OA) and trioctylphosphine (TOP) reactants. OA is the reductant and thus controls the nucleation rate, meanwhile TOP provides a tunable surface stabilization through coordination on the Ni(0) surface. This synthesis results in monodispersed nanoparticles (Fig. 1) around 12nm [2]. Ni and NiFe nanoparticles were annealed in different atmospheres and the effect of annealing on the structure and electrocatalytic activity of nanoparticles were investigated (Fig. 2). Furthermore, the role TOP to OA ratio on nanoparticles size and their electroactivity have been delineated and systematically studied.

Moreover, we have investigated the influence of Fe impurities in the KOH electrolyte on the experimentally measured OER activity. Ni nanoparticles show different electroactivity in rigorously purified (Fe-free) and not purified 0.1 M KOH electrolyte. Obtained results reveal that in purified electrolyte Ni nanoparticles show no significant OER current until >400 mV overpotential, different from previous reports which were likely affected by Fe impurities. Also our studies shows that rotation of the electrode (convection) drastically increases the activity of the electrode in no purified electrolyte.

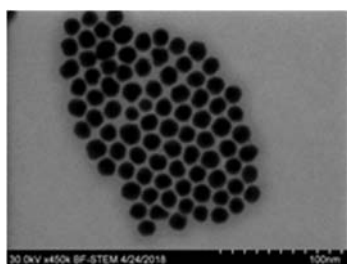


Figure 1. S(T)EM images of mono-dispersed Ni nanoparticles

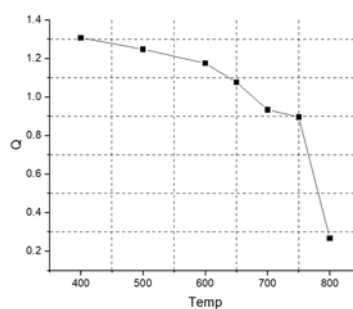


Figure 2. Effect of annealing temperature on the electrochemical surface area of Ni nanoparticles

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## A Passive Film in Motion

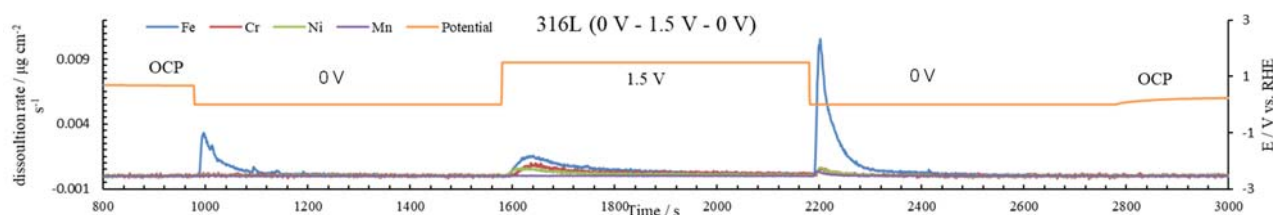
Sebastian Proch<sup>a</sup>, Manas Paliwal<sup>a</sup>, Jörgen Westlinder<sup>a</sup>, Peng Zhou<sup>b</sup>, Xuejie Li<sup>b</sup>, and Kevin Ogle<sup>b</sup>

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Stainless steels are scalable materials and, therefore, poised to address the “Terawatt Challenge”<sup>4</sup> and transform the current carbon into a future hydrogen economy. Moreover, from a corrosion perspective they are beautiful specimens, since their multicomponent nature introduces “transient transpassivity” leading to metal ion release upon potential changes for a couple of minutes and subsequent return to passive dissolution.<sup>5</sup> We have followed such “transients”, relevant for proton-exchange membrane fuel cells (PEMFCs), via atomic emission spectroelectrochemistry (AESEC), *i.e.*, an electrochemical flow cell coupled directly to an inductively-coupled plasma atomic emission spectrometer (ICP-AES)<sup>6</sup> (Figure 31). Steel dissolution can only be observed upon potential changes but cathodic dissolution at  $\pm 0$  V vs. RHE (reversible hydrogen electrode) can be found. **Error! Bookmark not defined.** AESEC is also suitable to investigate metal ion, especially Fe, dissolution at the cathode side of PEM electrolyzers. Iron release is detrimental since it can lead to severe electrolyzer membrane degradation due to its ability to catalyse Fenton-type reactions.<sup>7</sup> The goal of our research is to exploit the superior hydrogen embrittlement properties of stainless steels in electrolyzers.<sup>8</sup>



**Figure 31.** Simulated PEMFC anode transient (global hydrogen starvation). The curve has been recorded in aqueous 49 ppm  $\text{H}_2\text{SO}_4$  at 343.15 K and shows bright-annealed 316L stainless steel. The highest peaks in the AESEC (atomic emission spectroelectrochemistry)-traces correspond to Fe release upon potential changes (“transient transpassivity”). Especially, as  $\pm 0$  V vs. RHE are approached a second time (left side) cathodic iron dissolution is evident. This type of release is also relevant at the PEM electrolyzer cathode, however, since the electrolyzer cathode potential is enforced low it should be possible to remove this type of transient (transpassive) cathodic dissolution by proper electrochemical surface pre-treatments.

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Abstract No 179

## Utilizing low grade waste heat to produce hydrogen by reverse electrodialysis of ammonium bicarbonate

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The commercial renewable energy technologies mainly produce electric energy and intermittent power. The intermittency is balanced by utilizing energy storage. As storage technologies rely on optimal conversion efficiency and cost, a strong need for more chemical energy rises. We propose heat driven reverse electrodialysis (RED) technology with ammonium bicarbonate as salt for producing hydrogen. The study provides perspectives on the commercial feasibility of ammonium bicarbonate (AmB) RED for low grade waste heat (333 K - 413 K) to electricity conversion system.

The economic assessment includes parametric study and, scenario analysis of AmB RED system for hydrogen production. Various technical and, economic parameters including membrane cost, cost of heating, inter-membrane distance, membrane lifetime, and, residence time are studied. The results from the economic study suggests, RED system with membrane cost less than **3.12** €/m<sup>2</sup>, membrane life more than **7** years and a production rate of **1.13** mol/m<sup>2</sup>/h or more are necessary for RED to be economically competitive with the current renewable technologies for hydrogen production. In the present state, use of ammonium bicarbonate in RED system for hydrogen production is uneconomical. This may be addressed to high membrane cost, low (**0.6** mol/m<sup>2</sup>/h) hydrogen production rate and large (**1.28 x 10<sup>6</sup>** m<sup>2</sup>) membrane area required. From the scenario analysis it is clear that membrane cost and membrane life in the *“Present case”* regulates the levelized cost of hydrogen, LCH. In the *“Market case”*, and the *“Future case”* scenarios, the hydrogen production rate (which depends on membrane properties, inter-membrane distance etc.), cost of regeneration system and cost of heating regulates the levelized cost of hydrogen.



## Highly Efficient SOE System Design with Internal Steam Generation

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### Summary

A key element in the field of SOE system design is the energy-intensive vaporization of water. SOE systems are therefore mostly dependent on external steam, or heat sources. This might limit the application of SOE to certain dedicated industries. Supported by methods like pinch analysis and comprehensive system simulation, AVL developed highly efficient solutions for SOE systems to enable steam production internally, which makes future SOE systems more independent from external sources and therefore even more attractive for the industry by keeping its overall high efficiency of >80% (LHV based).

### Abstract

Power-to-X technologies offer high potentials concerning the realization of a highly integrated energy-sector landscape of the future. Emerging renewable electricity production with high stochastic characteristics together with targeted carbon dioxide emission reductions require appropriate solutions for energy storage, as well as the substitution of fossil-based- by renewable based fuels and chemicals. The high-temperature solid oxide electrolysis (SOE) technology meets those needs with its various application possibilities linking the electricity sector with the gas and energy storage sector, as well as with carbon dioxide emitting- and hydrogen/syngas demanding industry sectors. Compared to other power-to-X technologies, the SOE provides several promising capabilities, which include higher electrical efficiencies due to beneficial thermodynamic characteristics at elevated temperatures, the additional ability of direct conversion of CO<sub>2</sub> to produce pure CO or syngas (H<sub>2</sub>, CO) and related to those, a broad variety of application scenarios.

A central requirement of hydrogen-producing SOE systems concern the steam supply to the stack at high temperatures. Therefore, a common application focus relies on coupling with industrial processes which allow direct usage of excess steam or the usage of excess heat for steam production. However, for broad implementation of the technology it is necessary to find feasible and efficient stand-alone solutions to enable steam production internally.

To address these challenges, Pinch-analysis and comprehensive system simulations were conducted and process interface specification requirements of various SOE applications investigated. Process interfaces can differ significantly in terms of available heat, temperature levels and type of media (for input, as well as output interfaces of SOE). The conducted work aimed for finding highest efficient SOE layouts and integration solutions, while keeping the number of system components and complexity as low as possible. The resulting solutions were implemented in system designs of current projects ("AuRora" FFG850459, "HydroMetha" FFG864578), which include linking renewable electricity to gas storages as well as integrating a coupled Co-electrolysis and Methanation plant into industrial processes. These developments lead to highly efficient management of process streams and novel heat exchanger networks.

The resulted system layouts required further development of balance-of-plant (BoP) components, which are substantial to enable those highly efficient systems. Within this work, novel multi-step vaporizer heat exchangers were developed and tested for the first time. They enable internal steam production by utilizing the maximum available heat from several of the systems process streams. Furthermore, a novel and cost-effective hot-gas recirculation solution replaced former used recirculation blowers.

This paper provides the theoretical background, application scenarios, SOE system design variants, BoP development advancements, applied methods as well as results of current projects.

Abstract No 181

## **Stainless steel as OER electrode in alkaline water electrolysis**

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Electrodes made of Ni and Ni alloys are known to be stable and relatively good electrocatalysts for both the oxygen and hydrogen evolution reactions in alkaline media<sup>1, 2</sup>. Even though Ni belongs to the non-noble metals and have beneficial properties in alkaline electrolytes the price and durability of the nickel-based electrodes, which are currently in use by industry, are still needed to be improved. Recent literature claim that proper activated stainless steel can have high catalytic activity and durability compared to other nickel based electrodes and be served as electrode for the oxygen evolution reaction<sup>3, 4</sup>. In the first stage of this research, 316L stainless steel is activated through electro-oxidation at constant potentials in KOH solution in order to achieve an electrode surface with beneficial properties. Durability and activity of the samples are analysed using cyclic voltammetry and potential step measurements at a lab scale in alkaline environment and ambient conditions. The correlation between surface properties and the surface performances are considered with SEM, EDS, XRD and XPS. SEM equipped with EDS is used to evaluate the surface morphology along with elemental distribution on the surface. XRD is used for analysing the surface phases and XPS allows us to identify the oxidation state of the cations close to the surface.

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## Evaluating CRM-free anode and cathode catalysts for a 2 kW electrochemical methanol synthesis demonstrator plant

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As the energy landscape changes due to the introduction of renewable energy, new opportunities arise for power to fuels which enable the carbon cycle loop to be closed. One such process is converting CO<sub>2</sub> to methanol using electrolyser technology. Methanol is advantageous as an energy vector as it can be stored/transported in the liquid form and has a high energy density.

Within the framework of the EU H2020 project LOTER.CO<sub>2</sub>M “CRM-free Low Temperature Electrochemical Reduction of CO<sub>2</sub> to Methanol”, we aim to produce a 2kW demonstrator with ambitious goals of >60% conversion rate of CO<sub>2</sub> and a >90% selectivity towards methanol at the cathode and a critical raw material (CRM) free anode for oxygen evolution capable of 50 A g<sup>-1</sup> while only losing 1% activity over 1000 hours operation.

Electrochemical conversions of CO<sub>2</sub> to methanol and other alcohols have up till now suffered from low selectivity (~40%) or low turnover frequencies ( $r = \sim 10 \mu\text{mol m}^{-2} \text{s}^{-1}$  or  $\sim 1 \text{ mA cm}^{-2}_{\text{Geo}}$ ), Figure 1. Here we introduce the project and present recent results on material development and electrochemical testing procedures for both the CO<sub>2</sub> reduction reaction (CO<sub>2</sub>RR) and oxygen evolution reaction (OER) catalysts to be used in the demonstrator.

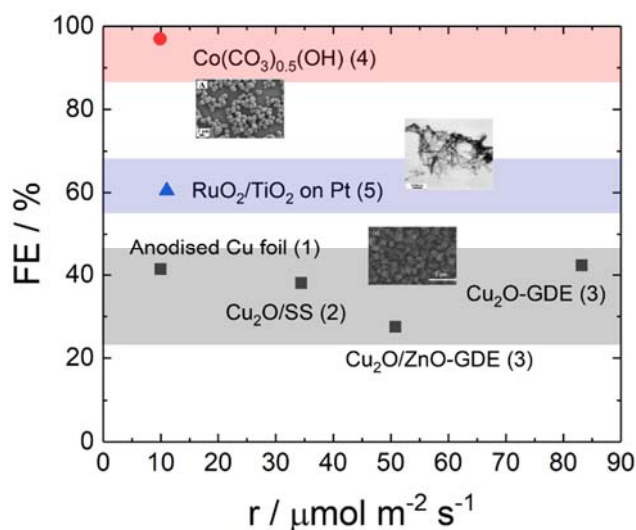


Figure 32. Literature comparison of current catalysts used for CO<sub>2</sub>RR to methanol showing the Faradaic efficiency and the geometric rate. Typical conditions include neutral pH and close to standard pressure and temperature.

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Abstract No 183

## Electrochemical performance of a novel LSFCO perovskite as electro-catalyst for gas diffusion electrodes in alkaline electrolyzers

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Water electrolysis is one of the most promising methods to produce hydrogen, especially when integrated with a renewable source that would allow its production without any emissions of air pollutant or greenhouse gases [1]. To further the development of alkaline electrolyzers it is important to develop novel electro-catalyst to give alternatives to the use of noble metals to promote the kinetics of both the oxygen and hydrogen evolution reactions (OER, and HER respectively). In this work we will present the performance of a novel  $\text{La}_{0.6}\text{Sr}_{0.4}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$  LSFCO-perovskite electro-catalyst that was used in the catalyst layer of our in-house gas diffusion electrodes originally for alkaline iron air batteries, but these type of electrodes could be developed as well for alkaline electrolyzers. In this work we will present the electrochemical characterisation of this novel electro-catalyst and will compare its performance with other reported catalysts.

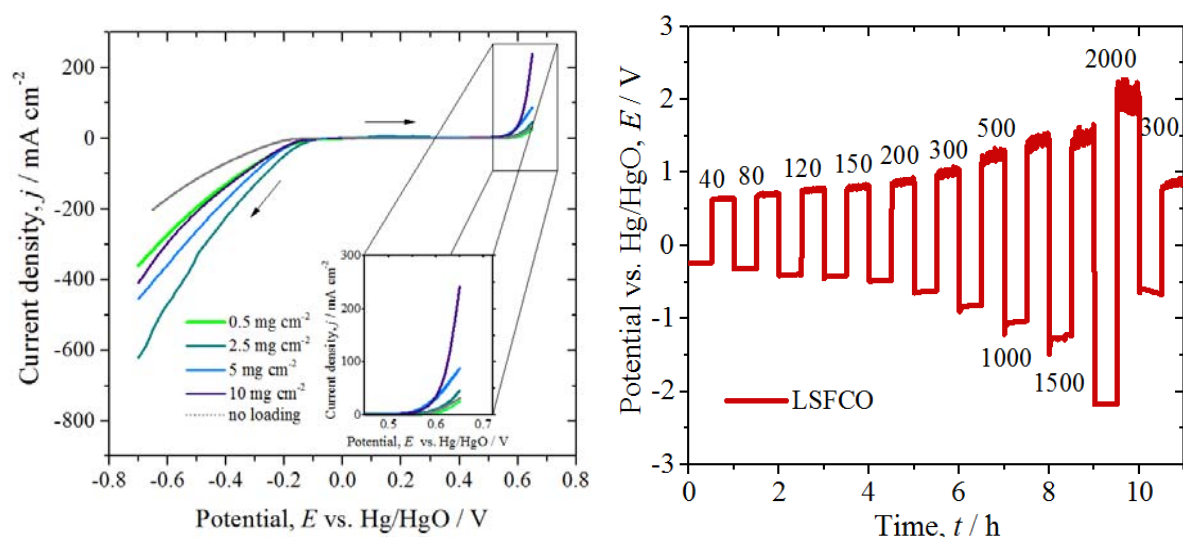


Figure 33. (Left) Cyclic voltammetry for both the ORR and the OER for LSFCO/C catalysed oxygen electrode with different loading of catalyst ranging from 0.5 – 10  $\text{mg cm}^{-2}$ . (Right) Charge-discharge profiles of oxygen electrodes with 5  $\text{mg cm}^{-2}$  LSFCO at various current densities ranging from 40 - 2000  $\text{mA cm}^{-2}$ . Oxygen was supplied at back end of the gas diffusion electrode at a flow rate of 100  $\text{cm}^3 \text{min}^{-1}$ .

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## Neocarbon Food pilot: Microbial protein from renewable electricity and ambient CO<sub>2</sub>

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**Summary.** Electrobioreactors with in situ water electrolysis offer a sustainable way to use renewable electricity to produce microbial protein for food and animal feed. A bench scale Neocarbon Food pilot with 20 L bioreactor volume is implemented. The production rate of microbial biomass is 4 g/h with hydrogen production of 2.7 g/h.

**Abstract.** Gas fermentation of hydrogen oxidizing bacteria is a long known method for single cell protein production. The in situ electrolysis was introduced to produce hydrogen for Knallgas bacteria already in 1965 [1]. In situ electrolysis helps to avoid the energy loss and safety issues related to the low mass transfer rate of gaseous hydrogen to the cultivation medium. The inorganic catalysts have been developed to achieve solar-to-biomass efficiency exceeding that of natural photosynthetic systems [2].

Neocarbon Food pilot setup in Fig. 1 demonstrates the microbial protein production based on renewable electricity and CO<sub>2</sub> captured from the ambient air. The direct air capture unit of CO<sub>2</sub> uses amine-functionalized

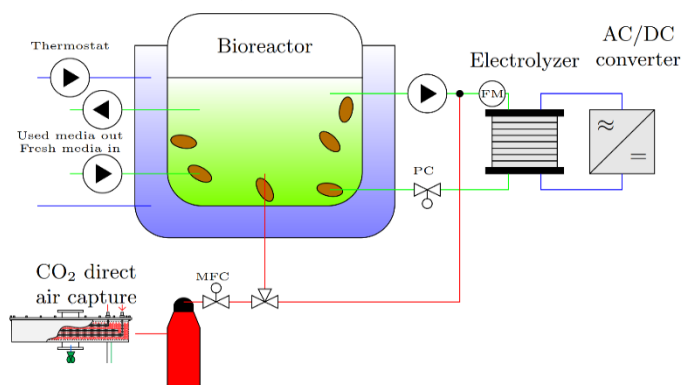


Fig. 34. Principle of the Neocarbon Food pilot setup: Microbial protein production with in situ water electrolysis and direct air capture of CO<sub>2</sub>.  
[www.neocarbonfood.fi](http://www.neocarbonfood.fi)

adsorbent with temperature and vacuum swing adsorption (TVSA) operation cycle [3]. The energy requirement is 10 kWh/kg 76% of which is thermal energy. Water is gathered as a side product.

Hydrogen is produced and mixed with the cultivation medium in an in situ water electrolysis stack. Stainless steel bipolar electrodes can be electrolytically coated with inorganic CoP catalyst. The current density is limited to tens of milliamperes per square centimetre not to harm the microbial growth. Furthermore, the low current density enables hydrogen production energy efficiencies around 50% despite the low conductivity of the cultivation medium. The stack with ten cells

connected in series has electrical supply power around 250 W.

The CO<sub>2</sub> supply and hydrogen production are controlled to maximize the bacterial growth rate of the continuous process. The target is to produce 4 g/h protein rich microbial biomass in the 20 L reactor vessel with hydrogen production of 2.7 g/h. The target overall energy consumption including CO<sub>2</sub> capture and hydrogen production is below 15 kWh per kilogram of biomass.

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Abstract No 185

## High temperature testing facilities at the Norwegian Fuel Cell and Hydrogen Centre

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Research and development within fuel cell and hydrogen technology now move from small scale and short duration tests to larger scale and long-term tests to become more application-oriented. The Norwegian Fuel Cell and Hydrogen Centre (N-FCH) is a new national research infrastructure, which focuses specifically on testing of fuel cells and electrolyzers from single cells to system integration and verification. N-FCH is a joint initiative taken by three major Norwegian R&D stakeholders (SINTEF, Institute of Energy Technology (IFE) and Norwegian University of Science and Technology (NTNU)) engaged in fuel cells and hydrogen technologies and has a clear nation-wide character and impact. The Research Council of Norway has funded N-FCH as a national research infrastructure and the main goal is to establish and operate a centre of advanced laboratories with the required instrumentation and personnel to facilitate high quality research and development of components and testing and validation of systems for fuel cells and electrolyzers.

N-FCH consists of three laboratories:

- Low temperature fuel cells & electrolyzers (SINTEF/NTNU, Trondheim)
- High temperature fuel cells & electrolyzers (SINTEF, Oslo)
- Fuel cell and electrolyser systems (IFE, Kjeller)

The centre also seeks to improve the education of new PhD, MSc and BSc students by ensuring a close interaction and use of the infrastructure, thus increasing the industrial relevance of education and training. The centre's infrastructure is accessible for both Norwegian and international actors within fuel cell and hydrogen technologies.

The High temperature fuel cell & electrolyzers lab (HT-lab) is located at SINTEF in Oslo. The HT-lab has testing facilities for solid oxide fuel cells (SOFC), solid oxide electrolyser cells (SOEC), proton ceramic fuel cells (PCFC) and proton ceramic electrolyser cells (PCEC). Cell sized that can be tested are from button cell size to short-stack 10×10 cm<sup>2</sup> and the geometry can be either planar or tubular. The HT-lab has several single cell ProboStat units for button cells and small tubes. For larger cells, three test stations from Greenlight (2 X40 + 1 X100) have been acquired and installed during the spring of 2019. An overview of the capabilities is shown in Table 1.

*Table 1. Overview of high temperature testing facilities at the HT-lab of N-FCH at SINTEF in Oslo.*

Test station	ProboStat	Greenlight X40	Greenlight X100
Planar/tubular	Planar and tubular	Planar and tubular	Planar
Planar cell size	2 cm diameter	5×5 to 10×10 cm <sup>2</sup>	5×5 to 10×10 cm <sup>2</sup>
Single cell/stack	Single cell	Single cell	Stack (~10 cells)
Pressure	0-20 bar	0-10 bar	0-3 bar
Humidity (max.)	Typically 3%	90 °C dewpoint	90 °C dewpoint + direct injection
Cathode flow (max.)	~0.2 L/min	4 L/min	40 L/min
Anode flow (max.)	~0.1 L/min	2 L/min	20 L/min
Load bank	~40 W	(1300 W)	1400 W
Power supply	~40 W	1320 W	5000 W

## High temperature steam electrolysis at DLR – from stack to system level

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Electrochemical hydrogen production can be crucial for the future energy system due to its many application benefits. Hydrogen is used in the chemical industry, is a promising energy carrier to store electric power from renewables and can play a large role in future mobility and steel industry. Currently, hydrogen is mainly produced by partial oxidation of natural gas. For defossilising the future energy supply new methods and processes for hydrogen production need to step in.

The German Aerospace Center (DLR) investigates environmentally friendly hydrogen production paths which do not use fossils fuels. One of the research topics is the high temperature steam electrolysis using solid oxide electrolysis cells (SOEC). This technology offers a great potential for a highly efficient energy conversion due to the high operating temperature. Additionally, previous studies have shown that the performance of solid oxide cells can be significantly improved by operating at elevated pressure [1]. A further reason for pressurization is the use of pressurized hydrogen in downstream processes for storage or fuel synthesis [2].

One of the test environments at DLR allows to run experiments of solid oxide cell stacks at elevated pressures between 1.4 to 8 bar. Experimental results of two commercially available planar 10-layer stacks with electrolyte supported cells will be presented. Both stacks run in SOEC mode under pressurized conditions. The performance of one stack was evaluated via steady-state and dynamically recorded  $U(i)$ -curves as well as via electrochemical impedance spectroscopy (EIS). It showed an increase of the cell voltages due to the elevated pressure and a slight positive effect on the overall stack performance was observed. Whereas the ohmic resistance is the major part of the entire resistance of an electrolyte supported cell, the activation and diffusion resistance play a subordinate role. The second stack was analysed within a constant-current operation over 1000 hours and at a pressure of 1.4 bar. This stack showed a comparably low degradation rate of 0.56 %/kh [3].

To play a significant role in the future energy system hydrogen production with SOECs from renewable electricity will have to be scaled up into the multi-MW range. To support this transition the DLR built a test environment for large SOC modules of up to 150 kW<sub>el</sub> which can be building blocks for the envisaged systems. The current status of the test rig's operational possibilities and the ongoing investigation of modules from two German SOC system manufacturers will be discussed.

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Abstract No 187

## Optimization and upscaling of porous ceramic separators and full cells for high temperature alkaline electrolysis

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A new generation of alkaline electrolysis cells that can operate at elevated temperature and pressure has recently been demonstrated for the production of pressurized hydrogen at high rate and high electrical efficiency. The concept relies on the development of mesoporous ceramic separators where aqueous KOH is immobilized by capillary forces, in combination with gas diffusion electrodes that overcome mass transport limitations at large production rates.[1, 2] Current densities of up to 3.75 A cm<sup>-2</sup> have been shown at a cell voltage of 1.75 V at 200 °C and 20 bar, with relatively stable performance over 400 h.[3]

Ensuring mechanical integrity of the brittle ceramic separator is required to avoid excessive mixing of the evolved hydrogen and oxygen gases, which could pose a safety threat. In this study the mechanical strength of zirconia based porous ceramic separators, produced by a continuous tape casting process, was measured by the Ball-on-Ring technique, and optimized by controlling their composition. Mesoporous ceramic separators with characteristic strength in excess of 250 MPa were achieved, as shown in Figure 35. Furthermore, the long-term chemical stability and mechanical durability of the separators was explored upon immersion in 8 M KOH solution at 200 °C and 20 bar for a period of 840 h, including 15 thermal and pressure cycles. No sign of degradation could be observed for the strongest separators, based on ZrO<sub>2</sub> doped with 3 mol% Y<sub>2</sub>O<sub>3</sub> (3YSZ). Our findings suggest that the nano-size of the 3YSZ crystallites suppressed the hydrothermally induced tetragonal to monoclinic transformation.[4]

Tape casted and sintered ceramic separators were produced at a size of 5x5 cm<sup>2</sup> and further employed for manufacturing high temperature alkaline electrolysis cells (HT-AEC) by application of gas diffusion electrodes. These cells were tested at elevated temperatures and pressures in a dual atmosphere cell holder with active pressure balancing, validating the HT-AEC concept at realistic technological operating conditions for the first time, since tests were so far carried out in a single atmosphere autoclave with passive pressure balancing [5] and at a cell size of < 1 cm<sup>2</sup>.

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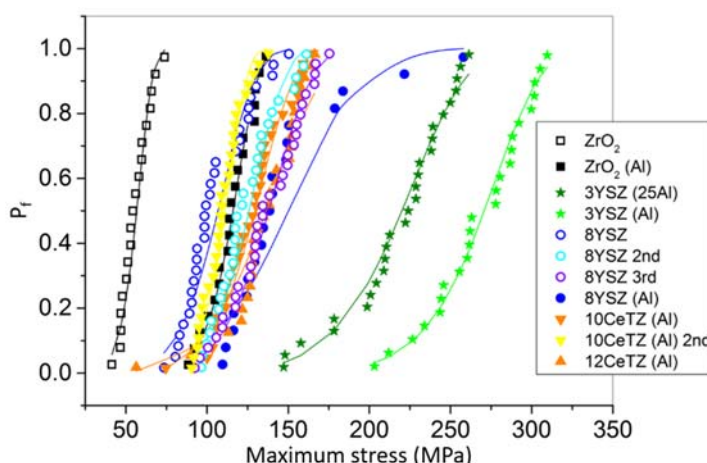


Figure 35 Probability of failure as a function of the applied stress (Weibull distributions) for fresh ceramic membranes with different compositions. (Al) stands for 1 mol% Al<sub>2</sub>O<sub>3</sub>.



## The Demo4Grid project: Demonstration of 4MW Pressurized Alkaline Electrolyser for Grid Balancing Services

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Demo4Grid is an ongoing project started on March 1st 2017, funded under H2020: FCH-02-7-2016 (Grant agreement ID: 736351) [1]. The main aim of this project is the commercial setup and demonstration of a technical solution using the Pressurized Alkaline Electrolyser (PAE) technology for providing grid balancing services under real operational and market conditions and the production of Green Hydrogen for industrial energy services.

In order to validate existing significant differences in local market and grid requirements Demo4Grid has chosen to setup a demonstration site in Austria (see Figure 1) to demonstrate a viable business case for the operation of a large scale electrolyser adapted to specific local conditions that will be found throughout Europe. To achieve that, Demo4Grid will demonstrate at this demo site with particular needs for hydrogen as a means of harvesting RE production:

- a technical solution to meet all core requirements for providing grid balancing services with a large scale PAE (4 MW) in direct cooperation with grid operators,
- a market based solution to provide value added services and revenues for the operation strategy to achieve commercial success providing grid services and those profits obtained also from the hydrogen application.

Aiming at the exploitation of the results after the project ends, Demo4Grid will assess the replicability and viability of various business cases.

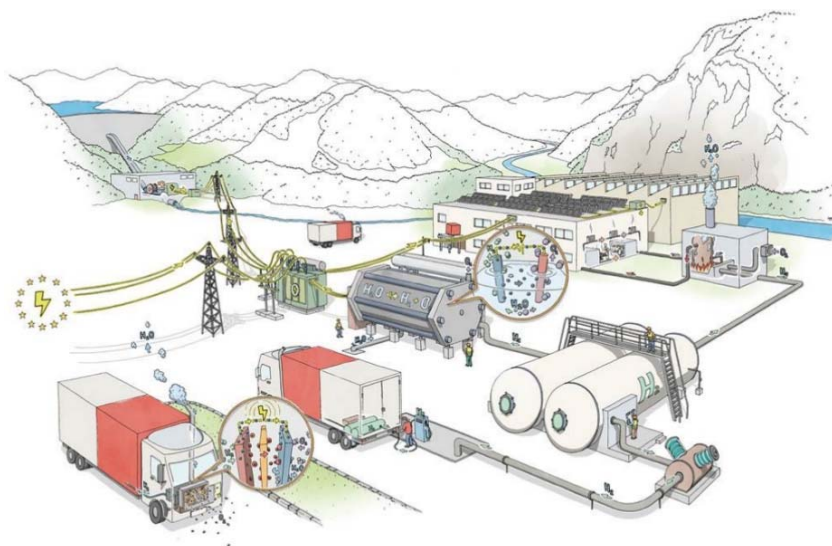


Figure 1. The Demo4Grid overall concept

[1] <https://cordis.europa.eu/project/rcn/207243/factsheet/en>.

Abstract No 189

## Key challenges in fabrication of metal-supported thin film proton conducting electrolyser cells

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Integration of porous stainless steel as metal support (MS) and proton conducting oxide (PCs) as electrolyte in the electrolysis cell structure (MS-PCEC) is a promising and emerging field. On one hand, MS can provide high mechanical integrity and tolerance to very rapid thermal and redox cycling. On the other hand, PC electrolytes such as Y-doped BaZrO<sub>3</sub> (BZY) allow lowering the working temperature compared to oxygen ion conducting electrolytes due to their high ionic conductivity. Moreover, PCEC avoids steam corrosion of the MS since pure dry H<sub>2</sub> is produced at the MS side and thereby reduces the oxidation problems. Since the MS can only withstand temperatures up to ~1300 °C without partly densification and/or melting, traditional powder-based synthesis of high temperature proton conducting ceramics at temperatures ≥ 1500 °C is excluded. In addition, PC electrolytes usually have lower thermal expansion coefficient (TEC) compared to nominal oxygen ion conductors e.g., Y-stabilized ZrO<sub>2</sub> (YSZ). This issue could hinder the application of proton conducting electrolyte in the cell since most other cell components such as porous metal backbone, air and fuel electrodes have higher TEC compared to that of the PC electrolyte, and this mismatch could initiate crack formation or spallation of the electrolyte layer.

In this work, we present manufacturing and testing of single MS-PCEC with a thin electrolyte. Key challenges in the fabrication will be discussed.

Plansee ITM porous metal is used as MS, while spray-coating or tape casting are used to deposit a conducting intermediate layer of La<sub>1-x</sub>Sr<sub>x</sub>Ti<sub>1-y</sub>Ni<sub>y</sub>O<sub>3-δ</sub> (LSTN). The intermediate layer needs to provide high n-type conductivity and for the fabrication be stable in both reducing and oxidizing conditions. It also needs to be smooth enough for deposition of a thin layer of electrolyte. Pulsed laser deposition (PLD) is applied to deposit a gas-tight, thin-film layer (< 2 μm) on top of MS/intermediate layer at low temperature (600-650 °C). In order to have a small TEC mismatch, we develop a new proton-conducting electrolyte by doping Sr and Ce into BZY, yielding a higher TEC (~10 × 10<sup>-6</sup> K<sup>-1</sup>) compared to BZY (~8 × 10<sup>-6</sup> K<sup>-1</sup>). The new electrolyte was deposited on MS/LSTN by PLD. The cell with Pt air electrode shows acceptable open circuit voltage and low overpotentials at 600 °C, demonstrating the opportunities of developing the first working metal-supported proton-conducting electrolysis cells.

Keywords: Barium zirconate, Ceramic processing, Thermal expansion, metal-supported cells

### Acknowledgements

Financial support from the Research Council of Norway (RCN) through the ENERGIX program (AH2A, Project No: 228819/E20 and DAICHI, Project No: 284289) is gratefully acknowledged.

## Titanium Porous Paper as PTL for PEM Water Electrolysis

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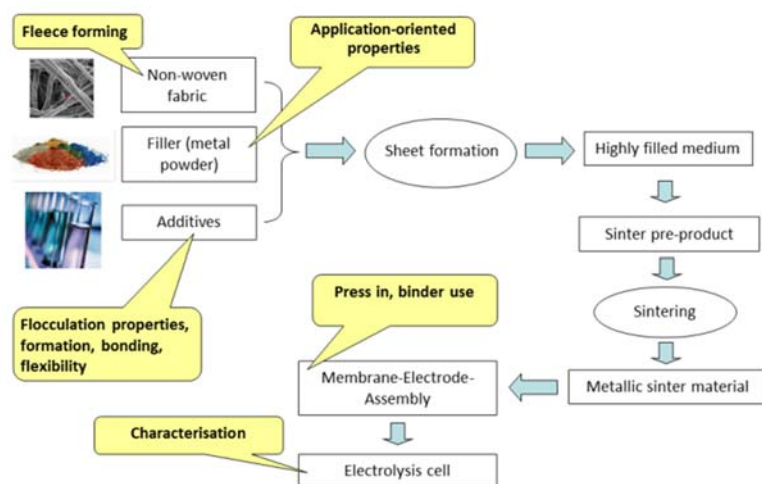
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Porous transport layers (PTLs) are used as power and water/gas distributors in Proton Exchange Membrane water electrolyzers (PEMWE). Nowadays usually thick titanium sintered layers or titanium fabrics are being used. An ongoing public-funded project aims to develop an alternative, cheap manufacturing route for functionally optimized PTLs. Additionally, this new route has the potential to produce thinner and more optimized PTL. In the frame of our project cost-effective paper technology is applied to produce green sheets from cellulose-based fibres and titanium powder. Fig. 5 shows the concept of manufacturing of such sintered material on a paper-structure basis.



In order to better meet the demands of the application, the pore morphology can be tailored via mixing the powder with the fibres and other additives of various shapes. After de-binding and sintering, a purely metallic porous titanium sheet is achieved. Here, the main challenge is the minimization of impurities such as carbon, nitrogen and oxygen. The thermal de-binding process can be greatly enhanced and optimized heat treatment procedures can be developed by means of in-situ infrared spectroscopy and mass spectrometry characterization during the heat treatment.

The obtained PTLs are in situ tested in a specially designed electrolysis single cell with an optically accessible flow field at the anode side and an active geometric area of 25 cm<sup>2</sup>. The design of the optically accessible anode side of the cell enables simultaneous electrochemical tests and characterization of the two phase gas-liquid flow between the electrically conductive lands on the anode plate consisting of produced oxygen and circulating water under industry relevant current densities (up to 2 A/cm<sup>2</sup>). Depending on the flow rate and current density the flow regime forms bubbly flow or slug flow, which is classified by the form of interface between the two phases. Effects of different configurations (channel height and width) and parameters of the titanium porous paper like thickness or pore size distribution have been investigated in detail. Fig. 6 shows a green sheet containing over 80 wt.-% of titanium powder.

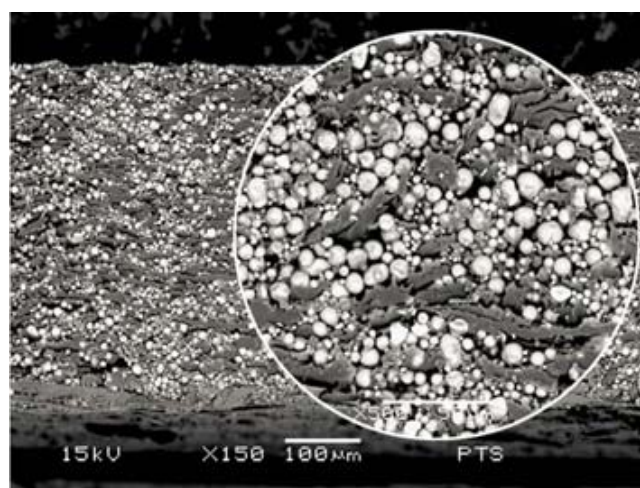


Fig. 6: SEM picture showing the green sheet containing metal powder (> 80 wt.-%)

This work has been funded by the Federal Ministry for Economic Affairs and Energy (IGF 19526 BG).

Abstract No 191

## **Degradation of PEM water electrolysis cells: Looking beyond the cell voltage increase**

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Identified development gaps for proton exchange membrane water electrolysis (PEMWE) include e.g. increasing current density, temperature and (hydrogen) pressure, as well as reducing catalyst loading and membrane resistance [1]. In order to close the aforementioned gaps without compromising the long-term stability and reliability of PEMWE cells/stacks, research activities in the field of degradation need to be intensified [2]. In this context, both the material- and stressor-specific degradation phenomena and mechanisms must be better understood and, on the other hand, specific accelerated stress tests (ASTs) must be developed in order to reduce the measurement time and effort required.

In this study, three points in particular are discussed: i) overpotential-specific degradation rates, ii) time-resolved changes in key parameters and iii) more accurate quantification of degradation effects. Regarding the first point, a simple but powerful high frequency resistance measurement already allows to distinguish between the main overpotentials, i.e. kinetic, ohmic and mass transport overpotentials. With respect to the second point, time-resolved changes in key parameters such as Tafel slope and apparent exchange current density allow a better understanding of stressor-specific degradation phenomena, rather than just having a picture of the begin and end state of health of the cell. Last but not least, the degradation rate unit is usually expressed in  $\mu\text{V/h}$ , which is a poor description as it does not take into account the productivity of the electrolyser, i.e. the amount of hydrogen (and/or oxygen) produced.

With this in mind, stressor-specific degradation experiments were carried out on commercial PEMWE materials. As often observed, a relatively large part of the cell voltage increase is of apparent nature, caused by changes in the oxidation states of the iridium oxide catalyst due to changing electrode potentials. Even if most of the cell voltage increase seems recoverable, catalyst-specific key parameters change anyway almost independent of the operating conditions. On the other hand, true degradation occurs mainly in the ohmic overpotential and depends strongly on the operating conditions. In summary, relatively simple electrochemical characterization methods and analysing tools provide a much more detailed insight into the degradation processes in PEMWE cells.

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## Performance degradation in proton-conducting ceramic fuel cells and electrolyzers

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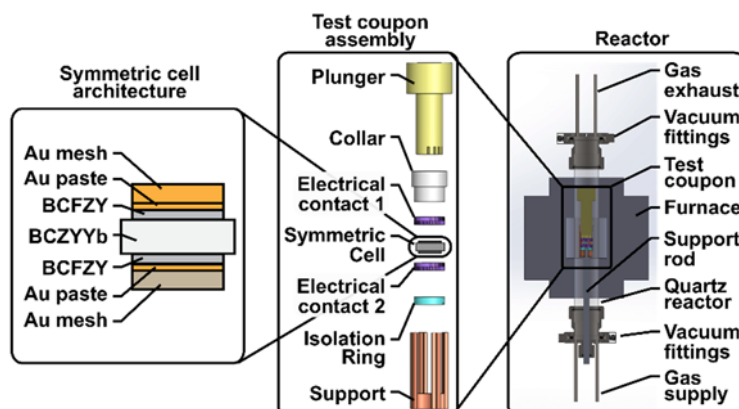
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In this presentation, we show our approach to understanding and mitigating performance degradation in proton-conducting ceramic electrochemical devices. Proton conduction in these ceramic materials is far higher than oxygen-ion conduction in more-conventional solid-oxide electrolysis cell (SOEC) materials. With operating temperatures of 500-600 °C, protonic-ceramic electrolysis cells (PCECs) may provide the benefits of low overpotential and high efficiency of higher-temperature operation without incurring the degradation found in SOECs operating near 800 °C. This can decrease materials requirements within the stack and hot module, and reduce operation and capital-equipment costs. While this presents an encouraging outlook for protonic-ceramic electrochemical cells, technological development of these emerging materials is at an early stage. Reports of long-term PCEC operation are limited, especially in the presence of stack-packaging materials. In this presentation, we will review our studies of performance degradation in proton-conducting ceramics for both fuel-cell and electrolyzer applications.

For commercial viability, developers target aggressive performance-degradation rates near 1% / 1000 hours. In order to meet such targets, the team at the Colorado School of Mines has developed careful experimentation shown in Figure 1 to accurately capture millivolt changes in PCECs over thousands of hours. Our approach is focused on understanding degradation in individual electrodes, and to decouple each electrode from the complete electrochemical cell or stack where competing physical processes can cloud our view of root causes. To facilitate this focus, our Degradation Test Stands utilize simpler “symmetric cells”, where the protonic-ceramic electrolyte material is sandwiched between identical electrodes. The symmetric cell is exposed to a single gas environment, further simplifying the experimentation. A power supply drives the cell, while electrode performance is continuously measured using an electrochemical impedance analyzer.

The symmetric cell packaging shown in Figure 1 is fairly unique and inspired by the research team at Institut de Chimie de la Matière Condensée de Bordeaux (ICMCB-CNRS, Bordeaux, France). All packaging materials are fabricated from high-purity alumina to prevent cell contamination. A heavy plunger is fitted through a collar to provide uniform and continuous compression of electrical contacts to the symmetric cell. The test coupon is supported within a quartz-tube reactor, and placed within a high-temperature furnace. Gases representative of those found under fuel-cell or electrolyzer operation are fed to the reactor, and electrode performance is measured over thousands of hours.

In this presentation, we will review our progress in understanding performance degradation in protonic-ceramic fuel and steam electrodes. Our primary materials set includes a  $\text{BaCe}_{0.7}\text{Zr}_{0.1}\text{Y}_{0.1}\text{Yb}_{0.1}\text{O}_{3-\delta}$  (BCZYYb) electrolyte sandwiched between two  $\text{BaCo}_{0.4}\text{Fe}_{0.4}\text{Zr}_{0.1}\text{Y}_{0.1}\text{O}_{3-\delta}$  (BCFZY) air/steam electrodes. The symmetric cells are exposed to steam-air mixtures at 550°C. In addition to understanding the effects of a changing environment, we are exploring the introduction of commercial ferritic steels. Using these tools, we seek to learn more about degradation in protonic-ceramic electrochemical cells, and develop operational strategies to enable long life.



Abstract No 193

## A 100 W seawater electrolyzer design with an inbuilt in-situ free chlorine and product gas analysis

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Ultra-purified water is indispensable for commercial electrolyzers. However, utilizing seawater for the water electrolysis has a tremendous potential in combination with renewable energy plants. Because of the various challenges seawater electrolysis is facing researches are still on a laboratory scale.

The scaling up of a laboratory experimental setup to a small testing plant on an industrial relevant scale contains additional obstacles. Herein, we want to show a practical device design from an in-house benchtop experimental setup to a modular electrolyzer setup with an in-situ free chlorine and product gas analysis for alkaline seawater electrolysis. The free chlorine concentration is detected by utilizing an amperometric method during operation. The product gas analysis will be conducted with a quadrupole mass spectrometer and specific gas sensors. The testing plant is conceived to measure the product selectivity [1], degradation processes and enables long-term stability testing under constant reaction conditions.

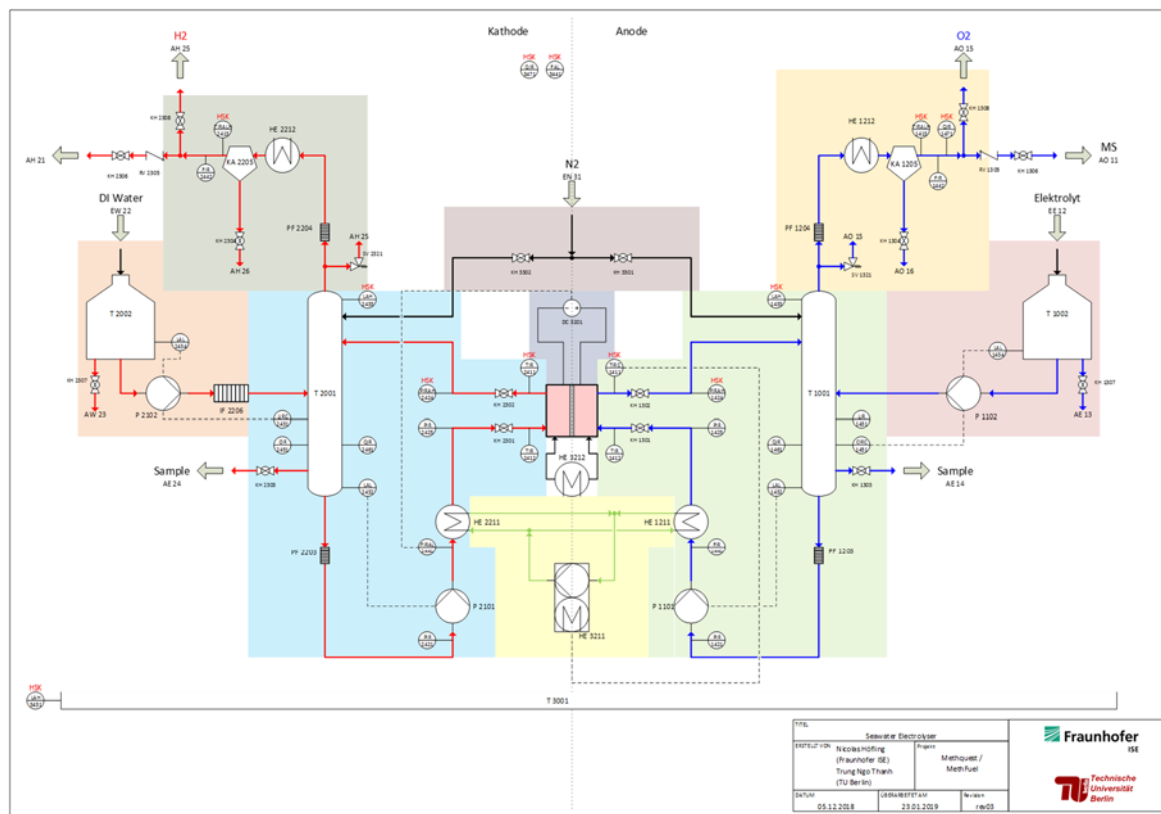


Figure 36 - P&I diagram of the seawater electrolyzer design



## Iridium deposition by galvanic displacement of Cu in a *one-pot* configuration

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In the transition to the hydrogen-based economy, high performance water electrolysis is a necessity. PEM water electrolysis (PEMWE) is particularly suited for incorporation in a power grid based on renewable energy sources[1]. The highly corrosive environment and high potentials on PEMWE anodes excludes the use of most materials, making Ir the only viable electrocatalyst[1]. Due to the scarcity of Ir, reducing the loading is an obvious point of improvement. This can be achieved in a number of ways, including dilution[1] or the usage of core/shell catalysts which can be made with methods such as further treatment of alloy particles[2] or the polyol method[3]. Galvanic displacement appears as a particularly simple method to produce precious metal core/shell structures[4]. Galvanic displacement is the reduction of metal cations coupled with the oxidation of a different, less noble metal. The reaction is driven by the difference in the electrochemical potential of the two metals.

Previous reports on Ir deposition *via* galvanic displacement has involved Ni platelets[5], Ni electrodeposits[6] and underpotential deposited monolayers of Cu on Pd[7]. Ni will oxidize in the low pH required for stable Ir solutions[8]. This suggest a more controllable system when Cu is used for the displacement reaction.

We show that Ir deposition from aqueous solutions of Ir precursors *via* galvanic displacement of Cu monolayers in a *one-pot* configuration is feasible. Figure 1 shows the voltammogram a modified Au electrode in H<sub>2</sub>SO<sub>4</sub>. The voltammogram shows the characteristic shape expected of IrO<sub>x</sub>. From analysis of open circuit potential transients and mass balances, the results show that the galvanic displacement reaction occurs *via* the reduction of Ir(IV) to Ir(III). The flux of Ir(III) species away from the reaction surface results in a very low yield. By successive formation and replacement of Cu monolayers on Au, we have demonstrated that at potentials insufficient for significant direct Ir electrolysis, the galvanic displacement reaction, (1), will proceed. The low yield due to flux of Ir(III) suggests that Ir deposition by galvanic displacement should be performed using Ir(III) precursor solutions.

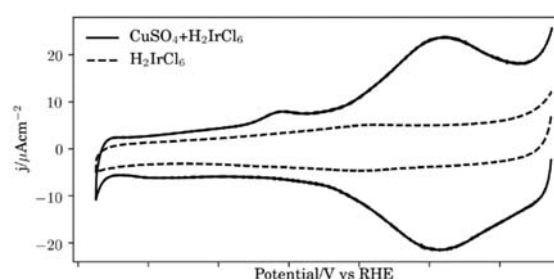
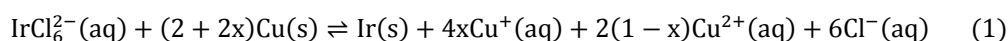


Figure 37: Cyclic voltammogram of a Au electrode after the galvanic displacement procedure in the presence (solid) and absence (dashed) of CuSO<sub>4</sub>.

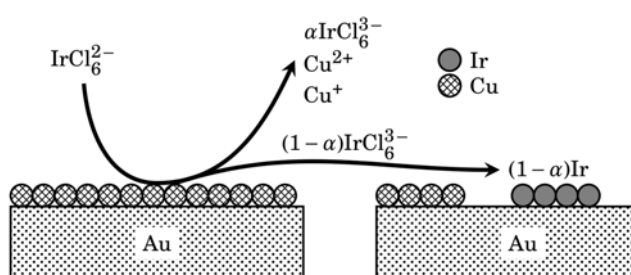


Figure 38: Schematic of the galvanic displacement reaction

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## Crossover in AEL and PEMEL: a direct Comparison of Mechanisms and Mitigation Strategies

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The alkaline water electrolysis (AEL) and the polymer electrolyte membrane water electrolysis (PEMEL) have a common issue – the gas crossover. This contribution performs a direct comparison of the hydrogen crossover for both water electrolysis technologies. For both it is hard to satisfy a H<sub>2</sub> in O<sub>2</sub> safety limit of 2 vol.%, if operating at higher pressures, which is evident from Fig. 1. The H<sub>2</sub> in O<sub>2</sub> content of the PEM water electrolysis is shown in Fig.1a) at different balanced and differential pressure conditions. Results of the alkaline electrolysis are shown in Fig.1b). The AEL was operated with mixed electrolyte cycles to balance the KOH concentration and also with separated electrolyte cycles at balanced pressure conditions.

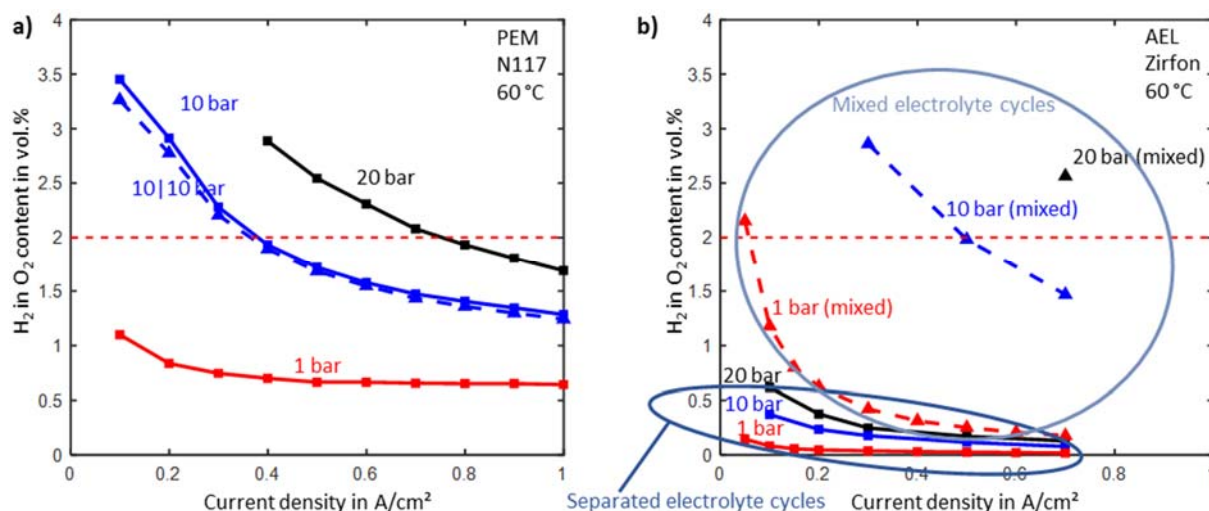


Figure 39. H<sub>2</sub> in O<sub>2</sub> content for PEMEL a) and AEL b). The AEL was operated with mixed and separated electrolyte cycles. Data are taken from [1].

Both technologies have problems to satisfy a safety limit of 2 vol.% H<sub>2</sub> in O<sub>2</sub> at higher pressures and mainly at low till moderate current densities. However, the AEL has this issue only, if the electrolyte cycles are mixed and consequently gas can enter into the wrong cycle. The crossover is much smaller, if separating the electrolyte cycles [2]. At this operation mode, the crossover is only caused by permeation. The permeability is for AEL one order of magnitude smaller than for PEMEC. Mainly, because of the smaller gas solubility in KOH in comparison to pure water [3] and also because of the thicker Zirfon separator of 500 μm compared to the N117 membrane with nearly 200 μm. Further reference measurements were applied to determine a detailed crossover breakdown for both technologies.

This contribution shows a direct comparison of both low temperature electrolysis technologies. Furthermore, different crossover mechanisms are discussed and quantified. Finally, mitigation strategies for both technologies are reviewed and discussed.

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## How Catalyst Layer Design affects Crossover and Cell Performance in PEM Water Electrolysis – by the Example of Ionomer Variation

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An experimental variation of the ionomer content within the cathode catalyst layer showed that both, the crossover and the cell voltage, increase with higher ionomer content. Fig. 1a) shows that the hydrogen crossover flux strongly increases for ionomer contents between 10 and 40 vol. %. From Fig. 1b) it can be seen that the variation of the ionomer content influences the  $iR$ -free voltage.

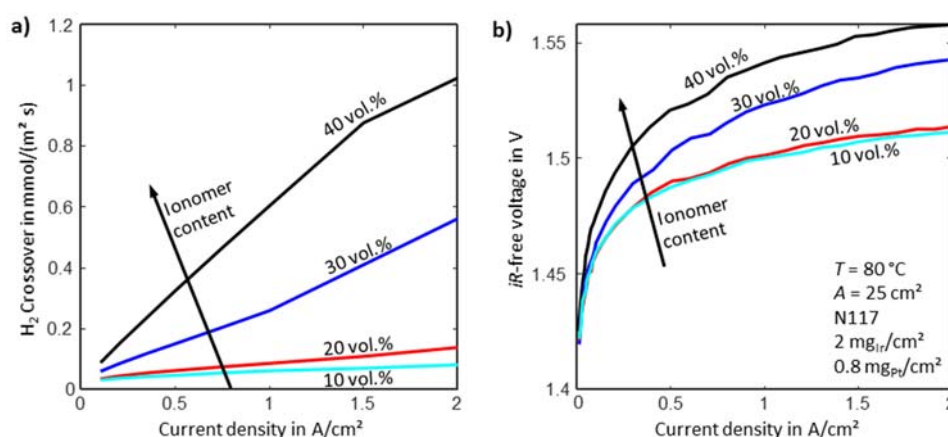


Figure 40. Ionomer variation within the cathode catalyst layer: a) hydrogen crossover flux and b)  $iR$ -free voltage.

The increase in  $iR$ -free voltage with increasing ionomer content was already measured for the anode [2]. It was also already shown that different cell setups cause different increases of crossover with current density, which was attributed to different mass transport resistances [3]. The special feature of the present contribution is that both, the increases in crossover and increases in  $iR$ -free voltage with increasing ionomer content can be explained by an increase in dissolved hydrogen concentration due to higher mass transport resistances. These increases in mass transport resistance can be explained by thicker ionomer films around the catalyst particles and reduced pore space if increasing the ionomer content. They cause higher dissolved hydrogen concentrations that lead to increases in crossover (higher concentration gradients) and increases in  $iR$ -free cell voltage (higher concentration causing mass transport losses). The experimental results are flanked by an advanced one dimensional model of the whole cell, which includes the description of electric/protonic potentials, temperature, gas/liquid pressures and dissolved gas concentrations. The structural parameters of the catalyst layers are adopted from FIB SEM data [4].

The measurement of the hydrogen crossover allows the determination of an integral mass transport coefficient and thus to characterize different structures. In addition, it is possible to determine the mass transport losses. The correlation between crossover and mass transport losses considering the dissolved gas concentration allows a more detailed loss breakdown and a further characterization possibility of different materials and layer structures.

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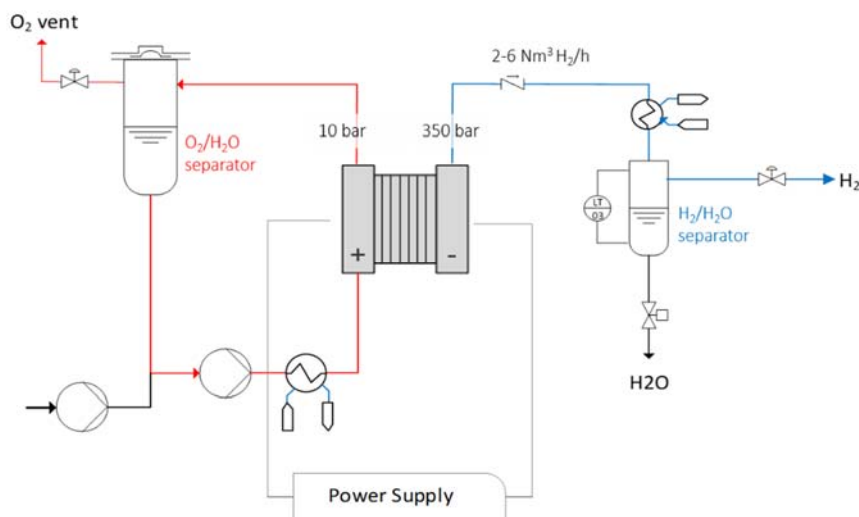
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## High Differential Pressure PEM WE System Laboratory

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Direct electrochemical compression of hydrogen offers several advantages over mechanical compression such as eliminating friction- and motor losses and being vibration free and noiseless. As the capital- and operational costs of hydrogen compressors make up a significant share of the hydrogen costs at dispensers [1], high-pressure electrolyzers delivering hydrogen at 350 bars may become a viable option for a more cost-efficient supply of hydrogen for heavy duty transport. These compact and durable systems are also envisioned suitable as home fuellers for early adopters [2]. Today, the standard delivery pressure of commercial PEM water electrolysis-based hydrogen production systems is 30 bar. This is considered the technoeconomic optimum due to reduced Faradaic efficiency (hydrogen crossover) and the losses associated with reinforced membranes and other necessary safety measures when going to even higher pressures [3]. High-pressure stacks operating at 350 and 700 bars have nonetheless been developed and demonstrated [4] and, to be able to deploy this technology, more research on the operation of high-pressure systems is needed.



**Fig. 1:** Sketch of the PEM WE test rig being built at Institute for Energy Technology (IFE).

As part of a new national research infrastructure in Norway (The Norwegian Fuel Cell and Hydrogen Centre) Institute for Energy Technology is installing a flexible water electrolyzer system test platform for testing of electrolyzers up to 33 kW. A state-of-art prototype high pressure (350 bar) PEM water electrolyzer stack with a production capacity of 2 Nm<sup>3</sup>/h is installed as reference technology. The test rig is constructed for differential pressure operation, where the anodic circulation loop can take up to 10 bar and the cathodic loop up to 350 bar. The electrolyzer and a 20 kW Li-ion battery module is connected to the same DC-bus via dedicated DC/DC converters. The DC-bus is connected to the local grid via an AC/DC converter which can be programmed to emulate different kind of loads and generators. This system laboratory can thus be used to emulate duty cycles (e.g. grid load profiles, solar and/or wind generation), test hybrid system configurations of water electrolyzers and batteries, investigate the performance of differential pressure systems at partial load operation and during startup and shutdown regimes, and research the components of the balance of plant.

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## PEM-based electrolyser developments at ECN part of TNO

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The general aim for the PEM-based electrolyser development within ECN part of TNO is to bring down the cost for electrolysers, which is required for economic viable hydrogen production. The two major cost factors are capital cost for the electrolyser system and energy cost. Especially flexible operation of the electrolyser systems due to the use of renewable energy sources requires a significant lowering of the capital cost of the electrolyser and an improvement of the electrolyser efficiency. In order to achieve the cost targets, cost efficient electrolyser components are required which perform in a robust manner with lifetime exceeding 4-8 years of operation.

The strategy of ECN part of TNO to realise cost reduction for the PEM-based electrolyser consists of the following items:

Development of accelerated stress test protocols: The challenge in the novel component development is to shorten-the-time to market as the demonstration of the feasibility of novel components is very much time-consuming due to the intensive testing required. In order to shorten the time-to-market of cost-efficient components ECN part of TNO has developed an accelerated stress test procedure, which helps to bring down the testing time required for the validation of novel components. The developed accelerated stress test and it's validation will be subject of this paper.

Development of novel cost efficient electrolyser components: ECN part of TNO is cooperating with component manufacturers and academia in the field of electrolyser component development. Involving component suppliers in the electrolyser development process and assisting them in electrolyser knowledge and testing facilities will contribute to bringing down the time-to-market for cost efficient electrolyser. In addition, the most added value in the electrolyser development is present within the supply chain. Currently the main emphasis is on the development of non-fluor-based electrolyte membrane materials in order to replace the relatively expensive Nafion membrane. This work is being performed in close cooperation with the Dutch Polymere Institute DPI and Fuji Film in the Netherlands. Other component development activities are development of blocking layers within the electrolyte membrane in order to prevent gas cross-over and alternative highly active low Ir-content anode electrocatalysts.

PEM-electrolyser stack and system development: A 50 kWe electrolyser system has been installed at ECN part of TNO for testing under renewable energy source conditions and long term testing. The electrolyser system has been developed by Frames and the 50 kW stack by Hydron Energy. Some initial results on the system performance will be shown. Further PEM electrolyzer stack and system scale-up concerns the development of a PEM-electrolyzer 1 MW open innovation platform, which has the possibility to exchange components in order to evaluate the performance of novel components in a MW-system environment. The 1 MW-system will be in operation end of 2020.

## Heat to H<sub>2</sub>

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This work presents a concept for hydrogen production in reverse electrodialysis using waste heat. Reverse electrodialysis converts energy stored in concentration differences between two solutions, using alternating anion and cation exchange membranes (Figure 1). The concentration difference can be restored using heat, where this research presents two separations techniques: The first is by evaporating the water from the concentrated draw solution and add it back to the dilute draw solution. The second is by precipitate salt from the dilute draw solution and add it back to the concentrated draw solution. The latter concept demands a significant change in solubility with temperature to precipitate salt, where KNO<sub>3</sub> fulfils this requirement. As part of a proof of concept, anion and cation exchange membranes from Fumatech were soaked in KNO<sub>3</sub> and conductivity was measured at different temperatures, and compared to the much-used salt in reverse electrodialysis; NaCl. Measurements indicate poorer conductivity for NO<sub>3</sub><sup>-</sup> in FAS-50 to Cl<sup>-</sup>, while the conductivity of K<sup>+</sup> in FKS-50 show similar conductivity as for Na<sup>+</sup>. An increase in temperature from 25 to 40 °C, indicates a significant better conductivity for K<sup>+</sup> in FKS-50, while the conductivity of NO<sub>3</sub><sup>-</sup> in FAS-50 does not change significantly. The conductivity measurements also show an indication of decreasing conductivity with increasing concentration.

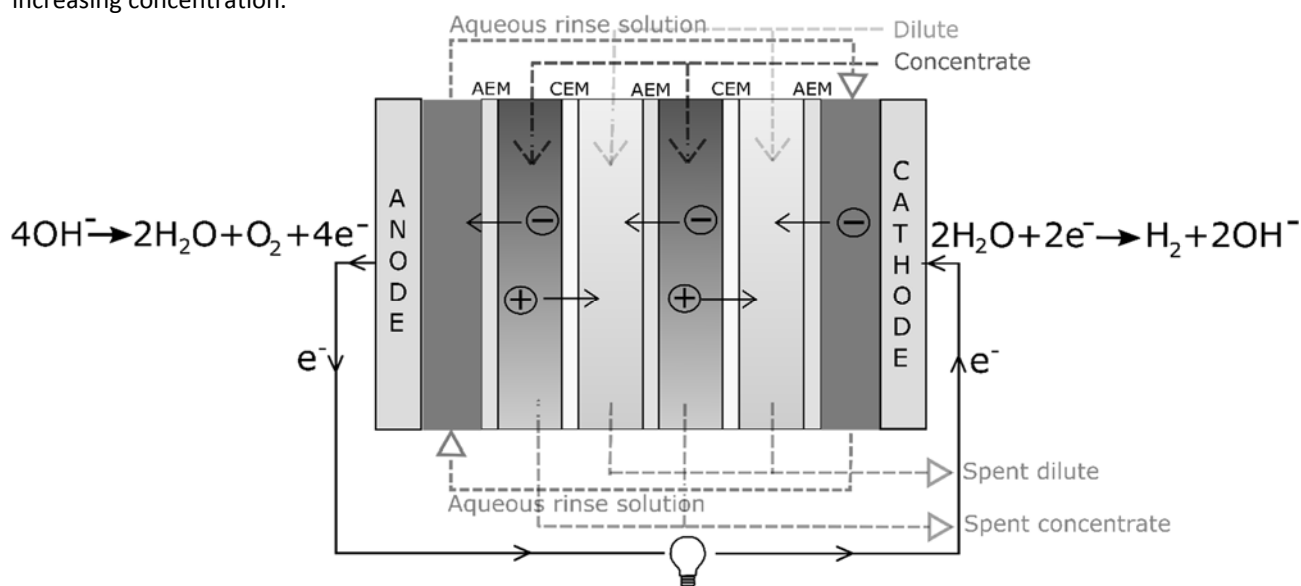


Figure 1 Illustration of a reverse electrodialysis stack

## Improved Electrocatalytic Water Splitting Reaction on CeO<sub>2</sub>(111) by Strain Engineering: A DFT+U Study

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Ceria is a promising cathode material in Solid Oxide Electrolysis Cells (SOEC) because ceria can become a mixed electronic and ionic conductor through doping, which enables a high surface area for electrocatalysis<sup>1,2</sup>. Here, we systemically investigate the effect of strain on the electrocatalytic water splitting reaction (WSR) for renewable hydrogen production<sup>3,4</sup> on CeO<sub>2</sub>(111) by using density functional theory corrected for on-site Coulomb interactions (DFT+U) with  $U = 4.5$  eV<sup>5,6</sup>. We find tensile strain stabilizes the reduced states of ceria such as oxygen vacancies and surface hydroxyls, while compressive strain destabilizes the reduced states. These trends are explained by a downshift of the Ce 4f orbital energy under tensile strain and agree with the larger size of the Ce<sup>3+</sup> ion than the Ce<sup>4+</sup> ion. Our results show that hydroxyl decomposition into H<sub>2</sub> has the highest activation energy along the WSR pathway ( $E_a$ ) and that the free energy of hydroxyl formation ( $\Delta G_H$ ) prior to hydroxyl decomposition can act as a thermodynamic barrier to the WSR. Compressive strain ( $< -3.0\%$ ) correlates strongly with increased WSR activity on CeO<sub>2</sub>(111) because it reduces the total barrier ( $\Delta G_H + E_a$ ). Strain also effectively engineers the reaction pathway of the WSR at  $T > 1000$  K. By comparing the total reaction barrier on different hydroxylated surfaces, the WSR is found to proceed readily via a Ce-H intermediate on excessively hydroxylated CeO<sub>2</sub>(111) under tensile strain because of the lower barrier, while the WSR proceeds preferentially and readily on the partially or fully hydroxylated CeO<sub>2</sub>(111) under compressive strain. Our investigations provide a better understanding on the efficient WSR on the CeO<sub>2</sub>(111) by strain engineering, which sheds new light on electrocatalysis on oxide catalysts.

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Abstract No 201

## Research on degradation mechanism of solid polymer electrolyte water electrolysis

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**Abstract:** An orthogonal test scheme L9 (3<sup>4</sup>) was designed considering four main factors, anode catalyst (IrO<sub>2</sub>) loading, cathode catalyst (Pt/C) loading, anode nafion proportion and cathode nafion proportion with three levels. The water electrolysis properties of different MEAs were characterized using polarization curve under 60 °C. The current density at 2V was used to assess the performance. The MEA had the best performance when the anode catalyst loading was 2.0mg/cm<sup>2</sup>, cathode catalyst loading was 1.0mg/cm<sup>2</sup>, anode nafion proportion was 20% and cathode nafion proportion was 25%. Polarization curve measurement, hydrogen yield calculation and stability test was conducted on the MEA with optimal condition. Under a constant current mode, the electrolytic voltage increased from 1.78V to 2.06V during the stability test. The water in tank was analyzed by inductively coupled plasma mass spectrometry (ICP-MS) every ten hours, the result showed that there was an obvious accumulation of the Ir and Pt elements in the feed water with the process of water electrolysis. The electrochemical impedance spectroscopy (EIS) indicated that the total resistance of electrolyser and charge transfer resistance both increased.

**Keywords:** SPE; Orthogonal test; MEA; Hydrogen yield; ICP-MS

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## Study on Ti based alloy coating corrosion mechanism by Cl and N Non-metallic Elements existence

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TiCr, TiNb and TiNbN coated 316L stainless steel metallic bipolar plates were prepared by magnetron sputtering method. The corrosion resistance performance of TiCr and TiNb under Chloride electrolyte solution and nitrogen atom doping were investigated respectively. The potentiodynamic polarization curves and electrochemical impedance spectroscopy study indicated that the presence of chloride ion and nitrogen atom decreased the Ti alloy corrosion resistance performance significantly. The corrosion mechanism were studied by Molecular dynamic simulation, XPS and SEM. Molecular dynamic simulation results indicated that Cl ion accelerated TiCr corrosion through adsorbing and dissolving surface metal atoms on passivation films. The XPS and SEM analysis showed that the doping of nitrogen atoms formed several nitrogen compounds and micro-concave defects, increased the H ion concentration in the defects, thus the Ti alloy corrosion rate was accelerated.

**Keywords:** Metallic bipolar plate, SPE water electrolysis cell, Ti alloy coating, Corrosion mechanism

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## Energy storage, ammonia synthesis and electric-power generation with reversible proton-conducting ceramic cells

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Ammonia synthesis and electric-power generation via high-performance proton-conducting ceramic cells is presented. Ammonia synthesis is enabled through use of a novel barium-calcium-aluminum oxide catalyst support with a low-loading ruthenium catalyst (Ru-B2CA) [1]. This catalyst and support has been patented by Starfire Energy, Denver, Colorado, USA, for both ammonia synthesis and ammonia cracking. The protonic-ceramic electrochemical cells feature a BaCe<sub>0.7</sub>Zr<sub>0.1</sub>Y<sub>0.1</sub>Yb<sub>0.1</sub>O<sub>3-δ</sub> (BCZYYb) electrolyte on a Ni-BCZYYb fuel-electrode support, and BaCo<sub>0.4</sub>Fe<sub>0.4</sub>Zr<sub>0.1</sub>Y<sub>0.1</sub>O<sub>3-δ</sub> (BCFZY) air-steam electrode. We utilize these cells for solid-state electrochemical-synthesis of ammonia. To date, we have achieved an ammonia-synthesis rate of  $1.3 \times 10^{-8} \text{ mol.s}^{-1}.\text{cm}^{-2}$  at ambient pressure from steam (carried by humidified air) and nitrogen feed streams. This rate is among the highest electrochemical ammonia-production rates ever reported, regardless of electrolyte type [2-5]. Other reported rates reach  $1.1 \times 10^{-8} \text{ mol.s}^{-1}.\text{cm}^{-2}$  using Nafion electrolyte and SFCN as ammonia formation catalyst [2], and  $1.0 \times 10^{-8} \text{ mol.s}^{-1}.\text{cm}^{-2}$  using molten NaOH-KOH as electrolyte and nano-Fe<sub>2</sub>O<sub>3</sub> as catalyst[3]. For higher-temperature electrochemical cells, an ammonia-production rate of  $8.2 \times 10^{-9} \text{ mol.s}^{-1}.\text{cm}^{-2}$  has been reported using GDC solid electrolyte and Ag-Pd catalyst [4]. When operated in fuel-cell mode, the same Ru-B2CA catalyst serves to crack the ammonia feed upstream of the protonic-ceramic fuel cell. We achieved a peak power density of 620 mW.cm<sup>-2</sup> at 600 °C using direct ammonia fuel with air oxidizer. Button cells and unit-cell stacks have been tested under direct ammonia fuel for up to about 800 h. This reversible protonic-ceramic approach presents a compelling technology for storage of intermittent renewable energy sources.

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