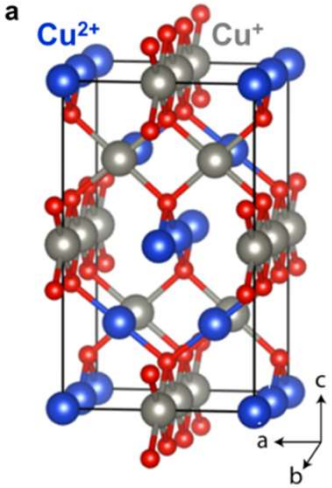
PhasKat

Phase-pure electrocatalysts and adjustment of conditions regarding the reduction of CO_2



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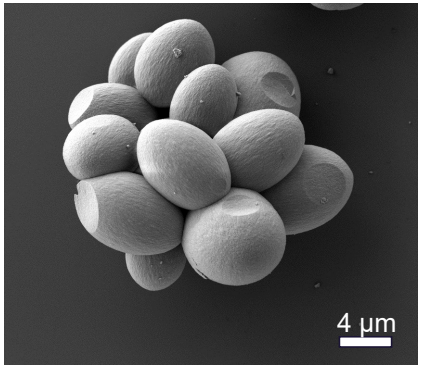
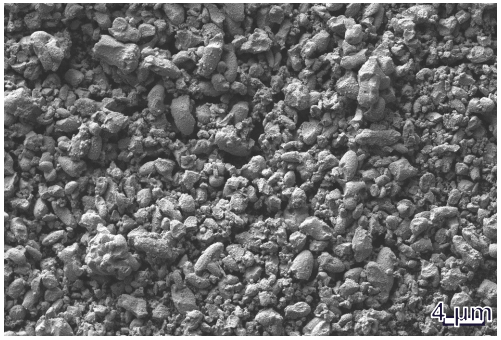


CO2- WIN: 1. Status Conference

June 8th, 2021

Dr. Wiebke Sarfert-Gast, Siemens Energy Global GmbH & Co. KG



Outline

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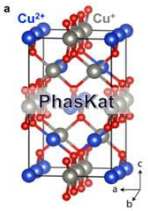

- Motivation
- Objectives
- Consortium & project setup
- Current status
  - WP1: Catalysts
  - WP2: Anion Exchange Ionomers
  - WP3:
    - Ink preparation & Electrode processing
    - Electrochemical characterization
  - WP4: Characterization by impedance spectroscopy
- Summary & Outlook

Motivation

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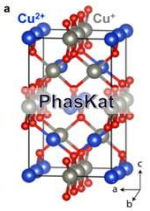


Resources | Direct Electrolysis | Target Products | Use

Solar Power

Wind Power

Renewable energy

H_2O

CO_2

Chemical resources

Electrolysis

Hydrocarbons (C_xH_y)/ Alcohols

TRL 2-3

Ethylene C_2H_4 [2]

Production:	140 Mio. t / year
Estimated CO_2 saving:	658 Mio. t / year
Price:	850 – 1200 € / t

Ethanol C_2H_5OH [3,4]

Production:	92,4 t / year
Estimated CO_2 saving:	191 Mio. t / year
Price:	360 € / t

Chemical Industry

Feedstock

Mobility (synthetic fuel)

Aviation

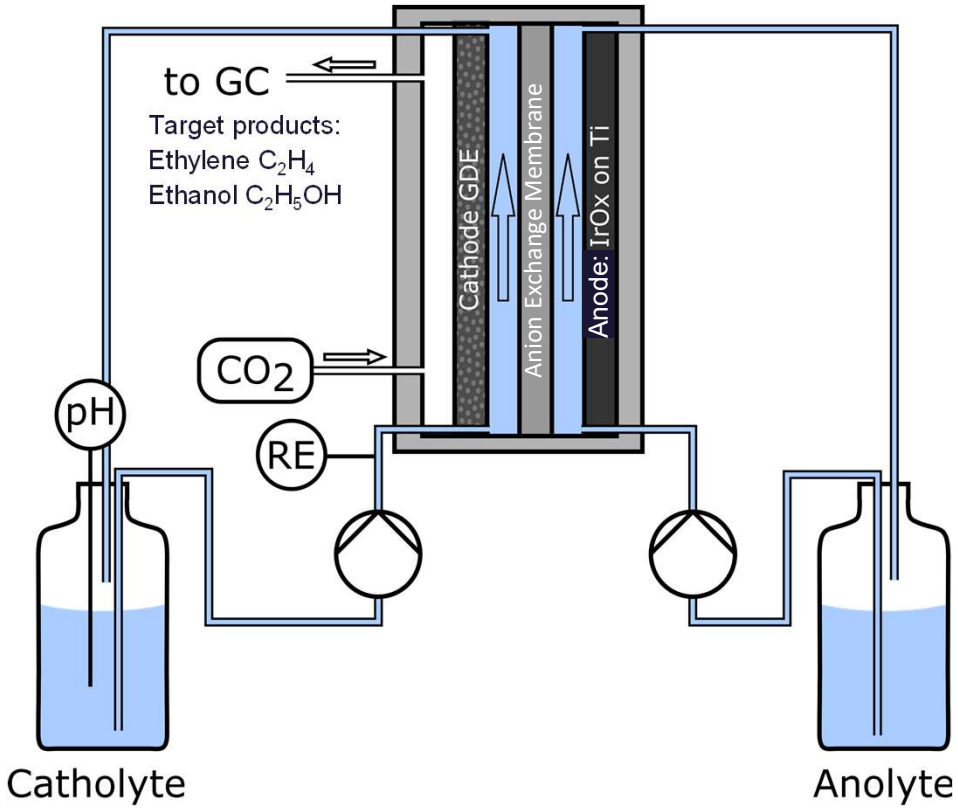
Marine

Heavy Road transportation

1) http://www.ceresana.com/de/marktstudien/chemikalien/ethylen/
2) https://www.statista.com/statistics/274142/global-ethanol-production-since-2000/
3) https://tradingeconomics.com/commodity/ethanol

Objective: CO_2-Electrolysis

Gas Diffusion Electrode (GDE) =
Gas diffusion layer coated with catalyst
and Anion Exchange Ionomers (AEI)



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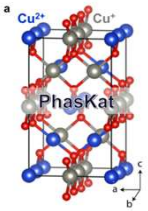

The performance of the GDE depends on:

- Properties of catalyst material
- Properties of Anion Exchange Ionomers
- Layer composition
- Morphology due to processing conditions
- Operation environment
- Operation mode

→ The aim of PhasKat is to develop a better understanding about the materials and the way of processing in order to be able to optimize the GDE performance at industrial relevant current densities (> 200 mA/ cm²).

Consortium

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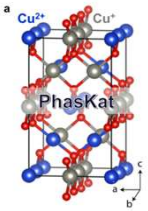
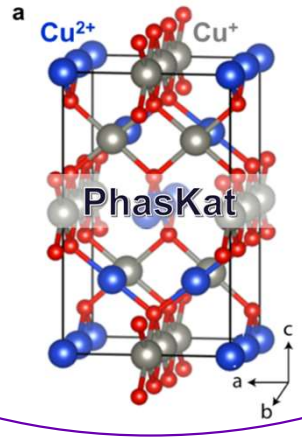


SIEMENS energy

EVONIK
Leading Beyond Chemistry
CREAVIS & Smart Materials



HI ERN
Helmholtz Institute Erlangen-Nürnberg
part of Forschungszentrum Jülich

JÜLICH
Forschungszentrum

Project Setup

Duration: 01.02.2020 – 31.01.2023
Funding code: 033RC028

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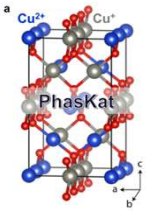
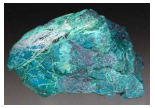


WP1 Catalysts (Evonik SM, SE)

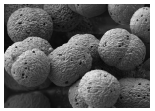
Reference Materials (known to SE)

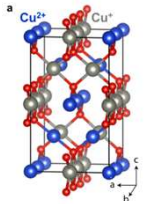
- Cu_4O_3 (Paramelaconite)
- $Ag_2Cu_2O_3$



New Target Materials

- $BaCuO_2$
- $CuFe_2O_4$
- Ag_3CuS_2





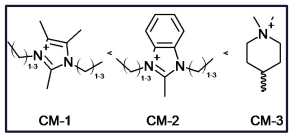
WP2 Anion Exchange Ionomers (AEI) (Evonik CREAVIS, SE, HIERN)

New AEIs under development:

- Evolon

Benchmark AEIs:

- Aemion (Ionomr)
- Sustainion (Dioxide Materials)
- ….

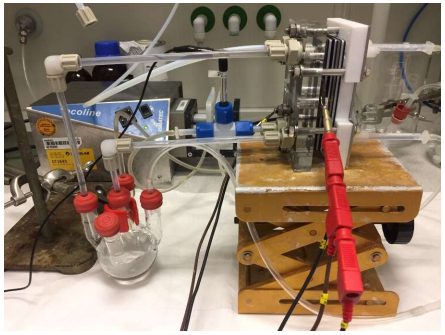


catalyst →
← property

AEI →
← property

WP3 Electrode Processing & Electrochemical Characterization (HI ERN, SE)

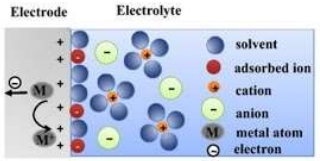
- Evaluation of various fabrication methods for GDE preparation.
- Evaluation of the catalysts and AEIs.
- Optimization of GDE (10 cm²) in terms of performance and stability to meet the final target: FE_{C2} > 80% @ 200 mA/cm², > 100 h.

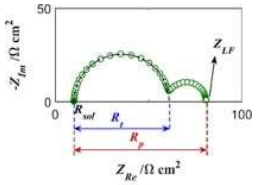


Basic understanding ↔

WP4 GDE Characterization by impedance spectroscopy (FZJ, IEK-9)

- Electrical fundamentals of GDE operation
- Local impedance of single components
- Impedance of electrodes and its interfaces





GDE preparation →

WP5 System Analysis & Demonstration (Evonik CREAVIS, SE)

- Up-scaling towards 300 cm² cell area.
- Demonstration of scalability.

WP1: Catalysts

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EVONIK Leading Beyond Chemistry

SIEMENS energy

Objective: Catalyst development, optimization and scale-up

Lab scale preparation

Transfer and implementation of SIEMENS protocol for:

- ✓ Cu_4O_3
- ✓ $Ag_2Cu_2O_3$

Novel Materials:

- ✓ $BaCuO_{2/2.5}$
- ▪ Ag_3CuS_2
- ▪ $CuFe_2O_4$

Project status: ongoing

Analytics

Transfer of SIEMENS standard analytical protocol for target materials

- ▪ Derive analytical tools for novel materials

Project status: implemented

Optimization and scale-up

Constant assessment of recipe and handling optimization as well as scale-up possibilities

Involved partners:
Dr. A. Reinsdorf, Evonik SM
S. Brinkmann, Evonik SM
N. Martic, Siemens Energy
Dr. G. Schmid, Siemens Energy

WP1: Catalysts

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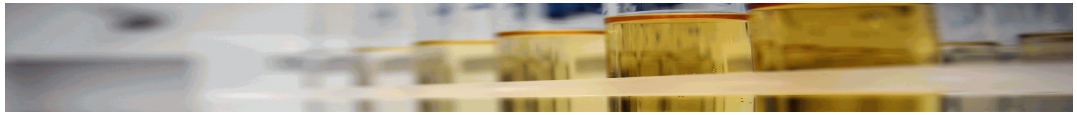
EVONIK Leading Beyond Chemistry

SIEMENS energy

Catalyst development, optimization and scale-up – Current status

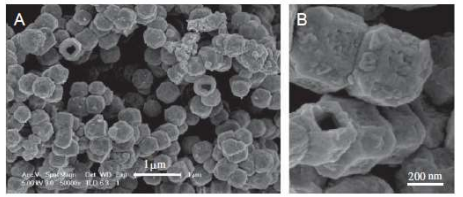
- Intensive literature search was done in close cooperation with SIEMENS
- Different recipes for silver and barium modified copper catalysts were analyzed and are currently in assessment at Evonik Catalysts
- Lab trials started with the preparation of Ag_3CuS_2 in February 2021
- Lab trials with $BaCuO_{2/2.5}$ started in March 2021, two samples are available



Synthesis and ammonia sensing property of Ag_3CuS_2 nanocages obtained from Cu_7S_4 18-facet hollow nanopolyhedra

Zhangxian Chen, Weixin Zhang *, Zeheng Yang

School of Chemical Engineering, Anhui Key Laboratory of Controllable Chemical Reaction and Material



A convenient hydrothermal route to mineral Ag_3CuS_2 nanorods

J.Q. Hu*, B. Deng, W.X. Zhang, K.B. Tang, Y.T. Qian

Structure Research Laboratory and Department of Chemistry, University of Science and Technology of China, Hefei 230026, PR China

Formation and Decomposition of $BaCuO_{2.5}$ Prepared from a Mixture of Nitrates

MASATO MACHIDA, KIYOSHI YASUOKA, KOICHI EGUCHI, AND HIROMICHI ARAI

Department of Materials Science and Technology, Graduate School of Engineering Sciences, Kyushu University 39, 6-1 Kasugakoen, Kasuga, Fukuoka 816, Japan

Precursor
- Choice
- Ratio
- Modification

Precipitation
- Temp.
- Time
- Energy dissipation

Drying
- Temp.
- Time
- Atmosphere[1]

Therm. Treatment
- Temp.
- Time
- Atmosphere[1]

| [1] Choice of Gases as well as possible convection

Involved partners:
Dr. A. Reinsdorf, Evonik SM
S. Brinkmann, Evonik SM
N. Martic, Siemens Energy
Dr. G. Schmid, Siemens Energy

WP2: Anion Exchange Ionomer (AEI)

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EVONIK Leading Beyond Chemistry

Objective

- Synthesis of anion exchange ionomers (AEI)

Approach

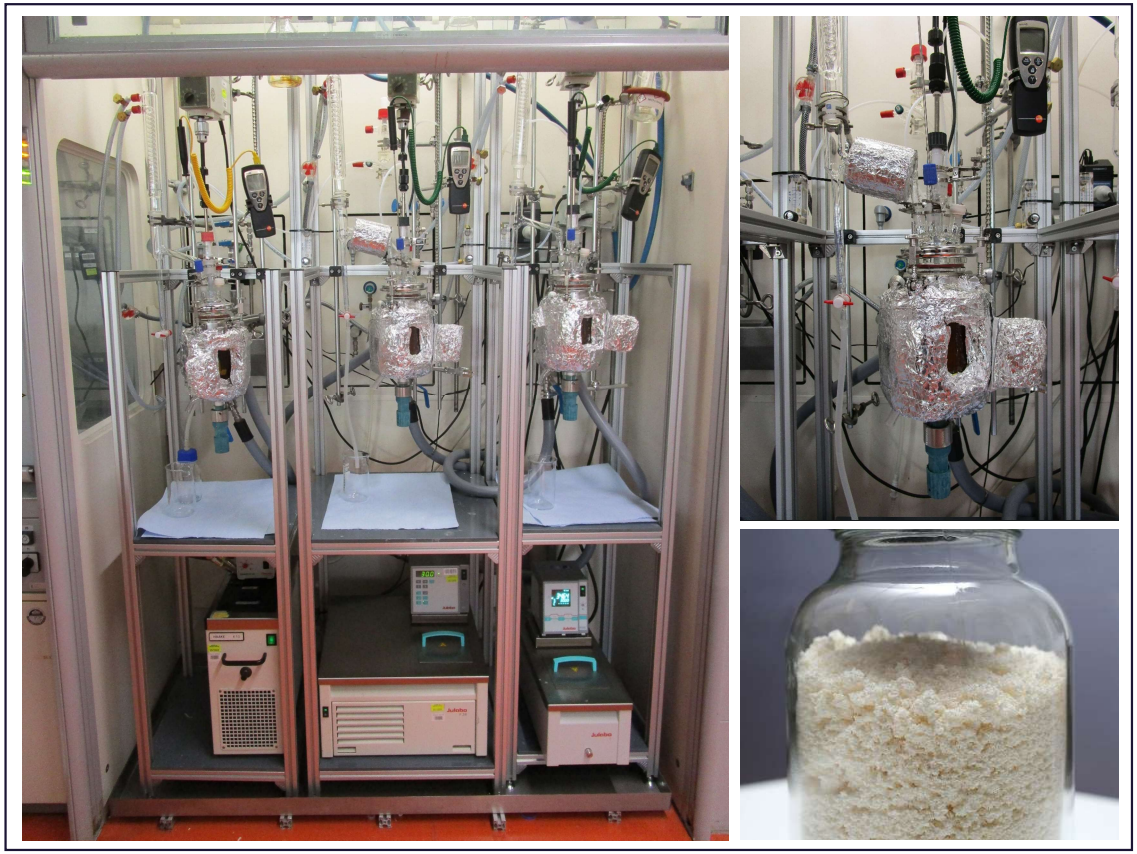
- Synthesis of AEIs using experience of Evonik in development of high-performance polymers

Result

- Evonik provided Siemens samples of EvoIon Type I AEI (5g batch)
- Synthesis scaled up to 10g batch

Outlook

- Synthesis of Type I AEI with different IECs & polymerization degree is in progress



Involved partners: Dr. A. Maljusch, Evonik CREAVIS

WP3: Catalyst ink composition optimization

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HI ERN Helmholtz Institute Erlangen-Nürnberg part of Forschungszentrum Jülich

Objective

- Optimizing ink composition to achieve more porous catalyst layer and better performance.

Approach

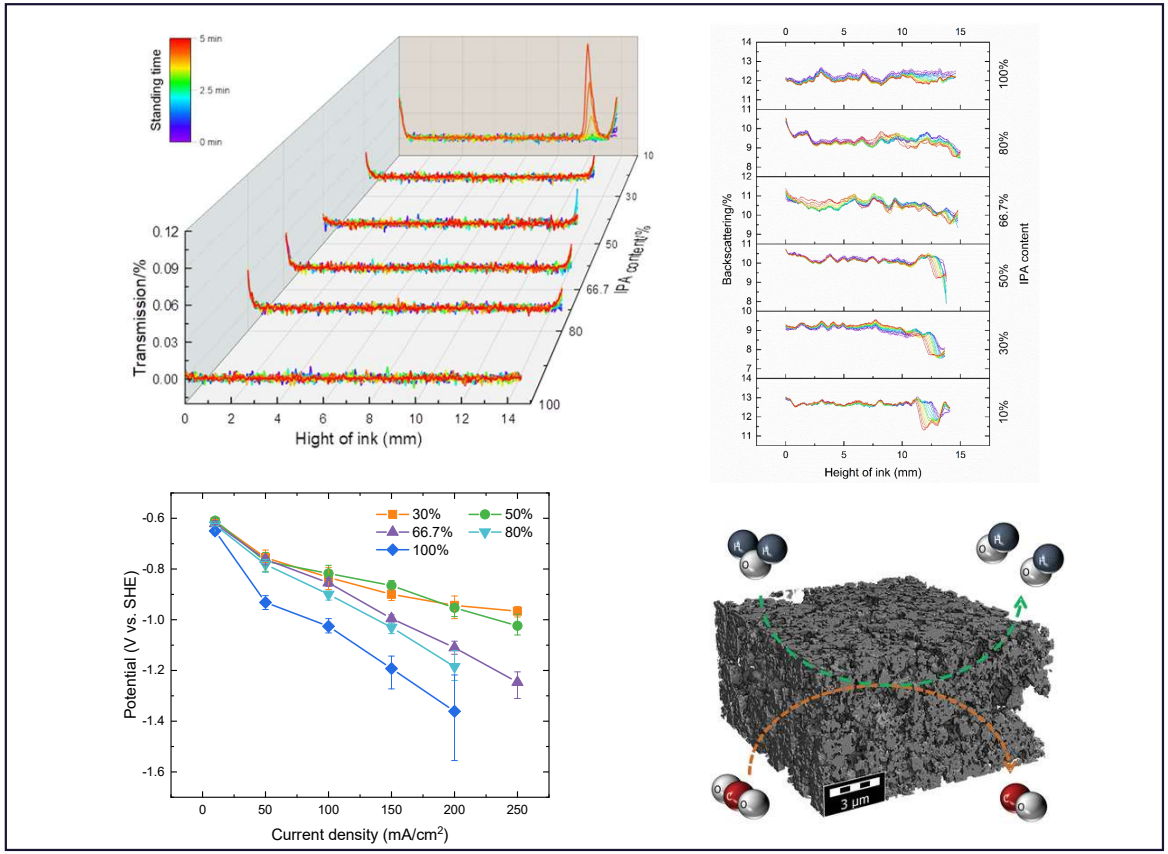
- Adopting an optical method for ink stability analysis, doctor blading for GDE manufacturing and 3D tomographic reconstruction for micro-structure characterization.

Result

- Higher IPA content contributes to stabilize the catalyst ink.
- Lower IPA content results more porous structure and better performance.

Outlook

- Correlating performance with ink composition and catalyst layer structure.



Involved partners: Prof. S. Thiele, L. Guangxin, D. McLaughlin

WP3: Evaluation of Catalyst Materials

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EVONIK Leading Beyond Chemistry

SIEMENS energy

Objective

- Paramelaconite (Cu_4O_3) based catalyst powders provided by Evonik have been evaluated as catalyst materials for electroreduction of CO_2 and compared to reference samples synthesized at SE.

Approach

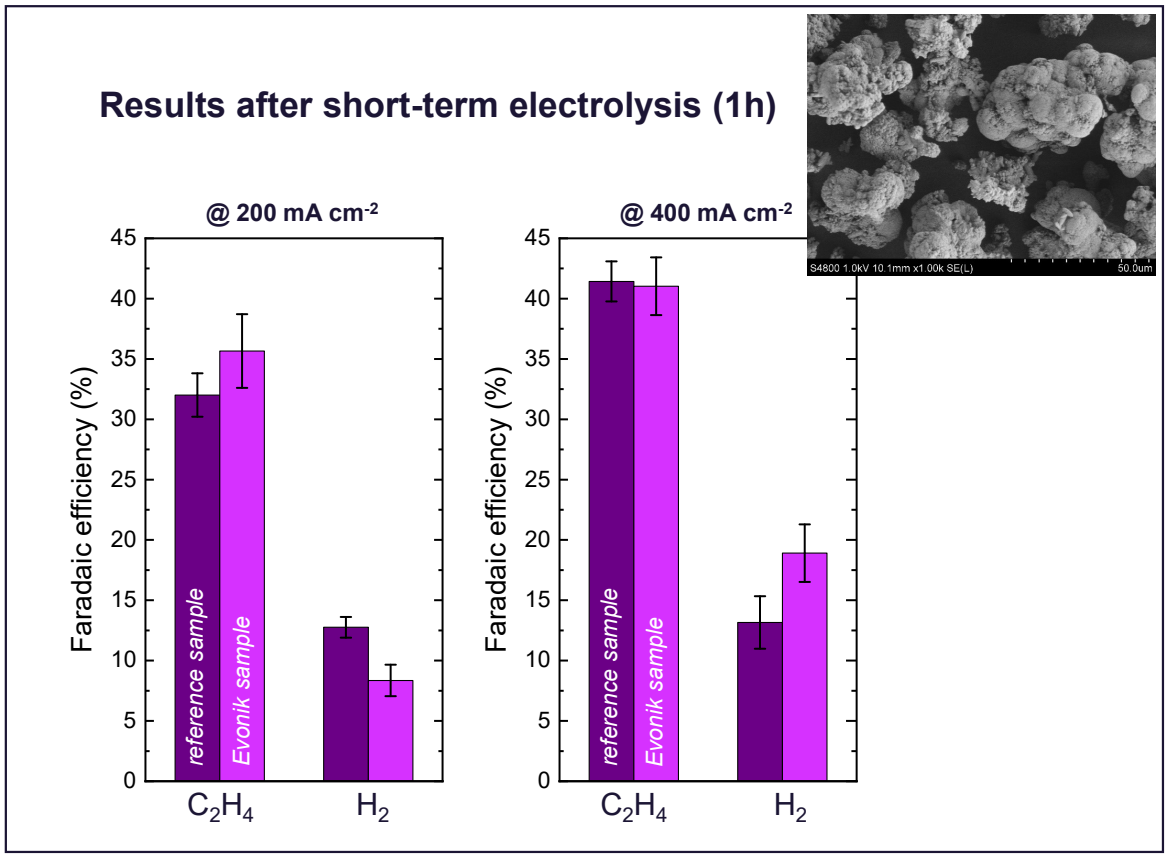
- Investigation via SEM.
- Processing into a GDE using spray-coating.
- Investigation of CO_2 electroreduction properties in a flow cell at industrial relevant current densities (> 200 mA/ cm²).

Result

- The catalytic performance of Cu_4O_3 powders provided by Evonik are comparable to the reference sample.

Outlook

- Evaluation of catalysts as prepared by Evonik.
- Selection of best catalyst for performance optimization and up-scaling.



Involved partners: Dr. A. Reinsdorf (Evonik); N. Martic, S. Szyszkowski, A. Maltenberger (SE)

WP3: Evaluation of Anion Exchange Ionomers

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EVONIK Leading Beyond Chemistry

SIEMENS energy

Objective

- Evaluation and benchmarking of different anion exchange ionomers (AEI).

Approach

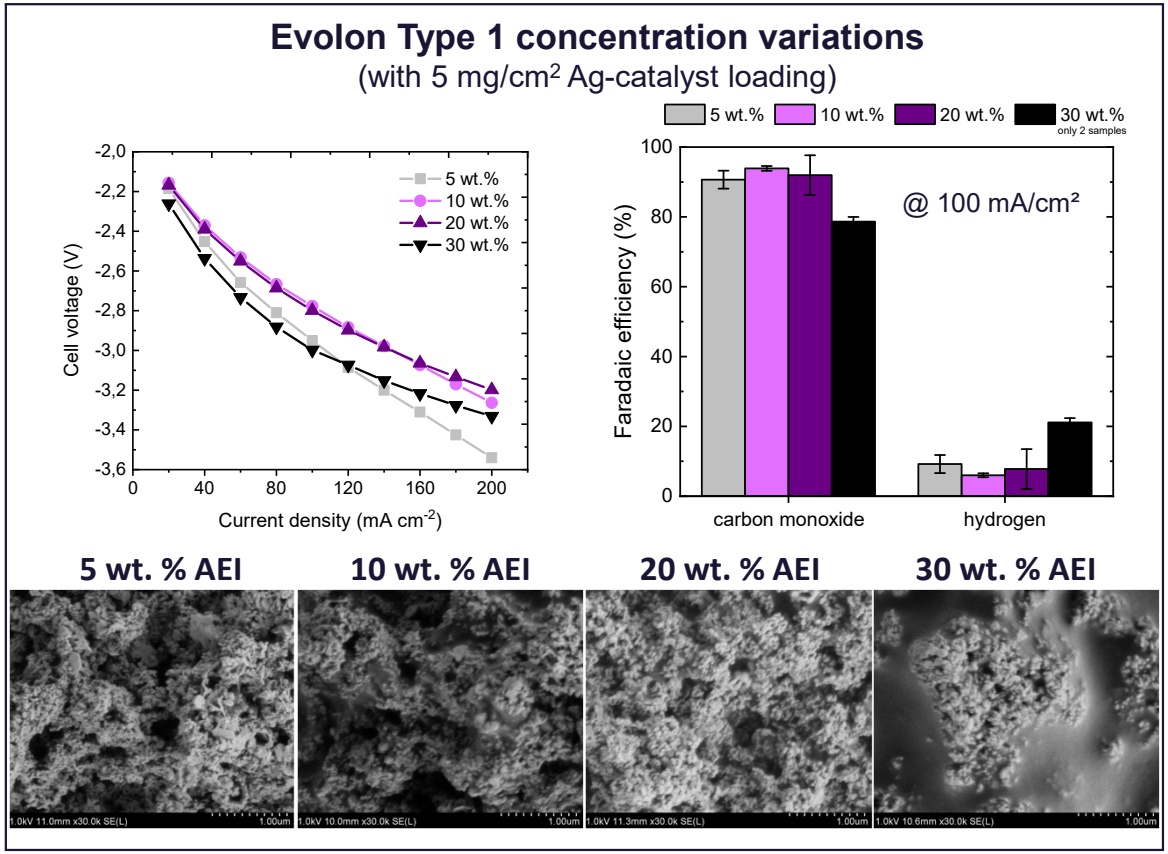
- Processing of the Ag-catalyst/AEI layers into GDEs using spray-coating.
- Investigation of electrochemical properties of the Ag-catalyst/AEI layers.

Result

- Testing procedure established.
- Cell setup is sensitive enough to display specific properties of different AEIs.

Outlook

- Evaluation of AEIs as prepared by Evonik and commercially available AEIs.
- Selection of best AEI for performance optimization and up-scaling.



Involved partners: Dr. A. Maljusch (Evonik); N. Martic, S. Szyszkowski, A. Maltenberger, A. Klinger, T. Reichbauer (SE)

WP4: GDE Characterization by impedance spectroscopy

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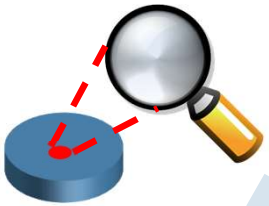
Objective

- Local impedance analysis



Approach

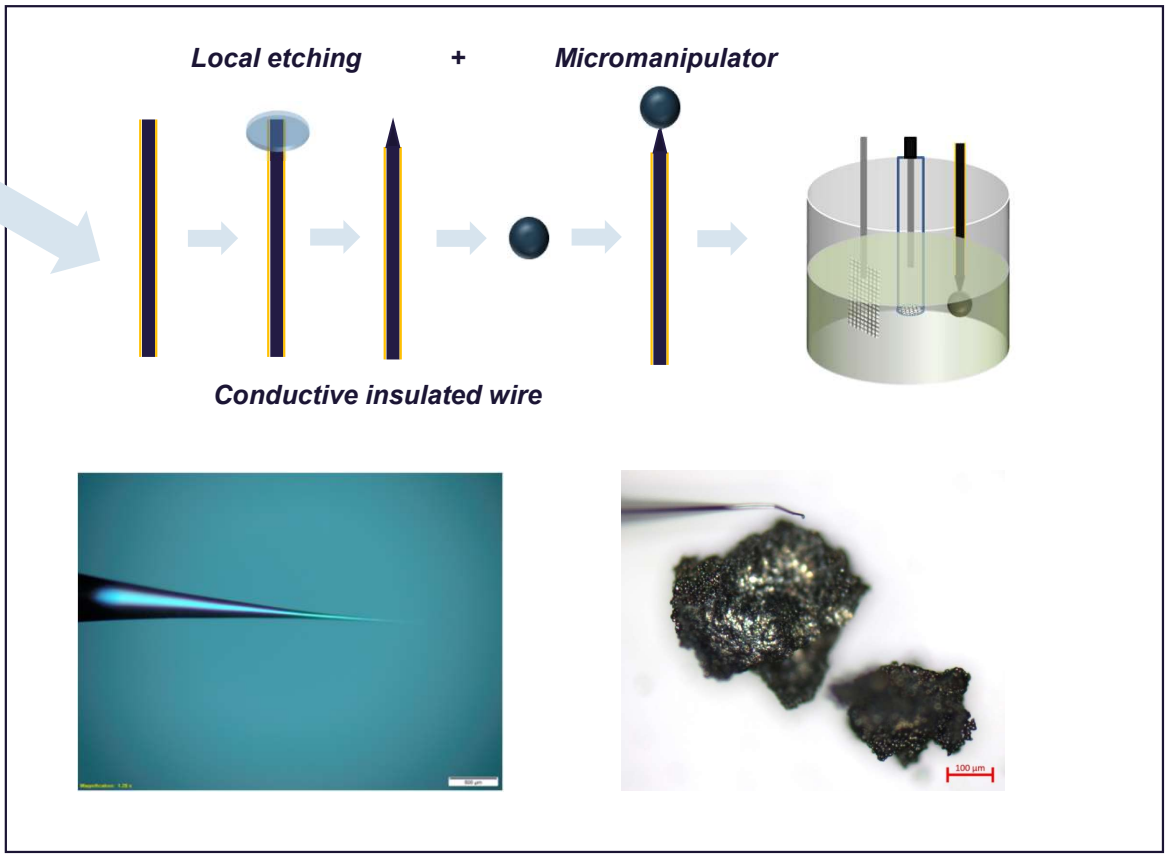
- Preparation of conductive tips
- Placing the particles at the tips and
- EIS investigations

Result

- The conductive tips were successfully produced by local electrochemical etching

Outlook

- Contacting the particles via inactive glues and performing EIS measurements



Involved partners: Dr. H. Tempel, Dr. Y. Emre, Dr. M. Schalenbach

WP4: GDE Characterization by impedance spectroscopy

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JÜLICH Forschungszentrum

Objective

- GDE characterization in the operating cell by impedance spectroscopy (EIS)

Approach

- Plane parallel anode (IrO_2) and cathode (GDE with catalyst). Reference electrode

Result

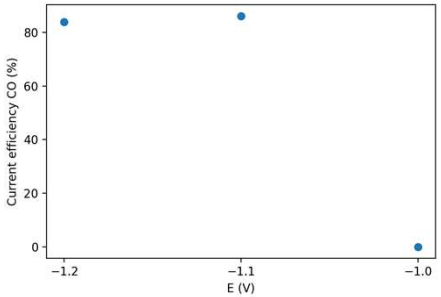
- Not possible to distinguish between hydrogen evolution and CO_2 reduction by EIS

Outlook

- Extract capacitance from impedance spectra and examine flooding/wetting

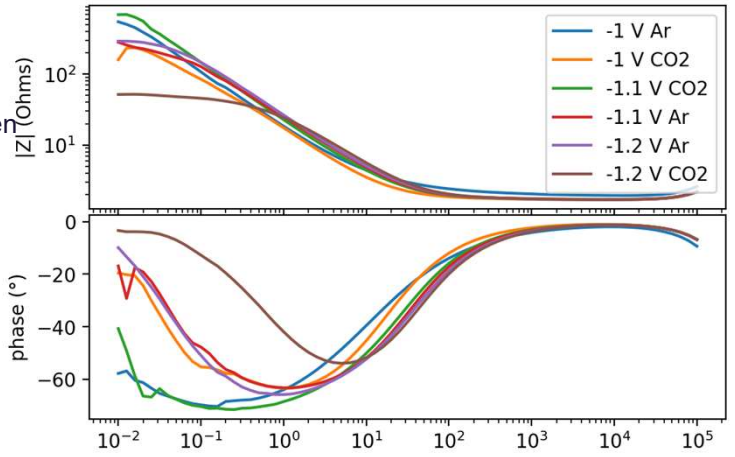
Current Efficiency:

Product composition varies as a function of the applied potential



Impedance spectra:

Not possible to distinguish between hydrogen evolution and CO_2 reduction, as all spectra can be characterized charge transfer resistance, capacitance and electrolyte resistance



Involved partners: Dr. H. Tempel, Dr. Y. Emre, Dr. M. Schalenbach

Summary

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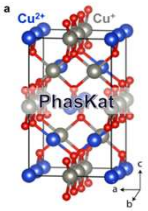

- **WP1: Catalysts**
  - Synthesis accomplished for three catalyst materials.
  - Cu_4O_3 prepared by Evonik showing similar performance to reference samples.
- **WP2: Anion Exchange Ionomers**
  - First AEI samples of EvoIon Type I (5g batch) prepared and evaluated.
  - Synthesis of EvoIon scaled up to 10g batch.
- **WP3: Electrode Processing & Electrochemical Characterization**
  - Inks with higher IPA content contributes to stabilize the catalyst ink.
  - Inks with lower IPA content results more porous structure and better performance.
  - Cell setup and testing procedure for the evaluation of AEIs developed.
- **WP4: Impedance Spectroscopy**
  - Conductive electrodes with sharp tips were successfully prepared.
  - Not possible to distinguish between different reactions in the cell from impedance spectra.

Outlook

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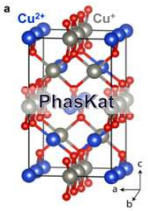

- Preparation of catalysts and AEIs and up-scaling of the synthesis ongoing.
- Continuing evaluation of catalysts and anion exchange materials.
- Selection of best catalyst and AEI for performance optimization and up-scaling.
- Correlating performance with ink composition and catalyst layer structure.
- The EIS analysis of the local electrode will be performed.
- Analyze wetting/flooding of the electrodes during operation in the cell with capacitance measurements.

Acknowledgement

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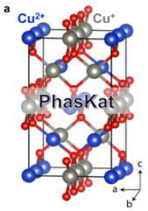

Thank you

for your attention!

Dr. Wiebke Sarfert-Gast

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