The development of a supported Iridium catalyst for oxygen evolution in PEM electrolysers

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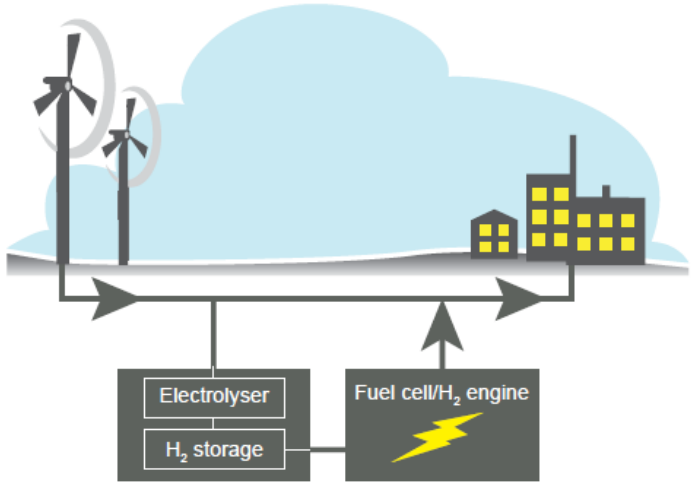
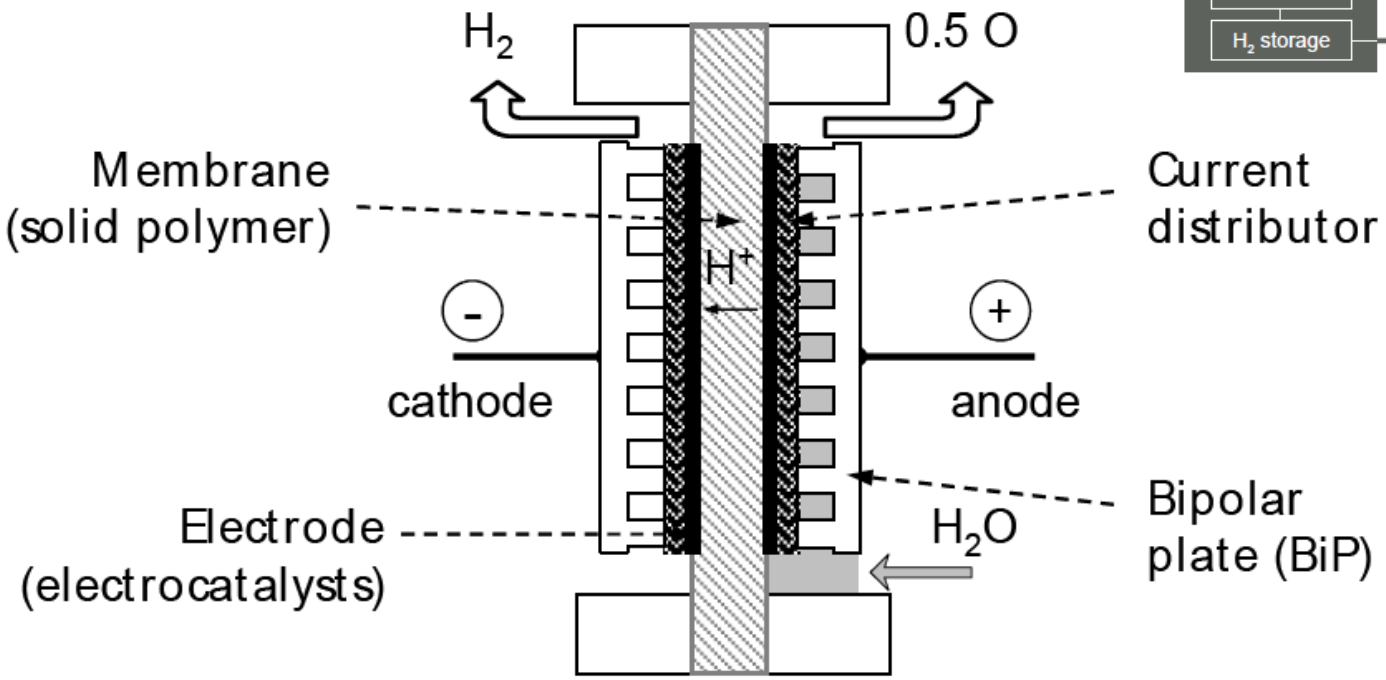
Water Electrolysis

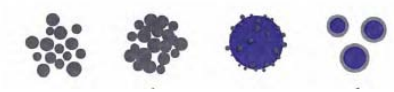




NEXPEL main objective:

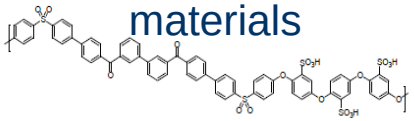
Develop and demonstrate a PEM water electrolyser integrated with RES: 75% Efficiency (LHV), H_2 production cost ~ €5,000 / Nm^3h^{-1}, target lifetime of 40,000 h

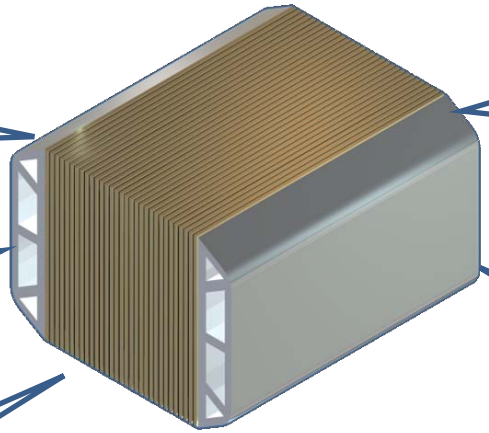
New catalysts



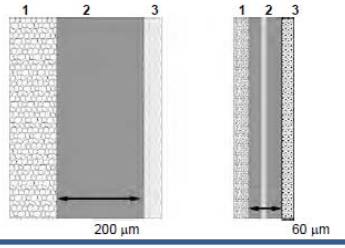
Improved DC-DC converter

New membrane materials

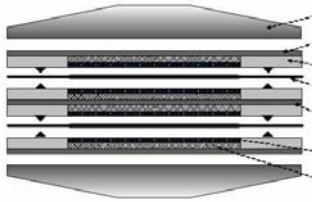




Improved MEAs



Novel stack design and new construction materials



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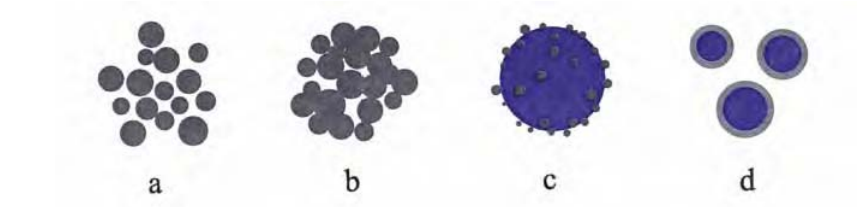
Catalysts for water electrolysis

Topic	Current State of Art	Current Research
O_2 evolution catalysts	Ru or Ir metal / metal oxide Often agglomerated hence not using active material to its full potential Loading: up to 6 mg cm^{-2}	Ir-Ru alloy or core shell catalyst Supported Ir / Ir-Ru or Ir oxide /Ru oxide catalyst also may be enhanced by the addition of transition metal elements Loading: 0.8 mg cm^{-2}
H_2 evolution catalysts	Material: Platinum Black Loading: 2 mg cm^{-2}	Material: Pt-Pd alloy or core shell catalyst Supported Pt or Pt-Pd on Carbon or carbon nanofibres Loading: 0.2 mg cm^{-2}

O_2 evolution catalysts

- **Catalysts**
  - Ruthenium oxide most active but also highly unstable
  - Iridium oxide More stable – very promising
  - Enhancement of Ir by alloying with other metal /metal oxides

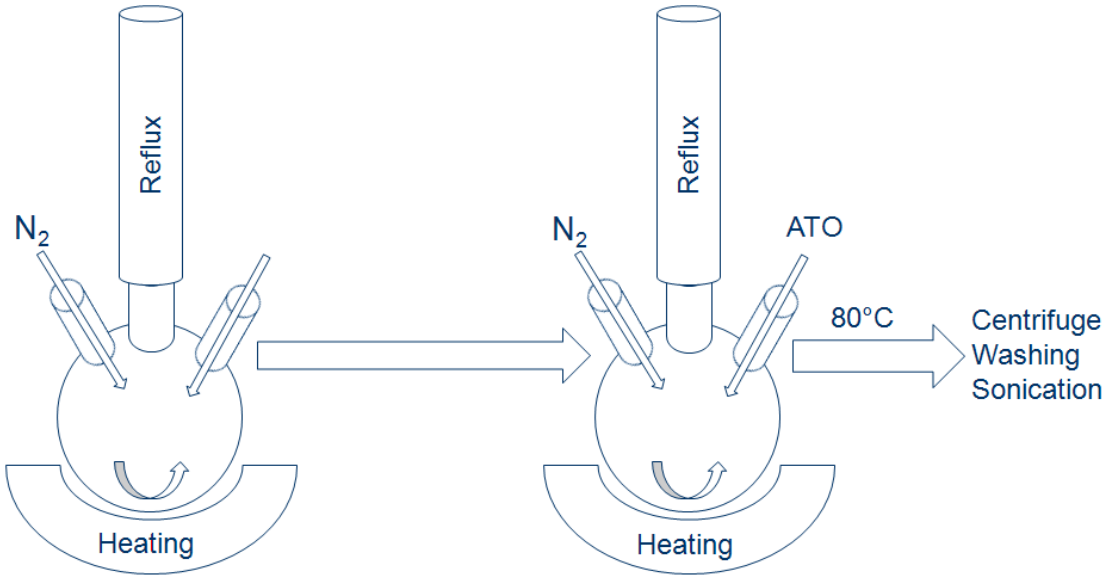
- **Support materials**
  - Must withstand potentials of up to 1.8V at temperature 80°C over long duration



Mono dispersed particles | *Sintered mono dispersed particles* | *Supported catalyst* | *Core-shell structure*

Polyol Synthesis Method

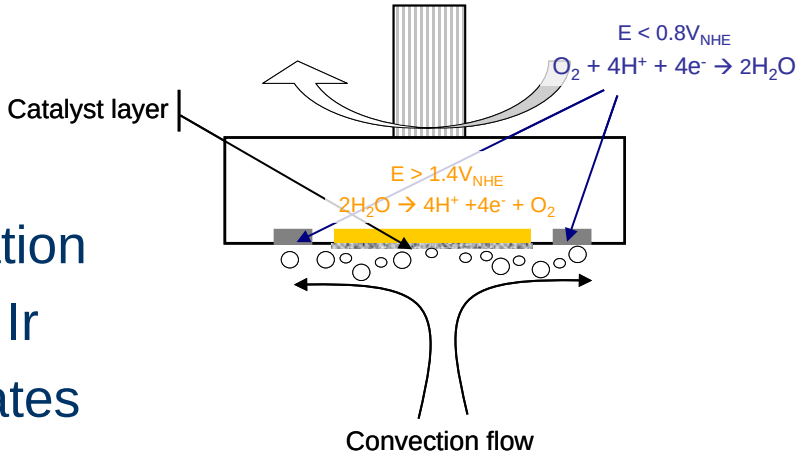
1. **Isolate Iridium**
   - Reflux percursor
   - pH adjusted solution
   - high temperature
2. **Add support**
   - Antomony Tin Oxide
   - well dispersed
   - reflux lower temperature
   - Adjust pH
3. **Isolate catalyst**
   - Centrifuge to remove EG
   - Sonicate and further centrifuge until pH is that of rinsing water



Electrochemical Characterisation

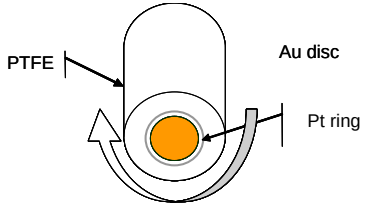
Cyclic Voltammetry

- Carried out at a variety of scan rates
- Establish inner and outer charge (mC)
- Use outer charge to normalise polarization
- Show characteristic oxidation peaks of Ir
- In case of Ir-Ru change in shape indicates presence of Ru

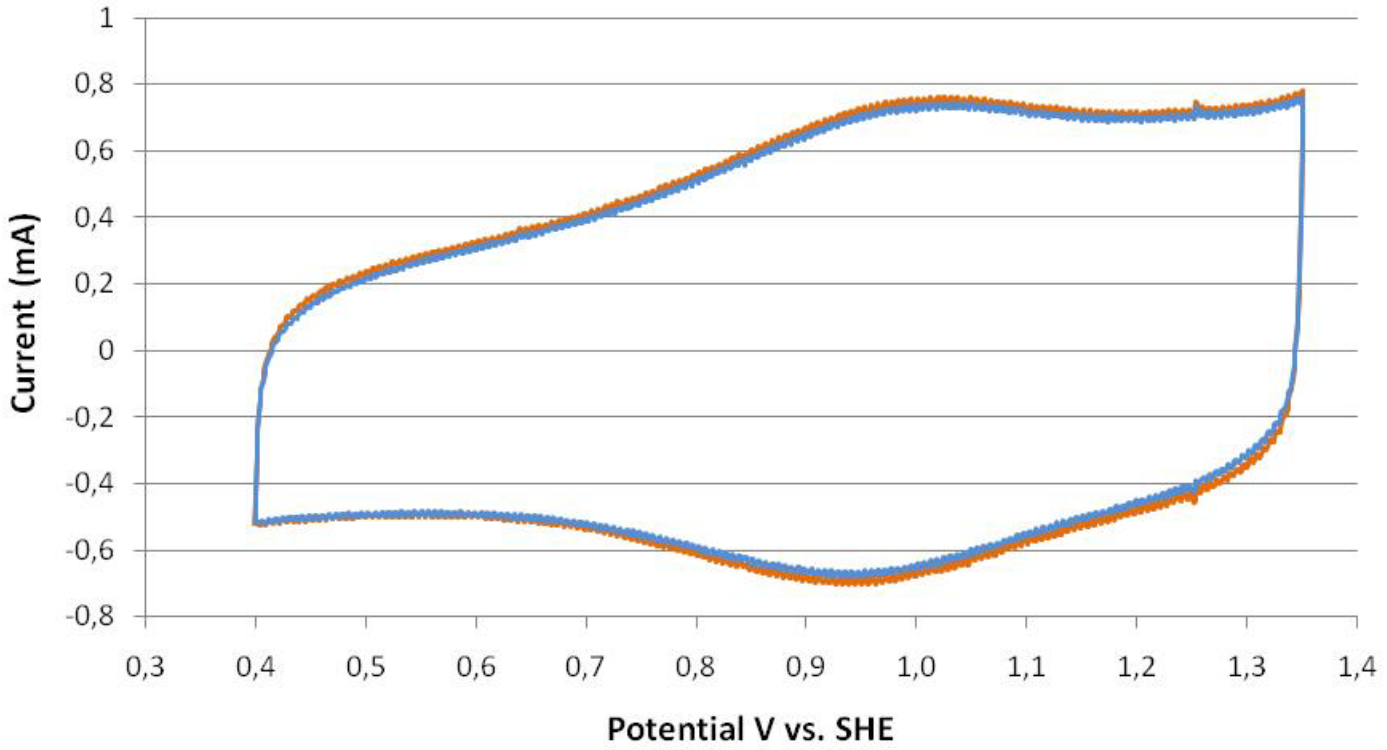


Linear Polarization at RDE

- Carried out with rotation to 1.6V vs. SHE
- Establishes specific activity of catalyst
- Method to compare catalyst performance



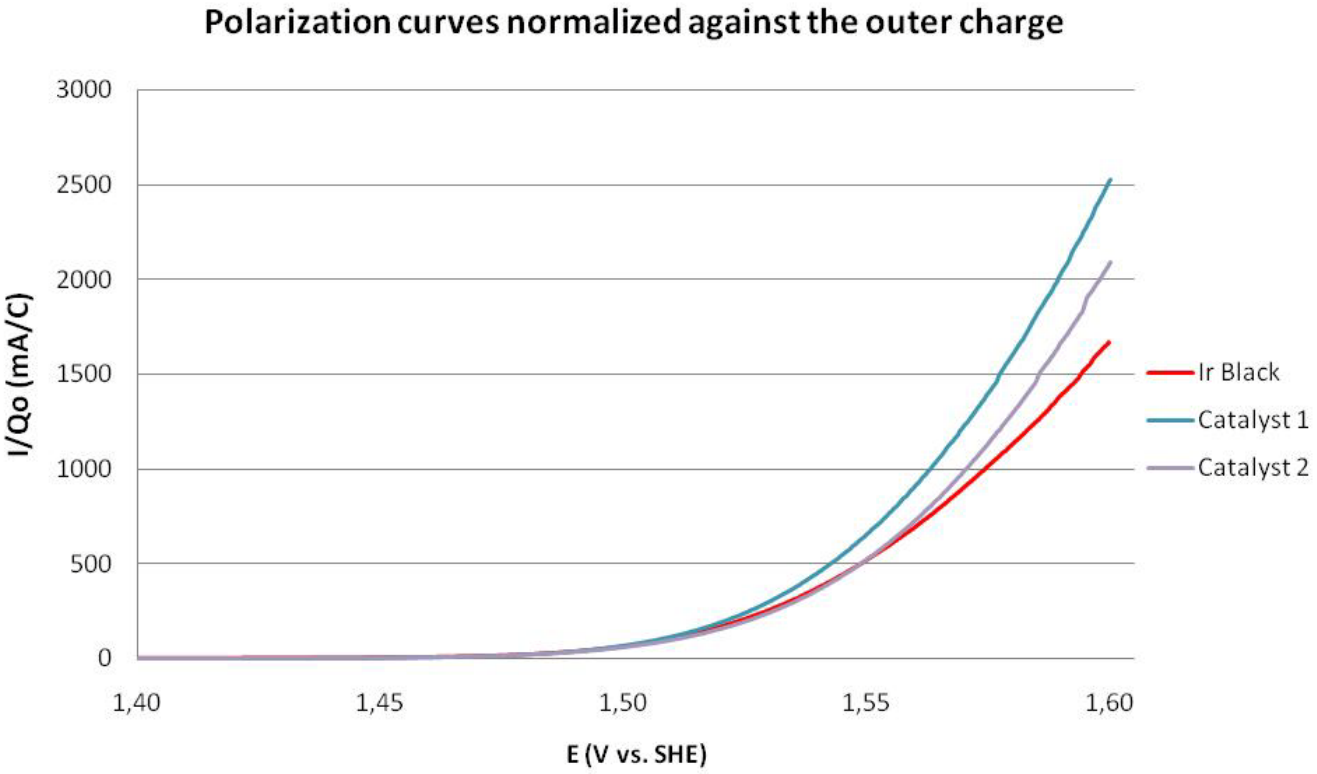
Cyclic Voltammetry



CV of Ir ATO catalysts carried out at 300mV s^{-1} in 0.5M H_2SO_4

- Cyclic voltamograms for 20% Ir on ATO catalyst exhibit peaks characteristic of Ir / IrO_2

Linear Polarization Results

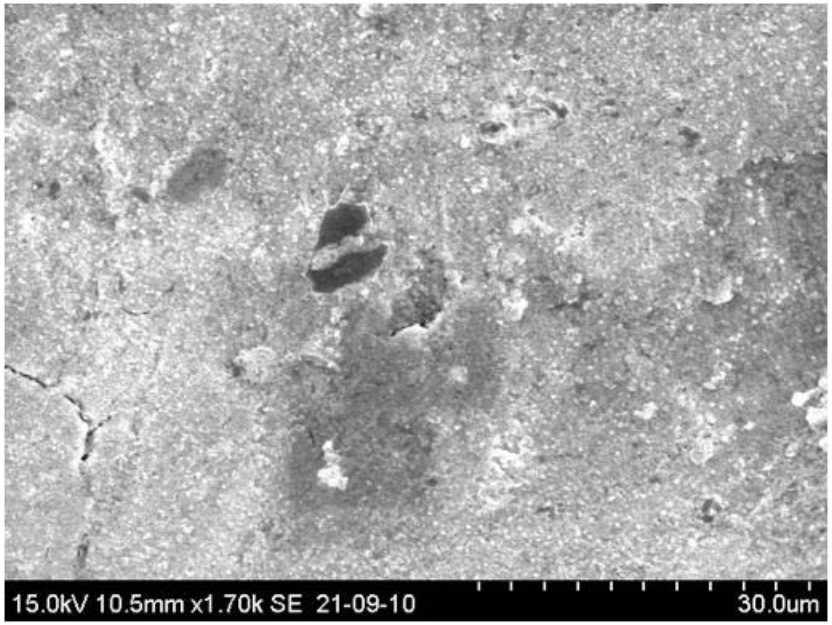


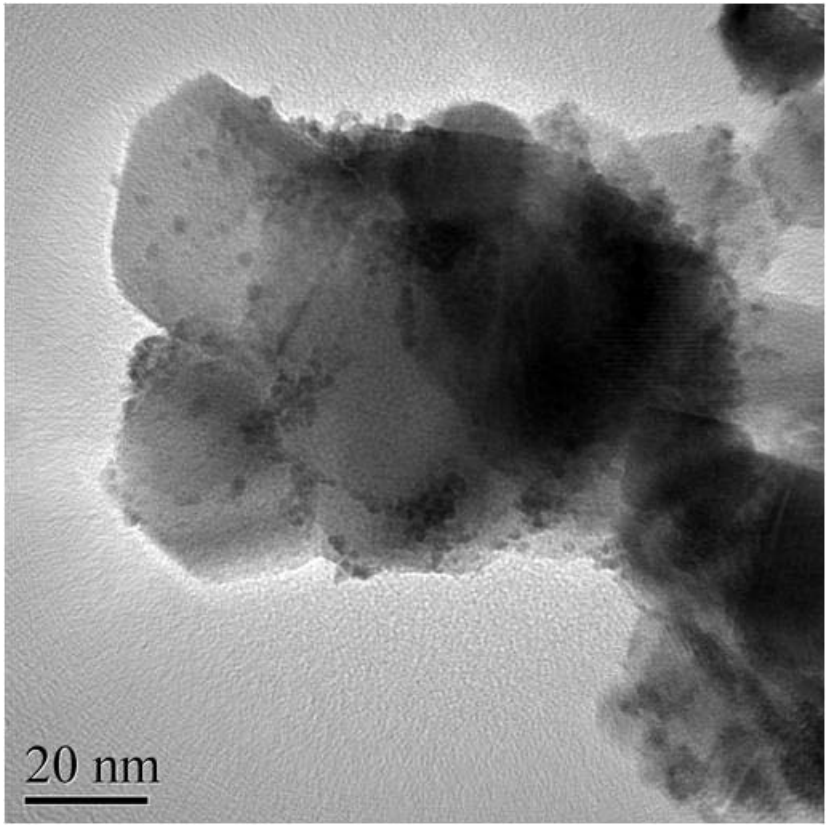
- Polarization curves are useful for initially comparing catalyst activity.
- Potentials higher than 1.6V are not recommended as even with rotation the O2 evolved blocks the electrode surface from electrolyte.

Physical Characterisation

- SEM and TEM
  - - information on catalyst particle size and particle size distribution
- EDS
  - - can give rough values concerning catalyst loading on the support
- TGA – DSC
  - - in the case of ATO Ir catalyst does not provide information
- XRD
  - – Has not provided information as Ir catalyst particles are too small
- ICP
  - - Current investigation to determine the exact catalyst loading

SEM and TEM images

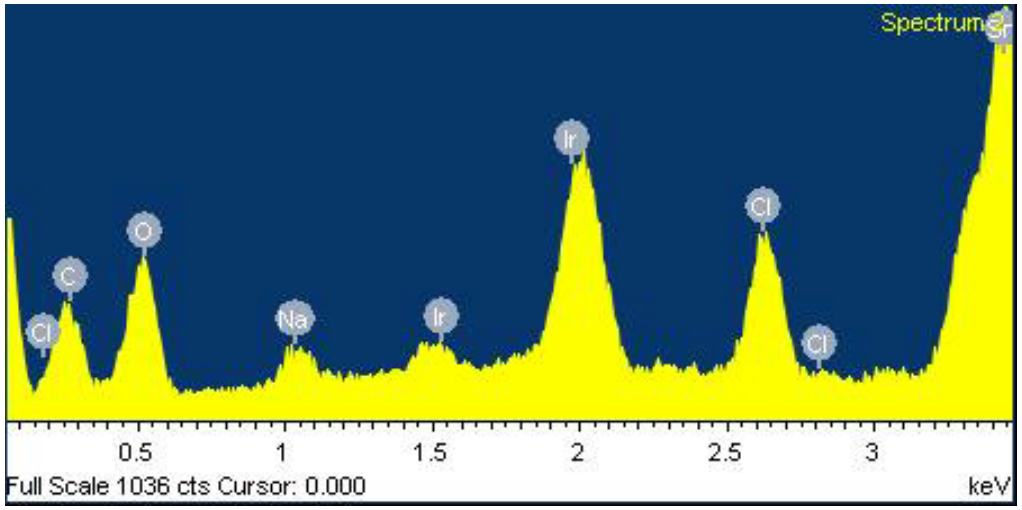


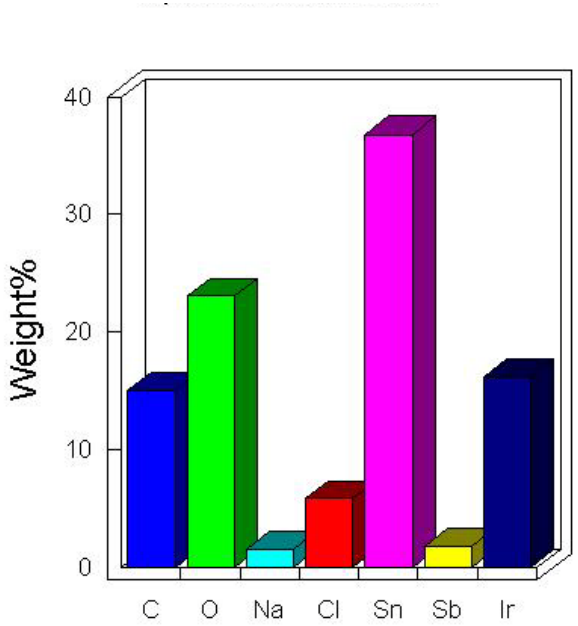


- **SEM** - Catalyst is dispersed using ultrasonic bath in isopropanol and then deposited by pipetting.
- **TEM** - 2nm Particles of Iridium on 50nm ATO support material. Some agglomeration of the Ir around edges of ATO particles.

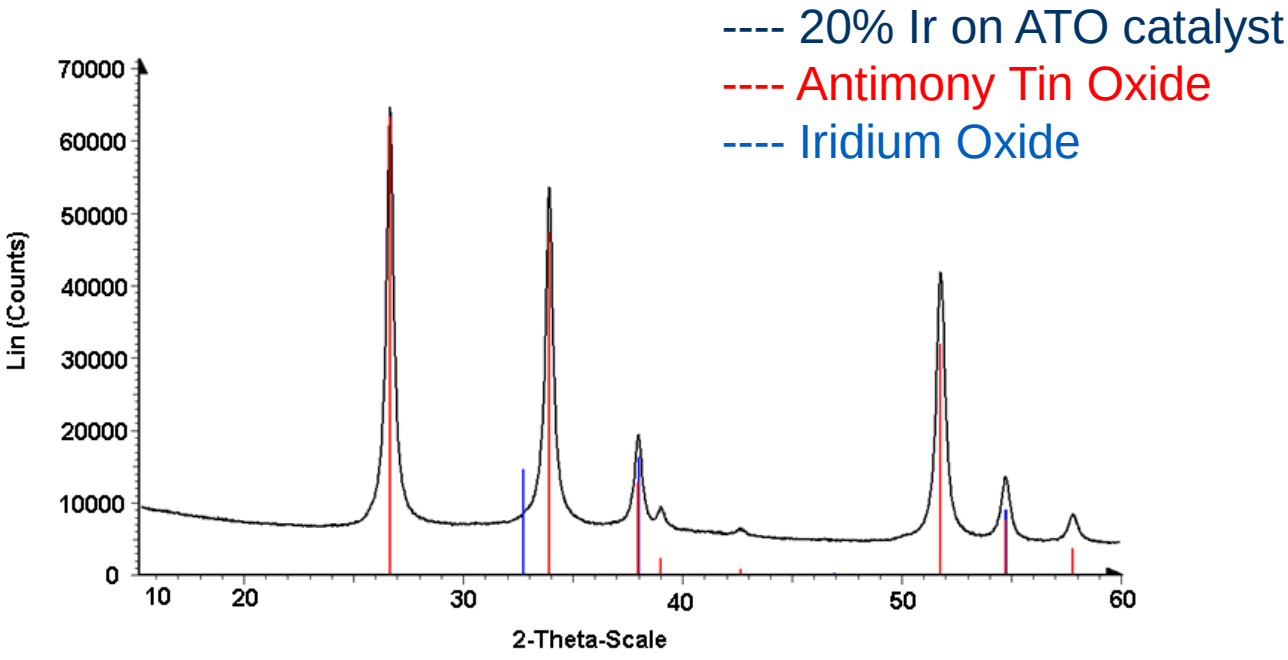
EDS Results

Catalyst	Loading target	EDS estimate loading	Current at 1,6V	Corrected current at 1,6V
Catalyst 1	20% Ir	18.5% ± 1.3	412	471
Catalyst 2	20% Ir	15.4% ± 1.2	238	309



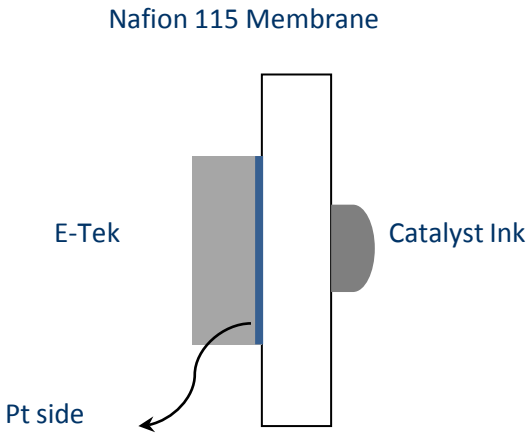


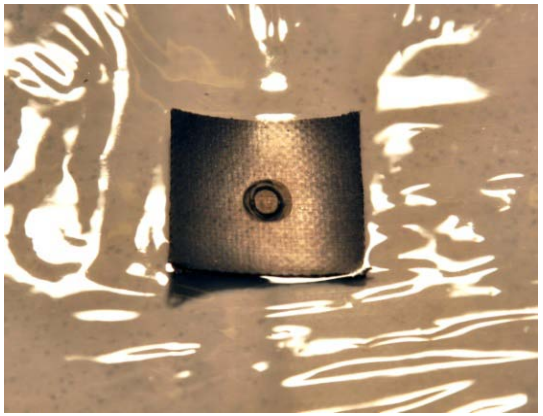
XRD Analysis



- No indication of presence of Iridium as particles are too small and non crystalline
- Conditions:15-60° ,Count time 15 s/step, Step size 0.02°

In-situ cell testing - Pipette method

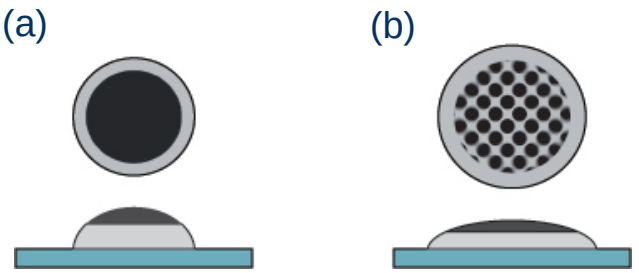




Advantage	Disadvantage
- Close to real MEA testing - Small amount of catalyst needed - Good control over total amount of catalyst - Relatively fast and easy method of making catalyst layer on Nafion membrane.	- Less control of catalyst loading (mg/cm^2) than by ordinary MEA preparation methods.

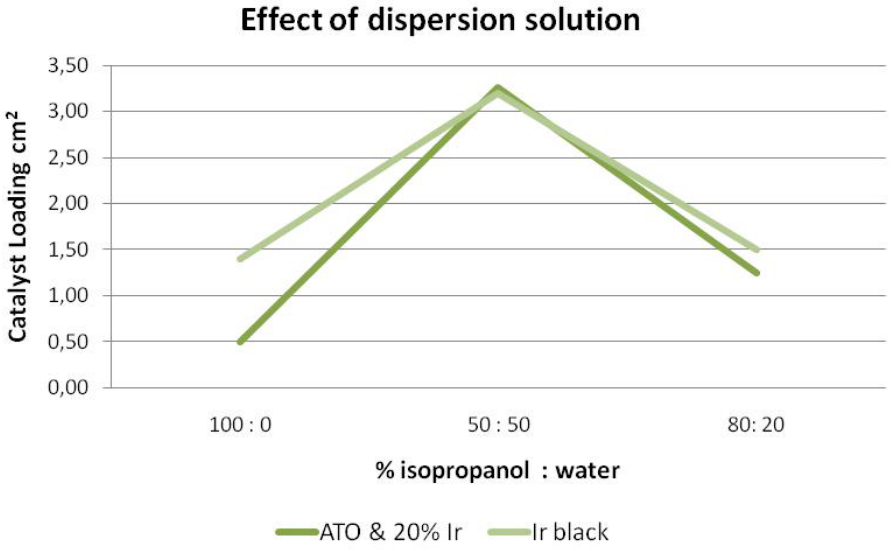
Catalyst loading & Pipette method

- **Procedure to load catalyst**
  - added to isoprop: water solution
  - Sonicated for 5 min
  - Stirred for 1 hour
  - 20 µl pipetted on membrane

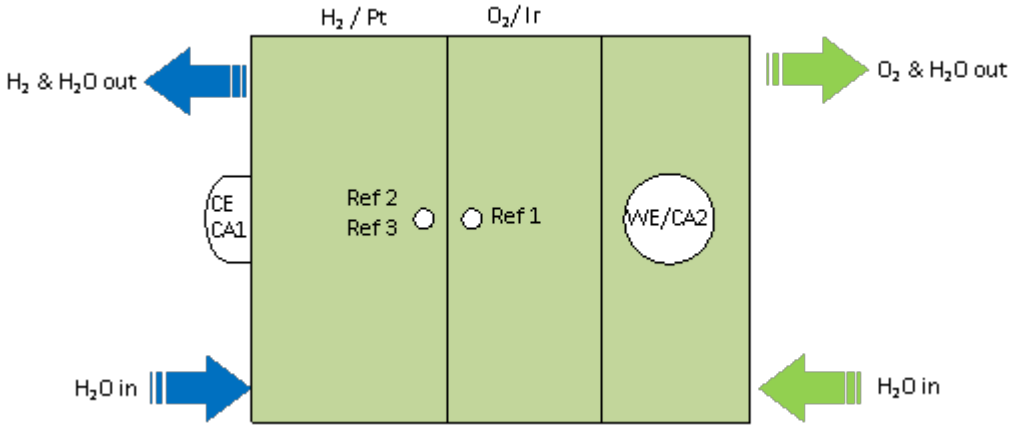


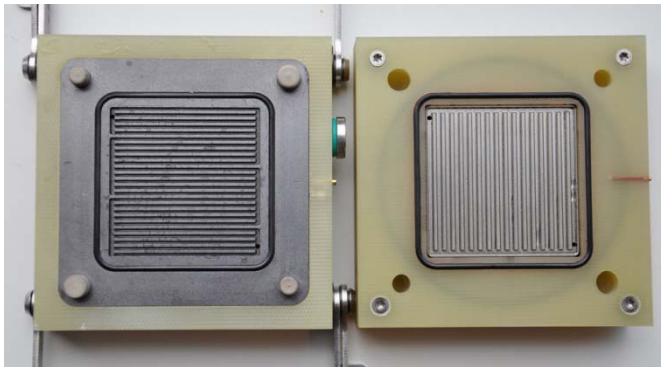
- Isoproponal surface tension too low : little control of dot symmetry
- 20% isopropanol water causes catalyst to clump (b)

- Ideal 50: 50 soln
  - Loading ca 3.2mg of catalyst
  - 20% Ir catalyst = 0.65g cm^2 Ir

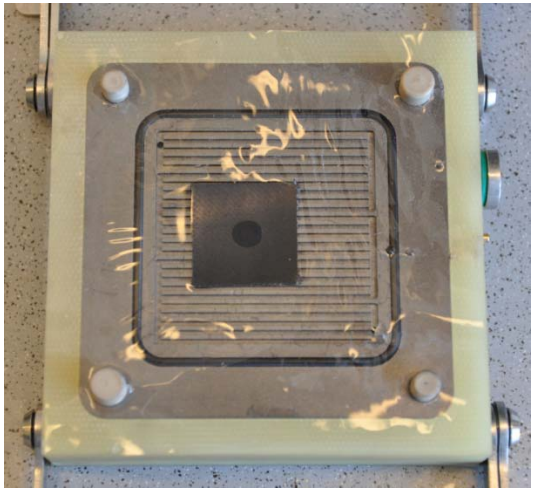


In-situ testing of catalyst in real electrolyzer cell

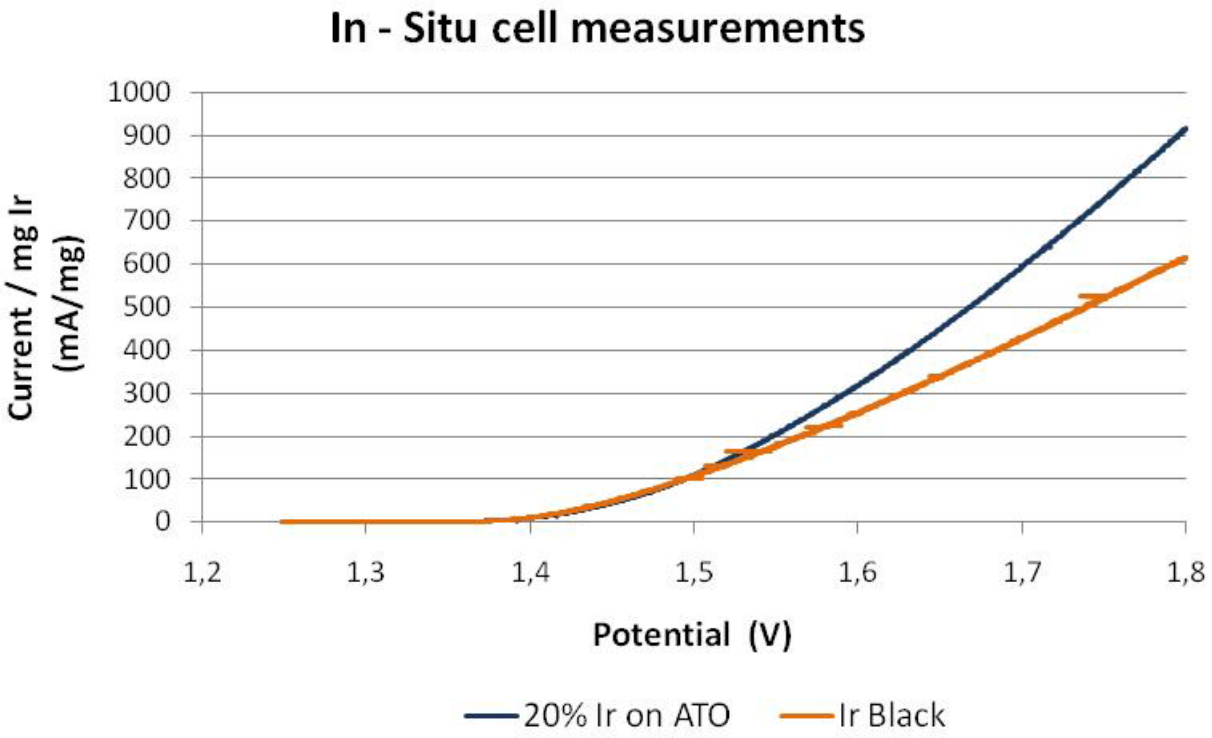




Titanium flow field



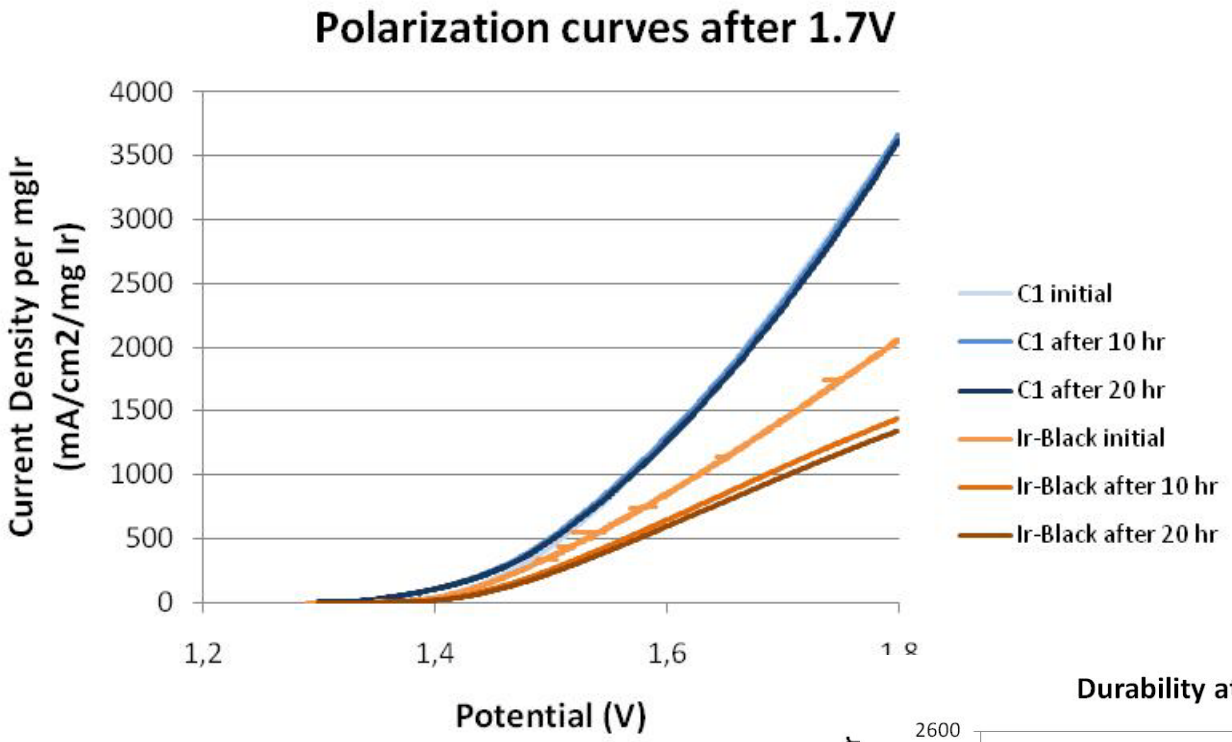
Experimental results from real cell



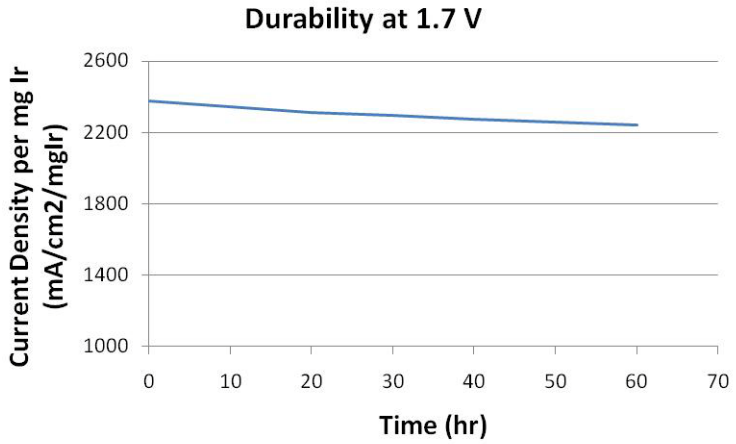
- 80°C
- 11 Bar clamping pressure
- H_2O 238 ml/min

Potential	Ir Black	20% Ir:ATO
V	A /cm^2/mg	
1.6	3.66	1.27
1.8	2.05	0.84

Durability

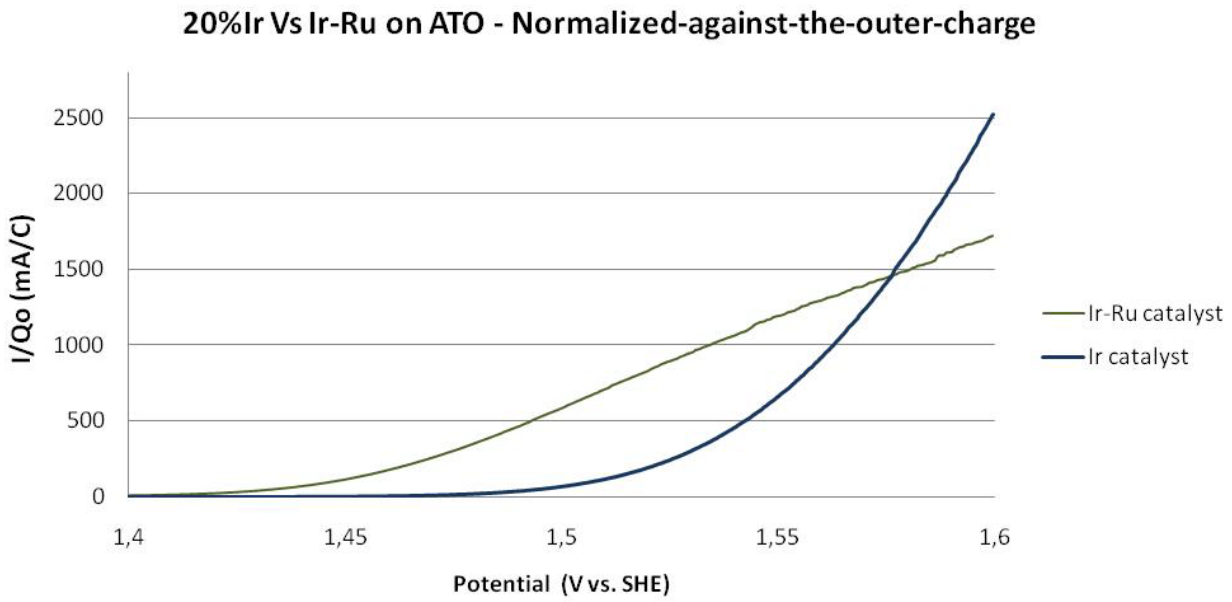


- **Durability test**
  - 1.7 V
  - 11 Bar clamping pressure
  - 80°C
  - Linear polarization after every 10 hrs



Future work

- Investigation to obtain optimal Ir loading values
- Incorporation of Ru with Ir – advantage O_2 evolution begins at significantly lower potential.
- Inverse polyol synthesis – more convenient method



Conclusions

- The deposition of fine 2nm particles of Iridium on Antimony Tin Oxide (ATO) by a polyol synthesis method has been demonstrated.
- XRD and SEM provides little information on this catalysts due to the small Ir particle size.
- The performance of the 20% Ir on ATO has been demonstrated to be considerably better providing a current of 3.66 A /cm^2/mg at 1.6V compared to that of 1.27 A /cm^2/mg for Iridium black.
- Antimony Tin Oxide (ATO) has been demonstrated as a suitable support material for water electrolysis catalysts with good durability.

Project Team and Acknowledgments

Thank you for your attention

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Effect of pressure