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SAVANNAH RIVER NATIONAL LABORATORY
Operated by Savannah River Nuclear Solutions, LLC

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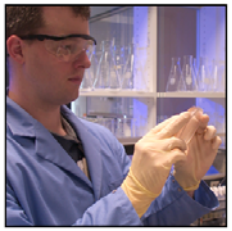
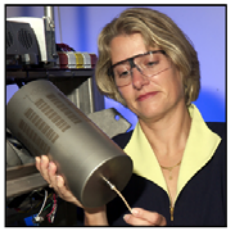
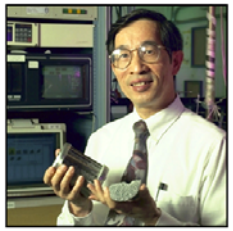
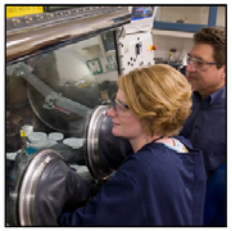
Hybrid Sulfur Electrolyzer Component Development Summary

David Hobbs

April 20, 2009

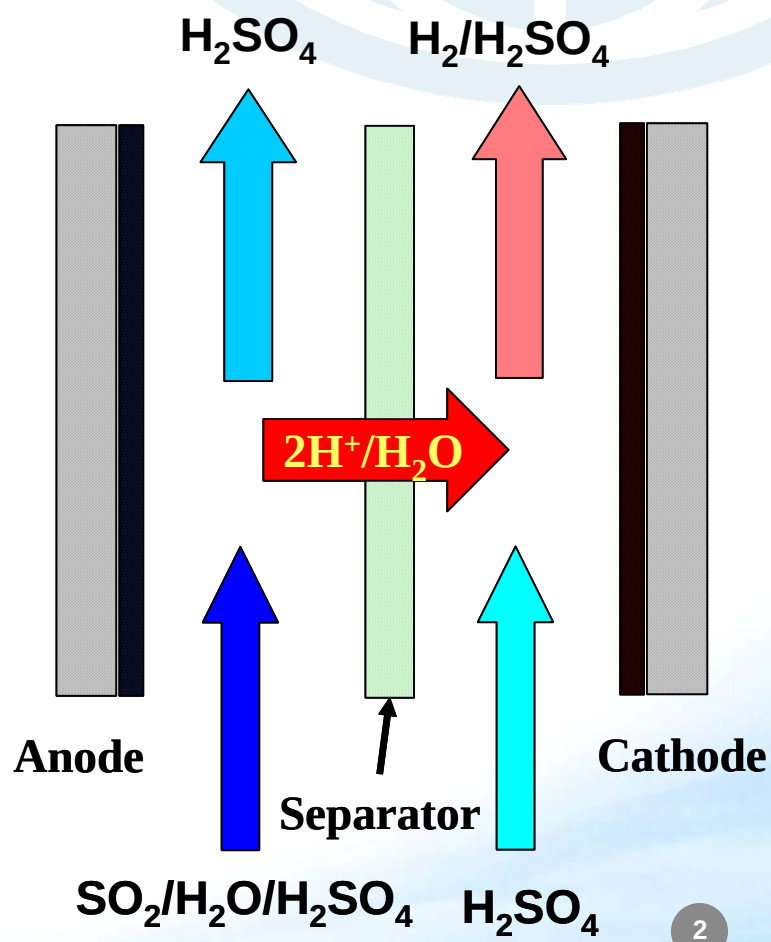
HyS Electrolyzer Workshop and Information Exchange

SRNL-STI-2009-00263

History of HyS Process Development

Patent for "Sulfur Cycle" issued to Westinghouse	1975
Two-compartment Diaphragm Cell Built	1977
Closed-loop Process Demonstration by (W)	1978
Solar-driven Process Design Completed by (W)	1983
Development "Hiatus"	1984-2003
New Process Design work by (W)	2004
Conceptual Design of HyS by SRNL	2005
Proof-of-Concept for PEM-based SDE	2005
Pressurized, Elevated Temperature SDE Testing	2006
Improved PEM Design; 100-hr Longevity Test; Multi-cell stack SDE	2007
Alternate Membrane & Catalyst Evaluations	2008

Original 2-compartment cell design



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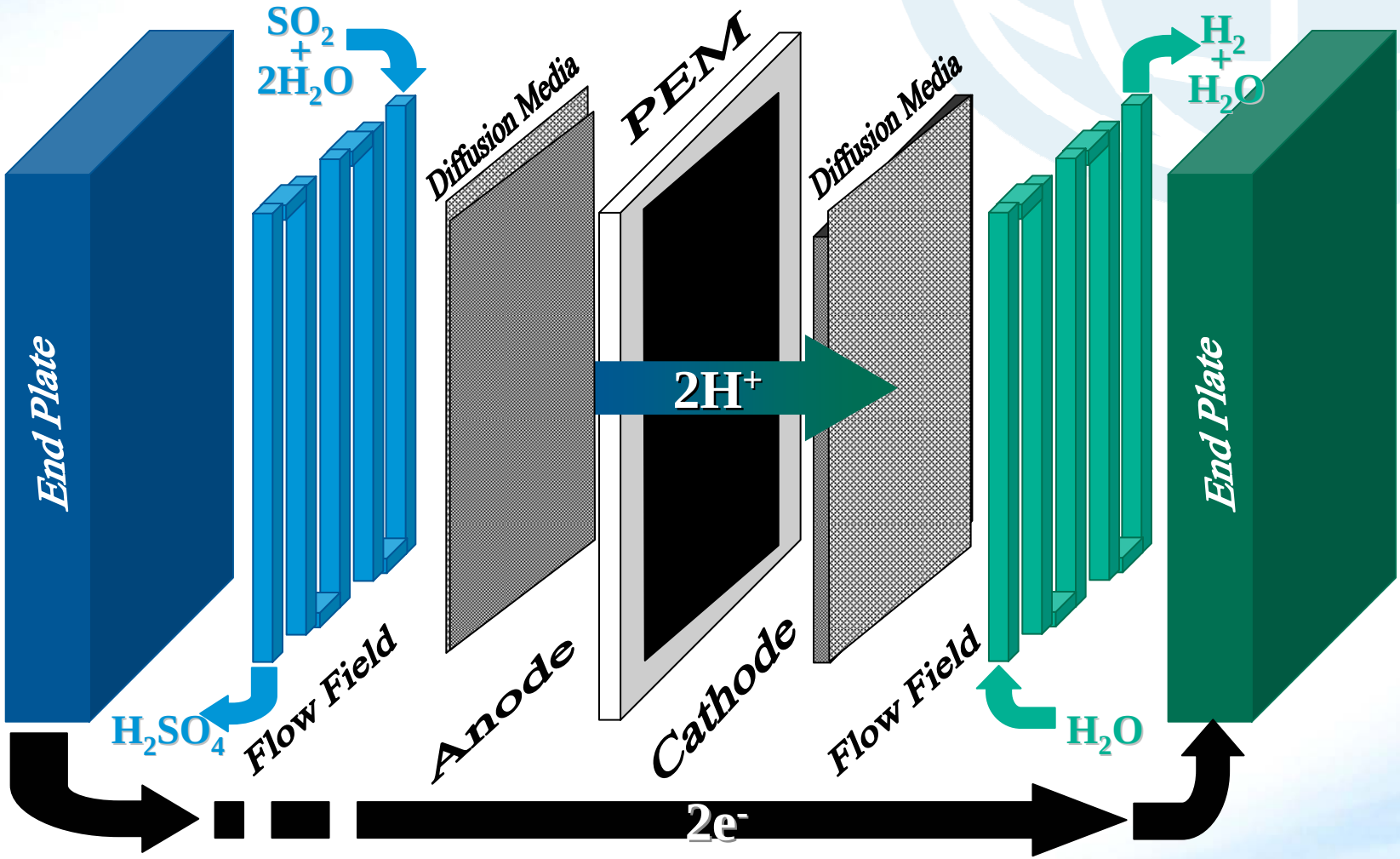
PEM Electrolyzer



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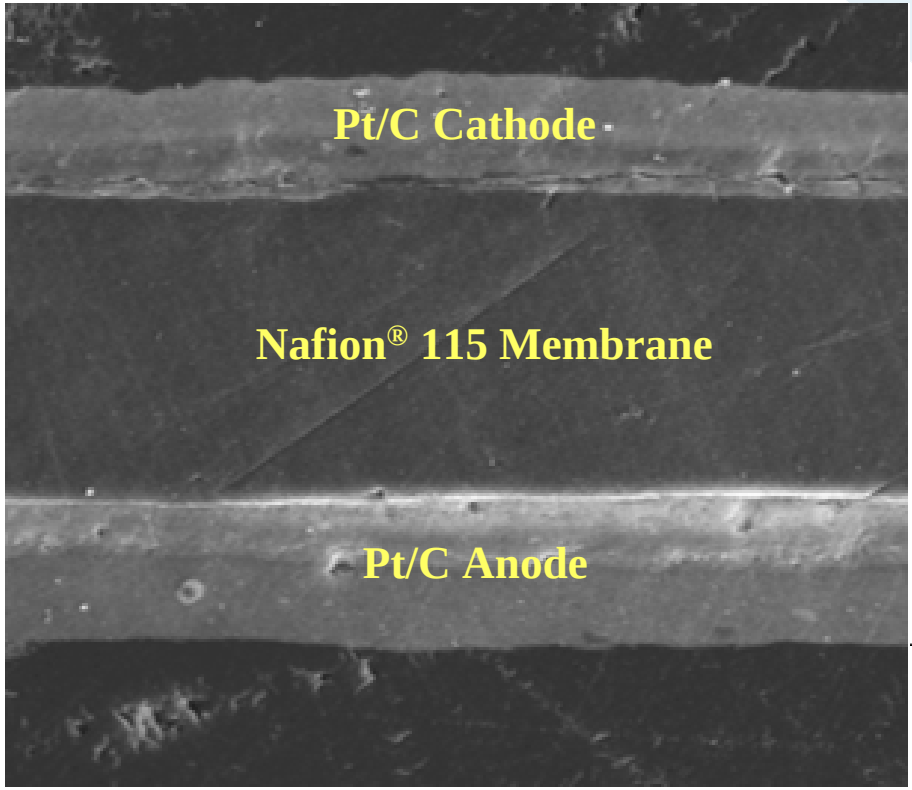
Membrane Electrode Assembly (MEA)

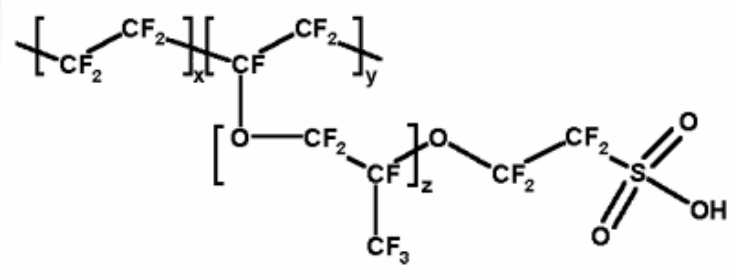


SEM Image

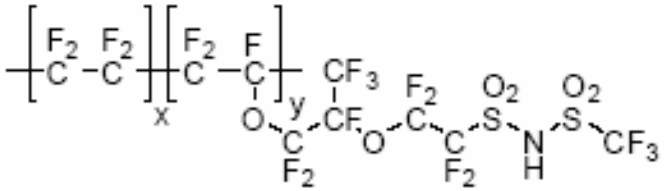
Key Electrolyzer Components

- Proton Exchange Membrane
- Anode
- Cathode
- Flow Field/Diffusion Media

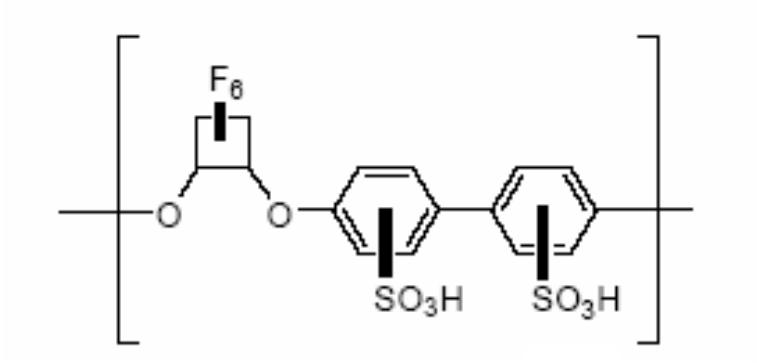
Membrane Candidates



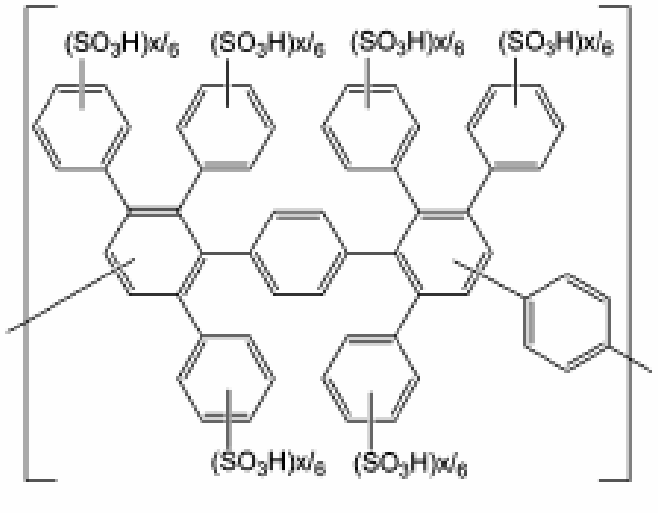
perfluorosulfonic acid (PFSA)



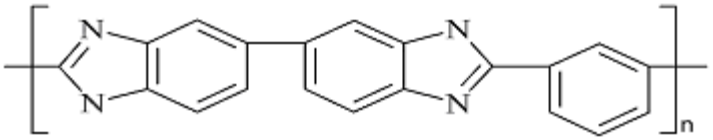
perfluorosufonimide (PFSI)



Sulfonated perfluorocyclobutyl (S-PFCB)



Sulfonated Diels Alder polyphenylene (S-DAPP)



Polybenzimidazole (PBI)

Electrocatalysts

- **Anode**
  - **Pt/C**
  - **Pt black**
  - **Pt/TM alloys (oxidation resistant)**
- **Cathode**
  - **Pt/C**
  - **Pt black**
  - **Pt/TM alloys (tolerate S)**

Flow Field/Diffusion Media

- **Graphite block**
- **Carbon paper**
- **Carbon cloth**

Analytical Needs

Analytical Needs

- R&D activities
  - understand electrochemistry
  - identify minor reaction products
- Electrolyzer operation
  - process control
  - performance & lifetime indicators

Analytical Needs

- Reagent concentrations
  - SO_2 , H_2SO_4
- Product concentrations
  - SO_2 , H_2SO_4 , H_2 , reduced S species
- Electrolyzer conditions
  - voltage
  - current
  - temperature
  - pressure
  - fluid flow rates

SO_2 Determination

- Standard method is iodometric titration using starch indicator (Std Method 4500-SO32-B)
  - Off-line measurement that requires sampling method
  - SO_2 converted to SO_3^{2-} to prevent outgassing
  - SO_3^{2-} susceptible to oxidation
  - High temperature and pressure can lead to errors
- AD evaluation recommended spectroscopic method for on-line measurement
  - UV-Vis or Raman

H_2SO_4 Concentration

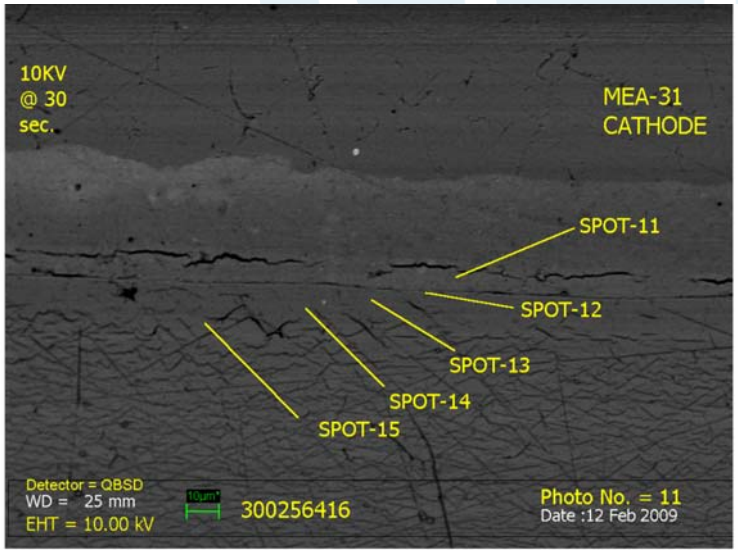
- Off-line: pH titration
  - presence of SO_2 can lead to errors
- On-line: density
  - currently used with the EDL electrolyzer
  - installing system on button cell

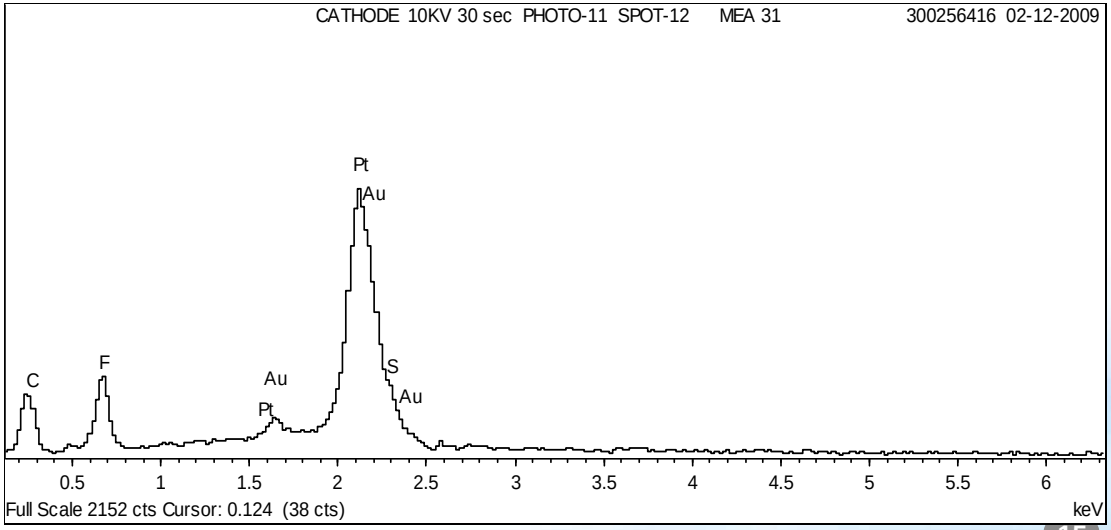
Catholyte Products

- Measure H_2 and S-containing species
- Gases identified by gas chromatography or mass spectroscopy
  - installing Agilent 3000A dual column micro GC on the EDL system
- Off-line measurement of particulates
  - SEM/EDAX
  - ICP-ES

Post-test Characterization of MEA

- Scanning electron microscopy (SEM)
- Mount small piece of MEA in epoxy
- Cure & polish surface
- Obtain images and elemental information (EDAX)
- Membrane subject to degradation by e^- beam





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