

# Neutron Tomography, Imaging, and PEM Fuel Cells

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The NIST logo consists of the letters "NIST" in a bold, black, sans-serif font. The "N" and "I" are connected, and the "S" is also connected to the "T".

National Institute of  
Standards and Technology  
Technology Administration  
U.S. Department of Commerce

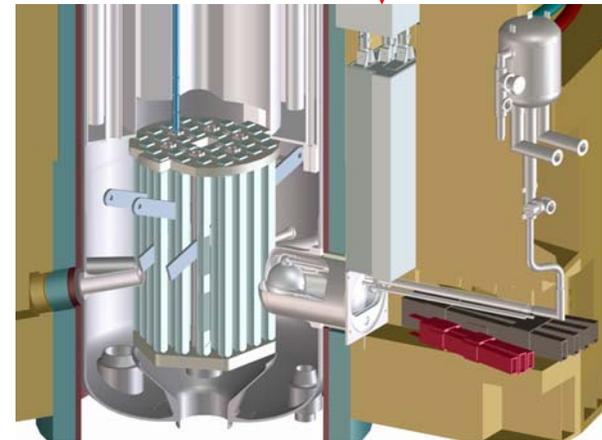


# National Institute of Standards and Technology Center for Neutron Research

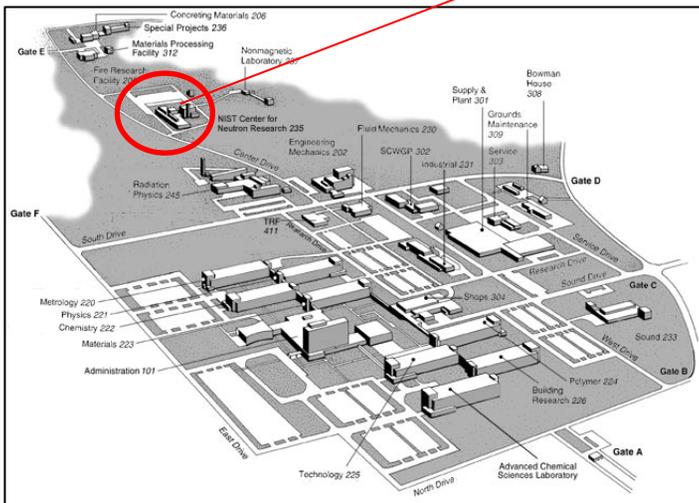
- Gaithersburg, Maryland Main campus 234 hectare (578 acre)
- Boulder, Colorado 84 hectare (208 acre)
- Annual operating budget of about \$771 million
- NIST employs about 3,000 scientists, engineers, technicians, and support and administrative personnel.
- About 1,800 guest researchers complement the staff.
- In addition, NIST partners with 2,000 manufacturing specialists and staff at affiliated centers around the country.



Cold Neutron Guide Hall

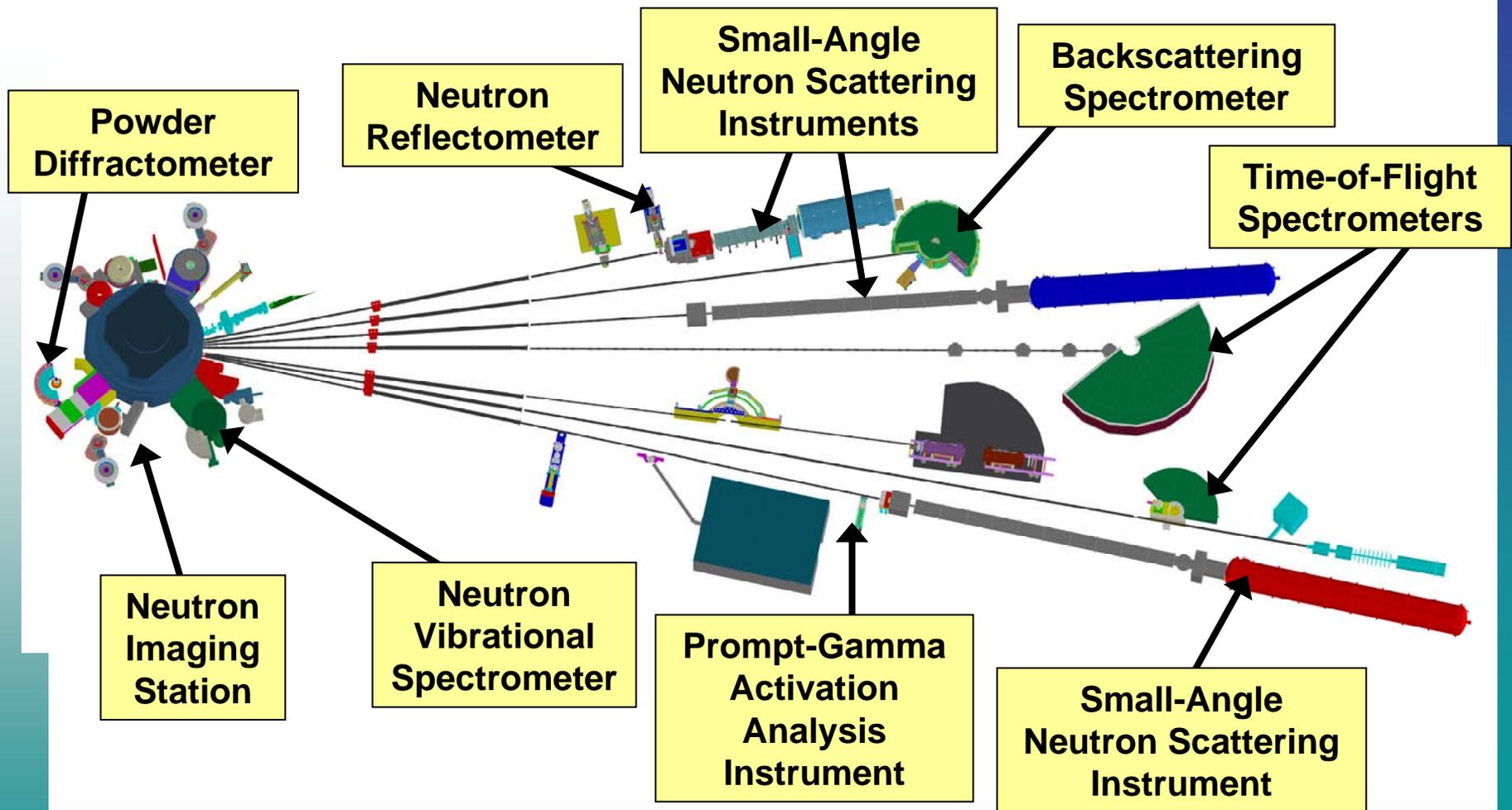


20 MW NBS Reactor



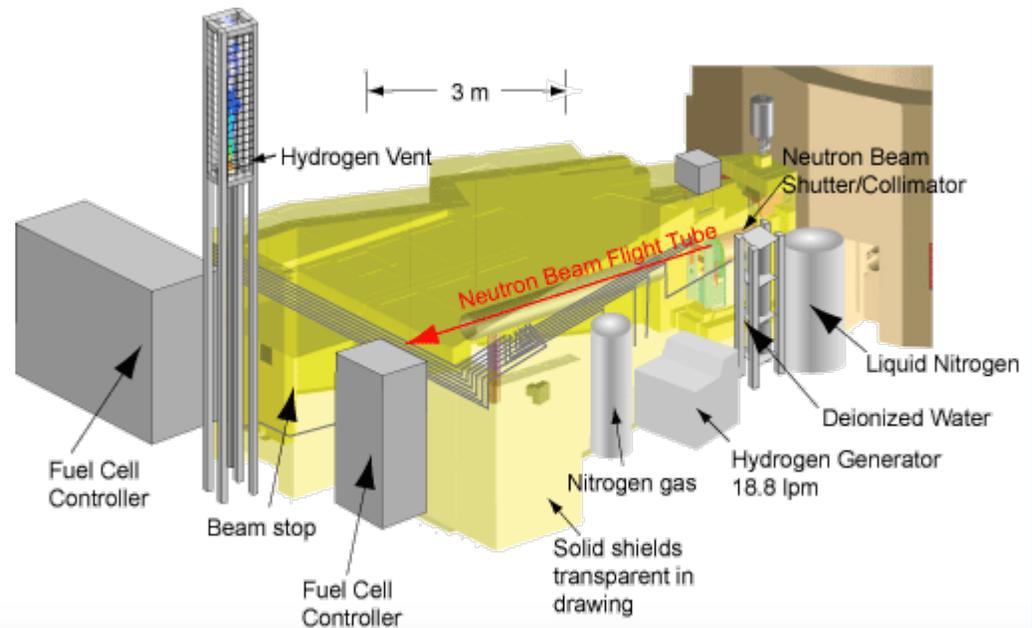
- NCNR provides a large cold neutron research facility.
- U.S. national user facility
- Instruments are staffed and run by many different divisions at NIST
- Reactor facility runs 38 days with an 11 day shutdown for roughly 240 – 280 days/year

# Neutron Instrumentation for Hydrogen-Materials Research at NIST

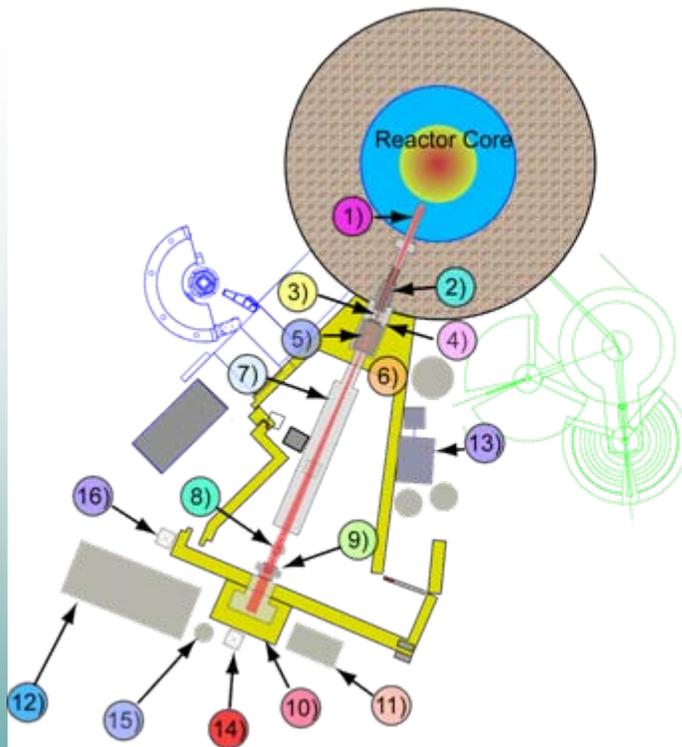


# Neutron Imaging Facility

- New facility replaced first facility built in 2003.
- New facility first users February 27, 2006
- Located at BT-2
- Much bigger 30 m<sup>3</sup>
- House gases/fluids
  - Hydrogen (18.8 slpm)
  - Oxygen (11.1 slpm)
  - Nitrogen
  - Air
  - Deionized water
  - Chilled water
- Freeze chamber for low temperature testing (-40 °C)
- Fuel cell test stands available and supported by NIST staff



# Schematic of New Facility



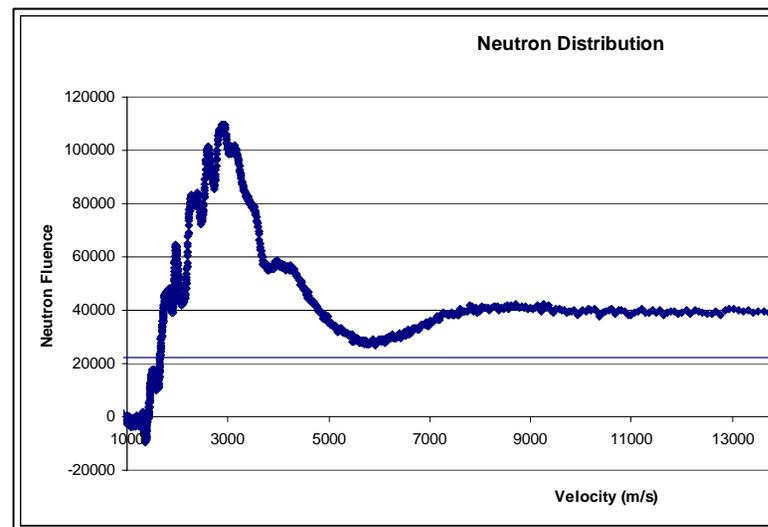
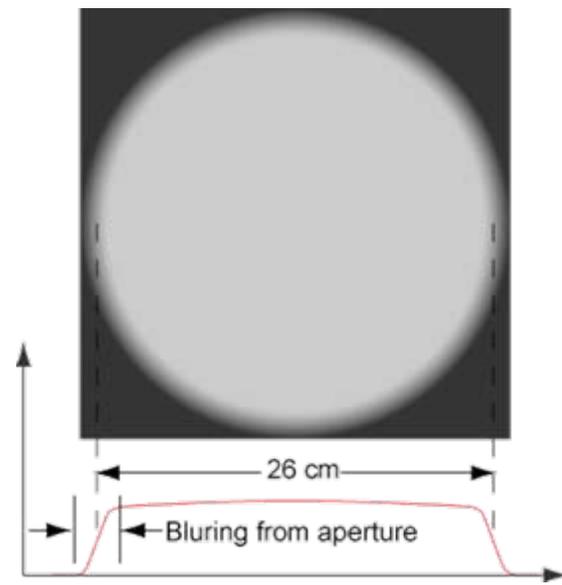
- 1) Neutrons from reactor
- 2) Tapered colimation
- 3) Bismuth filter
- 4) Apertures
- 5) Rotating Drum
- 6) Wax and shot filled shields
- 7) Evacuated flight tube
- 8) Sample position
- 9) Neutron camera
- 10) Beam stop
- 11) Small fuel cell test stand
- 12) Large fuel cell test stand
- 13) Hydrogen generator
- 14) Hydrogen vent
- 15) Oxygen bottle
- 16) Oxygen vent



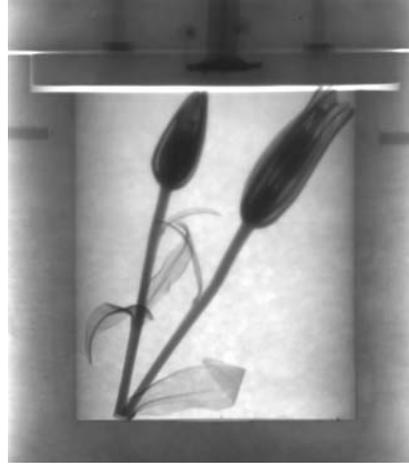
# Beam Properties

L (m)	D (cm)	L/d	Beam Dia. (cm)	Fluence Rate ( $\text{s}^{-1} \text{cm}^{-2}$ )	Fluence Rate No Bismuth ( $\text{s}^{-1} \text{cm}^{-2}$ )
2	2	100	8	$1.1 \times 10^8$	$3.0 \times 10^8$
3	2	150	13	$6.8 \times 10^7$	$2.0 \times 10^8$
4	2	200	17	$5.0 \times 10^7$	$1.5 \times 10^8$
6	2	300	26	$3.3 \times 10^7$	$1.0 \times 10^8$
6	1.5	400	26	$2.0 \times 10^7$	$5.9 \times 10^7$
6	1.0	600	26	$9.0 \times 10^6$	$2.5 \times 10^7$
6	0.5	1200	26	$2.0 \times 10^6$	$5.9 \times 10^6$
6	0.1	6000	26	$7.8 \times 10^4$	$2.5 \times 10^5$

At 6 m

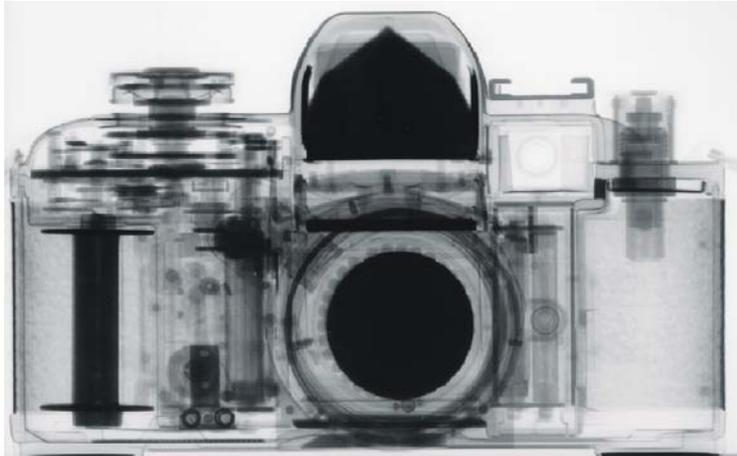


# Why Neutron Radiography?



- Neutrons see material differently than x-rays
- The fine details of the water in this Asiatic Lily are clear to neutrons even in a lead cask
- Neutron methods described as complimentary to x-ray methods see the images of the camera below.

**Neutron Image** of an SLR camera. The metal casing is nearly transparent, while the plastic film reel is easily seen. Also visible are the film advance gears and details of the shutter.

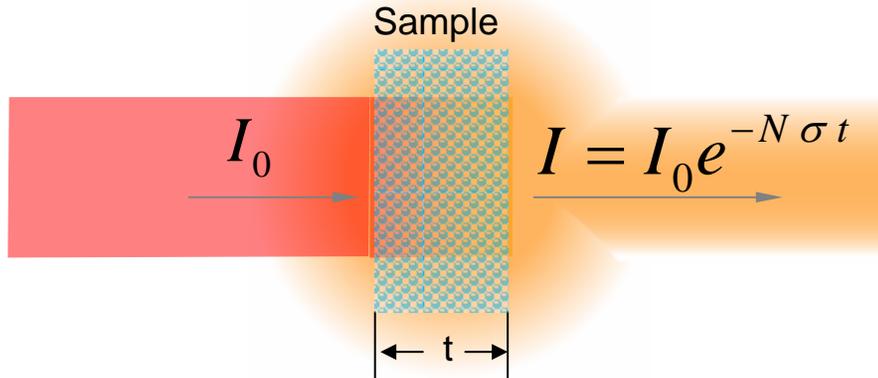


**X-Ray Image** of an SLR camera. The plastic film reel is invisible to the x-rays. The metal casing obscures the inner workings of the film advance mechanism and the shutter.



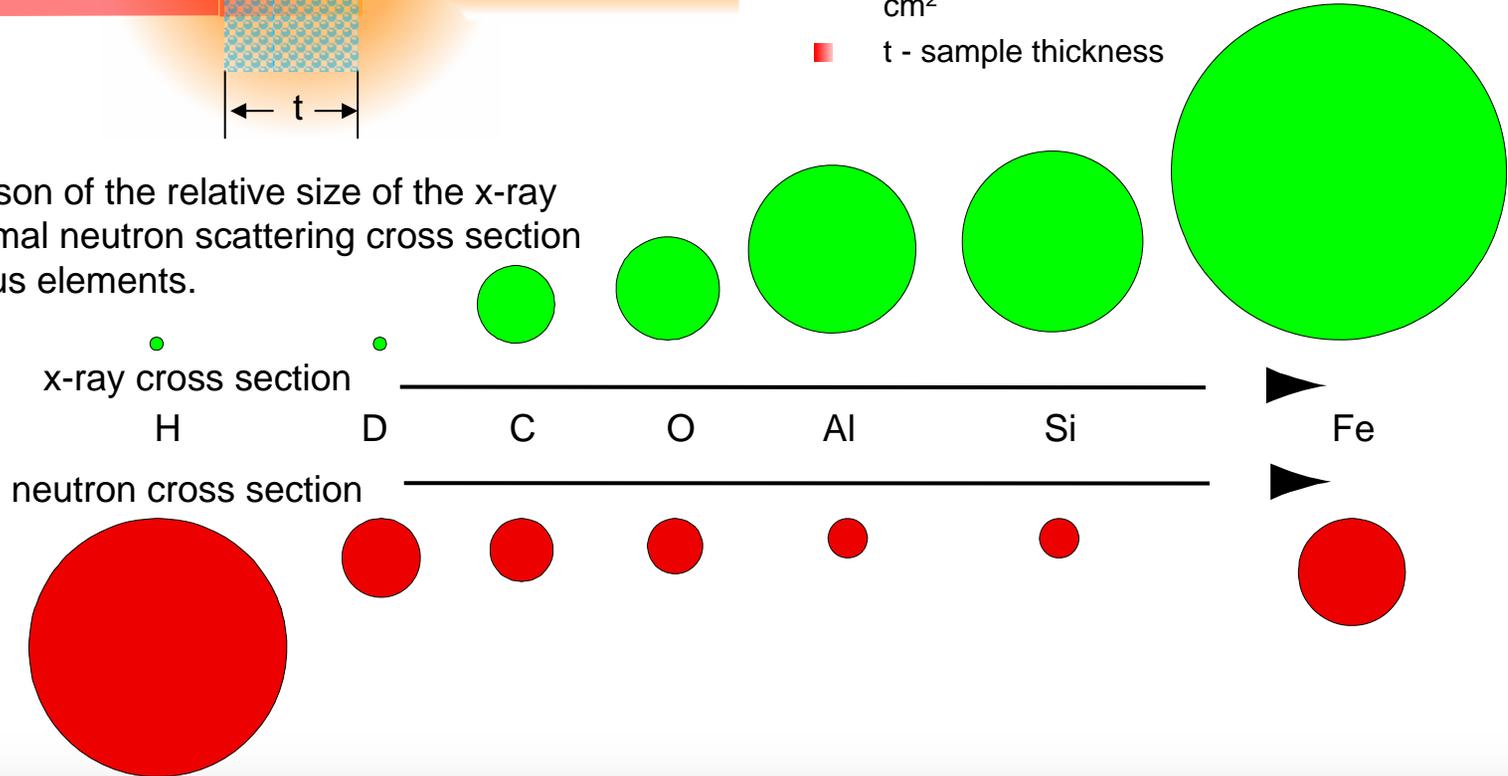
# Neutron cross section versus x-ray cross section

Neutrons are an excellent probe for hydrogen in metal since metals can have a much smaller cross section to thermal neutrons than hydrogen does.



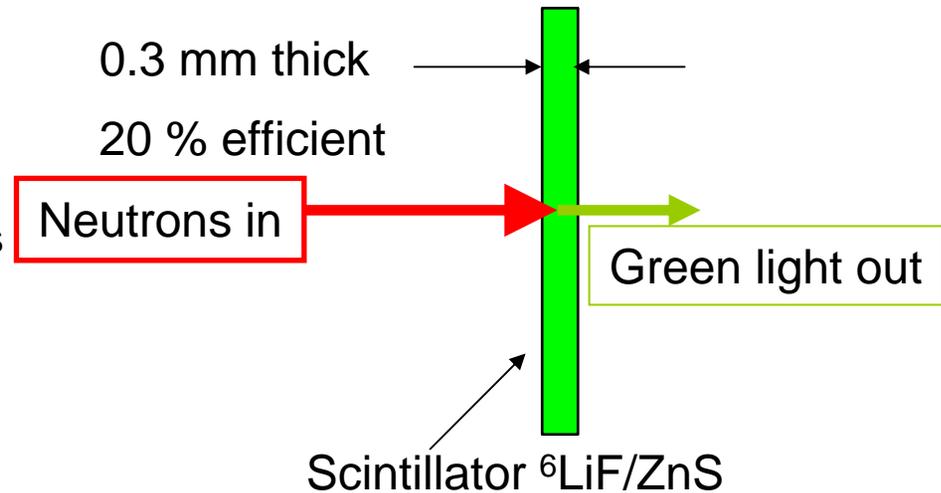
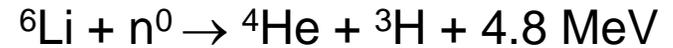
- $N$  – numerical density of sample atoms per  $\text{cm}^3$
- $I_0$  - incident neutrons per second per  $\text{cm}^2$
- $\sigma$  - neutron cross section in  $\sim 10^{-24} \text{cm}^2$
- $t$  - sample thickness

Comparison of the relative size of the x-ray and thermal neutron scattering cross section for various elements.



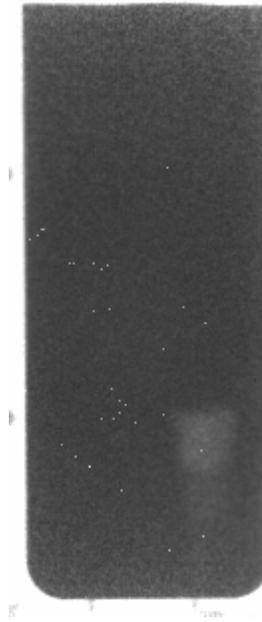
## Neutron Imaging (how we produce an image)

- We don't directly detect the neutron
- Special isotopes that are highly absorbing ( ${}^6\text{Li}$ ,  ${}^{10}\text{B}$ ) are used to capture neutrons.
  - ${}^6\text{Li} + n^0 \rightarrow {}^4\text{He} + {}^3\text{H} + 4.8 \text{ MeV}$
- Resulting nuclear reaction produces charged particles with Mega electron volt energies.
- Converter (traditionally ZnS) stops the particles (see top right photo).
- Energy lost by the particles generates a lot of light.
- The spatial resolution of the  ${}^6\text{LiF}/\text{ZnS}$  converter is limited not by the nuclear reaction (this has a 10 micron range), but by the spread of light in the converter. Typically this is about 200 to 300 micrometers or more due to the thick screens needed for high efficiency.



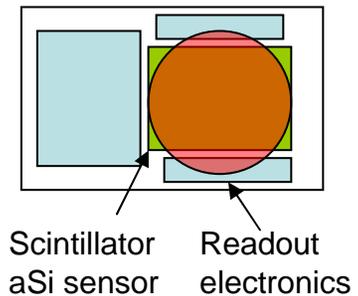
# Current Detector Technology

- Amorphous silicon
- Radiation hard
- High frame rate (30 fps)
- 127 micrometer spatial resolution
- Picture is of water with He bubbling through it
- No optics – scintillator directly couples to the sensor to optimize light input efficiency

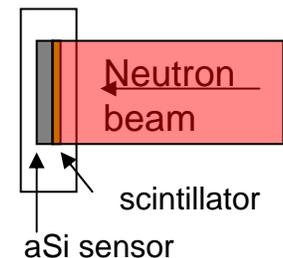


Helium through water at 30 fps

Front view



Side view



# High Resolution: What's the limit?

- ${}^6\text{LiF/ZnS:Cu,Al,Au}$  converter is limited in spatial resolution to about 100 microns.
- If the converter could make use of the 10 micrometer range of the charged particles, then 10 micrometer spatial resolution is possible.
- A current collaboration aims to achieve this using a radically new method to detect neutrons.
- Future speculative methods could achieve even sub-micrometer resolution by making use of the energy lost in the converter by the charged particles to further define the position of the neutron absorption event.

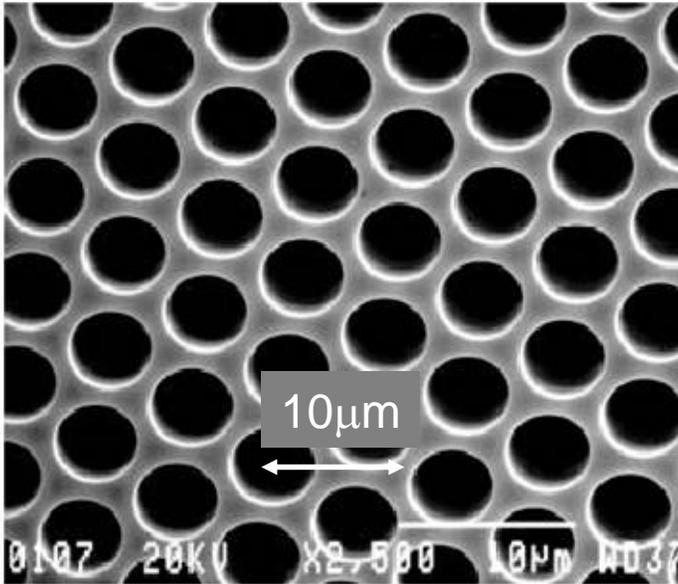
## Collaboration



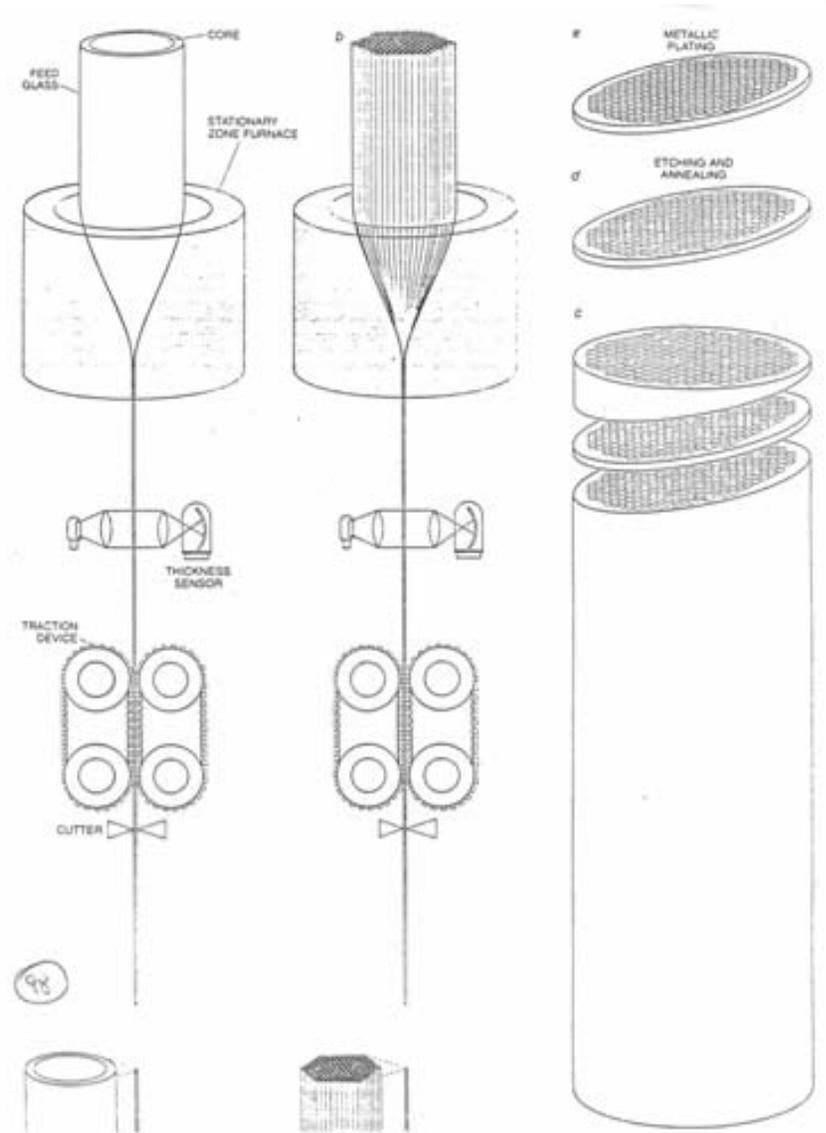
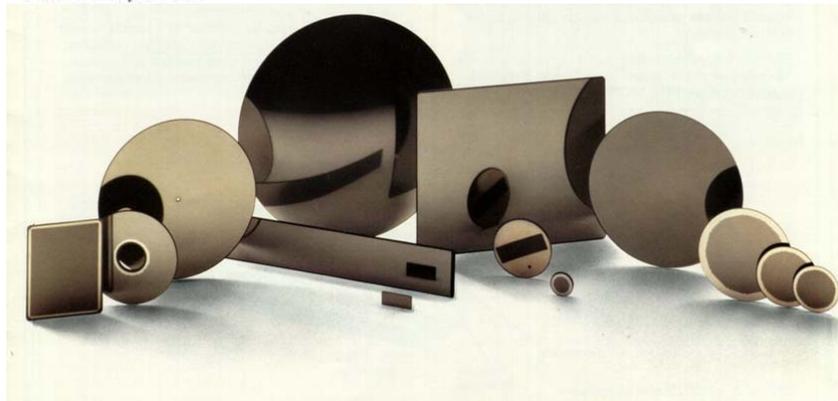
Space Sciences Laboratory, University of California, Berkeley,  
CA, 94720

Sensor Sciences LLC, 3333 Vincent Road, Suite 103, Pleasant Hill, CA 94523  
Nova Scientific Inc. 10 Picker Road, Sturbridge, MA 01566

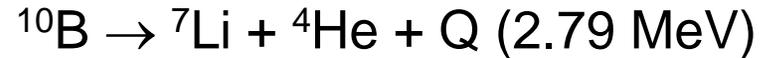
# Microchannel Plate



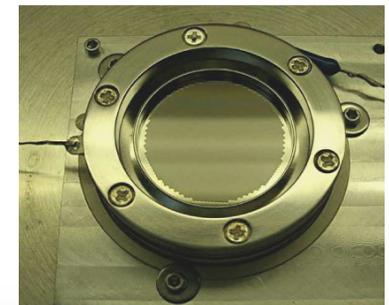
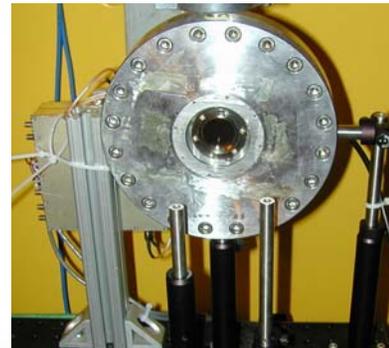
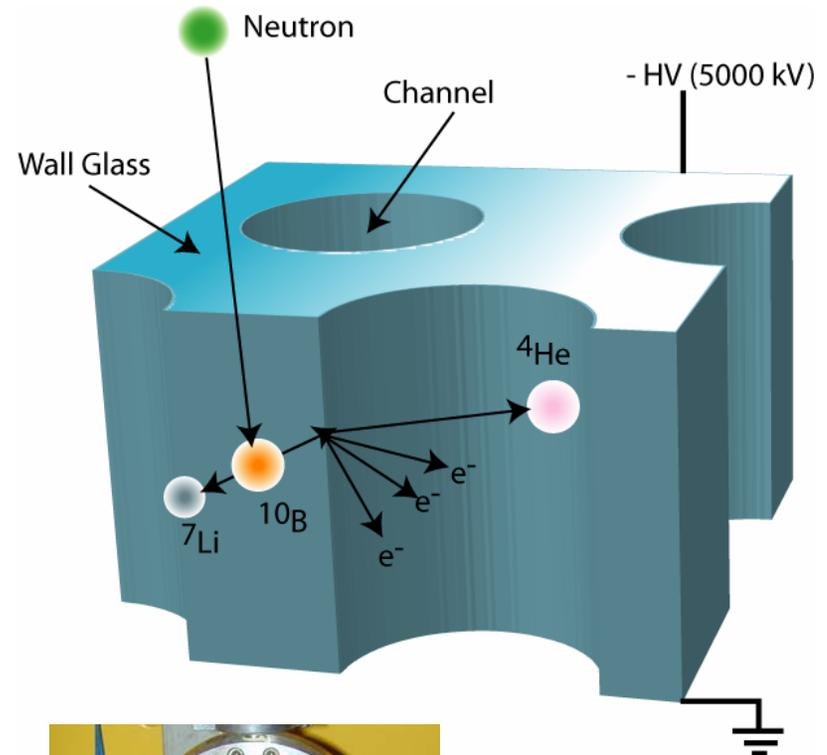
Microscopic view of a Glass Capillary Array with precision 5-micron pores.



# Neutron Imaging with MCPs



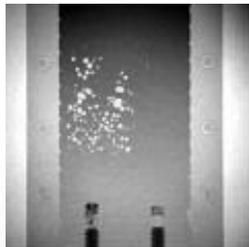
- $^{10}\text{B}$  or  $^{\text{nat}}\text{Gd}$  in wall absorbs neutron
- Reaction particles initiate electron avalanche down channel
- Charge cloud detected with position sensitive anode
- Spatial resolution limited by channel size and range of charged particle
- Current resolution is 0.025 mm
- Ultimate resolution about 0.01 mm
- Anode read-out is pulse counter
  - 1 % Deadtime at 200 kHz current event rate limit
  - FOV limited to 1 cm<sup>2</sup>
- New MCPS and readout expected Spring 08 with 0.01 mm resolution and higher count rate limit



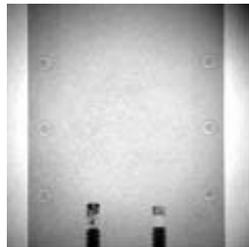
# Polymer Electrolyte Membrane Fuel Cells

# Water Sensitivity

Wet cuvet



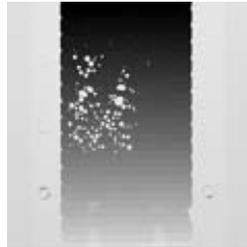
Dry cuvet



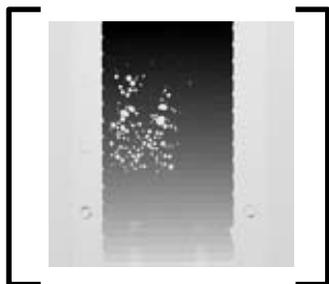
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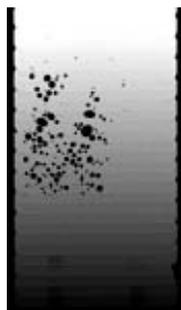
water only



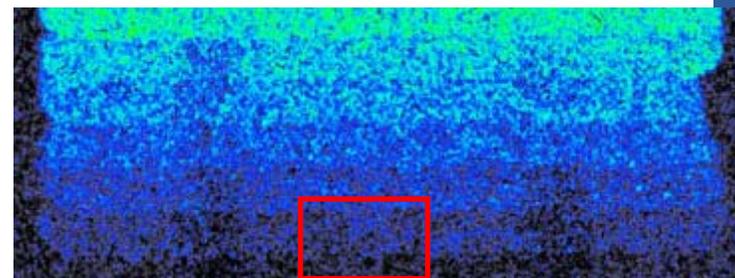
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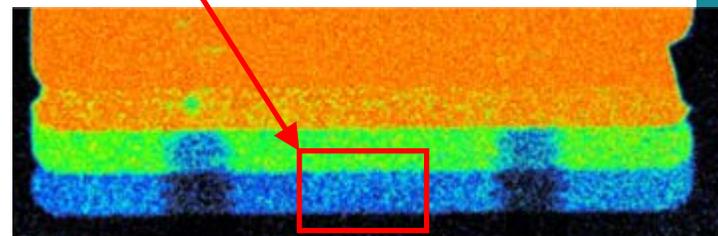


1 s exposure time



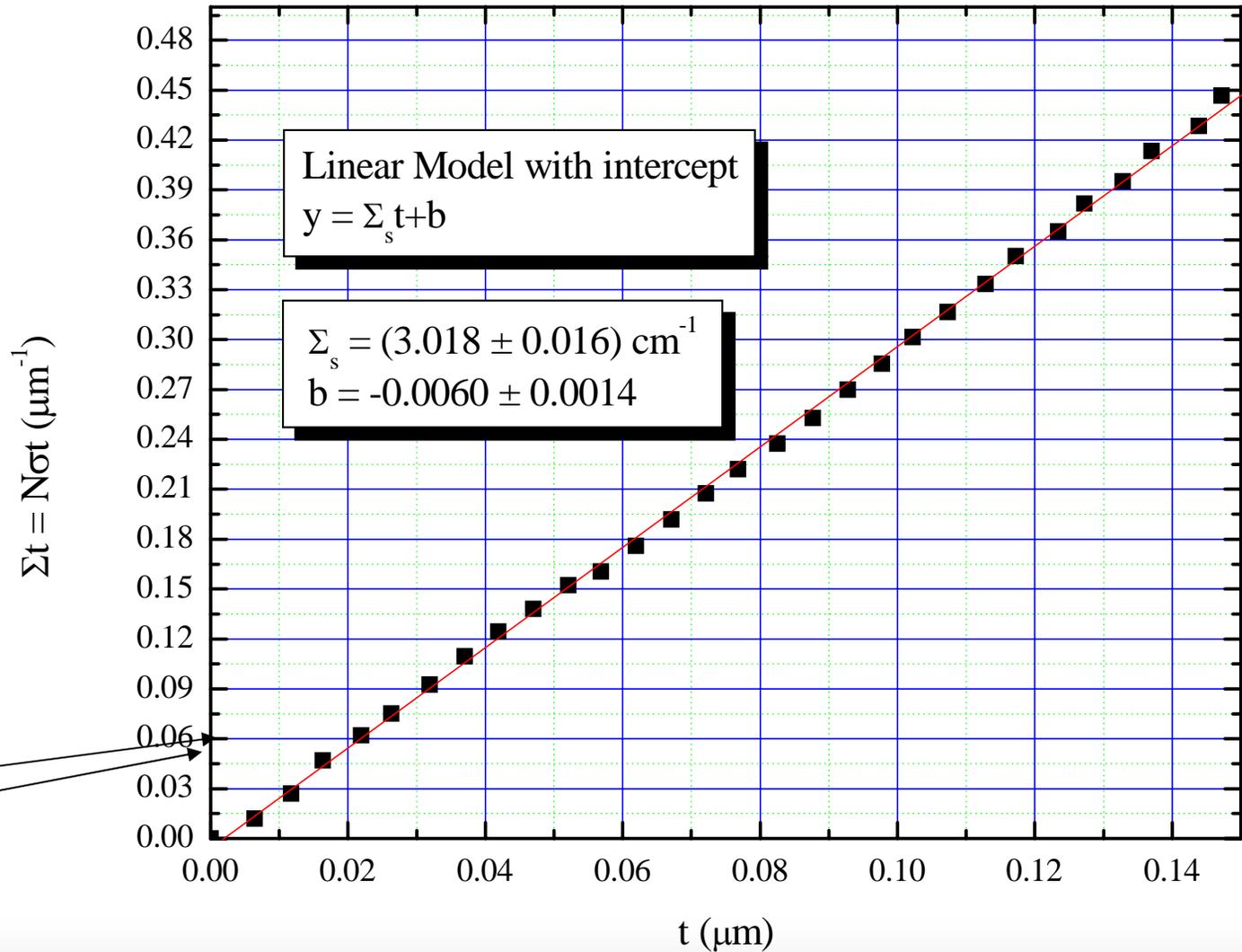
50 micron water thickness

- Steps machined with 50 micron.
- CCD camera exposure of 1 s yields a sensitivity of  $0.005 \text{ g cm}^{-2} \text{ s}^{-1}$
- After 100 s a factor of 10 improvement gives  $0.0005 \text{ g cm}^{-2} \text{ s}^{-1}$



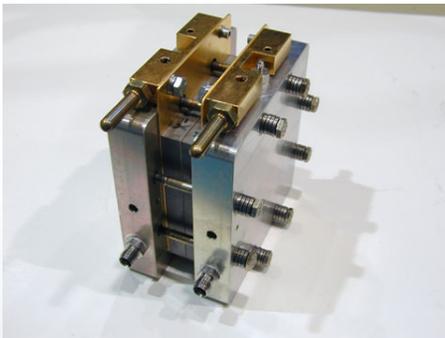
# Row average plotted for each pixel row

Measured Macroscopic Cross Section,  $\Sigma_s$ , for H<sub>2</sub>O

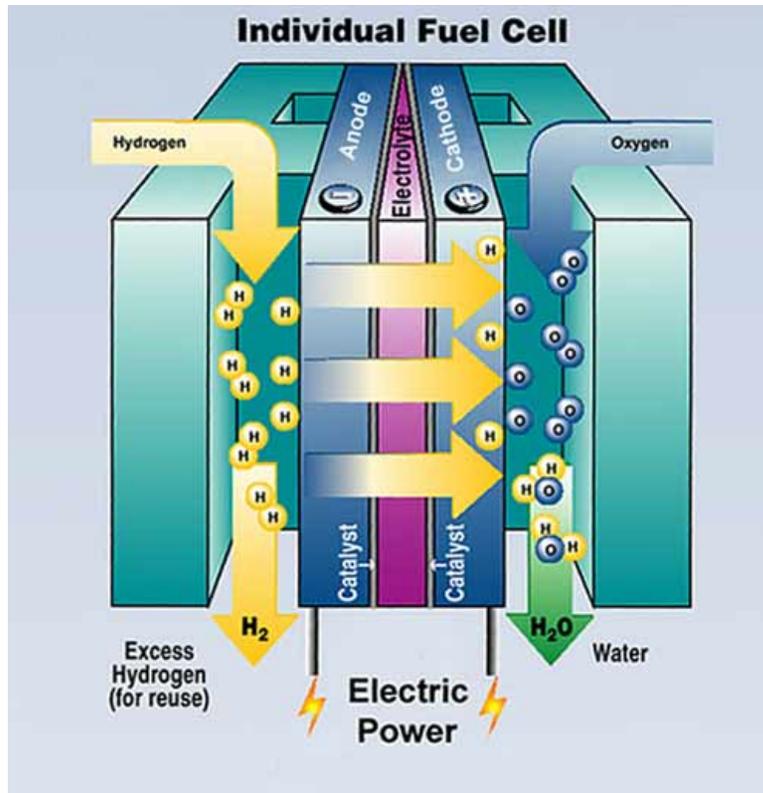
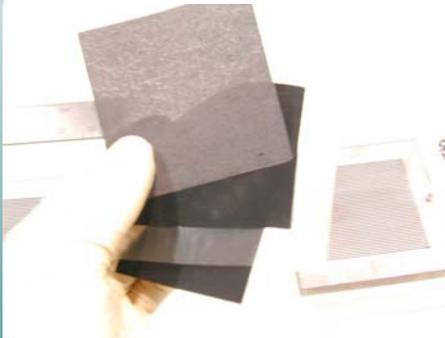


# Anatomy of a PEM Fuel Cell

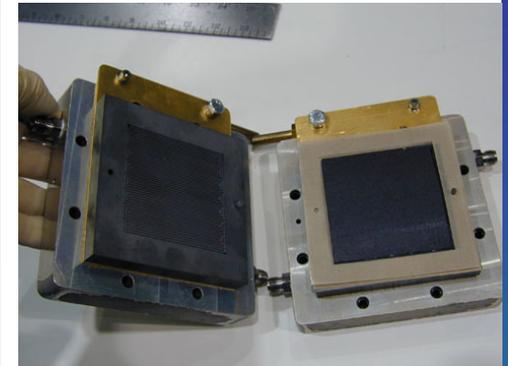
Assembled Cell



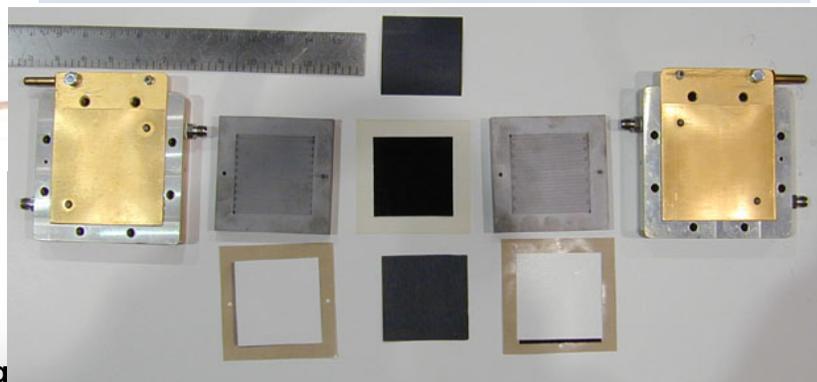
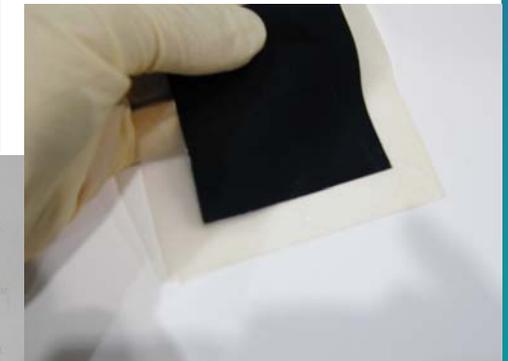
Porous gas diffusion layer  
GDL



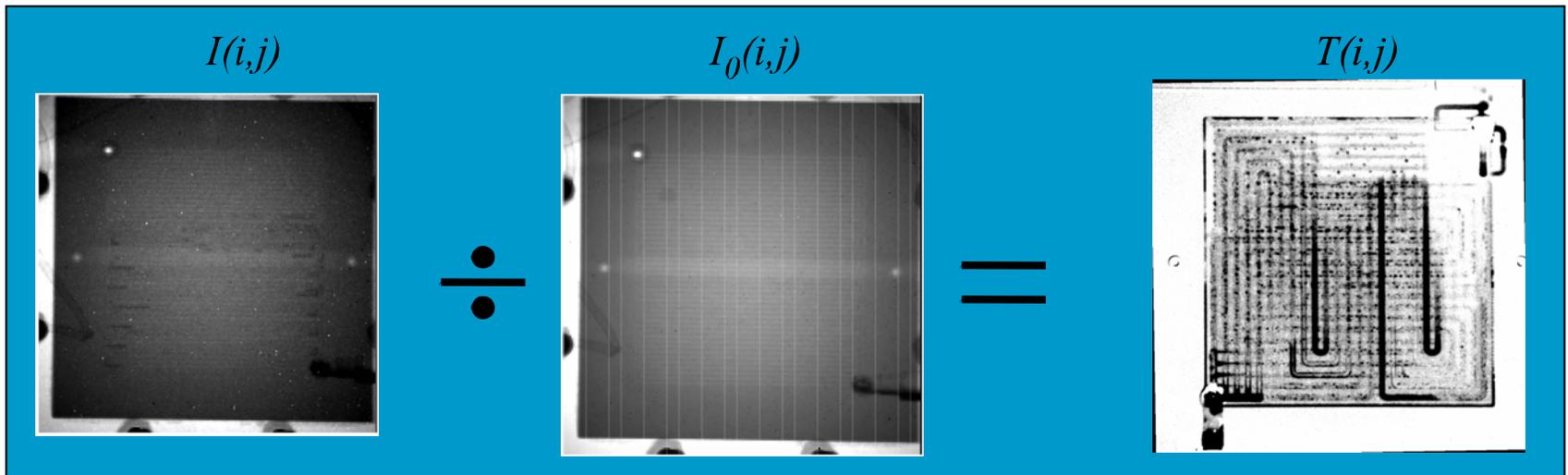
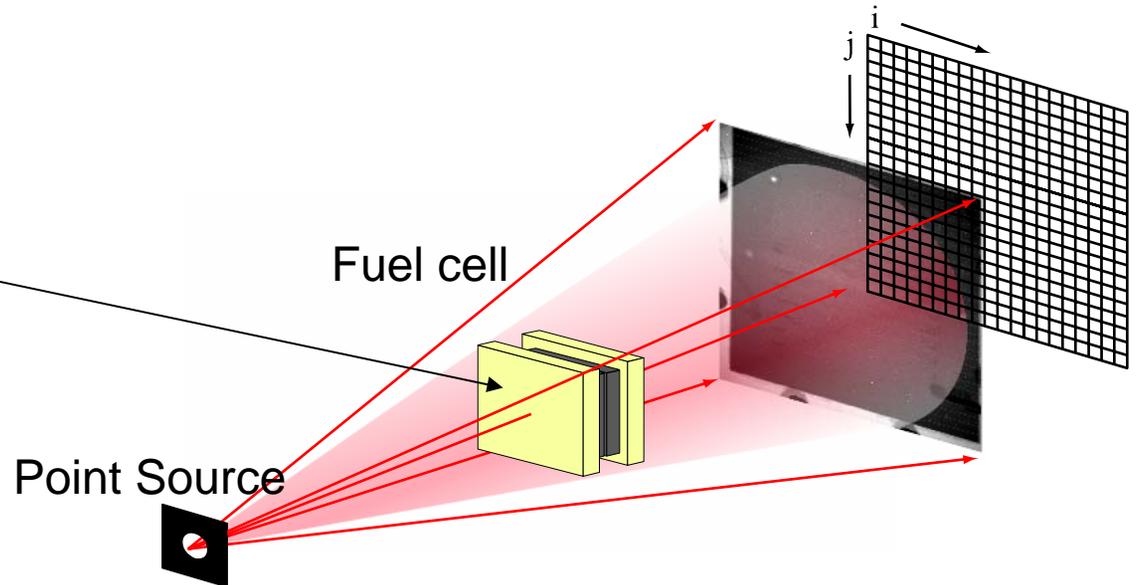
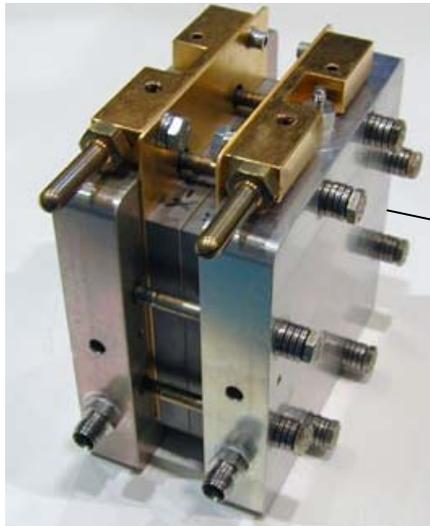
Flow field and soft goods



Membrane  
Electrode Assembly  
MEA

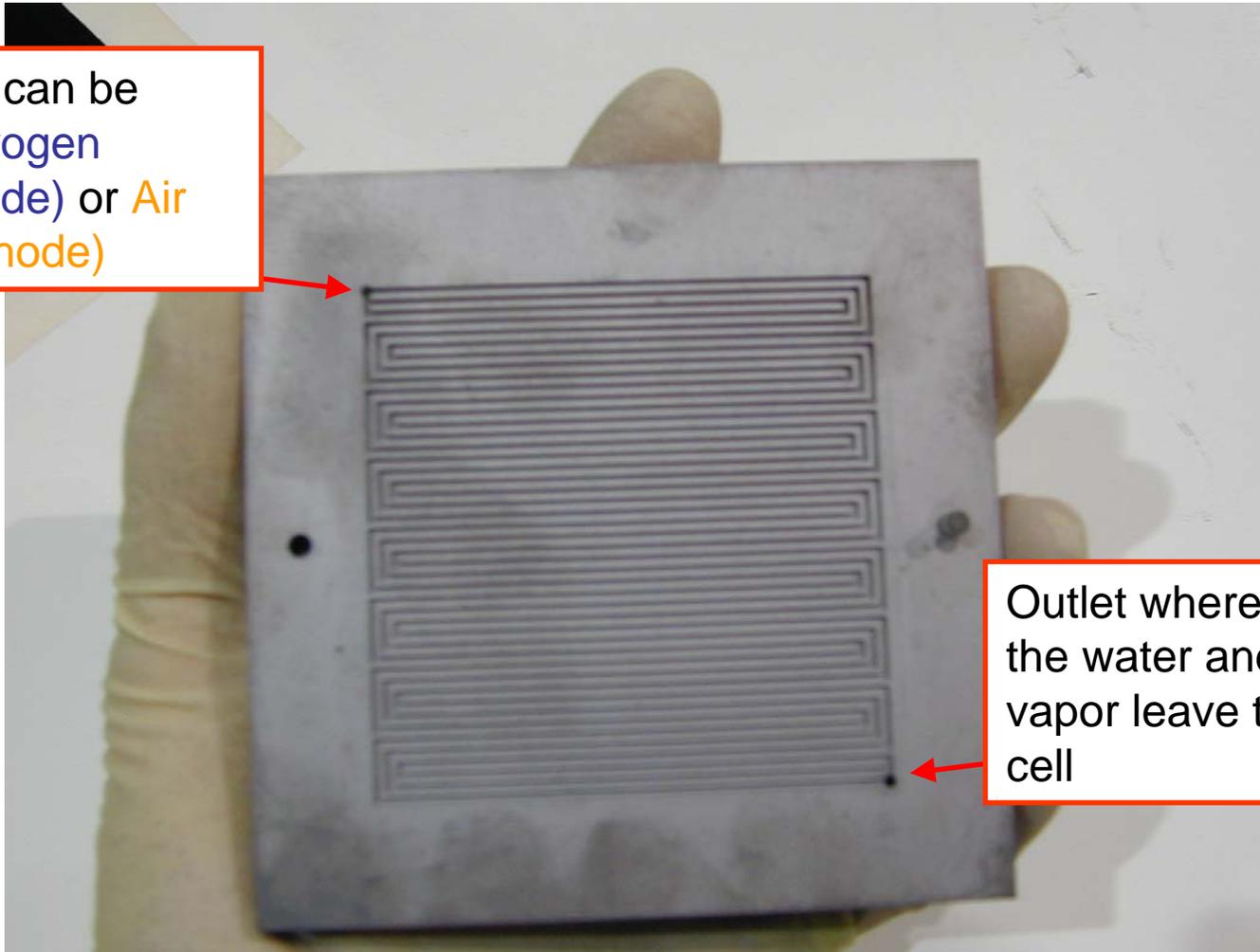


# Imaging Fuel Cells



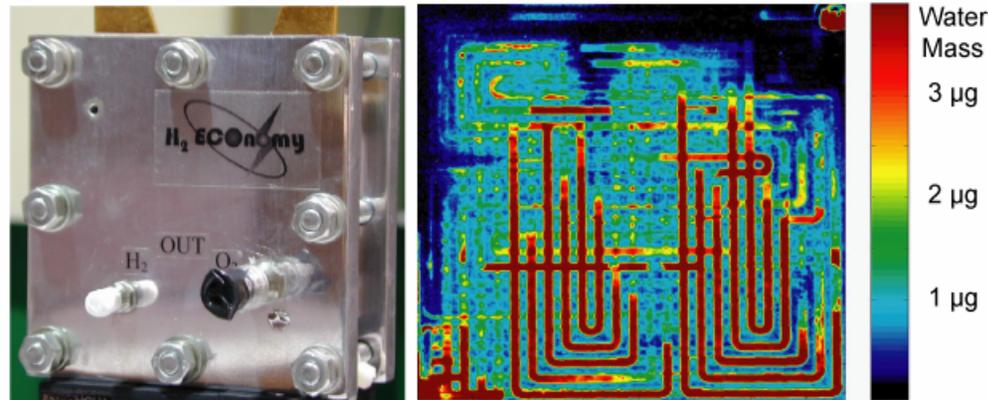
# The serpentine flow fields

Inlet can be  
Hydrogen  
(Anode) or Air  
(Cathode)



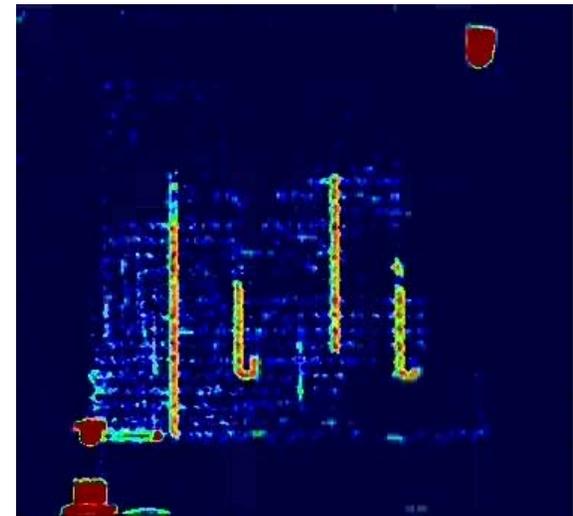
Outlet where the  
the water and  
vapor leave the  
cell

# Neutron Imaging for the Hydrogen Economy



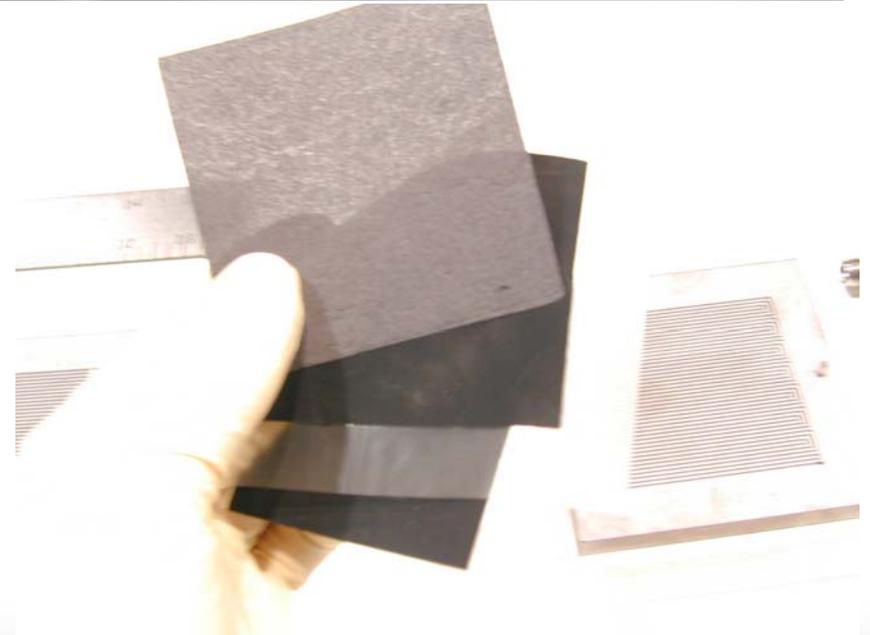
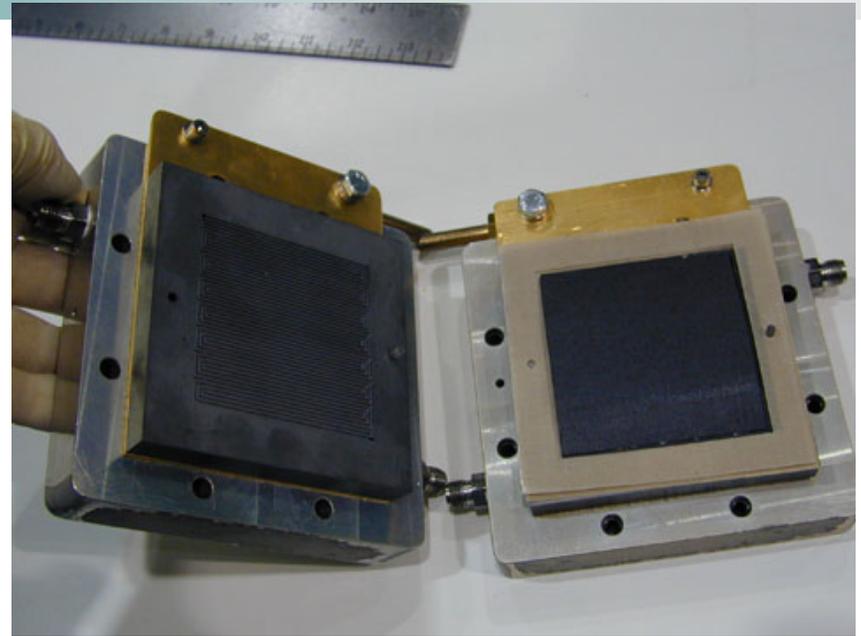
***Hydrogen Fuel Cells: A little water inside a metal matrix  
- an ideal problem for neutron imaging.***

- Neutrons see material differently than x-rays
- Subtle changes in the water distribution inside a running fuel cell impact performance and durability
- Neutron Imaging measures these small changes at video frame rate

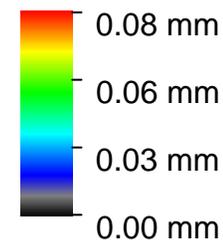


# Water Transport

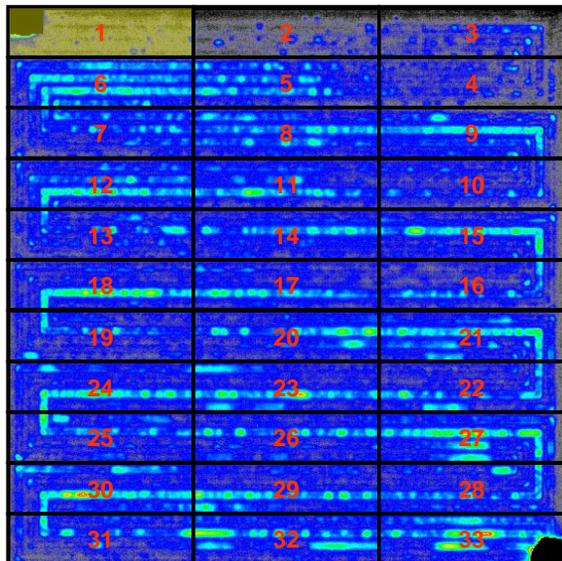
- Cells currently run at or less than 80 °C.
- Air streams are humidified
  - As fuel stream is used up the carrying capacity is reduced allowing condensation of water
- Evaporation
  - Heat generated locally by the chemical reaction
- Advection/Convection (also ejection)
  - Water is pushed out by the flow stream and/or capillary forces
- Capillary
  - Important but has a much lower impact on total water transport.



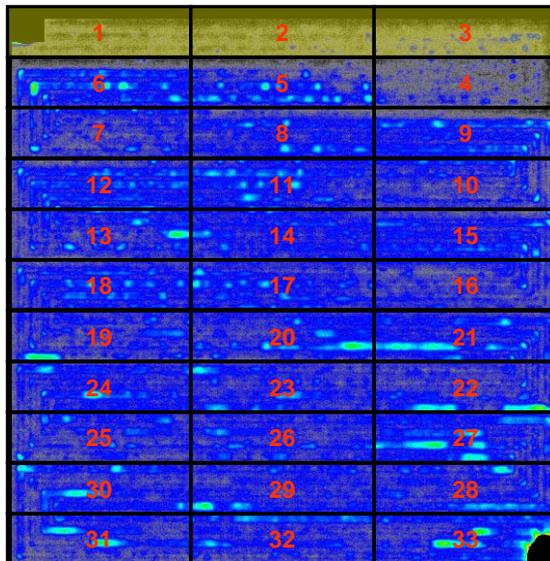
# Down-channel condensation – Bulk Cell Temperature of 60°C



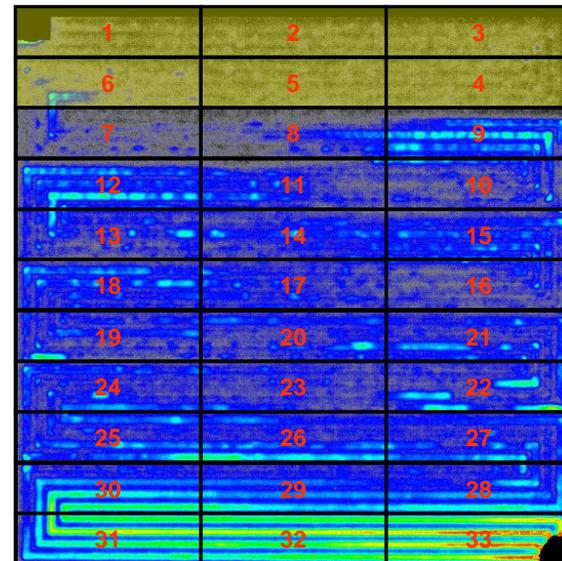
0.5 A/cm<sup>2</sup>  
cell 2 – predicted  
cell 2 – actual



1.0 A/cm<sup>2</sup>  
cell 4 – predicted  
cell 5 – actual

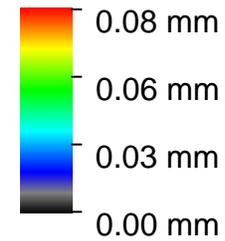


1.5 A/cm<sup>2</sup>  
cell 7 – predicted  
cell 8 – actual



In collaboration with M.A. Hickner, N.P. Siegel, K. S. Chen, D. N. McBrayer

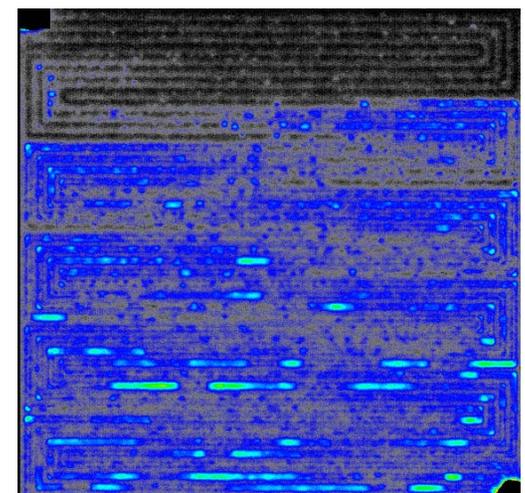
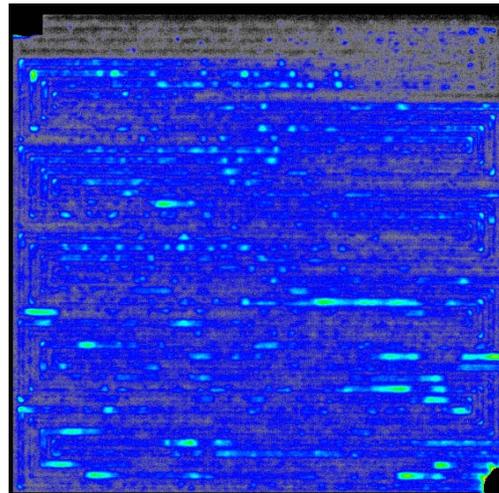
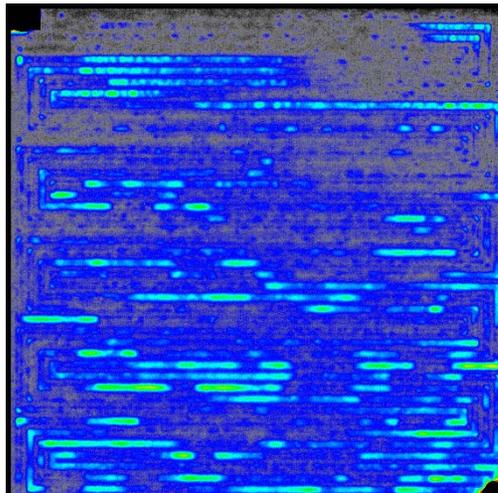
# Bulk Temperature Effect on Liquid Water Content



40°C

60°C

80°C



0.80 mL H<sub>2</sub>O

0.75 mL H<sub>2</sub>O

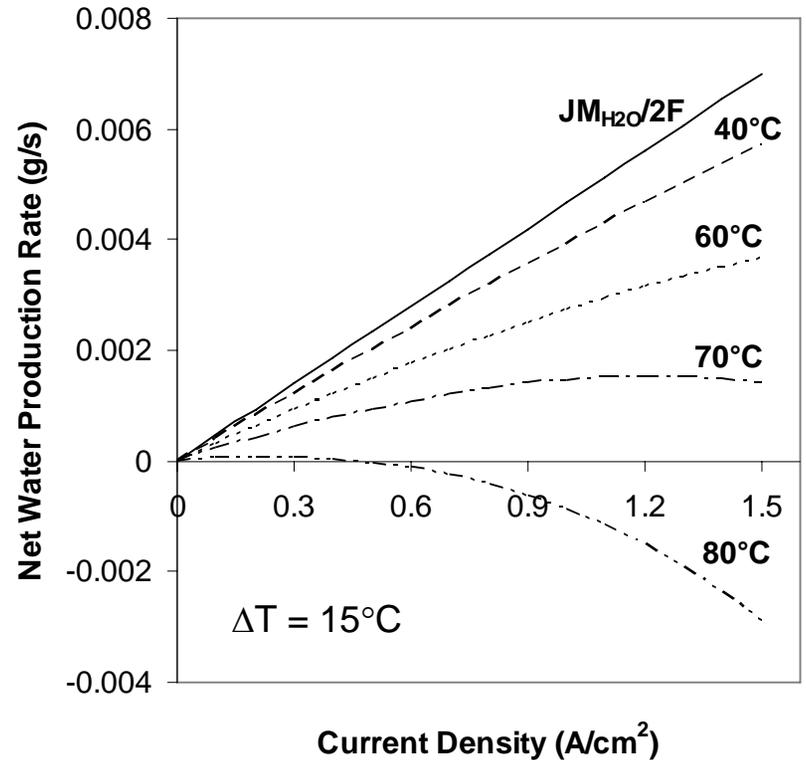
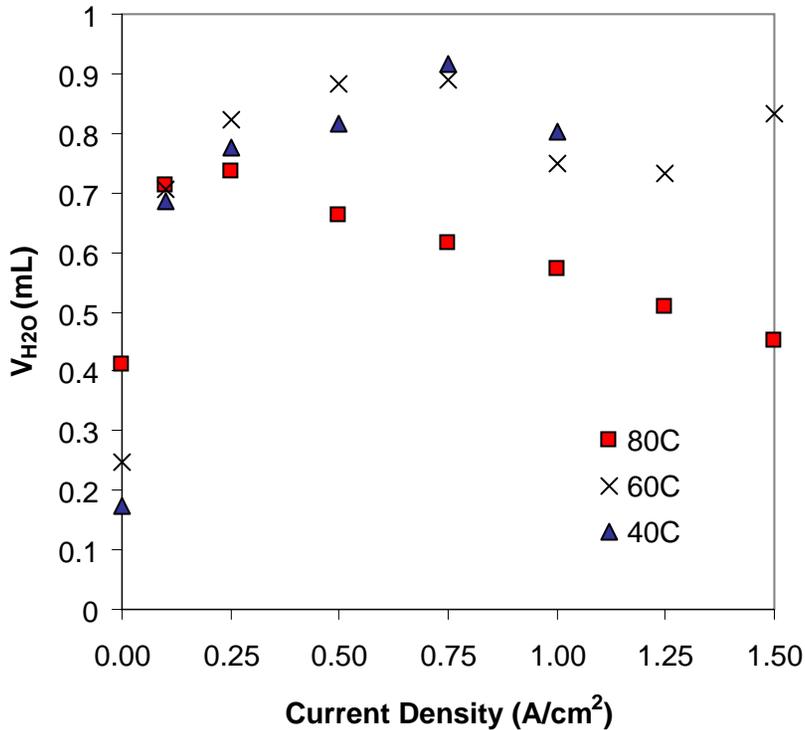
0.57 mL H<sub>2</sub>O



In collaboration with M.A. Hickner, N.P. Siegel, K. S. Chen, D. N. McBrayer

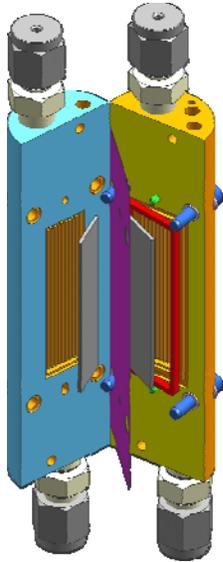
# Experimental Data and Current Model Data

Liquid water content

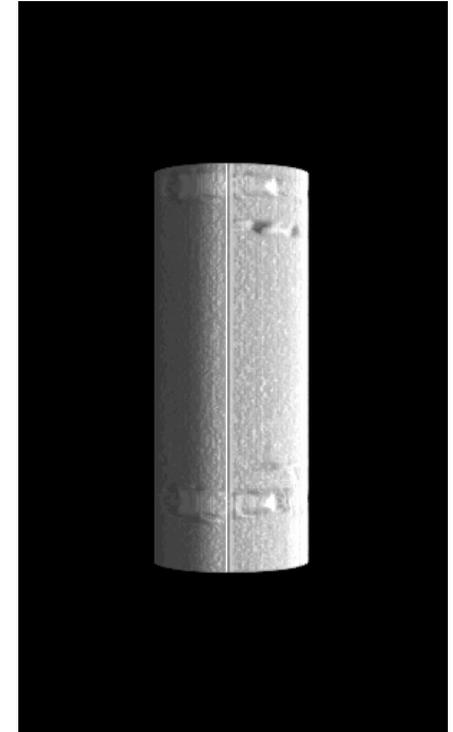


In collaboration with M.A. Hickner, N.P. Siegel, K. S. Chen, D. N. McBrayer

# Tomography Fuel Cell **Anode** v.s. **Cathode**

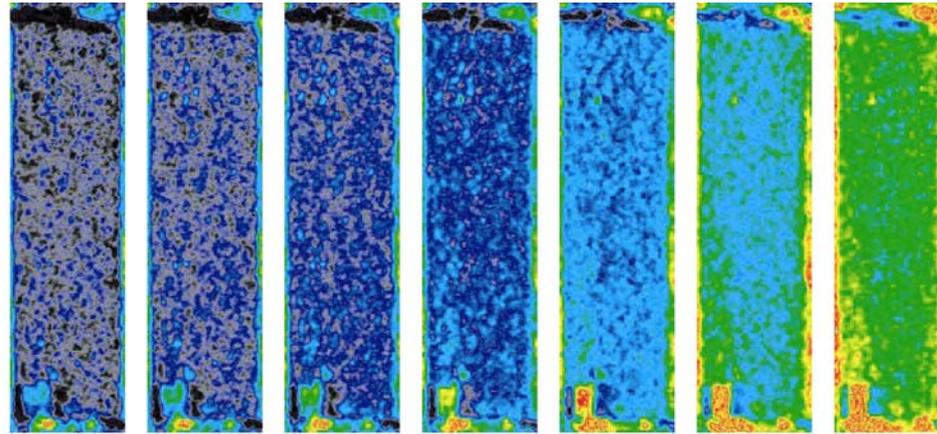


- Small Active area cell, 4 cm<sup>2</sup>
- Thick soft goods, 0.15 mm Membrane, 0.8 mm GDL
- 10 straight channels, 0.8 mm deep
- Blue is Anode, Orange is Cathode, inserts are on the cathode



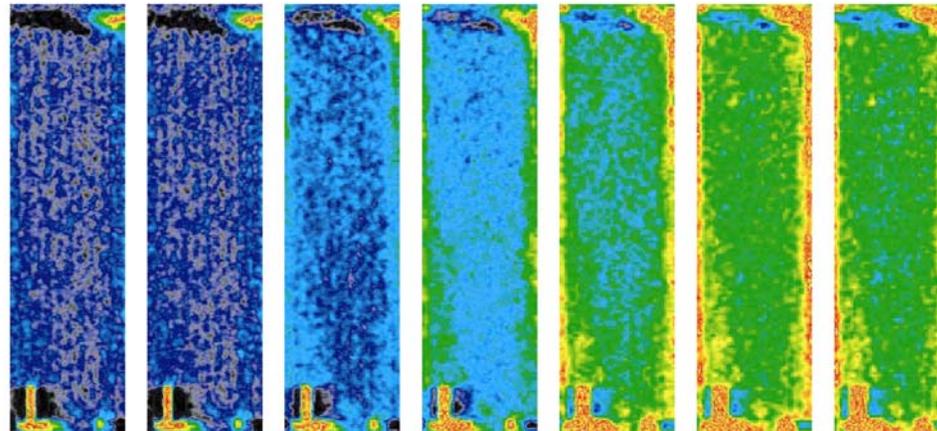
# Water content in the soft goods

Cathode



750  $\mu\text{m}$  625  $\mu\text{m}$  500  $\mu\text{m}$  375  $\mu\text{m}$  250  $\mu\text{m}$  125  $\mu\text{m}$  MEA

Anode



750  $\mu\text{m}$  625  $\mu\text{m}$  500  $\mu\text{m}$  375  $\mu\text{m}$  250  $\mu\text{m}$  125  $\mu\text{m}$  MEA

# Conclusions

- The NIST Neutron Imaging Facility is a productive addition to the the national user facilities at the Center for Neutron Research.
- In collaboration with colleagues we have and are pioneering new methods to reduce the spatial resolution available to neutron imaging by an order of magnitude.
  - For a little perspective - ZnS was invented around 1900 by Rutherford's laboratory to image the backscattering of alpha particles off the nucleus of gold.
  - Potentially we will reduce the resolution by as much as 2 orders of magnitude.
- Neutrons are the perfect probe to study hydrogen fuel cells.
  - Easily penetrates the metal exterior.
  - Sensitive to the water produced inside the fuel cell at the length scales needed to effectively understand the transport mechanisms in the fuel cell.

## Users/Collaborators 2007

- General Motors
- DaimlerChrysler
- Plug Power
- Sandia National Laboratory
- Los Alamos National Laboratory
- University of Kansas
- Rensselaer Polytechnic Institute (RPI)
- University of Michigan
- University of Waterloo
- University of Delaware
- Rochester Institute of Technology
- Wayne State University
- University of Connecticut
- Illinois Institute of Technology
- University of Tennessee
- Case Western University
- University of Mississippi
- University of California, Berkeley