Argonne NATIONAL LABORATORY

... for a brighter future

Ever Improving Sample Environment at the Intense Pulsed Neutron Source (IPNS)

Ken Volin

Intense Pulsed Neutron Source Division
Argonne National Laboratory
Argonne, IL

4th International Workshop on Sample Environment at Neutron Scattering Facilities

Argonne, IL USA
September 6-8, 2006

U.S. Department of Energy

THE UNIVERSITY OF CHICAGO

Office of Science
U.S. DEPARTMENT OF ENERGY

A U.S. Department of Energy laboratory managed by The University of Chicago

Many Crosscutting Science Research Areas Emerged from the Workshops

- New materials discovery, design, development, and fabrication, especially materials that perform well <u>under extreme conditions</u>
- Science at the nanoscale, especially low-dimensional systems that promise materials with new and novel properties
- Methods to "control" photon, electron, ion, and phonon transport in materials for next-generation energy technologies
- Structure-function relationships in both living and non-living systems
- Designer catalysts
- Interfacial science and designer membranes in both chemistry and materials sciences
- Bio-materials and bio-chemical interfaces, especially at the nanoscale where soft matter and hard matter can be joined
- New tools for:
  - Spatial characterization, especially at the atomic and nanoscales and <u>especially for in-situ studies</u>
  - Temporal characterization for studying the time evolution of processes
  - Theory and computation

HRMECS Harwood Pressure Generator

- Maximum Pressure Capacity
  200,000 psig (13.8 kbar or 1.4 GPa) helium
- Hydraulic pump restricted to limit maximum system pressure to
  4.5 kbar (0.45 GPa) helium. This corresponds to the Maximum Operating Pressure (MOP) of HRMECS inelastic scattering cell.

Harwood Engineering, Walpole, MA

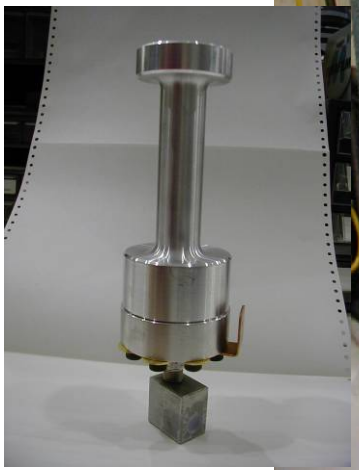
HRMECS Harwood Pressure Generator

- Maximum Pressure Capacity
  200,000 psig (13.8 kbar or 1.4 GPa) helium
- Hydraulic pump restricted to limit maximum system pressure to
  4.5 kbar (0.45 GPa) helium. This corresponds to the Maximum Operating Pressure (MOP) of HRMECS inelastic scattering cell.

Harwood Engineering, Walpole, MA

<u>Inelastic Scattering Cell</u>

- **Aluminum Body**
- **MOP = 70 kpsi**
- **Temperature Range**
  40 K < T < RT
- **360º Scattering**
- **Bridgman unsupported area seal**

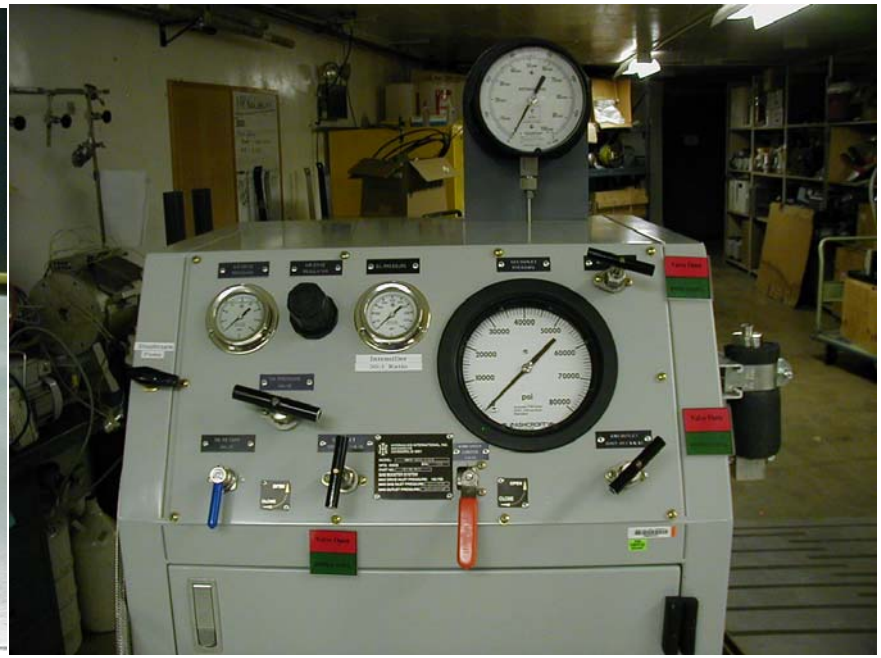


Hydraulics International Pressure Generator

- Gas Intensifier connected to a single diaphragm compressor.
- Maximum Pressure Generated = 60,000 psig (4 kbar or 0.4 GPa) helium

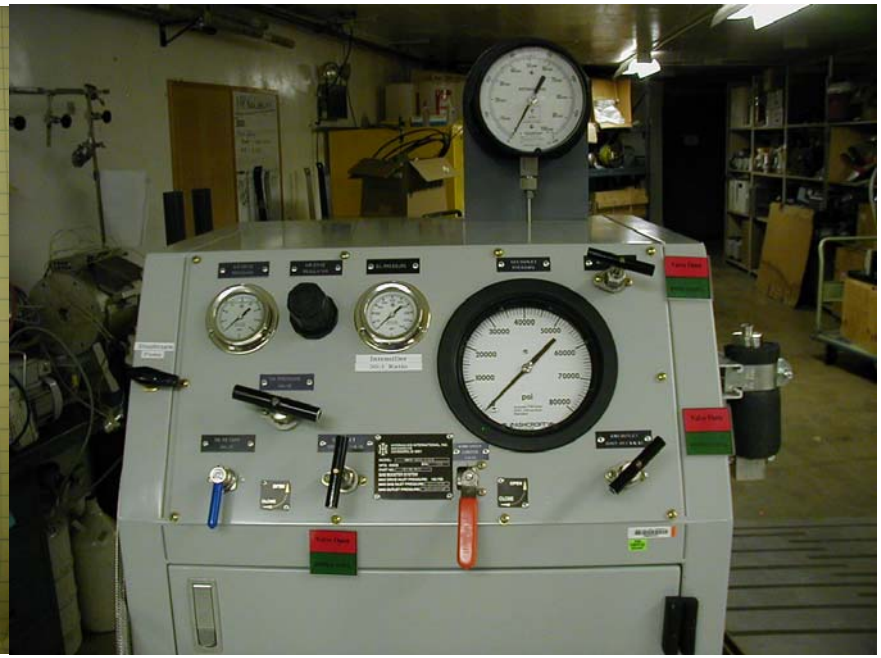
Hydraulics International Pressure Generator

- Gas Intensifier connected to a diaphragm compressor.
- Maximum Pressure Generated = 60,000 psig (4 kbar or 0.4 GPa) helium which is double the pressure generated by previous double diaphragm compressor without intensifier.



Hydraulics International Pressure Generator

- Gas Intensifier connected to a diaphragm compressor.
- Maximum Pressure Generated = 60,000 psig (4 kbar or 0.4 GPa) helium which is double the pressure generated by previous double diaphragm compressor without intensifier.
- Current aluminum cell designs are being upgraded to the new pressure regime.



SEPD 7T Magnet

- Maximum Field = 7T
- Sample Temperature Range
  1.3 K < T < RT
- 40 mm Diameter Sample Volume
- 1,1 cm diameter sample ~ 5 cc

SEPD "MAGNUS" 7T Magnet

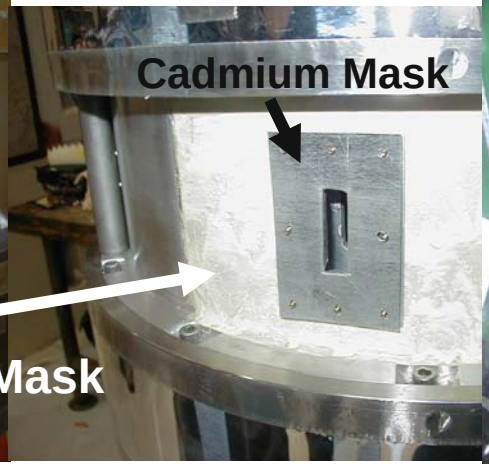
SEPD 7T Magnet

- Maximum Field = 7T
- Sample Temperature Range
  1.3 K < T < RT
- 40 mm Diameter Sample Volume
- 1,1 cm diameter sample ~ 5 cc
- Gd2O3 Masking with additional Cd Mask on incident and exit windows

SEPD 7T Magnet

- Maximum Field = 7T
- Sample Temperature Range
  1.3 K < T < RT
- 40 mm Diameter Sample Volume
- 1,1 cm diameter sample ~ 5 cc
- Gd2O3 Masking with additional Cd Mask on incident and exit windows

Next Year?
Radial Collimators
for the SEPD Instrument



LRMECS Lucas Furnace

DRYLID Furnace

- Tmax = 350ºC
- Aluminum pie pans
- Aluminum heat shields

LRMECS Lucas Furnace

DRYLID Furnace

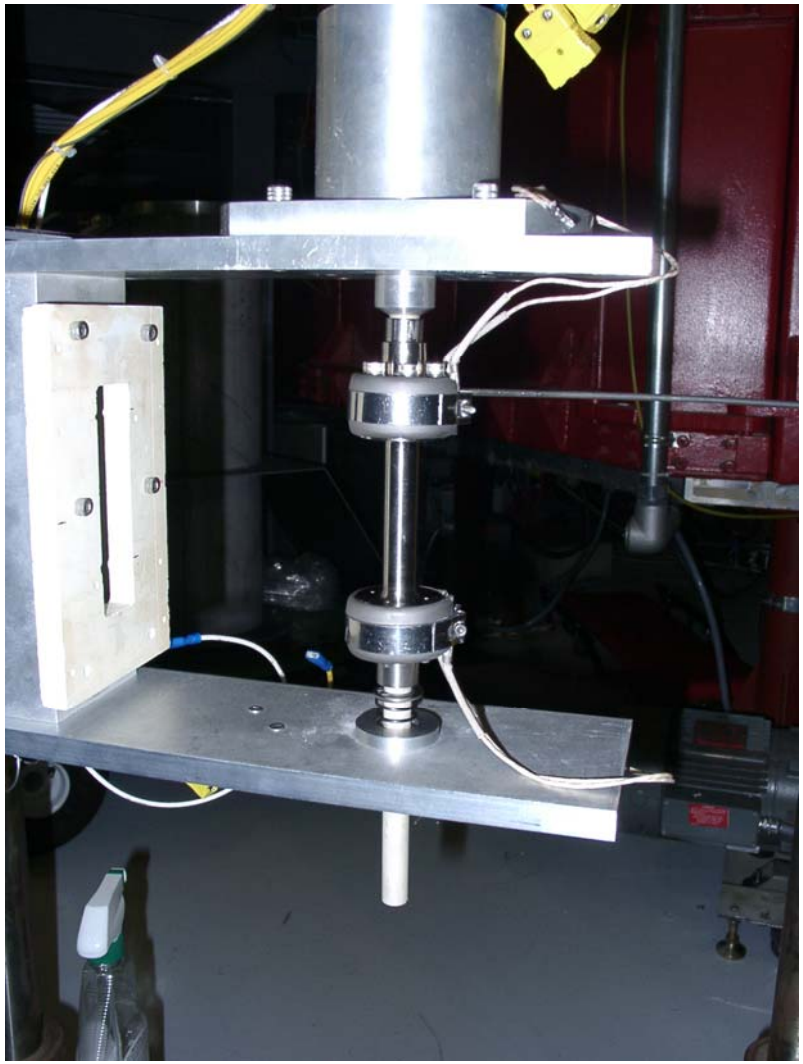
- Tmax = 350ºC
- Aluminum pie pans
- Aluminum heat shields

Lucas Furnace (CalTech)

- Tmax = 500ºC
- Niobium pie pans
- Aluminum heat shields

GPPD Ashfia Cell

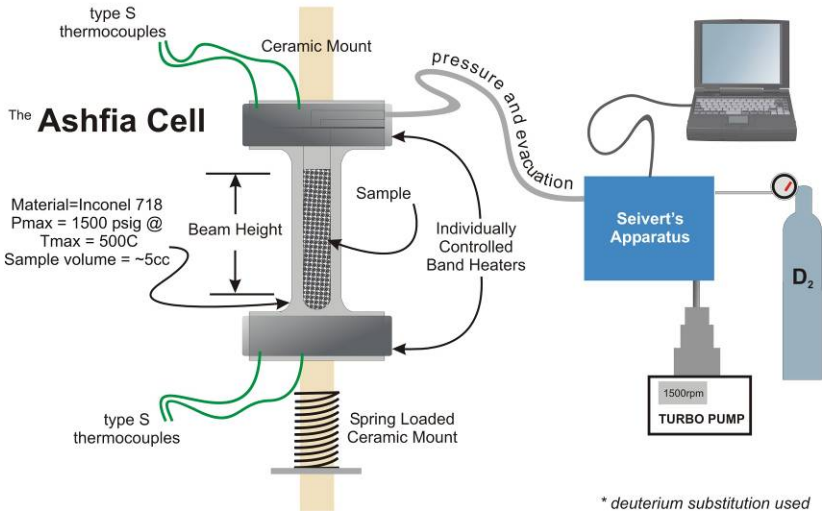
- Maximum Temperature = 500ºC Maximum Pressure = 1500 psig
- 1,1 cm diameter sample ~ 5 cc
- Dual controllers to individually control temperature top and bottom



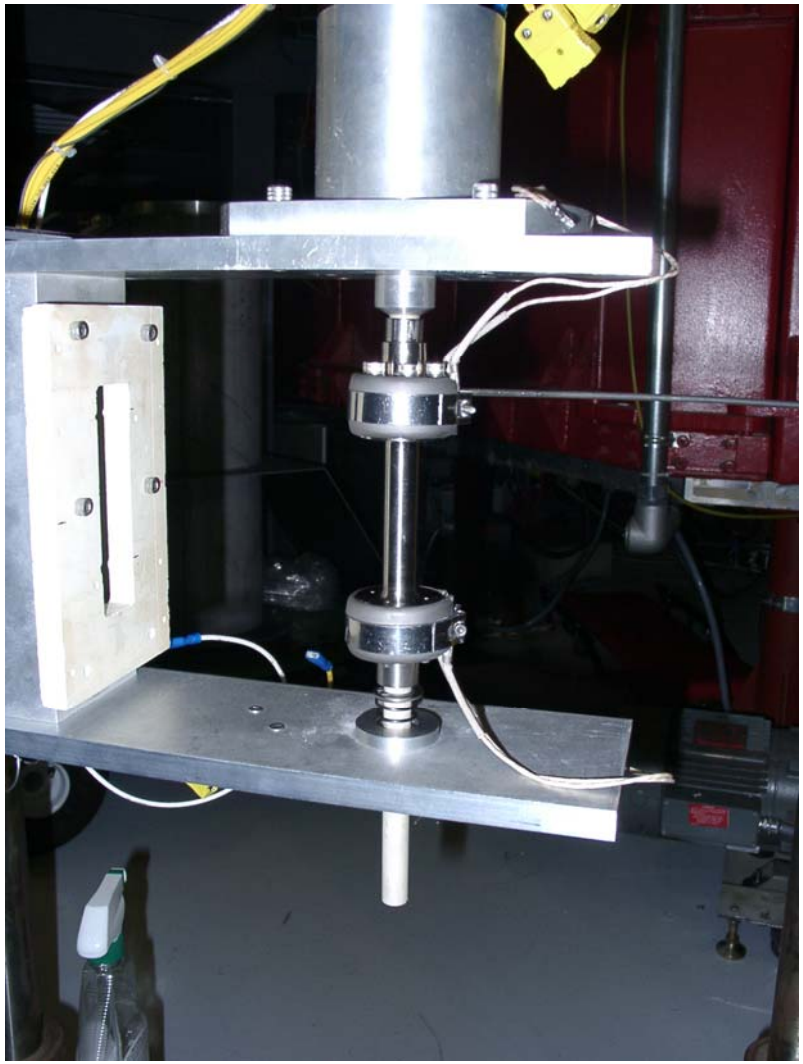
GPPD Ashfia Cell

- Maximum Temperature = 500ºC Maximum Pressure = 1500 psig
- 1,1 cm diameter sample ~ 5 cc
- Dual controllers to individually control temperature top and bottom
- Sievert Appatatus to control and measure hydrogen adsorption

In-Situ Hydriding and De-Hydriding* Experiment*

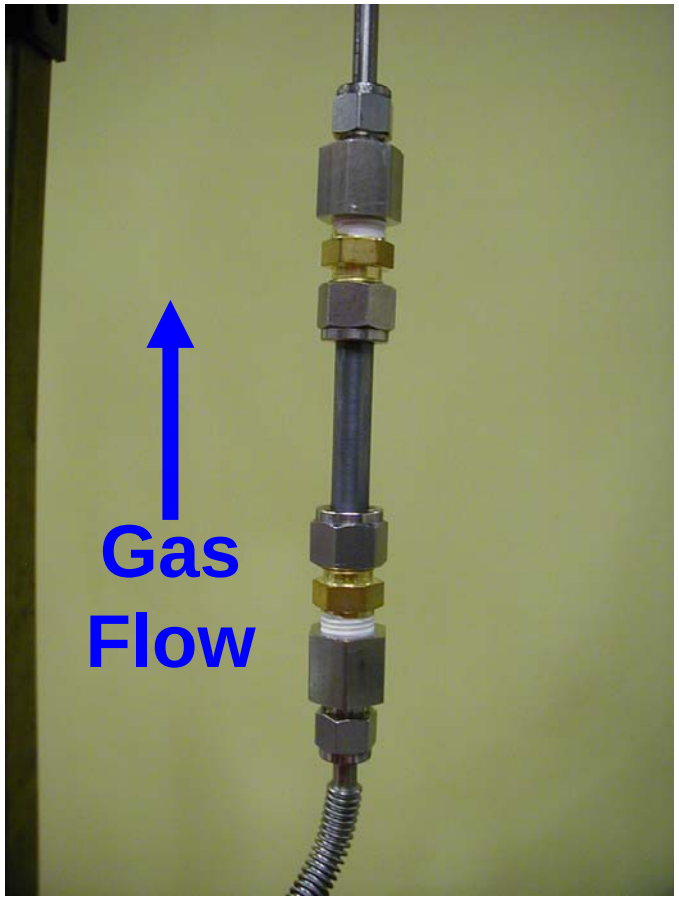


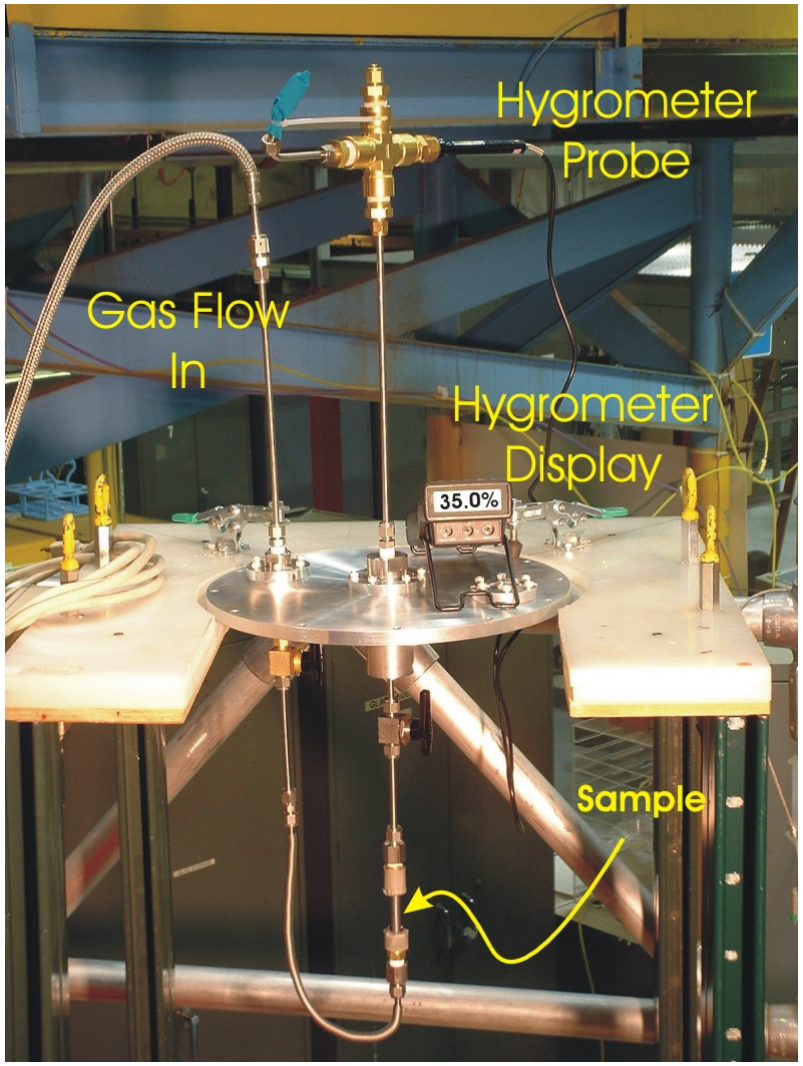
** deuterium substitution used*



GPPD Hygrometer Cell

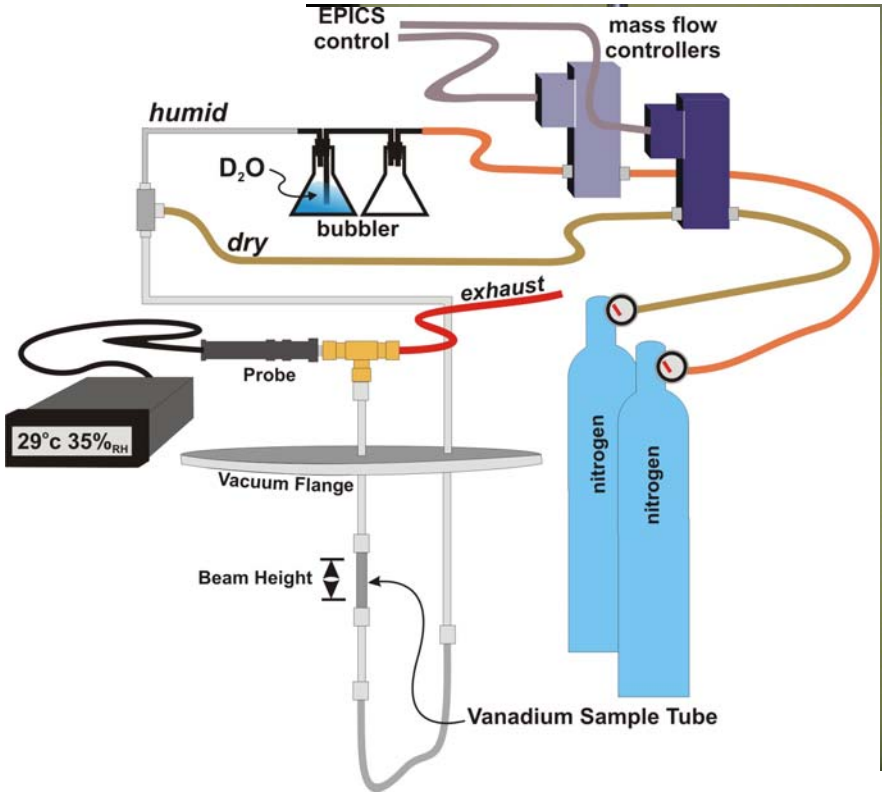
- Vanadium tube sample volume
- Inline Hygrometer: 2-99% RH

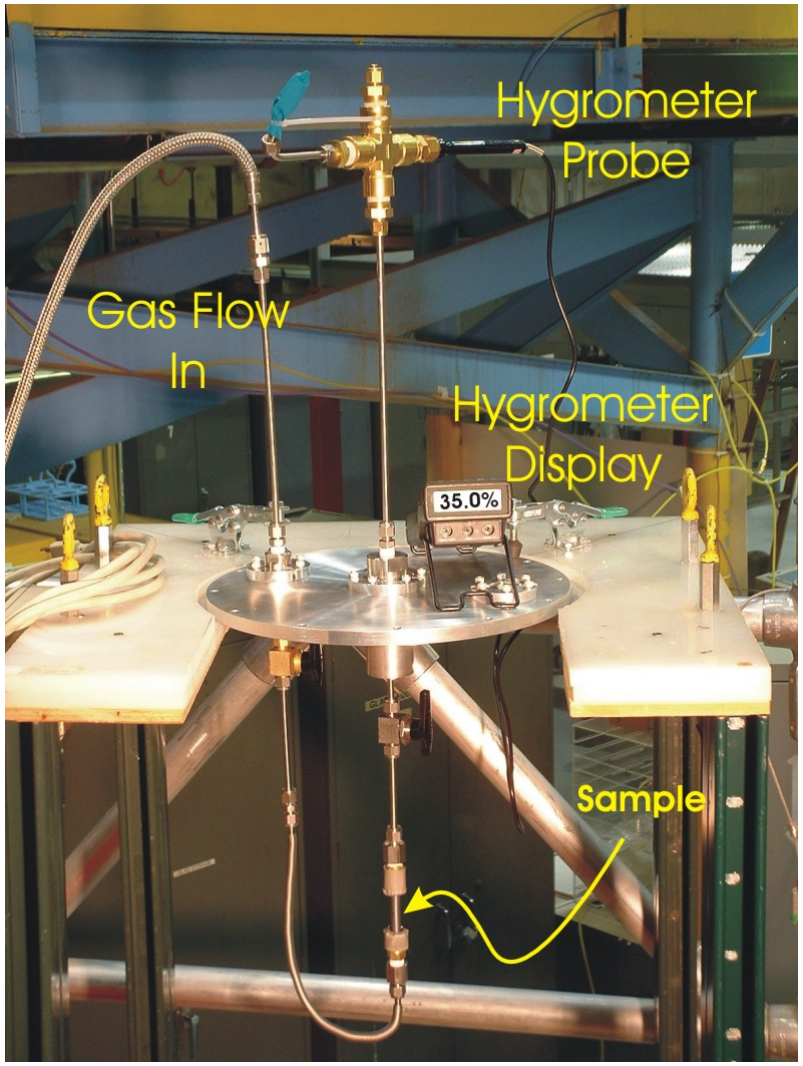




GPPD Hygrometer Cell

- Vanadium tube sample volume
- Inline Hygrometer: 2-99% RH
- Humidity control through dynamic mixing of dry and humid inert gases.





SAND – ESRF 7T Magnet

- History
  - Daresbury Laboratory
    Cheshire, UK
  - ESRF
    Grenoble, FR
  - IPNS
    Argonne, USA
- Maximum Field = 7T
- Sample Temperature Range
  1.3 K < T < RT

SAND – ESRF 7T Magnet

- History
  - Daresbury Laboratory
    Cheshire, UK
  - ESRF
    Grenoble, FR
  - IPNS
    Argonne, USA
- Maximum Field = 7T
- Sample Temperature Range
  1.3 K < T < RT
- Problems
  - Missing needle valve!
  - Spurious wiring/plumbing!
  - Quench protection missing!
- Ken's Summer, now Fall Project!

Acknowledgements

Many in the IPNS Division have contributed to the material presented but in particular the following have actively participated in the creation and commissioning of new sample environments.

- Ashfia Hug and Evan Maxey
  General Purpose Powder Diffractometer (GPPD)
- Simine Short (Retired) and Joe Fieramosca
  Special Environment Powder Diffractometer (SEPD)
- Rich Como
  Sample Environment and Ancillary Equipment Group
- Rich Vitt, Bob Kleb, Dave Leach, and Jon Baldwin
  Neutron Operations Group

The work reported in this presentation is supported by the U.S. Department of Energy, BES Materials Sciences, under contract W-31-109-ENG-38