Neutron Scattering Sample Environments

Presented at the

"The Neutron Lifecycle" Lecture Series

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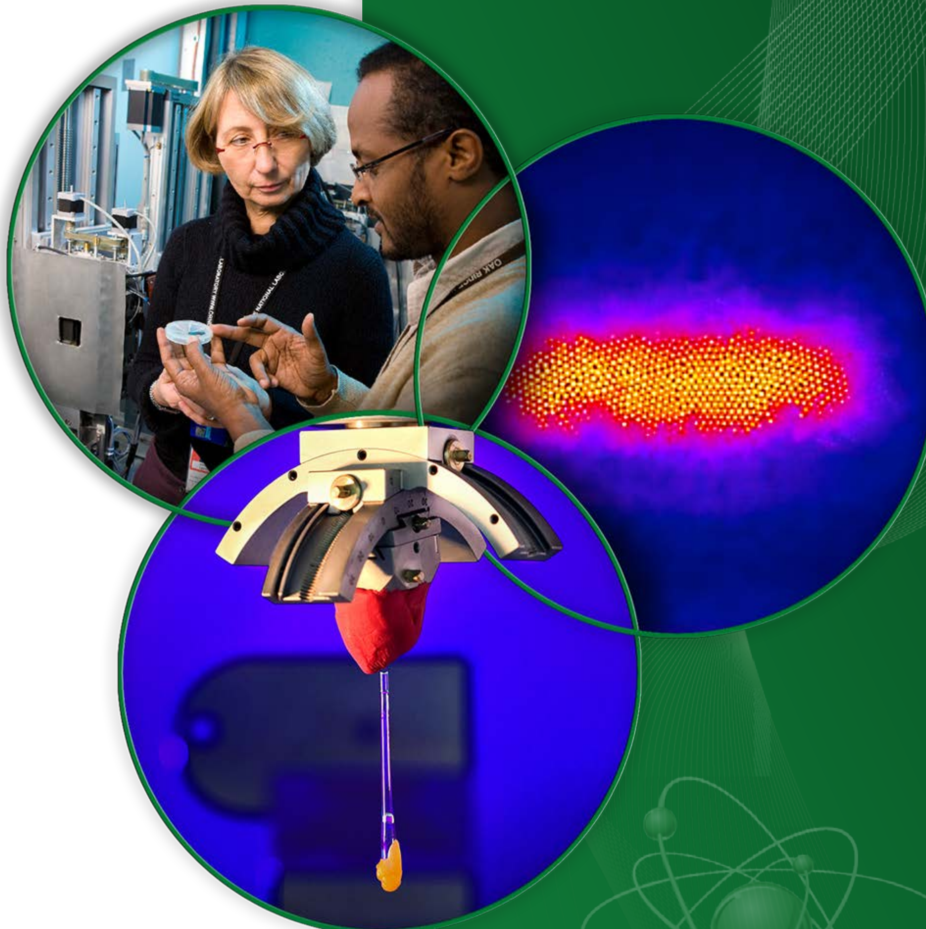
Group Leader for Sample Environment

June 30, 2016



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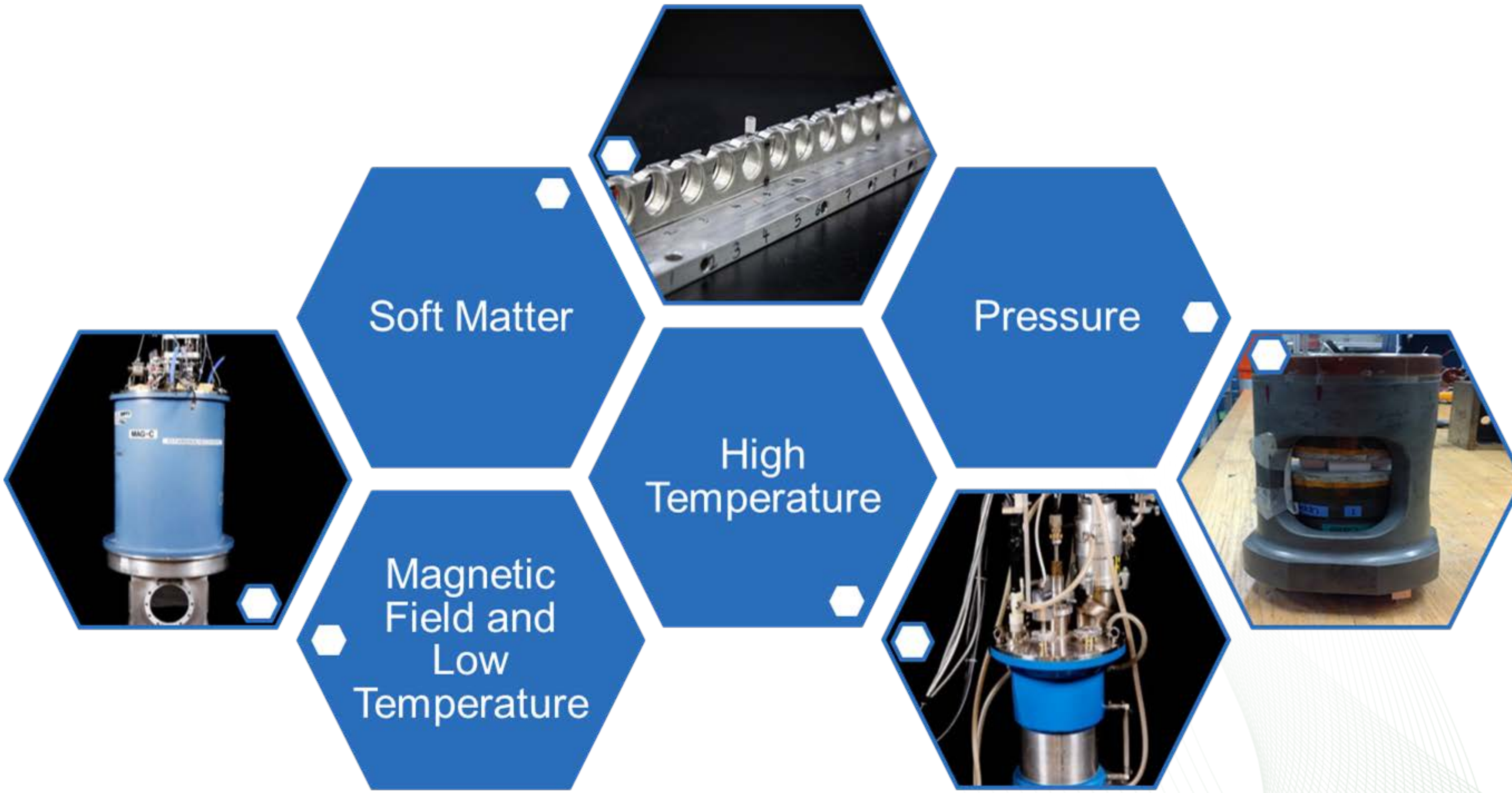
What is Sample Environment?

- Sample Environment is an integral part of the neutron scattering experiment where neutrons are used as an investigative probe
- Neutron Properties:
  - Neutrons have no electric charge: can penetrate into materials to be scattered by the nucleus
  - Neutrons have a magnetic moment and therefore are sensitive to the magnetic field of the atoms
  - Neutrons have an intrinsic energy that makes them sensitive to inter-atomic vibrations
  - Scattering and absorption cross-sections depend on the isotope: isotope labelling and contrast variation take advantage of the scattering differences between Hydrogen and Deuterium

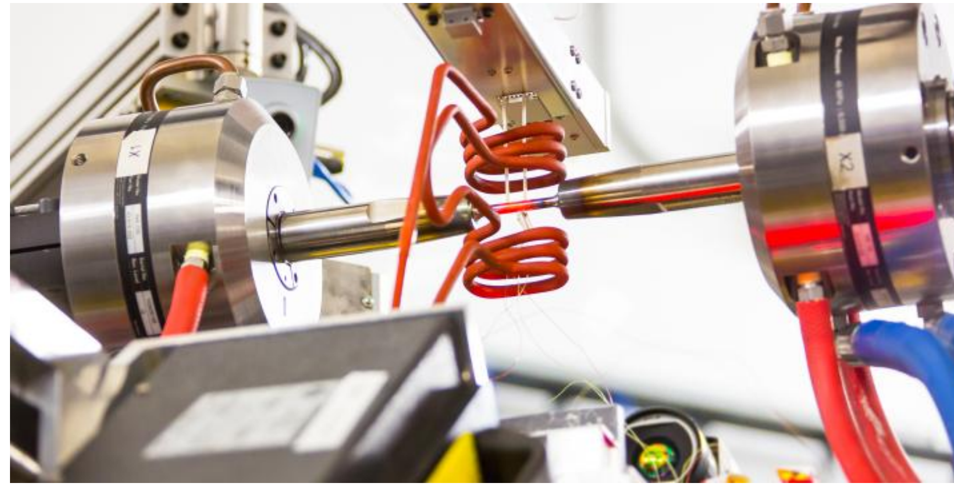
What is Sample Environment?

- Sample Environment equipment is used to precisely and accurately control experimental parameters such as temperature, pressure and magnetic fields



Neutrons Reveal Structure of Nickel-Based Superalloys Under High Temperature and Stress

- VULCAN instrument
- Nickel-based superalloys used in gas turbines
- Work by Yan Gao and Shenyan Huang of GE Global Research
- Combines microstructure measurements with mechanical testing

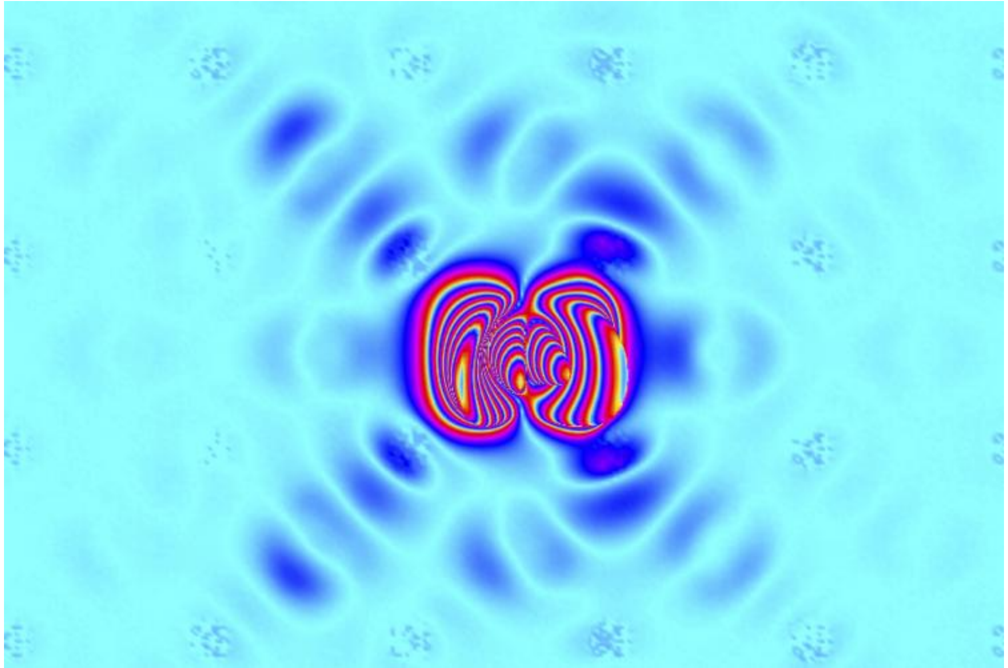


http://neutrons.ornl.gov/news/superalloys-under-high-temperature-and-high-stress

Ke An

Neutrons Probe Atomic Vibrations in Tin Selenide

- CNCS, CTAX and TAX instruments
- Explains the low thermal conductivity of tin selenide
- Efficient thermoelectric material (convert thermal gradients to electricity)
- Used in space batteries



http://neutrons.ornl.gov/node/6667

Olivier Delaire

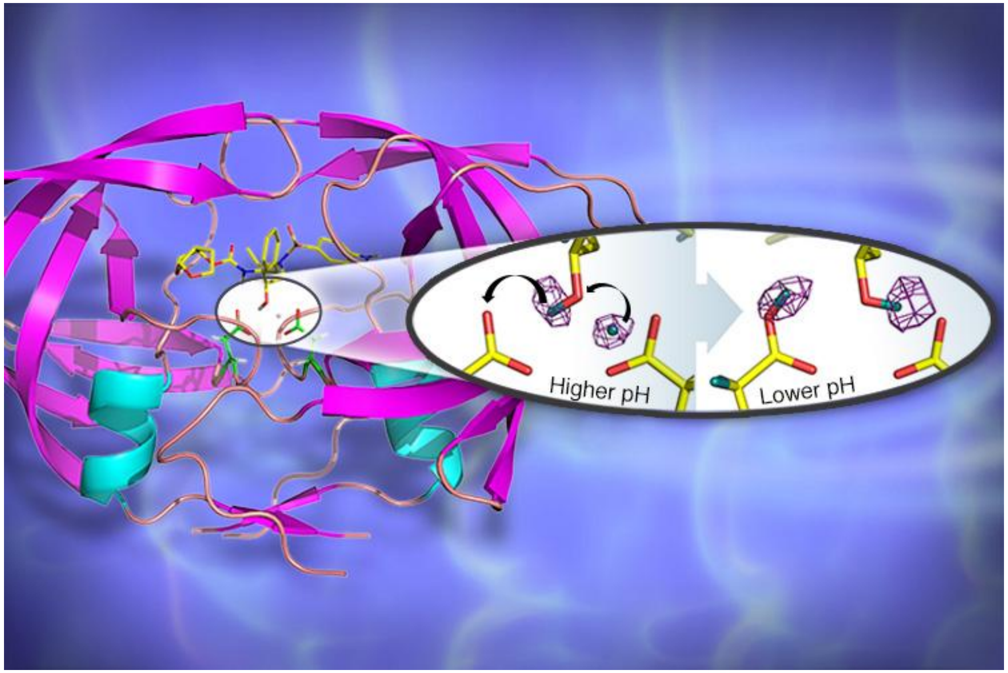
Tao Hong

Georg Ehlers

Songxue Chi

Neutrons Reveal Structure of the Enzyme HIV-1 Protease

- IMAGINE instrument
- HIV-1 Protease is a key drug target for HIV and AIDS therapies
- Deuterium labeling used to focus in on hydrogen-bonding sites related to drug binding



http://neutrons.ornl.gov/node/13745

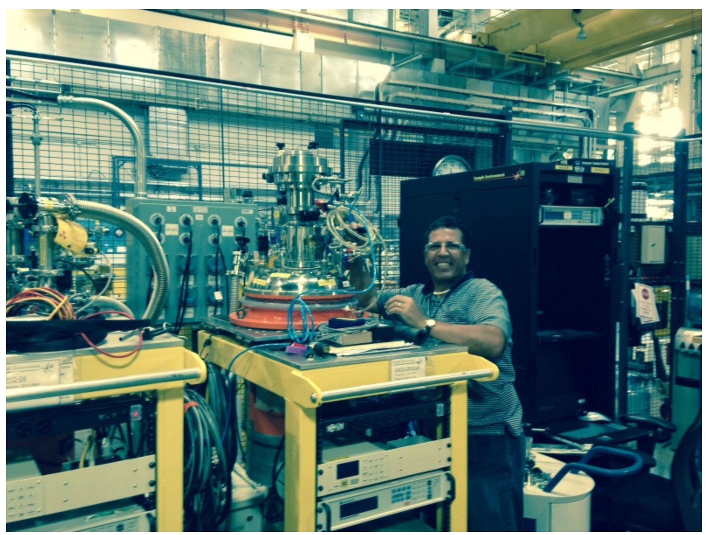
Andrey Kovalevsky

Kevin Weiss

Low Temperature

Low Temperature Equipment:

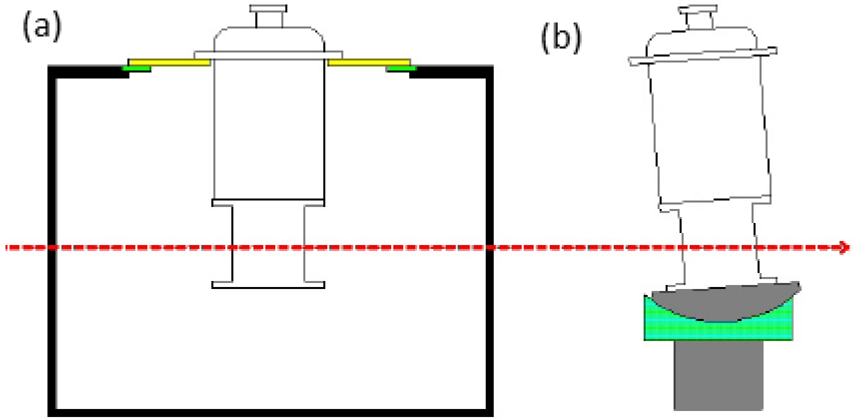
- Closed cycle refrigerators 4 K - 300 K
- Liquid Helium Cryostats 1.5 K - 300 K
- 3He inserts 0.3 K - 300 K
- Dilution refrigeration inserts 0.03 K - 300 K

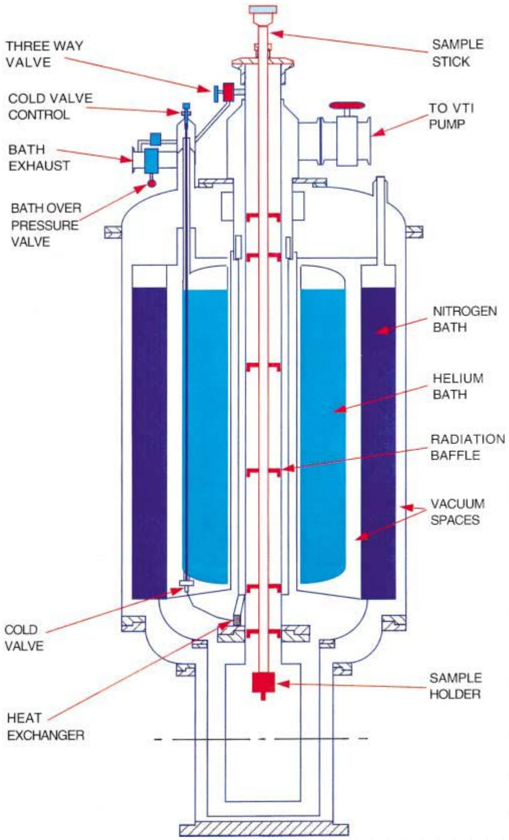


Low Temperature

Liquid Helium Cryostats

- Temperature range **1.5 K - 300 K**
- Sample exposed to 10 mbar helium exchange gas inside IVC
- Liquid helium exhausts through a heat exchanger integral to IVC
- Exhausting cold helium gas flows around and cools IVC
- Flange mount or tail mount
- Flange diameter defines maximum diameter allowed (700 mm typical)
- Outer Vacuum Chamber (tails) diameter 350 mm
- Distance from stick flange to beam center 950 mm
- Sample space diameter 43 mm





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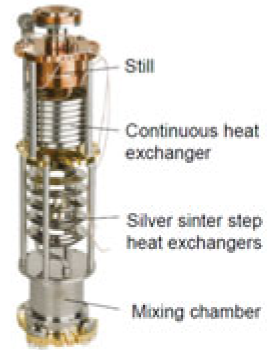
Low Temperature

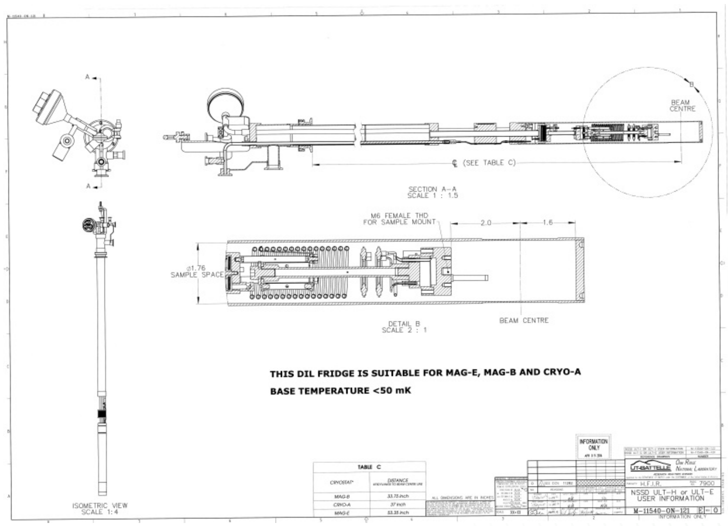
3He Insert

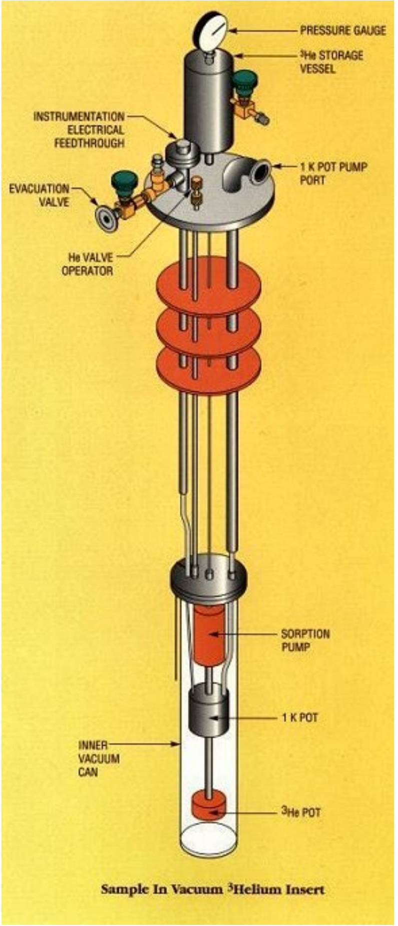
- Temperature range **0.3 K - 80 K** (up to 300 K with VTI)
- Achieve a base temperature less than 0.3 K for more than 40 hours
- Maintain a base temperature less than 0.35 K for more than 6 hours with a 50 µW heat load
- Temperature stability of ± 0.003 K below 1.2 K

Dilution Refrigeration Insert

- Temperature range **0.03 K - 1.5 K** (up to 300 K with VTI)
- Cooling power at least 40 µW at 0.1 K



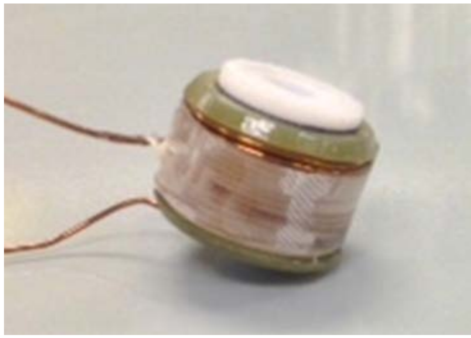




Magnets

Magnetic Field Equipment:

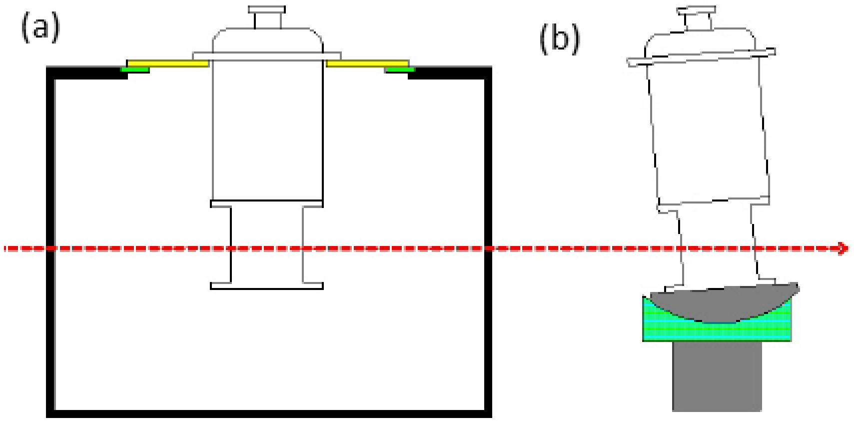
- 0.5-3 T electromagnet: specialized for Reflectometry or SANS
- 11 T superconducting cryomagnet, horizontal field
- 5-11 T superconducting cryomagnet, vertical field, symmetric or asymmetric
- 30 T pulsed magnet

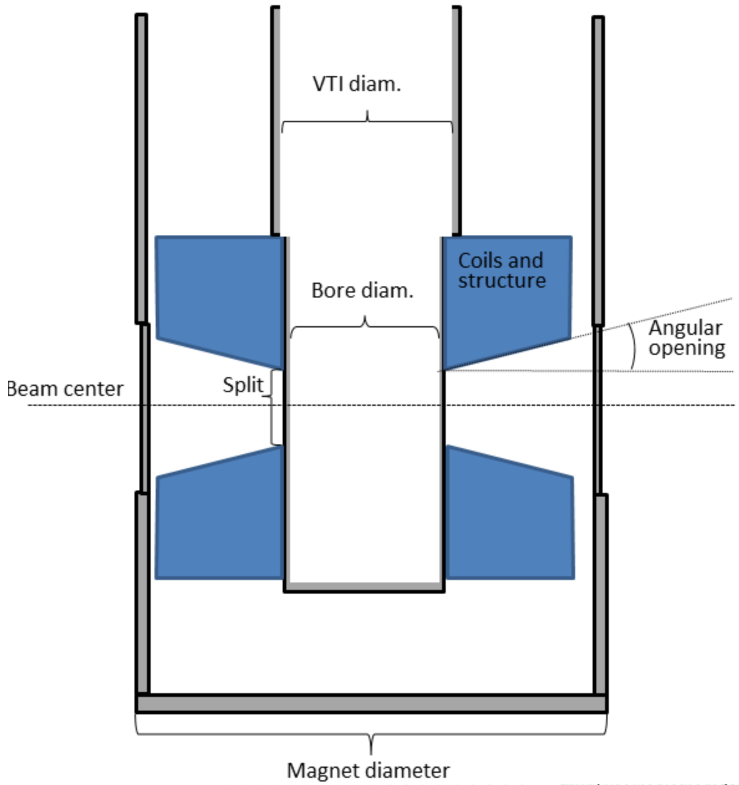


Magnets

Overall Physical Dimensions and Weight

- Flange mount or tail mount
- Flange diameter defines maximum diameter allowed (700 mm typical)
- Define flange: bolt holes, vacuum boundary, etc.
- Tail mount: distance from beam center to bottom of tail
- Maximum overall height (2200 mm typical)
    - Crane access: below the hook to mounting surface
    - Movement around the facility: through doors, etc.
- Total weight (including cryogens 450-680 kg) not to exceed crane capacity

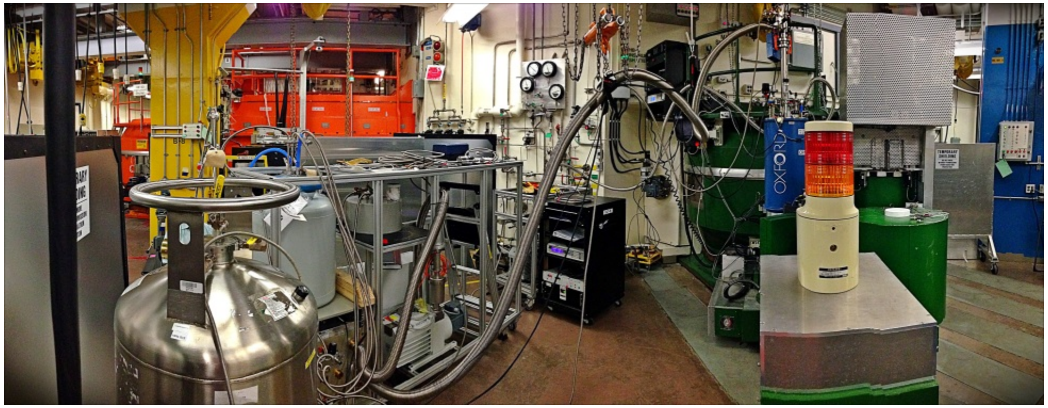




Magnets

Real Estate and Utilities

- Ancillary equipment such as power supply, Helium re-condensing equipment, vacuum pumps, etc. can take up several square meters of space around the instrument
- Routing of vacuum lines, power and signal cables can be a little tricky
- Electrical power (U.S.):
  - 60 Hz at 110 V and 20 A for instrumentation
  - 60 Hz at 208 V and 30 A for power supply
  - 60 Hz at 480 V and 30 A for cold head compressor
- Chilled water



Pressure

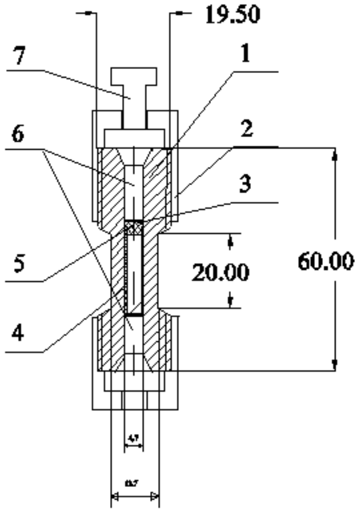
Pressure Equipment:

- 400 - 1300 bar V and TiZr cells for diffraction
- 6 kbar and 1.5 - 300 K Helium gas cells
- 4 GPa and 3.5 K Palm Cubic Anvil
- 1 - 3 GPa and 0.3 - 1.5 K Clamp cells for inelastic
- 10 - 40 GPa and 15 - 300 K Diamond Anvil Cells for diffraction

Pressure

Clamp Cell Design for Inelastic Scattering

- 500 mm^3 sample volume for inelastic scattering
- Fit in bore of magnet
- Non-magnetic material
- High thermal conductivity material to cool below 4 K
- Cell components have similar coefficients of thermal expansion
- Disadvantages: Peaks from the Material
- Choices of Material for High Pressure Cells:
  - NiCrAl yield strength 2 GPa
  - CuBe yield strength 1.2 GPa
  - CuTi yield strength 1.2 GPa
  - Maraging Steel yield strength 0.8 GPa
  - TiZr yield strength 0.7 GPa

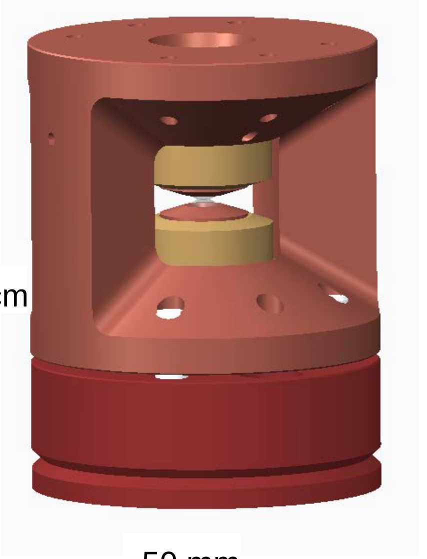


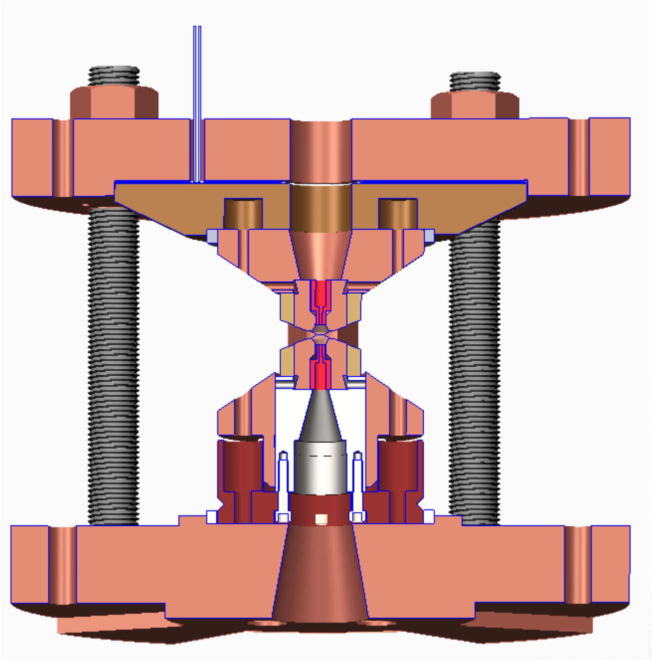
1- Body-nonmagnetic HNU (Ni-Cr-Al) alloy
2- Clamping nut-nonmagnetic Ti alloy
3- Extrusion ring-CuBe alloy
4- Capsule for sample (teflon or lead)
5- Capsule cap (teflon or lead)
6- Piston of a cell- nonmagnetic HNU alloy
7- Piston for pushing out the sample and for generating pressure- nonmagnetic HNU alloy

Pressure

Diamond Anvil Cell

- Max recorded Pressure for Neutrons: 94GPa.
- DAC can be made of Steel with Diamond anvils. For low temperatures, CuBe is used.
- Sample volume is limiting, on the order of 0.7mm in diameter and .16mm in gasket thickness
- Beamline Background reduction and collimation is of utmost importance for diamond anvil cell measurements.

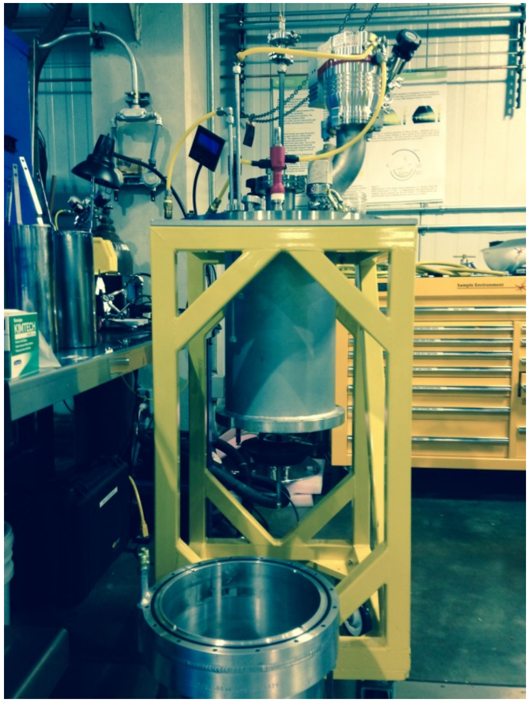


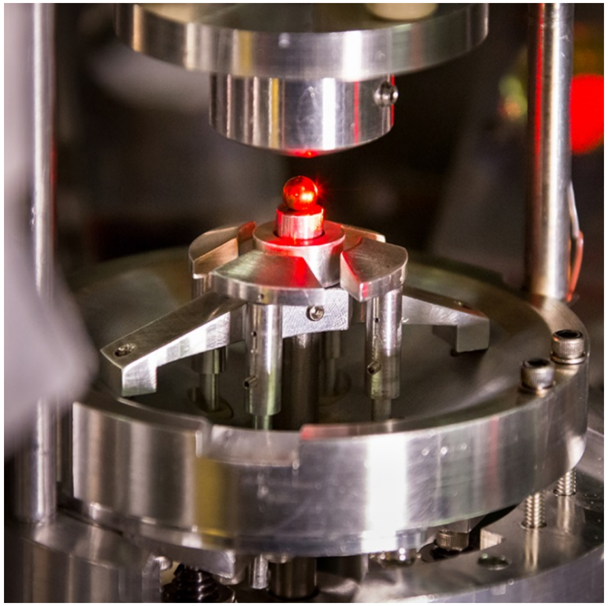


High Temperature

High Temperature Equipment:

- 1200 °C Vanadium ILL
- 1600 °C Niobium ILL
- 1200 °C or 1600 °C MICAS2
- 1500 °C Controlled Atmosphere
- 500 - 2000 °C Electrostatic Levitator

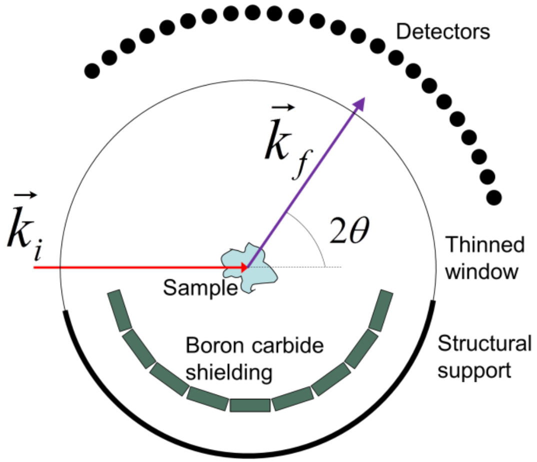


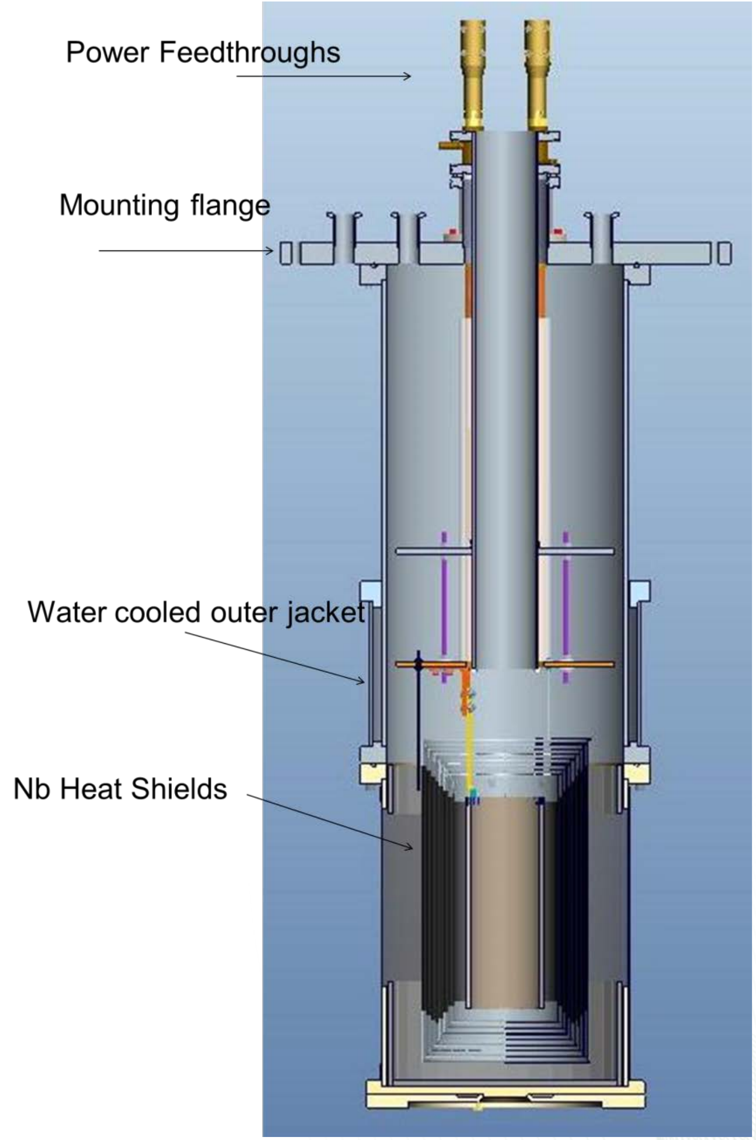


High Temperature

Radiative Heating Furnace

- Customize Outer Vacuum Chamber for detector coverage
- Minimize background using a thin (0.05 mm) Niobium window instead of Aluminum on Outer Vacuum Chamber
- Use of Boron Carbide to prevent multiple scattering

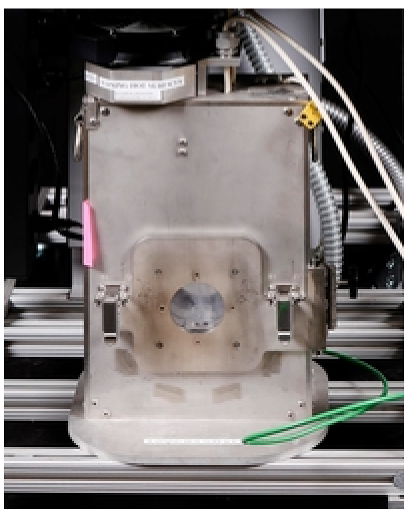




Soft Matter and Biological Materials

SANS Equipment:

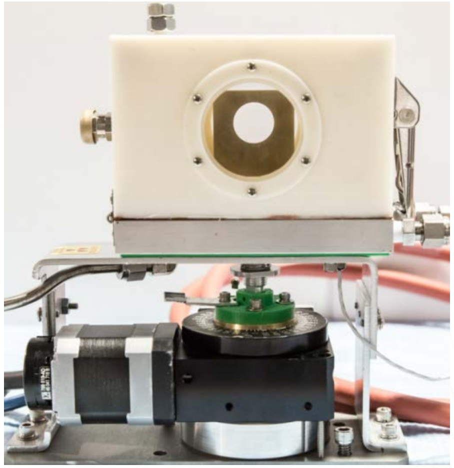
- 30 °C - 800 °C tube furnace with quartz windows
- 4.5 T superconducting cryomagnet, horizontal field, silicon windows
- Liquid Helium Cryostat 1.5 K - 300 K with sapphire windows, 200 bar pressure cell



Soft Matter and Biological Materials

SANS Equipment:

- 1 kbar pressure cell, Tantalum lined
- 1 kbar 4-position pressure cells
- Sample tumbler
- Relative humidity controlled cell



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The Ability to Control Experimental Parameters Comes with a Cost

- Avoid background issues: minimize amount of material in the incident and detected neutron beam paths
- There are always trade-offs and compromises: the best material to achieve high pressures or temperatures may not be the most neutron friendly