Neutron Scattering Sample Environments

Presented at the
“The Neutron Lifecycle” Lecture Series

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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


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

Andrey Kovalevsky
Kevin Weiss

http://neutrons.ornl.gov/node/13745
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
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 recondensing 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 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 - non-magnetic HNU (Ni-Cr-Al) alloy
2. Clamping nut - non-magnetic 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 - non-magnetic HNU alloy
7. Piston for pushing out the sample and for generating pressure - non-magnetic HNU alloy
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 0.16mm in gasket thickness.
- Beamline Background reduction and collimation is of utmost importance for diamond anvil cell measurements.
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
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