Extreme Sample Environment at ISIS Neutron Source

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Outline

• ISIS neutron scattering facility

• What is a neutron and why neutrons need low temperature

• Examples of Extreme Sample Environment
  – Extreme Sample Environment for Quantum physics
  – High Pressure & Low Temperature sample environment for Planetary Science
  – Stress in engineering components at cryogenic temperatures
  – Cryopreservation

• Acknowledgements
ISIS facility facts

• 34 neutron and 7 muon Instruments

• ~30 experiments per day simultaneously and 800 experiments per year

• about 2/3 of all experiments require cryogenic sample environment
Unlike X-rays and electrons, neutrons scatter from nucleus of an atom rather than the electron cloud.

**Neutron scattering advantages:**

- It is easier to sense light atoms, such as hydrogen.
- Isotopes of the same element have different neutron scattering lengths. That can be used for Isotopic substitution.
- The interaction of a neutron with the nucleus of an atom is weak. This allows the use complex sample environment such as cryostats, furnaces and pressure cells.
- Because of the weak interaction, neutrons are a non-destructive probe, even to delicate biological samples.
- Neutron diffraction determines the atomic structure of a material.
- Neutron spectroscopy measures how atoms and molecules move.
- Neutron has a magnetic moment that can couple directly to the magnetisation on atomic scale.
Why Neutron Scattering does need Low Temperatures?

The **thermal motion** of atoms is reduced at low temperature, significantly improving the precision of structural measurements.

Cryogenic temperature range allows the study of low temperature **phase transitions**.

**High magnetic field** sample environment is usually provided by cryogenic superconducting magnets.
Low Temperature - High Magnetic Fields Sample Environment

Magnetic field up to 17 T

Ultra low temperatures down to 0.02K

Superconductivity
Quantum criticality
Low temperature magnetism
Spintronics
Materials for Quantum Computers

Nature group journals – 34
Science – 3
Phys. Rev. Lett. - 128
Spin excitation spectrum of the Heisenberg spin ladder material

*Experiment*  

*Theoretical model*

D. Schmidiger et. al.  
*PRL* **111** (2014) 107202
Edinburgh Sputnik sell on E-18 dilution fridge
Joint project with University of Edinburg

Ability to change pressure at ~ 6K

Ruby luminescence in-situ pressure measurements

Pressure: \textit{up to 50 kbar} (5 GPa)

Sample size: 1 mm$^3$

Base temperature: < 100 mK

Magnetic field \textit{up to 4T}

\textit{Sputnik cell} on E18 fridge mixing chamber

(c) Sputnik cell with laser printed collimator
Europa’s cryovolcano

Enceladus cryogeysers

Crescent Jupiter and Ganymede

monster storms

Io

Pluto’s cryoglaciers

Titan’s cryolava

Comet 67P cryojets

Saturn’s diamond rain (or hail)
High Pressure & Low Temperature sample environment for Planetary Science

Temperature range: 20 – 200K (-253 to -73°C)

Pressure range: up to 300 kbar (30 GPa)

Titan from behind two of Saturn's rings and small moon Epimetheus
Variable temperature insert for the Paris Edinburgh Press

Based on two GM Cryo-coolers
Up to 300 kbar (30 GPa) pressure
Expected temperature range 20 - 300K

Paris Edinburgh anvils
**Neutron scattering measurements of bulk stress in engineering components at temperatures as low as 6.5K**

*Internal stresses* in materials have a considerable effect on material properties including *strength, fracture toughness* and *fatigue resistance.*
Engin-X is optimized for the measurement of strain, and thus stress, deep within a crystalline material, using the atomic lattice planes as an atomic 'strain gauge'.
One of the most popular Engin-X applications: measurement of internal stress in engineering materials under loads. The uniaxial load up to 100kN is provided by Hydraulic loading rigs. Stress rig cryostat provides sample environment temperature: 6.5K – 500K.
Engin-X Stress Rig Cryostat

Two CCRs: *Sumitomo RDK-415D*
Base Temperature: **6.5K**; Load up to: **100kN**
Cooling down to base temperature: **90 min**
2\textsuperscript{nd} generation HTS tape sample results

Load, N

Critical current, A

T = 77K

$I_c = 41.3$ A

Neutron diffraction data
Mechanical properties of superconducting wires and coil assemblies which are required for modelling and designing of advanced magnets for MRI and NMR.

Advanced magnets for Large Science Facility projects such as:

- **High Luminosity Upgrade** for the LHC
- **ITER** superconducting magnet

Variety of different superconducting applications based on newly developed $\text{MgB}_2$ wires and second generation HTS tapes such as Maglev trains.
Cryobiology

Cryopreservation of sperm, embryos, blood cells, stem cells, tissues and even small organs plays key role in IVF treatment, organ transplantation, preservation of animal genetic resources and other bio-medical applications.

Cryoprotectant solution used for fish embryo vitrification

In our research we used cryoprotectant solution formulated during a previous study of *Common carp* embryo cryopreservation. (B. Dzyuba et al. *Cryobiology* 61, (2010) 404)
Effect of cryoprotectant concentration on vitrified fish eggs

(a) Embryo at 77K dispersed in solution containing 15.4% PD (1,2-propanediol) and 11.4% methanol. Both medium and embryo appear opaque in cooled condition;

(b) Increased concentration of cryoprotectants (23% PD, 17% methanol) leads to appearance of transparent medium and opaque embryo;

(c) Finally the mixture with 23% PD, 17% methanol and 20% DMSO results in transparency of both medium and embryo.
**Neutron diffraction data**

All samples have been quench-cooled to 80 K and after that remained at this temperature during collecting neutron scattering data.
The water is confined into small volumes ~ 1 nm by the surrounding matrix of PD, methanol and DMSO. These chambers joined by a random network of water molecules, are too small to allow ice nucleation to occur on the practical time-scale for quench-cooling.

**Conclusions**

- Neutron scattering is a powerful tool which reveals atomic structure of a sample and allows to study movement of atoms.

- Weak interaction of a neutron with the nucleus of an atom allows the use of complex sample environments such as cryostats, superconducting magnets, furnaces and pressure cells.

- *Extreme conditions sample environment* in combination with *neutron scattering* allow experiments in frontier areas of science and technology that span from quantum physics and planetary science to engineering and bio-medical research.
I would like to thank:

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Thank you very much for your attention!