Cryogenic Sample Environment for neutron scattering experiments at ISIS

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ISIS facility facts

- 34 neutron and 7 muon Instruments

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

- ~95% of all experiments require some kind of Sample Environment (SE):
  - about 2/3 of all experiments require cryogenic SE
  - gas handling, high pressure and high temperature SE ~ 25%
  - magnetic field SE ~ 10%
Ultra Low Temperature Sample Environment
Experiments below 1K, often in High Magnetic Field

Number of ULT experiments per year

Long shutdown in 2014

Superconductivity
Quantum criticality
Low temperature magnetism
Spintronics
Quantum Fluids and Solids

Nature group journals – 38
Science – 3

About 2/3 of all ULT sample environment is used in muon spectroscopy experiments
### ISIS Helium Recovery

**Long shutdown in 2014**

<table>
<thead>
<tr>
<th>Year</th>
<th>Cost</th>
<th>Usage in Litres</th>
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ISIS Helium Recovery Building
Inauguration Event 30/06/2016
Europa’s cryovolcano

Enceladus & Io cryogeysers

Io

Crescent Jupiter and Ganymede monster storms

Pluto’s cryoglaciers

Titan’s cryolava

Comet 67P cryojets

Saturn’s diamond rain (or hail)
Variable temperature insert for the Paris Edinburgh Press

Based on two GM Cryo-coolers
Up to 300 kbar (30 GPa) pressure
Temperature range 20 - 300K (16/09/16)
Cooling down to base temperature: 2 h 40 min
ISIS Facility neutron spallation Target Station II (2009)

Solid Methane Moderator (47K)

Target
Experimental set-up of the “solid methane poke test”

OK et al. *Cryogenics* **52** (2012), 325
Bonding produced at 70K and breaking test conducted at the temperature of interest

Bonding procedure as well as breaking test conducted at the same temperature

OK et al. Cryogenics 52 (2012), 325
Ya. Yamashita and M. Kato Results of penetration tests: viscosity of solid methane at 77K $\mu = 3 \times 10^8 \text{ Pa s}$

Arrhenius form with activation energy of 1300 K ($T > 60K$)
E. Shabalin et. al. ~ 1100K

$25K < T < 50K$ - the creep rate follows Arrhenius form with activation energy of $\sim 300 – 400K$

**Dynamic viscosity:**
Honey: $2 - 10 \text{ Pa s}$
Molten glass: up to 1000 Pa s
Pahoehoe lava: 100 – 1000Pa s

*Andesite lava:* $10^6 – 10^7 \text{ Pa s}$
Rhyolite lava: $10^{11} – 10^{12} \text{ Pa s}$
This artist concept shows a lake and a mounting on the surface of Titan. Image credit: NASA/JPL

*Methane Ice* and *Snow* have been detected on a top of more than 1.5 km high mountings discovered by *Cassini–Huygens mission* in 2006 (NASA–ESA–ASI)

Titan average surface temperature 94 K
Methane melting point 90.7 K
Methane Snow on Pluto’s Peaks (3 – 4 km high)

NASA’s New Horizons mission, January 2015

Pluto temperature range: from 33K to 55K
Neutron scattering measurements of bulk stress in engineering components at temperatures as low as 6.5K

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'.

Science & Technology Facilities Council
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**

HTS Current Leads
Sample Grips
2\textsuperscript{nd} generation HTS tape sample results

Load = 200N
T = 77K

I_c = 41.3 A

Critical current, A

Load, N

Neutron diffraction data
Effect of cryoprotectant concentration on vitrified fish eggs

Sample vitrification cooling/rewarming protocol

(a) Embryo at 77K dispersed in solution: 15.4% PD and 11.4% methanol.

(b) Increased concentration of cryoprotectants 23% PD, 17% methanol

(c) Finally the mixture with 23% PD, 17% methanol and 20% DMSO
The water is confined into small volumes $\sim 1 \text{ nm}$ by the surrounding matrix of PD, methanol and DMSO. These clusters are too small to allow ice nucleation to occur.

### Sandals
- Small angle neutron diffractometer for liquids and amorphous samples
- Alan Soper, Boris Dzyuba, Barry Fuller, Bill Holt, Andy Church, Sam Callear, OK

Segregated water observed in a putative fish embryo cryopreservative *Royal Society Open Science* 3: 150655 (2016)

Strong isotope effects on melting dynamics and ice crystallisation processes in *cryo vitrification solutions* *PLoS ONE* 10(3): e0120611 (2015)

### Iris
- High-resolution QENS spectrometer
- Barry Fuller, Joana Mendonca Da Silva, Vicky Garcia Sakai, Ian Silverwood, Andy Church, Jeff Vine, OK

Cryopreserved liver cell spheroids used in artificial liver bio-transplant
- Research in progress…
I would like to thank:

All the members of *ISIS Cryogenics Team* as well as other members of *ISIS Sample Environment group*; *ISIS engineers and designers*; *ISIS administration* for not forgetting about Sample Environment; *ISIS scientists*, and also our colleagues from other Neutron Scattering Facilities particularly *ILL, HZB, PSI* and *ESS* and *Industry* for active involvement and support.

Thank you very much for your attention!