Extreme Sample Environment at ISIS Neutron Source

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CERN, European Cryogenics Days, 10th of June 2016

ISIS

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

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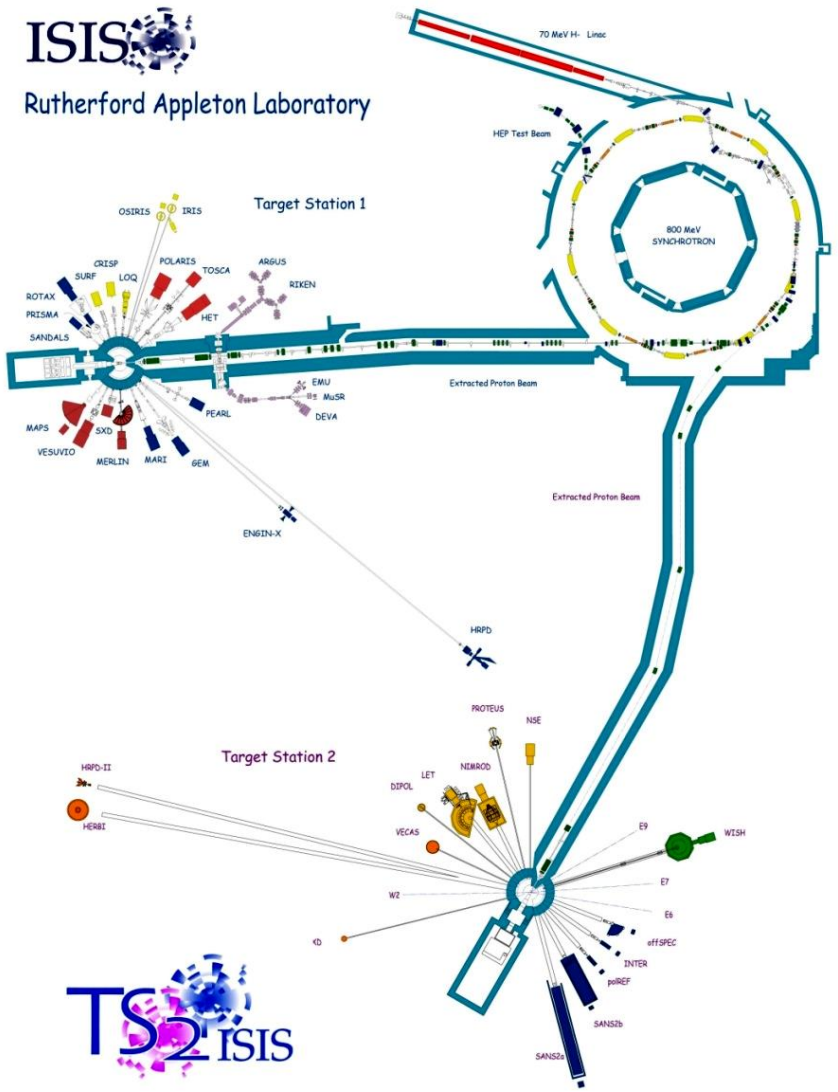
ISIS facility facts



- ***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***

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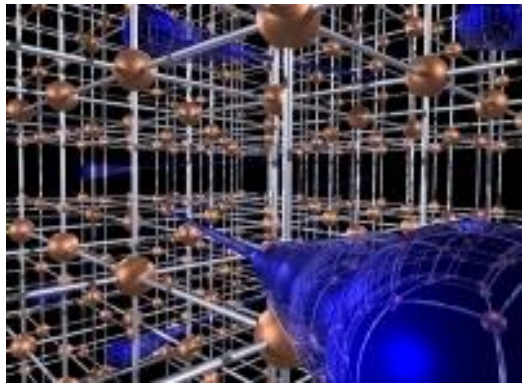
What is a neutron?

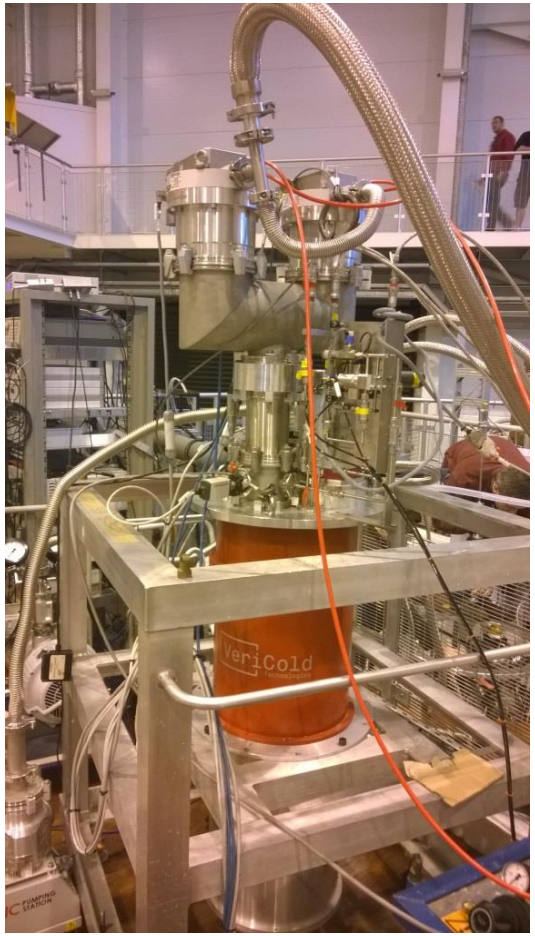
Unlike X-rays and electrons, ***neutrons*** scatter from nucleus of an atom rather than the electron cloud.

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

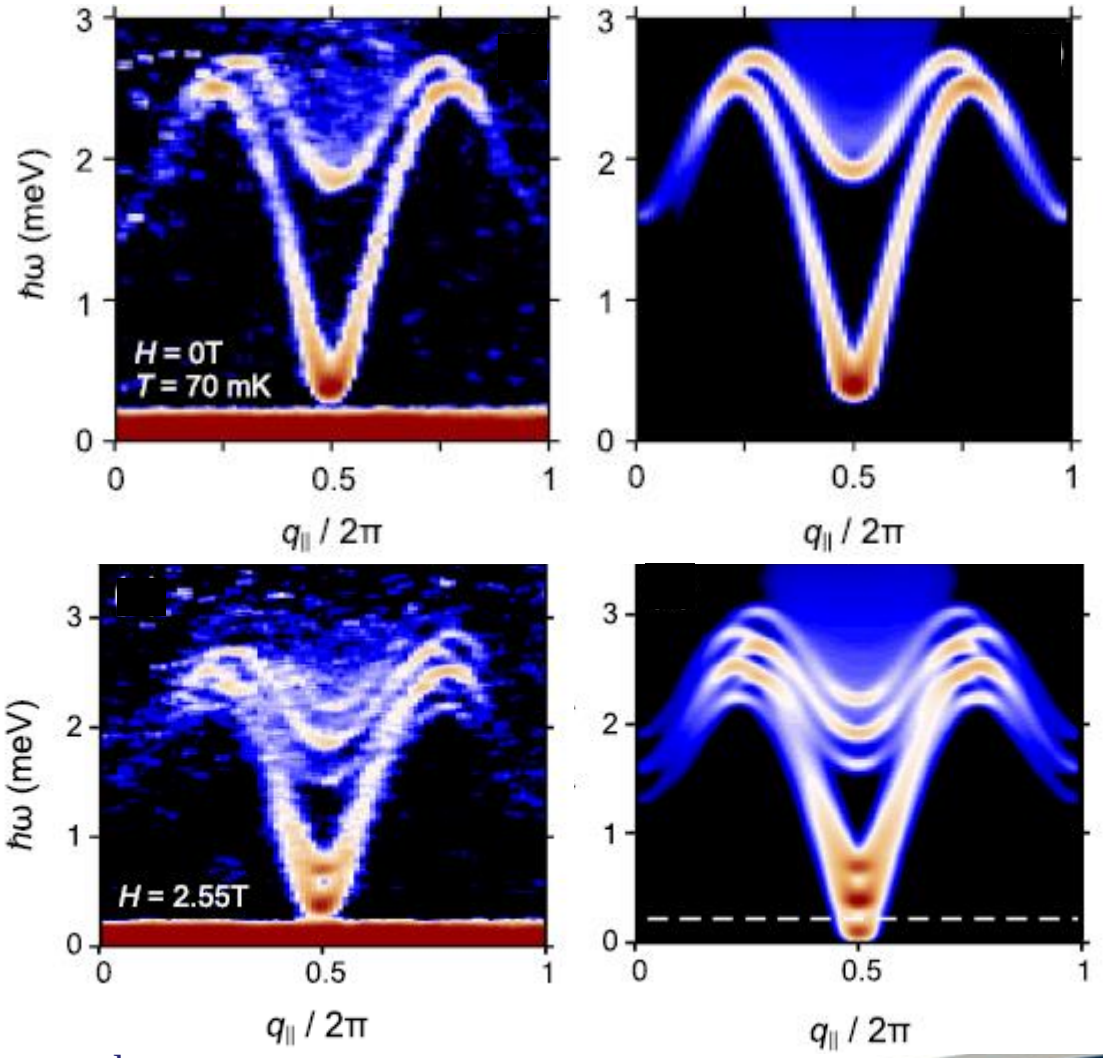
Ultra low temperatures down to 0.02K

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

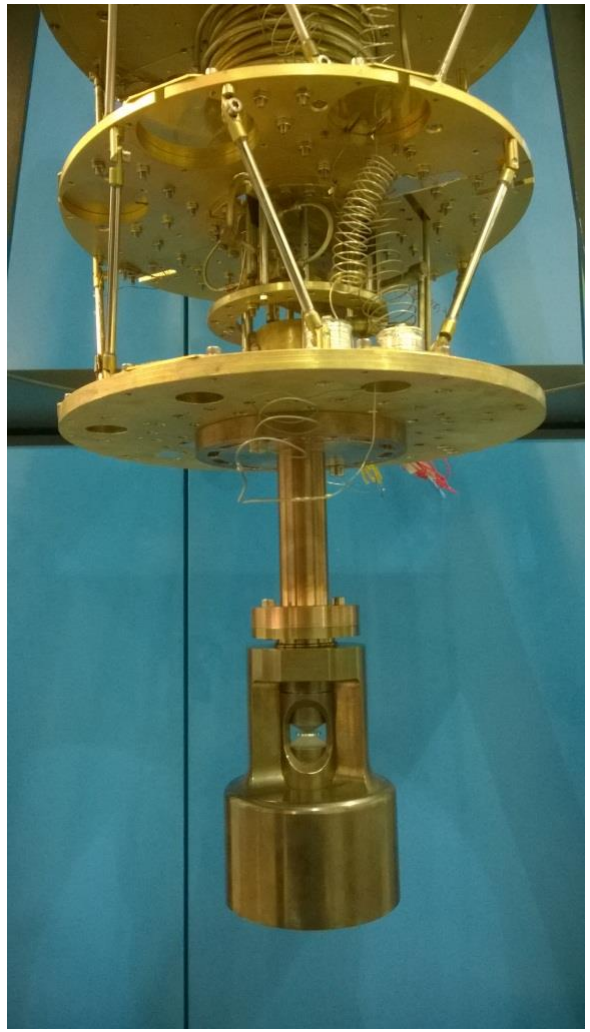
Experiment ***Theoretical model***



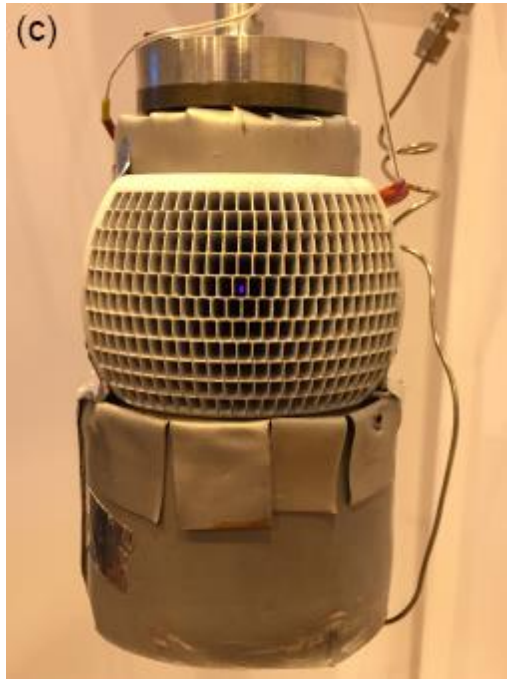
D. Schmidiger et. al.
PRL 111 (2014) 107202

Edinburgh Sputnik sell on E-18 dilution fridge

Joint project with University of Edinburg



Sputnik cell on E18 fridge mixing chamber



Sputnik cell with laser printed collimator

Ability to change pressure at ~ 6K

Ruby luminescence in-situ pressure measurements

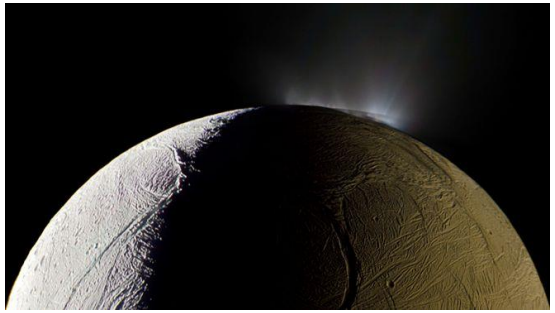
Pressure: ***up to 50 kbar*** (*5 GPa*)

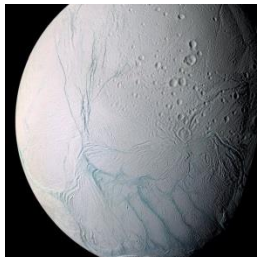
Sample size***: 1 mm^3***

Base temperature: ***< 100 mK***

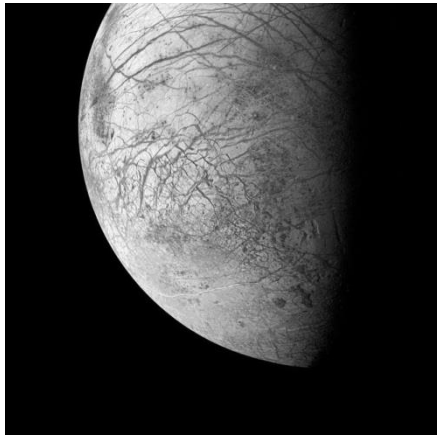
Magnetic field ***up to 4T***

Crescent Jupiter and Ganymede
monster storms





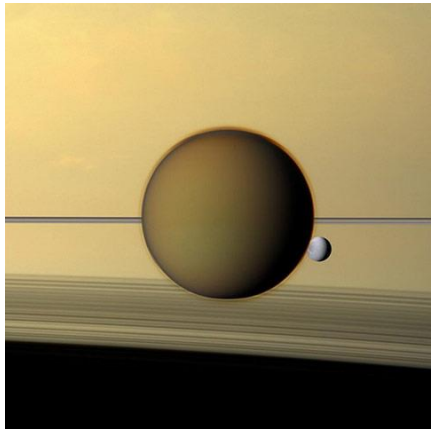
Enceladus
cryogeysers



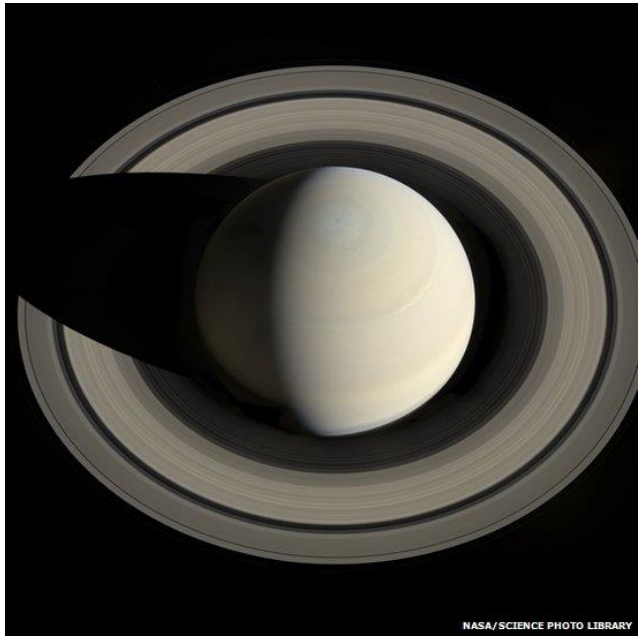
Europa's ***cryovolcano***

Io

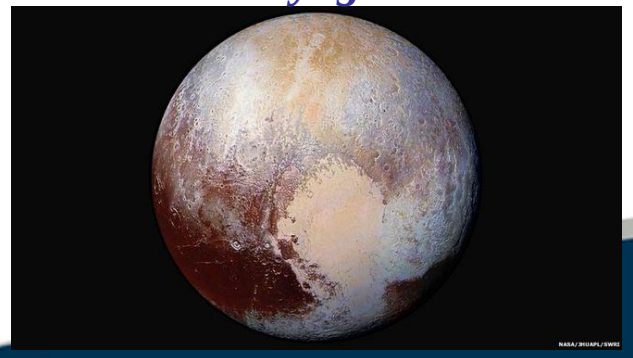
Pluto's ***cryoglaciers***



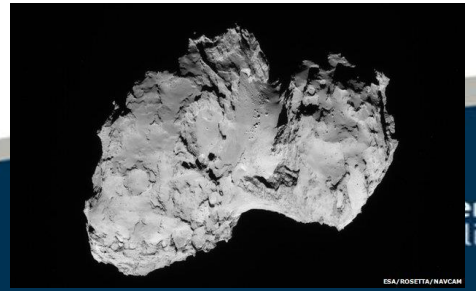
Titan's ***cryolava***



Saturn's ***diamond rain*** (or ***hail***)



Comet 67P ***cryojets***



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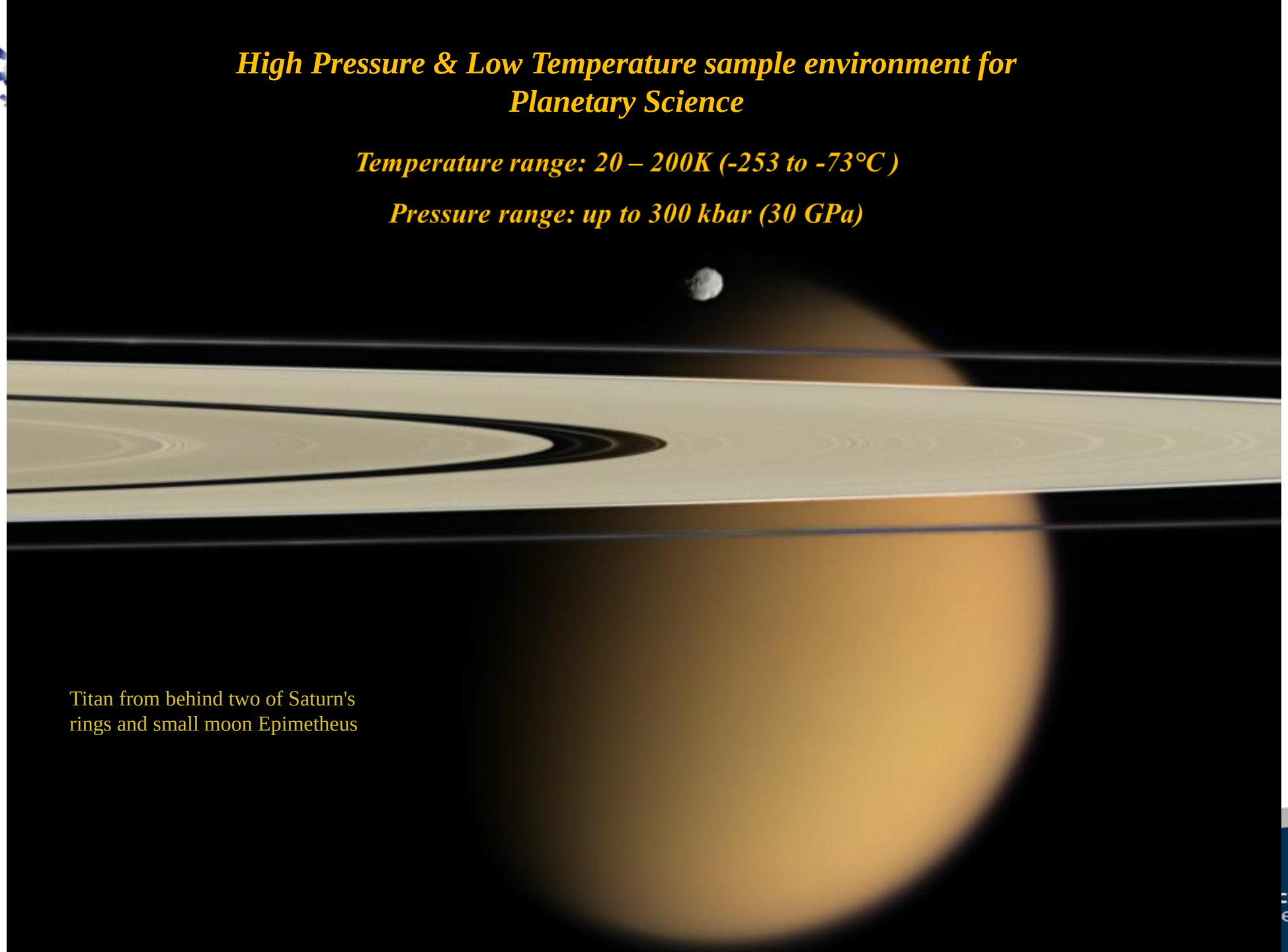
High Pressure & Low Temperature sample environment for Planetary Science

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

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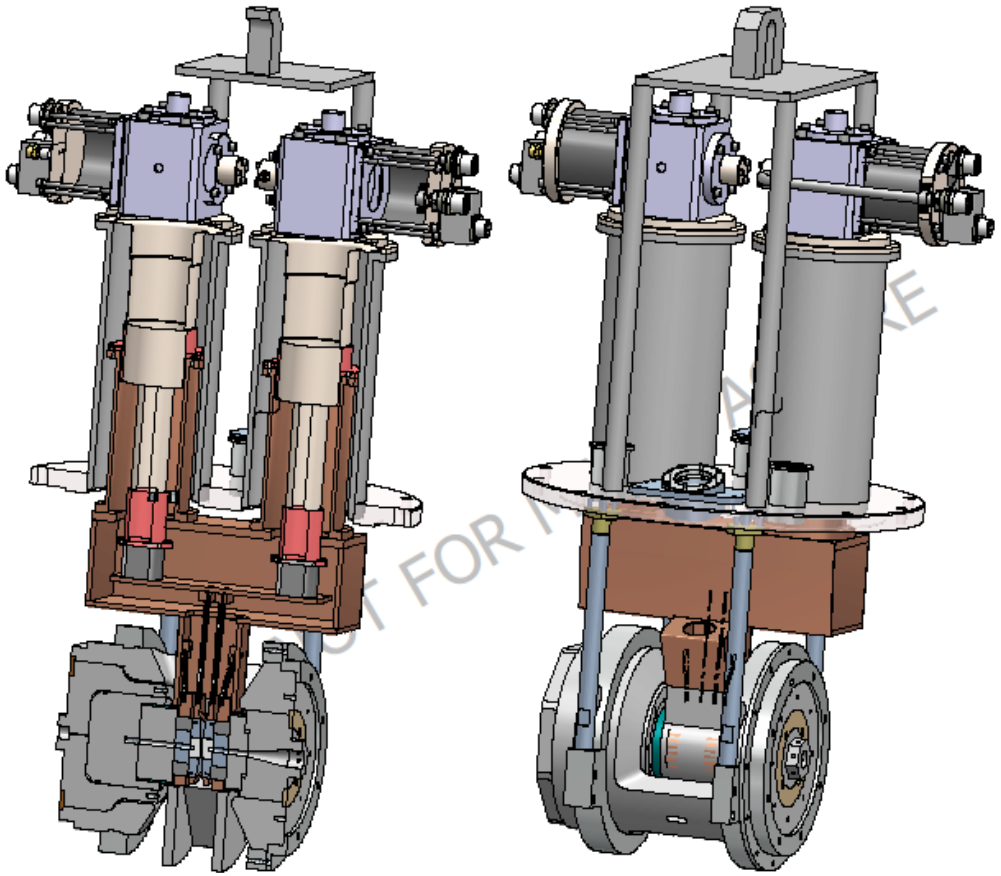
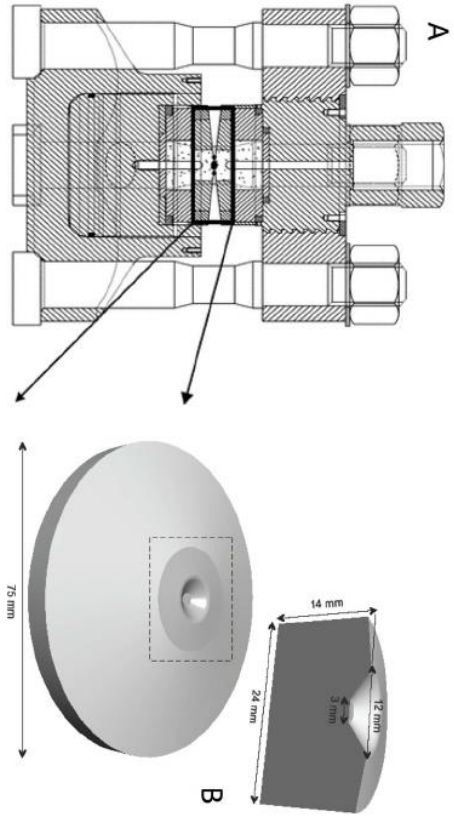
Variable temperature insert for the Paris Edinburgh Press



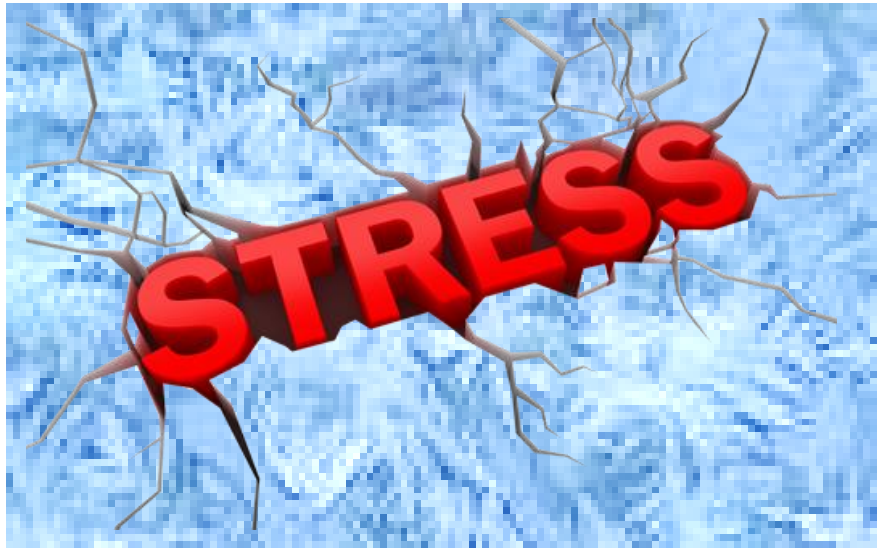


Paris Edinburgh anvils

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

ISIS

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***.

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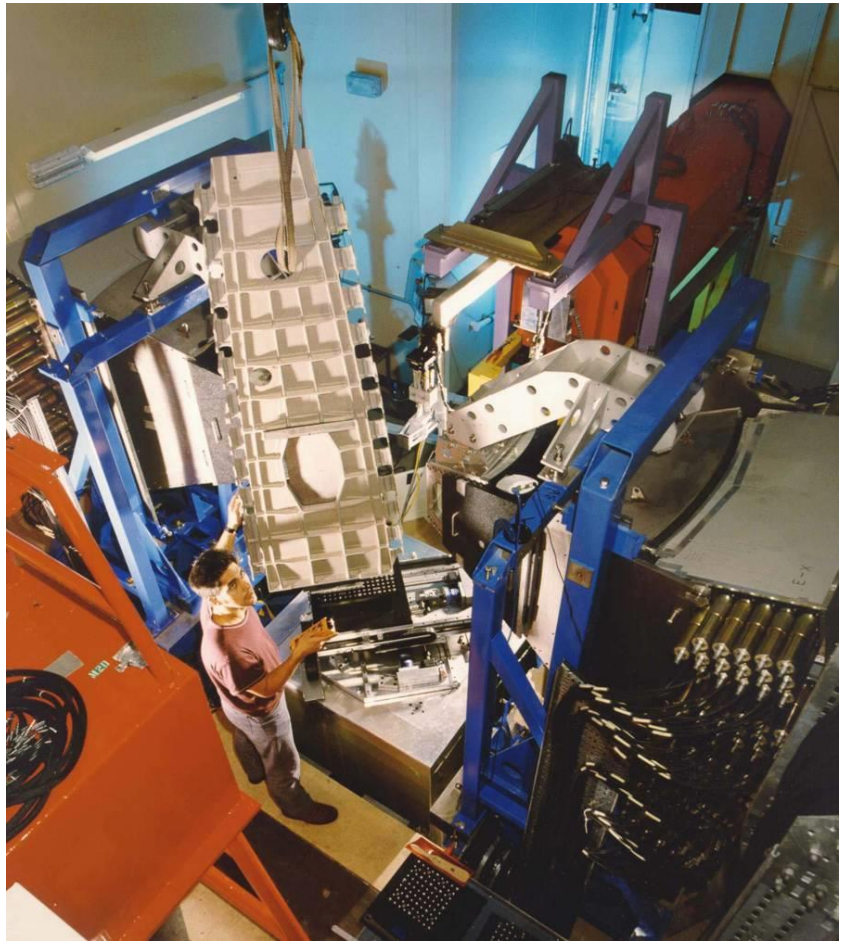
Engin-X: Engineering materials beam-line at ISIS Facility

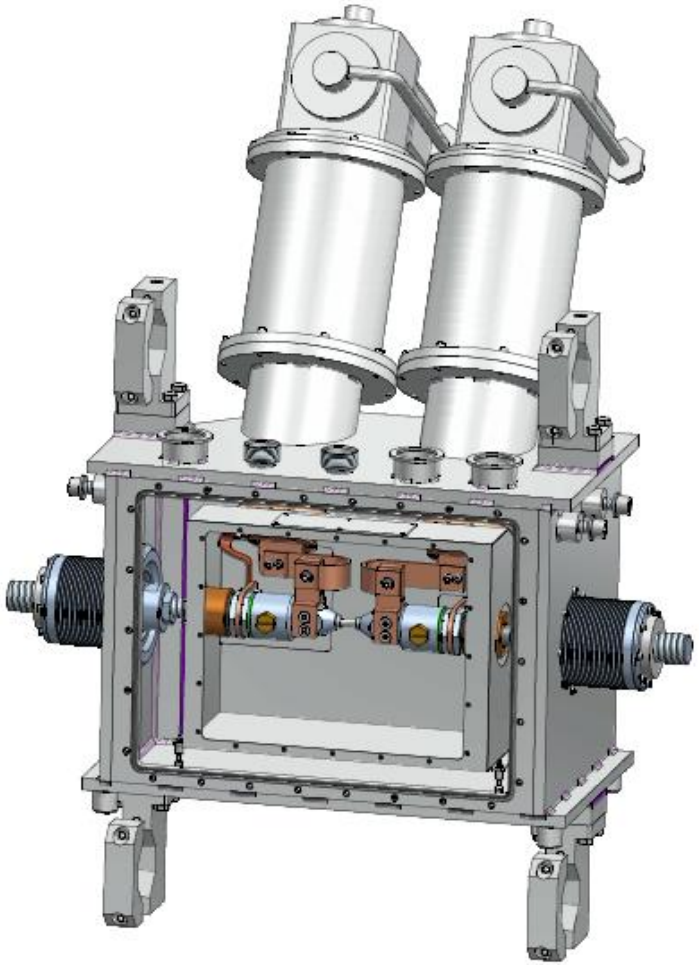


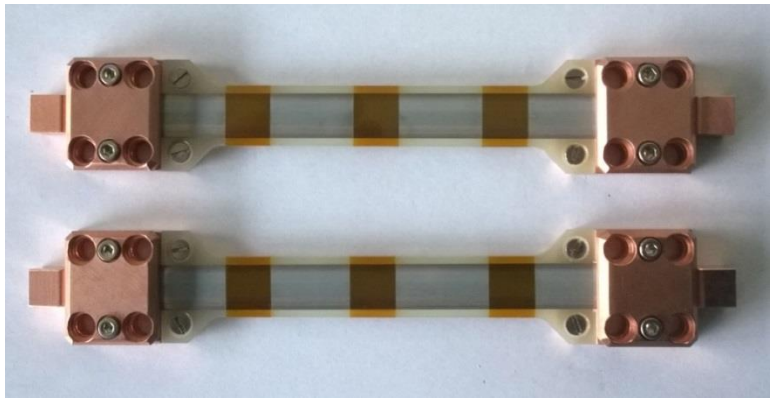
Engin-X is optimized for the measurement of ***strain***, and thus ***stress***, deep within a crystalline material, using the atomic lattice planes as an atomic 'strain gauge'.

Uniaxial hydraulic loading rigs

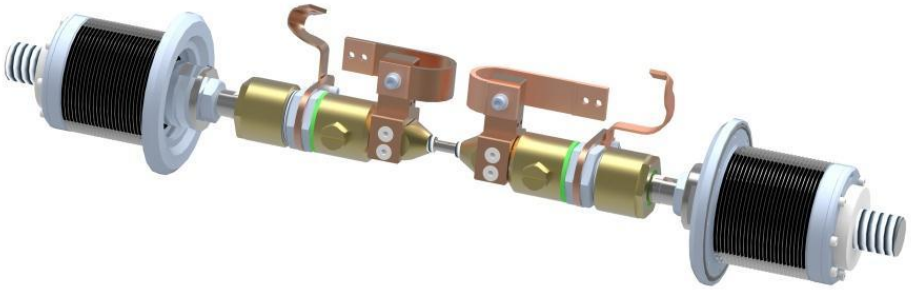
One of the most popular ***Engin-X*** applications: measurement of ***internal stress in engineering materials under loads***. The uniaxial load up to ***100kN*** is provided by ***Hydraulic loading rigs. Stress rig cryostat*** provides sample environment temperature: ***6.5K – 500K.***

Engin-X Stress Rig Cryostat





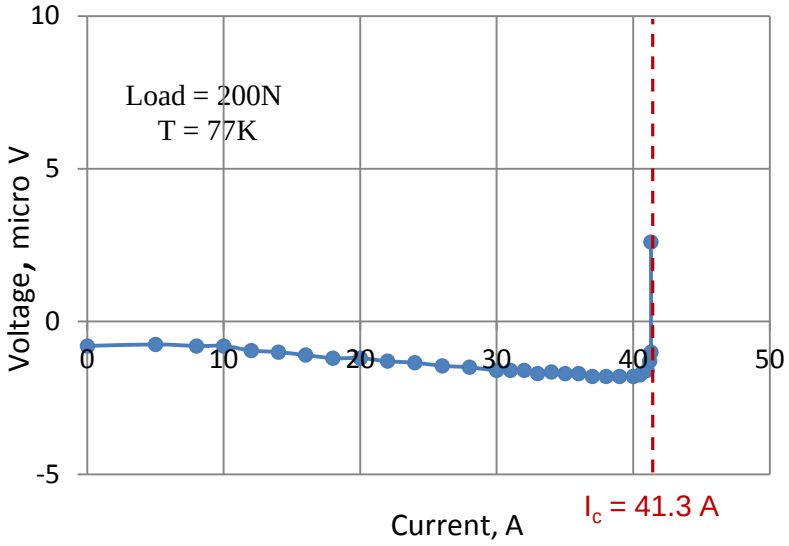
HTS Current Leads

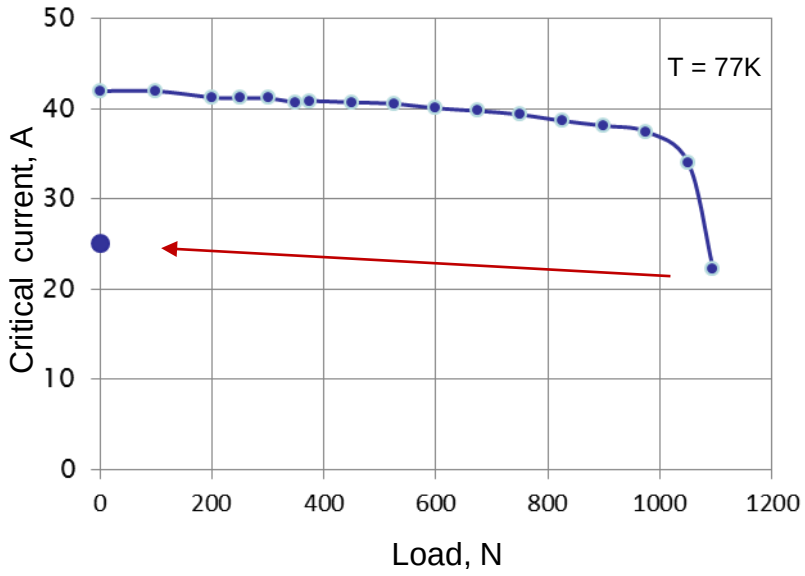


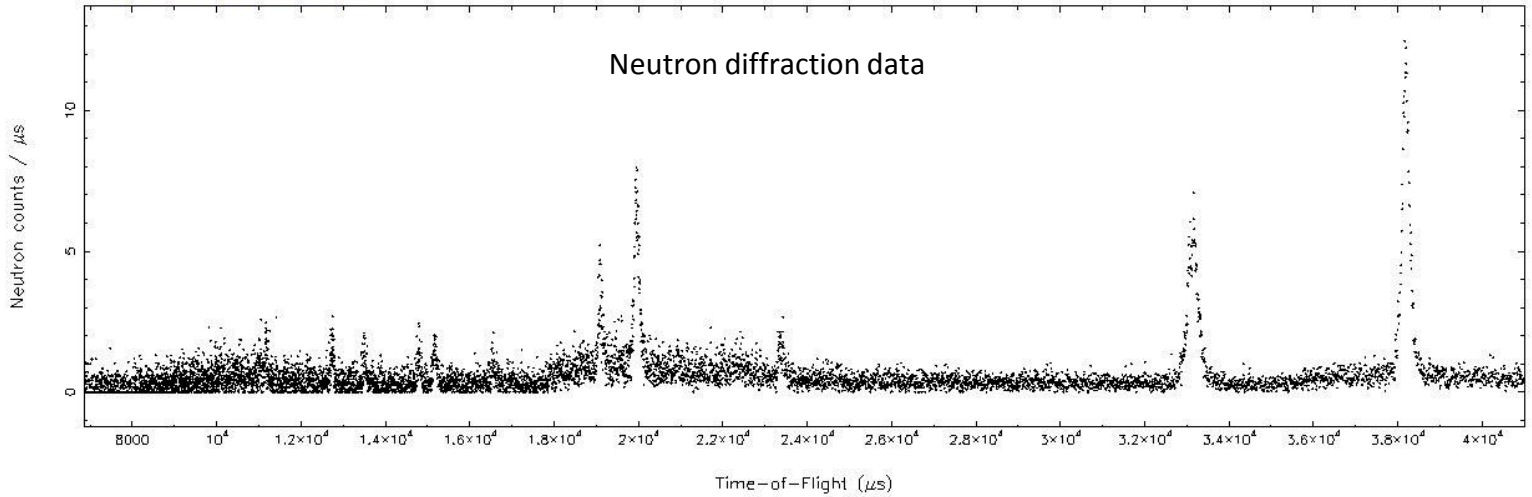
Sample Grips

Two CCRs: ***Sumitomo RDK-415D***
Base Temperature: ***6.5K;*** Load up to: ***100kN***
Cooling down to base temperature: ***90 min***

2nd generation HTS tape sample results



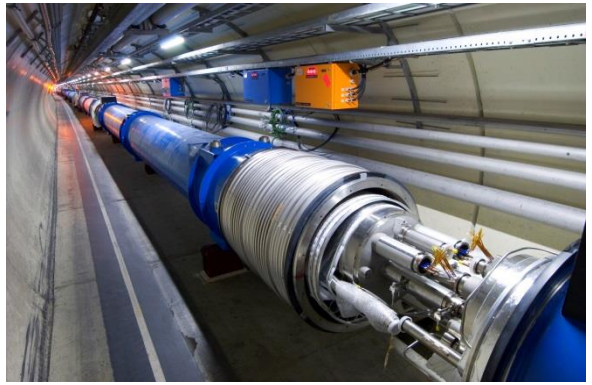


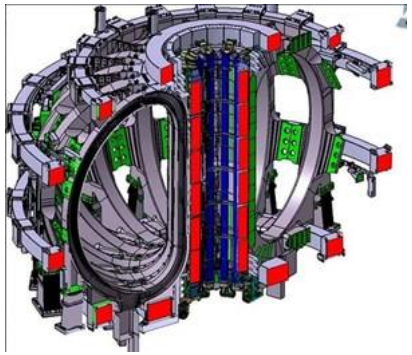


Mechanical properties of ***superconducting wires*** and ***coil assemblies*** which are required for modelling and designing of advanced magnets for ***MRI*** and ***NMR***

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 ***MgB_2*** wires and ***second generation HTS tapes*** such as ***Maglev trains***

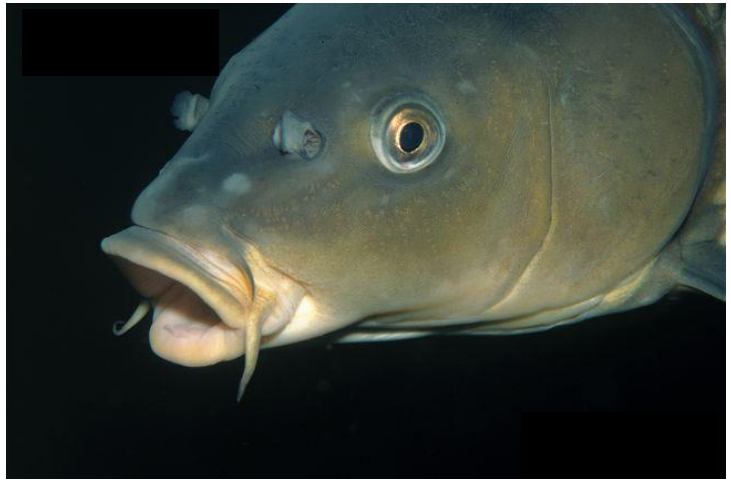
Cryobiology

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.

Main problem - crystallization of water in extra- and inter-cellular space.
Solutions – cryoprotectants and vitrification.

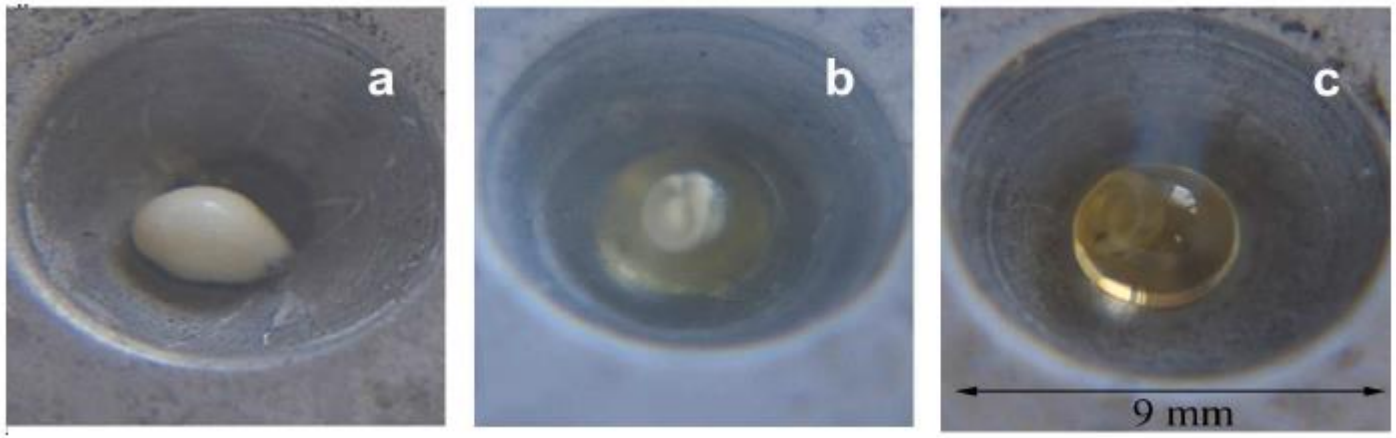
Cryoprotectant solution used for fish embryo vitrification

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)



Common carp

Effect of cryoprotectant concentration on vitrified fish eggs



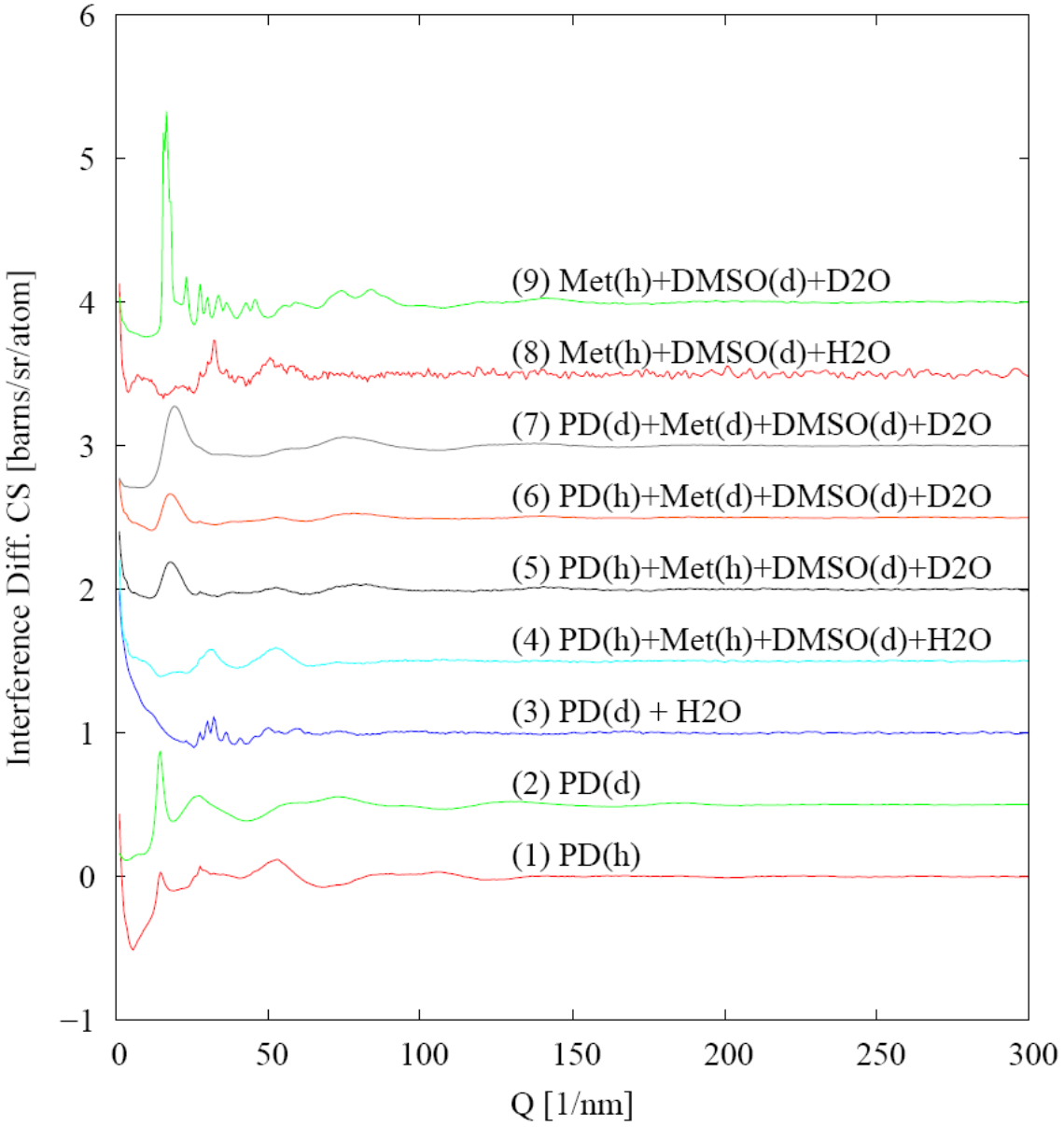
(a) Embryo at 77K dispersed in solution containing 15.4% PD (1,2-propanediol) and 11.4% methanol. Both medium and embryo appear opaque in cooled condition;

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

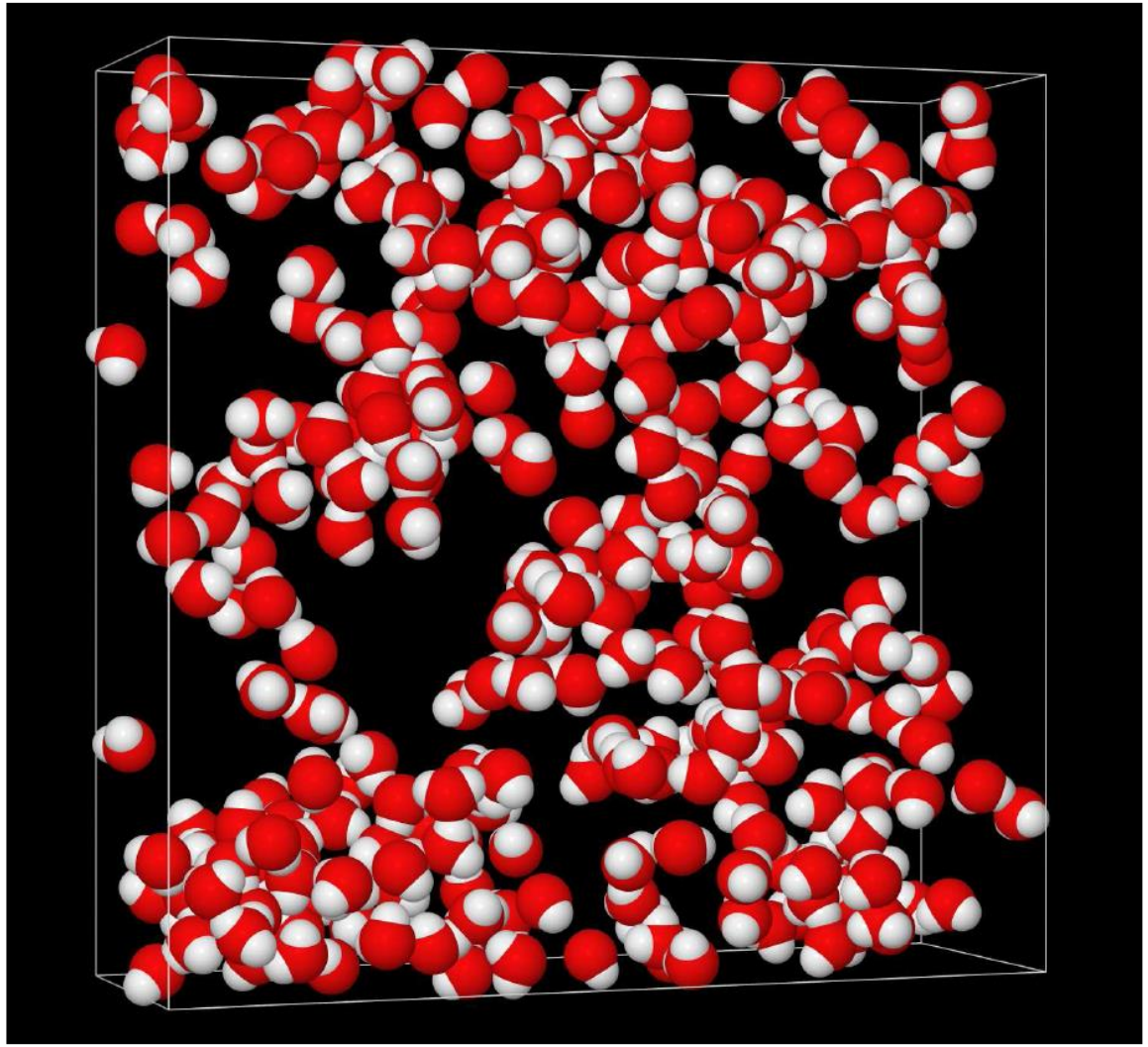
(c) Finally the mixture with ***23% PD, 17% methanol*** and ***20% DMSO*** results in transparency of both medium and embryo.

Neutron diffraction data

All samples have been quench-cooled to 80 K and after that remained at this temperature during collecting neutron scattering data.



View of only the water in the simulation box



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.

OK et. al. *Royal Society Open Science* **3**: 150655 (2016)

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.

ISIS

I would like to thank:

All members of ISIS Sample Environment group; ISIS scientists and engineers 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!

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