High Pressure Capabilities and Challenges at the NIST Center for Neutron Research

3rd ISRD-RCN Workshop (2023)

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The mission of the NIST Center for Neutron Research is to assure the availability of neutron measurement capabilities to meet the needs of U.S. researchers from industry, university and other Government agencies.

Towards this end we…

- **Operate** the NIST Research Reactor cost effectively while assuring the safety of the staff and general public;
- **Develop** neutron measurement techniques, develop new applications of these techniques, and **apply** them to science and engineering problems of national interest;
- **Serve** the needs of researchers from industry, university, and government by operating the research facilities of the Center as a national user facility.

Why Neutrons?

Wavelengths
Probing structures as small as atoms to as large as proteins.

Energies
Commensurate with motions of atoms in solids or liquids, waves in magnetic materials, or vibrations in molecules.

Magnetism
The neutron is “sensitive” to the magnetic spins of both nuclei and electrons.

Capture
Prompt gamma-ray activation used for stoichiometry analysis in minute amounts of material.

Neutrality
Non-destructive and highly penetrating technique allowing measurements under extreme conditions.

Selectivity in scattering
isotopes
Highly sensitive to isotopic variations within a sample.

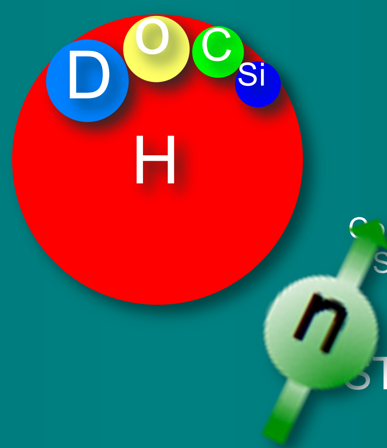
What do Neutrons "See"?

Isotopes have different scattering powers

H : 1 proton
1 electron

Incoherent scatterer
- Does not 'see' neighbor atoms

SPECTROSCOPY



D : 1 proton
1 neutron
1 electron

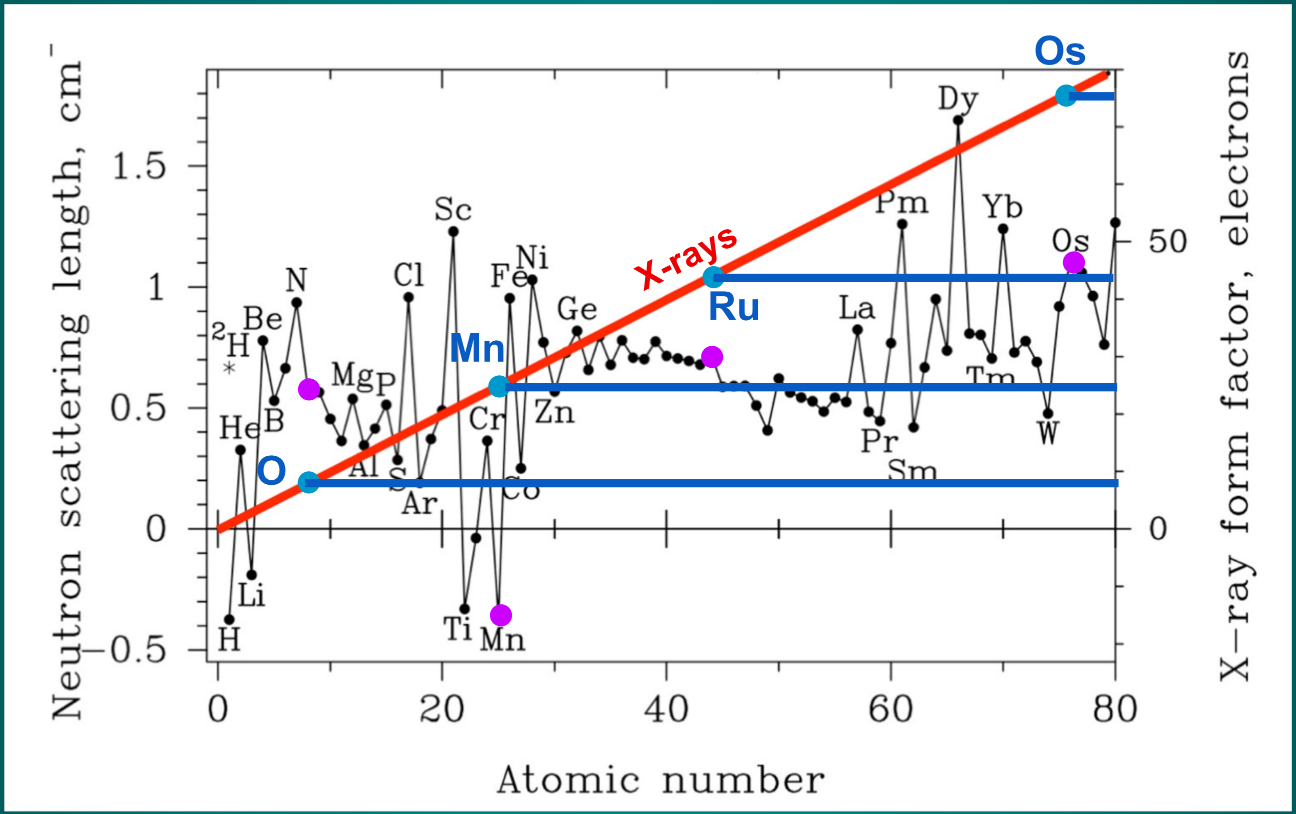
Coherent scatterer
'Sees' neighbor atoms

STRUCTURE

SPECTROSCOPY: "interesting" portions of the sample are hydrogenated and the "uninteresting" portions are deuterated.

Neutron magnetic moment interacts with spins

Neutrons Scattering Power

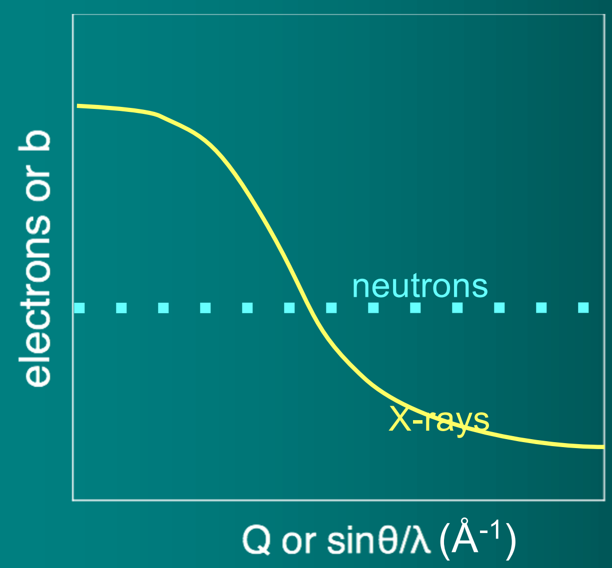


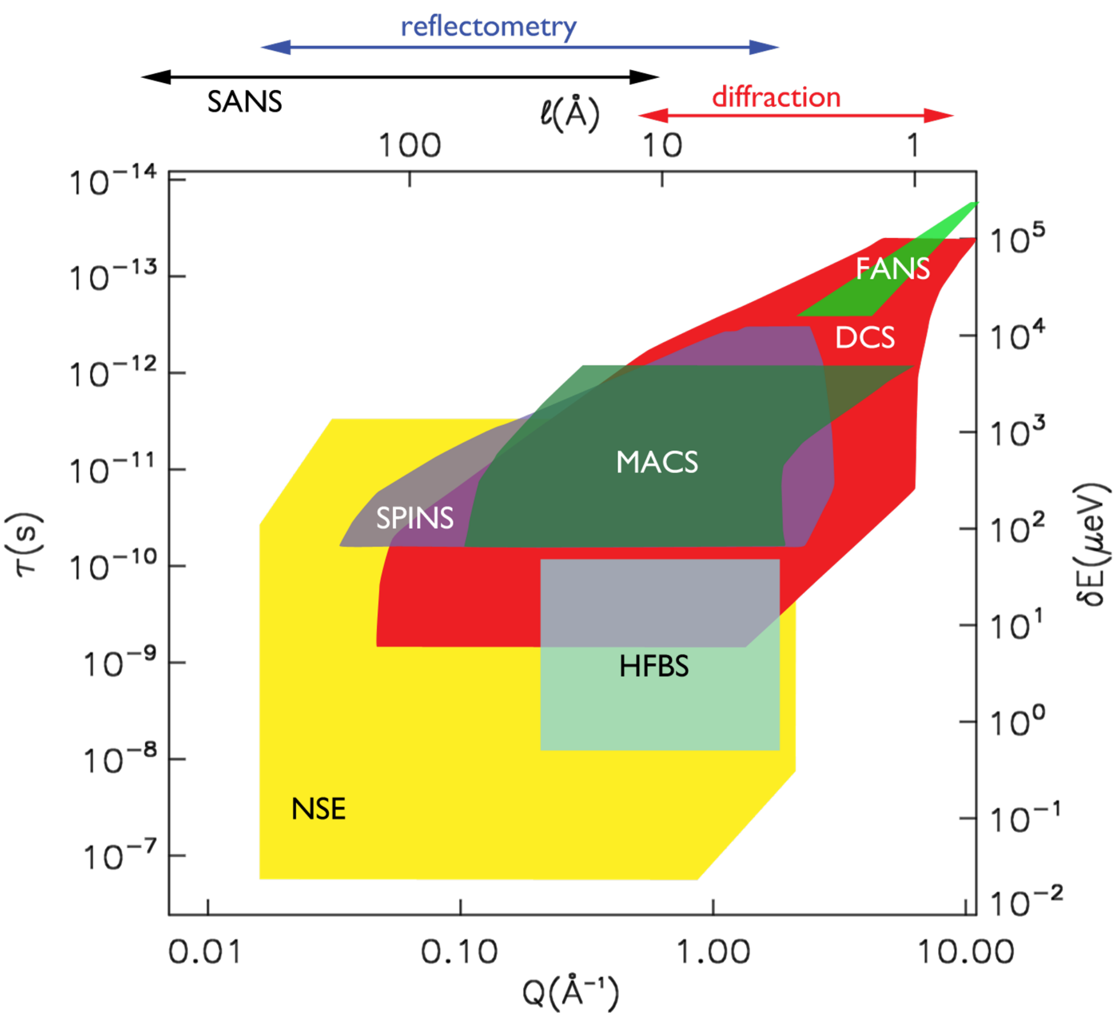
Form Factors

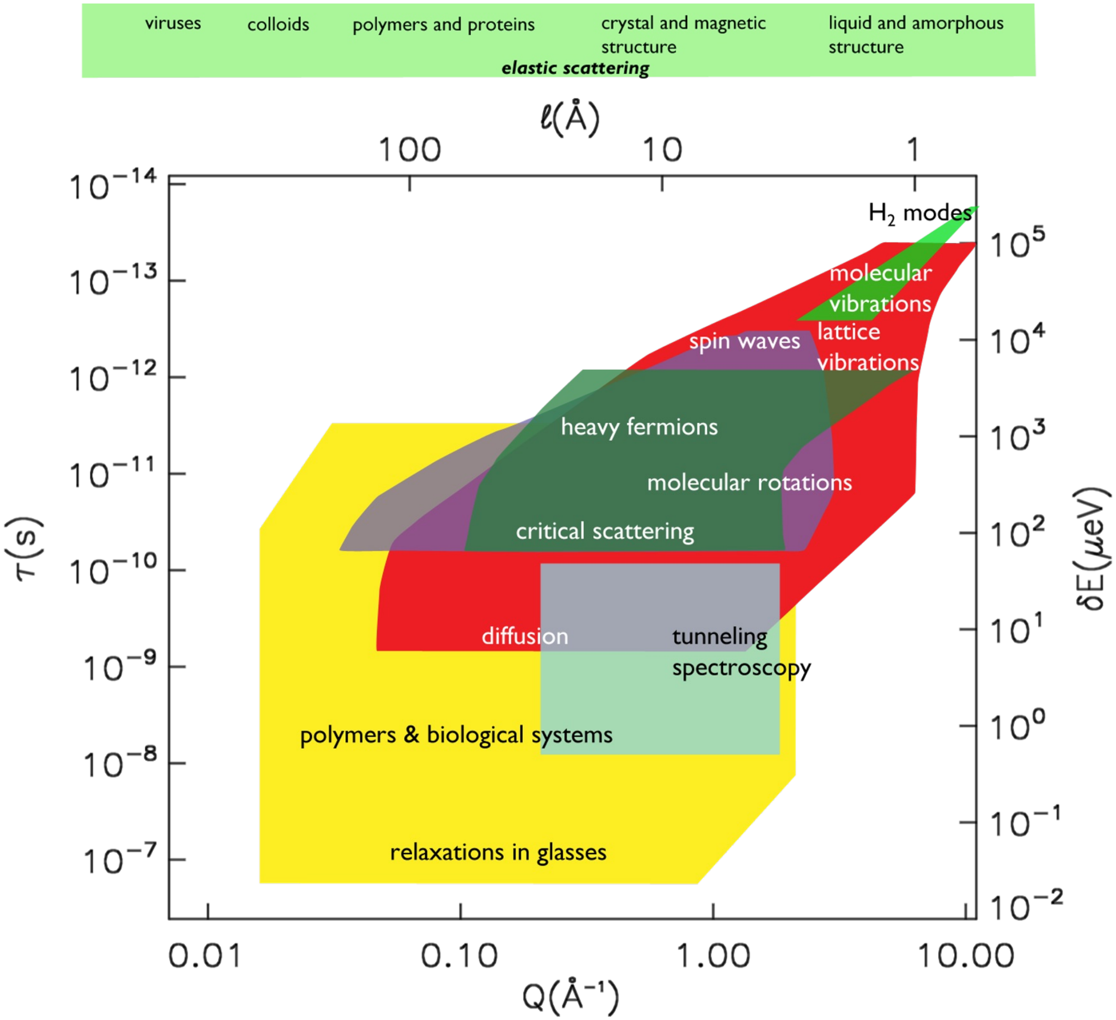
- X-rays (f_i): depends on number of electrons and Q
  – Can have resonance conditions

- Neutrons (b_i): depends on isotope but independent of Q.
  – some isotopes scatter with opposite phase; for these b_i is negative

  – Magnetic scattering is from electrons; $f_M(Q)$ similar to X-rays




reflectometry
SANS
ℓ(Å)
diffraction
100
10
1
10^{-14}
10^{-13}
10^{-12}
10^{-11}
10^{-10}
10^{-9}
10^{-8}
10^{-7}
τ(s)
10^{5}
10^{4}
10^{3}
10^{2}
10^{1}
10^{0}
10^{-1}
10^{-2}
δE(μeV)
FANS
DCS
MACS
SPINS
HFBS
NSE
0.01
0.10
1.00
10.00
Q(Å⁻¹)



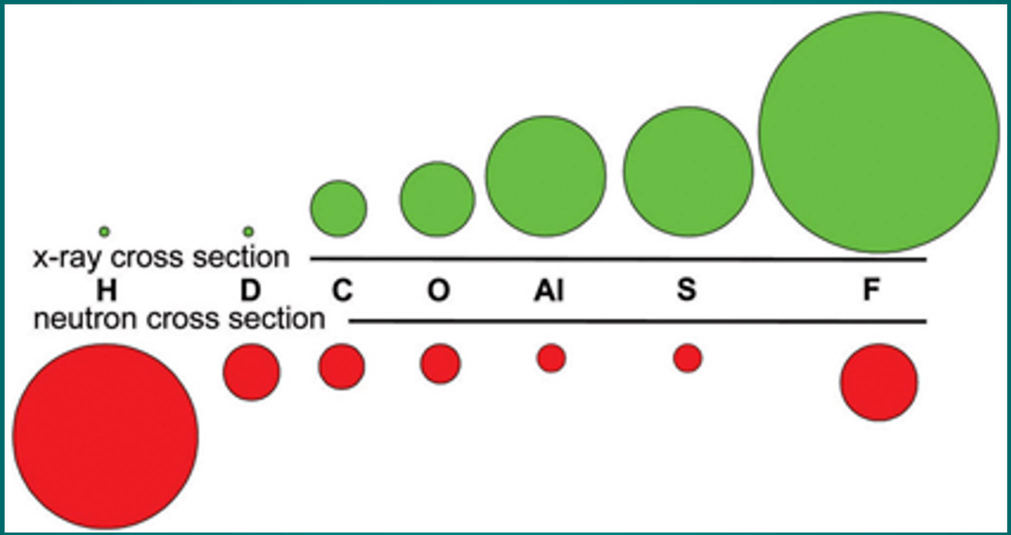
viruses
colloids
polymers and proteins
crystal and magnetic structure
liquid and amorphous structure
elastic scattering
ℓ(Å)
100
10
1
H₂ modes
molecular vibrations
lattice vibrations
spin waves
heavy fermions
molecular rotations
critical scattering
diffusion
tunneling spectroscopy
polymers & biological systems
relaxations in glasses
τ(s)
δE(μeV)
Q(Å⁻¹)
0.01
0.10
1.00
10.00

Considerations

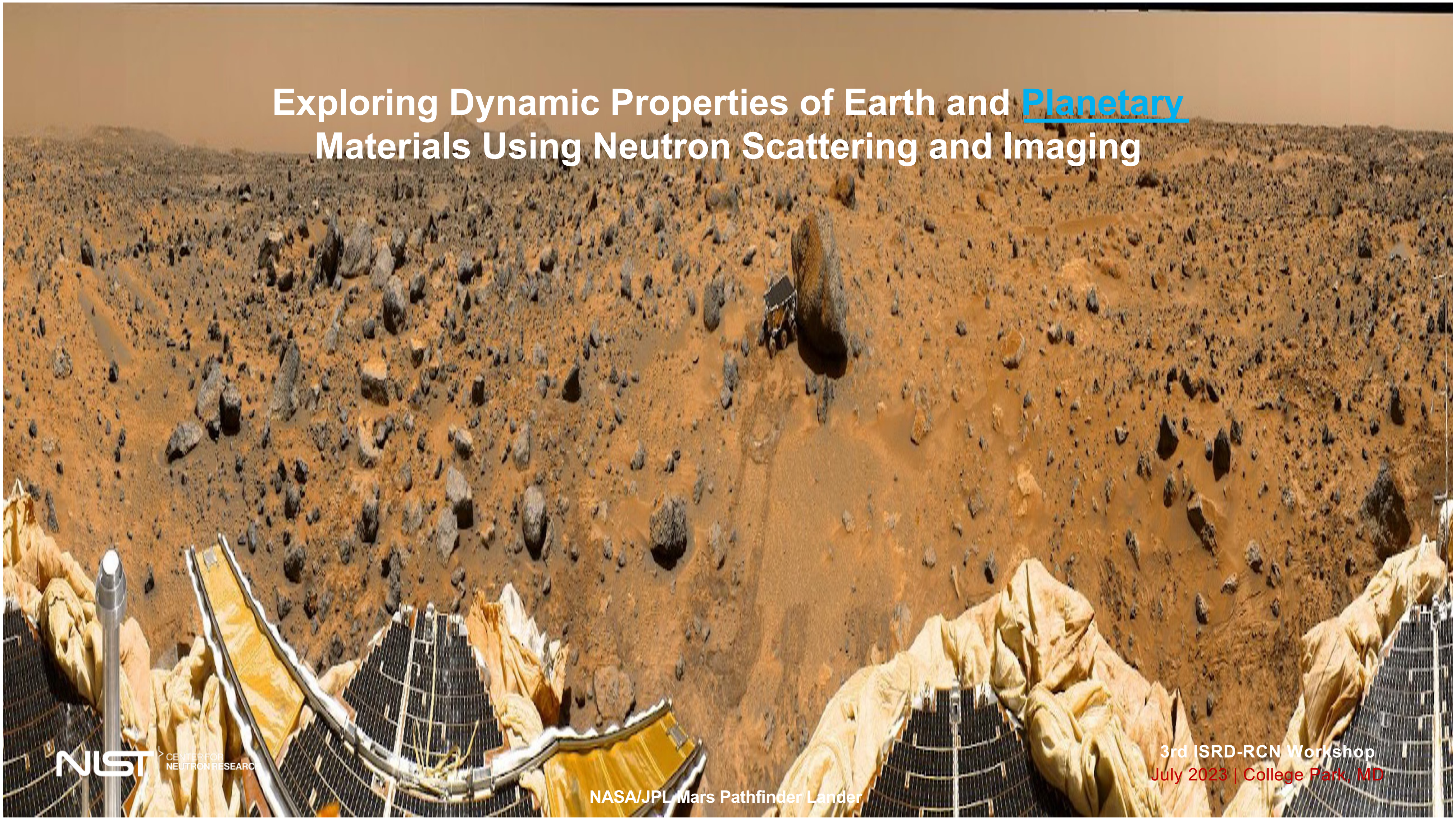
- **X-rays**
  - Limited penetration
  - Small sample sizes
  - Can't easily distinguish neighboring elements
  - hydrogen
  - High flux
  - Imaging/Tomography with very high spatial resolution
  - Can "burn" your sample!

- **Neutrons**
  - Electrically neutral : Penetrating
  - Isotopic substitution
  - Larger sample sizes
  - Some materials adsorb ^{0}n (Li, B, Cd)
  - Long counting time
  - In-Situ samples under extreme conditions of:
    - pressure,
    - temperature,
    - magnetic field,
    - chemical reaction vessels

What do Neutrons See?



Exploring Dynamic Properties of Earth and Planetary Materials Using Neutron Scattering and Imaging



NASA/JPL Mars Pathfinder Lander

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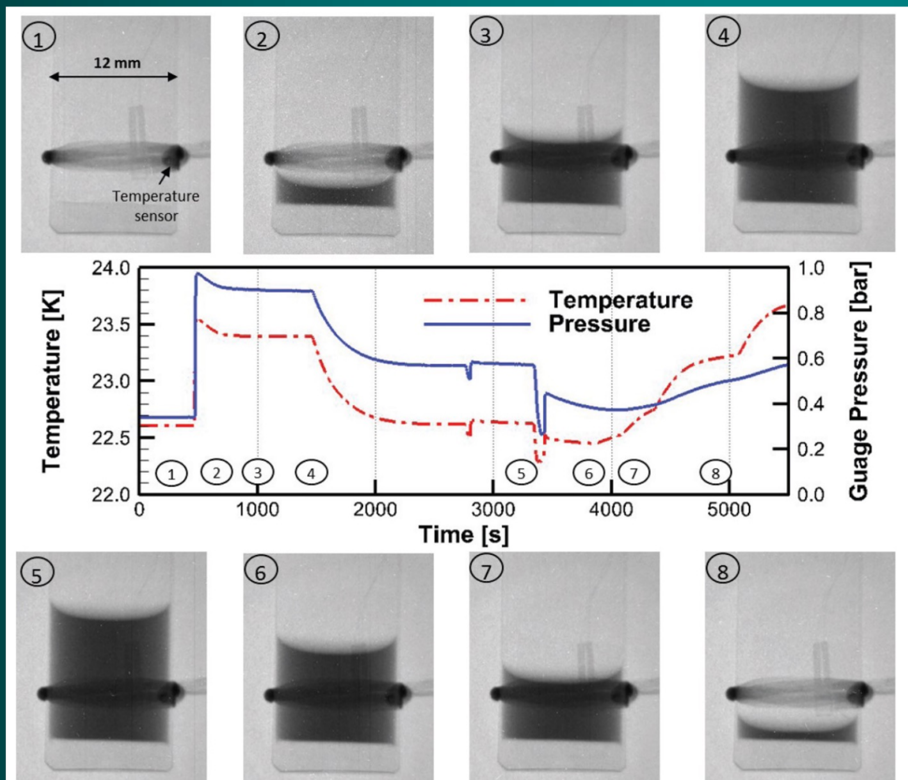
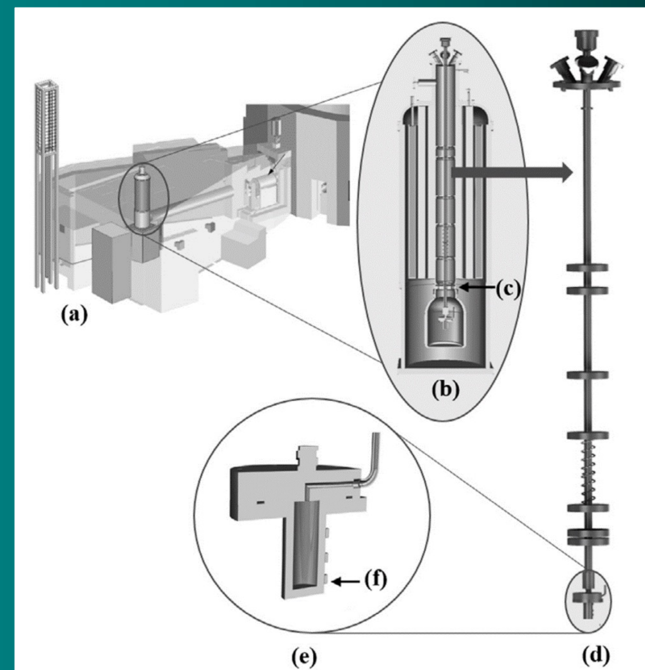
Understanding of Propellant Cryogenic Condensation/Evaporation In Microgravity

Kishan Bellur, et. al. J. Heat Transfer. August 2015, 137(8): 080901.





Set-up at the NIST Neutron Imaging Facility (NIF)

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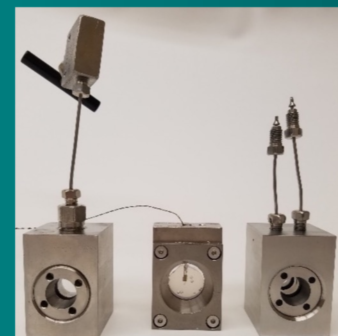
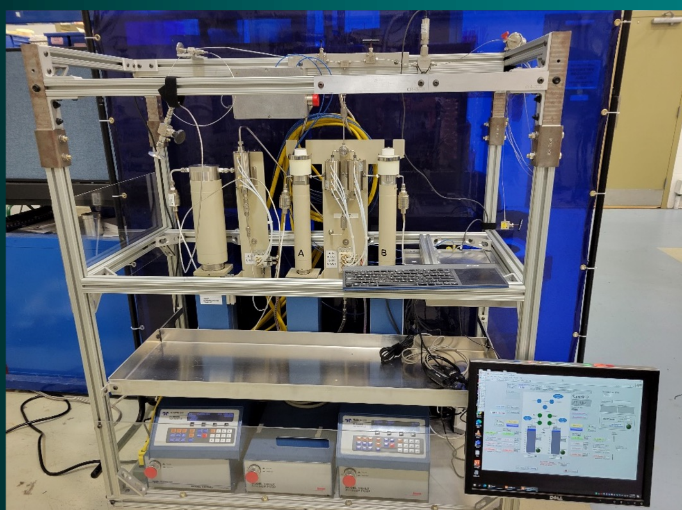
SANS 700 bar Pressure Cells

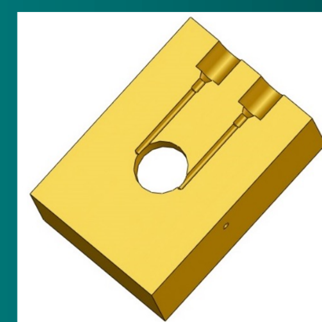
SANS Block Pressure Cells:
Static Pressure ranges:
  Sapphire windows (Pmax: 450 bar)
  Titanium windows (Pmax: 700 bar)
Maximum Volume: 0.7 cc
Adjustable beam path with optical inserts.
Aperture diameter 11 mm



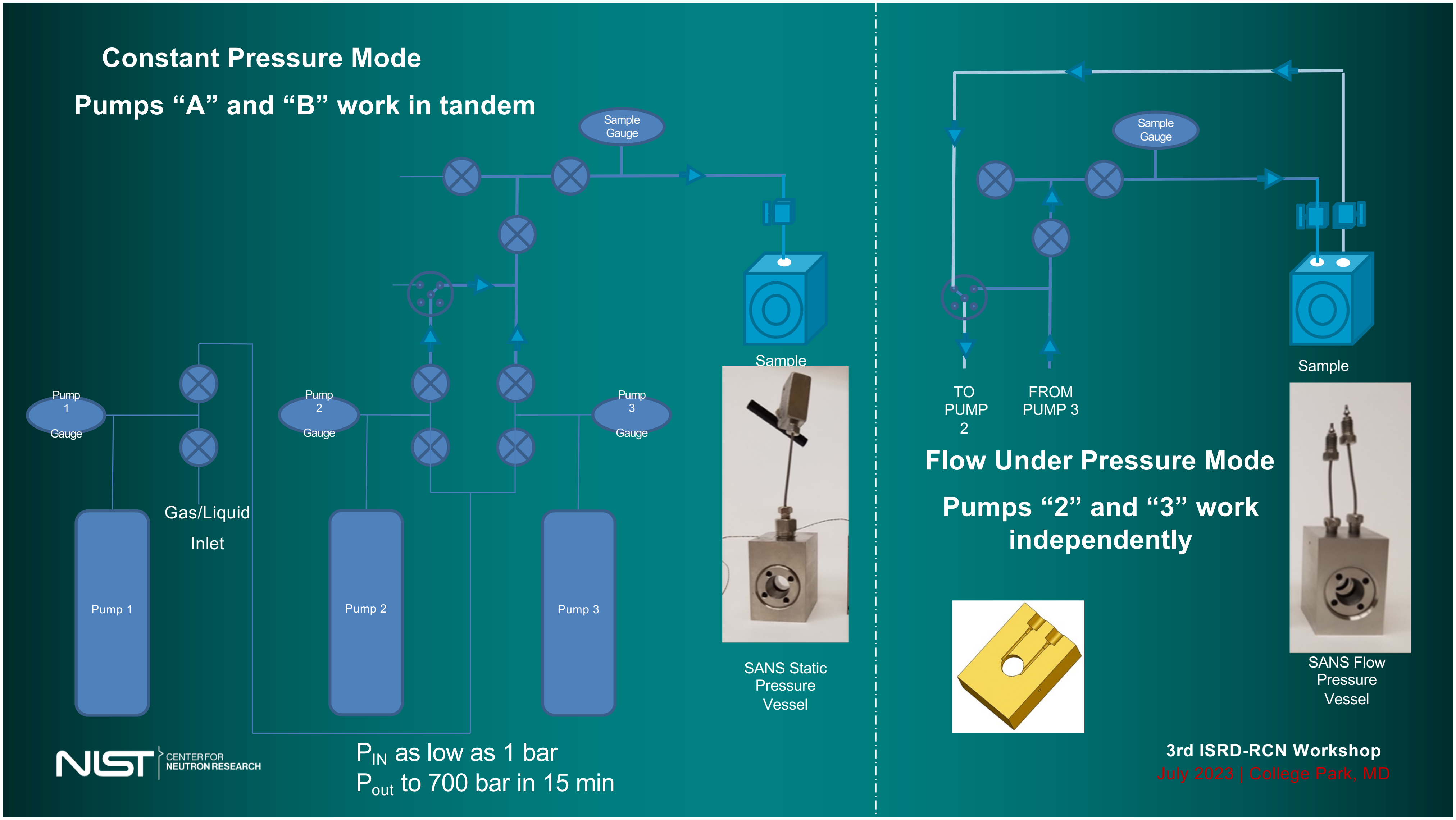


Pump Specifications:

- Two HLf100-Series pumps
- One HLf500-Series pump
- Pneumatic valve manifold
- Pmax = 700 bar
- Flow Range: 10^{-5} - 60 mL/min
- Compatible with most aqueous and organic liquids, corrosive solutions, heated fluids, liquefied gases, and viscous fluids
- Temperature control jacket



Flow cell interior cross section


Constant Pressure Mode
Pumps "A" and "B" work in tandem
Sample Gauge
Sample
Pump 1 Gauge
Pump 2 Gauge
Pump 3 Gauge
Gas/Liquid Inlet
Pump 1
Pump 2
Pump 3
SANS Static Pressure Vessel
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P_IN as low as 1 bar
P_out to 700 bar in 15 min
Sample Gauge
TO PUMP 2
FROM PUMP 3
Sample
Flow Under Pressure Mode
Pumps "2" and "3" work independently
SANS Flow Pressure Vessel
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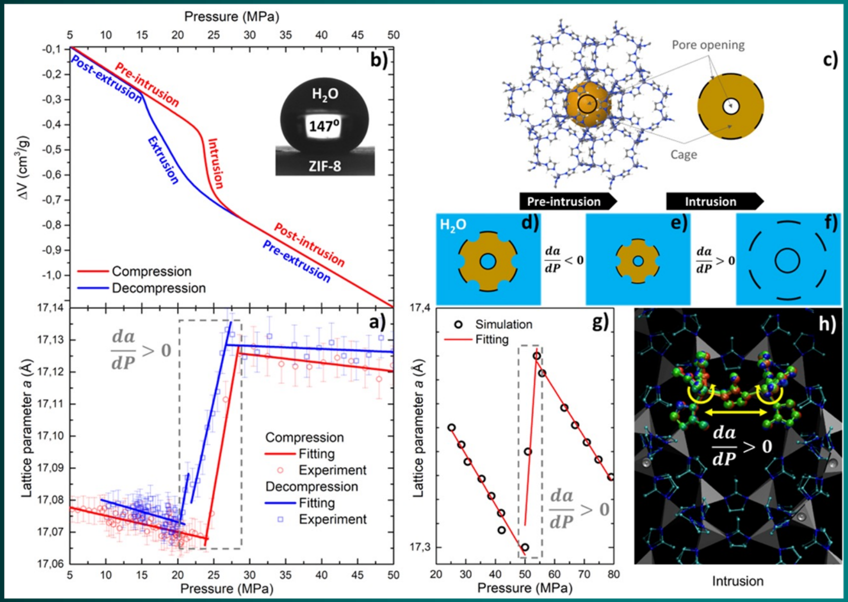
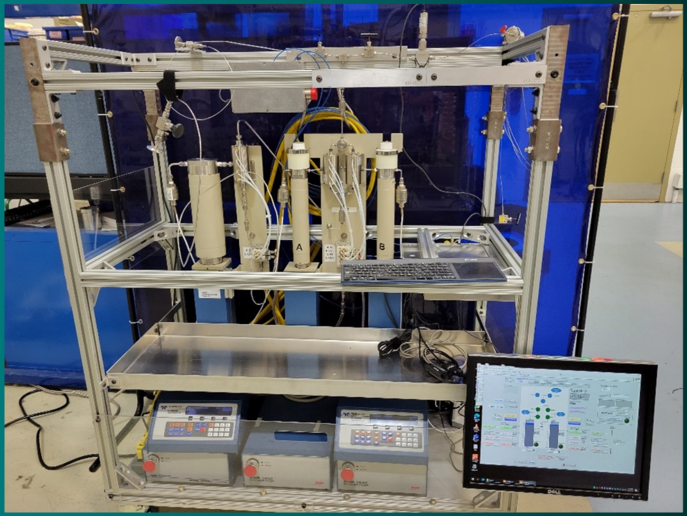
Giant Negative Compressibility by Liquid Intrusion into Superhydrophobic Flexible Nanoporous Frameworks

M. Tortora, et. al. Nano Lett. 2021, 21, 7, 2848–2853





P_{max} (bar)	Press. Medium	T Range (K)	Sample Volume (cm³)	Cell Material	^{0}n Transmission
1,000	Gases: N2, Ar, He Liquids: H2O, non-corrosive	1.5 - 400	2.5	Al 7075-T6	<90%

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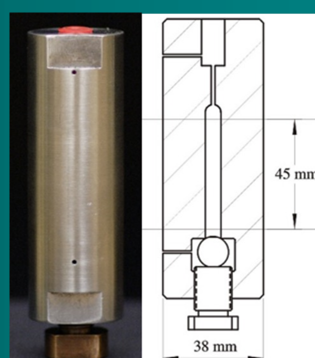
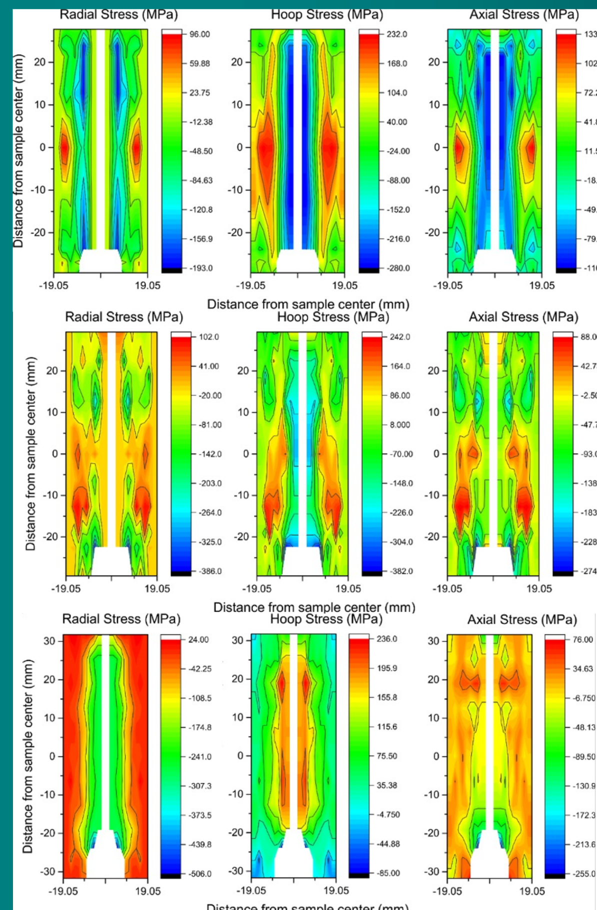
In-situ stress measurement comparison of aluminum pressure vessels under high working pressures

J. Leão, J. Milner. JNR 19 (2017) 65–72

Neutrons
- Penetrating and non-destructive
  - Thick-walled pressure vessels
- Isotopic substitution (H_2/D_2)
- Larger sample sizes compared with anvil cells
- Long counting times

In-Situ samples under extreme conditions





New pressure vessel stress contour maps after autofrettage.

Stress contour maps of a cycled pressure vessel.

Stress contour maps for the new pressurized vessel to an internal pressure of 6 kbar.

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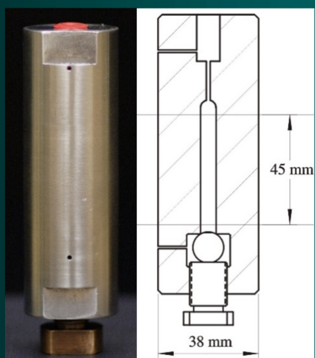
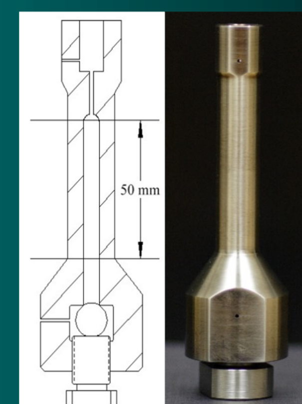
Pressure Cells



HW-02

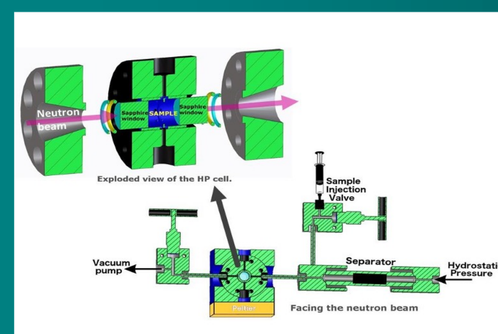
Cell	P_{Max} (GPa)	Pressure Medium	T Range (K)	Sample Volume (cm^3)	Cell Material	Expected Neutron Transmission
LIPSS (for solutions)	0.35		253 - 338	≥2.5	S.S. with Sapphire windows	80%
HW-02	0.65	He, N, Ar	1.5 - 300	1.5	Al 7075-T6	65% @ 2 Å
HW-03	1.0	He, N, Ar	1.5 - 300	1.5	13-8 Mo S.S.	25% @ 2 Å
Clamp Cell	2.5	Fluorinert	1.5 - 300	0.2	Al and CuBe	60% @ 2 Å
SANS T_{Low} Vessel for Powders	0.3	He, N, Ar	100 - 300	2.2	CuBe with Sapphire windows	80%

SANS T_{Low} Vessel



HW-03

Clamp Cell



LIPSS

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Additional Considerations

- Neutrons
  - Electrically neutral : Penetrating
  - Isotopic substitution
  - Larger sample sizes
  - In-Situ samples under extreme conditions of:
    - pressure,
    - temperature,
    - magnetic field,
    - chemical reaction vessels
  - Some materials adsorb ^0n (Li, B, Cd)

- In-situ measurements
  - Magnetism up to 10T
  - 50 mK ≤ T ≤ 2000 K
  - Gas adsorption (CO_2, H_2/D_2, CH_4/CD_4, other organics)
  - Pressure
    - Hydrostatic (Inert gases, CO_2, H_2/D_2, CH_4/CD_4)
    - Uniaxial

Neutron Instruments

Neutron Imaging

Neutron Imaging Facility (NIF)
Cold Neutron Imaging Facility

SANS Instruments

Ultra-Small Angle Neutron Scattering (uSANS)
nSoft 10 m Small Angle Neutron Scattering
NGB 30m Small Angle Neutron Scattering (SANS)
NG7 30m Small Angle Neutron Scattering (SANS)
CHRNS Very Small Angle Neutron Scattering (vSANS)

Reflectometers

Off-Specular Reflectometer (MAGIK)
Polarized Beam Reflectometer (PBR)
Cold Neutron Reflectometer- Horizontal Sample Geometry
CHRNS CANDOR White Beam Reflectometer

Analytical Chemistry

Cold Neutron Prompt-Gamma Neutron Activation Analysis
Radiochemical Neutron Activation Analysis (RNAA)
Instrumental Neutron Activation Analysis (INAA)
Cold Neutron Depth Profiling (CNDP)

Diffractometers

High Resolution Powder Diffractometer
Residual Stress Diffractometer

Spectrometers

BT4 Filter Analyzer Neutron Spectrometer
BT4 Triple Axis Spectrometer
BT7 Triple Axis Spectrometer
CHRNS Multi-Axis Crystal Spectrometer (MACS)
CHRNS Neutron Spin Echo Spectrometer (NSE)
CHRNS High-Flux Backscattering Spectrometer (HFBS)
Disk-Chopper Time-of-Flight Spectrometer (DCS)
Spin-Polarized Triple-axis Spectrometer (SPINS)

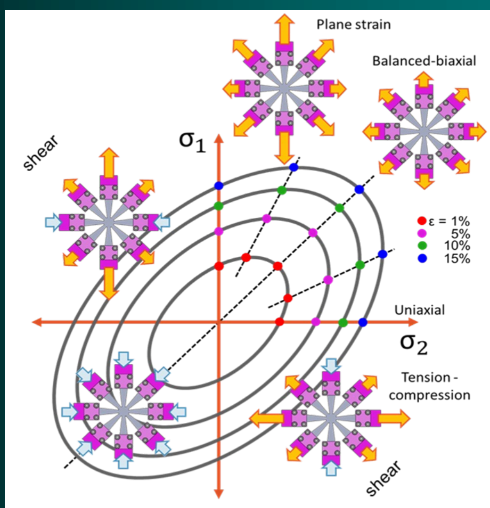
Neutron Physics

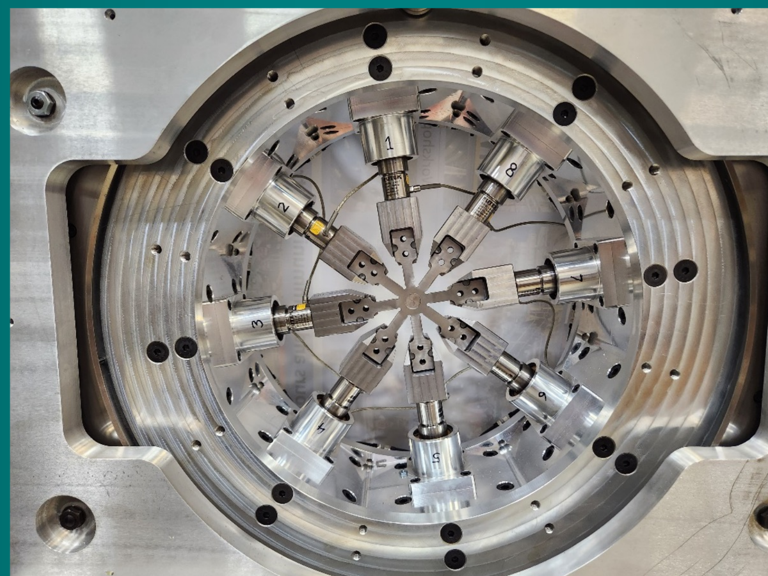
NGC Neutron Physics 0.5 nm
NG6M Neutron Physics
Neutron Interferometer (NIOF)

Exploring Dynamic Properties of Earth and Planetary Materials Using Neutron Scattering and Imaging

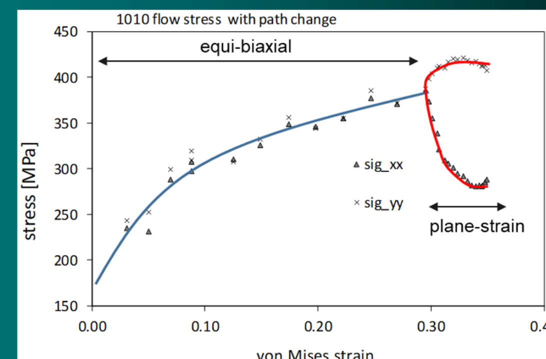
Octo-strain

Able to define, and switch between, various strain paths, such as: uniaxial, plain strain, balanced-biaxial and tension-compression.





Eight individually controlled actuators capable of 40 kN each.



Stresses along X and Y obtained by neutron diffraction using Octo-strain before and after a path change from equi-biaxial to plane-strain deformation.

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Thank you!

Questions?

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