# New Cell for Studying Porosity Under Mechanical and Chemical Stress

**Author:** Yeping Ji
**Co-authors:** Klaus Regenauer-Lieb, Tomasz Blach, Liliana de Campo, Andrzej Radlinski, Phung Nhu Hao Vu

Minerals and Energy Resources Engineering, UNSW Sydney

## Background and Motivation

• Most of the fuel in the unconventional reservoirs is stored in nano and micro pores, SANS and USANS are ideally suited to in-situ study sample rocks and the effects pressurized gasses and liquids have on them.

• Neutrons are the only way to measure inaccessible porosity using contrast matching techniques.

• We have developed a sample environment that allows directional and omnidirectional stress up to 1000 bar to be applied to the rock using hydraulic and pneumatic means.

• By carefully selecting the isotopic mixture of elements, it is possible to measure the extend of the working fluid penetration of pores, kinetics of diffusion and effects on closed porosity that the pressure has.

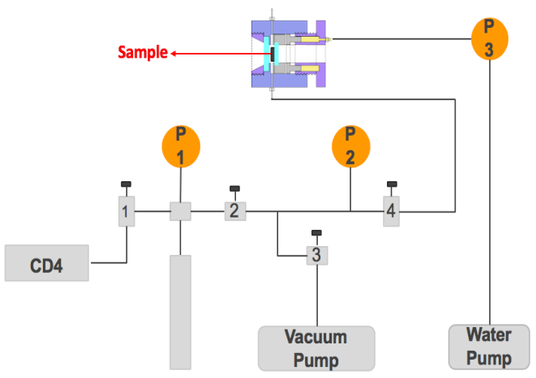
• The aim is to have a cell that can be moved between different instruments to allow SANS, USANS and Computer Tomography to be performed on our samples.

## Assumption

• 2-phase approximation for simple materials, 3-phase approximation for complex materials.

• Pores are spherical and under uniaxial stress become elliptical with the radius parallel to the stress direction getting smaller.

## Methodology

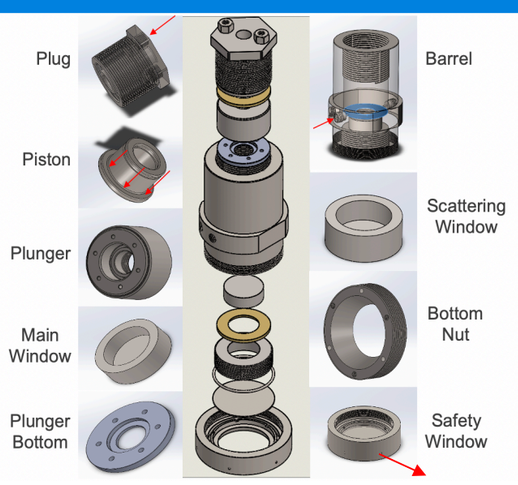



• Water pump to create the hydrostatic pressure

• CD4 as a matching fluid to make the open pores invisible

• The experiment conducted at vacuum condition firstly then at ZAC pressure

## High Pressure Cell




• The uniaxial stress in the direction parallel to the neutron flight path is controlled by a water based hydraulic system.

• The second system is the direct fluid port which allows the sample to be flooded by pressurized gas or fluid and is supplied by a fluid-controlled apparatus.

## Multiple Scattering

• Multiple scattering (MS) occurs when a neutron is scattered by more than one scattering point. This process degrades the data and should be avoided (Melnichenko 2016).

• The effect of MS becomes significant by increasing sample thickness and concentration, scattering contrast, and neutron wavelength (Jensen and Barker 2018).

• MS affects measurement accuracy in the micropore structures and results in an incorrect data estimation on the absolute scale.

• The higher transmission (0.5<Tsas<0.9), optimized sample thickness and wavelength are preferable.

## Future Study

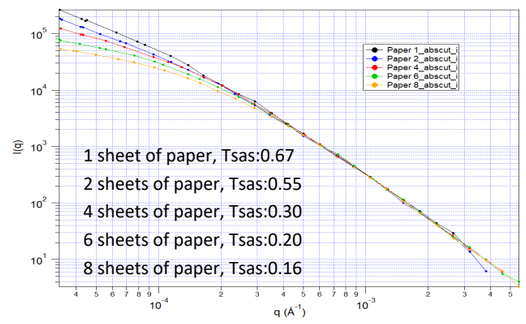
• Perform tensile strength thermal characterization for cell materials to optimize the cell window transmission (such as Alon).

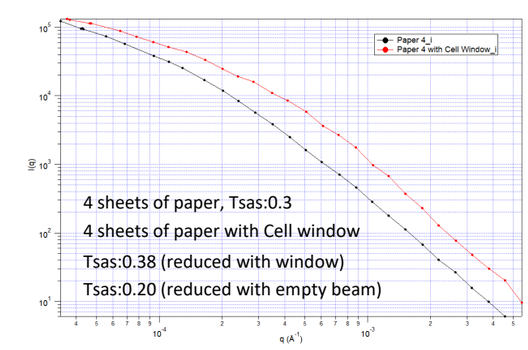
• Build a cell that can operate at 1000bar of gas pressure, 1000bar of stress pressure, and 500$^{o}$C+ for further applied thermal load.

## Result: Multiple Scattering

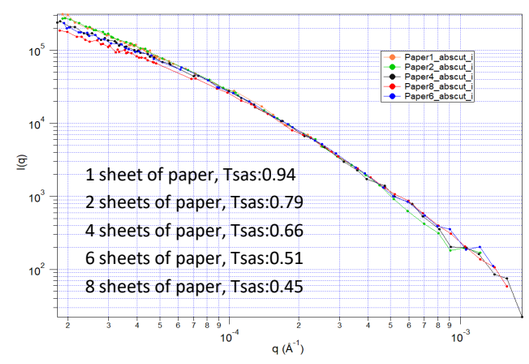
### Paper Experiment

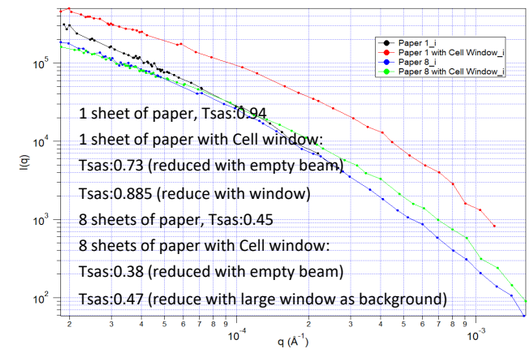
**Long Wavelength: 4.74 Å**



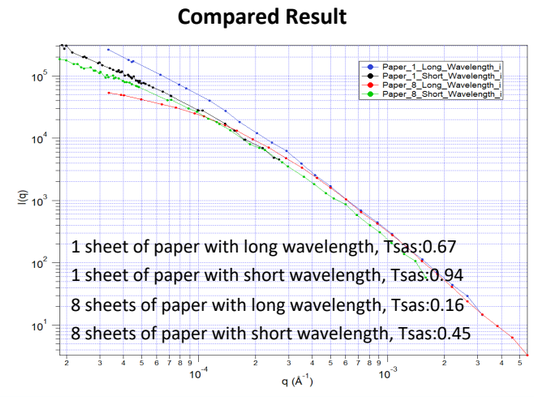




**Short Wavelength: 2.37 Å**



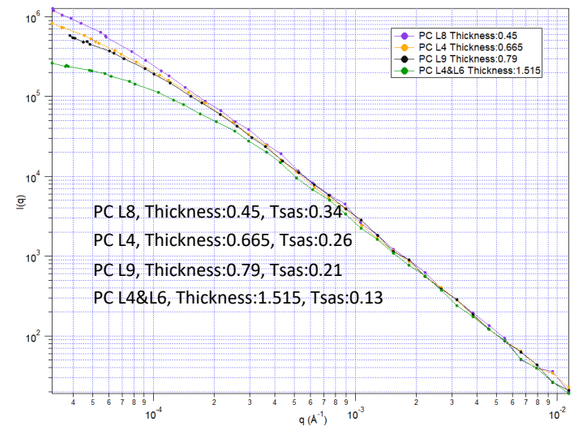



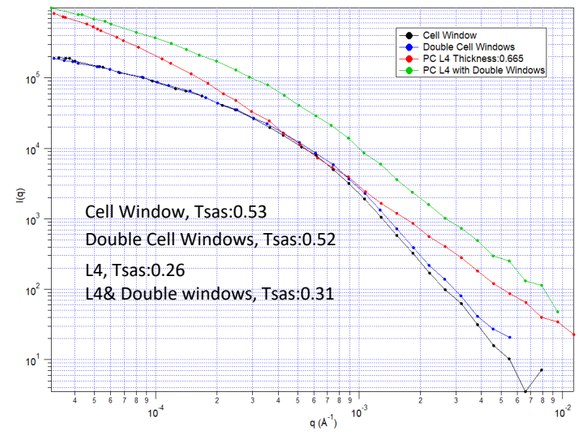

**Compared Result**



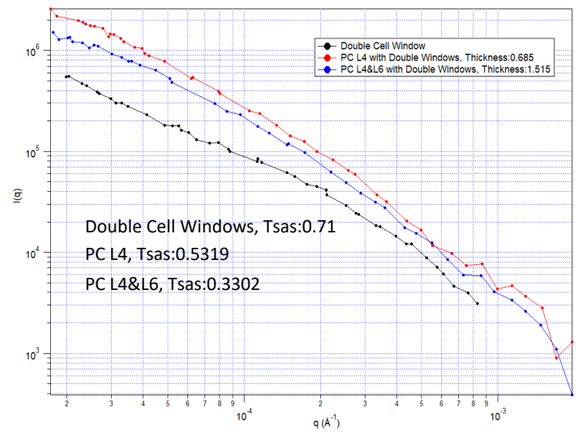

### Rock Sample Experiment

**Long Wavelength: 4.74 Å**

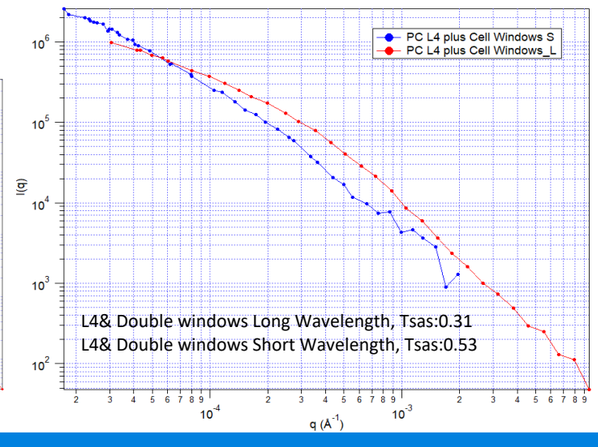



**Short Wavelength: 2.37 Å**




**Compared Result**




## Discussion and Conclusions

• The multiple scattering occurs significantly by increasing sample thickness and the neutron wavelength.

• The intensity value at the low Q region would be flattened by increasing sample thickness while remaining almost constant at the high Q region.

• The shorter neutron wavelength applied results in a preferable transmission value.

• A higher transmission value is preferable (0.5<Tsas<0.9) to minimize the multiple scattering effects.

• The transmission for cell windows with a long wavelength is around 0.5, while with a short wavelength is around 0.7.

## Reference

Blach, T., et al. (2020). "Pore anisotropy in unconventional hydrocarbon source rocks: A small-angle neutron scattering (SANS) study on the Arthur Creek Formation, Georgina Basin, Australia." International Journal of Coal Geology: 103495.

Cherfallot, G. l. J., et al. (2020). "Probing Multiscale Structure of Mineral and Nanoporous Kerogen Phase in Organic-Rich Source Rocks: Quantitative Comparison of Small-Angle X-ray and Neutron Scattering." Energy & Fuels **34**(8): 9339-9354.

Jensen, G. V. and J. G. Barker (2018). "Effects of multiple scattering encountered for various small-angle scattering model functions." Journal of applied crystallography **51**(5): 1455-1466.

Melnichenko, Y. B. (2016). "Small-Angle Scattering from Con ned and Interfacial Fluids."

Radlinski, A. P. (2006). "Small-angle neutron scattering and the microstructure of rocks." Reviews in Mineralogy and Geochemistry **63**(1): 363-397.