Commissioning the 1st High Pressure Cell @ the SNS

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Neutron Sciences

MOTIVE

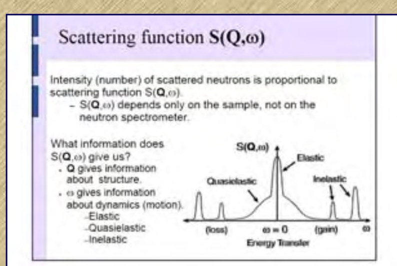
In 2007 a Lab Directed Research & Development (LDRD) project was proposed requiring a sample cell that could withstand temperatures of 450°C and pressures of 1500 bar. The proposed cell will be used in studies, such as the dynamics of fluids in nanopores at high temperatures and high pressures, this summer at the Spallation Neutron Source.

Why Study Fluids in Confined Geometries

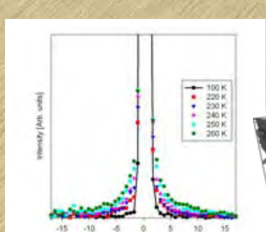
- They are very common in nature and engineering environments (chemical, oil and gas, pharmaceutical industries, catalysis).
- Their properties are very different from bulk counterparts (due to finite size effects, varying dimensionality, surface forces).
- Dynamics of fluids are affected dramatically by confinement (e.g. mobility of confined water – pore size, shape, distribution, connectivity.
- Interrogation of molecular mobility and transport is key to understanding the initiation and sustainability of reactions.
- Aqueous solutions form due to interaction with the matrix
- Flow, diffusion and selective adsorption of fluids are important in natural systems (e.g. oil and gas migration, soils and groundwater, geological CO_2 sequestration, waste disposal).

SCIENCE

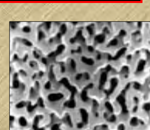
The best way to carry out dynamic studies of diffusion and relaxation processes is with QuasiElastic Neutron Scattering (QENS).



Typical temperature dependence of QENS data: The width of the QENS signal tends to grow as the temperature is increased: shorter relaxation times (faster diffusion). The relative intensity of the QENS signal tends to increase as the temperature is increased: more molecules become mobile on the time scale of the measurement.

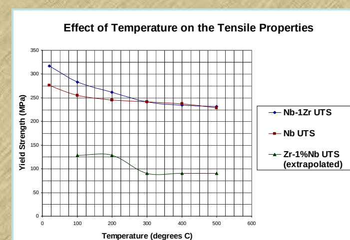


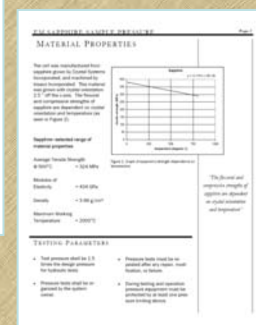
Example: confined aqueous solution: $CaCl_2$ – H_2O in Vycor Pore Glass



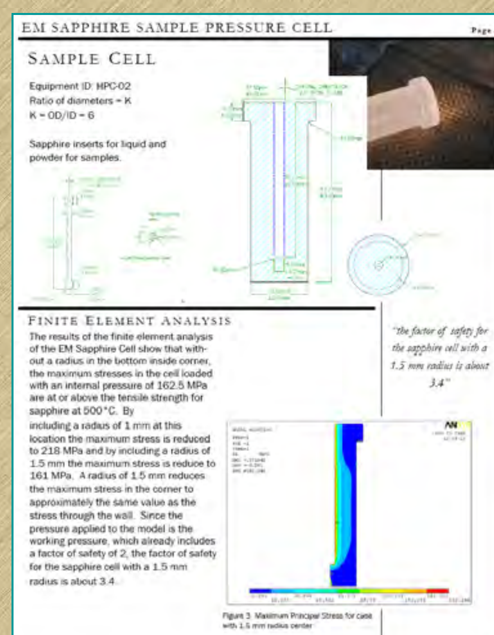
MATERIALS

Initially it was suggested the material best suited for this high pressure application would be a Niobium Zirconium alloy (Nb-1Zr), or a Zirconium Niobium alloy (Zr-1%Nb). Each of these materials were dually suited for applications using high pressure and neutrons at a spectrometer. Further research of these materials yielded data showing a decreased yield strength at 500°C. Low temperature high pressure testing at HFIR presented us with a unique solution. A Sapphire cell in use at their facility presented excellent neutron transparency (2.5° off the c-axis) as well as strength at high temperatures.





CELL



EM SAPPHIRE SAMPLE PRESSURE CELL — Page 3

SAMPLE CELL

Equipment ID: HPC-02
Ratio of diameters = K
K = OD/ID = 6

Sapphire inserts for liquid and powder for samples.

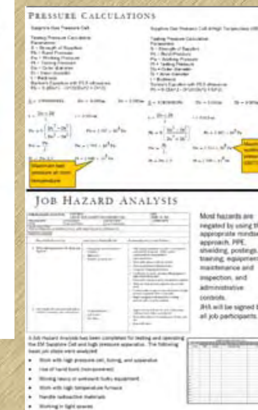
FINITE ELEMENT ANALYSIS

The results of the finite element analysis of the EM Sapphire Cell show that without a radius in the bottom inside corner, the maximum stresses in the cell loaded with an internal pressure of 162.5 MPa are at or above the tensile strength for sapphire at 500°C. By including a radius of 1 mm at this location the maximum stress is reduced to 218 MPa and by including a radius of 1.5 mm the maximum stress is reduce to 161 MPa. A radius of 1.5 mm reduces the maximum stress in the corner to approximately the same value as the stress through the wall. Since the pressure applied to the model is the working pressure, which already includes a factor of safety of 2, the factor of safety for the sapphire cell with a 1.5 mm radius is about 3.4.

"the factor of safety for the sapphire cell with a 1.5 mm radius is about 3.4"

Figure 3. Maximum Principal Stress for case with 1.5 mm radius corner

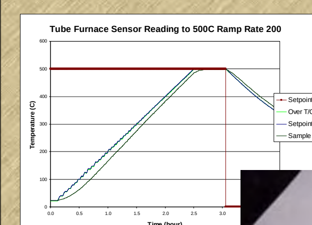
SAFETY



TESTING

Testing Status: In Progress

To date, one cell has failed due to a flaw in the sealing scheme, a temperature gradient has been measured perpendicular to the sample (no gradient across the sample), and a pressure rig has been identified and is being modified for liquid pressure applications.



CONTRIBUTORS

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