The development of a sapphire cell for neutron scattering experiments at high temperature and high pressure

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Abstract. A high pressure, high temperature sapphire cell has been designed to provide users at the Spallation Neutron Source with the ability to perform in-situ supercritical bulk water studies. The design criteria for the cell required the achievement of a maximum pressure of 150 MPa at the maximum temperature of 500˚C. Other conceptual constraints, such as a 360˚ viewing angle and top loading integration further complicated and limited available cell models. The sapphire cell was tested and modified due to pre-mature failure. Due to convenient geometry and ease of operation, interest in the cell for more moderate operating requirements has increased.

1. Introduction
High temperature and high strength characteristics make sapphire a perfect candidate for neutron scattering experiments where the material to be observed exhibits interesting behavior outside of typical experiment parameters (4 to 300 K, 0.1 to 5 MPa). One of these moderately extreme environment experiments was proposed for a high energy resolution neutron backscattering spectrometer at which Quasi-Elastic Neutron Scattering (QENS) can be performed. QENS probes diffusion and relaxation processes, and a typical requirement is an extended range of available temperatures. One long standing application of QENS has been the study of bulk and confined water [1].

Supercritical bulk water has generated interest for applications such as cleaning and dissolving pollutants and other non-polar substances like oils, as well as, nuclear reactor applications [1]. Both high pressure and high temperature are required to reach the supercritical phase. The thermodynamics of confined liquids, liquid confined in nano sized pores, are also of interest in this regime.

The original operational requirements needed to support these studies were a maximum temperature of 500˚C (773 K) and pressures to 400 MPa. Practical consideration required modification of these goals to 500˚C and 153 MPa. This article describes the design, fabrication, and test process of a high pressure and high temperature sapphire sample cell.

2. Cell Material Selection
The sample cell was manufactured from sapphire grown by Crystal Systems Incorporated [2], and machined by Insaco Incorporated [3]. Sapphire is often used in high pressure applications because of its strength, transparency to visible light, and low incoherent neutron scattering and absorption cross sections [4]. The relevant properties of sapphire, which where extrapolated from indexed single crystal data, are given in Table I. To further minimize reflections in the diffraction plane, the sapphire
cell was machined in such a way that its vertical axis is oriented 2.5° off the c-axis of the parent material [4].

| Material Properties                              | Value   | Reference |
|--------------------------------------------------|---------|-----------|
| Average Tensile Strength @500°C                  | 324 MPa | [5]       |
| Modulus of Elasticity                            | 344 GPa | [2]       |
| Density                                          | 3.98 g/cm³ | [2]    |
| Maximum Working Temperature                      | 2040°C  | [2]       |

Table I. Material properties for single crystal sapphire.

As designed, the sample cell has a total length of 93.75 mm, a minimum wall thickness of 12.5 mm, and a bore that is 5 mm wide by 81.25 mm tall as shown in figure 1. To minimize pressure build up within the cell the sample bore was modified from a design with sharp corners to a design with 1.5 degree radius corners. To optimize the amount of sample in the beam, sapphire inserts were designed. These inserts provide annular gap sizes of 0.1 mm and 1 mm. Gap sizes, or sample thicknesses, will vary based on the strength of the neutron scattering from the sample and are required in order to minimize multiple scattering from the sample.

![Figure 1. Sapphire cell dimensions](image1)

![Figure 2. 3-D Assembly of sapphire cell, stainless steel fittings, and Swagelok [6] sample stick adapter](image2)
3. Pressure Requirements for Cell

The design of the cell body is principally a flanged tube of sapphire ($\alpha - Al_2O_3$). The stainless steel fittings are designed in two parts. One part is a cradle for the cell flange and mates with the second part which acts as a cap for the sapphire cell. The assembly, as shown in figure 2, uses a metal c-ring as a sealing surface, and a HiP [7] high pressure fitting to connect the assembly to high pressure tubing and apparatus. Fully assembled the sapphire cell is designed to be operated at a pressure of 153 MPa at 500°C. Safety considerations at the SNS facility require a testing protocol at 1.5 times the maximum allowable working pressure (MAWP) at room temperature. The cell was thus designed for 270 MPa.

The design of the sapphire cell proceeded in two stages. First, a finite element analysis was performed to verify the theoretical calculations and pinpoint any weaknesses in the cell structure when under the maximum anticipated pressure. The results of the finite element analysis show (Figure 3) that without a radius of curvature on the bottom inside corner of the cell bore, the maximum stresses in the cell loaded with an internal pressure of 162.5 MPa are at or above the yield 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 reduced 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.

![Finite Element Analysis](image)

**Figure 3.** Finite Element Analysis shows Maximum Principal Stress distribution in the cell

Secondly, an FEA modified Barlow’s Equation (1) was used to calculate the bursting pressure of the sapphire cell. The working pressure is typically defined as $\frac{1}{2}$ the bursting pressure in systems where a safety factor of 2 is required [8].
where \( P_b \) is defined as the bursting pressure, \( S \) is the tensile strength of the sapphire, and \( d_o \) and \( d_i \) are the outer and inner cell diameter, respectively. With \( S = 324 \text{ MPa} \), \( d_o = 0.030 \text{ mm} \), and \( d_i = 0.005 \text{ mm} \), the MAWP for the sapphire cell is 153 MPa.

4. Cell Verification and Testing

Testing parameters were established to ensure safe work practices. As mentioned previously, test pressure will be 1.5 times the design pressure. In each instance pressure tests will be organized by the system owner. Also, pressure tests must be repeated after any repair, modification, or failure. Most importantly, during testing and operation, pressure equipment must be protected by at least one pressure limiting device.

The sapphire cell was inspected and tested upon delivery. The original cell failed unexpectedly after 10 MPa of pressure was applied. A design investigation was conducted and possible cell modifications were suggested. As a result, the cell body was returned to Insaco, Inc. [3] for exterior polishing (original cell had rough finish). During that time, interest in the use of the cell at lower pressures (1 to 5 MPa) increased. After polishing, the cell was tested and verified for operation at pressures up to 10 MPa and temperatures up to 80°C. The cell successfully performed at these conditions. After experiments at these conditions, the cell will be tested to allow operation at its maximum working pressure. Plans are also in place to test one or more cell bodies to failure. These tests will provide necessary feedback for future gas pressure cell designs.

5. Conclusions

A sapphire cell that maintains pressure up to 10 MPa at 80°C was fabricated at SNS to perform in-situ supercritical bulk water studies. A similar sapphire cell was fabricated at HFIR for operation at 35MPa and 12K [4]. Having both cells at ORNL provides a full temperature range to users wishing to perform QENS experiments. Two recent user experiments at the SNS facility have provided insight into background issues associated with the thick walled sapphire cell design. Feedback of this nature assists cell designers in making modifications that improve the cell’s strength, durability, and neutron transparency.

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