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# High-pressure cell for neutron diffraction with *in situ* pressure control at cryogenic temperatures

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Pressure generation at cryogenic temperatures presents a problem for a wide array of experimental techniques, particularly neutron studies due to the volume of sample required. We present a novel, compact pressure cell with a large sample volume in which load is generated by a bellow. Using a supply of helium gas up to a pressure of 350 bar, a load of up to 78 kN is generated with leak-free operation. In addition, special fiber ports added to the cryogenic center stick allow for *in situ* pressure determination using the ruby pressure standard. Mechanical stability was assessed using finite element analysis and the dimensions of the cell have been optimized for use with standard cryogenic equipment. Load testing and on-line experiments using NaCl and $BiNiO_3$ have been done at the WISH instrument of the ISIS pulsed neutron source to verify performance. © *2014 AIP Publishing LLC*. [http://dx.doi.org/10.1063/1.4870061]

## I. INTRODUCTION

The study of materials using neutron diffraction is an area of research in high demand, offering the ability to determine atomic and magnetic structures of materials. Where pressure studies are required, neutron diffraction remains more limited in range than for x-ray studies.[1–3] As a result, it is important to develop instrumentation to reach such conditions.

Instrumentation used in this area falls into categories including hydrostatic cells,[4,5] large volume/multi-anvil presses, and opposed anvil cells (diamond anvil cells,[6] sapphire anvil cells,[7–9] Paris-Edinburgh press[10]). These are covered in detail in several references.[11,12] In each case, there are limitations on the effective range in pressure-temperature space that can be reached. Systems with a large sample volume (i.e. multi-anvil or toroidal anvil presses) also have a large thermal load, limiting the low temperature range, and stability. Opposed diamond anvil cells (DACs) are compact enough that their temperature can be more easily regulated, and the load applied to the anvils can be controlled with a membrane. However, they have sample volumes significantly less than 1 $mm^3$, resulting in long count times and difficulty distinguishing sample from background signal. Hydrostatic cells (Gas & Piston-in-cylinder) are limited to the burst pressure of the container, for beryllium copper alloys this is generally below 3 GPa for double walled cells[5,13] and 1.5 GPa for single walled cells.[12,14] Beryllium-copper (or beryllium bronze) is frequently used as a cell material in neutron studies due to it being non-magnetic, and having a high tensile strength. Currently, Paris-Edinburgh presses, hydrostatic cells, and DACs form the primary instrumentation at central facilities, with the Institute Laue-Langevin and the ISIS neutron facility both reporting pressure cells of these types. The characteristic range of capabilities at central neutron facilities are shown in Table I.

Those most commonly used for cryogenic studies are piston-cylinder and opposed-anvil types, as the small size allows use with a cryostat or dilution refrigerator. However, the sample volumes required in neutron experimentation are orders of magnitude larger than x-ray work making use of large volume devices, such as the Paris-Edinburgh Press,[15] more prominent. These have the capacity to reach pressures approaching 30 GPa but have difficultly maintaining pressure at cryogenic temperatures. Some progress has been made[16–19] but such a temperature and pressure range remains challenging.

A more recent report illustrates the temperature and pressure range capable of Paris-Edinburgh presses,[20] which are presented as approximately 8–10 GPa and down to 4 K. One development comes from the work of Komatsu *et al.*[21] and involves a Paris-Edinburgh style press modified for independent cooling of the anvils. This has shown the capability of reaching 88 K, with continuous temperature control. The benefit of this design is the ability to use standard seals and hydraulic oil to generate load on the anvils, as these components are thermally isolated from the cryogenic temperatures. Other advances include the SNAP instrument of Oak Ridge National Laboratory, where a specially designed double membrane has been used with a custom diamond anvil cell design. With this they have been able to reach nearly 80 GPa[22] to date, though with a very limited sample volume.

The limitations of high-pressure neutron diffraction are well known and discussed in depth in many previous reports.[12,15,23–25] High priority considerations for new

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TABLE I. Representative high pressure capabilities of central user facilities around the world. The information in this table was obtained directly from the facility websites and is specific to neutron instrumentation only. Readers should note that such information is not necessarily the most up to date. The type of cell is listed in the first column, including what method used for pressurization. The central column lists the maximal pressure at both ambient and cryogenic temperatures. The final column lists the typical sample dimensions for that pressure. The notation N.R. means that this value was not reported on the particular cell.

| Type | Maximal Pressure (GPa), Temperature (K) | V ($mm^3$) |
|---|---|---|
| Hydrostatic[a] | 0.5 (300), 0.47 (1.5) | 5300 |
| Opposed anvil[a] | 10 (300), 2 (20) | 50 |
| Clamp[a] | 3.0 (300), 2.5 (1.5) | 650 |
| Hydrostatic[b] | 0.7 (1.5–400) | N.R. |
| Press[c] | 10 (85–300) | 16.8–68.1 |
| Clamp[d] | 3 (N.R.) | 100–770 |
| Press[d] | 10 (N.R.) | 30 |

[a]Insitut Laue-Langevin (ILL).
[b]ISIS neutron source.
[c]SNAP, SNS, Oak Ridge National Laboratory.
[d]Paul Scherrer Institut.

neutron instrumentation include large sample volume and operable pressure range. Previous reports on the use of various materials for the anvils[9] have shown that for large sample volumes, reasonable pressures can be achieved through the use of single crystalline sapphire anvils. However, due to the volumes associated with such anvils, the required forces become much larger than for the diamond equivalent. As such, another challenge associated with the design is the control of both pressure and temperature. The vast majority of instrumentation used for extreme conditions studies at neutron facilities uses direct gas pressure, screw driven pistons, or membrane style devices to control the applied load. However, continuous pressure change whilst at low temperatures remains challenging. To tackle this problem a new device has been developed that enables the change of pressure at cryogenic temperatures. The effectiveness of this setup has been demonstrated through load and on-line testing, using the facilities of the WISH beamline at the ISIS neutron source.

## II. TECHNIQUES FOR GENERATING LOAD AT CRYOGENIC TEMPERATURES

For instruments used in cryogenic conditions, there are three primary methods to generate the load. The first, mechanical generation, requires producing the load outside the cryostat and transmitting it down to the sample by means of a shaft or gearbox, resulting in significant thermal linking. The second possibility is through the use of piezo-electric elements. While there is some evidence of these being applied for the study of materials at high pressure,[26] such techniques are not generally viable due to load limitations on the piezo-electric elements.

As a result, only one option is feasible for the design of a moderately sized instrument – pneumatic loading. Such methods are the current mode of operation of standard presses (both multi-anvil and toroidal anvil). An under-represented

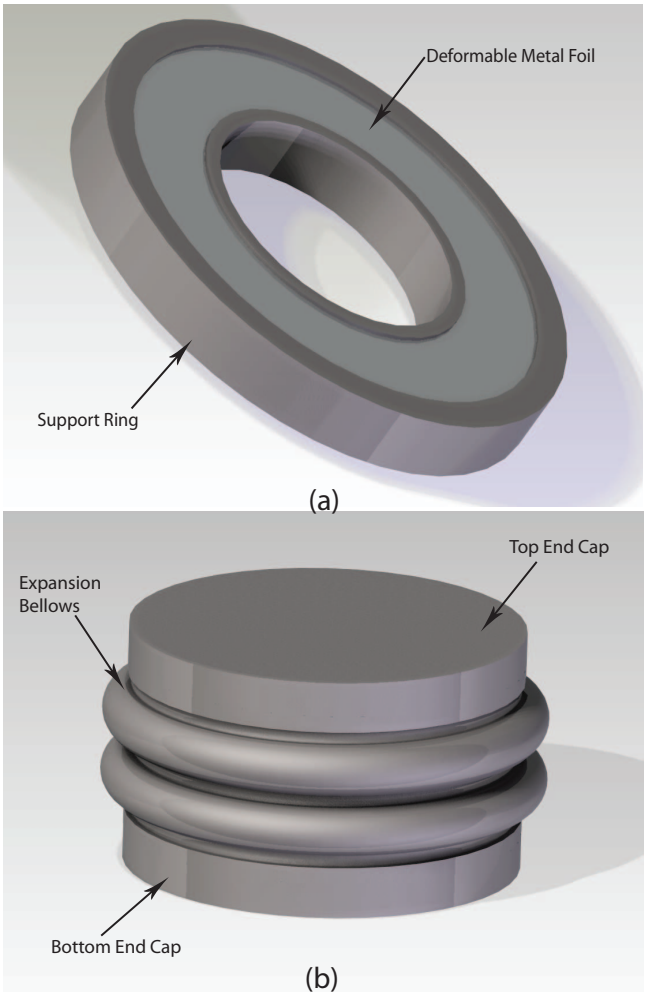


FIG. 1. (a) Small actuation membrane system for DACs. (b) Larger actuation bellows system.

method of pressurization is the fluid-bellows, where the load is generated by fluid pressure behind one or both of the anvils. Generally, such instruments are divided into those with minor actuation (membrane style) and those with major actuation (bellows style). In the past decade, the prevalence of membrane-coupled DACs has grown and now are widely available at most central facilities. Such setups consist of a metal foil sheet welded to a solid ring, as shown in Figure 1(a). These devices are ideal for small pressure cells as they provide the moderate forces needed, with pressures up to 14 MPa (140 bar of gas), and are reported[27] to have expansions up to 0.4 mm, which is sufficient for the small sample volumes used in diamond anvil cells.

One method to overcome both force and pressure control limitations is to move to a bellows-style device. An example of a bellows unit is shown in Figure 1(b). Previous attempts to incorporate a bellows into a pressure setup have been limited to x-ray investigations and DACs. Pfleiderer *et al.*[28] developed a uniaxial cell for attachment to a dilution refrigerator, which was capable of outputting 3 kN of force. This was equipped with a force amplification system, reported to apply a pressure of 2.5 kbar to their specimen. Chen and Weinstein[29] used bellows in series to generate an amplified

force to the sample. According to their report, the bellows had the capacity to generate 2.5 kN each and achieved a sample pressure of 20 GPa on a 150 $\mu$m diameter sample. Webb *et al.*[30] used a bellows driven DAC with an effective area of 15.5 cm$^2$ and a maximal sample pressure of 10 GPa, a maximal load of 2 kN. The application of a bellows pressurization system to a large volume device was reported at the Danish Research Establishment Risø.[31,32] Andresen *et al.*[32] describe a capillary coupled bellows used to actuate a piston. Through this setup, they were able to achieve 13 bar of applied pressure. This system is similar to the Paris-Edinburgh cell, though the absence of reported data suggests that it had limited use. Estimating from the available information suggests that a maximum pressure of 0.2 GPa could be generated on a 3 mm culet, making the system generally unsuitable for large sample volume investigations. The lack of temperature data demonstrating use under cryogenic conditions suggests that this device has a limited range of operation, perhaps only cooled through immersion in liquid cryogen.

## III. DESIGN ATTEMPTS

### A. Energized seal design

One possible design for this new instrument is based on an energized seal, as is shown in Figure 2(a). This seal is similar to that used in a gas loader for the Paris-Edinburgh press[33] and is composed of a tapered back-up seal combined with an energized seal,[34] comprised of a composite or plastic outer layer with an "energizing" spring inside. This spring serves the function of initially forcing the sides of the composite seal outward into the cell body. The introduction of internal gas pressure forces this further and creates a better seal due to the larger internal pressure in the notch of the composite seal.

### B. Packed seal design

Previous reports[12] have also suggested a layout of the internal components as shown in Figure 2(b). In this layout, a deformable metal seal (indium in this instance) is placed between two bronze filled Teflon (PTFE) spacers, with a metal backup seal on top and compressive ring on the bottom. The inner portion of the piston has been threaded to match a hole on the compression plate shown in the figure and, by tensioning the bolt, the metal seal is deformed to create a seal with the outer portion of the cell body.

### C. Limitations of packed and energized seal designs

For both of the previous two designs, loading under ambient conditions has proved reliable and containment of the internal gas was achieved. As such, testing was performed using a standard orange cryostat, cooled to a base temperature of 5 K. For the energized seal, operation was reliable down to nearly 30 K. For the packed seal design, containment was held down to nearly 50 K, where the stability fluctuated and was lost completely near 25 K. For each of these, the primary factor involved in the failure of the gas containment was thermal contraction differences between the seal and cell housing, determined through monitoring of the temperature stability of the cryostat. As each was cooled through these temperatures, the internal gas pressure and temperature of the cryostat rose sharply, indicating that the warm gas introduced into the cell body was leaking into the cryostat itself. At this point, the seals proved unreliable and the instrument was re-designed.

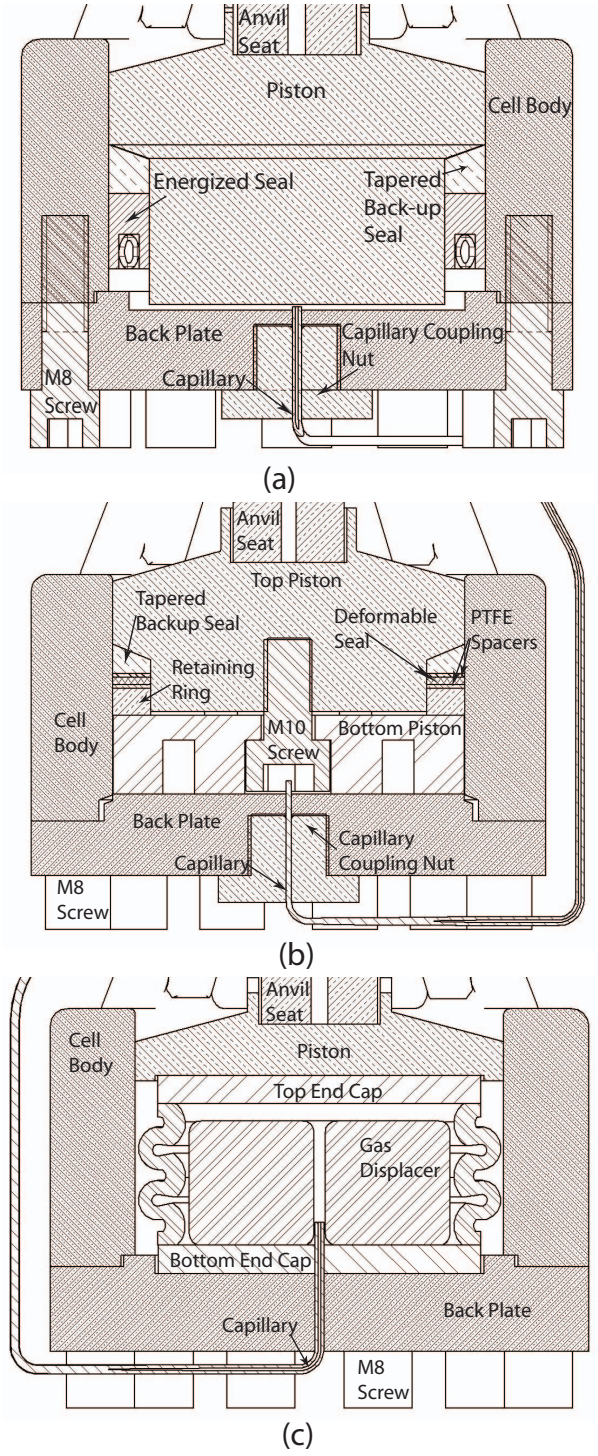


FIG. 2. Various seal designs. This figure shows the three different design attempts pursued during development of the new instrument. All parts are labeled for reference, with the different assemblies discussed in the text. (a) Energized seal design. (b) Packed seal design. (c) Bellows design.

### D. Bellows design

As was described previously, a bellows style device is capable of containing the fluid pressure medium completely. As such a setup avoids the thermal contraction issues presented in the energized and packed designs, the final version of this instrument involved the use of such a bellows. A schematic view is shown in Figure 2(c).

#### 1. Bellows unit

This bellows is composed of eight stacked concentric tubes of 0.25 mm thick stainless steel and two 5 mm thick stainless steel end pieces. The tube pieces were assembled together and hydro-formed into a single bellows collar, leaving no weld seam. The resulting collar was welded to the end pieces, with a gas displacer and a 1.6 mm diameter capillary welded to the bottom. This capillary line is fed through the back plate of the cell and along the cell body to be recovered from the top flange of the cryostat for connection to the helium source. In addition, the piston and back plate both have recesses to help locate the ends of the bellows unit inside the instrument. The bellows has been tested to have a leak rate of less than $10^{-9}$ mbar/s. It is capable of up to 1.5 mm of total actuation and can generate a maximal load of 78 kN on the anvils. This would correspond to a generated average axial stress on a 3 mm culet anvil of 11 GPa. The bellows unit is shown in Figure 3(d).

#### 2. Cell body and deformation

As the intent is for low temperature, the setup has been designed to minimize the thermal mass while retaining as much support as possible. The cell (including anvils and gasket) has a total mass of 4.65 kg, with the main body, piston, back plate, top plate, and anvil supports composed of beryllium copper (NGK Berylco). The cell is 95 mm in diameter and 150 mm tall when assembled with a taper to 60 mm diameter at the top of the cell, decreasing the thermal mass and improving the opening angle (55°). The cell body is shown covered in cadmium shielding in Figure 3(c). ANSYS® finite element analysis (FEA) software package was used to determine the stress distribution and deformation of the cell design due to loading. An example result from these simulations is shown in Figure 4 with a 1 kbar internal pressure creating a stress of 1.3 GPa at the base and top of the support pylons.

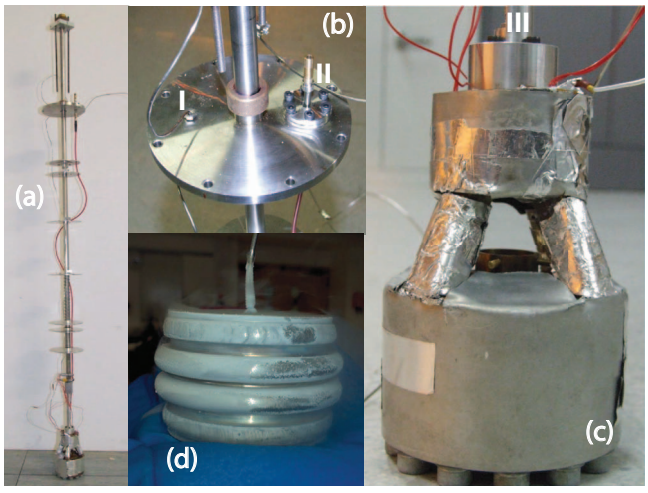


FIG. 3. Bellows operated cell with center stick. In this figure, (a) is the cell mounted on the center stick, (b) shows a view of the top flange of the stick with capillary and fiber ports indicated, (c) shows the cell housing itself, and (d) shows the bellows unit. The cell body is 150 mm tall (copper beryllium material only) and 95 mm in diameter. The cell shown in (c) is covered in cadmium shielding and has had the anvils removed. In (b) and (c), the label I refers to the capillary feed through and II and III refer to the fiber optic feed through and imaging optics, respectively.

#### 3. Anvils and gasket

This instrument has been designed to use a multitude of anvil possibilities. At the present, spherical tungsten carbide (WC) and sapphire anvils and sapphire cylinder anvils are compatible with the design. The gasket has also been tested as a single piece of 1–2 mm thick annealed beryllium copper 25, the "null-matrix" titanium-zirconium alloy, or aluminum. The tungsten carbide anvils are 9 mm diameter balls with culets polished to between 2 and 5 mm in diameter (corresponding to a average culet pressure of 15 GPa at 2 mm and 2.5 GPa at 5 mm) and are used both as anvils and indenter/sample packers. The sapphire cylinders have a diameter of 18 mm and a total initial height of 15 mm, but 9 mm sapphire balls have also been polished and used. These cylinders are polished to the same size culet and bevel angles as the tungsten carbide. With these parameters, the cell is capable of containing a volume between 0.75 (1 mm diameter by 1 mm thick) and 10 $mm^3$ (2.5 mm diameter and 2 mm thick) of sample, allowing for substantial sample in the beam. The anvils have been tested in multiple combinations, including individual or composite (WC on one side, sapphire on the other) combinations.

## IV. OFFLINE TESTING

For offline tests, a NaCl sample and BeCu gasket were used with anvil culets between 2 and 5 mm diameter and tested from ambient up to 200 bar in increments of 10 bar. Separate tungsten carbide anvils, with culets to match the test anvils, were used to indent the gasket and pack the sample until a very dense pellet was achieved. A background spectrum was taken to improve the ruby fluorescence patterns. A small ruby piece was placed into the sample chamber and the top nut of the cell locked into place, giving an initial pressure of 0.1 GPa. PeakFit[35] was used to fit the resulting spectra and extract the R1 line position, with the pressure calibration of Mao *et al.*[36] used. The results of these load tests are shown in Figure 5. From this, the maximal cell pressure ranges from 3 GPa for a 5 mm culet to 8 GPa for a 2 mm culet. Along with these tests, the system was tested for the application of load while at temperature. These tests resulted in similar pressure regimes and demonstrated that the cell was capable of continuous change of pressure whilst at temperature. The bellows were also repeatedly tested in a wet design cryostat prior to neutron experiment. During the tests the maximum gas pressure of 200 bar was supplied to the bellows at the temperature

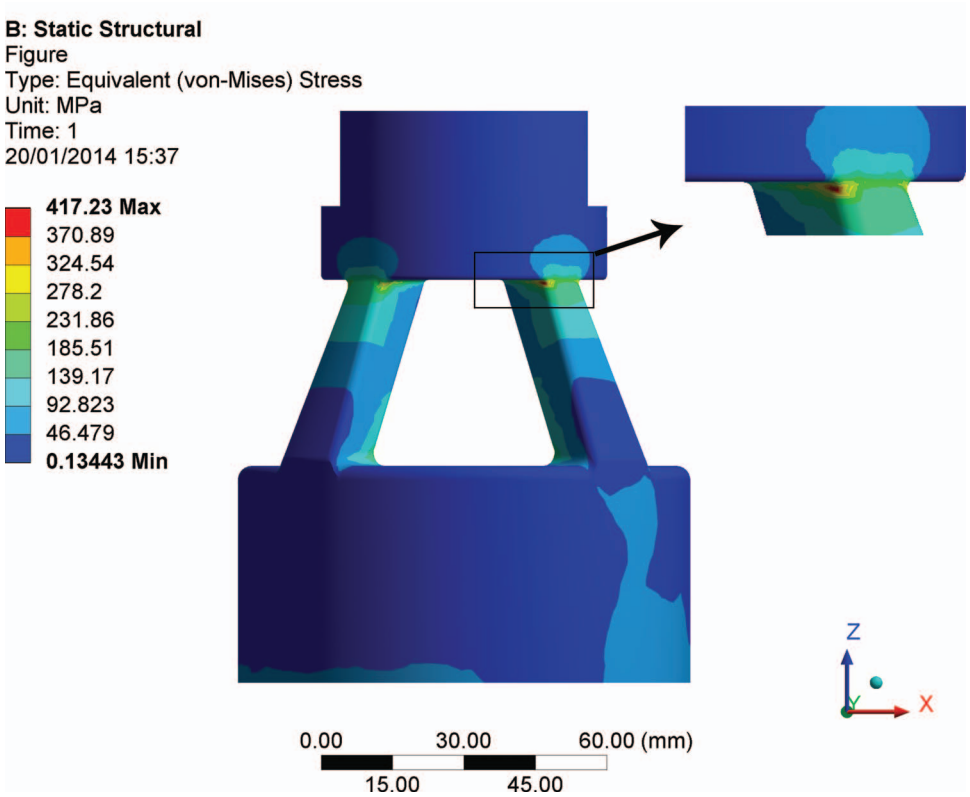


FIG. 4. Finite element study of cell body. The cell body was subjected to an internal pressure in the bellows of 1 kbar of gas (232 kN). The inset shows the maximal stress location from this study, which occurs at the top of the pylons. From the FEA study, the cell body is expected to stretch by approximately 60 $\mu$m in the z direction (along cell symmetry axis) and have a maximal stress of 400 MPa.

of 9 K, which is just above the solidification temperature of helium[37] at that pressure.

## V. ONLINE TESTING

In addition to the load testing, the cell was on the WISH instrument[38] of the ISIS pulsed neutron source, with NaCl as the specimen. The cell used a 1 mm thick titanium-zirconium gasket, indented to 0.6 mm thickness. A 1 mm hole was filled with the NaCl sample and ruby was added as a pressure marker. This was pressurized from ambient to 200 bar, with data collected at ambient, 100 bar, and 200 bar in 5 h runs and cooled to 140 K using the in-house Closed-Cycle Refrigerator (CCR) units. An example of the resulting data is shown in Figure 6, taken at maximum load. From the results, the internal pressure at 200 bar was 5.5 GPa,

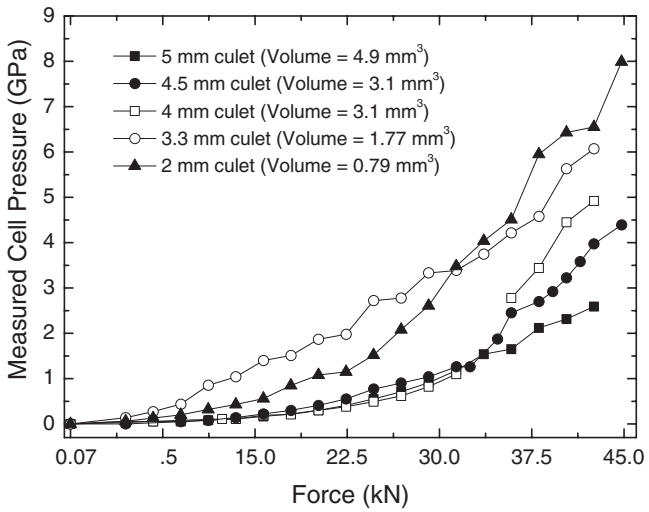


FIG. 5. Internal cell pressure vs. applied load. The points are the measured pressures with the lines serving as guides for the eyes. It should be noted that the cell was locked more tightly for the 3.3 mm diameter culet experiment, resulting in a lower take-off force than for the other diameters. In all cases, the loading measured was accomplished at room temperature (300 K).

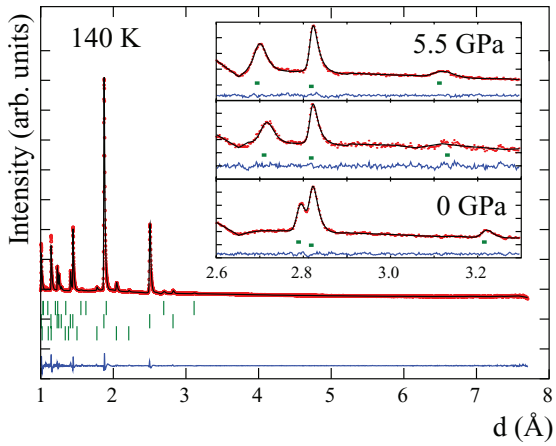


FIG. 6. Neutron diffraction patterns for NaCl sample at 140 K. The inset shows an enlarged view of the clearest peaks for NaCl and one WC peak. The ticks at the bottom represent (from top to bottom) NaCl, WC, and diffraction from the binder for WC.

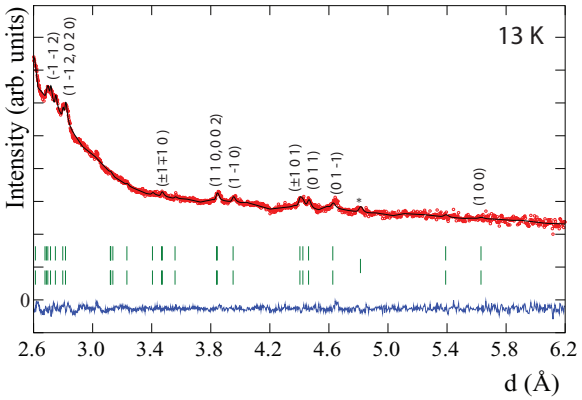


FIG. 7. Neutron diffraction pattern for $BiNiO_3$. This pattern was taken in a 12 h count at 13 K and 1.5 GPa. Although the gasket contribution makes determination difficult, sample peaks are clearly visible above the background. The tick marks along the bottom indicate (from top to bottom) crystal structure, NiO impurity, and magnetic structure peak locations. The sharp rise at low d-spacing is caused by the BeCu gasket. Prominent $BiNiO_3$ peaks are indexed and one impurity peak is indicated by the star.

according to the NaCl equation of state determined using EoSFIT.[39]

This system was also tested using $BiNiO_3$ as a sample, contained in an annealed beryllium copper gasket. In this situation, a 12 h count was taken at an internal pressure of 1.5 GPa and temperature of 13 K. This pressure was achieved by supplying 80 bar of helium gas to the bellows. Further pressurization resulted in failure of the sapphire anvils used and prevented higher pressures from being achieved on this sample. Due to the triclinic distortion of this perovskite-type material, the diffraction peaks are relatively weak against the background (Figure 7). A Rietveld fit, performed using FullProf,[40] confirmed that the magnetic structure is the same G-type spin ordering as observed at zero pressure,[41,42] but the crystal and magnetic structure parameters could not be refined.

## VI. CONCLUSIONS

A new instrument for neutron scattering experiments at high pressures and low temperatures has been developed. This apparatus is equipped with a bellows unit capable of generating up to 78 kN of force and altering the pressure while at cryogenic temperatures. The system has been used with a variety of culet diameters in the range from 2 to 5 mm on both tungsten carbide and single crystal sapphire anvils. It was determined that the internal pressure can reach up to 8 GPa when 200 bar of gas (equivalent to 45 kN of force) is applied through the bellows.

By contrast, it was expected on a theoretical basis that such an instrument should be capable of producing 11 GPa on a 3 mm culet, while only 6 GPa was achieved. The value of 11 GPa has been estimated as the theoretical value of the average pressure across the culet of the anvil generated by the maximum force of 78 kN which can be produced by the bellows with the 350 bar gas pressure on the culet of 3 mm diameter. The bellows have been tested to work to this limit; however the gas supply system used the helium gas at the maximum pressure available from a standard cylinder (200 bar). A pressure intensifying unit, with higher gas pressure capacity, is currently under development.

In addition the actual pressure generated in the sample depends on a number of parameters, such as the relative hardness between the sample and the gasket, the moduli of the sample and the medium, the hydrostaticity of the medium, the position of the ruby in the sample volume, etc. Discussions of such sources of discrepancy can be found in several literature sources.[43,44]

High pressure experiments have been performed on both NaCl and the magnetically interesting, low symmetry perovskite $BiNiO_3$ at low temperature. Good quality diffraction patterns were obtained within a few hours for NaCl and 10–12 h for $BiNiO_3$. Further improvements are currently being undertaken with this device, including optimization of the anvil design and testing of a wider variety of gasketing materials.

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