

# Simple high-pressure cell for neutron scattering

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A high-pressure cell, capable of 8 kbar, is developed for neutron scattering. It can be used with ILL type orange cryostats to obtain a temperature as low as 1.5 K. The simple seal design described here can easily be adopted to other high-pressure applications. © 1995 American Institute of Physics.

Temperature and pressure are important thermodynamic variables. While low-temperature is regularly used in neutron scattering, the use of high pressure is less standard. However, the interest in doing neutron scattering under high pressure has been increasing.<sup>1</sup> There are special requirements for high-pressure cells used in neutron scattering.<sup>2</sup> For example, the flux rates of current neutron sources require large enough sample volume in order to get acceptable signal-to-noise ratio in reasonable time. Also the cell body in the neutron beam gives rise to unwanted scattering and absorption. This demands a minimum of material in the beam path.

In this note, we report a simple cylindrical high-pressure cell for neutron scattering (see Fig. 1). The pressure is generated by a hydraulic compressor (not shown in the figure), made by Harwood Engineering Co. The pressure transmitting medium, helium, is connected to the high-pressure cell by a stainless-steel capillary (F). The pressure cell assembly is handy enough to be installed in a top-loaded ILL cryostat and a temperature as low as 1.5 K can be achieved. With the standard precautions,<sup>3</sup> quasihydrostatic pressure can be obtained at low temperature at which helium is solid. At elevated temperature above the helium melting point, the pressure is hydrostatic. The pressure in the cell can be monitored by the manganin resistance cell in the compressor, or by using neutrons to measure the lattice constant of a known crystal, e.g., graphite<sup>4</sup> or NaCl,<sup>5</sup> in the cell.

Sealing of the pressure cell is an important aspect of the pressure cell design.<sup>6</sup> It can be an Amagat "excess pressure" seal, as rendered in, for example, the tightened cone-to-cylinder contact. But the pressure which can be sealed is limited by the tightening stress. Bridgman-type or Poulter-type "self-sealing" seals need no pretightening. The high pressure inside the pressure cell provide the sealing pressure. But such seals usually only work at some elevated pressure. This leads to hybrid seals which combine features of both the excess pressure seal and the self-sealing seal. Sophisticated seals of this type have been designed.<sup>7</sup> The seal we describe here is a hybrid of the Amagat and Poulter types (refer to Fig. 1). When tightening bolt (C) against nut (D), the pushing cone (B) presses the cylinder-shaped copper ring (E) onto the step of the sample cell (A) thus deforming the ring into a wedged shape. This forms an Amagat seal which prevents leaking at low pressure. When the pressure increases,

the wedged ring acts as a Poulter seal. The copper ring (E) is not reusable. But it is very easy to make. The seal design is very simple yet reliable and it can easily be adopted to other high-pressure applications.

The high-pressure limit of the pressure cell is set by the material strength, the inner and the outer diameters of the sample cell (A). Both Maraging steel and beryllium-copper (Berylco 25) were used due to their mechanical properties from low to ambient temperature and their acceptable neutron absorption and scattering properties.<sup>2</sup> Berylco 25 has the added merit in that it is nonmagnetic which can be important when investigating magnetic samples. The inner diameter of the cell is determined by the sample volume desired and the thickness of the cell wall is determined by the compromise between the high-pressure limit and the background contributions. The sizes we settled on are 0.228 in. for the inner diameter and 1/2 in. for the outer diameter. A Maraging steel cell with these dimensions sustained 8 kbar without bursting.<sup>8</sup>

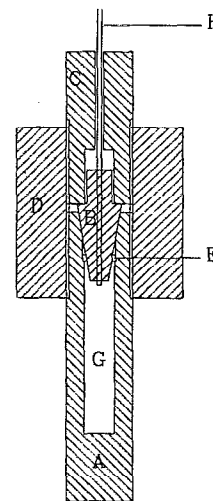


FIG. 1. Cross section of the high-pressure cell assembly: (A) sample cell, (B) pushing cone, (C) tightening bolt, (D) assembling nut, (E) sealing ring, (F) capillary from hydraulic compressor. (G) is the sample cavity. (A)–(D) are made of high strength material. (E) uses soft metal. The inner and outer diameters of the sample cell (A) we used are 0.228 and 1/2 in., respectively. The diameter in the opening step of (A) is 1/4 in. The matching cone surfaces of (A) and (B) have full angle of 20°. The cylindrical ring (E) is 3/64 in. in height and 1/32 in. in thickness, with the outer diameter of 1/4 in.

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Uses of this high-pressure cell in neutron scattering experiments are reported elsewhere.<sup>9</sup>

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<sup>1</sup>See, for example, D. B. McWhan, D. Bloch, and G. Parisot, *Rev. Sci. Instrum.* **45**, 643 (1974); J. Paureau and C. Vettier, *ibid.* **46**, 1484 (1975); H. Fujiwara, H. Kadomatus, and K. Tohma, *ibid.* **51**, 1345 (1980); A. Y. Wu, E. Whalley, and G. Dolling, *ibid.* **56**, 1409 (1985); A. Onodera *et al.*, *Jpn. J. Appl. Phys.* **26**, 152 (1987).

<sup>2</sup>For a comprehensive review, see C. J. Carlile and D. C. Salter, *High-Temp. High-Press.* **10**, 1 (1978).

<sup>3</sup>At high pressure and low temperature, the pressure adjustment should be conducted with helium warmed up above the solid phase and the cooling should be isobaric. Heating wire wound around capillary inside the cry-

ostat can be used to prevent blockage of capillary by crystallization of helium. See, for details, J. E. Schirber, *Cryogenics* **10**, 418 (1970).

<sup>4</sup>W. B. Gauster and I. J. Fritz, *J. Appl. Phys.* **45**, 3309 (1974).

<sup>5</sup>D. L. Decker, *J. Appl. Phys.* **42**, 3239 (1971); D. L. Decker and T. G. Worlton, *ibid.* **43**, 4799 (1972).

<sup>6</sup>S. Lee and K. Luszczynski, *Rev. Sci. Instrum.* **58**, 1262 (1987).

<sup>7</sup>See, for example, Yu. E. Gorbaty, *Rev. Sci. Instrum.* **65**, 505 (1994); C. J. Richards and M. R. Fisch, *ibid.* **65**, 335 (1994); P. T. T. Wong, *ibid.* **56**, 1417 (1985); J. Paureau and C. Vettier, *ibid.* **46**, 1484 (1975); R. Verbrugge *et al.*, *ibid.* **56**, 625 (1985).

<sup>8</sup>It is reported that when hydraulic autofrettage is performed, a beryllium-copper high-pressure cell can hold up to 30 kbar, which is three times larger than the tensile strength of the material. See, H. Fujiwara, H. Kadomatsu, and K. Tohma, *Rev. Sci. Instrum.* **51**, 1345 (1980).

<sup>9</sup>Wei Bao, C. Broholm, S. A. Carter, T. F. Rosenbaum, G. Aeppli, S. F. Trevino, P. Metcalf, J. M. Honig, and J. Spalek, *Phys. Rev. Lett.* **71**, 766 (1993).