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Hydrostatic optical cell with simple window structure for low temperature and hydrostatic pressure up to 5 kilobars

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A compact gas high-pressure cell with four windows for optical studies of phase transitions at low temperature and hydrostatic pressure up to 5 kilobars has been made. Techniques for sealing window components are discussed.

We describe here a four-window high-pressure cell with a cryostat, which was mainly designed for optical studies of the tricritical phenomena and dynamics in KH_2PO_4 (the tricritical point is at 111 K and 2.4 kilobars).[1] The principal design considerations are (1) the requirement of effective low-temperature pressure seals for helium gas as the pressure transmitting medium and (2) the need to minimize the temperature gradient of the cell in order to keep the sample temperature constant with high accuracy. There are several possible choices for the cell and seal materials. Beryllium–copper was chosen here for the cell body and various parts of the window and sample-holder ports, because of its ease of machining and heat treating and its good thermal conductivity. The technique for sealing high-pressure gas at low temperature differs in several details from conventional techniques[2,3] particularly in the use of unhardened Be–Cu O-rings whose linear thermal expansion has practically the same value as hardened (heat treated) Be–Cu in the temperature region from room to liquid nitrogen.[4] It is also noted that the linear thermal expansion of Be–Cu is very close to that of 304 stainless steel which is commercially used for pressure tubing, gland nuts, and collars.[4]

The optical pressure cell and the tail section of the cryostat are illustrated in Fig. 1. The dimensions of the cell present a compromise to keep the optical path length to a minimum and still have a reasonable aperture, in this case 3.17 mm diameter. The outside dimensions are 63.5 mm diameter by 76.2 mm length. The design of window ports owes much to an earlier design.[2] The conventional "unsupported area" seal was used. The optical windows (1) are Union Carbide[5] synthetic sapphires 6.35 mm in diameter by 6.35 mm long, with axes parallel to the c-axis and ends optically ground to 2λ flatness. The polished faces of these windows are glued with an adhesive mixture of varnish and toluene to the lapped and polished faces of the hardened Be–Cu window seats (2). The window seats are backed up with unhardened Be–Cu O-ring seals (3) and by hardened Be–Cu threaded plugs (4), which have a 12° tapered aperture hole to reduce shadowing. The detailed window structure is shown in Fig. 2. The window seat, seal, and plug are removed from the cell body (5) as a unit, using an extractor tool which screws into the aperture hole of the window seat. The structures of the sample holder (6), ring seal (7), and support plug (8) are similar to those of the window. The cell is connected to pressure tubing (9) of 6.35 mm o.d. by 1.59 mm i.d. with a standard gland nut (10) and collar (11), supplied by American Instrument Company.[6] The pressure cell is surrounded by a radiation shield can (12) and an outer can (13) of copper which is connected to the liquid nitrogen tank (14), which acts as a constant-temperature heat sink. The space inside of the Dewar (15) is evacuated to 10^{-5} Torr to minimize the heat losses and temperature gradient of the cell. The copper tubing (16) surrounding the

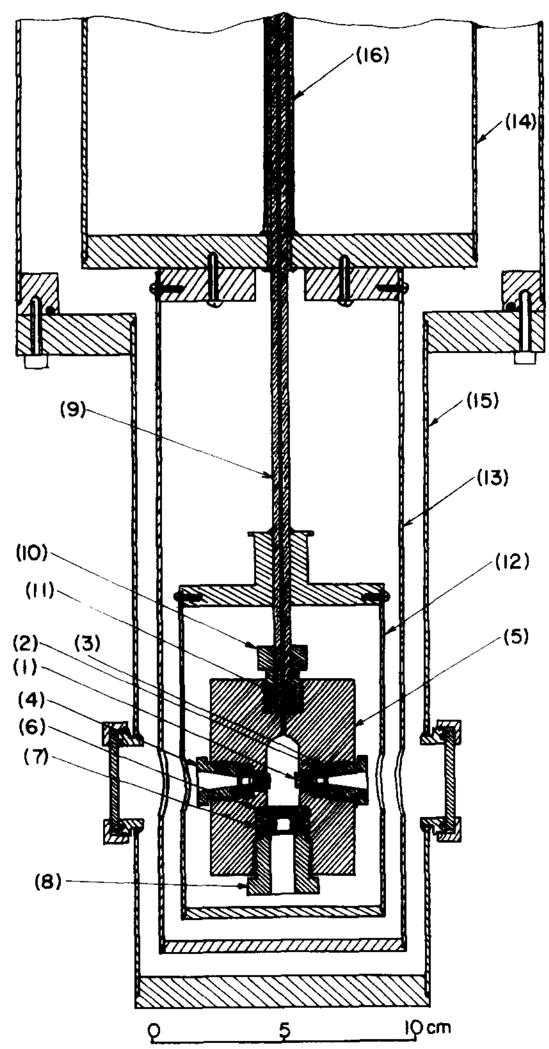

FIG. 1. The optical pressure cell and the tail section of the cryostat.

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pressure tubing inside the tank is provided to prevent changing of the temperature of the pressure tubing as the liquid nitrogen level varies. The temperatures of the cell and the inner copper can are controlled by a low-temperature capacitance controller (Lake Shore Cryotronics Inc. Model CSC 400)[7] and a homemade thermocouple controller, respectively.

In the present case, all parts of the cell were machined from a 63.5-mm-diam rod of ½ hard "Berylco 25" alloy, purchased from Kawecki Berylco Industries, Inc.[8] The chemical composition was Be (1.80%–2.05%), Co (0.20%–0.30%), and Cu (Bal.). The physical properties before hardening as listed by the manufacturer are tensile strength, 7.41 kilobars; elongation, 17%; and grain size, 0.020 mm. After hardening at 315 °C for 2 h, they are tensile strength, 13.4 kilobars; yield strength for 0.2% offset, 12.3 kilobars; and elongation, 5%. After machining, all parts of the cell except the ring seals were hardened by heat treating at 315 °C for 2 h, using an argon atmosphere to keep surfaces clean. The surfaces of the cell body, window seats, and sample holder in contact with each other and with the ring seals were polished with different grain sizes (1.0, 0.3, and 0.05 μm) of alumina on polishing cloths. The surfaces of the window seats on which the windows sit were carefully lapped until they were smooth and flat enough to adhere to and support the windows. After the window and seat were carefully cleaned, a thin coat of adhesive mixture was applied on the seat. The window was placed on the plug without sliding or rotation and gently pressed until it started to adhere. To assemble the cell, the seats and plugs were coated with Teflon oilless dry lubricant, supplied from American Durafilm Co.,[9] and the plugs were tightened to a torque of ~10 N·m to squeeze the ring seals. This procedure has provided an excellent initial seal and easily maintained a seal during thermal cycling.

The leak test of the pressure cell was made with helium gas at 0.24 kilobars in the temperature region from room temperature to liquid nitrogen temperature. The leak rate was small enough to allow us to maintain a pressure stability of ±0.2 bars which was necessary to match the temperature stability of 2 mK. The pressure cell was also tested at room temperature with oil as the pressure transmitting medium by using our homemade high-pressure generating system, and no leakage or window fracture has occurred at pressures up to 5 kilobars. Although

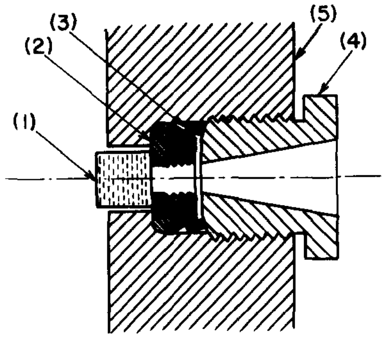

FIG. 2. The window assembly.

further high-pressure testing has not been done as the present cell was mainly designed for optical studies on the tricritical phenomena in KH_2PO_4, it may be used at higher pressures since the bursting pressure of the sapphire window including a safety factor of 4 is 13.8 kilobars based on extrapolation of bursting pressure versus thickness/diameter ratio data from Union Carbide.

To date, this cell has been used in birefringence measurements made on KH_2PO_4 at ambient pressure near its ferroelectric transition at 122 K. The capacitance controller held the cell temperature constant within 2 mK for periods of 5 h. We expect that this cell will be useful for a wide variety of optical experiments.

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[6] American Instrument Company, Division of Travenol Laboratories, Inc., 8030 Georgia Ave., Silver Spring, MD 20910.
[7] Lake Shore Cryotronics, Inc., 9631 Sandrock Rd., Eden, NY 14057.
[8] Kawecki Berylco Industries, Inc., 220 East 42nd St., New York, New York 10017.
[9] "Oil-es Oil", American Durafilm Co., Inc., 2300 Washington St., Newton Lower Falls, MA 02162.

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