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Four-Window Cell and Cryostat for High Pressure Studies at Liquid Helium Temperature*,†

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A compact gas high pressure cell for measuring the optical properties of solids at low temperatures under pressures up to 10 kilobars is described. Using helium gas as the pressure-transmitting medium, the pressure is purely hydrostatic at 78°K and nearly hydrostatic at 7–12°K. Techniques are discussed for sealing windows of sapphire for optical studies, of beryllium for x irradiation, and dummy windows for electrical connections. A cryostat which can be accommodated in most recording spectrophotometers is also described.

INTRODUCTION

IN extending techniques for the measurement of pressure effects to low temperatures, the principal design considerations are the need to keep the mass of the pressure cell to a minimum, the requirement of effective cryogenic pressure seals, and the problem of achieving and measuring hydrostatic pressure.[1] This paper describes a versatile four-window high pressure cell which has proved quite satisfactory for this temperature range. Because of its compact size, not only are the liquid helium requirements modest, but also the cell and its cryostat are small enough to fit directly into the sample chamber of many recording spectrophotometers. By using helium gas as the pressure-transmitting medium, hydrostatic pressures are possible at temperatures down to the freezing curve of helium, while at lower temperatures the solidified helium is sufficiently soft and plastic that nearly hydrostatic conditions exist after slow cooling. Since the sample does not undergo plastic deformation or shear, it is possible to study structure-sensitive properties. The techniques for sealing high pressure gas at low temperatures, while not novel in themselves, differ in several details from conventional techniques, particularly in the use of indium metal packing rings. With its small internal volume, the pressure cell is suitable for use with a tea-cart type of pressure system.

The cell has been used principally in a study of color center formation in alkali halides at low temperatures and high pressures.[2] In this application, the color centers were produced by x irradiation through a beryllium window, and were detected by measuring the optical absorption at right angles through sapphire windows. In this case, the helium gas had the additional advantage of transparency to both visible light and x rays, as well as being chemically inert under irradiation.

The apparatus is described and illustrated as it was used in this application. In addition, techniques for introducing electrical leads are presented, and the determination of pressure and temperature at the sample discussed.

DESCRIPTION OF APPARATUS

A. Pressure Cell

The pressure cell used throughout this work is illustrated in Fig. 1. The cell was designed for and tested at pressures up to 10 kilobars,[3] and no leakage or window fracture has occurred at the maximum operating pressure of 8 kilobars.

The dimensions of the cell represent a compromise to keep both the mass to be cooled and the optical path length to a minimum and still have a reasonable aperture, in this

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[1] For a recent review of high pressure techniques and measurements, see C. A. Swenson, "Physics at High Pressure" in *Solid State Physics*, edited by F. Seitz and D. Turnbull (Academic Press Inc., New York, 1960), Vol. XI.

[2] D. B. Fitchen, Ph.D. thesis, University of Illinois, Urbana, Illinois, 1962 (results to be published).

[3] 1 bar $=10^6$ dyn/cm^2 $=1.0197$ kg/cm^2 $=0.9869$ atm.

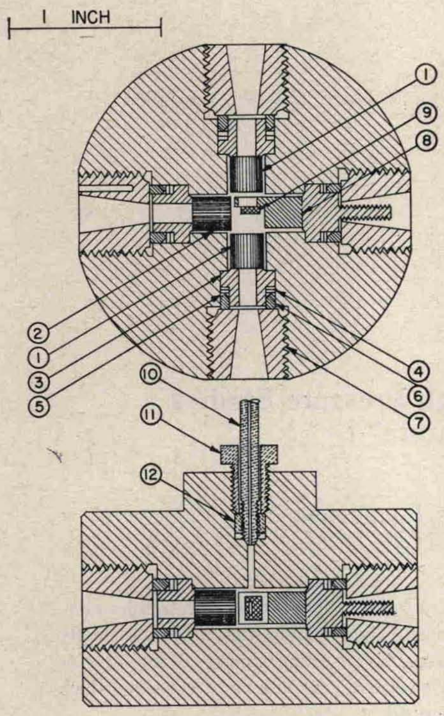

FIG. 1. The pressure cell as used for color center measurements.

case $\frac{1}{8}$-in. diameter. The outside dimensions are $2\frac{1}{4}$ in. in diameter by $1\frac{1}{2}$ in. in length, and the assembled unit weighs 23 oz.

Beryllium-copper was chosen for the cell body and various parts of the window ports because of its ease of machining and heat treating, and to avoid any problems of brittle fracture with steels at low temperatures. Beryllium-copper has been used for nonmagnetic pressure vessels, but has not been used widely in other high pressure work because of some early ruptures. The problems of selection of stock free of flaws and the performance of pressure vessels made of this alloy have been described in the literature.[4] Apparently, the trouble arises from bubbles and flaws in the hot-rolled stock used for the larger vessels. When only small-diameter vessels are needed, Paul[5] had indicated that the cold-rolled stock obtained from the supplier can probably be trusted without careful ultrasonic testing.

In the present case, the cell body was machined from the largest size of cold-rolled stock available, a $2\frac{1}{4}$-in.-diam rod of $\frac{1}{2}$-hard "Berylco 25" alloy. After machining, the piece was hardened to about Rockwell 40-C by heat treating at 600°F for 2 h, using an argon atmosphere to keep surfaces clean. Window parts were made from smaller rods of the same material.

The seats for the windows are of conventional Bridgman design[6–8] in a compact version. The optical windows (1) are oriented Linde synthetic sapphires $\frac{1}{4}$ in. in diameter by $\frac{1}{4}$ in. long, ground optically flat on the ends. The x-ray window (2) is "Berylco PX-20" extruded beryllium rod, supplied by the Beryllium Corporation of America as cylinders of the same size which were then lapped flat. The polished faces of these windows are glued with an adhesive mixture to the lapped and polished faces of the "mushroom plugs" (3). The mushroom plug rests on thin bronze, indium, and copper packing rings (4), prevented from extruding by small beryllium-copper rings of triangular cross section (5), which fit against the face of a hardened support ring (6). The support ring is held in place by the threaded plug (7) which has a 10° tapered aperture hole to reduce shadowing, and holes for a three-pin wrench so that it can be screwed in flush with the cell body.

In this experiment, the sample holder (8) is mounted on the fourth plug, a blind version of the other three, to provide easy access. The sample (9) is held on a fixed copper slit and surrounded by baffles to prevent light leaks around the sample holder.

The pressure connection is made with hard-drawn 316 stainless steel tubing (10), $\frac{1}{8}$-in. o.d. by 0.025-in. i.d., supplied by Harwood Engineering Company, and coupled to the cell with a standard Harwood gland nut (11) and collar (12).

B. Window-Sealing Techniques

The design of the window ports owes much to earlier designs of Warschauer and Paul,[7] and of Langer and Warschauer.[8] Various attempts to make a more compact window using soft metal O-ring seals proved unreliable at low temperatures, so the conventional "unsupported area" seal was used. The $\frac{1}{4}$-in.-diam stem of the mushroom plug is unsupported, thus maintaining a pressure in the packings twice that of the gas being sealed. The stem is backed up by the threaded support plug in case it should suffer "pinch-off" from the high pressures at the packing rings, but this has never occurred. The clearance between stem and plug is such that yield before rupture would probably bring them into contact and relieve the stress.

The packing rings selected were 0.035-in. bronze, 0.035-in. indium, and 0.005-in. copper. The bronze and copper rings serve mostly to contain the soft indium ring. Indium is very effective as a gas seal because it adheres well to the beryllium-copper walls to provide an excellent initial seal, and because it flows easily to maintain a seal during thermal cycling. The mushroom plug, packings, and support ring are inserted and removed as a unit, using an extractor tool which screws into the last $\frac{1}{16}$ in. of the aper-

[4] W. Paul, G. B. Benedek, and D. M. Warschauer, Rev. Sci. Instr. **30**, 874 (1959).

[5] W. Paul (private communication).

[6] P. W. Bridgman, *The Physics of High Pressures* (G. Bell and Sons, London, 1949).

[7] D. M. Warschauer and W. Paul, Rev. Sci. Instr. **28**, 62 (1957).

[8] D. Langer and D. M. Warschauer, Rev. Sci. Instr. **32**, 32 (1961).