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Apparatus for Optical Studies to Very High Pressures*

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An apparatus has been developed in two stages which permits optical studies to 200 000 atmospheres pressure. The use of NaCl windows permits studies in the near ultraviolet and in the infrared as well as in the visible. The design of the pressure cells and of a press suitable to fit in a spectrometer are described with essential dimensions. A calibration tested against Bridgman's data to 87 000 atmospheres is given.

EQUIPMENT has been developed which permits the observation of the effect of pressure on optical phenomena at least to 200 000 atmospheres pressure. The apparatus is in two stages, one of which is operable to 60 000 atmospheres while the second is used in the higher range. These are described as Cell I and Cell II below.

Both cells use an alkali halide (normally NaCl) as a pressure transmitting "fluid," and NaCl windows. Although sodium chloride has a relatively low shear strength and is effectively a fluid at very high pressure, it is a very viscous fluid. When fused in a long narrow hole by the repeated application of 30 000–40 000 atmospheres pressure at both ends, it is not extruded for a very long time even under the action of 60–80 000 atmospheres pressure. Using relatively intense light sources many optical and spectroscopic observations can thus be carried out on a sample at high pressure.

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CELL I

The outer steel jacket (*B*) is AISI 6150 steel hardened to 46-48 Rockwell C (Fig. 1). The inner cell (*A*) is made from Solar steel hardened to 58–60 Rockwell C and is pressed into (*B*). The carboloy pistons (*C*) are grade 883 or 999 jacketed with AISI 4140 hardened to 42-44 Rockwell C. Only the lower piston ($\frac{1}{8}$ in. diam) moves.

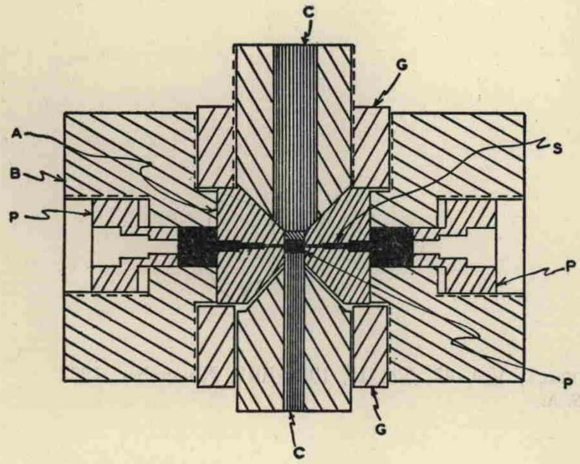

FIG. 1. Cell I—for 60 000 atmos.

The salt windows S consist of three sections in the Solar insert plus an outer section. From the inside a hole $\frac{1}{8}$ in. long 0.028 in. in diam, a hole $\frac{1}{8}$ in. long 0.047 in. diam, and a hole $\frac{3}{32}$ in. long 0.067 in. diam. The outer hole is $\frac{1}{4}$ in. in diam and is filled approximately $\frac{1}{4}$ in. deep. The windows are filled by applying pressure to heated NaCl crystals placed in the outer hole and in the center.

The inner chamber is $\frac{3}{16}$ in. long and is lapped to fit the pistons to 0.0005 in. The holes gradually stretch in service and the inserts must be replaced after 15–30 runs. The pressure is alternately applied in the center and at the outside until the windows become sufficiently clear. This may take as many as half a dozen applications of alternating pressure of about 30 000 atmospheres. The outer plugs P serve to minimize the breakup of the outer edges of the windows. The center NaCl can be removed and the sample inserted, usually in a very thin section between two alkali halide crystals. Thus it is not affected by any extrusion which takes place. The salt pellet is usually 0.040–0.060 in. thick. The spectra obtained are reversible with rising and falling pressure within about 5% on the pressure scale, indicating that friction is not large. Phase transitions which are not accompanied by a large "region of indifference" as described by Bridgman also show this reversibility.

The primary calibration was carried out by using the phase transitions observed by Bridgman[1] in bismuth at 24 700 atmospheres and in tellurium at 43 500 atmospheres. The secondary standard is the shift with pressure of the 2210 cm^{-1} vibration of CN-ion dissolved by fusion in NaCl.[2] This vibration shifts continuously to the "blue" (higher frequencies) with pressure. The data can be fitted by the equation

$$p=0.965\Delta\nu+4\times10^{-3}(\Delta\nu)^2+5.3\times10^{-5}(\Delta\nu)^3, \quad (1)$$

where $\Delta\nu$ refers to the change in frequency from the atmospheric pressure value in cm^{-1} and p is the pressure in thousands of atmospheres.

Cell I fails somewhere above 50 000 atmospheres because the moving carboloy piston breaks in compression.

CELL II

Cell II (Fig. 2) utilizes the same principles described for Cell I, plus one further development. The pistons are much larger in diameter with a corresponding change in insert size, window holes, etc. Typical dimensions are summarized in Table I. The pistons are tapered with a flat section $\frac{3}{32}$ in. in diam in the center. (This is the smallest flat conveniently operable in our experiments.) When the pressure is applied to the movable piston the center is compressed by a greater percentage than the taper. This results in a pressure gradient from the edge of the flat to the outside edge of the piston. The greater the taper, the higher the gradient, but also the higher the tendency to extrude from the flat section. After trying a wide variety of tapers and combinations of tapers, we found that a single taper of 6° on each piston was the most advantageous. The tapered piston accomplishes several objectives. The very high pressure is only on the center of the piston which does not break because of the principle of "massive support" mentioned by Bridgman. The salt also supports the piston along the taper. The salt from the flat to the outside under a continuously decreasing pressure acts as a series of "cells within a cell" and minimizes extrusion, permitting a pellet of useful thickness in the center. The average applied pressure and the drop in pressure at the outside of the piston is such that the windows have negligible tendency to extrude.

The sample is inserted in a thick slice in the center of the flat portion and perpendicular to the light path.

The pressure obtained is a distinct function of the thickness of the central flat portion (t_C) as well as of the average pressure across the piston (p_A). The pressure was calibrated by using the CN^- stretching frequencies obtained on Cell I and as upper points the phase transitions rated by Bridgman[3] on AgBr at

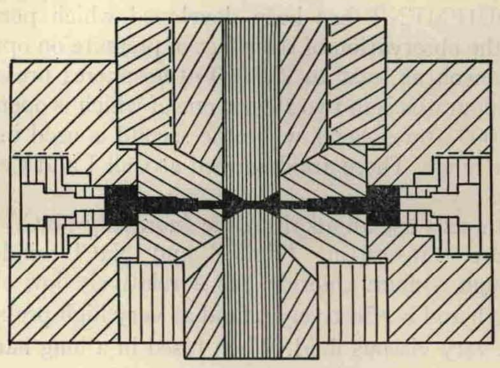

FIG. 2. Cell II—operated to 200 000 atmos.

[1] P. W. Bridgman, Proc. Am. Acad. Arts Sci. 74, 425 (1942).
[2] A detailed discussion of pressure effects on the CN^- stretching frequency will be published elsewhere.
[3] P. W. Bridgman, Proc. Am. Acad. Arts Sci. 76, 1 (1945).