B. INSTRUMENTATION FOR OPTICAL MEASUREMENTS AT HIGH PRESSURES [28]

The instrumentation necessary to make spectroscopic measurements at non ambient pressures includes an optical high pressure cell, a spectrophotometer, and the interface or optical link of the cell with the spectrophotometer. The optical link of the pressure cell to an IR spectrophotometer necessitates the use of a beam condenser, and to an interferometer a light-pipe or a beam condenser may be necessary. Very important considerations of this instrumentation are the windows used in the optical high pressure cell. The properties of the windows are of prime importance since they must be transparent to the electromagnetic radiation of interest and must be of sufficient strength to withstand high pressures. Ancillary equipment for some systems may include pressure-transmitting and pressure-measuring devices.

(i) Optical high pressure cells

The high pressure cells which can be used for optical purposes may be of three main types: (1) shock wave cell; (2) piston-cylinder cell; and (3) opposed anvil cell. Table 2 summarizes advantages and disadvantages of the three types of cells.

(1) Shock wave cell

The shock wave cell utilizes high explosives to obtain pressure and will give the highest pressures of any cell discussed in this paper [29]. However, the pressures are of short duration and may be destructive to the sample and optical equipment; moreover making the optical link to a spectrophotometer is difficult. Since the effects of pressure are of short duration (10^{-5} s) fast-scanning spectrophotometers are necessary. Except in specialized cases where extreme pressures are necessary to demonstrate some phenomenon, the shock wave cell and technique are less practical than the piston-cylinder or the opposed anvil cell. Many of the physical measurements that are possible with the latter two cells on in situ samples are not possible with shock-waves.

(2) Piston-cylinder cell

The piston-cylinder type cell or variations thereof have made notable contributions and can be used in an extended region of the electromagnetic spectrum from the UV region to the IR. The piston-cylinder cell has been used primarily for solids, although it has been adapted for use with liquids or solutions as well.

The best known cell of this type is the supported taper Drickamer cell, which was developed by Drickamer and his students [30—33], and is a cross between an anvil and a piston-cylinder apparatus. Several variations of this cell have now been made. Tables 3 and 4 summarize details of the pressure

TABLE 2

Types of optical high pressure cells

Type of cell	Pressure (kbar)	Advantages	Disadvantages
Shock wave	>>1000	1. Highest obtainable pressures	1. Pressure exerted over short time 2. May be destructive to optical equipment and sample 3. High explosives needed to obtain shock wave
Piston and cylinder [a]	180	1. Largest specimen volume 2. Gives more nearly hydrostatic pressures 3. Can be used for liquids or solutions	1. Insufficient optical clarity to permit microscopic observation or photography of sample 2. Specimen may interact with salt matrix 3. Difficult to use
Opposed anvils (diamonds)	1700	1. Only micro quantities of material necessary. 2. Compact — can be used with spectrophotometers and microscope easily 3. Can use for liquids or solutions 4. No matrix interference	1. Pressure gradient exists in cell 2. Absorption of diamonds may be troublesome 3. Cannot use large specimen of solids

[a] Or modifications thereof.

cells that are in use for optical studies of solids and liquids or solutions.

The piston-cylinder cell is considered to give essentially hydrostatic pressures, an important consideration in pressure studies. The Drickamer cell has two versions, one capable of providing pressures up to 60 kbar and the other to 200 kbar. Figure 1 shows the design of one of these cells. The pressure is exerted perpendicular to the light path. Sodium chloride serves as the spectroscopic window and the pressure-transmitting fluid.

The sample is loaded into the center of a small disc of NaCl. Although the cell accommodates the largest possible sample specimen for pressure measurements, the sample is surrounded by a salt matrix. If one wishes to study pressure effects on ionic solids, this could present a problem. Additionally, the hygroscopic nature of the salt matrix is detrimental. Other materials besides NaCl may be substituted for the center disc. This has the disadvantage in that it would shorten the life of the NaCl windows, and the matrix problem would always exist.