

Fig. 1 Microsectioning procedure - contour

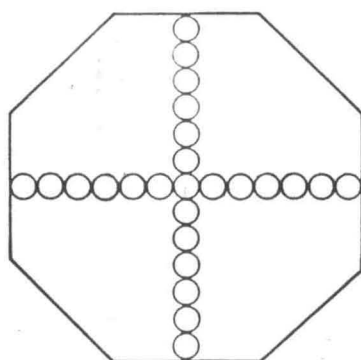


Fig. 2 Microsectioning procedure - gradient

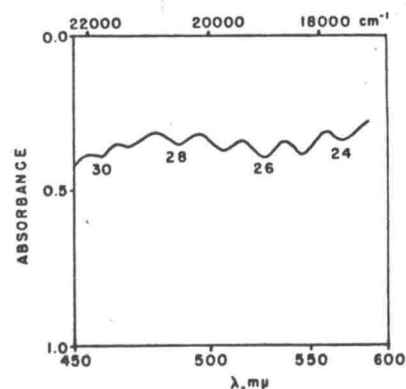


Fig. 3 Interference fringes from diamond cell

of the magnitude and especially the shape of the pressure distribution in high-pressure cells is indicated before very reliable state and spectroscopic data can be obtained using high-pressure cells with a pressure gradient.

EXPERIMENTAL METHODS

A microscope spectrophotometer described elsewhere (13) has been built for the determination of the spectra of selected areas of substances mounted in the diamond anvil high pressure cell. The physical dimensions of the cell make necessary the use of long-working-distance optics. Conoscopic optics normally used with the universal stage satisfy this requirement. Using these it is possible to select an area as small as 10^{-7} cm^2 . The area of the diamonds used in our experiments is $3.2 \times 10^{-3} \text{ cm}^2$. Thus, 3000 spectral determinations would be required to completely cover the diamond area under these conditions. Since 50-100 measurements are enough to characterize properly the pressure distribution within the cell, we use scan areas of approximately $4 \times 10^{-5} \text{ cm}^2$. Fifty-seven such circular sectors are taken from each specimen as indicated in Fig. 1.

After it was established that the pressure distribution was symmetric about the center of properly aligned diamonds, the sampling procedure was simplified to a pair of diametric measurements, with 17 such measurements from the scheme shown in Fig. 1 or by the use of 25 microsections with reduced area as shown in Fig. 2. Care was taken to get a reasonably good alignment of the diamonds. Microsections with an air pocket were avoided, as the spectra taken from such areas were not quantitatively correct and perhaps not qualitatively accurate.

Use was made of the larger of these voids, however, since the spectral pattern obtained often

has a sufficient interference pattern that the spectral positions of the fringes could be determined. Such fringes are often observed owing to reflections from parallel plates (in this case the diamonds) (14). The order, N , of the interference fringes can be assigned from the observed frequencies since

$$N/N + 1 = \lambda_N / \lambda_{N+1}$$

The thickness, t , of the air layer (and hence the sample thickness) can then be calculated by the relation

$$t = N/2 n_D N$$

where n_D is the index of refraction of the air. This procedure could be used only when the void was sufficiently large and the conditions for interference were met. Sample thicknesses of from 8 to 15 were so measured. An example of the typical interference pattern is given in Fig. 3 along with the assigned fringe order. This pattern was obtained from a sample of nickel dimethylglyoxime diluted with 3 parts KBr at an applied pressure of 4 kbar. An attempt will be made in the future to apply the method to the measurement of sample extrusion and the determination of the deformation of the diamond anvils under pressure, although it is now doubtful that the method will be sensitive enough for the latter determination.

For pressure-distribution studies it is best to select a substance which has an absorption band which shifts with pressure. Nickel dimethylglyoxime is known to have such an absorption band at $19,000 \text{ cm}^{-1}$ and has been shown by Zahner and Drickamer (15) to have a shift of $-80 \text{ cm}^{-1}/\text{kbar}$. This shift has been verified by measurements in our laboratory, by scanning the entire sample in the diamond high-pressure cell. Thus, if we determine the peak position of the absorption band for each