

Fig. 6 Pressure hysteresis in diamond cell

fraction of a turn about the position giving the desired pressure. (This procedure probably provides somewhat the same working of the sample as the anvil in Bridgman's cell.) The reduction in the hysteresis by this method is shown in Fig. 6.

After working the sample properly in the diamond high-pressure cell, it is placed on the stage of the microscope and positioned while viewing with the stage ocular at a magnification of 125X. The sample is secured to the stage and then viewed with the microsectioning ocular (at a magnification of 180X) without the fixed aperture in position. The desired fixed aperture is slid into position and the sample image is moved with respect to the fixed aperture (with the aid of the objective centering screws) until the desired position is found. A fine adjustment on the fixed aperture slide is provided which makes this adjustment still easier. However, if one is making quantitative measurements, it is necessary to maintain the position of the fixed aperture slide so the image will be always projected onto the same position of the photocell surface. The 3-mm fixed aperture is used in the microsectioning pattern shown in Fig. 1 and the 2-mm aperture for the pattern shown in Fig. 2.

The monochromatic light source is now used to illuminate the specimen and the alignment checked again before projecting the apertured sample image directly onto the photocell for the spectral determination. The spectrophotometric scans were made from $750\text{m}\mu$ down to $400\text{m}\mu$ in the case of nickel dimethylglyoxime. This range is adequate to cover the red shift from $19,000\text{ cm}^{-1}$ to $13,500\text{ cm}^{-1}$ corresponding to pressures up to 70 kbar.

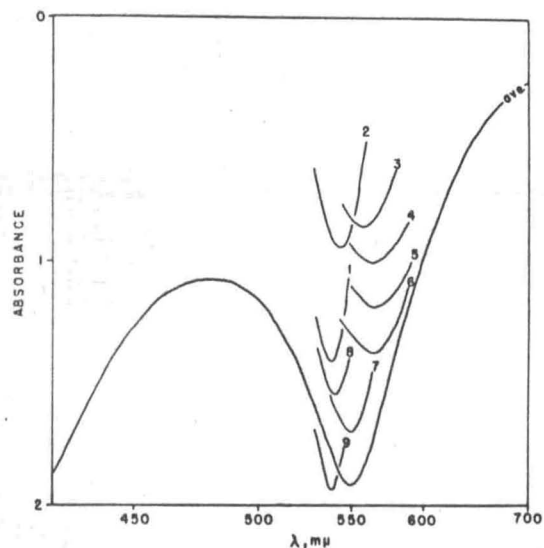


Fig. 7 Spectral data from positions along cell diameter, data taken from positions indicated in Fig. 1

A typical set of data from across one diameter of the diamond cell containing nickel dimethylglyoxime diluted with five parts of LiF is given in Fig. 7. The absorbance values are not accurate since some of the curves have been moved up or down to permit the reader to see the frequency shift with pressure.

The determination of pressure contours by the photographic method has been used with thallium bromide and nickel dimethylglyoxime. Zahner and Drickamer have reported a shift of $-115\text{ cm}^{-1}/\text{kbar}$ for the absorption edge of TlBr (16). Polaroid high speed (ASA 3000) film was used to make photos of these substances. The monochromator of the model 350 spectrophotometer was used for the illumination. The sample preparation is the same as given above except neither of the materials were diluted. The pressure contour being obtained from each photograph. The contour lines of five to ten photographs at different wavelengths were taken for each sample under pressure.

EXPERIMENTAL RESULTS

Optical observation of nickel dimethylglyoxime in the diamond high-pressure cell had led to the conclusion that the pressure decreases from the center of the cell to the edge and that the incremental change in pressure increases toward the edge.

These observations were supported by the data obtained in our early studies on nickel dimethylglyoxime diluted with 3 parts KBr. Fig. 5 is a contour map of a sample at 12 kbar mounted be-