Experimental

Virtually all the work to be reported upon has been conducted in opposed anvil-type apparatus. The general principles involved in the construction of the apparatus and the anvils themselves as well as details of the performance and calibration of such apparatus are given elsewhere.[2,3] A recent paper[3] describes various modifications and gives evaluations of different piston materials, etc. In another paper to be presented at this Symposium (see p. 77), criticism of calibration procedures and the evaluation of the opposed anvil pressure by utilisation of the Bi_I–Bi_{II} transition is presented. In apparatus of the type used, pressures up to 175 kb were reproducibly and very simply achieved at room temperature. Pressures near 150 kb have been sustained for several hours at temperatures below 400° and pressures near 100 kb may be sustained for several hours at temperatures as high as 600°.

Experimental procedure

In general, the glasses were ground to pass a 200-mesh sieve and were pre-pelleted as a very thin wafer of approximately 8 mils thick into a nickel ring of the same thickness (see Dachille & Roy[2a]). The wafer is surrounded on both sides by platinum–rhodium discs so that the entire system is quite free from contamination except by platinum or nickel. The system is usually taken to the derived temperature and the pressure applied for a short period ranging from $\frac{1}{2}$ minute to a few minutes. The pressure is released and then the temperatures lowered rather quickly. For certain runs it was necessary to make a series of experiments with times extending into 24 or 48 h. under these same conditions. In most cases, the glasses were recovered as a continuous transparent film which could then be examined for certain optical properties as a film or crushed for measurement of density and/or refractive index, etc. Densities were determined by a simple procedure with the usual heavy liquid media and centrifugation. Refractive indices were measured by the Becke line technique under a polarising microscope. Infrared spectra were obtained with the Perkin Elmer, Model 21, and visible spectra with a Beckman DK 2 spectrometer. For the studies on the kinetics of crystallisation, quantitative *X*-ray diffraction procedures were employed with use of a Norelco diffractometer, and standardisation with mixtures of the glass and the crystalline phase being formed.

Results

Data on densification of glass

Fig. 1 shows a graph of the refractive index of silica glass plotted as a function of the pressure to which the sample was exposed. It should be made quite clear that this is a refractive index of the glass after the entire quenching procedure and removal from the sample holder, etc. It is not, of course, the refractive index at pressure. Through frequent repetition and through storage of the samples for over 2 years, it is quite clear that there is no change in the silica glass caused by normal processing in the examination procedure. The variation in the refractive index of the product glass, if the sample is treated as a whole, can be quite large, ranging up to ±0·006, although most of the material may fall within a much narrower range. It appears in the data in Fig. 1 that silica glass may be compressed to a new structural form with a linearly increasing density and refractive index, depending on the pressure. The variation of refractive index across the sample is undoubtedly due to inhomogeneities in the pressure. Although the glass, when removed from the sample chamber, is indeed birefrequent, the change of refractive index is quite

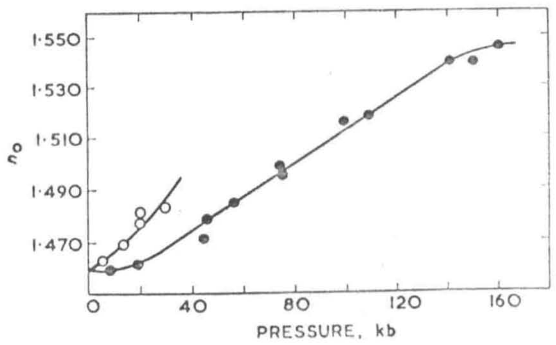

Fig. 1. *Refractive index of silica glass measured at atmospheric pressure and room temperature as function of the pressure of the run*
○ 600° ● 25°