Nature and uniformity of pressure

An extensive study in this laboratory has been made to use various ' fixed points ' in reaction equilibria as well as electrical resistivity measurements to find correlations in results with those obtained in other types of apparatus. In normal use, when the diameter-to-thickness ratio of a sample wafer assembly is about 25 : 1, the pressure distribution has been shown to be uniform. This has been demonstrated[10] in experiments where the ratio of the area of the metal ring to area of sample was varied from about 4 : 1 to 0 : 1 at the equilibrium pressure for the quartz–coesite reaction at 500°. The results showed that, for this wide range of sample distribution the transition pressure was within 0·4 kb of the 20·4 kb average for all cases. With this type of sample, univariant *P–T* equilibrium ' curves ' (Fig. 10) have been obtained for several important solid-state reactions. In some cases, where overlap permitted, these curves were carried down to low pressures where they join the hydrostatic pressure region. Furthermore, Bridgman's synthesis of the waxy ' polymer ' of CS_2 in liquid-pressure apparatus at 42 kb was duplicated by us at the same pressure in the anvil apparatus.

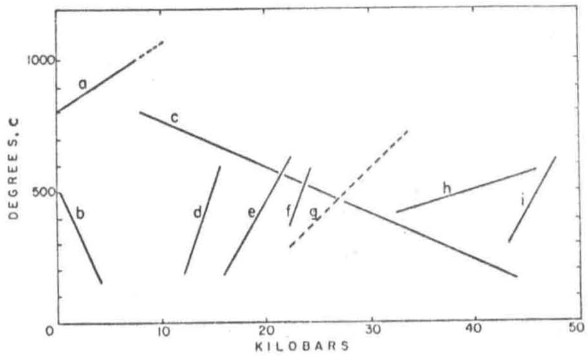

Fig. 10. ***Examples of univariant* P–T *curves obtained with our opposed anvil apparatus***

The dashed line is that for the theoretical graphite–diamond equilibrium. In order, they are (a) Mg_2GeO_4 olivine–spinel; (b) PbO litharge–massicot; (c) $ThSiO_4$ thorite–huttonite; (d) PbO_2 I–II; (e) SiO_2 quartz–coesite; (f) B_2O_3 hexagonal–monoclinic; (g) C, graphite–diamond; (h) $HoVO_4$ xenotime–scheelite; (i) BPO_4 cristobalite–quartz

Myers *et al.*[9] found that the pressure distribution changes radically (at temperatures below 200°) as the diameter thickness ratio is reduced. This one factor probably explains most of the variability obtained by some investigators. This behaviour is further modified by changes in the geometry and in the relative hardnesses of the materials of construction of the wafer assembly, and must be taken carefully into account both in experimental procedures and in reporting results which might be applied to calibration.

In Fig. 11 are shown the results of this study of pressure distribution in a relatively thick sample assembly (diameter/thickness ratio about 9). The assembly was identical in each run except for the placement across the diameter at different points of a short length of bismuth wire (0·025 in. dia.) whose I → II transition was used as a pressure indicator (25·4 kb[10]). The figure shows that at the centre of the sample the transition was obtained when the average pressure on the anvil face was only 11·5 kb. However, a calibration wire placed about 1/2 radius out from the centre went through the transition when the average load was 18 kb. The change in stress distribution at the metal–pyrophyllite boundary is probably due to the differences in the flow properties of the materials, causing a break in the continuity of compacted solids at the boundary. The relatively uniform distribution of pressure for a ratio of 14 in. is shown in Fig. 12.

However, if the bismuth wire is placed in the centre, the thickness of the wafer assembly being changed, it is seen from the results summarised in Fig. 12 that as the diameter thickness ratio approaches 16 : 1 the average stress approaches the transition pressure. (Photoelastic models of sample wafers also illustrate the general patterns of stress distribution observed in our direct experiments.)

Temperature measurements

Normally rather little is said about temperature measurements in opposed-anvil experiments because of the simplicity of the setup. This was pointed out by Griggs & Kennedy and has been found to be the general experience of all users of similar equipment. As indicated in Fig. 1, a thermocouple is looped on the shoulder of an anvil with the couple end against the edge of the sample. The assembly of anvils and thermocouple is then enclosed in loose-fitting, thick sleeves of stainless steel or equivalent. The temperature of the sample is taken as the temperature