

reactant plug and contact disk. A sectioned assembled unit is directly to the right, followed by a nylon sleeve which provides electrical insulation for the portions of the high pressure pistons extending into the high pressure cylinder bore. The above configuration has been found suitable for reaction studies because of its simplicity, and because reaction surfaces are broad and essentially isothermal. Where reaction between the reactants under study and pyrophyllite occurs, either a chemically inert protective sleeve or the arrangement shown in the bottom series of the

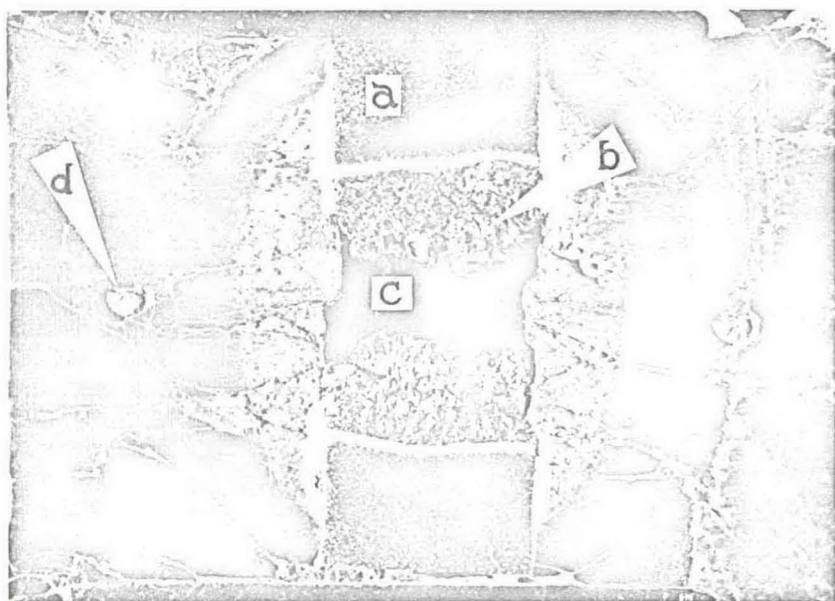


FIG. 4. A cross-sectioned specimen recovered from a supported stepped piston-cylinder apparatus after being subjected to high pressure and high temperature. Synthetic diamond can be observed at the metal-graphite interface. Width of the section is 12.5 mm.

Figure is used. The graphite—pyrophyllite interface is non-reactive. Contamination by diffusion through the graphite, however, must be considered. Since the basic geometry illustrated both for reactants and passive constituents is universally applicable to all present types of static pressure equipment, and is suitable for diamond growth, other more sophisticated techniques developed for special effects will not be described.

Figure 4 shows a cross sectioned complete specimen recovered from the pressure vessel. The constituents from center top to bottom are: (a) un-

reacted iron; (b) iron carbides<sup>1</sup> and diamond; (c) unreacted graphite. The thermal environment is illustrated by the light gray elliptical zone surrounding the reactants. Here pyrophyllite has been altered to coesite and kyanite (Giardini *et al.*, 1961). The pressure measuring circuit is shown at (d).

Figure 5 provides a view of a core recovered from an arrangement illustrated by the bottom row of Fig. 3. The reactants consisted of an iron rod placed within a graphite tube. The latter were both placed within a thin walled iron sleeve. Area (a) shows diamond crystals covered with unreacted graphite. Intermixed and behind the diamond are iron carbides.<sup>1</sup> Zone (b) constitutes a central core of iron silicate.<sup>2</sup> The latter formed as a result of silica diffusion from the outer pyrophyllite cylinder, and provides a graphic example of the surprising mobility generally encountered in solid state reactions carried out at high pressures and temperatures.

Analytical work has been performed by the following techniques: (a) optical microscopy, (b) electron microscopy, (c) emission spectroscopy, and (d) x-ray diffraction and fluorescence.<sup>3</sup>

These studies have shown that the mechanism of diamond synthesis under conditions of static high pressure and high temperature involving multi-component systems, is one of solution of carbon in a suitable solvent to a state of supersaturation. The thermodynamic instability of the resultant solution requires decomposition, and yields subsequent crystallization of the precipitated free carbon. Whether the latter crystallizes as diamond or graphite appears primarily dependent upon the local pressure-temperature environment. Theoretical data on the diamond-graphite equilibria derived by Berman and Simon (1955) have been experimentally verified within specified limits of probable error (Figs. 9 and 10). The minimum temperature for diamond growth at a given pressure in any usable system is defined by its carbon saturation limit. Some minimum values of pressure and temperature are given in Table 1.

The behavior of the metal carbon solvents listed above falls into two general categories: those prone to form stoichiometric carbon compounds (carbides), and those which are not. Examples of the former are Fe, Mn, Cr, and Ta. Pt, Ni and Co exemplify solvents not prone to form carbides.

<sup>1</sup> It may be of interest to mention here that the highest carbon iron carbide observed prior to diamond formation corresponds to a chemical formula of FeC. The material possesses a higher physical hardness and chemical stability than the common carbide of iron, Fe<sub>3</sub>C.

<sup>2</sup> Characterization is not yet complete. The weight ratio of Fe:Si is approximately 1:1. Additional data will be published later. In general, it resembles muscovite.

<sup>3</sup> See accompanying article by J. A. Kohn and D. W. Eckart.