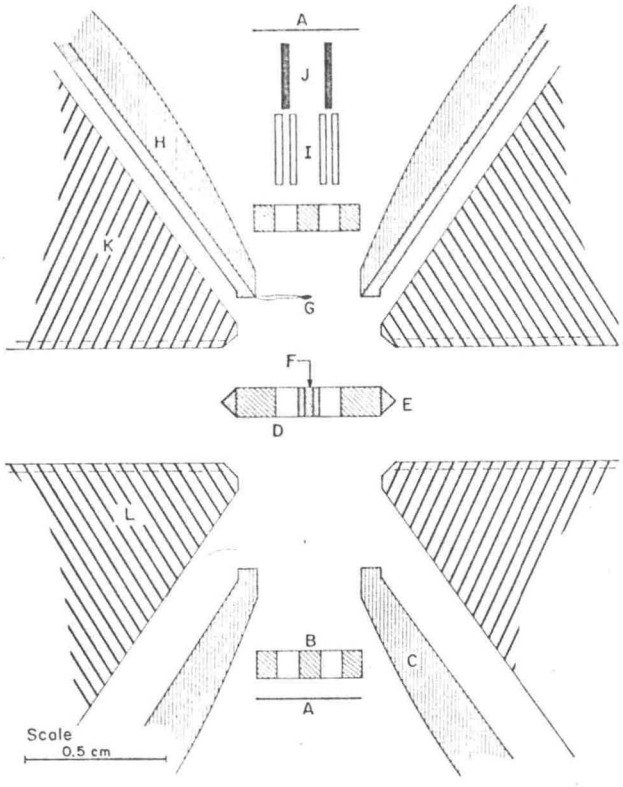

FIG. 4. Exploded view of internal components comprising the high-pressure volume of the apparatus. A—metal disk; B—baked pyrophyllite end pieces; C—pyrophyllite gasket; D—boron disk; E—Be ring pressure seal; F—sample; G—thermocouple junction; H—split pyrophyllite gasket; I—BN tubes; J—carbon heater rods; K—upper die half; L—lower die half.

Be ring fits in the taper and is supported by the die at all points except where the slots and fans have been removed. The ring and tapers on the die halves are shown in the cell assembly drawings (Figs. 4 and 5). At high internal pressures the ring is extruded slightly into the slot and fan regions but the ring seal has never failed by blowing out. The advantages of the Be ring seal are its low x-ray absorption coefficient (less than 10% loss) and high-temperature stability. Due to the slight flow of the ring, it is necessary to replace it after each run. Internal temperatures of 1000°C have been obtained with this method. Higher temperatures were not attempted since the steel die yields readily if the temperature of the die bore gets too high.

The medium surrounding the sample was a pressed amorphous boron pellet 0.51 cm diam by 0.1 cm thick. The sample is packed in a 0.03 cm hole drilled in the center of the pellet. Two carbon rods (0.038 cm diam) are placed in holes on either side of the sample hole and current is passed through the rods from piston to piston. To protect the carbon rods from the boron, they are sheathed with boron nitride tubes. A thermocouple junction of Chromel–Alumel or Pt/Pt–10% Rh is placed directly over the sample hole in the boron pellet. The temperature is therefore determined at a point less than 0.05 cm from the center of the compressed sample and less than 0.025 cm above the x-ray beam. The thermocouple is separated from the piston face by 0.075 cm of pyrophyllite.

PRESSURE MEASUREMENTS

Sample pressures are determined by calculating the change in the lattice parameter of NaCl which is intimately mixed with the sample under study. The equation of state of NaCl given by Decker[10–11] provides pressure calibration over the pressure–temperature range of 0–500 kilobars and 0–2000°C. An internal calibrant of this type is essential for accurate pressure–temperature studies. Decker's equation of state has been compared with other equations of state of NaCl by McWhan.[12] A problem involving recrystallization and grain growth of NaCl exists above 400°C. This results in spotty x-ray patterns that are difficult to read accurately. The problem can be minimized by diluting the NaCl with boron to separate the crystallites from each other.

X-RAY TECHNIQUES

The alignment of the x-ray beam through the die is accomplished by adjusting the press–die position relative to the fixed x-ray beam direction. Figure 1 shows the die in the press and the x-ray source facing the press. The entrance groove (0.05 cm diam circular cross section) is 5 cm long and provides the collimation. The press sits on an adjustable table which levels the press, moves it laterally, and rotates it about the x-ray entrance hole at the front of the die. The x-ray beam is observed by a phosphor painted on the die around the entrance hole. The

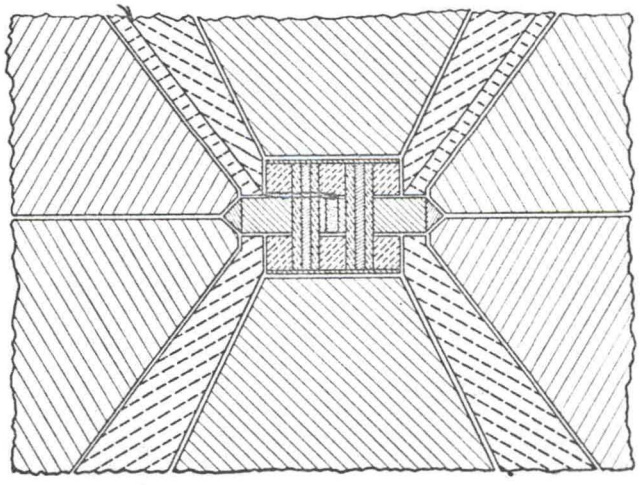

FIG. 5. Schematic diagram of assembled internal components filling the high-pressure volume of the apparatus.

[10] D. L. Decker, J. Appl. Phys. 36, 157 (1965).
[11] D. L. Decker, J. Appl. Phys. 37, 5012 (1966).
[12] D. B. McWhan, J. Appl. Phys. 38, 347 (1967).