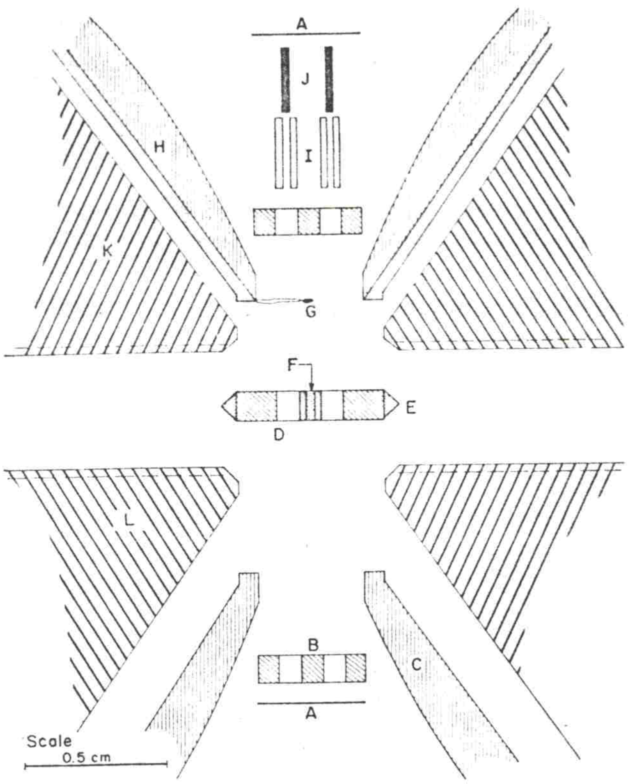


FIG. 4. Exploded view of internal components comprising the high-pressure volume of the apparatus. A—metal disk; B—baked pyrophyllite end pieces; C—pyrophyllite gasket; D—boron disk; E—Be ring pressure seal; F—sample; G—thermocouple junction; H—split pyrophyllite gasket; I—BN tubes; J—carbon heater rods;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

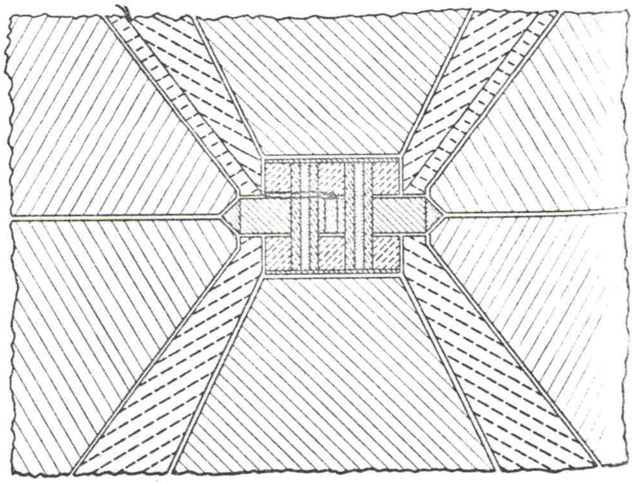


FIG. 5. Schematic diagram of assembled internal components filling the high-pressure volume of the apparatus.

[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).

emerging undeviated beam is detected at the exit slit with a Geiger–Müller counter when alignment has been attained. Press movement relative to the x-ray source does not affect the alignment of the sample relative to the x-ray beam since the dies, of which the sample is a part, do the collimating.

Powder diffraction patterns are recorded using standard x-ray film and a Debye–Scherrer geometry. The film cassette consists of two coaxial semicircular cylinders between which the film is sandwiched with a rubber gasket. The inner cylinder has a 0.6 cm high slit around the circumference to allow x rays to reach the film. Aluminum foil is placed between the film and slit to provide a light seal. The film cassette is clamped to the o.d. of the die, as shown in Fig. 6. This locates the film on a precise 57.3±0.03 mm radius as measured from the die center. At full load, the o.d. of the die expands less than 0.02 mm making the o.d. a stable reference distance for the film position. The film cassette can be removed from the press after each exposure without disturbing the sample alignment because it is the sample position relative to the die which determines the alignment.

For lattice parameter measurements, the precision of the split-die device is comparable to that of Bridgman anvil x-ray and tetrahedral x-ray devices (±0.2 to 0.4% depending on sample). Possible errors arise from film to die-center distance, sample positioning, and sample shifting. The film to die center is known to an accuracy of ±0.003 cm and the film is coaxial with the die center to that accuracy. In addition, the fan edges make sharp images on the film as is shown in Fig. 7. The angles of the fan edges relative to

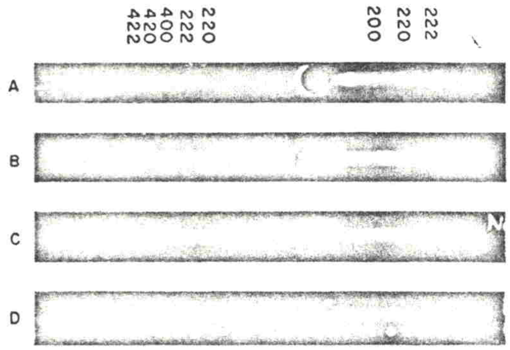
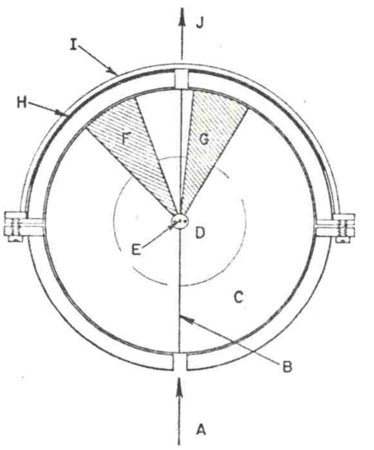


FIG. 7. X-ray powder diffraction patterns of NaCl under high pressure and temperature. A—14 kilobars, 25°C; B—53 kilobars, 25°C; C—70 kilobars, 600°C; D—60 kilobars, 800°C. Note the spotty patterns at high temperatures due to large grain size.

the x-ray beam are measured after they are ground to an accuracy of ±0.01° and thus can provide a precise determination of the center of the diffraction pattern and film shrinkage.

"Postmortem" microscopic examination of sample position indicates that shifts from center are less than 0.01 cm. The diffraction lines have a width which is proportional to the sample diameter inasmuch as the beam diameter is larger than the sample. Therefore, for precision measurements, the sample diameter is made as small as possible (less than 0.03 cm). Typical patterns of NaCl are illustrated in Fig. 7. Six lines are visible (200, 220, 222, 400, 420, 422) with the 220 and 222 lines appearing in both the 5–30 and 20–45° slots. The exposures were for 5–15 h using a Jarrell–Ash microfocus x-ray unit with Mo target (3 mA at 50 kV).

FIG. 6. Top view of mating surface of one-half of the split die. A—x-ray beam; B—beam entrance groove; C—die support ring; D—die; E—sample; F—20 to 45° diffraction slot; G—5 to 30° diffraction slot; H—x-ray film; I—film cassette; J—x-ray beam egress.



ADAPTATION FOR OTHER POSSIBLE USES

The split-die design might also be adapted for other kinds of studies. The apparatus is suited for high-pressure Mössbauer experimentation. The solid angle to the high-pressure region is large enough so that experiments with the absorber under high pressure are feasible. The high-temperature capability also makes the device unique for high pressure Mössbauer studies.

For high-pressure high-temperature optical studies, various windows could be cemented into the fan regions as was the epoxy for the x-ray application. Since the plain epoxy seal proves an effective pressure seal, a hard window material should be even more effective.