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

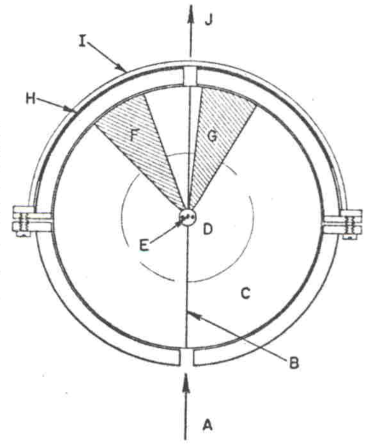

FIG. 6. Top view of mating surface of one-half of the split die. A—x-ray beam; B—beam entrance groove; C—die support ring; D—die; E—sample; F—20 to 45° diffraction slot; G—5 to 30° diffraction slot; H—x-ray film; I—film cassette; J—x-ray beam egress.

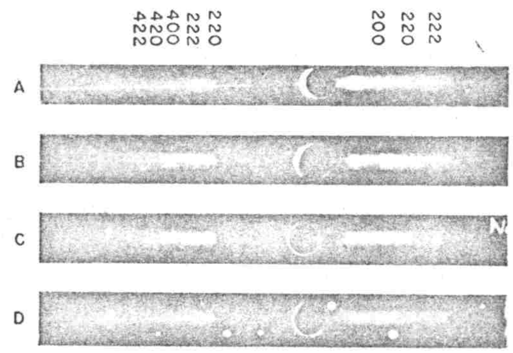

FIG. 7. X-ray powder diffraction patterns of NaCl under high pressure and temperature. A—14 kilobars, 25°C; B—53 kilobars, 25°C; C—70 kilobars, 600°C; D—60 kilobars, 800°C. Note the spotty patterns at high temperatures due to large grain size.

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).

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.