of interference. The faces are ground parallel. The die is made of tool steel to facilitate the grinding operations described below. A higher pressure range could probably be attained with the use of tungsten carbide.

An entrance–exit groove is ground across the mating surfaces of the two halves to a depth of 0.025 cm with a grinding wheel dressed to a 0.025 cm radius. The groove is centered on a diameter of the die and passes through the die center to within ±0.001 cm. A fan-shaped slot for diffracted rays is ground into the assembly on each side of the groove. The fan on one side of the groove covers the 2θ diffracted angles 5 to 30° and the fan on the other side covers 2θ angles 20 to 45°. This provides a range of measurable "d" values with Mo K_α radiation from 8.1 to 0.93 Å with overlap of the two slots from 1.37 to 2.04 Å. The fans have a vertical taper of 2°, which, at a film distance of 57.3 mm, gives an x-ray pattern 3 mm high. An initial flat region 0.025 cm deep is left in the fan for a distance of 1.25 cm from the bore center before the vertical taper is started. The flat region aids in effecting the pressure seal. The two halves are placed together and aligned by placing a 0.05 cm drill rod in the entrance and exit groove.

Two methods are used to prevent extrusion of the pressure medium into the slots and grooves. The first method employs epoxy resin to fill the slots and grooves to a distance of 0.5 cm from the periphery of the bore of the die. A clear epoxy loaded with 50–70% by weight of amorphous boron is suitable. An excess of the mixture is applied and then lapped parallel to the die face after curing. The epoxy–boron mixture has a linear absorption coefficient for Mo K_α x radiation of approximately 1.0 cm^{-1}, which results in an attenuation of intensity for the described configuration of 65%. When using the epoxy seal the internal temperature of the sample is limited to 500–600°C, since the temperature of the bore is approximately one-third the internal temperature and the epoxy begins to soften above 200°C.

The second method of sealing the pressure is by means of a beryllium ring with a wedge-shaped cross section. The bore is tapered where the two die halves mate so that the

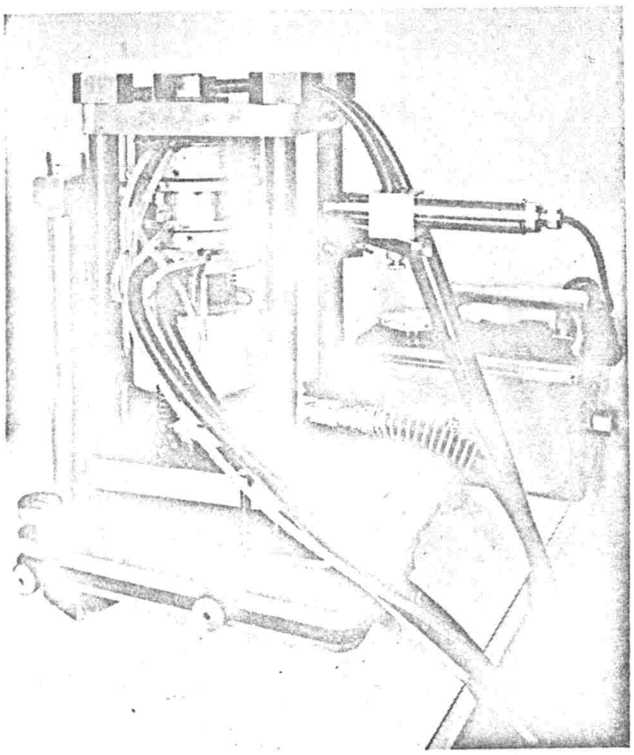

FIG. 1. Photograph of the high-pressure apparatus showing the ress frame and hydraulic ram, the positioning table, the x-ray tube ad (left), and the Geiger tube for aligning the press. The ebye–Scherrer camera is not in position so that one of the slots for e diffracted rays is shown. The scale is in centimeters.

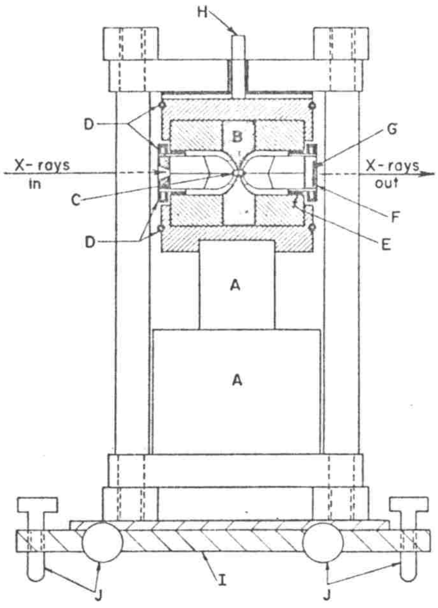

FIG. 2. Schematic drawing of the high-pressure apparatus. A—hydraulic 50-ton ram; B—WC pistons; C—die assembly; D—water cooling tubes; E—rubber shim to position die water-cooling tube; F—film cassette; G—x-ray film; H—insulated current lead; I—press positioning table; J—adjusting screws for vertical and horizontal positioning.

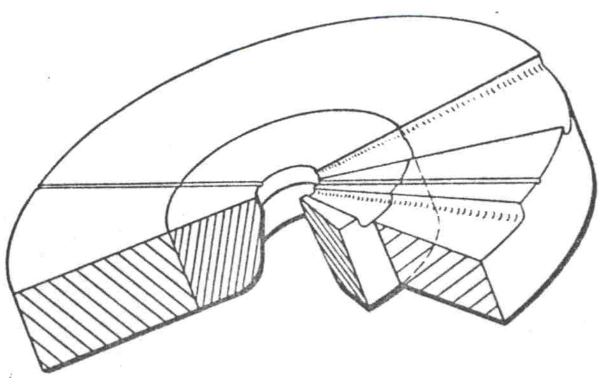

FIG. 3. One-half of split-die assembly showing mating surface with groove for entrance of x-ray beam and egress of undeviated beam and fan-shaped slots for egress of diffracted rays.