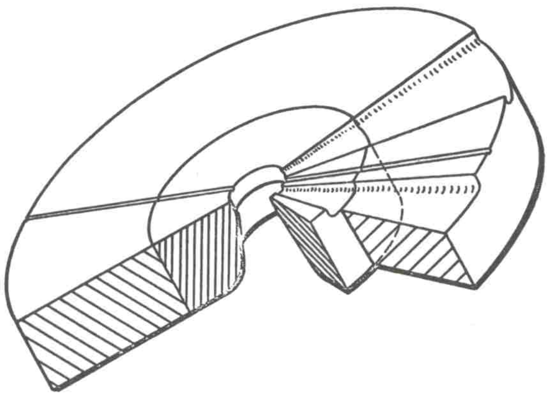

Fig. 2. One-half of the split-die assembly showing mating surface with groove for entrance for X-ray beam and exit of undeviated beam and fan-shaped slots for exit of diffracted rays.

The fan shaped slots allow diffracted X-rays to emerge from the high pressure regions. They cover the 2θ diffraction angles of 5 to 30° and 20 to 45° when using dispersive analysis. With MoKα radiation one measures a d-value range of 8 to 0.9 Å. For the dispersive analysis technique a film cassette is positioned around the outside diameter of the die; this provides an accurate and stable reference of sample to film distance of 114.8 mm.

Two methods are used to prevent extrusion of the high pressure medium into the grooves and fans. Epoxy loaded with amorphous boron is placed into the fans and grooves for a distance of 0.5 cm from the die bore. Alternately, a beryllium ring of wedge shaped cross section is placed in a similarly shaped taper ground into the bore edge. Both methods contain the pressure medium to sample pressures of 100 kilobars.

The details of the high pressure cell are shown in Figure 3. The relatively large high pressure volume makes possible internal heating, and temperatures greater than 1000°C are attainable. Carbon rods sheathed in boron nitride tubes provide resistive heating when current is passed through them from piston to piston. A thermocouple monitors the sample temperature and is brought out through a split lavite gasket. The pressure medium is a pressed amorphous boron disc with the sample packed in a 10 mil hole at the center. A beryllium ring is shown here providing the pressure seal. The X-ray beam would pass through the sample in a direction perpendicular to the plane of the figure and between the carbon heater rods.

Non-Dispersive Technique

For non-dispersive analysis, the sample is irradiated with white radiation and an energy-intensity analysis is made of the X-rays diffracted at a fixed angle from the incident beam. From Bragg's equations written in terms of energy instead of wavelength, the angular and energy range requirements can

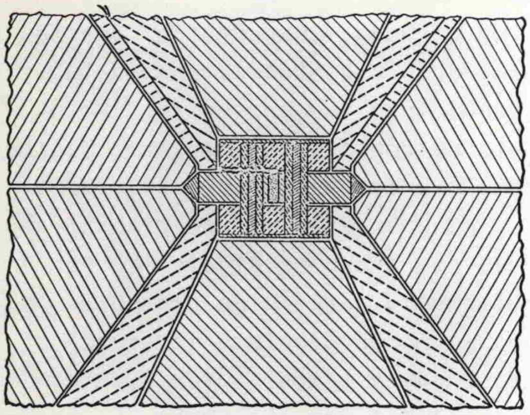

Fig. 3. Schematic diagram of assembled internal components filling the high-pressure volume of the apparatus.

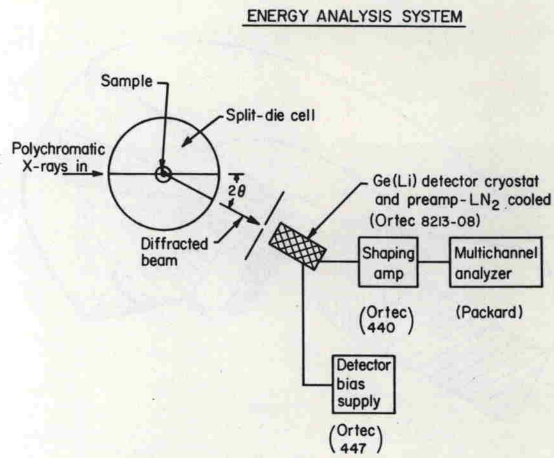

Fig. 4. Energy analysis system.

be calculated for a given range of d-values of interest.

$$\lambda = 2d \sin \theta = \frac{12.40}{\text{Energy}} \text{ (kilovolts)}$$

For example at an angle of 15° = 2θ and with a useful energy range of 15-50 Kev the d-value range of 3.2 to 0.9 Å will be observed. By using different angles different d-value ranges may be obtained for a fixed energy range of white radiation.

The split die system was modified by placing a narrow slit along the outside diameter of the die opposite one of the diffraction fans and at a fixed angle from the entrance exit groove, Figure 4. White X-radiation was produced in the microfocus unit with the use of a tungsten target run at 55-60 kv and 6 ma. The diffracted radiation was analyzed with an Ortec lithium drifted germanium detector placed behind the slit. The associated equipment consists of a dewar to cool the detector, preamplifier located in the detector housing, shaping amplifier, detector bias supply, and multichannel pulse height analyzer. The natural line width of the detector system is about 500 eV, FWHM at 50 keV. The angular spread of the diffraction geometry produces a $\Delta 2\theta$ of 1/2° and thus 1000 eV line width.

To monitor the intensity-energy distribution of the white radiation, the detector is rotated behind the exit-groove of the split die system. This will provide a