Rh thermocouple into the one atmosphere form of Li_3BN_2 in the cell and plotting temperature versus time on a recorder and noting the break in the curve. The temperature was increased at a rate of about 200°/min up to $1000^\circ C$, 400°/min to $1300^\circ C$ and 500°/min above $1300^\circ C$; the variable rate is primarily a function of rapid changes in the heater characteristics above $1000^\circ C$. Heat effects were observed for the melting of the high pressure phase. The thermal discontinuity upon melting is accompanied by a physical break in the thermocouple since the noble metals react

FIG. 1

Pyrophyllite-Tantalum Cell for High Pressure Studies Using the "Belt" Apparatus
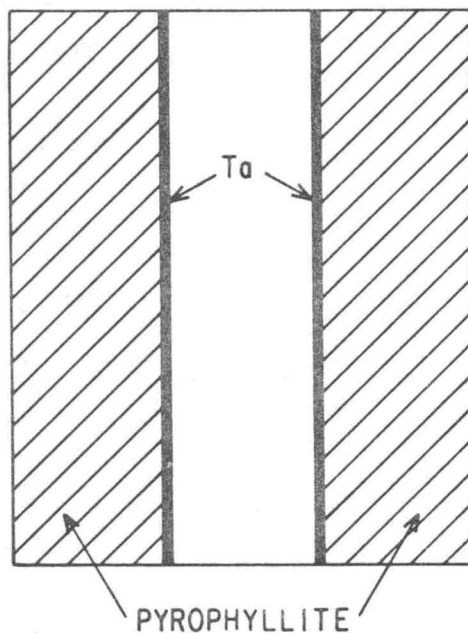
rapidly with the lithium in the melt. The data taken by this technique were reproducible within $\pm 25^\circ C$ and aided in the interpretation of quench data near the melting curve.

Measurement of Temperature and Pressure

The commonly accepted practice of using a pressure calibration made at room temperature and assuming some validity for this calibration at high temperatures was followed. The cell core, normally occupied by Ta and the sample, was filled with AgCl. Wires of Bi, Ba and Tl were inserted along an axial hole in the AgCl and were used as a part of a bridge circuit to detect the changes in resistance which occur in the materials at 25, 58, and 37 kb, respectively. The plot of these values versus psi on the ram was used to determine pressure.

Temperature measurement was based on the reproducibility of the relationship between temperature and the power requirements of the heater. To determine the watts versus temperature curves for the cell at various pressures, Pt-Pt/10Rh thermocouples were centered in the cell and the graphitic form of BN was packed around the bead. Corrections to the emf as a function of pressure were made (5), and these corrected curves were used in subsequent runs to determine the temperature.

Identification of Phases

The phases in the quenched sample were characterized by both optical and x-ray diffraction techniques. By both methods the two quenchable forms of Li_3BN_2 are easily distinguished. The agreement between our x-ray data for the high pressure form and that of Wentorf's "complex" (2) is sufficiently close to ascertain that the "complex" is in reality the high pressure modification of Li_3BN_2. (Table 1). This phase is labelled $Li_3BN_2(W)$ in Fig. 2. Our x-ray data for the one atmosphere form agree well with those of Goubeau and Anselment (3). (Table 1).

When observed with a petrographic microscope in transmitted light, the one atmosphere form of Li_3BN_2 appears as equant grains with refractive indices greater than 1.700. This phase appears to be biaxial negative with a large 2V. The refractive index of the high pressure form, $Li_3BN_2(W)$, is also greater than 1.700, but this phase is uniaxial positive and is easily distinguished by its lath-like or prismatic habit and its parallel extinction. The higher refractive index is along the length of the laths (length slow) as determined with a Berek compensator. These crystals are also pleochroic with the stronger absorption parallel to the lower refractive index; i.e., at right angles to the length of the laths. Both the thermal and mechanical breakdown of this phase appears to take place preferentially along the plane perpendicular to the length of the laths, i.e. it has a preferential parting or weaker bonding perpendicular to the "c" axis. This combination of optical, mechanical, and thermal properties suggests a layer-like structure for $Li_3BN_2(W)$.

As a first approximation it is reasonable to assume that the structure of Li_3BN_2 at one atmosphere would be closely related to