

## THE SYSTEM $\text{Li}_3\text{BN}_2$ AT HIGH PRESSURES AND TEMPERATURES

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### ABSTRACT

The system  $\text{Li}_3\text{BN}_2$  was studied over the pressure and temperature range from 10 to 65 kb and 300-1900°C, respectively. The stability region of a quenchable high pressure modification,  $\text{Li}_3\text{BN}_2(\text{W})$ , was defined, and the data also suggest the possibility of another nonquenchable high pressure modification.  $\text{Li}_3\text{BN}_2(\text{W})$  is the phase which appears to be in equilibrium with borazon (cubic BN) during growth of this phase from the system  $\text{Li}_3\text{N-BN}$ , and the P-T data obtained on the melting of  $\text{Li}_3\text{BN}_2(\text{W})$  are useful for defining the growth conditions for borazon.

### Introduction

Within the pressure-temperature space of the system Li-B-N is a volume in which cubic BN (borazon) is stable at high temperatures and pressures (1,2). One of the phases intimately associated with borazon when "quenched" from this system is the "complex" reported by Wentorf (2). One of the results of the present study is that this "complex" is a high pressure form of  $\text{Li}_3\text{BN}_2$ . This paper is concerned with a study of the P-T conditions for stability of both the one atmosphere and the high pressure modifications of this compound. The results suggest the possibility of a second high pressure polymorph.

### Experimental

#### Starting Compositions

Mixtures were made from  $\text{Li}_3\text{N}$  (Foote Mineral Co.) and BN

(graphite form, 99.9%, Gallard Schlesinger) in 1:1 mole ratio. Quench runs were made both with unreacted mixtures and with the one atmosphere 1:1 compound made by the reaction of the nitrides using the method of Goubeau and Anselment (3). The results were identical with either starting material because  $\text{Li}_3\text{BN}_2$  forms readily in the high pressure cell during the heating process. Thermal analysis runs were made only with the one atmosphere form of  $\text{Li}_3\text{BN}_2$ . Those making this compound should be cautioned about the strong exothermic reaction at  $650^\circ\text{C}$  associated with the formation of this compound, and the nitride mixture should be heated slowly (about  $50^\circ\text{C}/\text{hour}$ ) through the temperature range  $600\text{-}750^\circ\text{C}$ .

### Cells

All experiments were done in pyrophyllite-tantalum cells as shown in Fig. 1 using the "belt" apparatus (4). The tantalum tube (0.430" x 0.127" OD x 0.116" ID) is simultaneously the container and resistance heater and probably acts as a getter for hydrogen and oxygen from the decomposition of pyrophyllite. No evidence for nitrides or borides of tantalum was found. A flame test on a tantalum heater after a run shows that the lithium contamination, if any, is small.

Since there is a steep temperature gradient in the cell from ends to center, all results were based on material taken from a nearly uniform temperature region 1 to 2 mm high at the center of the cell. The separation of a homogeneous sample presents some problems.

### Quench Run Procedure

Most of the data on phase changes were obtained by the following procedure. Pressure was applied to the cell to the desired value and then the temperature was raised at a rate of about  $400^\circ\text{C}$  per minute. Temperature and pressure were held at the predetermined levels for 10-15 minutes, the power to the cell was switched off, and quenching to room temperature was accomplished in about 30 seconds with the pressure still applied. The pressure was then released.

### Thermal Analysis Runs

Thermal analysis data were obtained by inserting a Pt-Pt/10-