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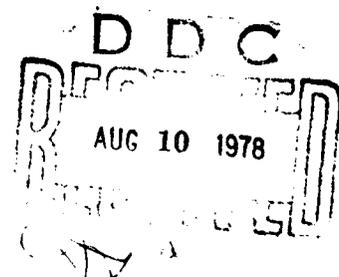
LITHIUM-BORON ALLOY ANODES FOR MOLTEN SALT BATTERIES (II)

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RESEARCH AND TECHNOLOGY DEPARTMENT

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SUMMARY

The lithium-boron system has been studied by differential scanning calorimetry (DSC), metallography, and X-ray diffraction. The "alloy" was found to consist of a high-melting, sintered, porous, crystalline compound with a stoichiometry of Li_7B_6 , into which has been wicked excess elemental lithium. This material exhibits both the high-energy electrochemical properties of lithium and stability in the presence of molten salts. This report describes progress toward the development of lithium-boron anode material for improved molten salt batteries.

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INTRODUCTION

Voltaic cells with calcium metal anodes discharging in molten lithium chloride - potassium chloride eutectic electrolyte are employed in thermally activated primary batteries. Such batteries have specialized uses for high-power, short-term discharges in a wide range of military and aerospace applications (1). They typically yield less than 30 percent of theoretical capacity because the calcium anode exhibits severe ohmic polarization at current densities above 0.6 kA/m^2 . Insoluble reaction products form a solid passivating barrier between the anode and the molten salt electrolyte (2) that limits the anode reaction.

The electrochemical superiority of lithium as an anode material is well-known. Its reaction product, lithium chloride, is soluble in molten salt electrolyte. Liquid lithium anodes exhibit very little polarization even at current densities up to 400 kA/m^2 (3). Practical thermally activated primary batteries with lithium anodes are capable of discharge at current densities up to 30 kA/m^2 . They employ massive and bulky porous metal or wire screen substrates as carriers for the liquid lithium (4,5,6). These anodes are subject to leakage, short-circuiting, and corrosion of cell components. The active lithium content is typically less than one-fourth of the anode assembly mass.

Studies of rechargeable solid anodes of lithium alloyed with aluminum (7,8) or with silicon (9,10) have led to their use in rechargeable molten salt cells operating at current densities less than 1 kA/m^2 . Compound formation in these alloys lowers the anode potential significantly below that of lithium. Less than one-fourth of the alloy mass is electrochemically active.

Because boron is similar to aluminum in atomic structure but has only 38 percent of its atomic mass, alloys of lithium and boron are of interest as anode materials. Early attempts to prepare lithium-boron compounds resulted in substances of high boron content generally believed to be borides (11 - 15). Data on the preparation and physical properties of lithium-rich alloys (16-19) suggested the existence in the alloys of a high-melting crystalline compound with stoichiometry near 1:1. Lithium-rich alloys discharged anodically up to molten lithium chloride-potassium chloride eutectic at current densities up to 80 kA/m^2 showed only slight polarization (20). A discharge residue corresponding to the stoichiometry Li_2B was inferred from capacity measurements.

The current work was undertaken to identify and characterize the stable compounds formed in lithium-rich lithium-boron alloys, to determine the conditions under which they form, and to provide a basis for estimating the theoretical anodic discharge capacity.

The primary experimental method employed to study the alloys was Differential Scanning Calorimetry (DSC). This thermal analysis technique, in effect, simulates the preparation procedure (controlled heating) of the alloy while measuring enthalpic effects of chemical or physical changes in the sample. In addition to DSC, X-ray and chemical analysis and metallography were used as tools in characterizing the material.

EXPERIMENTAL

A. Preparation of Lithium-Boron Alloys

Lithium-boron alloy ingots were prepared in a helium atmosphere glove box with total impurities (O_2 , N_2 , H_2O , and CO_2) maintained below 10 ppm. Lithium (Foote Mineral Co., 99.9%, ingot or ribbon) was scraped clean of impurities. Crystalline boron (Atomergic Chemetals Corp., 99.9%, lump) was ground in a mortar until it passed through a 40 mesh screen and was retained by an 80 mesh screen. The lithium and boron were then weighed into a stainless steel crucible having a detachable bottom to facilitate the removal of the finished ingot. The crucible and contents were heated with stirring with a stainless steel spatula in a furnace at about 723 K ($450^\circ C$) until the boron was completely reacted. Coinciding with the reaction of the boron with lithium, a solid phase formed at the bottom of the crucible. This material was a finely-divided solid that became dispersed throughout the melt during stirring. Metallographically, the substance was similar in appearance to powdered boron, but, unlike boron, it was quite malleable. When the formation of this product was complete, as judged by the absence of any further formation of solid material in the melt, the temperature was slowly raised. At about 803 K ($530^\circ C$), another solid product began to form at the bottom of the crucible, until, at about 853 K ($580^\circ C$), this solid product occupied most of the crucible; the remainder of the material was molten lithium. Between 853 K ($580^\circ C$) and about 873 K ($600^\circ C$), the particles of the solid product coalesced suddenly, imparting rigidity to the entire product mixture. The excess lithium was then apparently wicked into the solid lithium-boron compound matrix, and the alloy contracted away from the container walls. The ingot was then removed from the crucible and allowed to cool.

B. Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) was employed to measure changes in enthalpy during controlled heating of known mixtures of elemental lithium and boron. DSC measurements were obtained between room temperature and 953 K ($680^\circ C$) at a programmed rate, usually $10^\circ C/min$. ($0.167 K/s$), using a calibrated DuPont Model 990 DSC apparatus in which a flowing atmosphere of dry argon was maintained. The DSC samples of about 10 mg mass were prepared by thoroughly kneading weighed portions of lithium (99.9%, Foote Mineral Co.) and powdered crystalline boron (99.9%, Atomergic Chemetals Co.) together and then hermetically sealing the mixture in a specially-designed Armco iron sample cup. Some samples were weighed portions of previously-prepared alloy ingots.

A preliminary DSC experiment using a sample mixture of 5 mg of Li and 5 mg of B yielded the curve shown in Figure 1. Three major thermal transitions were observed: (a) An endotherm at 454 K (181° C), (b) a first exotherm at 603 K (330° C) and (c) a second larger exotherm at 803 K (530° C).

These three thermal transitions corresponded to the changes observed in the reaction mixture during the preparation of the alloy in the glove box, i.e., the fusion of lithium and the formation of the two solid products.

Based on these results, a multi-step DSC procedure was devised (Figure 2). The sample was alternately heated and then cooled to room temperature, progressing to higher temperatures with each re-heating. Each heating was terminated just after the completion of an observed transition. The procedure thus consisted of four heatings at a constant rate, followed by cooling to room temperature.

The approximate temperature ranges were:

<u>Run</u>	<u>Temperature Range</u>	<u>Thermal Transitions Observed</u>
1	298 - 493 K (25 - 220° C)	(a) only
2	298 - 725 K (25 - 450° C)	(a) and (b)
3	298 - 953 K (25 - 680° C)	(a) and (c)
4	298 - 953 K (25 - 680° C)	(a) only

C. Metallography and X-ray Analysis

Samples for X-ray and metallographic examination were prepared from alloy ingots and also from the samples which had been heated beyond the second exothermic transition in the DSC. These metallographic samples were mounted in epoxy, polished with silicon carbide paper, etched with methanol or ethylene glycol, and then coated with mineral oil to protect the sample from reaction with the atmosphere. X-ray patterns were obtained for these same samples with a Norelco diffractometer using Cu K α radiation. The diffractometer shield was packed with Drierite desiccant and covered with a tightly-fitted Saran Wrap cover to protect the sample from reaction with atmospheric water vapor.

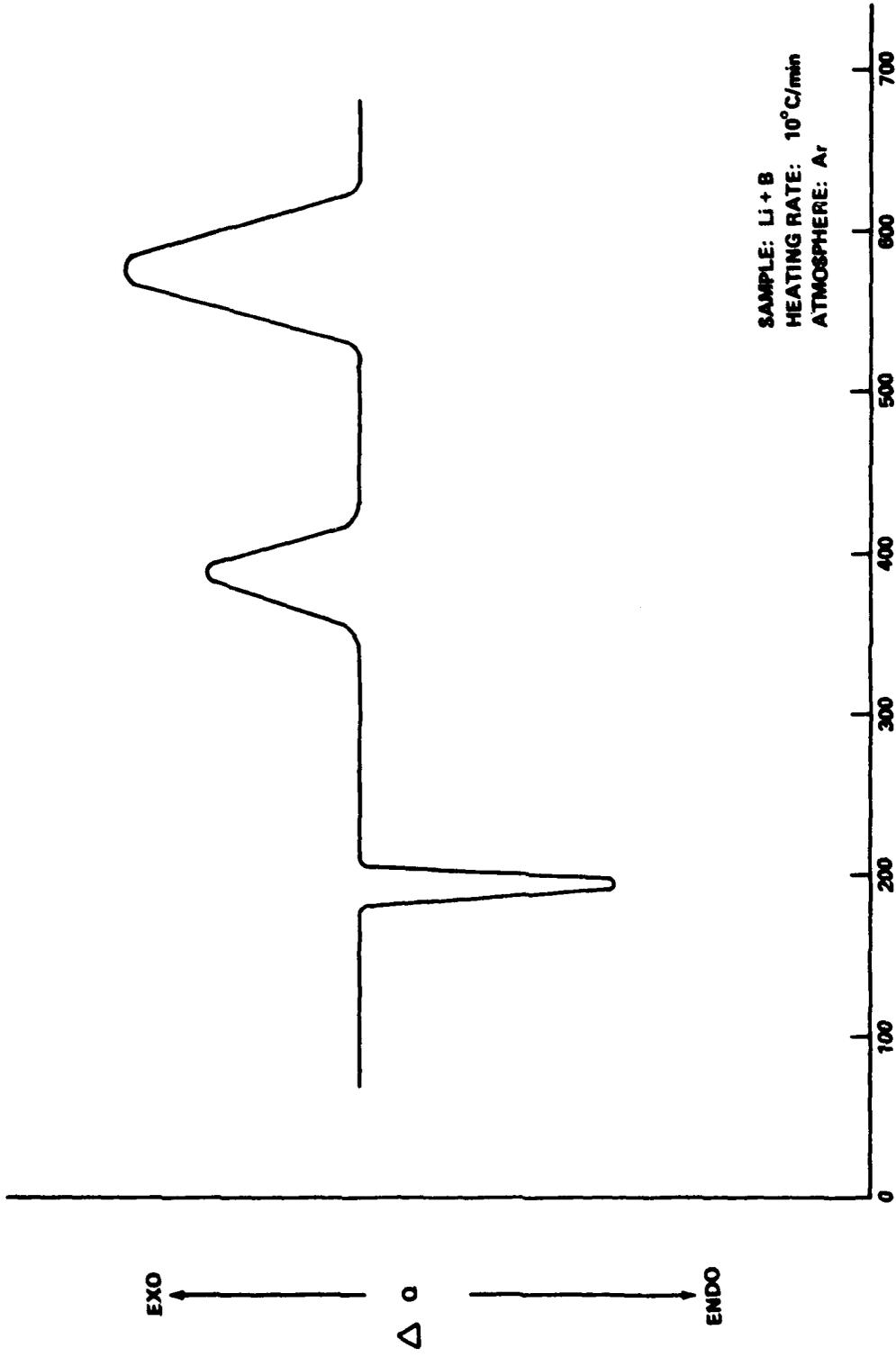


FIGURE 1. DSC CURVE OF LITHIUM + BORON MIXTURE

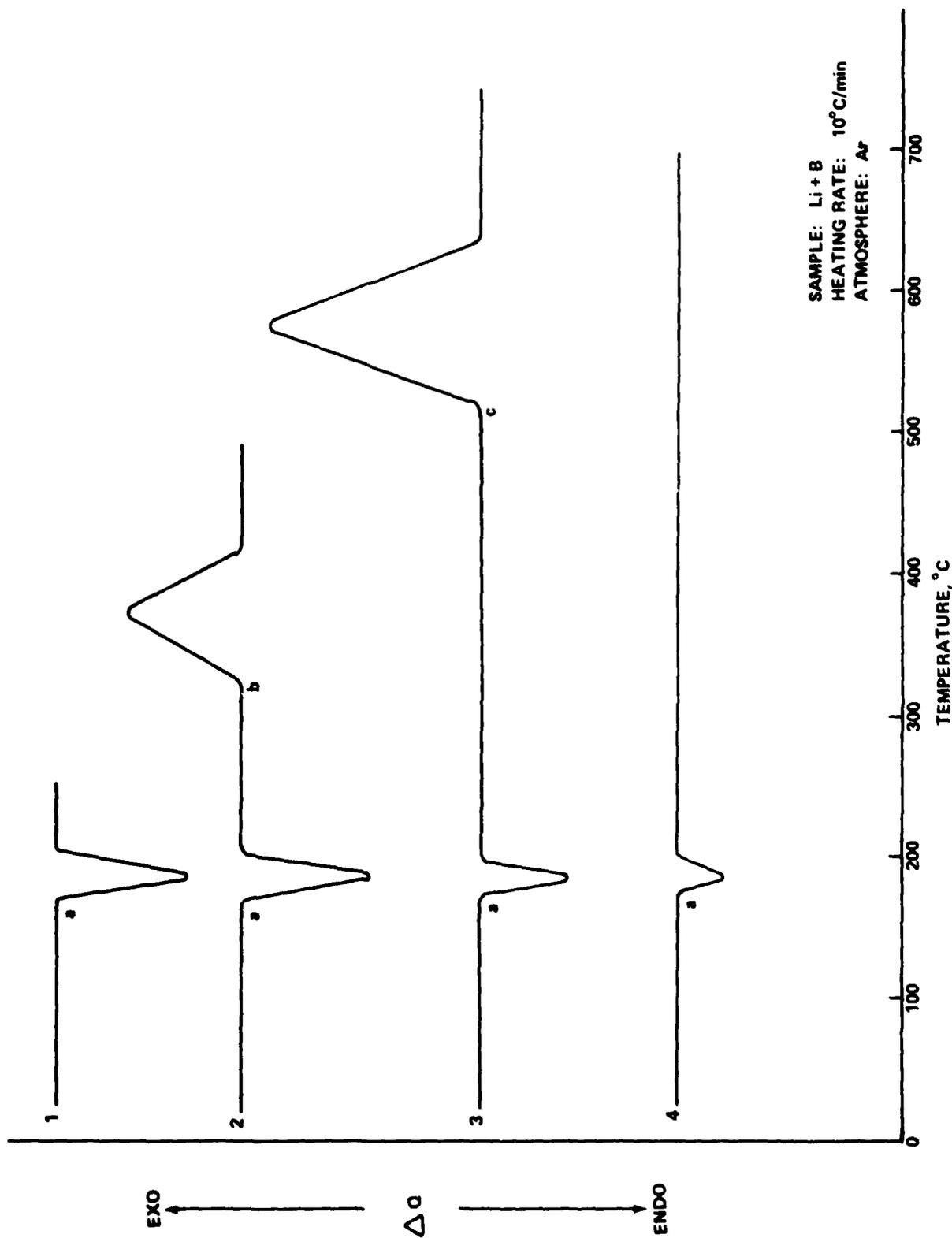


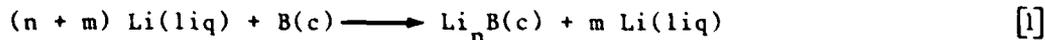
FIGURE 2. DSC CURVE OF LITHIUM + BORON (MULTI-STEP PROCEDURE)

RESULTS AND DISCUSSION

Approximately 35 DSC experiments were performed employing samples containing between 40 and 90 atomic percent total lithium mixed with boron. Figure (2) is a composite showing four curves typical of those obtained in a single experiment. The endothermic transition occurred in runs 1, 2, and 3 for all samples at 454 ± 1 K ($181 \pm 1^\circ$ C); it also always occurred in run 4 for samples richer in lithium than 53 atomic percent. The two exothermic transitions, one in run 2 and the other in run 3, occurred for all samples. No exothermic transition occurred in run 4. Curves identical to run 4 were obtained when this run was repeated.

For a given sample, the measured enthalpies of the endothermic transitions for runs 1 and 2 were always the same. The endothermic enthalpies were smaller in run 3 and still smaller in run 4. With an initial mixture of 53 atomic percent lithium or less, the endothermic enthalpy in run 4 was zero (Figure 3). The endothermic transition always occurred in all runs at the melting point of pure lithium. There was no evidence of freezing point depression or elevation.

A chemical reaction between liquid lithium and crystalline boron may be described by:



The method employed for determining the value of n in reaction (1) was based on the DSC measurements of the heat of fusion of the amount of unreacted lithium present in the sample. The difference between two such measurements, one made before the reaction and the other after, provides a direct measure of the lithium content of the product. The success of this method is based, in part, on the complete reaction of the boron and on the negligible solubility of any products in molten lithium.

A reaction was judged to be complete when the exothermic transition associated with it was absent in a subsequent run and when the calculated value of n remained constant. Since there was no detectable freezing point depression or elevation in the melting point of lithium, the endotherm could be assigned solely to the fusion of pure lithium ($\Delta H_{\text{fus}} = 432.3 \pm 2.3$ kJ/kg) (21).

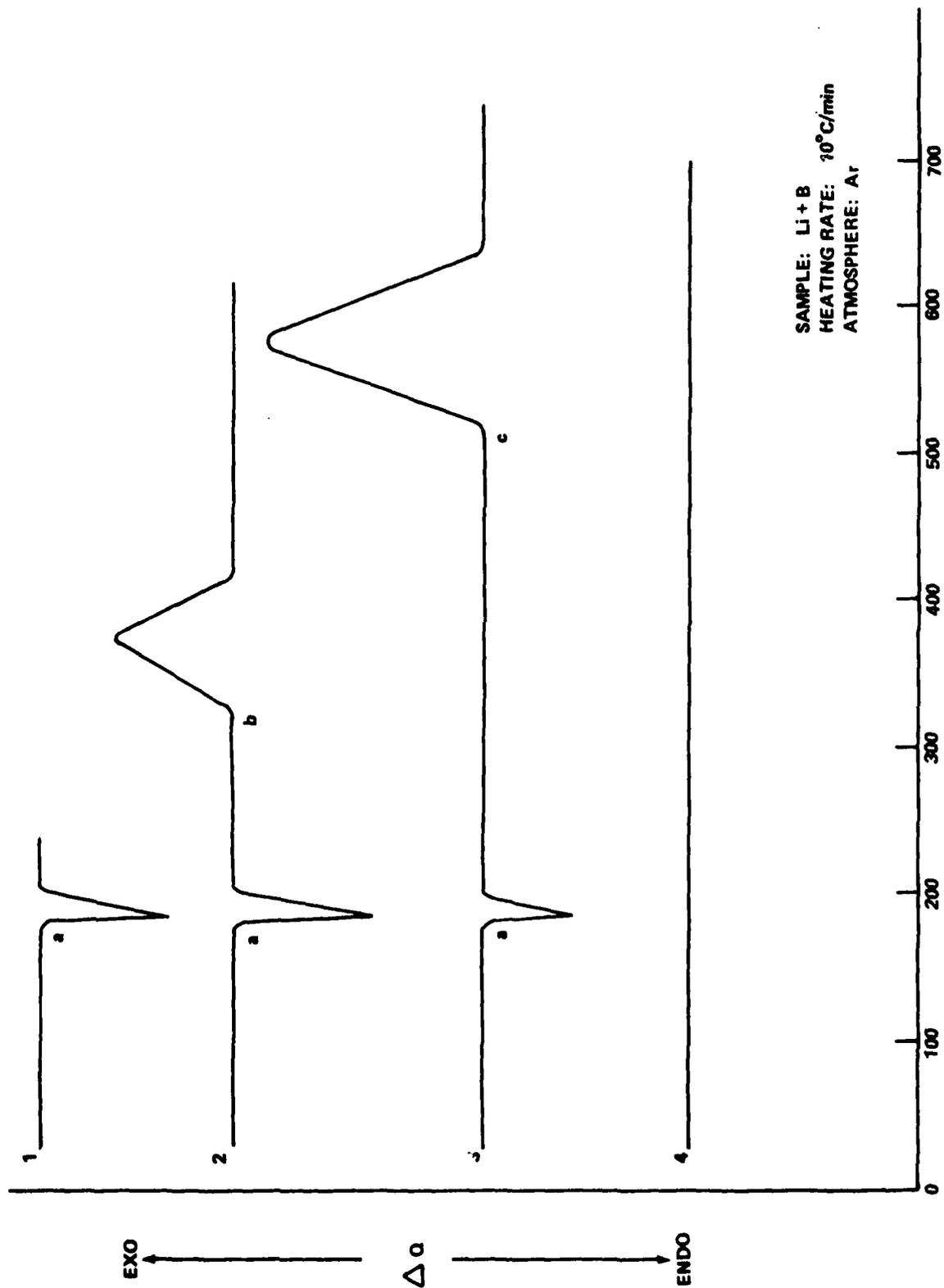
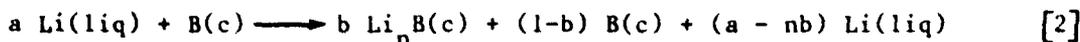


FIGURE 3. DSC CURVE OF LITHIUM + BORON (MULTI-STEP PROCEDURE)
FOR 53.0 a/o Li + 47.0 a/o B

Several experiments were performed at very slow heating rates, 1° or 2° C/min (0.0167 or 0.0334 K/s), to see what effect this would have on the calculated n values. The n value was strongly dependent on the heating rate for the product of the first exotherm. However, the value of n calculated for the product of the second exotherm was totally independent of heating rate and independent of the apparent stoichiometry of the product of the first exotherm. It was realized that the first reaction was not going to completion as shown by



wherein the amount of excess lithium, a-nb, measured by the DSC, varied, depending on the extent of reaction in a particular run.

Thus, although it was postulated that a single lithium-boron compound, $\text{Li}_n\text{B(c)}$, was being formed during the first exothermic transition, the amount of unreacted lithium varied in each run, because varying amounts of boron apparently remained unreacted in the mixture. The difficulty in achieving complete reaction of the boron is probably a direct consequence of the inability to stir the reaction mixture during a DSC run. It is not at all surprising that this problem was encountered in a heterogeneous reaction mixture. Nevertheless, the results obtained for the final product, whether it was prepared in the DSC or as an ingot, were identical.

In order to resolve the problem of a varying n value for the first product, a series of isothermal experiments was performed on several lithium-boron mixtures contained in DSC sample cups. The results are presented in Figure 4. The procedure involved holding the reaction mixture isothermally for a certain period of time, usually 20 - 30 minutes, and then cooling below the melting point of lithium and determining the amount of unreacted lithium in the mixture by DSC from the endothermic enthalpy. In this manner, the stoichiometries of the products of the two exothermic reactions were determined to be $\text{Li}_{.333}\text{B}$ and $\text{Li}_{1.166}\text{B}$, respectively.

The primary focus of our investigation was on the second exothermic reaction, during which the product of interest as battery anode material is formed. The results for this reaction on mixtures of elemental lithium and boron and on samples of the previously-prepared ingots are presented in Figure 5. The atomic percent of lithium remaining after the second exotherm goes to zero when the initial amount of lithium in the mixture is about 53.8 a/o. A statistical analysis of the 31 data points for which a measureable quantity of unreacted lithium remained yields the following results for Li_nB :

\bar{n}	s	a/o Li
1.166	0.051	53.83

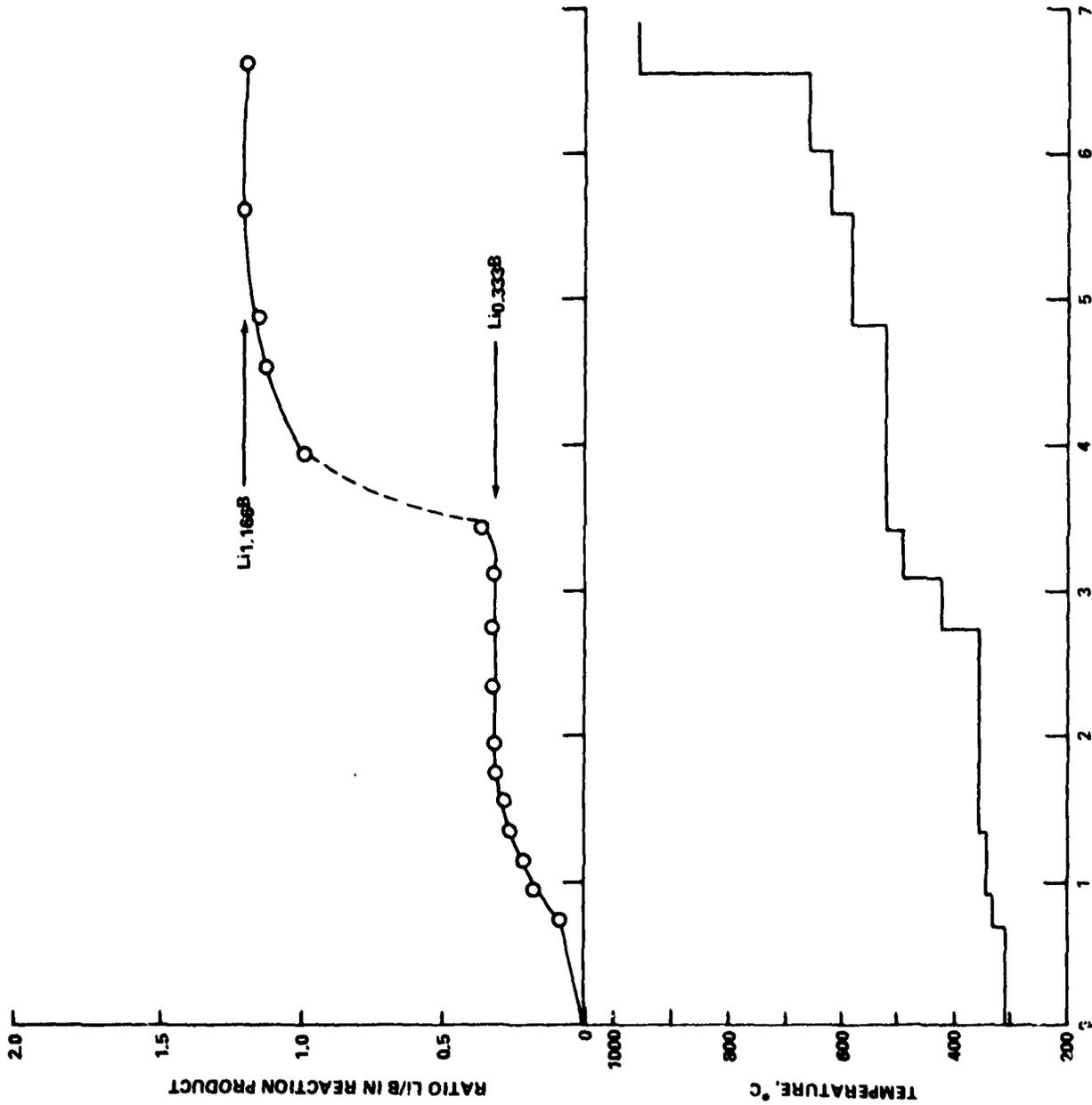


FIGURE 4. ISOTHERMAL REACTIONS IN LITHIUM - BORON MIXTURES

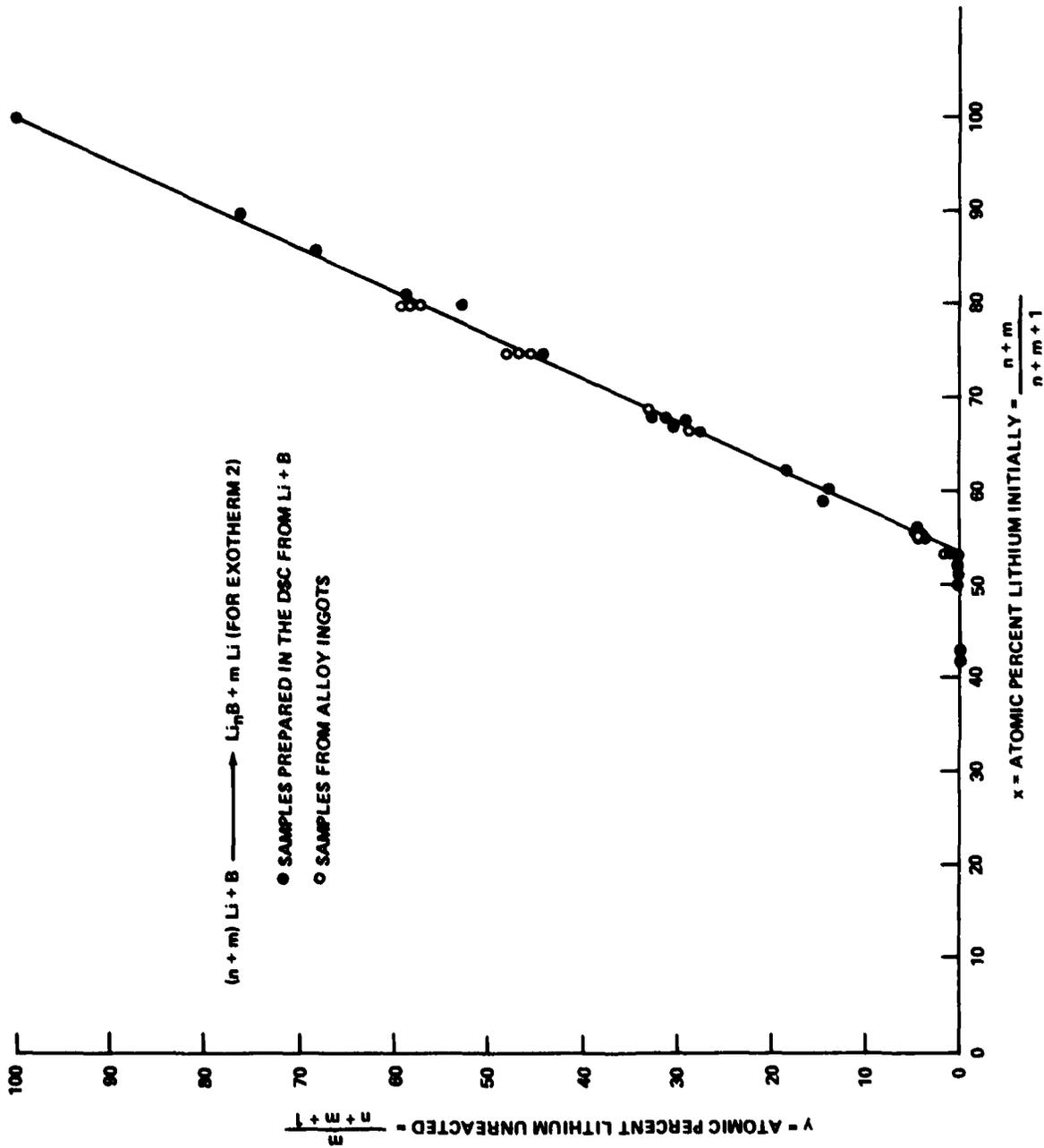
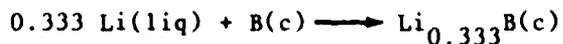


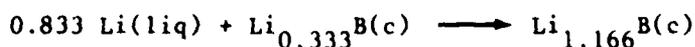
FIGURE 5. RESULTS OF DSC EXPERIMENTS ON LITHIUM - BORON MIXTURES AND INGOT SAMPLES. DETERMINATION OF Li7B6.

This result is extremely close to a value of $n = 1.167$ (a/o Li = 53.85) calculated for the stoichiometry Li_7B_6 . These results indicate that the lithium-boron "alloy" apparently is a two-phase material consisting of the metallide, Li_7B_6 , and excess lithium. (The term "metallide" was first proposed by Kurnakov (22) to describe certain metal-metal or metal-metalloid compounds).

The enthalpies of formation of the two products were determined from the areas under the exothermic peaks. Because the reaction to form the product of the first exothermic transition did not consistently go to completion in the DSC experiments, the measured enthalpies could be interpreted only semi-quantitatively. The enthalpies measured for the first exothermic transition were in the range 18 ± 4 kJ per gram atomic mass of boron. The enthalpies measured for the second exothermic transition were in the range 28 ± 4 kJ per gram atomic mass of boron. Thus, it is estimated that for the reaction



the heat of reaction in the range $320^\circ \text{C} - 450^\circ \text{C}$ is approximately $\Delta H = -18$ kJ, and for the reaction



the heat of reaction in the range $520^\circ \text{C} - 650^\circ \text{C}$ is approximately $\Delta H = -28$ kJ.

The results of the X-ray and metallography studies were consistent with the DSC results. Only one, single, consistent X-ray pattern was obtained over the entire range of compositions from approximately 55 to 90 a/o lithium. The pattern was that of a two-phase material, a lithium-boron phase plus lithium. A unique pattern was obtained for a 53 a/o lithium sample; it was identical to the other patterns but contained no peaks attributable to elemental lithium (see Table I). Metallographic examination of the alloys also revealed a two-phase material. One phase, the lithium-boron compound, Li_7B_6 , was present throughout the range of compositions studied; the amount of the other phase, elemental lithium, was consistent with reaction (1) with $n = 1.166$ and $0 \leq m < 8$, the highest value studied. However, below about 66 a/o Li, the free elemental lithium could not be detected metallographically; it is postulated that the lithium is in micro-pores and cannot be seen below this composition. The presence of free elemental lithium found by DSC in mixtures between 53.8 a/o Li ($m = 0$) and 66 a/o Li ($m = 0.83$) was confirmed qualitatively by X-ray diffraction.

A photomicrograph of an 80 a/o Li (B) sample ($m = 2.83$) is presented in Figure 6. The Li_7B_6 phase is seen as the dark area, while the light material is elemental lithium.

Table I.
X-RAY DIFFRACTION RESULTS FOR Li_7B_6

2θ	d	I/I_0
25.70	3.466	100
40.80	2.212	23
45.10	2.010	66
52.60	1.740	<5
62.25	1.491	<5
79.90	1.201	<5
82.75	1.166	<5
99.55	1.010	<5

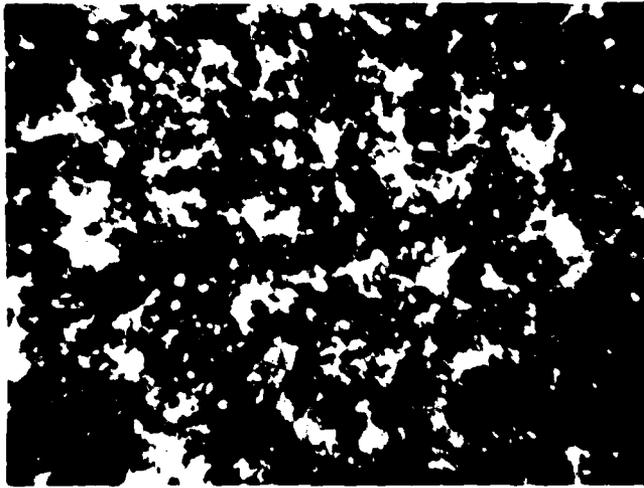


FIGURE 6. 80 ATOMIC PERCENT Li (B) ALLOY (150X)

Alloy samples were also analyzed for free lithium by Kilroy and Angres (23). The excess lithium in the sample was extracted with an excess of naphthalene dissolved in tetrahydrofuran to form a lithium naphthalide solution. The solution was separated from the ingot residue, reacted with water, and the resulting lithium hydroxide solution was titrated with standard hydrochloric acid. This analysis technique was performed over the same range of compositions as in the DSC studies. The stoichiometry of the residue compound calculated from the analysis results was $\text{Li}_{1.15+05}\text{B}$, which is in very good agreement with the DSC results, and was independent of the initial alloy composition from 55 to 90 a/o lithium.

Based on the anodic discharge behavior of lithium-boron alloys in the LiCl-KCl eutectic melt, DeVries et al. (24) have determined the anode composition at the end of the discharge to be $\text{Li}_{1.16}\text{B}$, or Li_7B_6 . They also found evidence of a compound in the alloy with the apparent stoichiometry Li_2B . However, we have found no evidence for the existence of this compound.

A summary of the results obtained from Differential Scanning Calorimetry, Metallography, and X-ray analysis, and the results of the extraction analysis (23) and the electrochemical discharge data (20, 24) are presented in Table II.

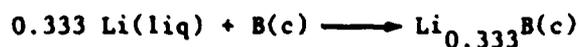
Table II.
 SUBSTANCES FOUND IN LITHIUM-RICH Li-B ALLOYS
 AFTER SECOND EXOTHERMIC TRANSITION

Method	Total Atomic Fraction Lithium In Alloy	
	<u>.53 to .66</u>	<u>.67 to .90</u>
Metallographic Examination	Li ₇ B ₆ Only	Li ₇ B ₆ and Lithium
X-Ray Diffraction	Li ₇ B ₆ and Lithium	Li ₇ B ₆ and Lithium
Differential Scanning Calorimetry	Li ₇ B ₆ and Lithium n = 1.166 ± .051	Li ₇ B ₆ and Lithium n = 1.166 ± .051
Extraction Analysis	Li ₇ B ₆ and Lithium n = 1.15 ± .05	Li ₇ B ₆ and Lithium n = 1.15 ± .05
Electrochemical Discharge	Li ₇ B ₆ and Lithium n = 1.16 ± .03	Li ₂ B and Lithium n = 2.00 ± .04

CONCLUSIONS

When elemental lithium and elemental boron are mixed together and heated (in the absence of any other reactive substances), two successive irreversible exothermic chemical reactions occur forming solid compounds of lithium and boron that are apparently quite insoluble in molten lithium. The apparent stoichiometries of the products that have been identified are represented by the following reactions:

- a. At temperatures between about 573 K (300° C) and 723 K (450° C), the predominant reaction is



- b. At temperatures between about 753 K (480° C) and 923 K (650° C), the predominant reaction is



The lithium-boron alloy system consists of this $\text{Li}_{1.166}\text{B}$ phase (Li_7B_6), which is a high-melting, sintered, crystalline compound, into which has been wicked excess elemental lithium. The excess lithium, which is the anodically active substance, is immobilized by the solid Li_7B_6 matrix during electrochemical discharge.

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