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The Preparation of a Lithium Deuteride Casting
and Some Properties of Lithium Deuteride

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Berkeley, California

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THE PREPARATION OF A LITHIUM DEUTERIDE CASTING
AND SOME PROPERTIES OF LITHIUM DEUTERIDE

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8 November 1950

Introduction

It is thought that the neutrons produced by proton bombardment of a lithium deuteride cyclotron target might be more nearly mono-energetic than those obtained from targets currently in use. The work described below, then, is the result of an attempt to produce a uniformly dense casting of lithium deuteride for this purpose.

Apparatus

The apparatus finally adopted (Fig. 1) consists of a zirconia support upon which rests a $\frac{1}{4}$ " transite plate. A zircon stand sits in the center of the plate and a porcelain tube surrounds it. The annular space between the stand and the tube is filled with sand. A graphite induction-heating unit rests upon the stand and contains the casting cell. This entire assembly is enclosed in a pyrex bell jar (O.D. = 6") which has two openings, one at the center of the top, the other near the bottom. This jar is sealed to the base by Apiezon grease. The induction heating coil is outside of this bell jar. A brass tube carrying a tamping rod and a lifting hook is inserted through the upper jar opening. Helium gas is introduced through the tube and exits through the lower opening in the bell jar. A thermocouple is sealed through a glass tube in the exit line and passes up into the chamber and then curves downward into the space between the graphite crucible and the casting cell. A second heating element, consisting of an iron cylinder $1/8$ " thick and $1\frac{1}{2}$ " high is used for the local heatings. The graphite crucible is removed and a magnesia

crucible is used with the iron heating element.

The casting cell was made of stainless steel; 0.010 inch thick, welded on the bottom and along one side; and was $\frac{1}{2}$ " x 1" in sectional area, and $5\frac{1}{2}$ " long. The metal was quite clean except in the weld, where oxidation occurred during the welding processes.

Procedure

The procedure adopted was as follows: Fill the casting cell to about an inch from the top with granular LiD. Assemble the apparatus and heat the cell in He to 720-740°C and maintain this temperature until the LiD is completely molten. Cool slowly. Refill the cell to give about 10% excess LiD and re-assemble using the short heater. Remelt the material at the top of the cell, keeping the bottom below the melting point. After the liquid drains into the cavity, allow it to cool. Usually a second top-heating is required to completely eliminate the cavity. When the cell contents have cooled to room temperature, carefully strip off the mold and saw (carefully) or grind the casting to the desired dimensions. Apply two or three light coats of clear glyptal to inhibit attack by the atmosphere.

Discussion

The procedure above was developed using LiH, and yields equally good castings of LiH. Helium was used in these preparations since the LiD and LiH were of technical grade (~90% pure). The preparation of a pure casting would involve making pure LiD or LiH as a first step, and then using a D₂ or H₂ atmosphere above the cell during the casting operation.

In every case, when either LiH or LiD is completely melted in the cell, the casting produced has a very deep cavity. This has been thought to be the result of 'crawling' of the material, but it seems more likely that it is the result of the large volume change in freezing. The density of liquid LiD was

measured at 720°C and was found to be 0.76 gm/cm³. The ideal density of LiD crystals is 0.878 ± 0.002 gm/cm³,⁽¹⁾ so that there is a 15% decrease in volume on freezing. This amount of volume change is easily enough to cause a large void or crater in the final melt.

Although 'crawling' is not the cause of the cavity formation, it is present, particularly in regions where the cell wall is oxidized. In this respect Li metal, LiH, and LiD behave similarly, in that only a small amount of crawling (less than 2 cm) is evidenced by these substances when the walls are clean, but if oxidized surfaces are present, the crawling may easily be 5 times as great. The procedure outlined above virtually eliminates the problem of crawling. During the first heating the LiD crawls up the weld seam, then, when the small heater is used to melt the top portions only, the LiD in the seam is the first to melt and run down to the bulk of the casting. The seam is thus cleaned and the tendency to crawl is inhibited.

The cells were found to break along the side seam, but due to the high surface tension of LiD, very little molten salt escaped. Breakage is probably due to expansion of the solid castings during the re-heating periods. The use of heavier walled cells with stronger seams would eliminate this cracking, but the walls should then be scored to facilitate removal from the final casting.

The product obtained by the procedure above consisted of a dense aggregation of blue-white crystals on the lower end, and a blue-green glassy-appearing crystal on the upper end (re-heated end). The density of this LiD target was 0.84 gm/cm³, which is 4.4% less than the theoretical density. Therefore there is presumably 4.4% void in this target. The physical dimensions of the target are 1.31 cm x 2.55 cm x 6.19 cm.

Conclusion

Lithium deuteride and lithium hydride have been found to be castable, and a suitable specimen has been cast for use as a cyclotron target.

Appendix - Properties of LiD and LiH

Some physical and chemical properties of LiD and LiH are given in Tables I and II.

Table I
Properties of LiD and LiH

Property	LiH	Ref.	LiD	Ref.
m.p., °K	953	(2)	959 ± 5	(1)
density, gm/cm ³				
crystal	0.76 ± 0.01	(1)	0.878 ± 0.002	(1)
liquid			0.76	(this paper)
$\Delta H_f(298)$ kcal/mole	44.0 ± 2.	(2a)		
Cp, cal/gm °C	0.98 at 273°K 1.07 at 323°K	(3)		

Table II
Decomposition Pressures of H₂ over LiH

Temp.	% LiH	P _{H₂} mm Hg	Ref.
825°C	100	714	(2b)
	90	425	
	56	425	
770°C	100	317.5	(2b)
	97	160	
	36	160	
700°C	100	268	(2c)
	98	35	
	18	35	

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FIG. 1
CASTING ASSEMBLY

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