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NOVEMBER 8, 1963

SPECIAL REREVIEW FINAL DETERMINATION	Reviewers	Class.	Date
	KAW	U	5-4-82
Class: <u>U</u>			

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INFORMATION CATEGORY	
CRD	
E. L. Layland	11/12/63
Authorized Classifier	Date

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Classification cancelled (or changed to)
by authority of _____
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**FABRICATION
DEVELOPMENT
OF SHIELDING MATERIAL**

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FABRICATION DEVELOPMENT
OF SHIELDING MATERIAL

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TABLE OF CONTENTS

	<u>Page</u>
1. Abstract	1
2. Introduction	2
3. Material and Equipment	3
4. Experimental Procedure	4
5. Discussion	6
(A) Effect of Particle Size on Pressed Density of LiH	6
(B) Effect of Dwell Time on Density of Pellets Compacted From As-Supplied Powder	7
(C) Effect of Length on the Density of Pellets Pressed From As-Supplied Powder	8
(D) Heat Treatment of LiH Compacts	9
6. Conclusions	11

LIST OF TABLES AND FIGURES

TABLE I	A display of the Accuracy of Equation 2	12
FIGURE 1	Density Vs Pressing Pressure for LiH Pellets Compacted From Various Size Powders	13
FIGURE 2	Density Vs Dwell Time for LiH Pellets Compacted From As-Received Powder at Various Pressures	14
FIGURE 3	Density Vs Pressing Pressure for Dwell Times in Excess of 4 Seconds (As-Received Powder)	15
FIGURE 4	Density Vs Ratio of Length to Diameter for 0.502" Diameter LiH Pellets Compacted from As-Received Powder at 50,000 psi	16

LIST OF FIGURES

(Cont.)

		<u>Page</u>
FIGURE 5	Density Vs Ratio of Length to Diameter for 0.502" Diameter LiH Pellets Compacted From As-Received Powder at 60,600 psi	17
FIGURE 6	Density Vs Ratio of Length to Diameter for 0.502" Diameter LiH Pellets Compacted From As-Received Powder at 70,800 psi	18
FIGURE 7	The Constant C From $D = M (L/d) + C$ of Figures 4, 5, and 6 Vs Pressing Pressure	19
FIGURE 8	Photomicrographs of LiH Compacts Produced From As-Received Powder For As-Pressed and Heat-Treated Conditions	20

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ABSTRACT

A study was undertaken to establish the feasibility of producing dense lithium hydride pellets by cold pressing commercial powder. This study stemmed from the requirement to investigate methods of fabricating the material for NERVA shielding applications. It was found that commercial powder (-16+40 mesh) could be easily cold pressed to densities within a few percent of theoretical. A rough density (gms/cc) - pressure (psi) relationship is:
$$D = 1.12 \times 10^{-6} P + 0.682$$
 for pellets of moderate length to diameter ratio. The effect on density caused by the pressure dwell time and length to diameter ratio is slight. An approximate relationship for the effect of length/diameter on density is $D = -0.027 (L/d) + C$, where C is a constant dependent upon pressing pressure and dwell time. These results established cold powder compaction as an acceptable lithium hydride pelletizing procedure, and cold pressing methods similar to those used in the nuclear industry for UO_2 were eventually used to fabricate LiH for the first NERVA shield.

Heat treatment of LiH pellets results in particle fusion with the loss of discrete particle interfaces. This phenomenon is thought to be a result of sintering, and may improve the low temperature thermal conductivity of the material by eliminating discrete particle boundaries.

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INTRODUCTION

A lithium hydride-stainless steel radiation shield is part of the NERVA system. The shield consists of stainless steel tubes filled with lithium hydride pellets and stainless steel discs. Attenuation of gamma radiation is accomplished by the stainless steel while the lithium hydride serves to slow down fast neutrons and capture thermal neutrons by the $\text{Li}^6 (n, \alpha)$ Tritium reaction. The low density of lithium hydride, 0.78 gms/cc, makes it an attractive choice for space nuclear shielding applications where light weight systems are demanded. Use of lithium hydride, however, necessitates fabricating nearly theoretically dense shapes from commercially available powder starting material. Possible methods for producing the desired shapes are casting, swaging, hot pressing, cold pressing, and cold pressing and sintering. This report summarizes an investigation into the cold pressing characteristics of lithium hydride. The study was undertaken to determine the feasibility of using this method to produce the NERVA shield pellets. Some mention is also made of the heat treatment characteristics of the material.

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MATERIAL AND EQUIPMENT

The lithium hydride used for this study was purchased from the Lithium Corporation of America. The mesh size of the as-received powder was -16 to +40, and its composition was guaranteed to be at least 95% LiH.

Because of the hygroscopic nature of LiH, operations pertaining to powder processing such as crushing, sizing, pressing, weighing and measuring were performed in a glove box under the protection of argon.

All heat treatment of cold pressed LiH was performed in flowing hydrogen inside an electric tube furnace retort. The retort was loaded and unloaded in the glove box. Air was prevented from entering the retort during transfer from the glove box to the furnace and back by closing gas regulating valves on its entrance and exit ports.

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EXPERIMENTAL PROCEDURE

The pressed density of a solid cylindrical pellet formed from a powder can most generally be described as:

$$D = f(P, T, t, L, d, P.S., P.S.D., D.C., P.A.)$$

where:

D	=	density
P	=	pressing pressure
T	=	pressing temperature
t	=	time of pressure application (dwell time)
L	=	length of pellet
d	=	pellet diameter
P.S.	=	particle size
P.S.D.	=	particle size distribution
D.C.	=	die surface condition
P.A.	=	pressing action (e.g. single, double)

A study into the effect each variable has on the pressed density is impractical. Thus, the investigator is left with two possible courses of action: namely, (1) he may assume that certain variables only slightly affect the compacted density and can be neglected, and/or (2) he may fix certain "variables" with the understanding that his results apply only for these conditions or for conditions closely approximating these. Both courses of action have been taken in the investigations described herein. These are:

1. The effect on the pressed density caused by the particle size distribution within the powders used was neglected. This is reasonable, since, with one exception only, the range of particle sizes in the powders studied was not great.
2. The pressing temperature was fixed at room temperature.

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3. The pellet diameter was arbitrarily fixed at 0.502 inch. (The die body was also slightly tapered to facilitate ejection of long pellets.)
4. Double action pressing was simulated through use of the floating die.

With these conditions imposed on the studies, the effect on the pressed density caused by powder particle size, dwell time and pellet length were investigated at several fixed pressing pressures.

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DISCUSSION

(A) Effect of Particle Size on Pressed Density of LiH:

This study was carried out on four different particle size powders - their effect on density being studied at three fixed pressing pressures. The powders evaluated can be described by mesh as:

- (1) As-supplied (-16 +40)
- (2) -40 +60
- (3) -60 +100
- (4) -100 (50% -200)

The parameters - pressure, dwell time, length and die conditions were fixed as follows:

- a. Pressure - 30,300; 50,500; and 70,800 psi
- b. Dwell Time - 5 seconds $\pm$ 1 second (timed with stop watch)
- c. Length - roughly fixed for each pressure by keeping weight of the die charge at 1.520 gms.
- d. Die Condition - the walls of the die cavity were cleaned after the pressing of every pellet involved in this investigation.

The results of this study are in Figure 1. The data, to a certain extent, implies that for a fixed pressing pressure the compacted density decreases as the powder particle size decreases. The reason for this is believed to arise in part from the decrease in bulk density of the material with decreasing particle size. This increase in "fluffiness" probably causes the material to entrap more atmospheric gas during compaction. Also, as the particle size decreased, an increasing tendency for the powder to creep up between the pins and die wall during compaction was noted. This undoubtedly affected the friction on the pins which was reflected in the density of the compacts. In general, there do not appear to be any advantages gained by pressing powders which are finer than the as-supplied material.

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Probably the most significant aspect of this data is the finding that the as-supplied -16 +40 mesh LiH could be cold compacted to relatively high densities at pressures which are not excessive. For example, if 0.78 gms/cc is assumed to be the theoretical density of this material, then compacted densities approaching within a few percent of theoretical are realizable at pressures in the range 60,000 to 70,000 psi. (Cold compaction of as-received powder was the method used to produce the LiH pellets for the first NERVA shield.)

B. Effect of Dwell Time on Density of Pellets Compacted from As-Supplied Powder:

The effect of dwell time on the pressed density of as-received (-16 +40 mesh) powder was studied for times in the range 1/2 to 7 seconds at four fixed pressures. An industrial timer was wired into the electrical system of the press in place of the pressure relief valve in order to accurately control the pressure dwell. (Dwell times of between 0 and 1/2 second could not be studied because of limitations in the press and industrial timer.) The parameters, pressure, particle size, length and die condition were fixed as follows:

- a. Pressure - 40,400; 55,600; 70,700; and 80,800 psi
- b. Particle Size - -16 +40 (as-supplied)
- c. Length - fixed per pressure by keeping die charge at 1.520 gms.
- d. Die Condition - the coating of LiH which smears on the die walls during pressing was not removed. This, in a sense, duplicates the conditions imposed by production.

The results of this study are plotted in Figure 2. In general, each curve shows that density approaches an asymptotic value at a dwell time of about four seconds. None of the curves, however, show more than a 1 or 2% difference between the asymptotic density and that at 1/2 second dwell time. Thus, the effect of the pressure dwell for times in excess of 1/2 second is very slight.

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Figure 3 shows a graph of density versus pressing pressure for the as-supplied powder.* The plot was derived from the curves in Figure 2 by plotting against pressure the density to which the dwell time curves became asymptotic. The linear relationship obtained is:

$$(1) \quad D = 1.12 \times 10^{-6} P + 0.682$$

where D = density in gms/cc, and P is the pressing pressure in psi

(C) Effect of Length on the Density of Pellets Pressed from As-supplied Powder:

The effect of pellet length on pressed density is usually studied by considering the dimensionless ratio of length to diameter (L/d). In this study the L/d was varied between approximately 0.8 to 1.8, and its effect on the pressed density of as-received (-16 +40 mesh) material was examined at several pressures for several dwell times.

The parameters, pressure, particle size, dwell time and die condition were fixed as follows:

- a. Pressure - 50,500; 60,600; and 70,800 psi
- b. Particle Size - -16 +40 (as-supplied)
- c. Dwell Time - 1/2, 2 and 4 seconds (with exception of study at 70,800 psi where effect was evaluated at 1/2 and 4 seconds only).
- d. Die Condition - smeared LiH was not removed from the die walls.

The results of the study are plotted in Figures 4, 5, and 6. The equations describing the lines drawn through each group of data points are least square equations. Note that the decrease of density with increasing L/d is roughly a constant for all eight experimental plots. If one assumes that these slopes should be equal, and that the individual slopes only

*The Figure 3 graph differs from that presented for as-supplied powder present in Figure 1 because (1) it is derived from a larger amount of data and is thus more accurate, and (2) the die conditions differ.

represent some fluctuation about a mean, one equation can be written to describe density as a function of L/d . That equation is:

$$(2) \quad D = -0.027 (L/d) + C$$

where: C is a constant which depends upon the pressing pressure and dwell time, and has units of gms/cc.
-0.027 is the average of the slopes of the eight equations of Figures 4, 5, and 6, and has units of gms/cc.

The proper constant to use in the above equation can be picked from the plot in Figure 7. Here the constants from the equations presented in Figures 4, 5, and 6 are plotted against pressing pressure.

The accuracy of equation (2) can be judged through use of the data from Figure 2. Some examples of this are shown in Table I. A comparison of the calculated densities (column 6) with the actual densities (column 3) shows relatively small deviations (column 7). The equation can, therefore, be used to give a fairly good representation of the pressing conditions necessary to yield a pellet of given dimensions and density. Calculations of pellet density for a set of dimensions and pressing conditions, and pellet dimensions for a set of pressing conditions and densities can, of course, also be made.

(D) Heat Treatment of Lithium Hydride Compacts

Heat treatment of lithium hydride compacts has been considered as a possible means for driving the hydrogen gas producing $\text{LiOH (impurity)} + \text{LiH}$ reaction to completion. It has been feared that this reaction might result in excess gas pressure build-up in the NERVA shield capsules during reactor operation. Recent irradiation studies*, however, have shown that pellets which are not heat treated do not result

*WANL irradiation studies on lithium hydride, David Jacobs. (This study will be reported in a future document.)

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in significant capsule gas pressure build-up even when subject to conditions in excess of those imposed by NERVA operation.

Pellet heat treatment at high enough temperatures results in a loss of particle boundary definition. This phenomenon is especially pronounced at 600 °C, and is apparent in the photomicrograph for this condition in Figure 8. Sintering has probably rapidly progressed at this temperature and has eliminated the original particle boundaries. This elimination of the particle interfaces could be advantageous in increasing the low temperature thermal conductivity of the material.

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CONCLUSIONS

- (1) Cold compaction of commercially available lithium hydride powder is a realistic method of producing dense pellets for NERVA shielding requirements. An approximate density (gms/cc) - pressure (psi) relationship is: $D = 1.12 \times 10^{-6} P + 0.682$. The pressure application time (dwell time) and the length to diameter ratio do not significantly affect the finish pellet density. A rough relationship for the effect of length to diameter ratio on pellet density is given by $D = -0.027 (L/d) + C$ where C is a constant dependent upon pressure and dwell time.
- (2) Heat treatment of compacted LiH results in the loss of discrete particle interfaces and may improve the material's thermal conductivity.

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TABLE I
A DISPLAY OF THE ACCURACY OF EQUATION 2

1 Pressure (psi)	Data From Figure 2*		Calculated From Figure 2 Data	Figure 7	Density Calculated From Equation 2	Deviation of Calculated Density
	2 Dwell Time (seconds)	3 Density (gms/cc)	4 L/d	5 C (gms/cc)	6 Density= $-0.027(L/d)+C$ (gms/cc)	7 (gms/cc)
55,600	0.5	.725	1.285	.759	.722	-.003
"	2	.741	1.259	.766	.732	-.009
"	4	.745	1.255	.772	.738	-.007
70,700	0.5	.739	1.263	.788	.754	+0.015
"	2	.758	1.233	.794	.761	+0.003
"	4	.762	1.225	.800	.767	+0.005
80,800	0.5	.761	1.229	.806	.773	+0.012
"	2	.769	1.215	.810	.777	+0.009
"	4	.772	1.211	.818	.785	+0.013

*Pellet weight 1.520 gms.

Pellet diameter 0.502 inches.

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FIGURE 1 - DENSITY VS PRESSING PRESSURE FOR LITHIUM HYDRIDE PELLETS COMPACTED FROM VARIOUS SIZE POWDERS

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FIGURE 2 - DENSITY VS DWELL TIME FOR LITHIUM HYDRIDE PELLETS COMPACTED FROM AS-RECEIVED POWDER AT VARIOUS PRESSURES.

DIE CHARGE = 1.52 GRAMS
DIE DIAMETER = .502 INCHES

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FIGURE 3 - DENSITY VS PRESSING PRESSURE FOR DWELL TIMES IN EXCESS OF 4 SECONDS
(AS-RECEIVED POWDER)

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FIGURE 4 - DENSITY VS RATIO OF LENGTH TO DIAMETER FOR .502" DIAMETER LITHIUM HYDRIDE PELLETS COMPACTED FROM AS-RECEIVED POWDER AT 50,000 PSI

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FIGURE 5 - DENSITY VS RATIO OF LENGTH TO DIAMETER FOR .502" DIAMETER LITHIUM HYDRIDE PELLETS COMPACTED FROM AS-RECEIVED POWDER AT 60, 600 PSI

FIGURE 6 - DENSITY VS RATIO OF LENGTH TO DIAMETER FOR .502 DIAMETER LITHIUM HYDRIDE PELLETS COMPACTED FROM AS-RECEIVED POWDER AT 70, 800 PSI

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FIGURE 7 - THE CONSTANT C FROM THE EQUATIONS OF FIGURES 4, 5 AND 6 OF THE FORM $D = -M (L/D) + C$

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8X AS COLD PRESSED

8X HEAT TREATED
400 °C - 1/2 HR.

8X HEAT TREATED
450 °C - 1/2 HR.

8X HEAT TREATED
500 °C - 1/2 HR

8X HEAT TREATED
550 °C - 1/2 HR

8X HEAT TREATED
600 °C - 1/2 HR.

FIGURE 8 - PHOTOMICROGRAPHS OF LITHIUM HYDRIDE COMPACTS 595726A
PRODUCED FROM AS-RECEIVED POWDER FOR AS PRESSED
AND HEAT TREATED CONDITIONS

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