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To: Mr. K. J. Kelly

Contract AT(30-1)-2789
CNLM-5757

Date: June 24, 1964

From: H. Slotnick

cc: Messrs.

Subject: Irradiation of Lithium Hydride

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Authorized Classifier6/24/64
Date

A comprehensive review of the literature revealed a limited number of lithium hydride irradiation tests. The salient aspects of these tests are presented in the attached (1) condensed summary tabulation and (2) review enumeration.

It is noted that high quality lithium hydride which has been outgassed and enclosed in evacuated or inert gas-filled stainless steel capsules will undergo negligible swelling, powdering, or gas evolution when irradiated at 10^{16} - 10^{19} nvt for 100-180 hours at 200-1000F. Although these test conditions approximate the temperatures and total doses expected for SNAP-50 reactor application, the test durations were no more than 1/50 of the required 10,000-hour life. In view of the importance of time in a diffusion-controlled mechanism for radiation damage, an inpile lithium hydride program to evaluate dimensional stability and physical integrity under temperature, time, and dose conditions approaching those expected in SNAP-50 appears warranted.

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SUMMARY OF LITHIUM HYDRIDE IRRADIATION STUDIES

Agency	Report	LiH Form *	Container		Test Conditions			Observations	Comments
			Material	Atmosphere	Temp. F	Time, hr	Dose, nvt		
Argonne (1948)	ANL-4208	C	Steel	H ₂ , 50 psi	N.R. **	2160 (3 mos.)	8 x 10 ¹⁷	1. LiH, blackened 2. No pressure increase.	LiH purity unknown.
Oak Ridge (1950)	ORNL-629	N.R.	N.R.	N.R.	305; 462	2160 (3 mos.)	N.R.	1. Dissociation press. not affected.	LiH purity unknown.
NDA (1956)	NDA-23	C-P	Type 347	Vacuum or helium	100-610	240-335	1.55 x 10 ¹⁵ - 1.12 x 10 ¹⁸	1. LiH, blackened. 2. LiH, expansion, dis- integration. 3. Pressure increase. 4. No change in lattice spacing.	1. Growth and disintegration attributed to irradiation. 2. Hydrogen evolved from LiOH impurity.
GE-ANPD (1956)	APEX-323; APEX-915	C; C-P	Type 304 (outgassed)	Vacuum	970-1000	100	4 x 10 ¹⁸ thermal 1 x 10 ¹⁸ fast	1. Pressure increase, < 10 mm. 2. No change in contain- er dimensions. 3. LiH, blackened. 4. No change in lattice spacing.	Test temperature probably contributed to recovery from radiation damage,
GE-ANPD (1959)	TID-6248; APEX-915	C; C-P	SS (outgassed)	Vacuum	160-400	130-444	3.7 x 10 ¹⁵ - 1.4 x 10 ¹⁸	No powdering or swelling; limited fracturing during post-test handling.	No deleterious effect.
					560-1000	118-181	2.5 x 10 ¹⁶ - 1.3 x 10 ¹⁹	" "	" "
					400-550	744/583	2.1 x 10 ¹⁹ / 3.1 x 10 ¹⁸	Swelling, powdered, gas evolved.	Capsule leak (?); effect attributed to exposure to small amounts of air and/or moisture.

* Form: C, Cast; C-P, Cold Pressed

** N.R.: Not Reported

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SUMMARY OF LITHIUM HYDRIDE IRRADIATION STUDIES
(CONTINUED)

Agency	Report	LiH Form*	Container		Test Conditions			Observations	Comments
			Material	Atmosphere	Temp. F	Time, hr	Dose, nvt		
GE-ANPD (1961)	DC 61-7-15; APEX-915	C-P	Type 321	Helium	800	100	1.7×10^{18}	1. No gas evolution. 2. No powdering or swelling. 3. Slight discoloration.	No deleterious effect.
Lockheed (1962)	Y-1415	C-P Block	Al	Not inert	N.R.	N.R.	N.R.	1. Can rupture; rapid growth of LiH. 2. Hydride spalling. 3. LiH (post-test)-11.2% LiOH	Dry atmosphere not available to supplier. Exposure to atmosphere during compaction caused LiOH contamination. Subsequent reaction produced hydrogen.
Oak Ridge (1963)	Y-1415	C-P Block with Honeycomb (outgassed)	Type 304	Helium	300-335	45	1.6×10^{17}	1. LiH, blackened. 2. No cracking, spalling or disintegration. 3. Diameter increase: 2.4% Length increase: 1.4% 4. X-ray diff. shows 0.03% volume increase in crystal lattice.	Test prematurely terminated due to air leak.

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ENUMERATION OF LITHIUM HYDRIDE IRRADIATION STUDIES

- I. Argonne National Laboratory, Experimental Nuclear Physics Division
Report July-September 1948, ANL-4208, October 4, 1948.

Conditions:

Lithium hydride: Cast in steel cans.

Atmosphere: Hydrogen, 50 psi

Exposure:	Time	-	3 months
	Flux	-	10^{11} ny
	Dose	-	8×10^{17} nvt
	Temperature	-	Not reported

Observations:

1. Lithium hydride turned pitch black.
2. Casting shrinkage hole present.
3. No change in pressure following irradiation.

Comments:

1. Purity of hydride unknown.
2. Could not observe induced dissociation with 50 psi hydrogen overpressure.
3. Nature of test prohibits further evaluation of radiation damage.

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II. Oak Ridge National Laboratory, ANP Quarterly Report through February 28, 1950 - ORNL-629, April 19, 1950.

Conditions:

Lithium hydride: Form not reported.

Atmosphere: Not reported.

Exposure:	Time	-	About 3 months
	Flux	-	About half maximum flux (?)
	Dose	-	Not reported
	Temperature	-	152C (305F), 239C (462F)

Observations:

Negligible change in dissociation pressure as a result of irradiation.

Comments:

1. Purity of hydride unknown.
2. Nature of test prohibits further evaluation of radiation damage.

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III. Nuclear Development Corporation of America (B. Minushkin)
Lithium Hydride Radiation Stability, NDA-23, March 15, 1956.

Conditions and Observations:

Lithium hydride: Powder-pressed

<u>Dose,</u> <u>nvt</u>	<u>Temp.</u> <u>F</u>	<u>Max.</u> <u>Press.,</u> <u>psia</u>	<u>Observations</u>
0	400-600	25	2-7 days, no blackening or growth.
1.55×10^{15}	100	10	6 psia He overpressure. Slight blackening. No growth or disintegration.
2×10^{16}	400	7.8-10	Blackened, expanded to fill capsules.
$1.05-1.12$ $\times 10^{18}$	400-610	11.9-15	Blackened, partially or completely disintegrated to coarse crystalline powder.

Comments:

1. Gas evolution attributed mostly to reaction between lithium hydroxide impurity and the lithium hydride which forms hydrogen, and only slightly affected by radiation.
2. Growth and disintegration of cold-pressed pellets were attributed to irradiation.
3. Aitken and Henry (APEX-323) note that self-annealing probably did not occur at temperatures as low as 600F. Also the cohesive energy of powder-pressed lithium hydride was probably insufficient to resist radiation damage caused by thermal spikes and thermal stresses.

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- IV. General Electric, ANPD (E. A. Aitken; D. L. Henry)
Radiation Damage to Lithium Hydride, APEX-323, December 6, 1956.
(Also APEX-915, March 1, 1962)

Conditions and Observations:

Lithium hydride: Type 304 capsule

1. Cast into primary capsule.
2. Cold pressed cylinder, machined and close-fitted into capsule.
3. Cold pressed cylinder, machined undersize to permit 1/16-inch gap between hydride and capsule.

Exposure:

Time - 100 hours
Flux (nv) - 1.25×10^{13} thermal; 0.3×10^{13} fast
Dose (nvt) - 4×10^{18} thermal; 1×10^{18} fast
Avg. temperature, F

	Cold Pressed		
	<u>Cast</u>	<u>Confined</u>	<u>Unconfined</u>
Center	970	972	1000
Periphery	818	738	602
Capsule evacuated prior to irradiation			
Pressure change above LiH, mm	< 10	< 10	< 10
Change in dimensions of container	none	none	none

Further Observations:

1. Gas build-up did not exceed dissociation pressure.
2. No change in freshly cut surfaces except blackening.
3. No change in lattice spacing (X-ray measurement).
4. Preliminary evidence of microcrystalline fracturing indicated by line broadening of X-ray diffraction patterns.

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IV. General Electric, etc. (continued)

Comments:

1. Hydride-hydroxide reaction driven to completion by evacuation at elevated temperature.
2. No measurable effects noted with regard to gas evolution or dimensional change. A total of 0.5 millimole of helium was anticipated from thermal neutron capture by lithium-6. Lithium hydride becomes plastic at about 900F; operating conditions such that recovery from radiation damage probably occurred.

Helium apparently had sufficient mobility to diffuse to heterogeneous sites, such as low angle boundaries, dislocations, and voids, none of which contained sufficient helium atoms to exert a gas pressure high enough to move dislocations. This mechanism is consistent with (1) no change in lattice parameter, (2) no volume change, and (3) evidence of microcrystalline fracturing.

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- V. General Electric, ANPD (P. Bauer; F. H. Welch; E. P. Kilb)
Irradiation Testing of XMA-1A Shield Materials, TID-6248, TISE,
October 31, 1959. (Also APEX-915, March 1, 1962)

Conditions and Observations:

Lithium hydride: Stainless capsule

1. Cast (C)
2. Cold pressed cylinder (C-P)

Atmosphere: Outgassed and evacuated

<u>Form</u>	<u>Approx. Temp. °F</u>	<u>Time, hr</u>	<u>Dose*</u> <u>nvt</u> <u>/N = x 10^N</u>	<u>Observations</u>
C	250	155-444	5.0/17 - 1.4/18	No effect**
C-P	160-250	157-444	1.1/16 - 1.4/18	No effect
C	300-400	155-444	5.0/17 - 1.4/18	No effect
C-P	400	130-444	3.7/15 - 1.4/18	No effect
C-P	400	744	2.1/19	Powdered, swelled. Capsule leak during test.
C	580	148	6.9/17	No effect
C-P	560-580	118-149	2.5/16 - 9.9/17	No effect
C-P	550	583	3.1/18	Swelled, gas evolved. Capsule leak suspected.
C	800	149-181	5.9/17 - 1.6/18	No effect
C-P	770-800	118-150	4.1/16 - 8.5/17	No effect
C	1000	181	5.9/17	No effect
C-P	1000	117-155	3.3/16 - 1.3/19	No effect

* Dose measured external to capsule.

** No effect: No powdering or swelling of hydride. Limited fracturing during post-test handling.

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V. General Electric, ANPD etc. (continued)

Comments:

1. No deleterious effects to cast or cold pressed lithium hydride enclosed in evacuated capsules after 10^{16} - 10^{19} nvt exposure for 100-180 hours up to 1000F.
2. Swelling and powdering are attributed to exposure to small amounts of air and/or moisture.

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VI. General Electric-ANPD (F. H. Welch)
Properties of Lithium Hydride. IV. Irradiation in the Materials Testing
Reactor, DC 61-7-15, July 13, 1961. (Also APEX-915, March 1, 1962)

Conditions:

Lithium hydride: Cold-pressed in Type 321 capsule.

Atmosphere: Helium

Exposure:	Time	-	Approx. 100 hours
	Flux	-	5×10^{12} nv, thermal
	Dose	-	1.7×10^{18} nvt
	Temperature	-	800F

Observations:

1. No gas evolution.
2. Slight discoloration of hydride. No swelling or powdering.

Comments:

No deleterious effect to hydride enclosed in helium-filled capsule for
100 hours at 800F, 1.7×10^{18} nvt.

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- VII. Lockheed Aircraft-Sunnyvale (1962). Investigation cross-reference:
Union Carbide Corporation (C. W. Hamill; F. B. Waldrop)
Shielding Studies: Neutron Irradiation Damage to a Lithium Hydride Compact,
Y-1454, November 11, 1963.

Conditions:

Lithium hydride: Cold-pressed blocks in aluminum cans. Low humidity
areas not available to supplier during pressing and
handling of hydride. Samples not outgassed.

Exposure: Irradiated at Convair-NARF. Conditions not reported.

Observations:

Can ruptured early in test. Blocks removed and recanned. Further trouble
experienced by apparent rapid growth of the blocks. Spalling of hydride
apparent during post-test examination. Chemical analysis of irradiated
lithium hydride: 11.2% LiOH.

Comments:

Significant atmospheric exposure of the lithium hydride powder during
compacting would lead to formation of hydroxide films on the particles.
The following reaction would take place with rise in temperature during
irradiation:

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VIII. Union Carbide Corporation, Y-12 (C. W. Hamill; F. B. Waldrop)
Shielding Studies: Neutron Irradiation Damage to a Lithium Hydride Compact,
Y-1454, November 11, 1963.

Conditions:

Lithium hydride: Cylinder (6-inch dia. x 6 inches) with stainless steel
honeycomb; cold pressed, sintered at 1100F. Handling
in dry atmosphere (< 35 ppm water). Enclosed in
Type 304 container.

Atmosphere: Outgassed; back-filled with 3 psia helium.

Exposure:	Time	Approx. 45 hours
	Dose (nvt)	1.6×10^{17} (E = 7.1 Nev)
	Temperature, F	300-335

Test interrupted at given time to permit evaluation of
another item. Leak in pressure transducer became
apparent.

Observations:

1. Gas sampled; found to be air.
2. Hydride specimen black throughout.
3. No cracking, spalling, or disintegration of hydride.
4. Diameter increase - 2.4% facing radiation source.
Length increase - 1.4%.
X-ray diffraction indicates 0.03% volume increase in crystal
lattice structure.

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- IX. Los Alamos Scientific Laboratory (F. E. Pretzel, et al.)
Radiation Effects on Lithium Hydride, LA-2463, September 15, 1960.

The effects of radiation upon lithium hydride containing lithium tritide were studied. Isothermal expansions of samples were observed from -196°C (-321°F) to 400°C (752°F) exposed to a flux of tritium β radiation equivalent to 16.8 Mr/hr. In addition to dose rate (or concentration of LiT), volume expansion depends upon time and temperature of exposure.

It was concluded that diffusion, which is time and temperature dependent, plays the predominant role in controlling net radiation damage. Volume expansion occurs by mechanisms involving vacancy diffusion. Radiation damage products (helium and hydrogen) can reduce the expansion rate by escape from the sample. In such cases vacancies diffuse to free surfaces of the crystalline grains and thus escape. To physically outgas, helium and hydrogen must reach the sample surface by (1) direct diffusion, (2) gaseous diffusion through fissures at grain boundaries, and (3) gaseous diffusion through microfissures developed by aggregation of cavities along subgrain boundaries.

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