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To: Mr. K. J. Kelly

From: H. Slotnick

Subject: Irradiation of Lithium Hydride

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 200-1000°F. 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.
**SUMMARY OF LITHIUM HYDROXIDE IRRADIATION STUDIES**

<table>
<thead>
<tr>
<th>Agency</th>
<th>Report</th>
<th>Form</th>
<th>Material</th>
<th>Atmosphere</th>
<th>Temp. F</th>
<th>Time, hr</th>
<th>Dose, nvt</th>
<th>Observations</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>Argonne</td>
<td>ANL-4208</td>
<td>C</td>
<td>Steel</td>
<td>H₂, 50 psi</td>
<td>N.R.**</td>
<td>2160 (3 mos.)</td>
<td>8 x 10¹⁷</td>
<td>1. LiH, blackened</td>
<td>LiH purity unknown.</td>
</tr>
<tr>
<td>NDA</td>
<td>NDA-23</td>
<td>C-P</td>
<td>Type 304</td>
<td>Vacuum or helium</td>
<td>100-610</td>
<td>240-335</td>
<td>1.55 x 10¹⁵-1.12 x 10¹⁸</td>
<td>1. LiH, blackened. 2. LiH expansion, disintegration. 3. Pressure increase. 4. No change in lattice spacing.</td>
<td>1. Growth and disintegration attributed to irradiation. 2. Hydrogen evolved from LiOH impurity.</td>
</tr>
<tr>
<td>GE-ANFD</td>
<td>APEX-323; APEX-915</td>
<td>C; C-P</td>
<td>Type 304 (outgassed)</td>
<td>Vacuum</td>
<td>970-1000</td>
<td>100</td>
<td>4 x 10¹⁸ thermal 1 x 10¹⁸ fast</td>
<td>1. Pressure increase, &lt; 10 mm. 2. No change in container dimensions. 3. LiH, blackened. 4. No change in lattice spacing.</td>
<td>Test temperature probably contributed to recovery from radiation damage, no deleterious effect.</td>
</tr>
<tr>
<td>GE-ANFD</td>
<td>TID-62A8; APEX-915</td>
<td>C; C-P</td>
<td>SS (outgassed)</td>
<td>Vacuum</td>
<td>160-400</td>
<td>110-444</td>
<td>3.7 x 10¹⁵ 1.4 x 10¹⁸</td>
<td>No powdering or swelling; limited fracturing during post-test handling.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>Notes:</strong></th>
</tr>
</thead>
<tbody>
<tr>
<td>* Form: C, Cast; C-P, Cold Pressed</td>
</tr>
<tr>
<td>** N.R.: Not Reported</td>
</tr>
<tr>
<td>** H.S.: Not Reported</td>
</tr>
<tr>
<td></td>
</tr>
<tr>
<td>** Confidential - D.D. **</td>
</tr>
</tbody>
</table>
To: Mr. K. J. Kelly

Summary of Lithium Hydride Irradiation Studies
(Continued)

Test Conditions

<table>
<thead>
<tr>
<th>Agency</th>
<th>Report</th>
<th>L1H Form*</th>
<th>Container</th>
<th>Atmosphere</th>
<th>Temp. F</th>
<th>Time, hr</th>
<th>Dose, nvt</th>
<th>Observations</th>
<th>Comments</th>
</tr>
</thead>
<tbody>
<tr>
<td>GE-ANPD (1961)</td>
<td>DC 61-7-15; APEX-915</td>
<td>C-P</td>
<td>Type 321</td>
<td>Helium</td>
<td>800</td>
<td>100</td>
<td>$1.7 \times 10^{18}$</td>
<td>1. No gas evolution.</td>
<td>No deleterious effect.</td>
</tr>
<tr>
<td>Lockheed (1963)</td>
<td>T-1415</td>
<td>C-P Block</td>
<td>Type 304</td>
<td>Helium</td>
<td>300-335</td>
<td>45</td>
<td>$1.6 \times 10^{17}$</td>
<td>1. Can rupture; rapid growth of LiH.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>with Honeycomb (outgassed)</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>2. Hydride spalling.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>3. LiH (post-test)-11.2% LiOH</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>test prematurely terminated.</td>
<td>due to air leak.</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Test prematurely terminated due to air leak.
ENUMERATION OF LITHIUM HYDRIDE IRRADIATION STUDIES


Conditions:
- Lithium hydride: Cast in steel cans.
- Atmosphere: Hydrogen, 50 psi
- Exposure:
  - Time: 3 months
  - Flux: $10^{11}$ ny
  - Dose: $8 \times 10^{-7}$ 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.

Conditions:

Lithium hydride: Form not reported.

Atmosphere: Not reported.

Exposure:

<table>
<thead>
<tr>
<th>Time</th>
<th>Flux</th>
<th>Dose</th>
<th>Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>About 3 months</td>
<td>About half maximum flux (?)</td>
<td>Not reported</td>
<td>152°C (305°F), 239°C (462°F)</td>
</tr>
</tbody>
</table>

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.
III. Nuclear Development Corporation of America (B. Minushkin)

Conditions and Observations:
Lithium hydride: Powder-pressed

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

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 600°F. Also the cohesive energy of powder-pressed lithium hydride was probably insufficient to resist radiation damage caused by thermal spikes and thermal stresses.
To: Mr. K. J. Kelly

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) - $1.25 \times 10^{13}$ thermal; $0.3 \times 10^{13}$ fast
Dose (nvt) - $4 \times 10^{18}$ thermal; $1 \times 10^{18}$ fast
Avg. temperature, F

<table>
<thead>
<tr>
<th></th>
<th>Cast</th>
<th>Confined</th>
<th>Unconfined</th>
</tr>
</thead>
<tbody>
<tr>
<td>Center</td>
<td>970</td>
<td>972</td>
<td>1000</td>
</tr>
<tr>
<td>Periphery</td>
<td>818</td>
<td>738</td>
<td>602</td>
</tr>
</tbody>
</table>

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.
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.
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

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

* Dose measured external to capsule.

** No effect: No powdering or swelling of hydride. Limited fracturing during post-test handling.
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 1000°F.

2. Swelling and powdering are attributed to exposure to small amounts of air and/or moisture.
VI. General Electric-ANPD (F. H. Welch)

Conditions:
Lithium hydride: Cold-pressed in Type 321 capsule.
Atmosphere: Helium
Exposure:
- Time: Approx. 100 hours
- Flux: $5 \times 10^{12}$ nvt, thermal
- Dose: $1.7 \times 10^{18}$ nvt
- Temperature: 800°F

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 800°F, $1.7 \times 10^{18}$ nvt.

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:

\[ \text{LiH} + \text{LiOH} \rightarrow \text{Li}_2\text{O} + \text{H}_2 \]
To: Mr. K. J. Kelly

VIII. Union Carbide Corporation, Y-12 (C. W. Hamill; F. B. Waldrop)

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:

<table>
<thead>
<tr>
<th>Time</th>
<th>Approx. 15 hours</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dose (nvt)</td>
<td>$1.6 \times 10^{17}$ (E = 7.1 Mev)</td>
</tr>
<tr>
<td>Temperature, F</td>
<td>300-335</td>
</tr>
</tbody>
</table>

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 - 1.2%.
   X-ray diffraction indicates 0.03% volume increase in crystal lattice structure.
IX. Los Alamos Scientific Laboratory (F. E. Pretzel, et al.)

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 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.