Identification of Lithium Hydride and its Hydrolysis Products with Neutron Imaging

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Abstract

In this study, lithium hydride (LiH) and its hydrolysis products were investigated non-destructively with neutron radiography and neutron computed tomography. Relative neutron transmission intensities (I/I_0) were measured for LiOH, Li₂O and LiH, and their linear attenuation coefficients calculated from this data. We show that ⁷Li is necessary for creating large differences in I/I_0 for facile identification of these compounds. The thermal decomposition of LiOH to Li₂O was also observed with neutron radiography. Computed tomography shows that the samples were fairly homogeneous, with very few macroscopic defects. The results shown here demonstrate the feasibility of observing LiH hydrolysis with neutron imaging techniques in real time.

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1. Introduction

Lithium hydride (LiH) is a light, hydrogen dense material which has received attention due to its use as a hydrogen storage medium [1–7], whilst its corrosion products, particularly Li₂O, have been studied as candidate tritium breeding materials for use in commercial nuclear fusion [8–10]. LiH reacts readily with moisture to irreversibly form surface corrosion layers of Li₂O, LiOH and LiOH·H₂O [11–18]. This poses problems for storage and handling and the material must be kept and processed in vacuum or an ultra-dry inert gas glove box. Even in these conditions, the material slowly hydrolyzes due to trace quantities (less than 1 ppm) of moisture [19, 20]. Corrosion layer thickness varies in size and morphology with the environmental conditions [21–23]. On the other hand, controlled LiH hydrolysis has been proposed as an efficient hydrogen generator for portable applications [3, 5, 6]. Hydrolysis is given by either:

Alternatively:

In both these reaction schemes, a thin layer of Li₂O forms at the interface between LiH and LiOH. This process is thought to be a diffusion limited process and results in a trilayer LiH – Li₂O – LiOH corrosion system. There is a growing consensus, both experimentally and theoretically, that under ambient conditions, the hydrolysis reaction proceeds via the formation of Li₂O before LiOH (*i.e.* formed by equations 1 and 2) [11, 15, 24, 25]. In other words, Li₂O is favored by lower moisture exposures and at higher temperatures. At 298 K, the thickness of the Li₂O layer is reported to be in the 10 nm range [15]. Upon heating to 523 K, the conversion of LiOH to Li₂O has been observed on a LiH substrate [13, 21, 26]:

Several analytical techniques have been applied to study these reactions, including infrared spectroscopic methods, x-ray diffraction, gas pressure measurements or thermal decomposition methods. By measuring the –OH resonance, diffuse reflectance infrared Fourier transform (DRIFT) spectroscopy has shown the presence of LiOH on the surface of LiH following exposure to moisture. DRIFT spectroscopy has also shown that –OH is lost upon heating, with hydroxide being converted to oxide [13, 20, 27]. Raman spectroscopy has also been recently applied to the study of LiH corrosion, in particular the measurement of the Li₂O phonon [28–30]. Thermal decomposition and diffraction have provided kinetic and mechanistic information on the hydrolysis of LiH [19] and the subsequent thermal decomposition of the LiOH layer [21–23].

Neutron characterization offers an alternative and as

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yet unexplored method of probing the nature of LiH and its hydrolysis products. Antecedent studies of LiH with neutrons have, for example, focussed on crystallography [31] and Compton scattering experiments [32]. The use of LiH as a neutron shielding material has also recently been discussed [33]. As opposed to x-ray radiation, neutrons interact strongly with Li and H and thus are attractive as a probe of LiH and its corrosion products. X-ray attenuation is related to the atomic number of the atoms present in a material and therefore LiH is amongst the poorest x-ray absorbers. In the case of neutrons, attenuation is determined by the nuclear structure of a particular atom, with natural Li and H exhibiting very high attenuation characteristics. In this report, neutron radiography and computed tomography (CT) data are obtained for the LiH corrosion system. The intentions of these measurements were: to experimentally determine the relative transparencies of LiH, Li₂O and LiOH to cold neutron irradiation; to observe the thermal dehydration of LiOH; and to evaluate the possibility of imaging LiH corrosion in real time with neutron imaging.

2. Experimental

2.1. Materials

Samples were produced from the compaction of powders at ~ 20.7 MPa (3000 psi) in a uniaxial Carver die press using a 6 mm diameter die set. To ensure sufficient neutron transmission, each 6 mm sample was sanded down by hand to a thickness of 3 mm parallel to the neutron transmission axis. Table 1 shows the isotopic composition of the samples used here. The reason for selecting Li compounds enriched with ⁷Li is two fold. Firstly, samples containing natural Li have sufficient ⁶Li to prevent any neutron transmission being observable. Secondly, we wish to observe LiH chemistry and thus we are interested in spatial distributions of H and O and therefore variations in attenuation due to Li is not of particular interest; O and H are the mobile species in this system. Instead of using LiH enriched with the ⁷Li isotope, the decision was made to use Li²H which reduces neutron attenuation via the use of the ²H (D) isotope. We note that the use of ⁷Li [34] and D [35] in neutron characterization experiments has previously been described. Also the use of D afforded the possibility of observing D mobility in the Li₂O and LiOH layers after thermal treatment. As shown in table 1, the LiH samples are predominantly ²H and will be referred to henceforth as LiD. Also for brevity 99.99% ⁷LiOH will be referred to as LiOH. LiD samples were approximately 90% dense compared to the theoretical maximum calculated from x-ray diffraction [15, 36]. Li₂O and LiOH both exhibited densities in the region of 95–98% of their theoretical densities [15].

Five samples were produced, four layered and one a mixture of powders. A summary of sample composition, nominal layer thickness and thermal treatment is presented

Table 1: Isotopic composition of the samples used in this study. Note that Li₂O (2) was produced via heat treatment of LiOH, see table 2 for details. Natural O was used in all samples.

Compound	Isotopic Composition (%)			
	⁶ Li	⁷ Li	¹ H	² H
LiH	7.59	92.41	8	92
LiOH	0.01	99.99	99.99	0.01
Li ₂ O (1)	2	98		
Li ₂ O (2)	0.01	99.99		

in table 2. Of the four layered samples, the thickness of the Li₂O layer was varied (0.05 mm and 0.16 mm) to assess its effect on reducing further hydrolysis of the LiD during the thermal decomposition of LiOH (equation 5). This effect is known to occur in samples containing native corrosion products of LiD, in that further hydrolysis is a diffusion limited process across the Li₂O layer [21, 23]. We note that these Li₂O layers are too large to be considered representative of the ambient LiD hydrolysis system but they still have the ability to give useful H₂O transport information across the Li₂O layer. Thermal treatment was performed *ex-situ* on samples 1 and 5. Samples were removed from the glovebox, wrapped in Al foil before being transported briefly in air to be loaded in an Al vessel. The Al vessel was immediately evacuated to a pressure of approximately 3 Pa. The vessel was placed in a tube furnace and was heated in 100 K increments to a final temperature of 673 K in about 30 minutes. The temperature was monitored using a K-type thermocouple positioned on the vessel. Pressure increased to 7.4 Pa immediately after the temperature of 673 K was reached and returned to 3 Pa after 45 minutes at temperature. After 3 hours, the furnace was turned off and samples were left in the tube to cool, while maintaining the vacuum.

Table 2: Chemical composition and thermal treatment of samples used in this study. The dimensions given are nominal. The pressure refers to the base pressure during heat treatment. Thermal treatment of LiOH converted it to Li₂O (2).

Sample	Layer thickness (mm)	Thermal Treatment
	LiD : Li ₂ O (1) : LiOH	
1	1 : 0.05 : 1	673 K, 3 h, 3 Pa
2	1 : 1 : 1	
3	powder LiD / LiOH	
4	1 : 0.16 : 1	
5	1 : 0.16 : 1	

2.2. Neutron Imaging

Neutron radiography was performed using the CG-1D imaging instrument at the High Flux Isotope Reactor, sit-

uated within Oak Ridge National Laboratory (Tennessee, USA) [37]. The instrument provides a cold polychromatic neutron beam with a wavelength spread of 0.08 – 0.6 nm, with an intensity maximum at 0.26 nm. The overall neutron flux is estimated to be $5 \times 10^6 \text{ cm}^2\text{s}^{-1}$. Neutron radiographs were captured on a LiF/ZnS scintillator attached to a charge coupled device (CCD) camera. The camera was Peltier cooled to about 210 K to reduce thermal noise in the electronics. The resolution of the instrument was determined with a Gd coated grid to be approximately 100 μm . To collect CT data, each sample was rotated through 180° at an increment of 0.17°.

Neutron imaging relies on the attenuation of a neutron beam as it traverses a given material. For each pixel (x, y) the observed neutron flux, I , as a beam of neutrons transverse a sample is given by the *Beer-Lambert law* [38–40]:

$$I(x, y) = I_0(x, y) \exp(-\Sigma(x, y)d(x, y)) \quad (6)$$

where I_0 is the incident neutron flux, Σ is the linear neutron attenuation coefficient and d is the sample thickness, in the direction parallel to the neutron beam.

At each angle a two dimensional radiograph was obtained with an acquisition time of 85 s. Prior to imaging the samples, a dark field and an open beam image were recorded. The dark field image was obtained with the neutron beam stopped to record any dark currents in the CCD apparatus. The open beam image was collected for a neutron beam without any sample, which is equivalent to the incident beam intensity, I_0 . With a sample placed in the beam, the intensity on the detector was then the uncorrected transmitted beam intensity, I' . Since the samples were enclosed within an Al vessel (to prevent atmospheric corrosion), the effect of the neutron attenuation from the sample holder must be accounted for. The corrected relative transmission (I/I_0) of the sample is therefore:

$$\frac{I}{I_0} = \frac{I'}{I_0} + \frac{I_a}{I_0} = \frac{I' + I_a}{I_0} \quad (7)$$

where I' is the uncorrected transmission and I_a is the attenuation due to the Al can. The relative transmission of the Al can was measured to be 0.93 in a region close to the samples, hence the relative attenuation (I_a/I_0) is 0.07. This value is added to each measurement of I'/I_0 to give I/I_0 . In all figures and tables, the corrected relative transmission is given.

2.3. Chemical Characterization and Microscopy

In addition to neutron imaging, the pressed samples were studied with optical microscopy and Raman spectroscopy. Specifically, similar samples were evaluated by optical microscopy in air, before and after the heat treatment to evaluate if dimensional changes with temperature and pressure occurred. Raman spectroscopy was conducted on the Li_2O sample to confirm its integrity and also on a heat treated sample at different locations. Each sample was contained in a CF40 cell with a glass window

for Raman data acquisition. The instrument utilized was a B&W TEK i-Raman Plus with a microscope attachment (20x objective) with 532 nm excitation.

3. Results and Discussion

3.1. Preliminary Characterization

Optical images of a LiD / Li_2O (1) / LiOH sample is shown in figure 1. It shows a sample of nominal dimensions of 1 mm : 0.16 mm : 1 mm, pre and post heating (equivalent to samples 4 and 5, respectively, in table 2). The layer thickness was measured to be LiD – 1255 μm , Li_2O – 118 μm , and LiOH – 1000 μm . The observed roughness of these layers is $\sim 50 \mu\text{m}$ and $\sim 10 \mu\text{m}$ for the LiD – Li_2O (1) interface and the Li_2O (1) – LiOH interface, respectively. After heat treatment, it was observed that LiD was covered with a white coating, especially at the edges of the layer and there was also evidence of separation of LiOH from Li_2O . Within the limits of the interface roughness, there was no measurable change in layer thickness after the heat treatment. The white coating on this sample was identified by Raman to be LiD with a minor amount of Li_2O , figure 2(a). This Raman spectrum shows a broad feature centered around 2600 cm^{-1} which is likely to be LiD fluorescence similar to that previously observed for LiH [29]. The symmetric stretch for Li – O – Li is barely discernible at 517 cm^{-1} . There was spatial variation in the intensity of the Li_2O feature, indicating that there was a dispersion of Li_2O within the LiD matrix. Also characterized by Raman was the Li_2O layer. This was done to check for Li_2O hydrolysis when the sample was briefly exposed to atmosphere for pressing and thermal treatment. Figure 2(b) show a typical Raman spectrum for this layer, revealing that it was predominantly Li_2O with minor amounts of LiOH (at 3666 cm^{-1}) and Li_2CO_3 (at 1594 cm^{-1} and 1089 cm^{-1}) [13]. We conclude by stating that both LiD and Li_2O layers are of high purity with only a minor amount of other compounds detectable.

3.2. Neutron Radiography

3.2.1. Neutron Radiography – Imaging

Representative neutron radiographs of LiD and its related corrosion products are shown in figure 3. The composition of samples in figure 3 are annotated on the figure and described in more detail in table 2. They show strong contrast between the LiD, LiOH and Li_2O (2) (99.99% ^7Li) layers of each sample. The LiD layer in each sample is rather opaque to neutrons, while the Li_2O (2) layer is highly transparent to neutrons. The Li_2O (1) (98% ^7Li) and LiOH layers are both relatively strong neutron absorbers due to their ^6Li and H content, respectively. Samples (2) and (4) particularly show that there is very little difference in relative transmission between the Li_2O (1) and the LiOH layers. The intention of sample (3), a mixture of LiD and LiOH, was to observe the solid state reaction between LiD and LiOH (equation 4). It is clear that

Figure 1: Optical micrograph of LiD / Li₂O (1) / LiOH sample prior to and after thermal treatment (673 K) in rough vacuum (3 Pa). The left hand layer is LiD, the middle layer is Li₂O (1) and the right hand layer is LiOH.

Figure 2: Raman spectra of (a) LiD portion of thermally treated LiD / Li₂O (1) / LiOH sample, showing a minor Li₂O feature; (b) as fabricated Li₂O sample, containing minor amounts of LiOH and Li₂CO₃.

there is an increase in neutron transparency compared to pure LiD, which is a result of the presence of the less attenuating LiOH. The generation of Li₂O would reduce neutron attenuation further and thus may cause localized variations in neutron transparency. Although there is evidence that there are variations in neutron transparency, it is difficult to say whether these are a result of variations in O distribution due to Li₂O or LiOH; or are simply an artefact of the mixing process. Layered samples provide much more certainty in the assignment of compounds and the chemistry involved. Samples (1), (4) and (5) were prepared to explore the thermal decomposition of LiOH (equation 5) and the effect of the evolved H₂O on LiD, albeit with a artificially thicker Li₂O (1) layer than occurs naturally. Comparing samples (4) and (5), it is clear that the LiOH layer has decomposed to Li₂O. The only plausible explanation is that the strongly attenuating H has been lost from this material, as per equation 5. To ascertain the effect of LiOH decomposition on LiD, it was necessary to examine line profiles of the neutron radiographs, which are described in the next section.

3.2.2. Neutron Radiography – Line Profile Analysis

To further analyze radiographs, line profiles were obtained across the layers, which are displayed in figure 4. Line profiles are displayed for samples (1), (2) and (5). A line profile for sample (3) does not make sense and sample (4) has essentially the same profile as sample (2) albeit with a smaller Li₂O (1) layer, hence these profiles are not displayed. The large differences in neutron transparency, among LiD, Li₂O (2) and LiOH represents a chemical fingerprint to easily identify the compound involved in the hydrolysis of LiH. This effect could be exploited in future studies to view hydrolysis in real time or to map the extent of the hydrolysis reaction in three dimensional space, similar to what has been demonstrated for H₂O transport in concrete [41], soil [42] and fuel cells [43]. The red line in figure 4 shows that the difference in neutron transparency between Li₂O (1) and LiOH is rather slight, the two layers are only just discernible from each other, with the difference being about 4%. A direct comparison can be made of the neutron absorption between Li₂O (1) and Li₂O (2). A 3 mm sample of Li₂O (1) attenuates 49% of neutrons while Li₂O (2) attenuates just 4% of neutrons.

The line profile of sample (2) indicates that the interface is ~200 μm wide. This is slightly broader than the interfacial roughness measured by optical microscopy,

Figure 3: Representative neutron transmission radiographs obtained for samples 1 to 5 (see table 2 for details). The isotopic composition of Li_2O (2) and LiOH is 99.99% ^7Li while Li_2O (1) has 98% ^7Li . LiD is natural Li .

probably due to the fact that neutron radiography penetrates the whole sample while optical microscopy is essentially a surface measurement. This sample has had no thermal treatment and thus the roughness measured from this sample is the natural roughness of the interface. The line profile of sample (1) (blue line in figure 4) shows that the interface is broader than sample (2), $\sim 450 \mu\text{m}$. Sample (1) contains a Li_2O (1) layer that is nominally $50 \mu\text{m}$ thick. This leaves a difference of about $200 \mu\text{m}$, which is either: an increase in roughness due to a degree of delamination as in sample (5); or thermally induced hydrolysis of the LiD layer, through liberation of H_2O from the LiOH layer. Optical microscopy (figure 1) would suggest that thermally induced hydrolysis cracking of this sample is rather small (of the order of $10 \mu\text{m}$), while the whitening (Li_2O formation) at the interface is of the order of $100 \mu\text{m}$, which is consistent with the neutron imaging. As LiD is formed of natural Li , hydrolysis of this layer would mean that the neutron transparency of this layer would tend toward Li_2O (1) with a relative transmission of 0.51, consistent with line profile analysis. The outside of the LiD layer has a similar roughness, $\sim 220 \mu\text{m}$, in neutron radiographs of both samples before (sample (2)) and after (sample (1)) thermal processing. Optical microscopy, figure 1, shows that oxide layer at the outside of the LiD layer is $\sim 150 \mu\text{m}$ thick. The lack of any difference in the neutron radiography (a bulk measurement) between pre and post thermal treated samples would suggest that hydrolysis at this interface is a surface effect and does not penetrate particularly far into the bulk of the material. Raman spectroscopy (figure 2) is supportive of this assertion as the data shows that Li_2O is only present in small quantities on the LiD surface.

The linear attenuation coefficient, Σ , can be measured from experiment, as described by equation 6. It can also be estimated from the physical properties of the material. Σ is given by [44]:

$$\Sigma = \sigma(\lambda)N = \sigma(\lambda)\frac{\rho N_A}{M} \quad (8)$$

where Σ is the linear attenuation coefficient, $\sigma(\lambda)$ is the total neutron cross section for neutrons of wavelength λ , N is the atom density, ρ is the material density, N_A is Avogadro's number and M is the molar mass. The total neutron cross section is the sum of the scattering and absorption cross sections. Table 3 shows the absorption and scattering cross sections for isotopes relevant to this study. Table 4 shows the physical properties; derived and measured values of Σ ; and the expected and measured I/I_0 for a 3 mm thick sample.

The value obtained for Σ for LiD is in reasonable agreement with the calculated value. It is slightly lower than expected from calculation. For Li_2O (1) and Li_2O (2), there is also good agreement between calculation and experiment. The value of Σ is within error in the case of Li_2O (2) and close to in the case of the Li_2O (1). On the other hand, there are significant differences between the value obtained for Σ from calculation and experiment for

Table 4: Relative neutron transmission (I/I_0) of compounds relevant to this study. ρ is material density, M is molar mass, N is the atom density, Σ is the linear attenuation coefficient, which was calculated from equation 8. Percentages refer to the isotopic concentration of ^7Li , except LiD where it is labelled individually.

Compound	ρ (g cm ⁻³)	M (g mol ⁻¹)	N ($\times 10^{22}$ cm ⁻³)	Σ (cm ⁻¹)		I/I_0	
				Calc.	Exp. (± 0.15)	Calc.	Exp. (± 0.01)
92% LiD 8% LiH	0.86	8.86	5.86	5.00	4.36	0.22	0.27
99.99% $^7\text{LiOH}$	1.47	23.98	3.65	3.21	1.99	0.38	0.55
98% $^7\text{Li}_2\text{O}$ (1)	2.02	29.98	4.05	1.81	2.24	0.58	0.51
99.99% $^7\text{Li}_2\text{O}$ (2)	2.02	29.99	4.05	0.29	0.13	0.92	0.96

Figure 4: Line profiles derived from figure 3. Blue line: line profile from sample (1), Li_2O (2) / Li_2O (1) / LiD. Red line: sample (2) LiD / Li_2O (1) / LiOH. Black line: sample (5) and LiD / Li_2O (1) / Li_2O (2). Li_2O (2) and LiOH have 99.99% ^7Li content while Li_2O (1) has 98% ^7Li .

Table 3: Thermal neutron ($\lambda = 0.18$ nm) cross section data for relevant elements and isotopes. σ_s is the neutron scattering cross section and σ_a is the neutron absorption cross section. Data sourced from [39, 45–48].

Element	σ_s (barns)	σ_a (barns)
^1H	82.02	0.33
^2H (D)	7.64	5×10^{-4}
^6Li	0.97	940
Li	1.37	70.5
98% ^7Li	1.39	18.85
^7Li	1.40	0.05
O	4.23	2×10^{-4}

LiOH. The experimentally obtained attenuation parameters for LiOH is significantly less attenuating than would be expected from calculation. There are several plausible reasons for these discrepancies. For Li_2O (1), the measured I/I_0 is lower (more attenuating) than expected from calculation. This is likely a result of contamination. The Li_2O (1) layer was formed in ambient laboratory conditions, which has been shown in figure 2 to contain small but detectable levels of LiOH and Li_2CO_3 . LiOH contains H, hence it is a more effective neutron moderator and may explain why the observed attenuation is slightly greater than the calculated value. For Li_2O (2) and LiOH, the measured I/I_0 is higher (less attenuating) than expected from calculation. The cause may be due to the fact that I/I_0 in table 4 is calculated for a single neutron wavelength ($\lambda = 0.18$ nm), while the neutron beam here is polychromatic ($\lambda = 0.08 - 0.6$ nm). Beam hardening, whereby low energy neutrons are preferentially removed, may also contribute the observed deviations in I/I_0 and Σ [49–51]. This effect will shift the observed I/I_0 to a higher value and will therefore reduce the observed Σ . It is known to be particularly evident in strong neutron absorbers, hence it would be expected to contribute here. The calculation also assumes idealized material density. As the materials are fabricated from the compaction of powders, there will inevitably be residual porosity present. In the samples used here, LiD was measured to be $\sim 90\%$ dense, while Li_2O (1) and LiOH were measured to be 95–98% dense. Clearly, a decrease in the actual material density, compared to the calculated value, will increase neutron transparency. Furthermore, the Li_2O (2) layers have been formed by the dehydration of LiOH. Escape of H_2O during this process has been shown to cause spallation and cracking and thus will increase the porosity of the material [21–23], further increasing neutron transmission. All of the above factors are likely to have contributed to an increase in the experimentally measured I/I_0 , in comparison with the calculated value.

3.2.3. Neutron Radiography – Tomography

CT allowed for the three dimensional visualization of the LiD, Li_2O (1), Li_2O (2) and LiOH layers. A three dimensional rendered volume of samples (4), (2) and (1) are shown in figures 5(a), (b) and (c), respectively. In sample (4), the LiD layer is clearly discernible from the

LiOH layers; however, the Li_2O (1) / LiOH interface is indistinct with a boundary that is not easily identifiable. There is a small defect evident at the surface of LiD layer. The sample appears to have been chipped which probably occurred during sample fabrication.

Figure 5(b) shows a three dimensional rendered volume of sample (2). The main difference between this and figure 5(a) is that the Li_2O (1) layer is larger. Again the boundary between the Li_2O (1) and LiOH layers is unclear. It also reveals that this sample is highly homogeneous within each layer, with only a small defect visible at the LiD / Li_2O (1) interface, which was possibly introduced into the sample during pressing. Line profile analysis, figure 4, is the more reliable method for distinguishing between these two layers. In addition, the tomography of this sample reveals highly neutron attenuating impurities, which appear as two near spherical intense red features in figure 5(b). These impurities are likely to be a result of contamination during sample preparation.

Figure 5(c) shows a two dimensional cut through sample (1). The Li_2O (2) layer was too transparent to be effectively depicted as a colormap, hence a grayscale image is presented here. The two dimensional cut through the LiD / Li_2O (2) sample revealed that each layer was almost entirely homogeneous, with only a small amount of roughness observable at the interface. During thermal decomposition of LiOH, H_2O escapes from the material, which has been previously shown to cause cracking and spall from the surface [23]. However, there is no conclusive evidence of cracking in the Li_2O (2) layer in this sample. Small variations in neutron transmission (less than 2%) are apparent, but this is too low to assign to cracking. The fact that Li_2O (2) is highly transparent ($I/I_0 = 0.96$, table 4) hinders the observation of cracking. Indeed, the resolution of the instrument (100 μm) may not be sufficient to observe cracking, which has been previously reported to be of the order of 10 μm .

4. Conclusions

Neutron radiography has been shown to be an effective technique in observing LiH and its hydrolysis products. The large differences in the relative transmission of neutrons through LiD, $^7\text{LiOH}$ and $^7\text{Li}_2\text{O}$ allow for unambiguous identification of these compounds. For these compounds, Σ was measured and compared to calculated values. Agreement was generally good, however, there was a tendency for calculation to overestimate the observed values of Σ . This deviation was ascribed to a combination of beam hardening, the fact that a polychromatic neutron source was employed, and to residual sample porosity. Neutron imaging has also proven to be effective in the observation of the thermal conversion of LiOH to Li_2O , where the loss of H in this process is clear. There is also an increase in the width of the LiD – LiOH interface, suggestive of further hydrolysis of the LiD layer. Tomographic methods give further insight into the three dimensional

Figure 5: Three dimensional rendered volume of samples. (a) sample (4), LiD / Li_2O (1) (160 μm) / LiOH specimen. The intense red area is LiD and the intense green area is LiOH. The diffuse green area surrounding the sample is the sample container. (b) sample (2), LiD / Li_2O (1) / LiOH specimen. The intense red area is LiD and the intense green areas are Li_2O and LiOH. The diffuse green area surrounding the sample is the sample container. (c) sample (1) LiD / Li_2O (1) (50 μm) / Li_2O (2) sample. The bright area is LiD and the transparent area is Li_2O (2).

structure of these layers, primarily impurities and structural defects. The only detriment identified is the spatial resolution, approximately 100 μm , which precludes observing the initial stages of the hydrolysis reactions. The neutron imaging data provided here gives a basis for future research into LiH hydrolysis, especially on the mesoscopic and macroscopic scales. In particular, the data presented here shows that three dimensional, time dependent observations of processes such as LiH hydrolysis and LiOH thermal decomposition are feasible with neutron imaging and may contribute significant additional insight into these phenomena.

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