**Introduction**

- The recent interest in a hydrogen-based fuel economy has renewed research into metal hydride chemistry.
- Many of these compounds react readily with water to release hydrogen gas and form a caustic.
- Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFT) has been used to study the hydrolisis reaction. The LiOH stretch appears at 3670 cm⁻¹.
- Raman spectroscopy is a complementary technique that employs monochromatic excitation (laser) allowing access to the low energy region of the vibrational spectrum (<600 cm⁻¹).
- Weak scattering and fluorescence typically prevent Raman from being used for many compounds.
- The role of Li₂O in the moisture reaction has not been fully studied for LiH. Li₂O can be observed by Raman while being hidden in the infrared spectrum.

**Fluorescence**

- The significant feature in the Raman spectrum of LiH at most excitation wavelengths is fluorescence.
- As a result, high concentrations of LiOH or Li₂CO₃ are required to be observed.
- Baseline correction and exotic deconvolution routines did not improve spectra or sensitivity.
- FT-Raman (λ = 1064 nm), which typically is used to overcome fluorescence resulted in two distinct fluorescence features.

**Photoluminescence**

- Absorbed laser photons are spontaneously emitted from LiH at specific wavelengths.
- The intensity of these photons is much greater than Raman scattering limiting Raman sensitivity.
- The Raman spectral region (100-4000 cm⁻¹) is highlighted for multiple excitation wavelengths in order to pick the best laser probe.
- UV wavelengths offer high intensity and low luminescence.

**UV Raman**

- Excitation with a UV (<400 nm) laser has been shown recently to effectively overcome autofluorescence in many organic and biological compounds.
- The energy gap between the UV excitation and initiation of autofluorescence is such that the entire Raman scattering spectrum can be observed free of obstruction.
- Raman intensity is also much stronger since intensity is proportional to 1/λ⁴. Stretching and phonon vibrations for LiOH observed.

**Fiber Optic Delivery**

- Due to pyrophoricity concerns, spectroscopy within a controlled environment is desired. This can be achieved through fiber optics.
- Advances in UV fibers and optical filters make fiber optic delivery possible at 325 nm.
- Renishaw, Inc. designed a UV fiber optic Raman probe for use in environmental enclosures.

**In Situ Hydrolysis**

- Compacted LiH was exposed to 100 %RH air for 1 day.
- The UV Raman probe measured spectra every minute.
- Both Li₂CO₃ and LiOH grew in intensity
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  \text{LiH} + \text{H}_2\text{O} \rightarrow \text{LiOH} + \text{H}_2(\text{g})
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  \[
  \text{LiOH} + \text{CO}_2 \rightarrow \text{Li}_2\text{CO}_3 + \text{H}_2(\text{g})
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- Fluorescence bleached away over time
- Heating the sample (350 °C) under vacuum converts LiOH to Li₂O which is observed by Raman directly.