Thermal Desorption Spectroscopy studies of hydrogen retention on FLIRE

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Outline

- Introduction
- Thermal Desorption Spectroscopy (TDS) system description
- Experimental results
- Conclusions/Future work

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Importance of H retention measurements

- QUESTION: How much hydrogen gets carried away by a liquid PFC?
- If too high, tritium inventory may be unacceptable – (not enough tritium in existence)
- If too low, recycling benefits may not be realized
- FLIRE TDS system can provide answers!

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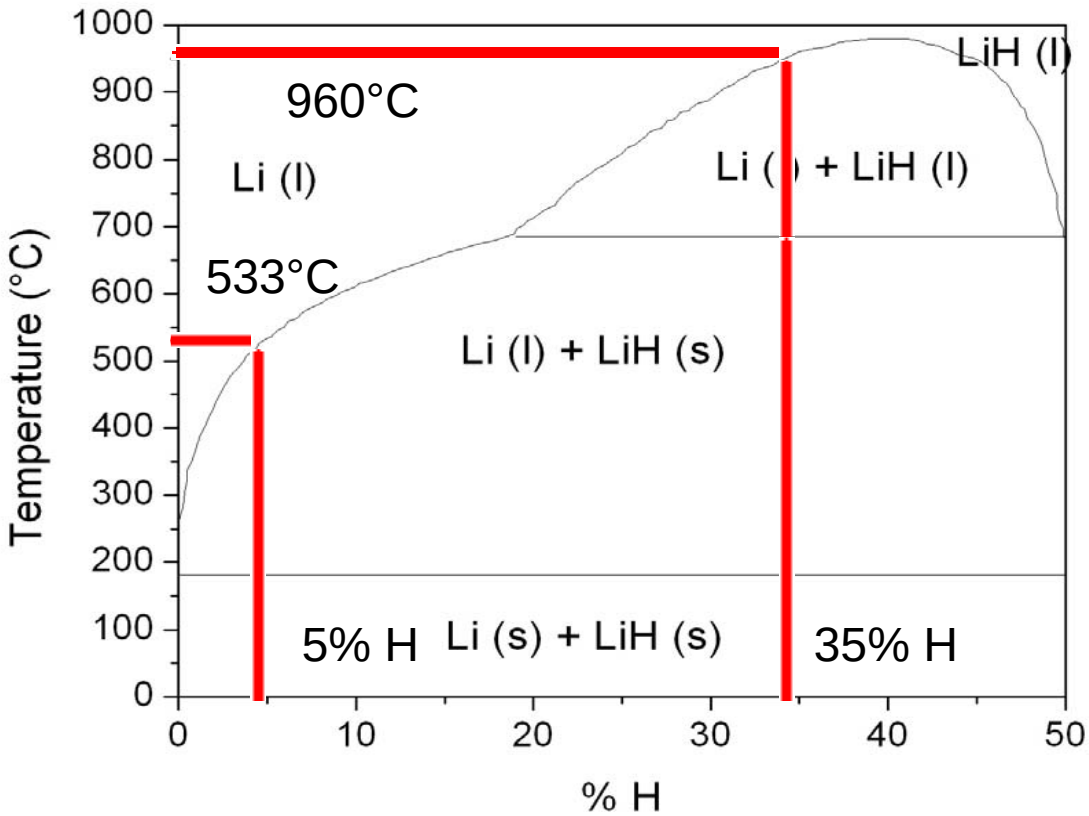
Solubility of H in Li

- Unlike He, H may be trapped in the Li for longer time
- Hydrogen is soluble in lithium
- Hydride formation is very likely.
- Below 19% no liquid LiH phase is formed



* Data taken from: The chemistry of the liquid alkali metals, C.C. Addison. John Wiley & Sons. 1984.

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Need for a Thermal Desorption Spectroscopy (TDS) system

- Quantification of hydrogen content – how much is actually retained in the flowing liquid Li
- Study of hydrogen desorption mechanisms from lithium surfaces
- TDS can offer detailed temperature measurements of hydrogen release
- Higher temperatures can be achieved in the TDS chamber than anywhere else on FLIRE

What do we expect?

- Strong hydrogen desorption from the liquid lithium at a specific temperature, correlated to the hydrogen content (from the phase diagram)
- Evaluate the effect of gaseous impurities on the desorption process
- Quantify hydrogen long-term retention on the Li metal

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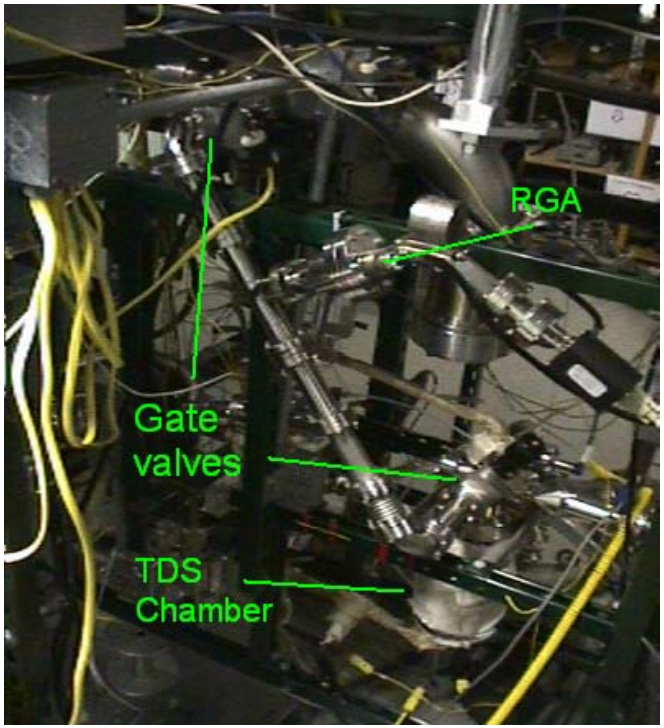
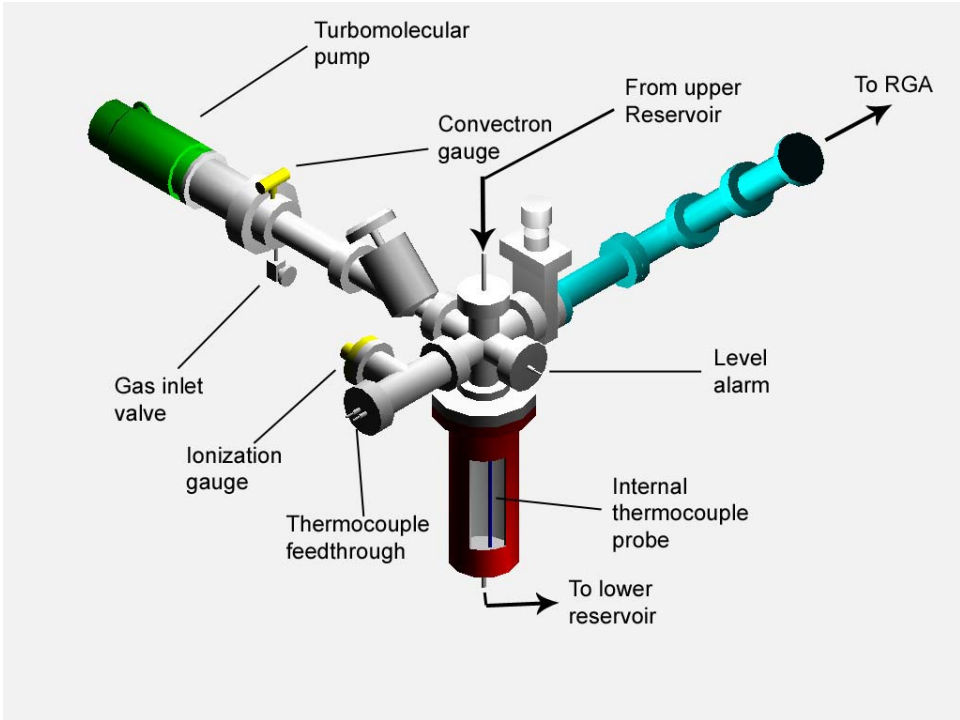
The Thermal Desorption Spectroscopy (TDS)





- Small TMP evacuates the TDS chamber
- RGA shared with the lower chamber
- Heat tapes and PID controller can ramp up TDS temperature at a constant rate

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Experimental conditions

- Irradiation conditions
  - Li stream irradiated with 2 μA,1 keV D beam (multiple ions)
  - Exposed to 5 x 10^{-5} Torr of D_2 neutral gas (Needed for ion source operation)
  - Flow velocity of 60 cm/s
- TDS experiment
  - Lithium mass (~ 400 g) moved into the TDS chamber
  - Heated from 230 to 600 °C in ~ 3 hrs (2 °C/min)
  - Soaked at 600 °C for 1 hour
  - Natural cool down after 1 hour to 230 °C
  - Thermal treatment repeated after cooldown to verify removal

Estimation of gas absorption

- Correlation for wetted-wall cylindrical absorption tower used:

$$\frac{x_H - x_H^*}{x_{H,0} - x_H^*} = 1 - 3\sqrt{\frac{DL}{\pi \delta^2 v_{\max}}}$$

x_H:	mole fraction at end of path
$x_{H,0}$:	mole fraction at the entrance to the system
x_H^*:	interface concentration
D:	diffusion coefficient
L:	length traveled by the film
δ:	film thickness
V_{max}:	maximum velocity

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Estimation of gas absorption

- Values for the parameters during the experiment were:

  $x_{H,0} = 0$ $x_H^* = 0.5$ $D = 1\text{x}10^{-4}$ cm^2/s $L = 10$ cm $\delta = 1$ mm
  $v_{max} = 60$ cm/s

- If all D forms LiD, the mole fractions at the end of the ramp is at most 3.4 %
- Contribution from ion implantation is minimal (2μA for 45 seconds gives $5.6*10^{14}$ ions implanted, and 400g of Li gives $3.5*10^{25}$ atoms)

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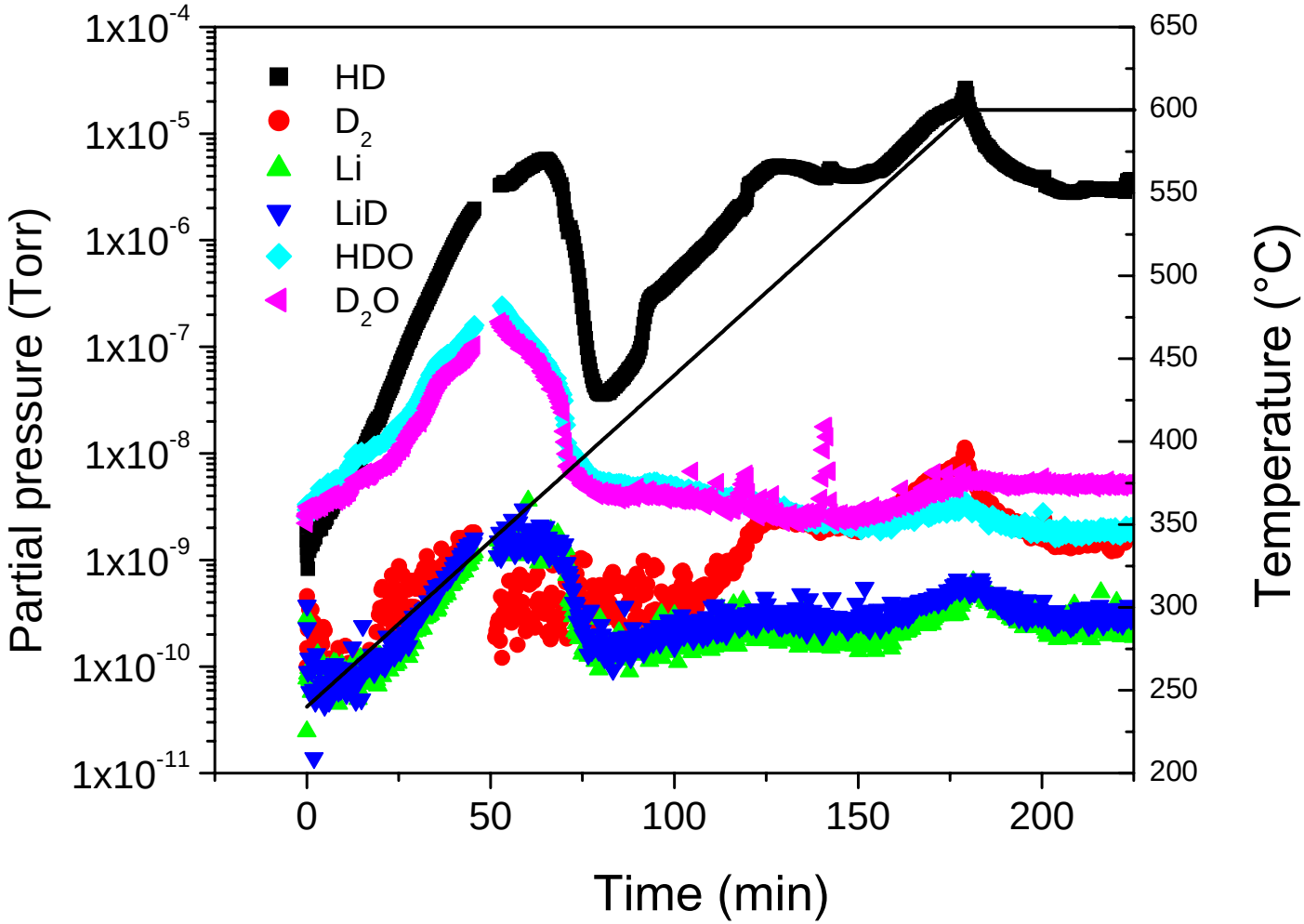
TDS traces, after D exposure



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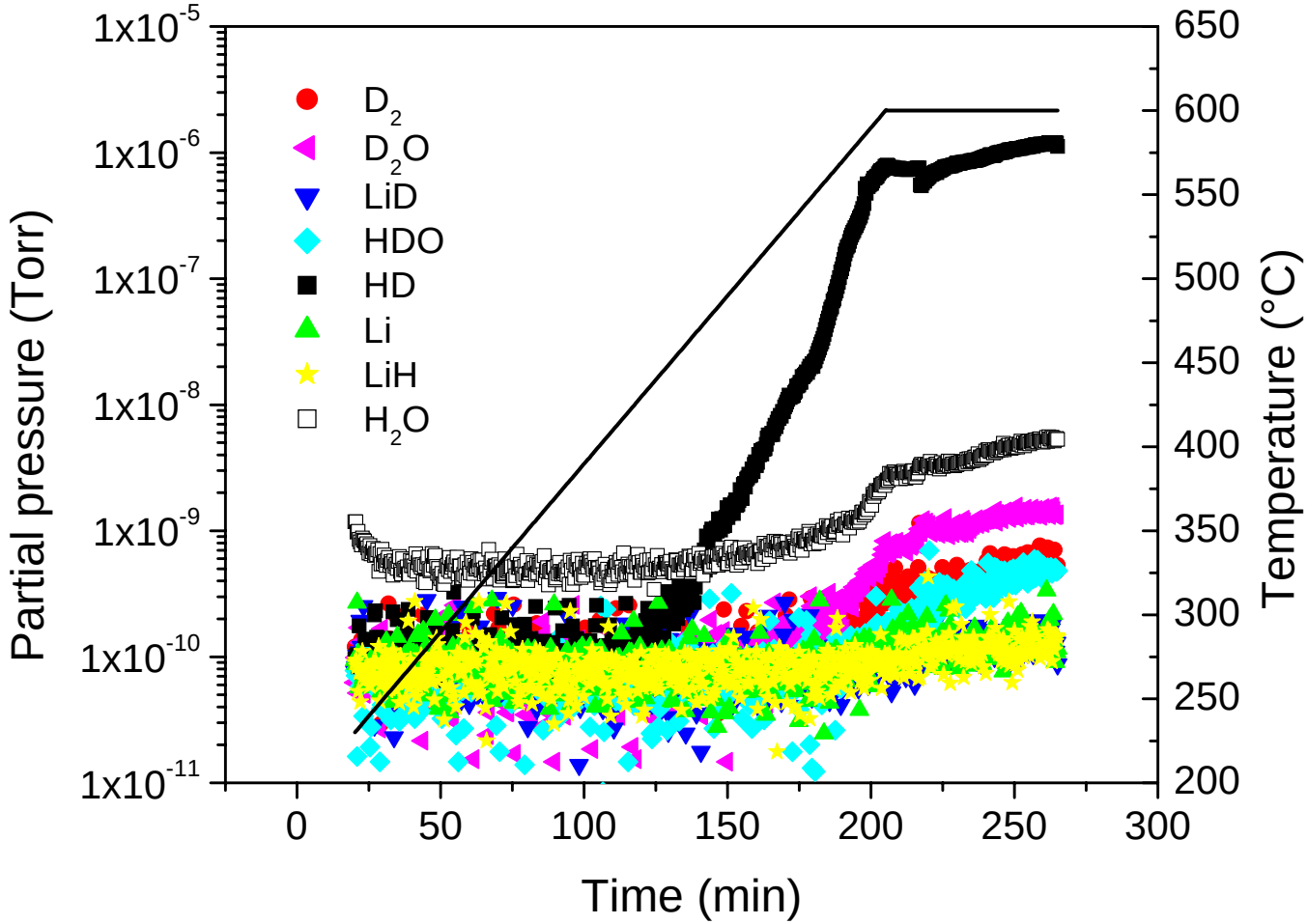
TDS traces, re-heated in chamber



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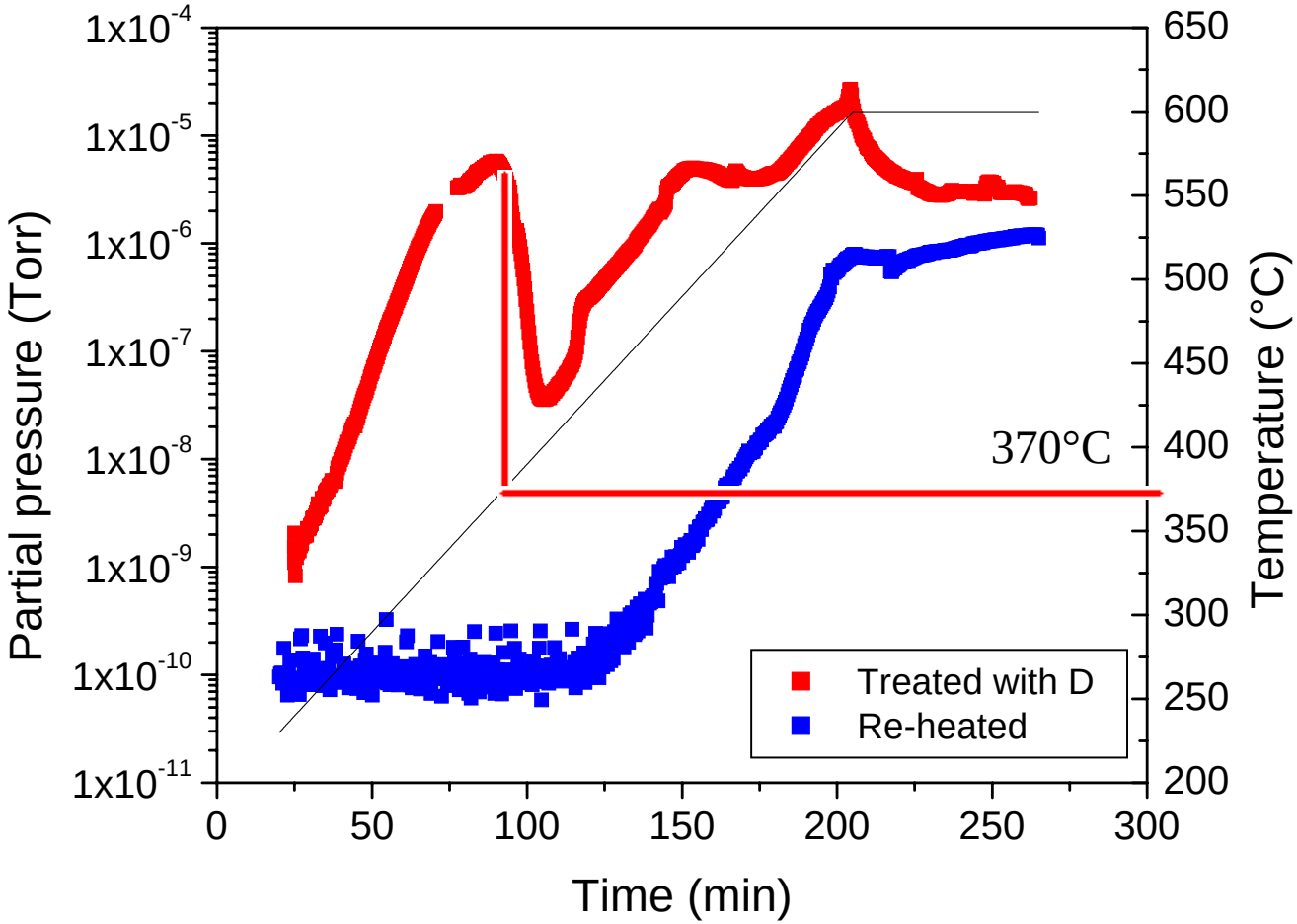
Experimental Data for HD



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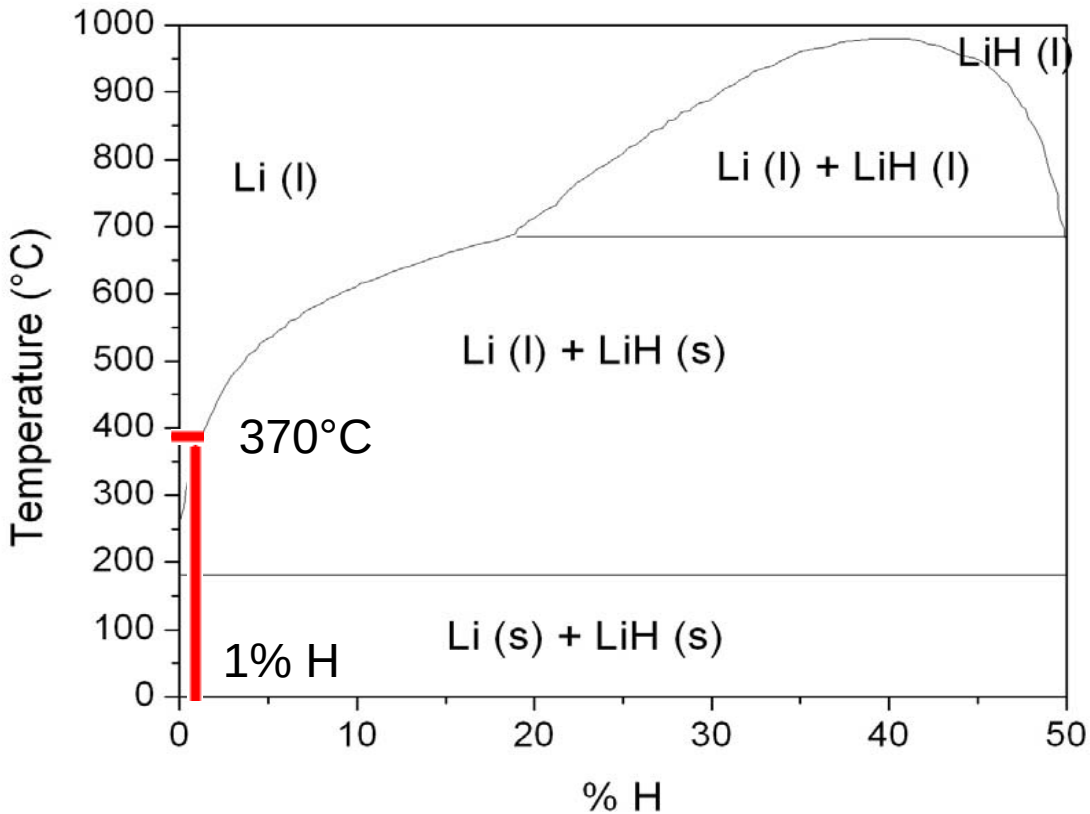
Solubility of H in Li



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Results

- 1% concentration of D was measured compared to 3.4% predicted. Therefore 70% of initial D was promptly released
- This temperature result is consistent with previous work at UCSD*
- Re-heating the batch confirms complete LiD removal from the lithium (no peak at 370°C)

* M.J. Baldwin, R.P. Doerner, S.C. Luckhardt and R.W. Conn "Deuterium retention in liquid lithium," October 2002

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Results (cont.)

- Increase in partial pressure of HD at higher temperatures (~500°C) is most likely caused by desorption of D from colder surfaces adjacent to TDS Chamber
- Lithium signal was lower than expected (condensation?)

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Conclusion

- The TDS system in FLIRE was tested successfully and performs as expected
- Initial estimates indicate at most 3.4% LiD could be formed
- Experiments showed that the maximum deuterium release occurs at 370 °C, corresponding to 1% D content, therefore other 2.4 % did not form LiD
- No Li vapor interference with measurements was observed

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Future work

- Irradiate lithium stream under various conditions (Energy, Ion current, flow velocity, pressure, etc.)
- Quantify the amount of D trapped as LiD in the liquid Li as a function of flow parameters
- Upgrade TDS heating system (clamp furnace)

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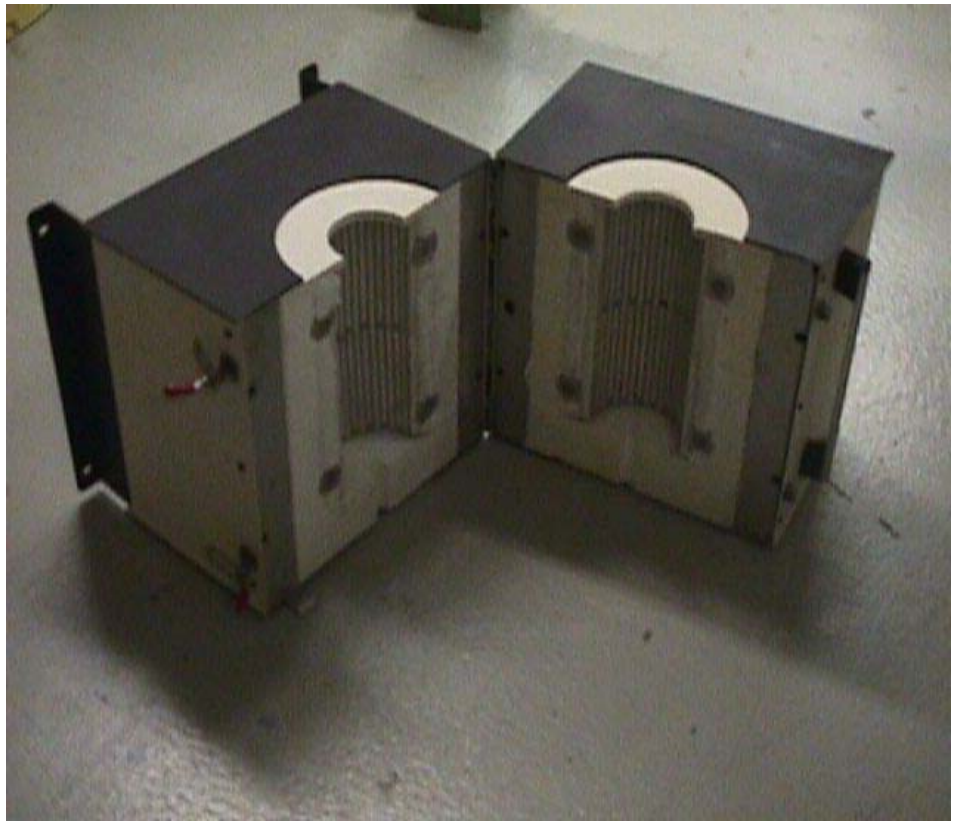
New furnace for TDS

- Manufactured by ThermCraft inc.
- Capable of achieving temperatures up to 1200°C



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