

# Surface chemical/binding reaction of coated Li layer by lithium vapor injectors in LIGHT-1

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# Introduction : LIGHT-1 for Li Vapor Exp.



**LIGHT-1**

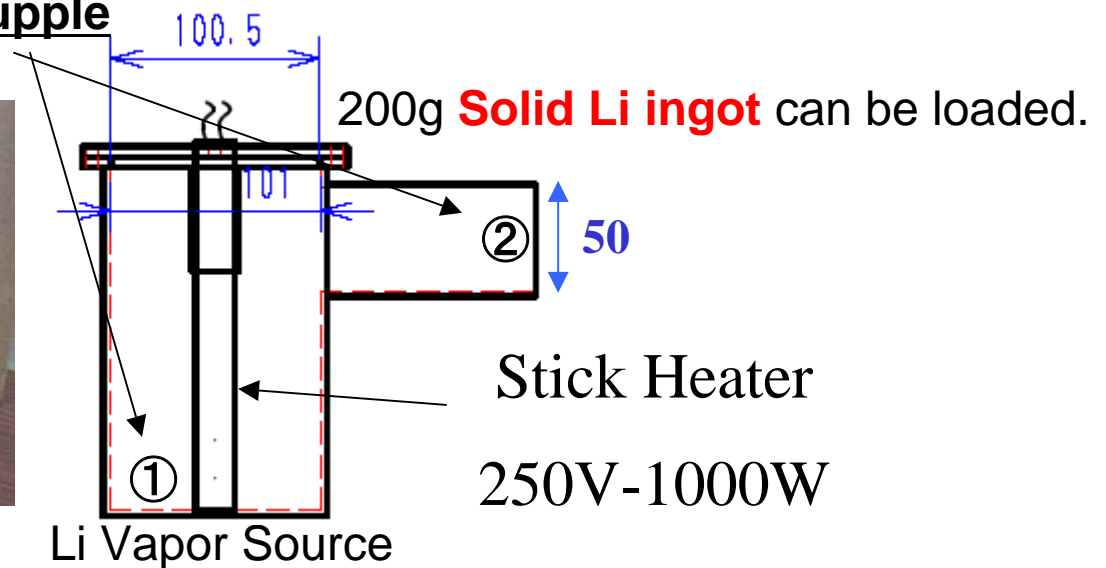
- LIGHT-1 (Lithium Injection Gettering of Hydrogen and its Transport exps.) experiment was started from 2009 for understanding characterizations of Li Vapor process.

- Initial experimental issues in LIGHT-1 are
  - ①Control of Heating temperature ( $650\text{ }^{\circ}\text{C}$ @max)
  - ②Temperature dependence of Li Vapor flux
  - ③Li flux profile along the injected direction

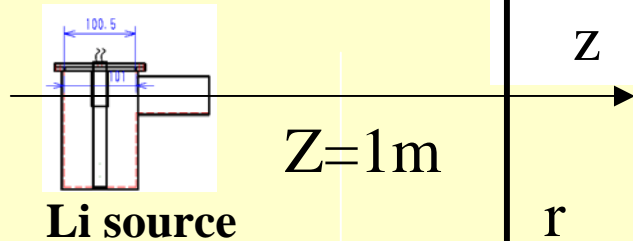


*See, H.Tsuchiya in this WS*

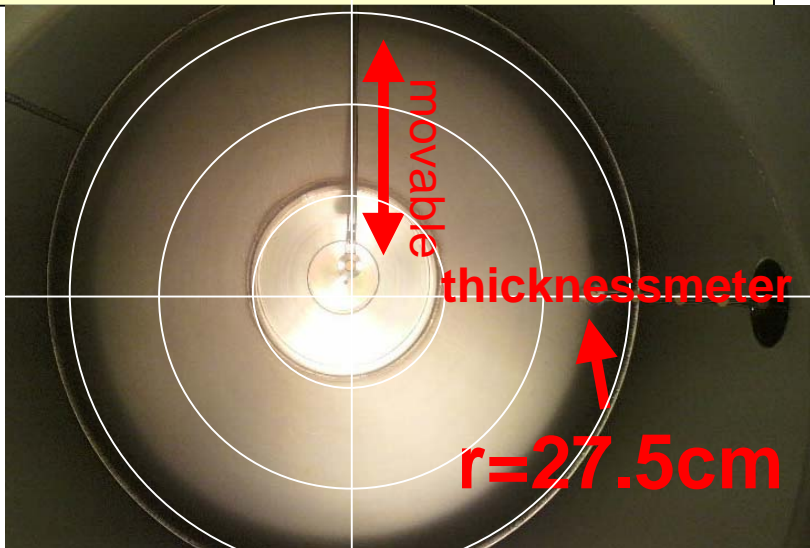
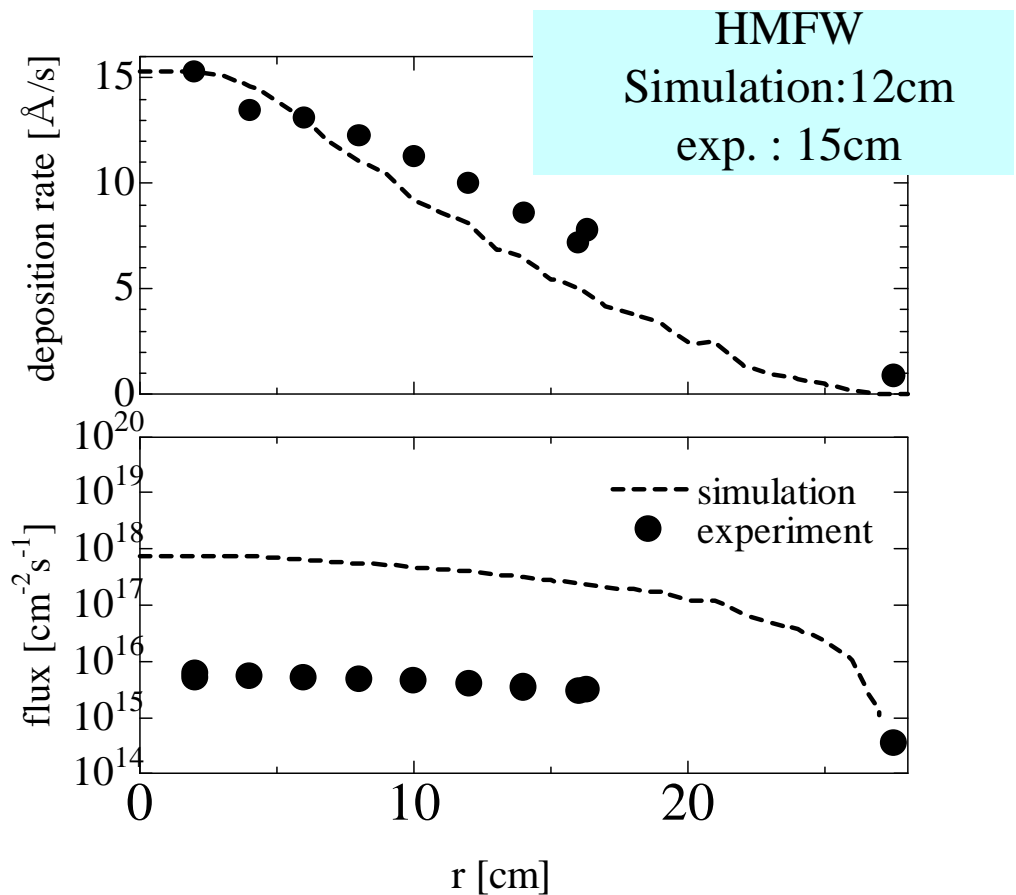
thermocouple



# Radial profile of Li beam by QMB



Li Vapor deposition rate are measured on radial cross-section (r-direction) by movable QMS

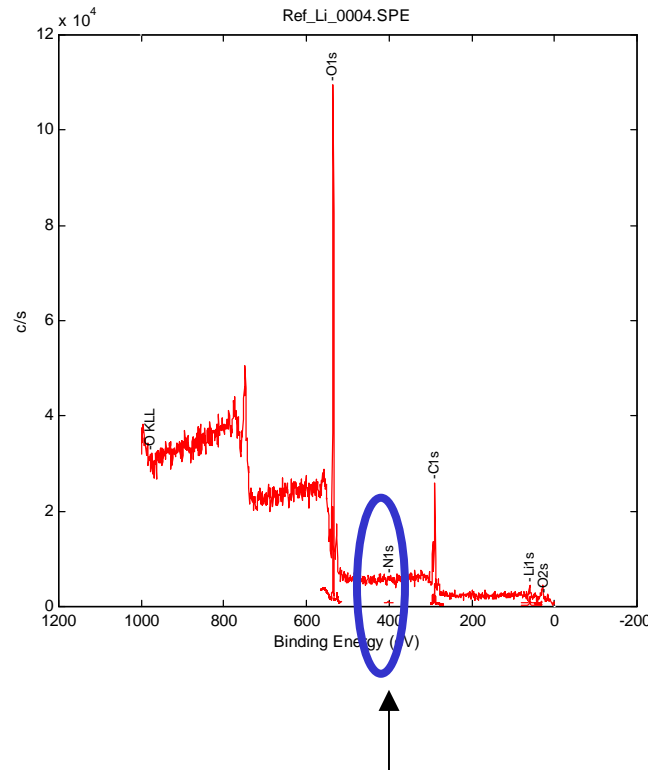


# Motivation

- Using surface analysis by X-ray photoelectron spectroscopy (XPS), depth profile of chemical binding between Li and other impurities are observed.
- From these depth profile for different conditions, operational histories can be estimated.
- For understanding these operational histories, it is presented about initial experimental data with reference data.

# Solid Li Before sputtering

Ref\_Li\_0004.SPE: CompanyName  
1.0947e+005 max 20.02 s  
SUR/Area1/1

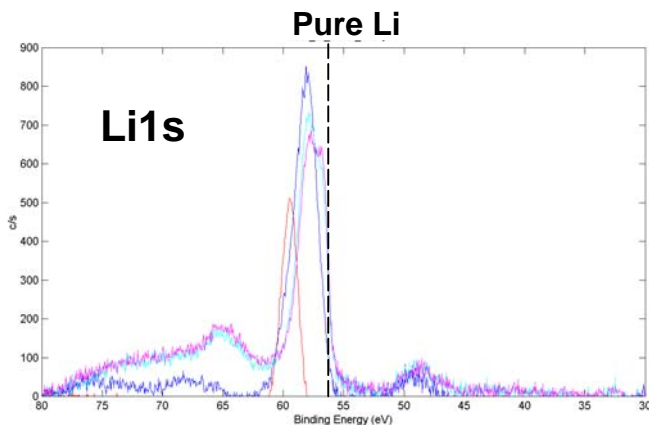
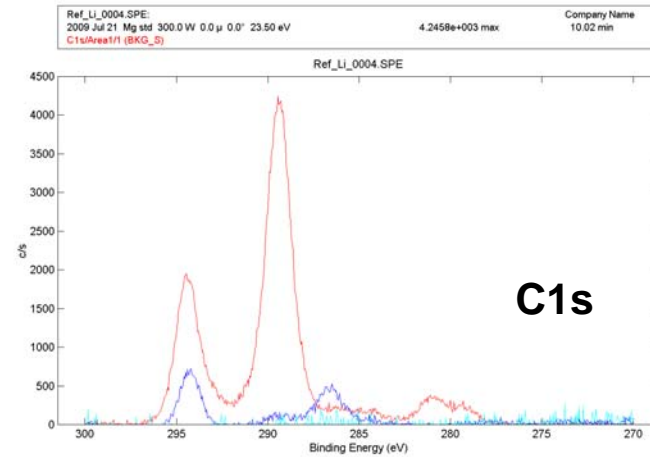
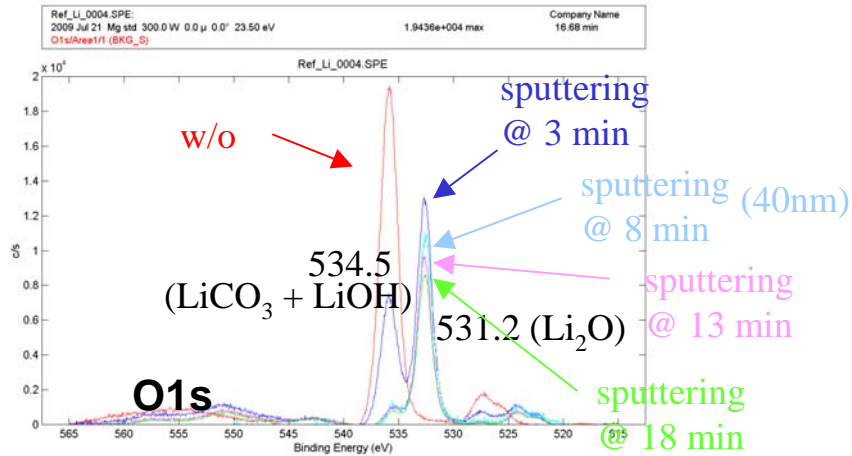


**Stainless steel 316  
material is used as  
base target.**

**(SS316 is the first wall  
material in LHD)**

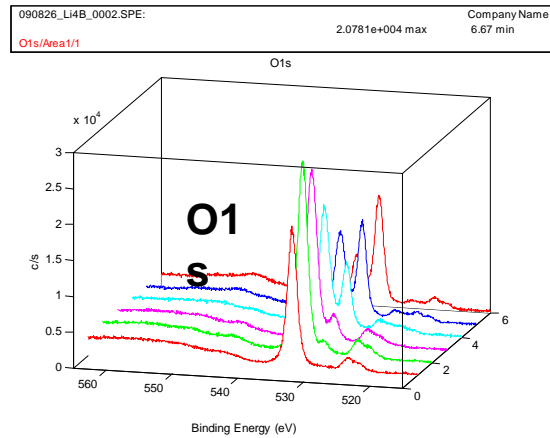
No peak around 400eV.  $\Rightarrow$  it is not observed Li-nitride(Li<sub>3</sub>N).

# Depth profile of solid pure Li

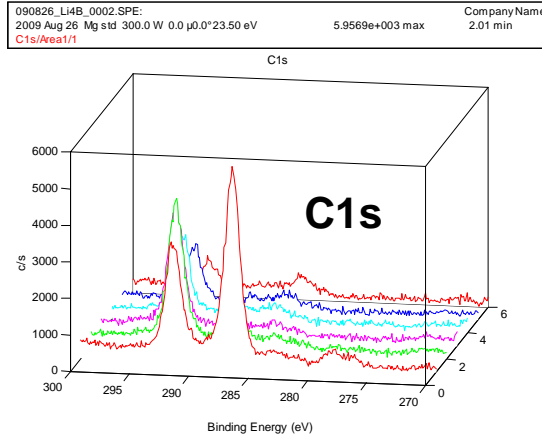


- About depth of 40nm, C1s did not observed  
— > Oxide layer due to air is removed.
- O1s peak shifted from LiCO<sub>3</sub>+LiOH to LiO
- Li1s peak shifted under 40 nm.

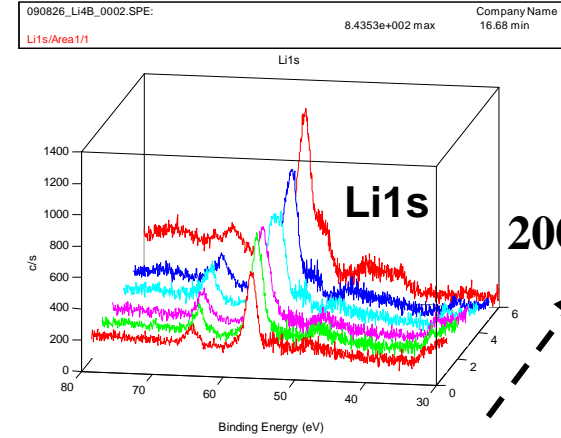
# (1) Vapor test (w/o glow plasma)



At the top surface, LiCO<sub>3</sub>+LiOH(534.5eV) is main peak. A main peak shifted to Li<sub>2</sub>O (531.2eV).

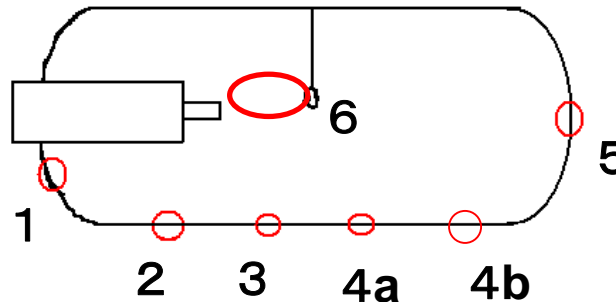


At only the top surface, Cox and CxHx impurities are observed.



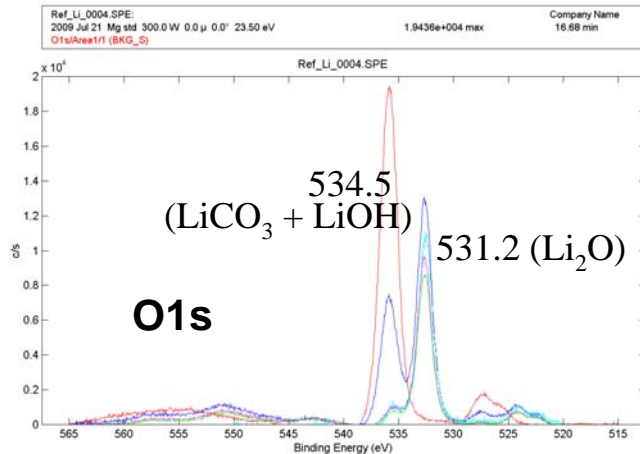
(0nm)

Li intensities are increasing toward deeper region from the top surface.

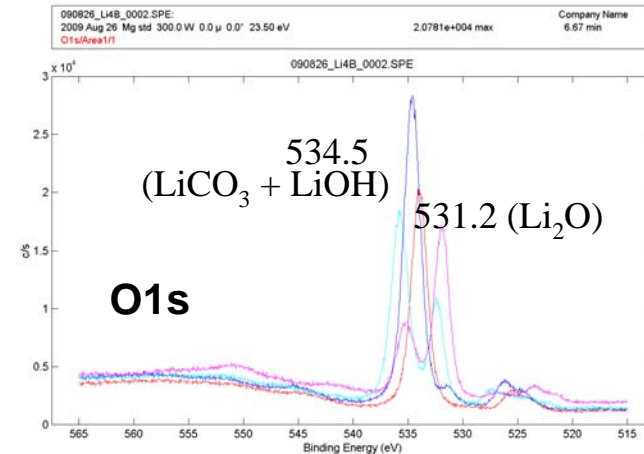


# Comparison of Solid Li and Coated Li on SS316 sample ; O1s

## Solid Li

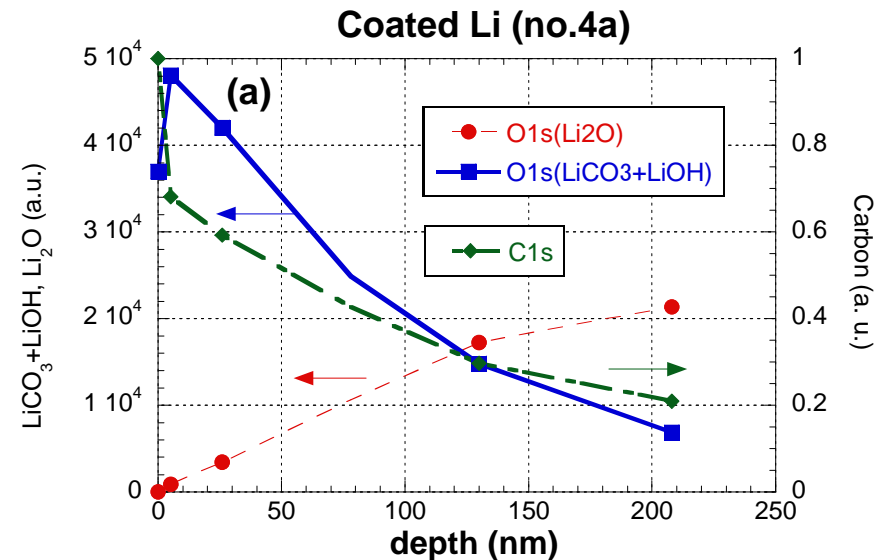
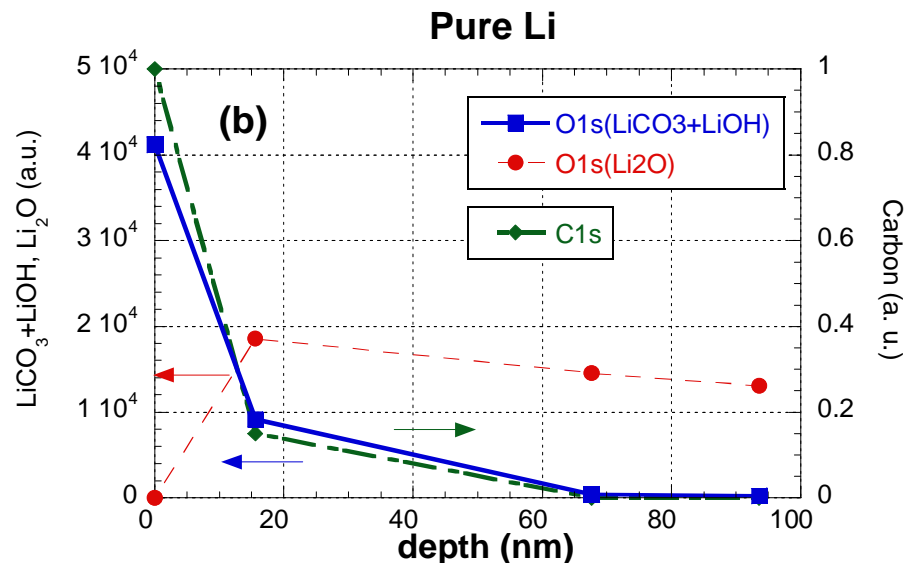


## Coated Li in LIGHT-1 (No.4a)



- Two kinds of peak positions for LiCO<sub>2</sub>+LiOH and Li<sub>2</sub>O is similar. At coated Li, shifted peak positions are not uniform.
- Coated Li have thick oxide layer due to vacuum vent, and thickness of Li<sub>2</sub>O layer is larger than solid pure Li.
- These peak shifts are similar to previous work by J.P.Allain (Ref.JNM(2009))

# Estimation of oxide layer due to exposed to air



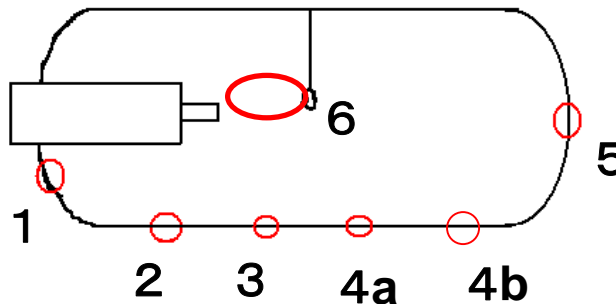
- From comparison of Li1s shift and intensities of C1s, oxide layer due to vacuum vent are estimated.
- For solid pure Li, Li1s peak shifted under 20nm (near the top surface)
- Carbon intensities are depending on the exposed to air.
- On coated Li sample, O1s peak shift is kept until 150nm. => Oxide layer is thick.

Depth profiles of Li<sub>2</sub>O are different on both sample.

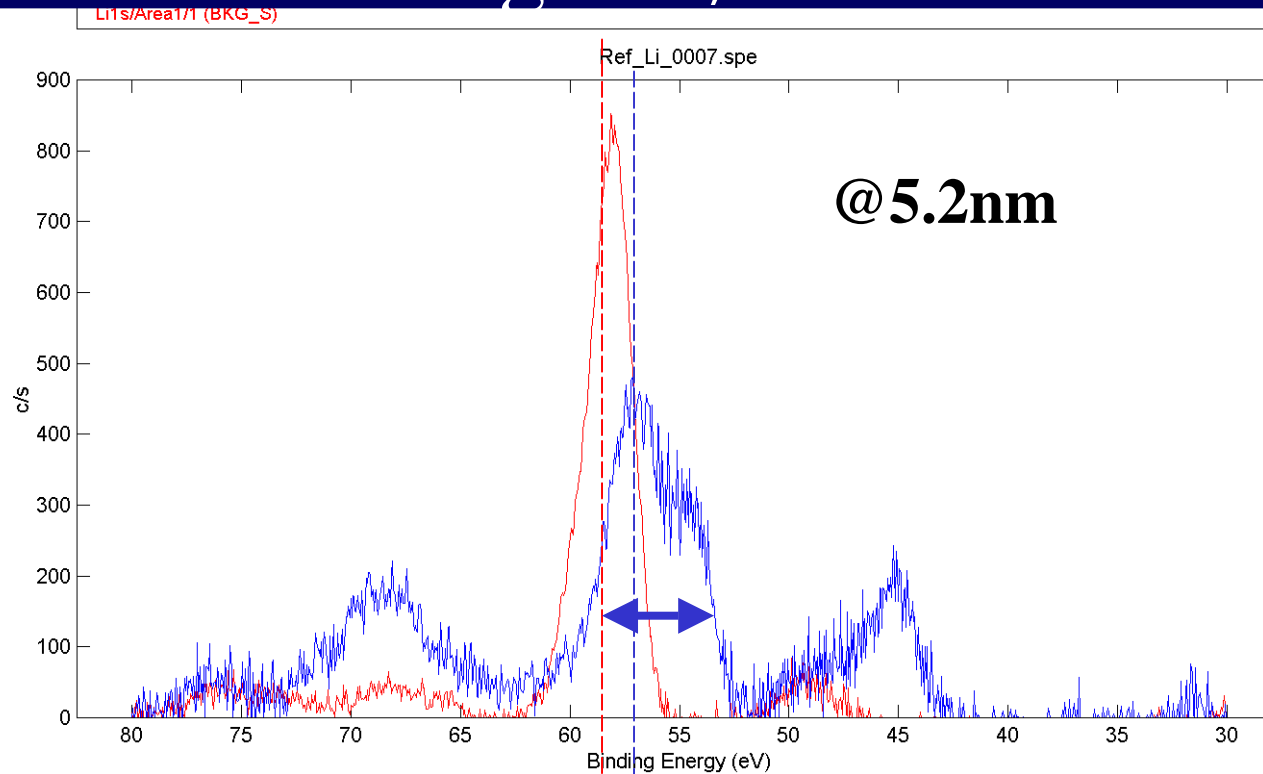
## (2) Li Vapor with glow plasma

### Operational history

- Ar glow plasma for wall conditioning (5h)
- Li vapor during hydrogen glow plasma (2h)
- Ar glow plasma (2h)



# Comparison of Solid pure Li and Coated Li with H2 glow; Li1s

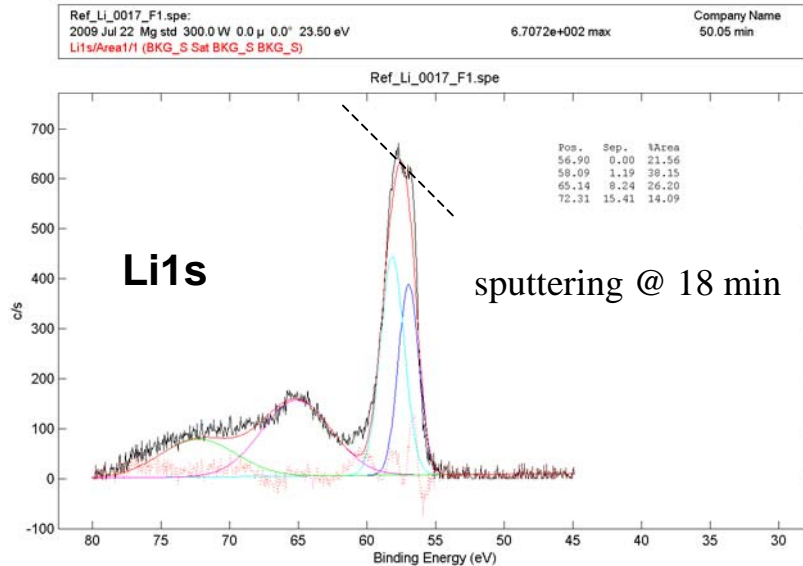


**Pure Li**

**Coated Li**

**At near the top surface, chemical binding between metal and Li was observed. =>eroded metals by glow?**

# Li1s peak (Solid Li)



From data of O1s peaks,  $\text{Li}_2\text{O}$  is increasing toward far distance from the top surface.



At depth of 100nm, Li1s peak shifted to low energy region. This region is low influence of oxygen by vacuum vent

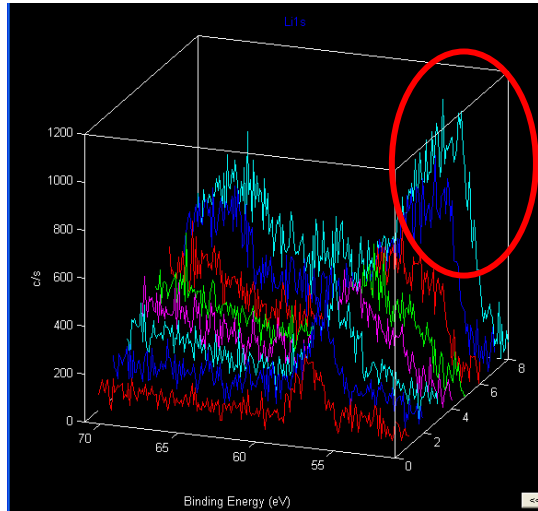
**-Is  $\text{Li}_2\text{O}$  increasing ?**

- It is effect by satellite peak of Fe (Fe3p)
- $\text{Li}_2\text{O}$  peak also closes to iron-oxide ( $\text{Fe}_2\text{O}_3$ ) peak

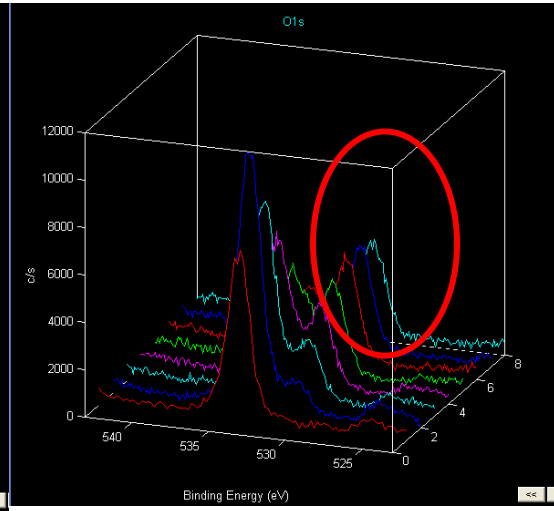
**-Li thickness is estimated using peak positions of  $\text{Li}_2\text{O}$  and Li**

# Li Vapor with glow plasma

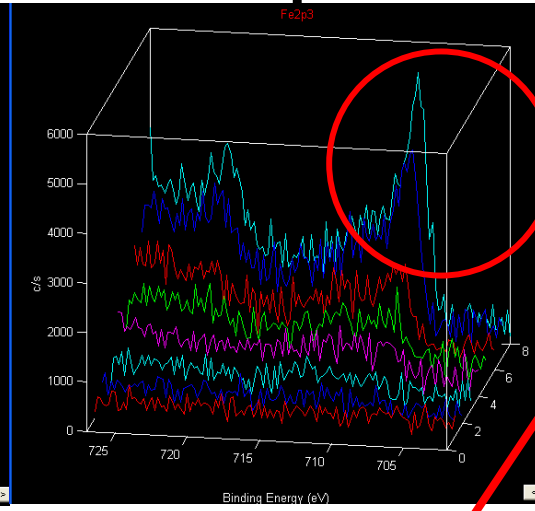
**Li1s**



**O1s**



**Fe2p3**



**21nm**

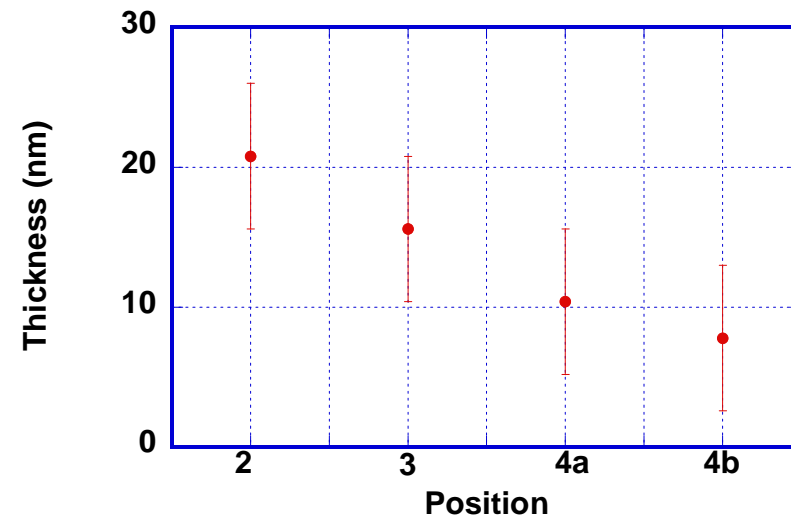
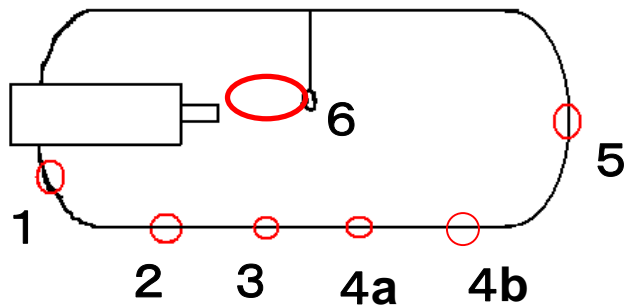
**0 nm**

**Fe intensities are increasing around 15 nm.**

# Li vapor with glow plasma

## Operational history

- *ArGlow for wall conditioning (5h)*
- *Li vapor during hydrogen glow plasma (2h)*
- *Ar glow plasma (2h)*



Thickness of Li is almost uniform on the injected direction

# Summary

- Using surface analysis by X-ray photoelectron spectroscopy(XPS) analysis, depth profile of chemical binding between Li and other impurities are observed.
- Using peak positions of  $\text{Li}_2\text{O}$  and Li, Li thickness is estimated.
- Comparison of Pure solid Li and coated Li in LIGHT-1, these characterizations including vent effect due to exposed to air are shown.
- From Li Vapor with glow plasma experiment, thickness of coated Li are similar at each position.