

Surface analysis of retention and lithium wetting of molybdenum

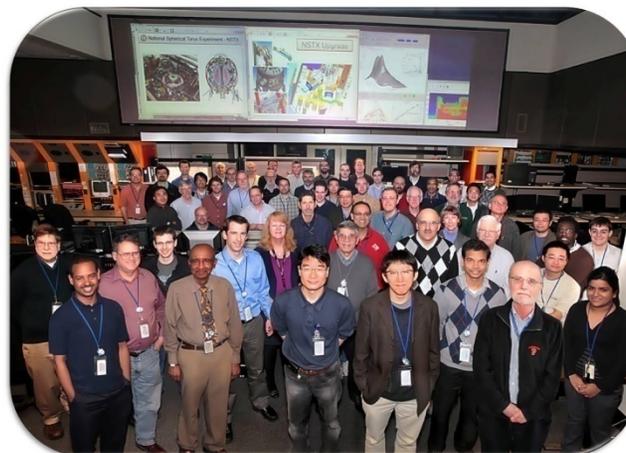
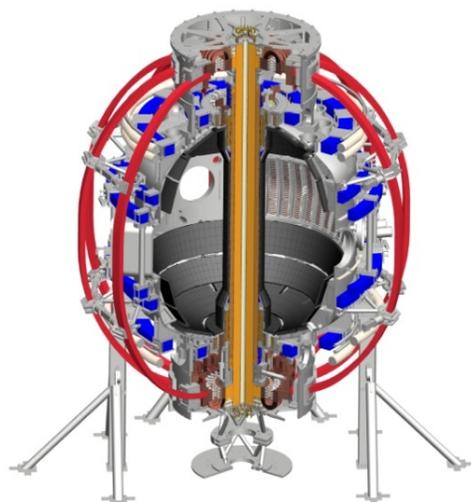
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A.M. Capece, J.P. Roszell, B.E. Koel

and the NSTX Research Team

**55th Annual Meeting of the APS Division of Plasma Physics.
Denver, CO
11-15 November 2013**

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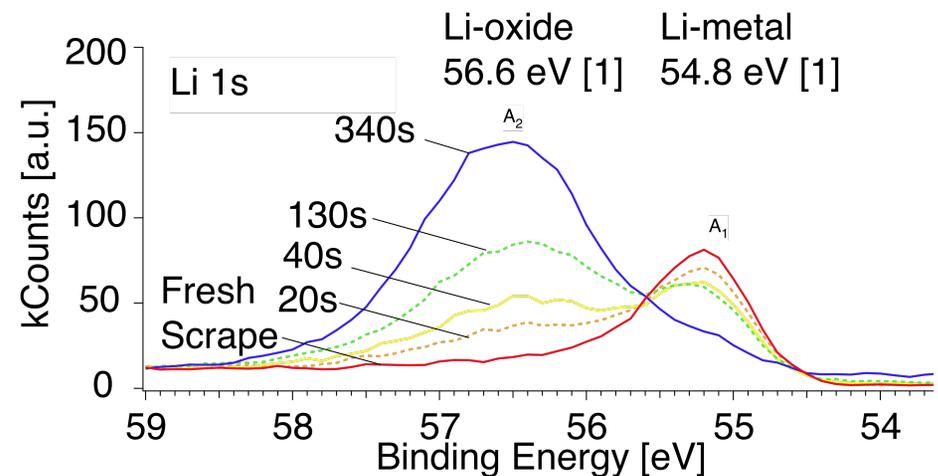
Motivation:

- Lithium conditioning has led to remarkable advances in reducing the divertor heat flux while at the same time increasing the energy confinement time in NSTX and other machines. *[Maingi this session]*.
- Predictive understanding is needed for confident application of liquid metals and coatings in NSTX-U and future machines. *[Abrams, Ruzic this session]*
- This talk presents work in progress to investigate elemental and chemical composition of surface layers in atomistic detail in a controlled lab environment.
- Aim is to elucidate complex bi-directional plasma-surface feedback loops that are very difficult to diagnose in a tokamak environment

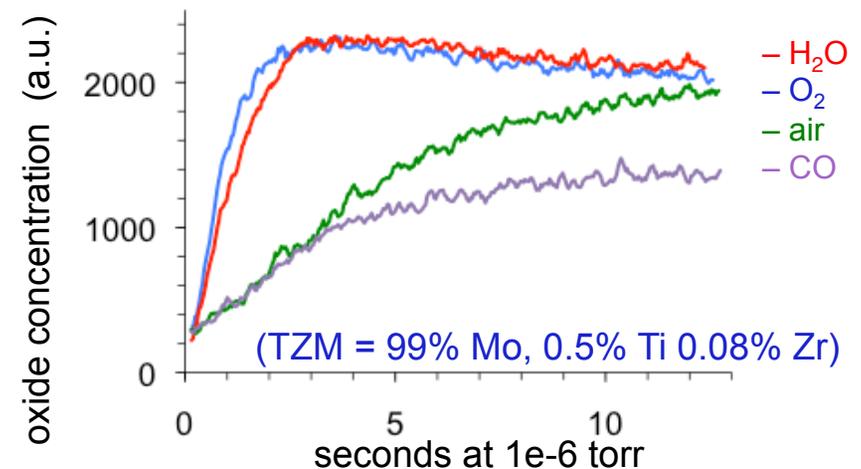
Lithium plasma facing surface is mixed material:

- Measure lithium oxidation with high resolution X-ray photo-electron (XPS) and Auger spectroscopy (AES)
- Find Li oxidized in 10s of seconds from residual H₂O at typical NSTX intershot pressures ~1e-7 torr
- Plasma facing surface after Li evaporation is a mixed material rather than 'lithium coating'
- **Short Li reaction times motivate development of flash Li evaporator**

XPS: O₂ exposure 6.9 e-8 Torr solid Li

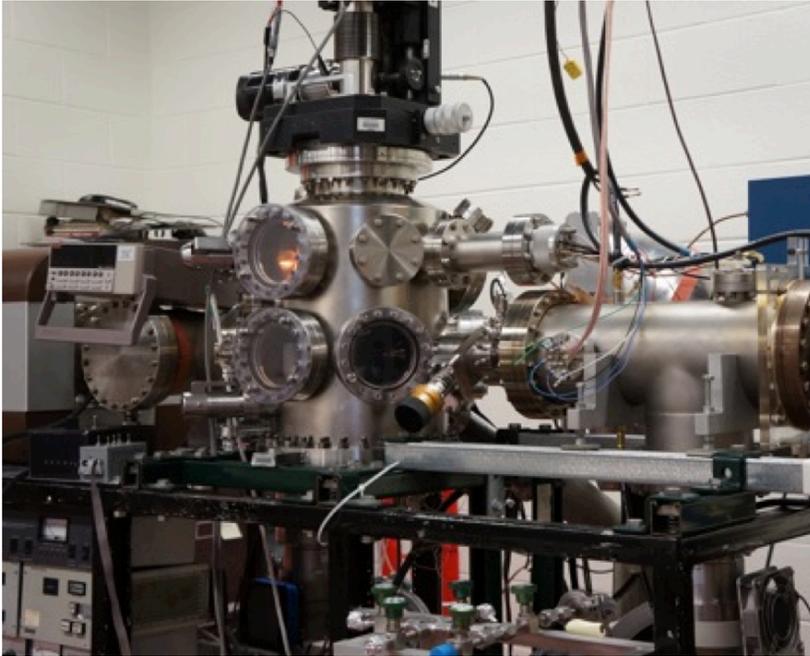


AES of 1 monolayer of Li on TZM Mo

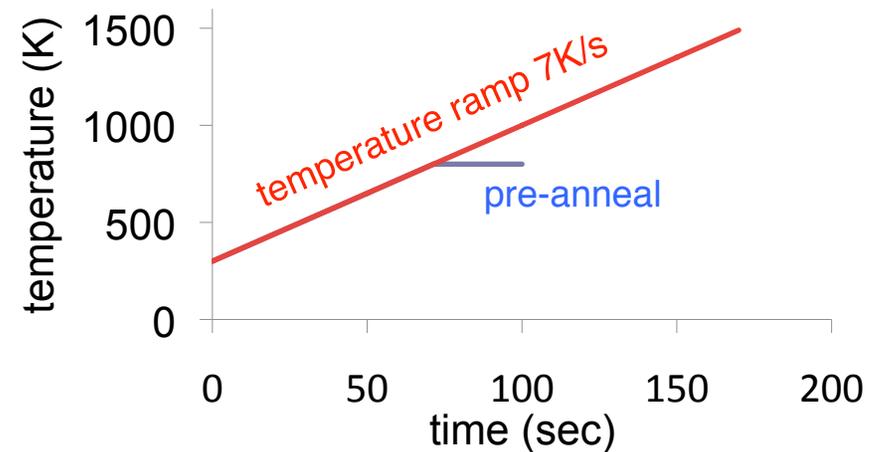


R. Sullenberger, Thesis (2012)
 C.H. Skinner et al., J.Nucl. Mater. 438 (2013) S647
 C.N. Taylor 10:00 Wednesday NI3.00002 "Differentiating...
 A.L. Roquemore 9: 30 AM Friday YP8.00086 "Flash evaporator..."

Thermal desorption analysis of well-defined Mo surface



Thermal desorption temperature is related to chemical binding energy (Temperature Programmed Desorption, TPD)

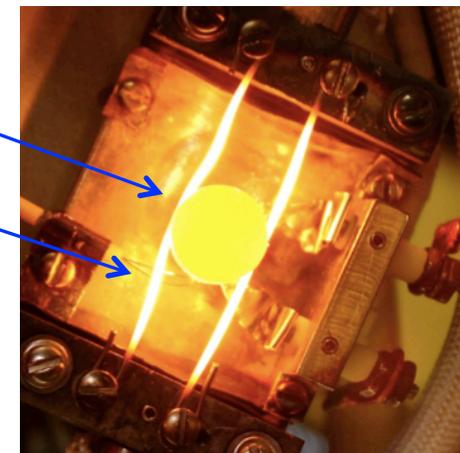


‘Department store’ of surface analysis capabilities:

- Elemental analysis (XPS, AES, ISS, TPD)
- Surface structure (LEED, ALISS, XPD)
- Li film growth in UHV with sub-monolayer control
- Gas dosing for studying effects of background gases on surface growth and chemistry
- Low energy, differentially pumped, mass analyzed ion source for retention experiments

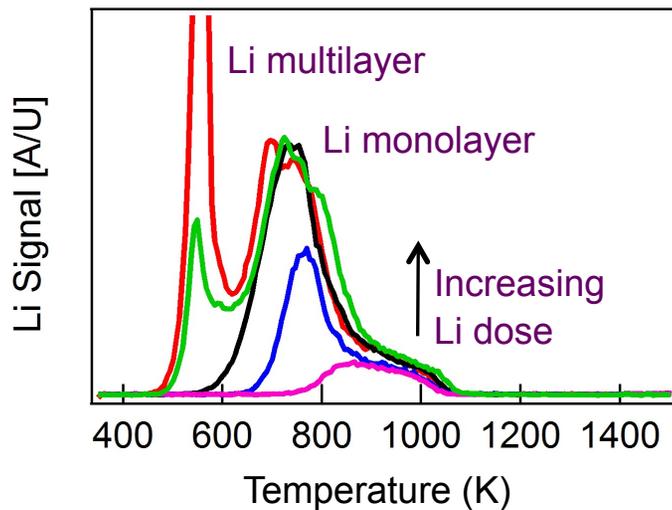
Mo(110) xtal

heater wires



Oxidation of Li film on Mo(110) crystal studied with TPD

- Formation of Li monolayer and multilayer films is observed in desorption spectrum of clean Li
- Desorption analyzed with QMS



- Lithium oxide formation shifts desorption peaks
- Individual peaks isolated by annealing away lower temperature peaks before TPD

pre-annealed to 1050 K

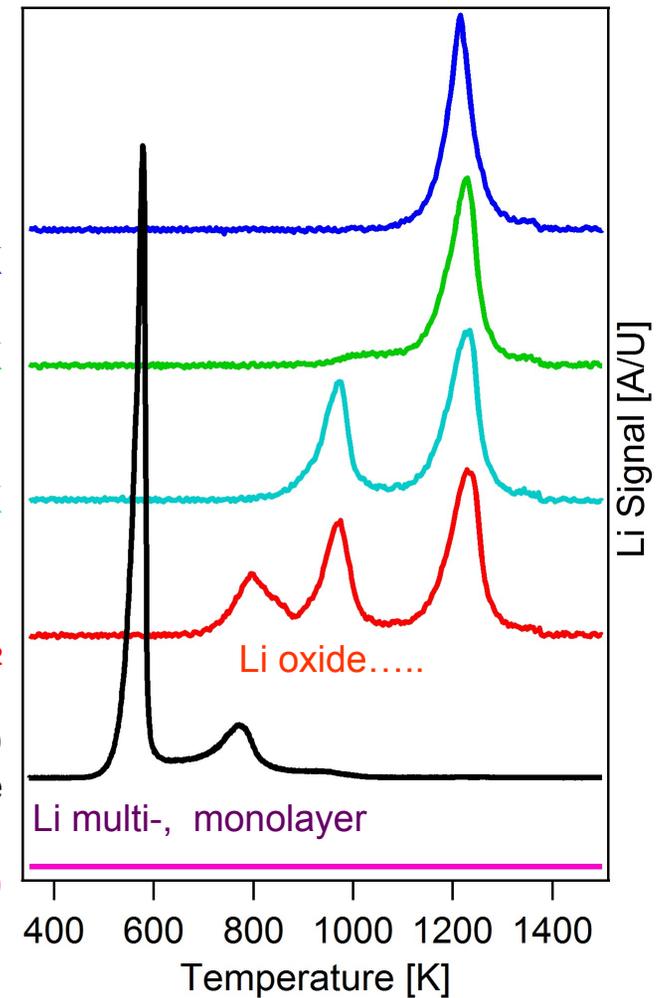
pre-annealed to 950 K

pre-annealed to 800 K

3 ML Li deposited on Mo(110); oxidized with 5 Langmuir O₂

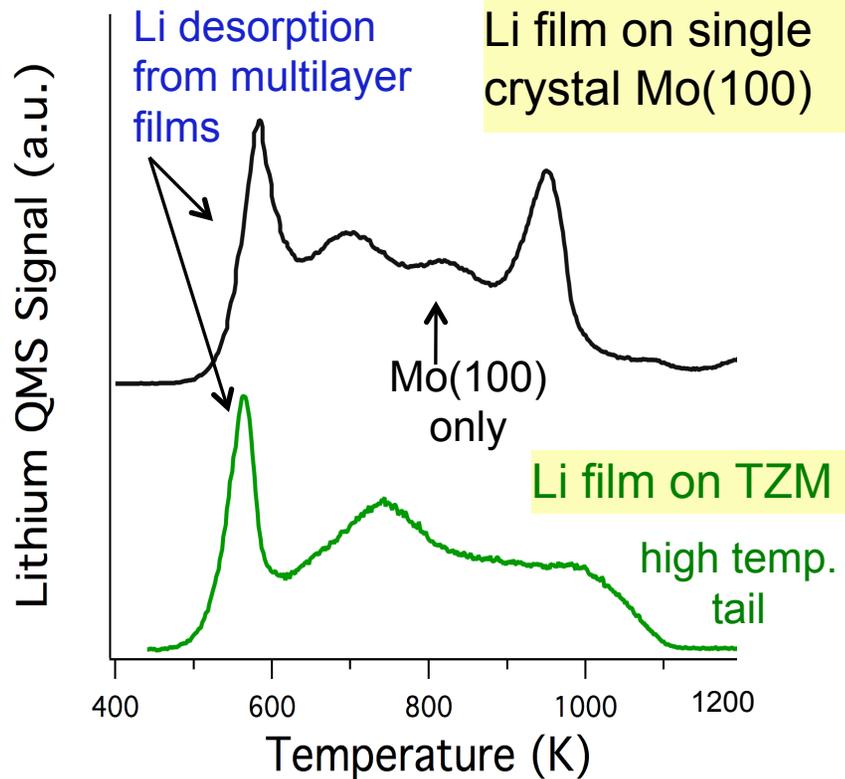
3 ML Li deposited on Mo(110) surface, no O₂ dose

Clean Mo(110)

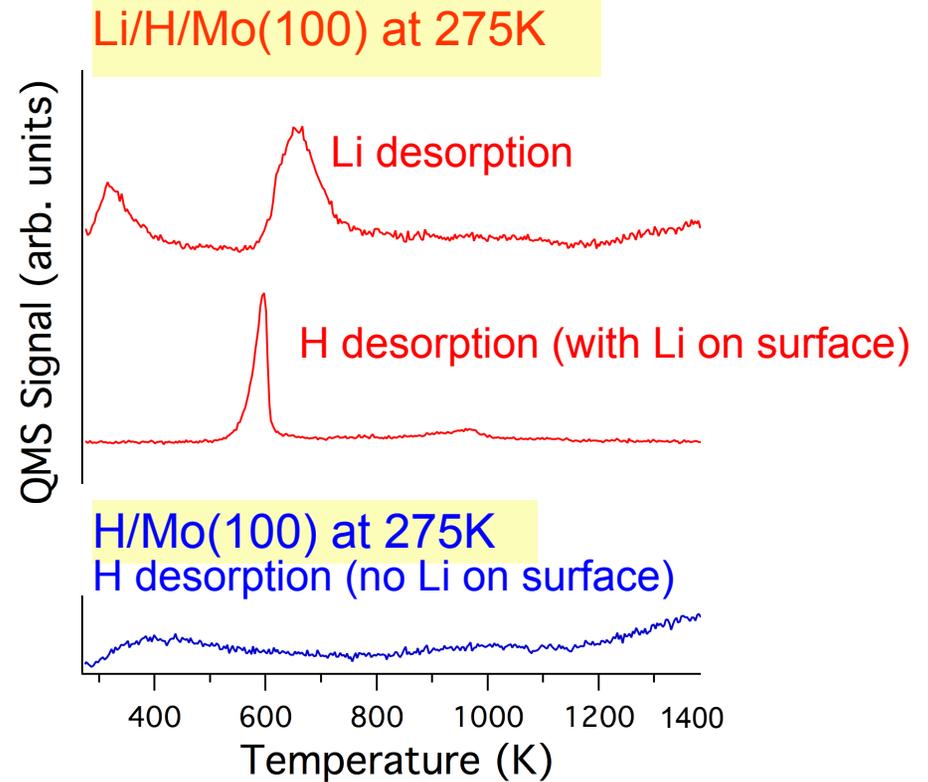


Wed 9:30 AM: Roszell, NP8.00011 Deuterium retention and hydride formation by

Carbon contamination and/or grain boundaries affect surface



- Tail on TZM β_1 peak reveals other chemical bonding or enhanced diffusion of Li into subsurface region via grain boundaries.

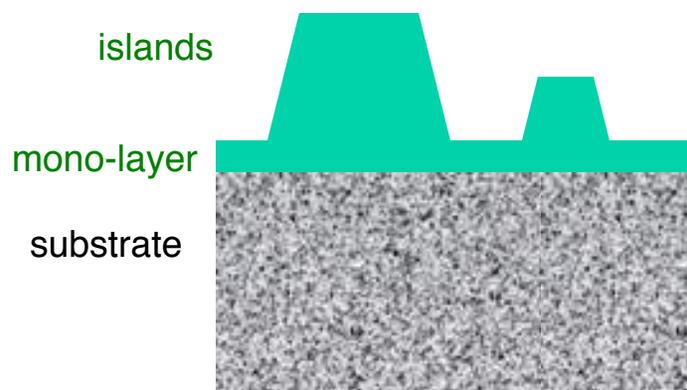


- H pre-adsorbed onto Mo(100) at 275 K
- Without Li, H desorbs near room temperature.
- With 2 ML Li deposition – Li and H react to form LiH, which decomposes near 600 K.
- H retained only when Li is present

Wed 9:30 AM: Capece, NP8.00010 The Influence of ...

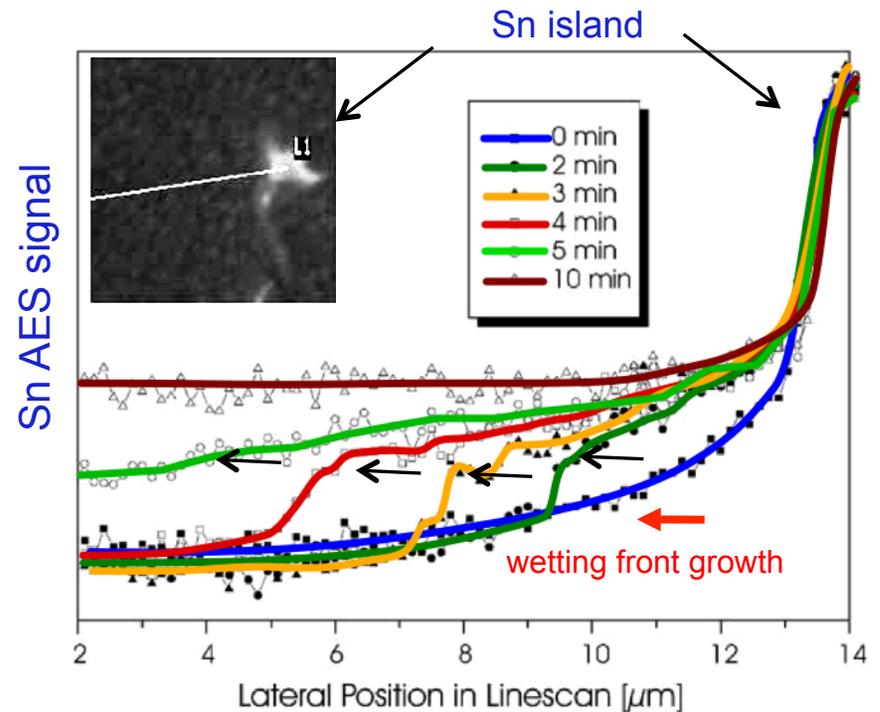
Chemical bonds will affect Li wetting

- Strong Li-Mo and weak Li-Li bonding suggests lithium wetting of Mo proceeds by layer + island growth
- Thermally activated surface diffusion - influenced by impurities and surface roughness
- Experiments with Li on SS or TZM underway with scanning Auger microprobe at PPPL



Wetting of Sn on Al revealed by scanning Auger microscopy:

Klein, et al., Vacuum 80 (2005) 74-80.



- Sputter deposition of Sn, then etching of wetting layer, then re-wetting from Sn islands.
- Auger linescan shows Sn re-wetting on Al surface @ room temp. (< Sn melt temp of 231°C)

Summary:

We are investigating the elemental and chemical composition of PFC relevant surface films in a controlled lab environment

Aim to provide predictive understanding for application of liquid metal PFCs and coatings in NSTX-U and future machines

1. Li plasma facing surface in NSTX was Li-oxide
2. Thermal desorption of Li monolayer, multilayer and oxide identified
3. Formation of LiH studied
4. Li wetting experiments in progress

Posters:

Wed 9:30 AM: Capece, NP8.00010 The Influence of Temperature and Oxygen ...

Wed 9:30 AM: Roszell, NP8.00011 Deuterium retention and hydride formation by

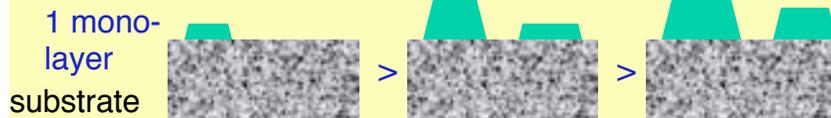
Back up slides

Lithium wetting:

'Atomistic' approach, complementary to 'thermodynamic' approach via contact angle.

Three modes of film growth:

- a. Island growth (Volmer-Weber)
(adatom-adatom interactions are stronger than those of the adatom with the surface)



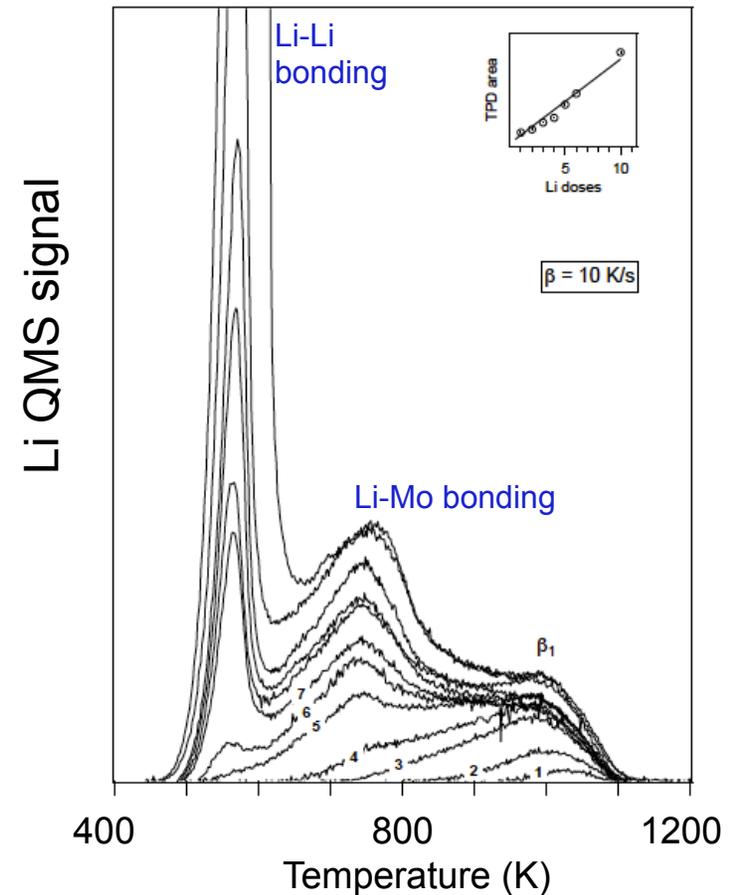
- b. Layer-by-layer growth (Frank-van der Merwe)
(adatoms attach preferentially to surface sites resulting in atomically smooth, fully formed layers)



- c. Layer + island growth (Stranski-Krastanov)
(transition from the layer-by-layer to island-based growth)



TPD of Li on TZM molybdenum alloy, reveals strong Li-Mo bonds and weaker Li-Li bonds



Stranski-Krastanov wetting of Sn on Al

Klein, E., et al., Vacuum 80 (2005) 74-80.

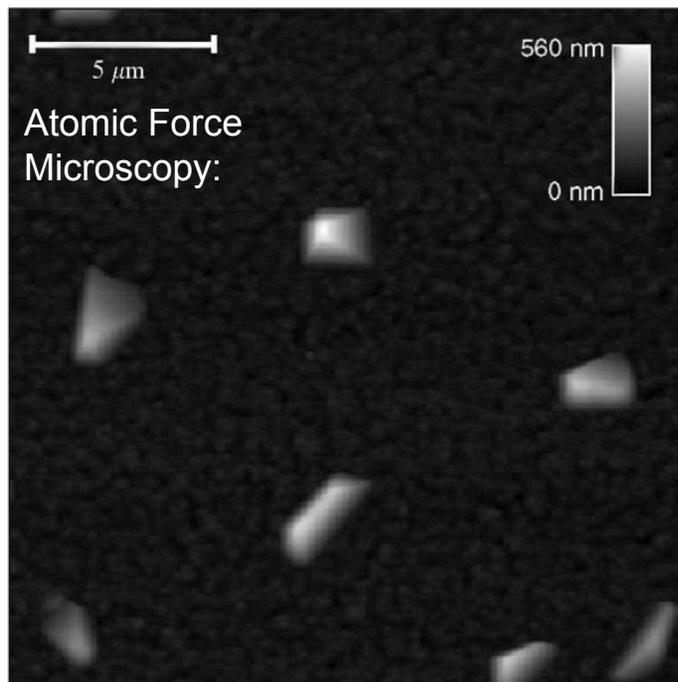
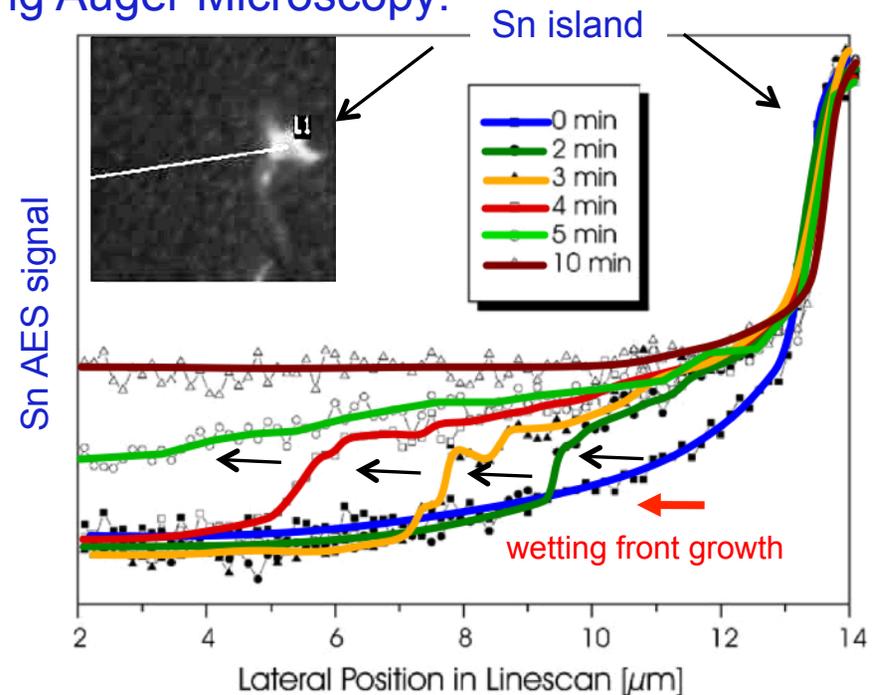


Fig. 1. AFM topograph of the surface morphology of a typical Al-Sn bilayer system: base layer: 400 nm Al; top layer: nominally 10 nm Sn; deposition temperature: 453 K (180 °C). Three dimensional Sn islands with well-defined crystallographic surfaces (bright regions) are located on top of the Al base layer.

Scanning Auger Microscopy:



- ‘Solid’ wetting from thermally activated diffusion - influenced by impurities and surface roughness
 - ‘Atomistic’ approach, complementary to ‘thermodynamic’ approach via contact angle.
 - Similar experiments with Li on SS or TZM in progress at PPPL
- Sputter deposition of Sn, then etching of wetting layer, then re-wetting from Sn islands.
 - Auger linescan shows Sn re-wetting on Al surface @ room temp. (< Sn melt temp of 231 °C)