SAND2014-4354P

Slides for ISEEM

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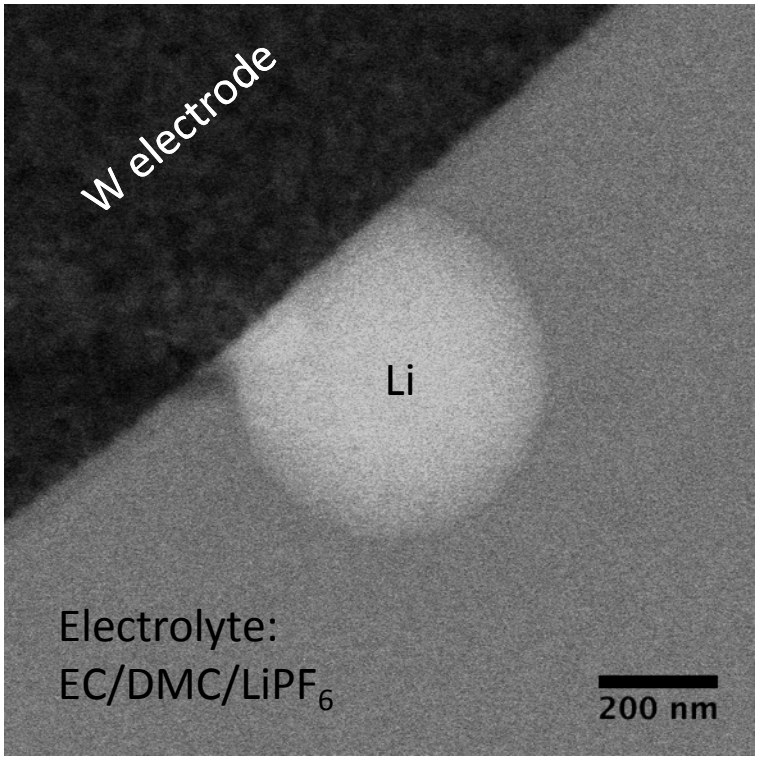
Images show Li electrodeposition in Sandia TEM Liquid Cell.

Beam effects in aprotic electrolyte:

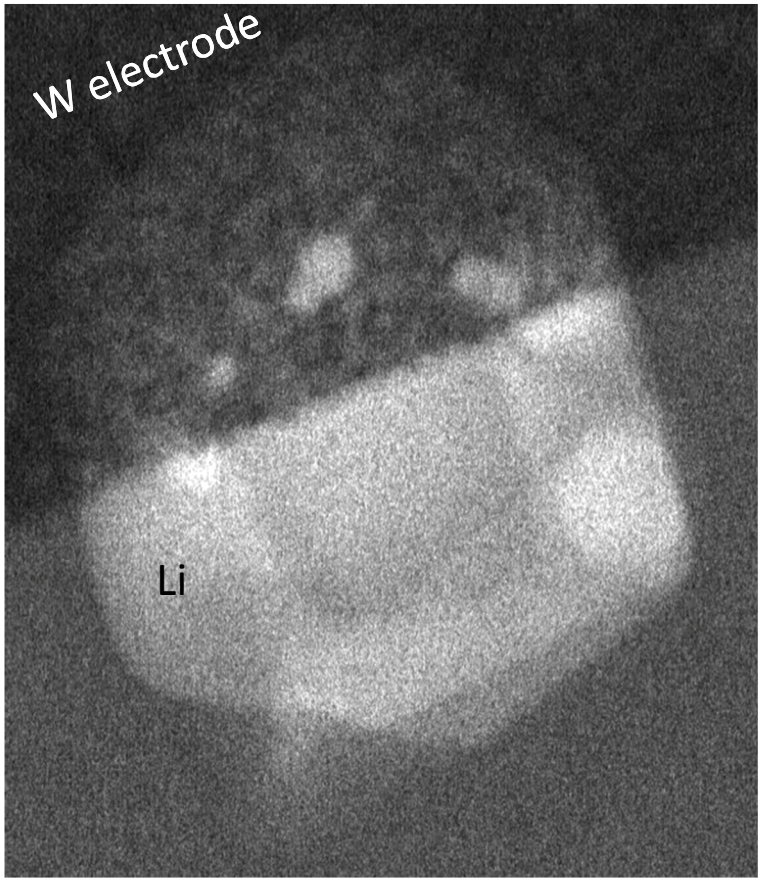
Dose low enough that <u>little noticeable degradation occurs in liquid electrolyte</u>.
However, chemistry induced by beam affects surface and deposition morphology.
Also, prolonged e-beam exposure induces significant (100 nm) surface film on Li.

Deposited with <u>beam on</u>:
Circular (spherical?) grain
Dose < 200 e^-/nm^2



(BF STEM)

Deposited with <u>beam off</u>:
Faceted grain



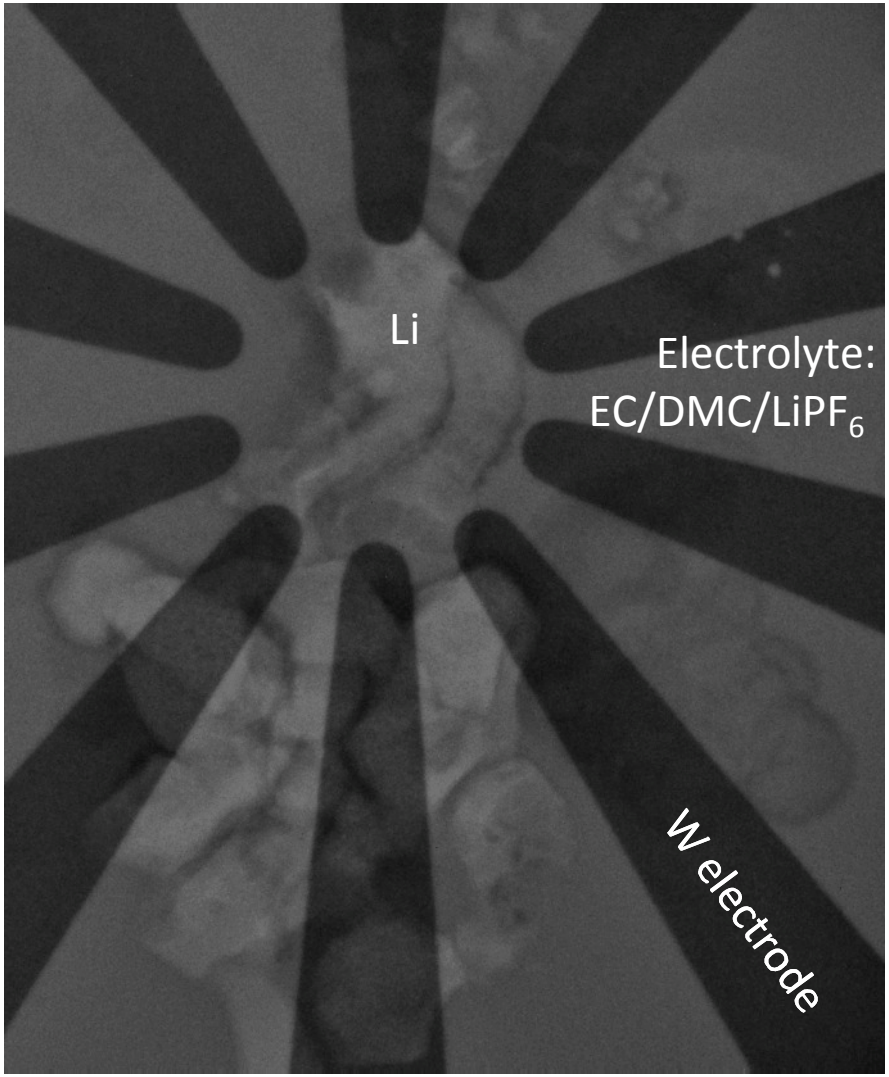
(BF STEM)

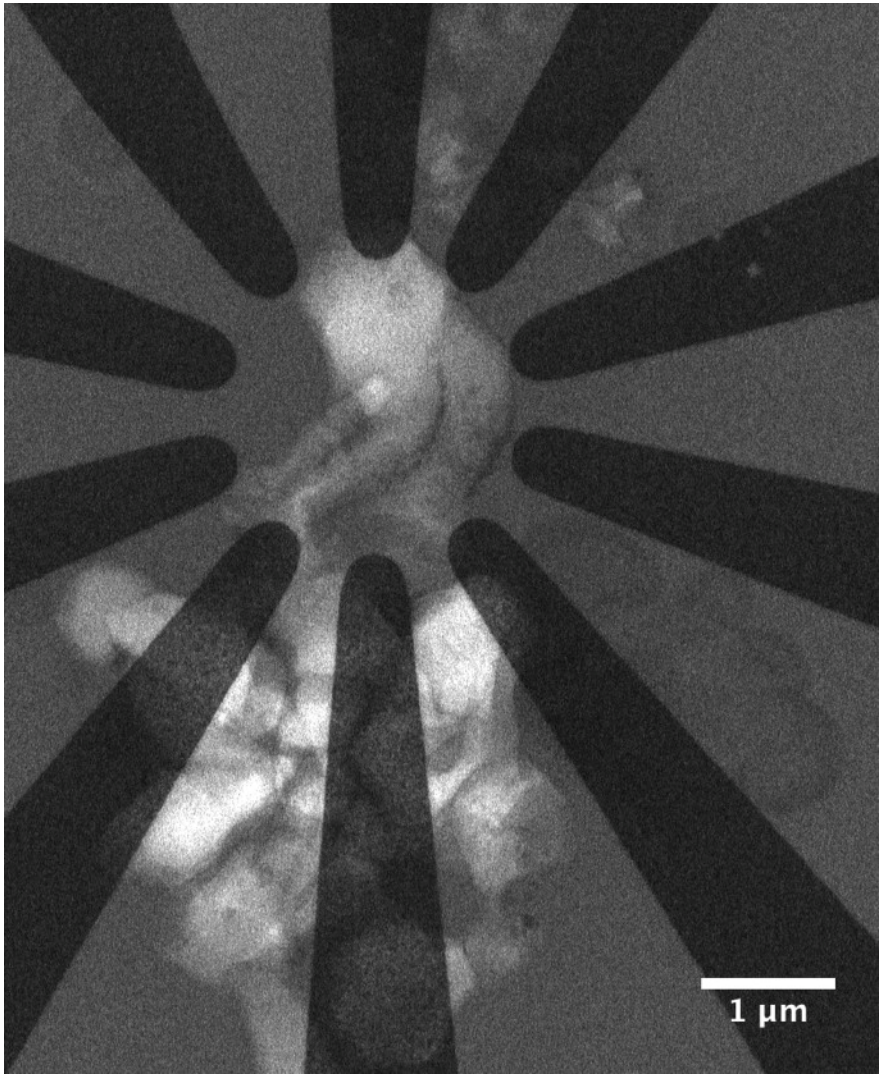
STEM gives higher contrast for low-density Li:

BF TEM: 145 pA beam, 5 sec exposure
7.8 nm/pixel
40 e^-/nm^2 but much outside image

vs.

BF STEM: 11 pA beam, 5 sec exposure
8.3 nm/pixel
4.8 e^-/nm^2 confined to image





In situ Electrochemical Electron Microscopy (ISEEM) 2014 Workshop

Name and Research Group: **Andrew Leenheer**, Center for Integrated Nanotechnologies. Research group includes Tom Harris, Katie Jungjohann, John Sullivan, and Kevin Zavadil.

Institution: Sandia National Laboratories

Microscope(s) used: FEI Tecnai F30 at 300 kV, usually STEM mode

In situ cell(s) used: CINT TEM Liquid Cell Discovery Platform

Potentiostat(s) used for ISEEM:
Solartron Modulab + femtoammeter card, chosen for good accuracy during low-current control

Systems studied: Volatile liquid electrolytes *in-operando*:
Li electrodeposition
Cu electrodeposition
Li alloying

Research questions you use ISEEM to address:
Li-ion battery degradation (Li dendrites, SEI dynamics)

Technical difficulties commonly encountered:

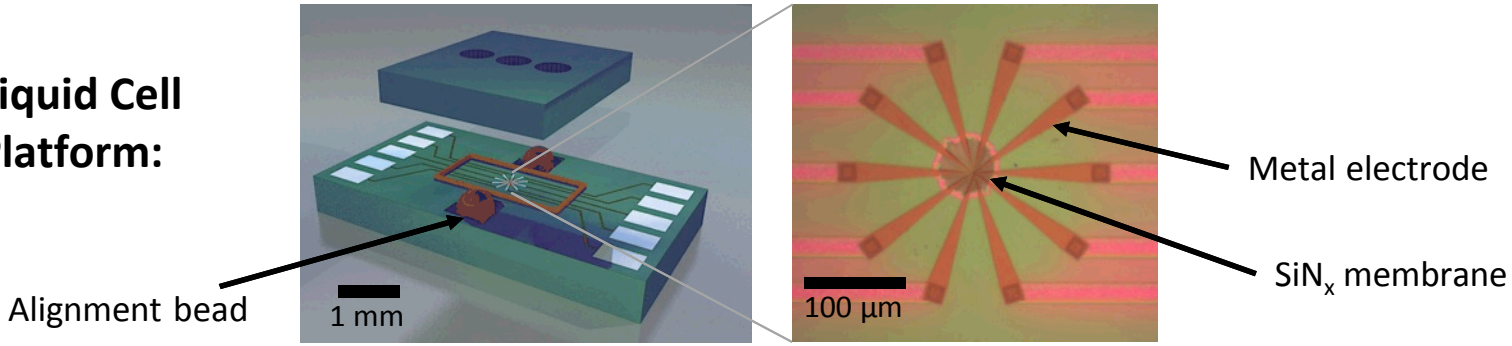
Water in aprotic electrolyte.
Current/voltage transients upon connecting potentiostat cables.

ISEEM publications from your group (0-3):

ISEEM publications from elsewhere you most refer to:

Information is non-confidential and will be physically distributed at workshop.

CINT TEM Liquid Cell Discovery Platform:



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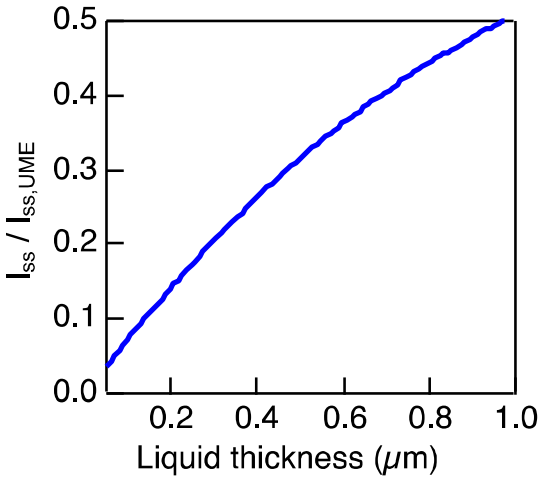
Consider **contaminants in liquid electrolyte** when performing quantitative electrochemistry.

Steady-state diffusive current at ultramicroelectrode (UME):

$$i_{ss,UME} = 4nFc^*Dr \sim 0.2 \text{ nA}/\mu\text{m radius/mM}$$

A 2D confined TEM liquid cell electrode can act much like a UME which is very sensitive to low-concentration contaminants. Calculation at right shows steady-state diffusive current at a 1 µm² electrode with a reservoir 500 µm away.



Example: Dissolved oxygen at 0.3 mM reduced at a 1-µm radius electrode contributes ~100 pA current.