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Nanoscale Characterization in a Controlled Liquid Environment

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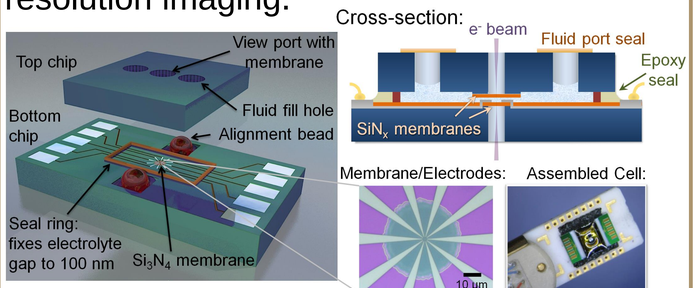
Problem: Understanding Dynamics and Nanoscale Processes in a Liquid

- Visualization of nanoscale processes can lead to mechanism discovery for liquid-solid systems; such as nanomaterial growth, assembly, corrosion and electrochemical cycling.
- Energy storage in batteries can involve detrimental, irreversible electrochemical processes that are inherently nanoscale.

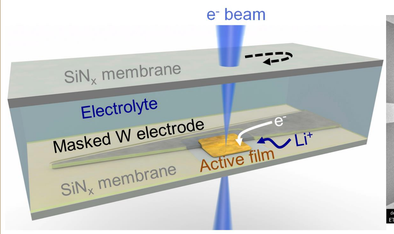
Approach: Microfabricated TEM Liquid Cell

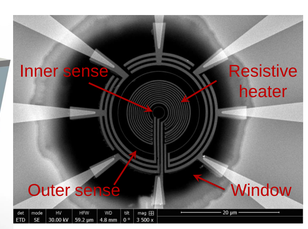
The Center for Integrated Nanotechnologies (CINT) has developed a MEMS device[1], fabricated at MESA, to environmentally control a liquid sample within a transmission electron microscope for nanometer resolution imaging.

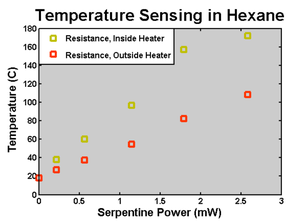


Advantages over a Commercial TEM Liquid-Cell:

- Flat ~120-nm thick liquid layer
- Picoampere current control, quantitative data
- Customized 10 electrodes materials/patterns
- Metallic Li incorporation for stable CE and RE



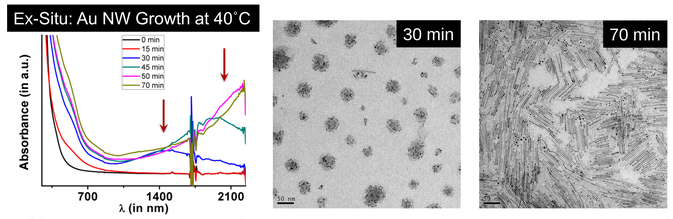




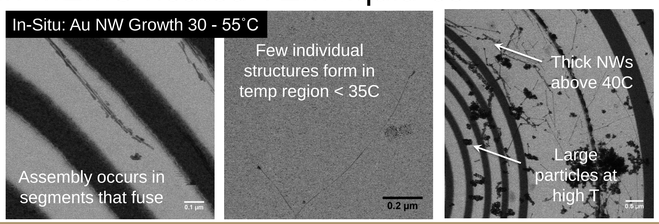
Custom patterning for temperature control and sensing using Pt resistive heating buried under an insulating film.

Elevated Temperature: Au Nanowire Growth and Assembly

Au nanowires for nanoelectronics and biosensors are limited by production of individual ultra-thin single crystalline wires using solution growth.

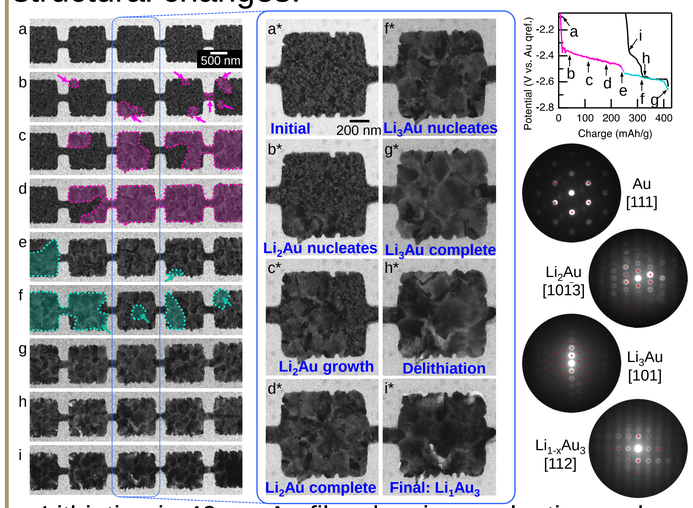


Different growth modes were observed simultaneously due to temperature gradient in MEMS heated TEM liquid-cell.



Li-Ion Battery Degradation: Energy Security, Electricity Storage

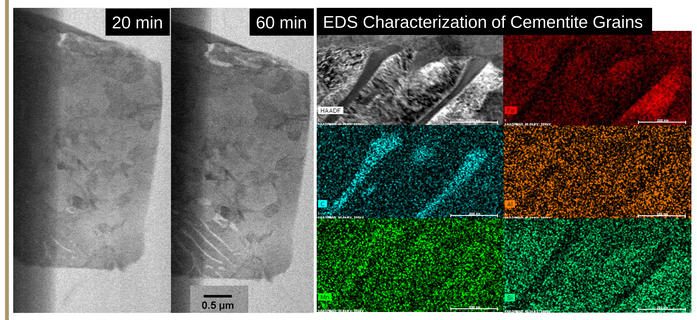
Li-ion battery anode materials may be studied in commercial electrolytes, with relation in voltage plateaus to nanoscale structural changes.



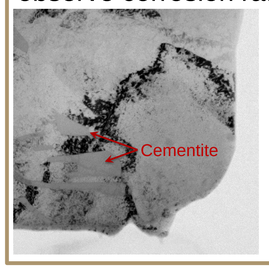
Lithiation in 40 nm Au film showing nucleation and phase front propagation.

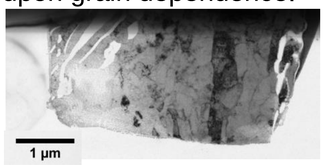
Microfluidic Corrosion: CO_2 and H_2S on Steel

Steel corrosion is a global problem with many factors that influence rate and mechanism, we are looking at processes that occur during oil and gas production.



Focus ion-beam section of 1018 steel was contained in a liquid environment of 1ppm H_2S and 9ppm CO_2 to observe corrosion rates upon grain dependence.





After 18 hours, rapid corrosion of Fe_3C grains and slow overall thinning of Fe-rich grains.

Accomplishments: Quantifiable In-Situ Characterization

Developed a capability to study material systems under external and environmental control with nanoscale real-time imaging.

- Elevated temperature control, up to 175˚C, relative to commercial systems that cannot sense during the experiment and are limited to 80˚C
- High-resolution imaging in thin-flat liquid layers, where commercial systems exhibit thickness gradients and are difficult to obtain <200 nm thick
- Customization for a particular experiment allows for flexibility in electrode layout and ability to use a shadow mask for patterning metallic Li or Na electrodes within confined geometries

Impact:

- **Provides Sandia with characterization of processes previously unobtainable using commercial products, ability to tailor the device design to the experiment**
- **Providing a test device for benefiting the Power on Demand Research Challenge**
- **Solving the fundamental understanding of material properties under working conditions, able to define intermediate states unobservable with general postmortem analysis techniques**

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1. Leenheer *et al.*, *J. Microelectromech. Syst.* **2015**. DOI 10.1109/JMEMS.2014.2380771