

# In situ Electrochemical Electron Microscopy (ISEEM) 2014 Workshop

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Microscope(s) used: FEI Tecnai F30

In situ cell(s) used: solid or liquid open cell

Potentiostat(s) used for ISEEM: Nanofactory controller, or Gamry

Systems studied: Li-ion battery, Na-ion battery, solid-state battery, Li-air battery and Li-S battery.

Research questions you use ISEEM to address: Generally, to understand the mechanisms of the electrochemical reactions and electrodes degradation from a microscopic view. Some examples:

1. Understand the anisotropic volume expansion in Si;
2. Understand the effects and evolution of ALD  $\text{Al}_2\text{O}_3$  layer coating on the active electrodes;
3. Study and control the ionic transport in battery electrodes;
4. Quantitative electrochemistry on individual nanostructures inside the TEM.

Technical difficulties commonly encountered:

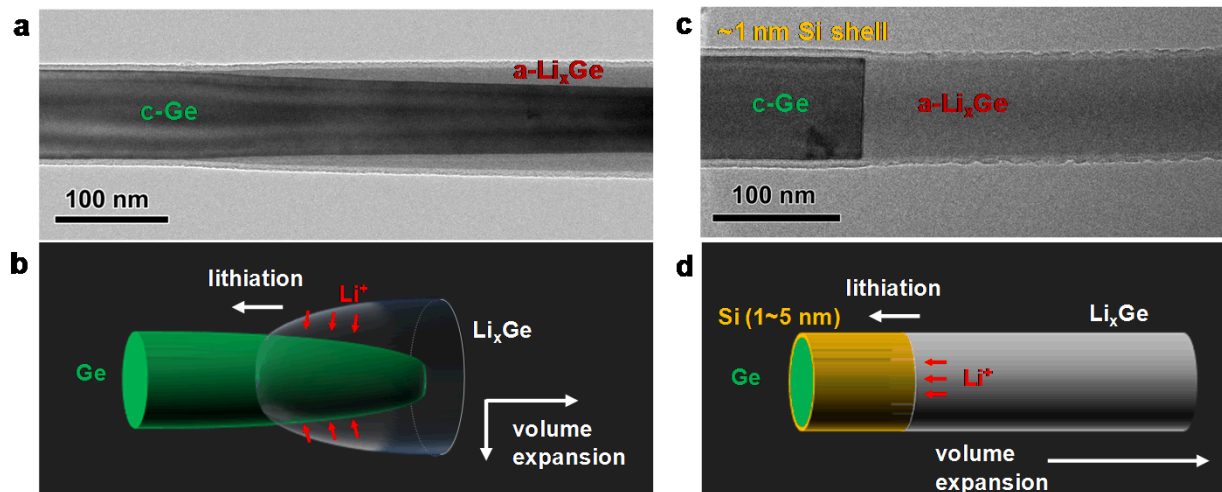
1. How to construct a nanobattery for in-situ electrochemical TEM study?
2. How to minimize and even eliminate the beam effects?
3. How to control the charging/discharging rate?
4. How to control the reaction potential?
5. How to correlate the in-situ results with the ex-situ coin cell results? Bulk electrode v.s. nanosized electrode.

ISEEM publications from your group (0-3):

1. Y. Zhu, J. W. Wang, Y. Liu, X. H. Liu, A. Kushima, Y. H. Liu, Y. Xu, S. X. Mao, J. Li, C. Wang and J. Y. Huang, In Situ Atomic-Scale Imaging of Phase Boundary Migration in  $\text{FePO}_4$  Microparticles during Electrochemical Lithiation, **Advanced Materials**, 25, 5461, (2013).
2. K. E. Gregorczyk, Y. Liu, J. P. Sullivan and G. W. Rubloff, In Situ Transmission Electron Microscopy Study of Electrochemical Lithiation and Delithiation Cycling of the Conversion Anode  $\text{RuO}_2$ , **ACS Nano**, 7, 6354 (2013).
3. Y. Liu, N. S. Hudak, D. L. Huber, S. J. Limmer, J. P. Sullivan and J. Y. Huang, In Situ Transmission Electron Microscopy Observation of Pulverization of Aluminum Nanowires and Evolution of the Thin Surface  $\text{Al}_2\text{O}_3$  Layers during Lithiation-Delithiation Cycles, **Nano Letters**, 11, 4188 (2011).
4. Y. Liu, Z. Y. Zhang, X. L. Wei, Q. Li and L.-M. Peng, Simultaneous Electrical and Thermoelectric Parameter Retrieval via Two Terminal Current-Voltage Measurements on Individual ZnO Nanowires, **Advanced Functional Materials**, 21, 3900 (2011).
5. Y. Liu, H. Zheng, X. H. Liu, S. Huang, T. Zhu, J. W. Wang, A. Kushima, N. S. Hudak, X. Huang, S. L. Zhang, S. X. Mao, X. F. Qian, J. Li and J. Y. Huang, Lithiation-Induced Embrittlement of Multiwalled Carbon Nanotubes, **ACS Nano**, 5, 7245 (2011).

ISEEM publications from elsewhere you most refer to: Work by Chongmin Wang's group @ PNNL

# Tailoring Lithiation Behavior by Interface and Bandgap Engineering at the Nanoscale



Comparative close views of the reaction front area (a) in pure Ge NW with the tapered shape phase boundary, and (c) in Ge/Si core/shell NW with the axial lithiation where the moving phase boundary moves parallel to NW cross section that converted c-Ge into a-Li<sub>x</sub>Ge. Schematic illustrations of (b) core-shell lithiation in pure Ge NWs and (d) axial lithiation induced by an ultrathin Si shell on Ge NWs.

## Research Details

- Core-shell lithiation was observed on pure Ge nanowires (NWs) indicating inward diffusion from surface to core.
- Pure axial lithiation on Ge/Si core/shell NWs showed Li<sup>+</sup> ions do not penetrate the ultrathin surface Si shell.

Yang Liu, Xiao Hua Liu, Binh-Minh Nguyen, Jinkyong Yoo, John P. Sullivan, S. Tom Picraux, Jian Yu Huang, and Shadi A. Dayeh, "Tailoring Lithiation Behavior by Interface and Bandgap Engineering at the Nanoscale", *Nano Letters*, 13, 4876-4883 (2013). DOI: 10.1021/nl4027549