Easing in-situ TEM biasing experiments with movable probes

Rodriguez Manzo, J.A.[1], Karki, K.[1], Sun, J.[2], Ni, Y.[2], Yang, Z.[2], Du, F.[3], Alsem, D.H.[1], Salmon, N.J.[1], Ying Yang, H.[3] and Chen, S.[2]

[1] Hummingbird Scientific, United States, [2] University of Houston, United States, [3] Singapore University of Technology and Design, Singapore

Transmission electron microscopy (TEM) sample holders with biasing capabilities in which one of the electrodes is a movable probe (Figure 1) allow to investigate, simultaneously, the electrical properties of materials or micro-fabricated devices and their structural and chemical state, by taking advantage of the characterization techniques available in a TEM (high-resolution imaging, electron-based spectroscopy, electron diffraction, etc.). This in-situ biasing microscopy technique has been used to investigate, for example, the conductance through rows of atoms [1,2], the switching of oxide resistive memories [3], and anode materials for batteries [4,5]. Here, we discuss practical aspects of the technique with the aim of facilitating this type of in-situ experiments - which can be challenging because of the lack of standards - and present examples in where anode materials where biased and characterized simultaneously.

Regarding the technique's practical considerations, we discuss how to approach the sample with coarse and (piezoelectrically-driven) fine movements and how to prepare the movable probe, inside the TEM column, to obtain a good electrical contact between the sample and the probe prior to the experiment; by "good" we mean contacts bare of contamination or oxide layers that can mask the contact's intrinsic electrical properties. We will show experiments performed with a new in-situ TEM biasing sample holder that allows to approach the sample with the movable probe - usually a scanning tunneling microscopy (STM) tip - in manual coarse mode with smooth and uncoupled steps of µm or fractions of µm on the three spatial axes. Likewise, this holder allows to do the final contact approach with a piezoelectric controlled mechanism that allows uncoupled nanometer-sized (or less) steps on the three spatial axes. We present two applications for this technique: 1) observation of the Li-alloying process in yolk-shell p-Si nanorods@void@nitrogen-doped carbon nanostructures relevant for anode materials in Li-ion batteries (Figure 2), and 2) the investigation of the high capacity retention of Sb2Te3/C composites relevant for sodium-ion batteries. These examples illustrate the convenience of this in-situ biasing technique when creating a contact prior to the experiment is difficult or not possible.

[1] Hideaki Ohnishi, Yukihito Kondo, Kunio Takayanagi. Nature 395(1998), p. 780.

[2] Ovidiu Cretu, et al., Nano Letters 13(2013), p. 3487.

[3] David Cooper, et al., Advanced Materials 29(2017), p. 1700212.

[4] Fei-Hu, et al., ACS Nano11(2017), p. 8628.

[5] Ze Yang, et al., Energy Storage Materials 9(2017), p.214

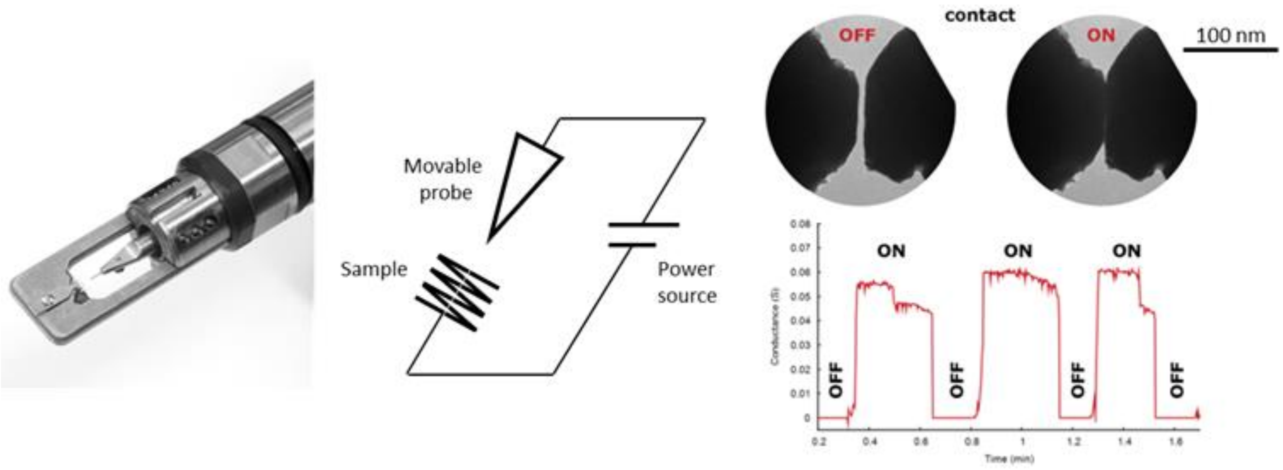

Figure 1: Detail of in-situ TEM biasing sample holder with a movable probe. From left to right, tip of in-situ TEM biasing sample holder, detailed circuit diagram, and conductance vs time plot, showing the conductance between two STM probes (Pt/Ir) as they connect and disconnect continually with the fine piezoelectrally-controlled movement inside a TEM. Contacting two STM probes requires high mechanical precision.

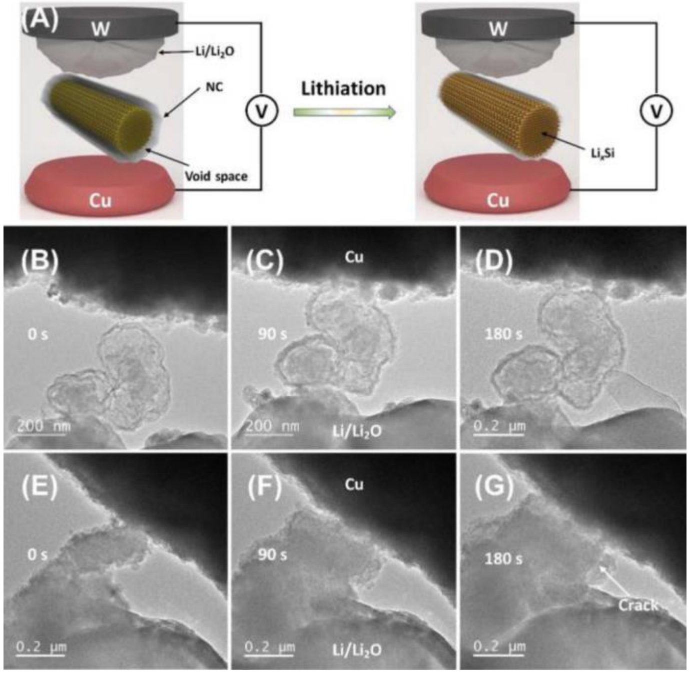

Figure 2: (A) Diagram of the nanoscale electrochemical cell for in-situ (de)lithiation within the TEM. (B–G) Time-lapse images of the lithiation of (B–D) p-Si NRs@void@NC and (E–G) p-Si NRs@NC composites (adapted from reference [4]).

Shuo Shen acknowledges support from the Defense University Research Instrumentation Program (FA2386-14-1-3007).