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AirSEM: Electron Microscopy in Air, without a Specimen Chamber

Kayla Nguyen[1], Megan Holtz[2] and David Muller[2]

[1.]Department of Chemistry and Chemical Biology, Cornell University, Ithaca, NY, USA.
[2.]School of Applied and Engineering Physics, Cornell University, Ithaca, NY, USA.

The development of environmental scanning electron microscopes (ESEMs) has allowed the study of specimens in a gaseous environment. The ESEM typically operates at below atmospheric pressure due to the long gas path length and need for differential pumping apertures. A new generation of Atmospheric scanning electron microscopes (ASEMs) allow samples to be imaged in liquid or at atmospheric pressure through an electron-transparent window that separates the column of the microscope from the sample [1, 2]. One approach to dealing with the short required working distance has been to image directly into the liquid with an inverted SEM column below a silicon nitride window held by a petri dish [1]. Here, we explore an alternative design for a general-purpose field-emission AirSEM from b-Nano [2]. This is an upright geometry where the sample is mechanically positioned 50-200 microns below electron-transparent window after computer-controlled alignment with an optical microscope (Fig. 1a). This decouples the sample from the window, allowing for its reuse and enables multiple signal collection such as gas detection, in addition to the backscattered electron detector (BSE) [2,3].

The highest resolution images are obtained from interactions with the unscattered beam. Monte Carlo simulations [4] (Fig 1b) show the electron mean free path (mfp) changes from 75 μm in air to ~900 μm in He. Therefore, in order to achieve high resolution, we placed our specimen 50 μm away from the SiNx window and mix helium into the flow across this gap. With a standard Au on carbon resolution test sample, we were able to image Au nanoparticles smaller than 10 nm in BSE mode at 15 and 30 keV (Fig 1c).

Due to the AirSEM's flexible design, we can work with in-situ cells and detectors with volumes several inches in each dimension without using vacuum feedthroughs, and samples can simply be placed on an optical microscope slide. The registration between the optical microscope and the electron beam is useful for correlative microscopy. Fig. 2 shows the comparison between optical and BSE images of a proton-exchange membrane fuel cell after 30,000 cycles of operation where there is coarsening and migration of the Pt_3Co nanocatalysts, some of which have grown from their original 5 nm diameter to sizes of 10s to 100s of nm. Fig 3a shows the electrode of an aged Li-ion battery. Fig 3b shows wet moss imaged on a glass side, illustrating the minimal preparation needed for wet biological samples. Fig 3b shows a time sequence from the in-situ growth of Pb dendrites on the surface of a liquid. From these experiments, two notable features of the instrument are the rapid specimen turn-around time (a few minutes per sample) and the ready access for in-situ cells and detectors. [5]

References:
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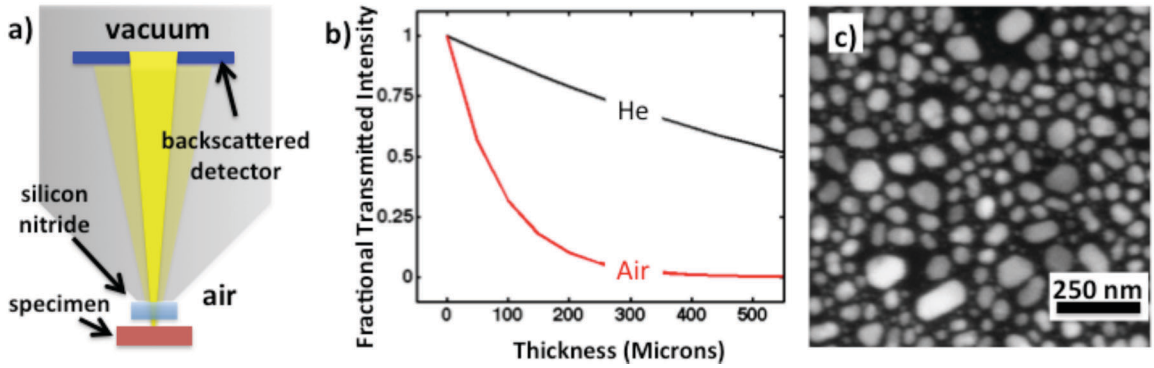

Figure 1. a) Schematic of the AirSEM. b) Fraction of the unscattered beam remaining as a function of distance through helium and air. c) Au nanoparticles as resolution test-smallest particles are ~ 10 nm and are clearly resolved with smaller gaps also visible.

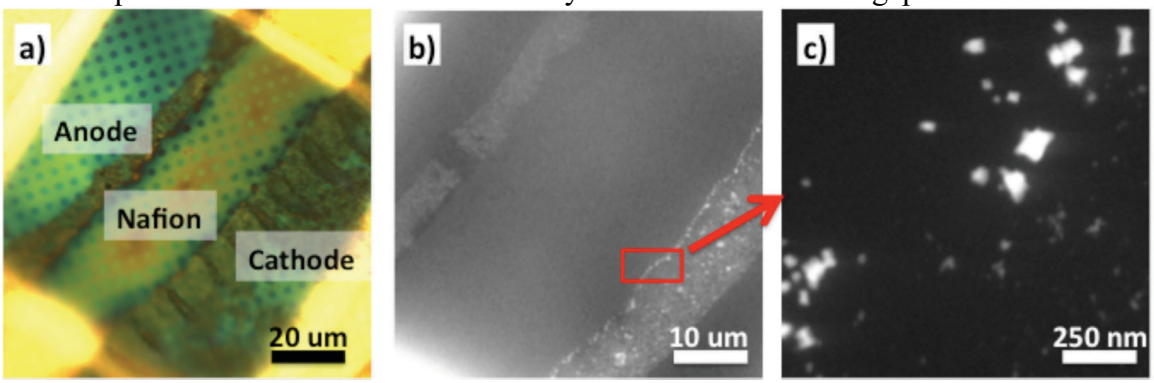

Figure 2. a) Optical image of a cross-sectioned proton-exchange membrane fuel cell. b) AirSEM image of the same fuel cell. c) Higher magnification of the cathode showing the Pt_3Co catalysts, that have coarsened after 30,000 cycles aging.

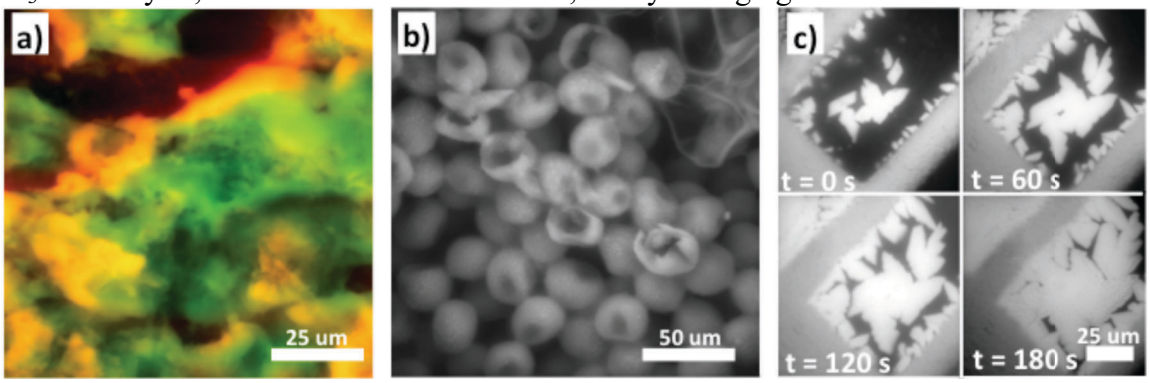

Figure 3. a) Aged Li-ion battery electrode with false color scale overlapping BSE (green) with Surface Detector signals (red) b) Wet moss. c) In-situ growth of Pb dendrites from solution.

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