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AirSEM: Electron Microscopy in Air, without a Specimen Chamber

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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 directly image 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 a computer-controlled alignment with an optical microscope (Fig. 1a). This decouples the sample from the window, allowing for its reuse. The accessibility of the sample, without the need for vacuum feedthroughs makes it very simple to add imaging modes, including secondary ion detector, x-ray mapping, and cathodoluminescence.

Nanoscale resolution can be retained at atmospheric gas path lengths (GPL) as long as 400 μm, and beam spreading in the electron-transparent window is the dominant effect on contrast at typical operating conditions [3]. High-resolution imaging is possible only if a usable fraction of the incident beam remains unscattered, and the scattered electrons are so dispersed as to provide a broad, structureless background. The contrast reduction will then follow a product of Beer's law exponential decays for the window, and the gas atmosphere. The key is to ensure that the window and GPL thickness should not exceed a few mean free paths (mfp). The mfp at 30 keV for the window is ~30 nm for silicon nitride, and 75 μm for air. Graphene is an ideal window material due to its high mechanical strength, single atomic layer thickness and long electron mfp, which can keep the combined path length to within one mfp, a single-scattering regime comparable in resolution and contrast to vacuum SEM (Fig 1b) [4].

With these thinner windows, lower voltage (5-7 keV) imaging becomes practical. Ionization of gas molecules from ejected secondary electrons produces a measurable ion current that is read off from the conducting window, allowing a form of gas amplification imaging that produces a surface sensitive signal in air. As a check of surface sensitivity, Fig. 2a shows surface image (SI) of a monolayer of graphene on a copper foil. Fig 2b,c compares resolution from vacuum SEM and the airSEM using the thin graphene window[4]. Low beam voltage work is also possible with a thin nitride membrane. Fig 2c,d shows the simultaneous BSE and SI images of wet moss, using a 5nm SiN_x window and a 7 keV beam energy.

The addition of an x-ray detector allows for the rapid compositional mapping of samples in air. This can be in either SEM mode, or STEM for higher spatial resolution. Fig 3 shows a sample from a candidate Li-S battery electrode with hollow carbon spheres infiltrated with sulfur. Sulfur sublimates in the vacuum of an SEM, making this an ideal candidate for airSEM/STEM [5]. From the x-ray maps of Fig 3c,d we see sulfur has infiltrated the walls, but not the hollow interior. [6]

References:

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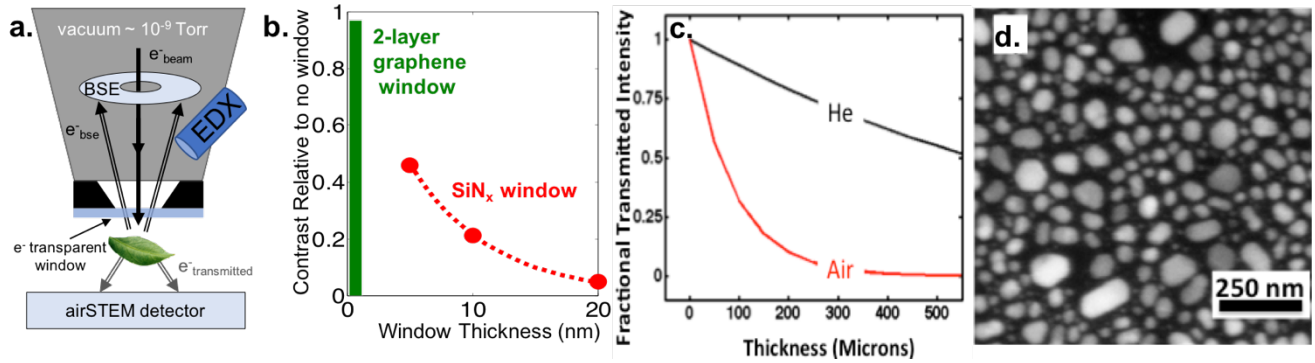

Figure 1. a) Schematic of the airSEM. b) Monte Carlo simulation of relative contrast for graphene window and SiN_x windows vs no window at 7keV. c) Fraction of the unscattered beam remaining as a function of distance through helium and air. (d) Au nanoparticles as resolution test for BSE imaging at 30 keV– smallest particles are ~ 10 nm and are well resolved, with smaller gaps also visible.

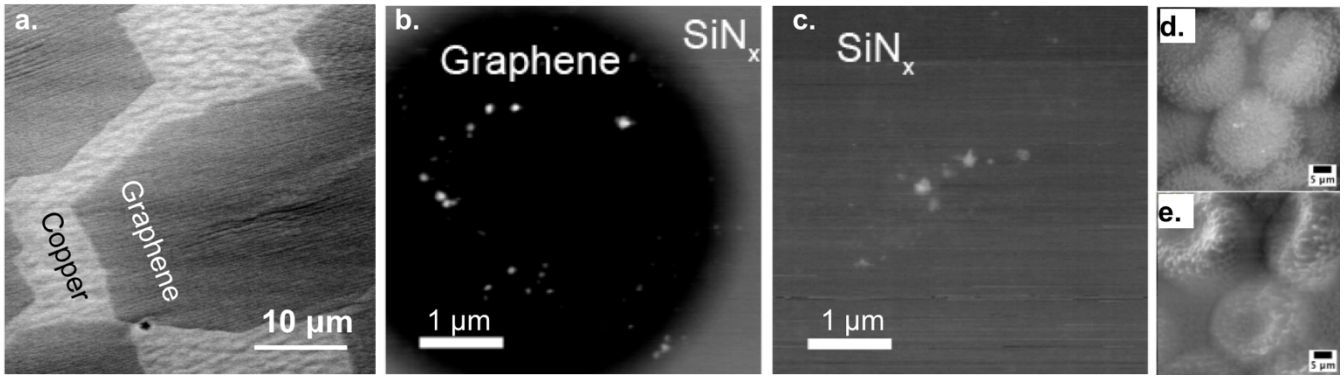

Figure 2. Surface Imaging (SI) using ion current from the window. a) Partial graphene monolayer on a copper foil. Pt catalyst nanoparticles in an aged proton-exchange-membrane fuel cell cathode imaged with a conventional FESEM (b) and the airSEM (c) with a 15 keV beam energy. d) Backscattered images of wet moss in air at 7 keV with a 5 nm-thick SiN_x window. c) Simultaneous SI image of wet moss.

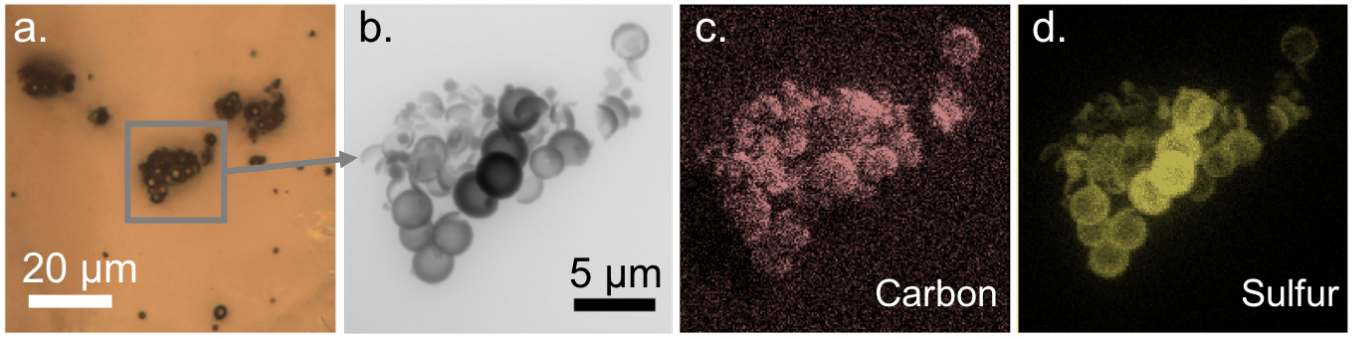

Figure 3. (a) Extended depth of field optical microscope image of clusters of carbon-sulfur spheres recorded on the airSEM's optical microscope. b) 30 keV airSEM BF-STEM image of single cluster of carbon spheres from the same region as (a). c) & d) XEDS maps showing carbon and sulfur signals that confirm sulfur infiltration into the sphere walls, but suggests no infiltration into the sphere interior.

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