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New electron microscopy tools for characterizing air-sensitive samples.

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Lithium-metal- or lithium sulfur-based systems can provide safe, high capacity and longer cycle life batteries compared to the state-of-the-art liquid electrolyte-based batteries that have serious safety issues. Successful use of solid electrolytes holds promise of not only providing batteries with increased safety but also higher energy density, for example by allowing the use of lithium metal as an anode in Li-ion batteries [1]. For example, it has been shown that use of block co-polymer electrolytes can substantially suppress dendrite formation in a lithium anode [2], [3], thus preventing shorts that result in thermal runaway and fires in batteries. There is a clear need for understanding the failure mechanisms in these solid-state batteries, that can be done by closely studying the materials and the interfaces between them while they are in operation. A major problem related to in situ study of solid-state batteries using electron microscopy is that they often contain materials such as lithium metal, that transform quickly when exposed to air or moisture before they are inserted into the microscope when we use the commercially available biasing or cycling TEM holders. Moreover, it is also challenging to prepare electron microscopy samples of lithium and other air-sensitive materials.

ZoNexus has developed several products to address aforementioned issues related to electron microscopy of air-sensitive materials. In this presentation two products for in situ and ex-situ characterization of air-sensitive samples will be demonstrated:

1) **Air-free transfer in situ TEM holder**: This holder was developed for performing in situ experiments such as solid-state battery cycling, biasing and heating on air-sensitive samples. The in situ TEM holder was developed with special considerations to difficulties in handling specimens in a glovebox and features a user-friendly connector for accepting MEMS devices coupled with a double-tilt cradle. The retractable tip with an O-ring, which is operated using a knob, helps in sealing the sample in inert gas or vacuum as shown in Figure 1A. Figure 1B shows preliminary results obtained on electrochemically deposited lithium filaments. Lithium quickly transforms to lithium oxide and lithium carbonate when exposed to air. It is worth noting that the change in crystal structure occurs without substantial change in the morphology of the lithium filament.

2) **Air-free transfer module for SEM/FIB**: As shown in Figure 1C, this device consists of a small chamber for air-free transfer of air-sensitive samples from glove box to SEM/FIB and vice versa. The low-profile design and small footprint allow tilting of module to more than 52 degrees towards the ion-column without interfering with the pole piece, probes and detectors in the FIB/SEM. The module also features and elevator to raise the sample by ~ 15 mm. A motor-controlled swinging lid is used to seal the sample in the glove box, which can be opened inside the FIB/SEM using a touch-screen control mounted on the SEM or a remote. It has a customizable design for addition of wiring for heating, temperature measurement, electrochemistry or biasing experiments.

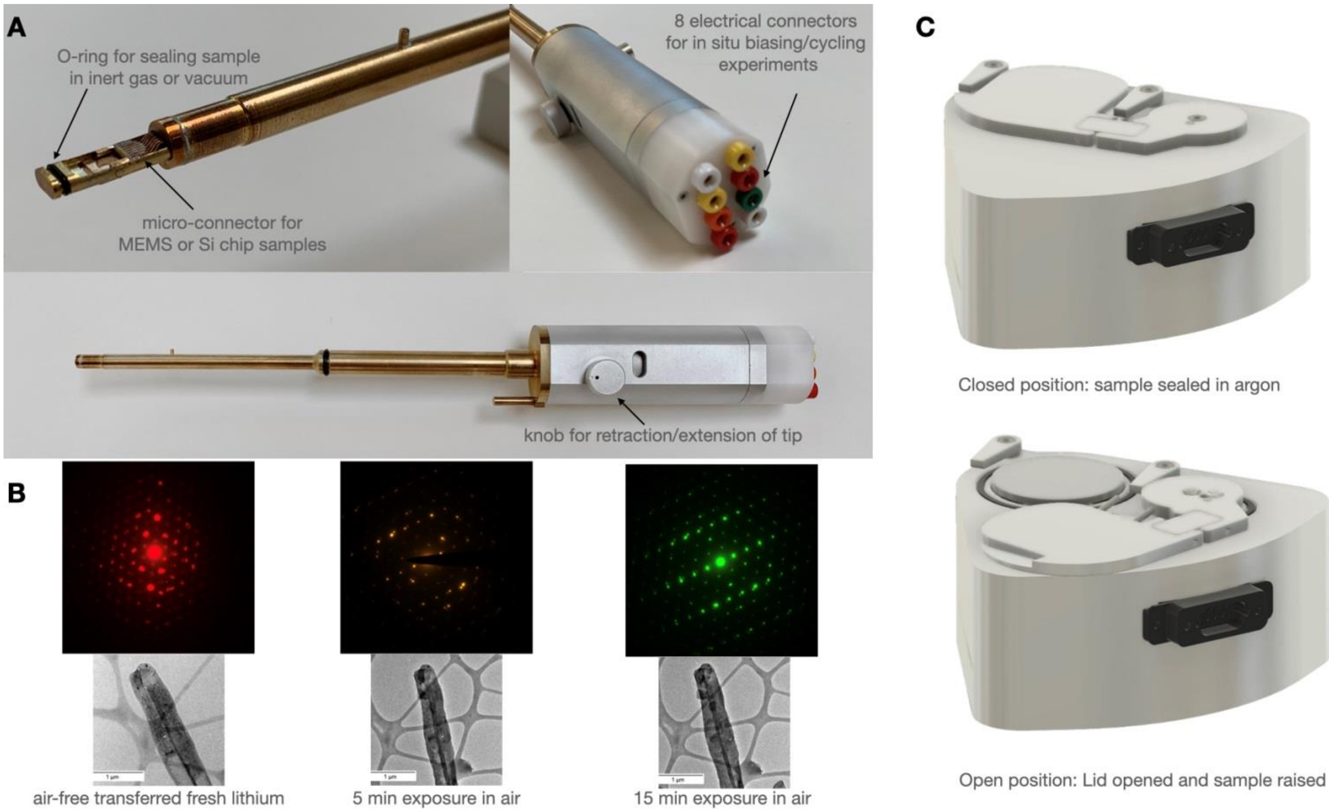

Figure 1. Tools for electron microscopy of air-sensitive samples: A) Air-free transfer in situ TEM holder, B) Selected area electron diffraction patterns and bright field image showing transformation of electrochemically deposited lithium metal, C) Air-free transfer SEM/FIB module.

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