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Correlative UHV-Cryo Transfer Suite: Connecting Atom Probe, SEM-FIB, Transmission Electron Microscopy via an Environmentally-Controlled Glovebox

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For certain samples, the application of advanced microscopy is limited by the requirement for the specimen to be inserted from the outside atmosphere into a high vacuum environment. Transfer through air means that it is challenging to study air sensitive materials, or processes that occur in environments other than air, such as catalysis and corrosion. Also, under normal circumstances, the vacuum chamber of most microscopes supports only solid samples, also preventing the study of wet and/or organic structures in their native state.

To tackle this issue, many microscopy centres have established vacuum or cryo workflows that are designed around a particular microscopy technique (e.g. TEM or light microscopy (LM)). Modern microscopy workflows for materials science typically involve the preparation of a specimen in one type of instrument (e.g. a focused ion beam (FIB)) and examination of the specimen in another (e.g. a transmission electron microscope (TEM) or even an atom probe). In the life sciences, correlative microscopy via cryo transfer systems is becoming well-established. A number of research labs worldwide are working on the development of correlative transfer systems that incorporate atom probe tomography, and some excellent results have already been achieved [1-7].

The University of Sydney has recently installed its own suite of advanced microscopy tools that allow the preparation and transfer of samples into and between instruments via ultra-high vacuum and/or cryogenic transfer. These facilities include a purpose-built controlled-atmosphere glovebox (Microscopy Solutions), a Zeiss Auriga scanning electron microscope-focused ion beam (SEM-FIB) equipped with a custom-designed cryogenic stage (also Microscopy Solutions), and a CAMECA laser-assisted local electrode atom probe (LEAP) equipped with a Vacuum and Cryo Transfer Module (VCTM) on the load lock, all which are connected by a Ferrovac UHV suitcase. It is also possible to transfer cryo specimens prepared in the FIB to a JEOL 2100 TEM, by transferring them through a liquid nitrogen bath in the glove box into a ThermoFisher Scientific Vitrobot sample box. The precise hardware configuration has been developed in collaboration with Microscopy Solutions (with support from the University of Queensland) and CAMECA.

The new transfer suite enables production of the nanoscale geometries required for APT and TEM under controlled cryogenic temperatures. In this talk, we will provide an overview of this system, and initial results that validate the capabilities of the integrated cryogenic and transfer system. Results include atom probe data from deuterated alloy samples and clean samples with organic self-assembled molecules. TEM and atom probe results will also be presented from lithium battery materials that require vacuum transfer for atom probe and TEM.

References:

[1] Y Chang et al., Nature Communications **10** (2019), p. 942.
[2] DK Scheiber et al., Ultramicroscopy **194** (2018), p. 89.
[3] P Dumitraschkewitz et al., Adv. Eng. Mater. **17** (2017), 1600668, doi:10.1002/adem.201600668 .
[4] Y.S. Chen et al., Science **355** (2017), p. 1196.
[5] SSA Gerstl et al., Microscopy and Microanalysis **23**(S1) (2017), p. 612.
[6] S Gerstl, W. Wepf, Microscopy and Microanalysis **21** (2015), p. 517.
[7] DE Perea et al., Scientific Reports **6** (2016), p. 22321.

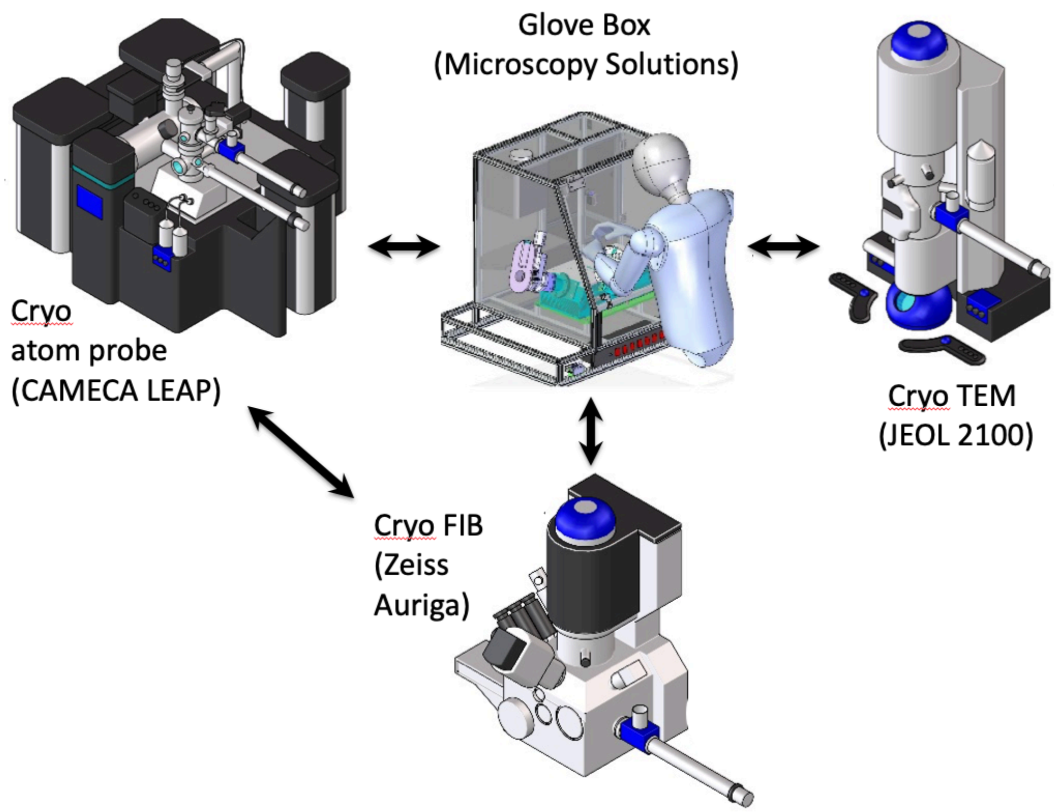

Figure 1. Schematic of the apparatus that comprise the correlative cryo microscopy suite.