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Advanced Cryo-FIB Specimen Preparation and Handling of Environmentally Sensitive Materials for APT Analysis

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The liquid-solid interface plays an essential role in many phenomena encountered in biological, chemical, and physical processes relevant to both fundamental and applied science [1]. However, hydrated materials, or more generally liquids, present a technical challenge for high-resolution microscopy as they are generally incompatible with a broad range of analytical tools that require high to ultrahigh vacuum conditions. One strategy to preserve and probe the liquid-solid interface is to cryogenically solidify the liquid [2]. By doing so, the surface composition and morphology, as well as local ionic chemical gradients, can be preserved within the solid liquid structure, making it more amendable to vacuum-based high-resolution analyses of environmentally sensitive materials.

Motivated to apply the analytical technique of Atom Probe Tomography (APT) to study the liquid-solid interface relevant to materials science and biology, much effort has been dedicated at the Pacific Northwest National Laboratory to develop custom hardware and protocols that allow the use of a cryogenic FIB (cryo-FIB) to prepare specimens, and a modified specimen shuttle suitcase device for environmental transfer to the atom probe for APT analysis. Through this unique cryo-based approach [3, 4], the APT analysis of a liquid-solid interface within a nanoporous material can be revealed [4, 5].

In this tutorial, I will walk you a detailed journey that is not only enables the near-atomic scale analysis of the liquid/solid interface with APT but also enables access to new science [1, 3]. The beginning of such a journey requires the development of a plan to prepare specimens in a cryo-FIB, have a means to transfer the specimen between the FIB and the atom probe within a protected environment, and then a means get the specimens into the atom probe. To prepare specimens cryogenically in a FIB, a cryogenic specimen preparation system from Quorum Technologies was modified to be compatible with atom probe specimen puck carriers on a FEI Helios 600 dual beam FIB-SEM (Fig. 1A). From the atom probe side, an environmental transfer hub station (ETH) high vacuum chamber with integrated specimen shuttle suitcase load lock was custom designed and fabricated for the acceptance, temporary cryo storage, and transfer of cryo-FIB prepared specimens into the atom probe for analysis (Fig. 1B). To facilitate the environmentally protected transfer of specimens between the cryo-FIB and the atom probe, a modified specimen transfer shuttle suitcase device from Quorum Technologies is used (Fig. 1C). A full description of these devices are described in Ref [3].

With the hardware technology in place, a procedure was developed to actually perform a liftout, mounting, and sharpening of cryogenically frozen specimens for APT analysis (Fig. 2A). This procedure utilizes nanowelds and backfilled notch cuts to create a mechanically robust attachment of the specimen to micropost substrates compatible for APT analysis. As a working example of this approach, the liquid-solid interface making up a nanoporous corroded glass specimen was prepared and analyzed via APT, while maintaining the liquid water as frozen ice (Fig. 2B). A full description of these results are described in Ref [4, 5]. Throughout this tutorial, I will describe in detail, the challenges and considerations involved in applying this approach to a broad range of materials spanning materials science, geology, and biology, and discuss the potential for cryo-based correlative TEM and APT studies

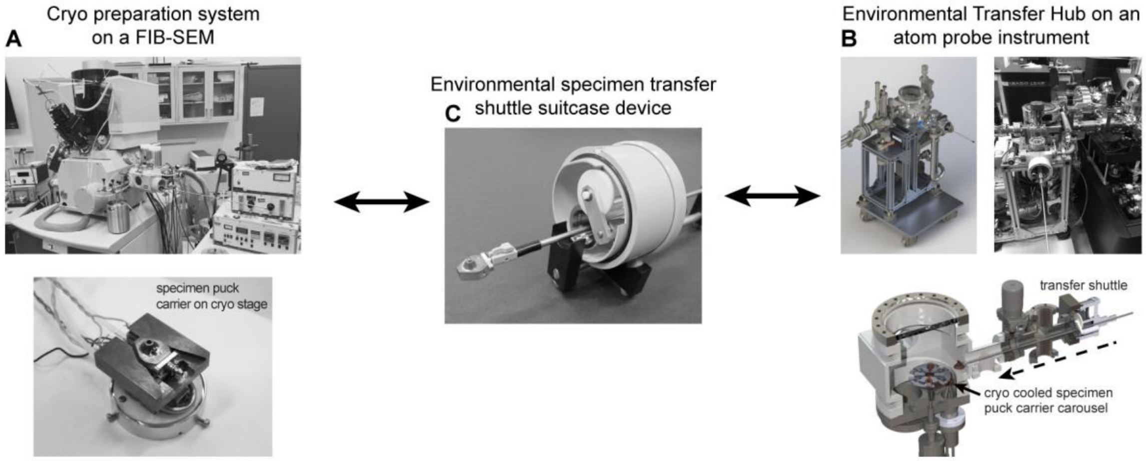

Figure 1. Development of the handling, preparation, and transfer of cryogenic specimens for APT analysis. A). A modified cryo specimen preparation workstation by Quorum Technologies attached to a FIB. B). The environmental transfer hub attached to an atom probe to enable to acceptance of cryogenically prepared specimens for APT analysis. C). Environmental specimen transfer shuttle suitcase device used to facilitate the transfer of cryogenic specimens between the FIB and atom probe.

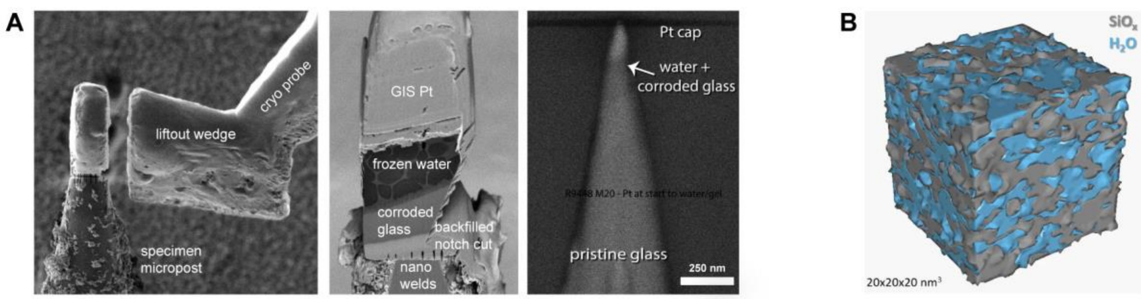

Figure 2. Cryo-FIB fabrication and APT analysis of a specimen targeted at the liquid/water interface of a corroded nanoporous glass. A). Liftout, attachment, and final specimen geometry of the liquid/solid interface in a corroded glass. B). APT reconstruction of a corroded nanoporous glass structure showing the silicate network (grey) infiltrated by water (blue). The structure is delineated by 9 ion% water isoconcentration surface.

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