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Developing cryogenic and vacuum transfer capabilities at the Australian Centre for Microscopy and Microanalysis

Ingrid McCarroll[1], Levi Tegg[1], Luke Daly[2], Takanori Sato[1] and Julie Cairney[1]

[1]The University of Sydney, United States, [2]The University of Glasgow, United States

Through new developments in the area of cryogenic and vacuum transfer, it is now possible to study non-traditional materials and phenomena using atom probe tomography, including soft matter and the distribution of solute hydrogen. At the University of Sydney we have been developing one of only a handful of systems that enable the transfer of samples at, or near to liquid nitrogen temperatures between multiple characterisation platforms. Our cryogenic interconnected laboratory incorporates a local electrode atom probe, a focused ion beam-scanning electron microscope (FIB-SEM) with a cryogenic stage and a glovebox that enables the transfer of samples between a nitrogen atmosphere and high-vacuum at cryogenic temperatures. To facilitate the transfer of these samples we utilise an ultra-high vacuum cryogenic transfer suitcase, pictured herein.

A number of similar infrastructures have been developed around the globe to expand the current capabilities of atom probe tomography, each providing slightly different features [1]. The system at the University of Sydney differs from many of these in that it employs active cooling and pumping during sample transfer, and maintains ultra-high vacuum in the transfer suitcase. Furthermore, the transfer suitcase is interfaced to a purpose-built glovebox, which provides a dry nitrogen atmosphere and the capacity to introduce large quantities of liquid nitrogen. Experiments can be undertaken within the glovebox, followed by immediate and rapid cooling of the samples before transferring them to the suitcase. Samples can then be transferred directly from the transfer suitcase to the atom probe or first to the FIB-SEM. The glovebox also has a port for cryo-transmission electron microscopy holders, enabling a pathway for future transfer of cryogenic samples between scanning electron microscopes/atom probes and transmission electron microscopes. The system is designed in such a way that these transfers can be undertaken within the one lab, but also has the potential to enable samples to be transferred between instruments at different institutions.

A key challenge in preparing samples for atom probe tomography is producing the correct specimen 'tip-shape' geometry. For certain experiments it is possible to use a pre-shaped tip and directly transfer the specimen between the atom probe and the glovebox. For other experiments it is necessary to first transfer samples to the FIB-SEM to either prepare the tips via bulk annular milling [2] or site-specific lift-out methods [3]. It is currently unclear what effect the focused ion beam may have on the distribution of hydrogen within a material. Understanding this influence is critical to the preparation of materials, such as geological samples, deuterium charged steels, and particularly biological specimens. Through undertaking complex experimental workflows we have explored the effects of focused ion beam milling on the distribution of deuterium within steels and on the distribution of elements within geological specimens.

This new frost-free transfer suite enables production of the nanoscale geometries required for atom probe analysis under controlled cryogenic temperatures. In this presentation we will highlight the developments that we have been making towards enabling the study of vitreous ice, critical to the study of biological specimen via atom probe tomography. We will also highlight some of the recent results that we have obtained exploring the distribution of hydrogen in steels, light alloys, and geological materials, including the influence of FIB.

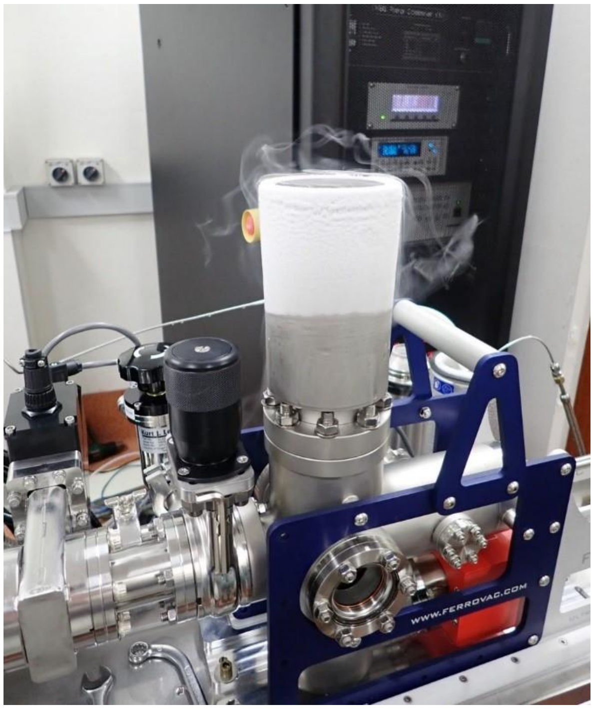

Figure 1. Transfer suitcase attached to a LEAP4000.

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