doi:10.1017/S1431927617003737
Microsc. Microanal. 23 (Suppl 1), 2017

Enabling Atom Probe Analyses of New Materials Classes with Vacuum-Cryo-Transfer Capabilities

Stephan S.A. Gerstl[1], Sebastian Tacke[1], Y-S.Chen[2], Julia Wagner[3], Roger Wepf[4]

[1.] ETH Zurich, ScopeM, Zurich, Switzerland.
[2.] Oxford University, Dept. of Materials, Oxford, UK.
[3.] Inst. Materialwissenschaft, U. Stuttgart, Stuttgart, Germany.
[4.] University of Queensland, Centre for Microscopy and Microanalysis, Brisbane, Australia.

Atom probe tomography (APT) has become an increasingly integral part of the characterization toolbox due to its capability of measuring nanometer chemical details in 3-dimensions – of predominantly hard & dense materials. Developments at ETH Zurich [1] enables the transfer of cryogenically arrested materials into the analysis chamber, thereby facilitating the analysis of 'dynamic' – rapidly diffusing, soft, or even liquid – materials to be investigated via APT.

In order to transport frozen samples, a high vacuum or inert gas environment with temperature control is necessary to prevent changes to the sample surface (Fig.1).[2] Both environments are realized to enable the cryo-transfer method: cryogenically frozen samples are stored in either (1) a cryo-vacuum chamber or (2) LN_2. In case (1), samples are moved directly with Leica's Vacuum Cryo Transfer (VCT) shuttle to the analytical machine of choice. In case (2), samples are transferred into an inert gas glove box (such as dry N_2), to manipulate them into a shuttle, used for transporting them to a post-processing unit to achieve high vacuum, followed by (as in case 1), VCT to either Cryo-FIB or Cryo-transfer-enabled LEAP.

Cryo-transfer-enabled LEAP is achieved with a VCT port on the Load-lock (Fig.1, inset), where the sample is transferred at < 3E-7 mbar through a cryogenically cooled anti-contamination tunnel into a cryo-carousel, which has been actively cooled to below -190°C from a Dewar mounted on the LL. Thermal measurements recorded during the cryo-transfer show the sample remains below recrystallization temperatures of ca. -140°C until it reaches the analysis chamber at < -200°C. Comparative electron microscopy images from multiple VCT movements between FIB to LEAP to FIB have confirmed the vacuum and low temperature fidelity, such as when transferring ½-grids with plunge frozen aqueous solutions on them, demonstrated in Fig. 2.

By enabling the transport of specimens in vacuum and cryogenically arrested states, a variety of new materials classes can be analyzed via APT. These include, but are not limited to, 'dynamic materials' such as: alloy surfaces prone to corrosion & other environmentally induced alterations, materials with rapidly diffusing solutes, H or D in steels [3], polymers, and ultimately cellular subunits. An example of the rapid aggregation of Mg in an Al 6XXX alloy (first solution annealed, then air quenched), reveals the growth, Fig. 2, of nanometer sized chemical features resulting from just 20 min. aging at room temperature. For even more dynamic materials, early results [4] show APT measurements are possible of small volumes of aqueous solutions.

The capability to cryogenically transfer specimens into an atom probe enables the interrogation of materials details not accessed previously, thereby increasing the materials space APT can investigate.

References:

[1] Gerstl S.S.A.; Wepf R. Microsc. Microanal. 21, 517 (2015).
[2] Wepf R. et al. High Resolution Cryo SEM of Macromolecular Complexes in "Biological Field Emission Scanning Electron Microscopy," edited by R. Fleck and B. Humble (Wiley, submitted)
[3] Chen Y-S.; Haley D.; Gerstl S.S.A.; London A.J.; Sweeney F.; Wepf R.; Rainforth W.M.; Bagot P.A.J.; Moody M.P., (accepted in Science).
[4] Moody M.P., Vella A., Gerstl S.S.A., Bagot P.A.J. (2016). MRS Bulletin, 41, pp 40-45.
[5] S.G. acknowledges funding from ScopeM (formerly EMEZ) R&D grants.

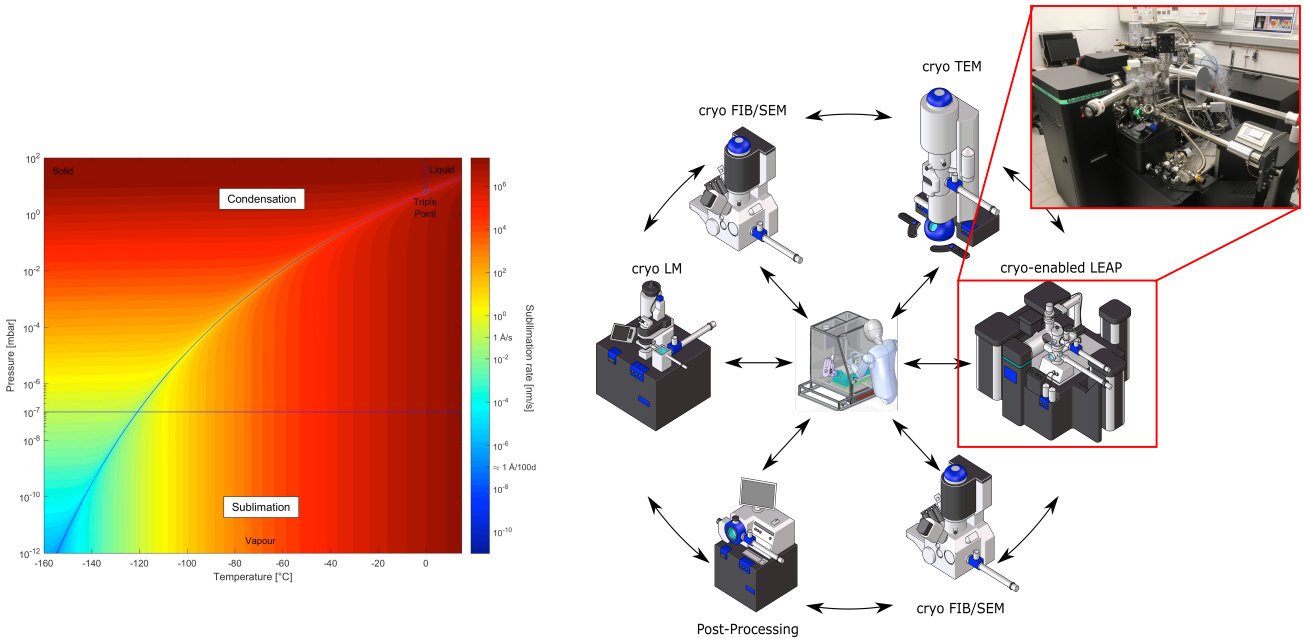

Figure 1. (Left) The portion of the water phase diagram showing critical relationships to regard while transferring cryogenically arrested materials between chambers. (Right) The typically dry N2 filled glove box (at center) is instrumental in transferring specimens without contamination between multiple stations & microscopes; inset image of Cryo-enabled LEAP tomograph.

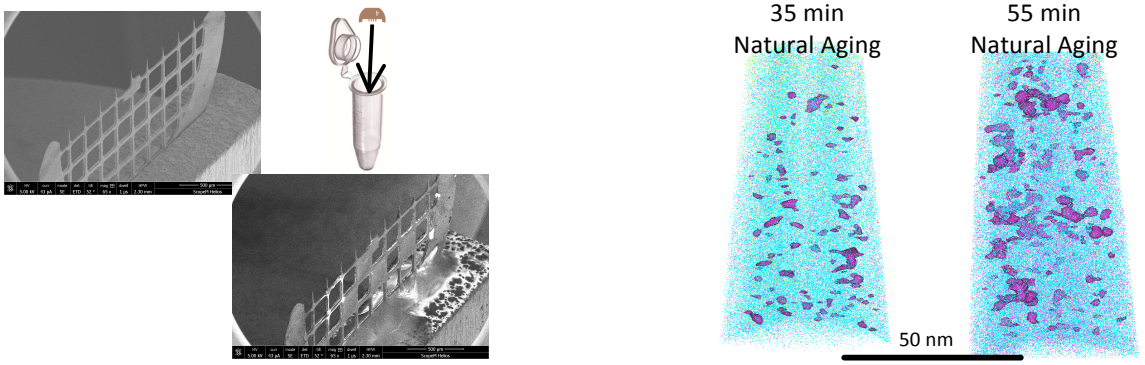

Figure 2. (Left) ½-grid field developed tips, dipped in a fluid ROI, then plunge frozen & cryo-transferred is a method applied to analyze aqueous solutions. (Right) Two reconstructions from two analyses (on the same needle specimen) displaying the *same* Mg isosurface, after 35 or 55 min. aging following cryo-transfer from an as quenched state; aged in the buffer chamber at room temperature.

https://doi.org/10.1017/S1431927617003737 Published online by Cambridge University Press