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Applications, Technical Challenges, and Recent Implementation of a UHV/Cryogenic Specimen Transfer System for Atom Probe Tomography

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The ability to control or prevent the change of a sample from its native state prior to analysis is a desire of nearly every microscopy technique. For a wide variety of microscopies and associated applications, changes related to exposed surfaces or the bulk temperature history have little or no effect on the goal of the analysis, e.g. characterization of stainless steel grain size. For other studies, a very carefully controlled environment (temperature, pressure, atmosphere, etc.) is critical, e.g. recording the oxidation state of an exposed lithium metal sample. For atom probe tomography (APT), sample preparation is critical as the region of interest must be placed within a needle geometry with a typical diameter of less than 200 nm at the apex. Like transmission electron microscopy, the surface-area-to-volume ratio of a typical APT sample (Figure 1) can be tens of orders of magnitude larger than a typical metallographic mount [1].

Even with such a surface rich sample, APT can be applied successfully to many applications without stringent environmental requirements. For example, the vast majority of APT samples are sharpened into the required needle shape by either electrochemical polishing (followed by rinsing and drying), or shaping in a FIB-SEM after mounting to a carrier. The requirement is that either exposure to normal laboratory air creates a very thin and protective oxidation layer (as in silicon or aluminum), or is relatively inert over the minutes or hours a specimen may wait prior to entry into the UHV system of an atom probe microscope. These restrictions strongly limit the ability for APT to be applied to a variety of applications, for example:

- Rapid oxidizers (e.g. uranium, lithium)
- Surface contamination (e.g. catalysts)
- Characterization of hydrogen content in steels, semiconductors, etc.
- Analysis of "soft" materials potentially encased in vitreous ice (e.g. biological)
- Transport between various microscopic analysis/treatments (e.g. FIB-SEM, reaction chambers)
- Observing specimen apex shape at intervals during APT analysis to improve reconstruction

Example use cases such as a specimen transferred from one microscope to another, or modified in a treatment chamber are depicted in Figure 2. The most challenging specimens are those that need to be kept at cryogenic temperatures after specimen preparation is complete. To maintain a clean surface, the sample must be kept in an environment with an extremely low partial pressure of water and other contaminants that may condense and or freeze on the surface. For some microscopies, solutions to these problems are commercially available, but only a few sites in the world have even tried to do cryo transfer successfully into an atom probe. Teams at ETH in Switzerland, Oxford University in England, Pacific Northwest National Laboratory, and the University of Michigan in the United States have built or adapted commercial UHV transfer systems with different capabilities [2][3]. Based on demonstrated success, and growing interest in applications that require this capability, a new design has been

completed in a collaboration between the Max Plank Institute for Steel Research in Dusseldorf, CAMECA, and Ferrovac in Zurich Switzerland.

The transfer design is based on the mobile UHV Suitcase and Fast Pump Down Dock from Ferrovac, customized to transfer samples held in a standard LEAP specimen puck (Figure 2). This can be attached to a specially modified UHV buffer chamber of a LEAP 5000 system. The very low moisture UHV transfer system includes cryogenically cooled (< 100K) stage, shielding, an ion and getter pump, and a load lock chamber with a dedicated turbo molecular pump and cryogenic cooling that can achieve less than 1E-8 torr in less than 30 minutes. Inside the LEAP system the sample is transferred onto a specially designed carrier puck that can be pre-cooled on the standard LEAP specimen stage and transferred to a carousel with a position that is comprised of a low heat transfer UHV compatible polymer (Figure 1c.). Transfer can be completed in less than 30 seconds, maintaining the sample well below the recrystallization temperature of vitreous ice (which is near 130 K). Further work remains to determine if a more simple and ergonomic load-lock entry into the LEAP would be possible while without condensing potential contaminants on the specimen. This would remove the requirement for the Fast Pump Down Dock and the dedicated pumping system.

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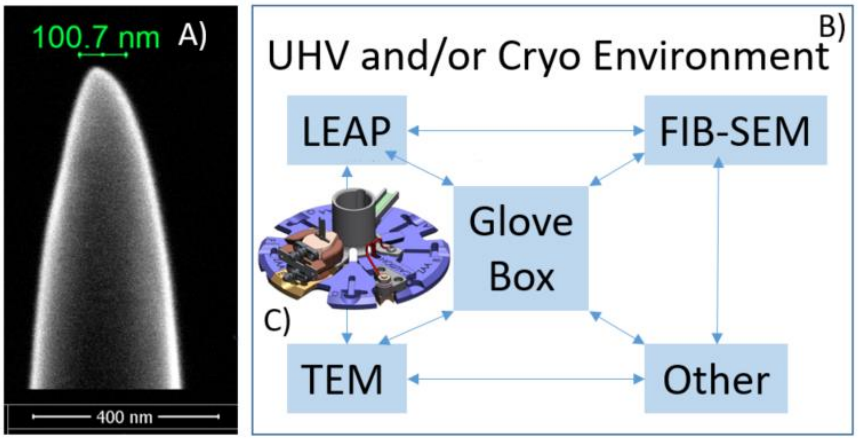

Figure 1. A) The typical needle geometry of an APT specimen, B) The desired flexibility of a cryogenic transfer system with a specially designed specimen carrier (inset).

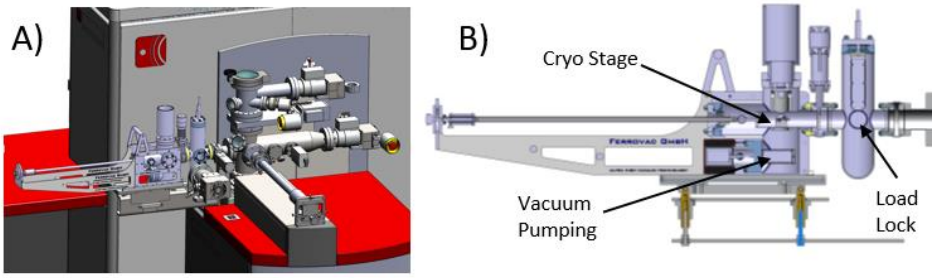

Figure 2. A) A solid model of a LEAP 5000 with the UHV Cryogenic Suitcase from Ferrovac attached. B) The Suitecase cross sectioned to show the cryogenically cooled stage, integrated pump, and fast pump down load lock.

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