Modular UHV cryogenic protocols for environmentally-sensitive atom probe

Stephenson, L.[1], Mouton, I.[1], Chang, Y.[1], Szczepaniak, A.[1], Vogel, D.[1], Tezins, U.[1], Sturm, A.[1], Kelly, T.[2], Raabe, D.[1] and Gault, B.[1]

[1] Max-Planck-Institut für Eisenforschung, Germany, [2] Cameca Instruments, United States

Atom probe is an essential characterization tool for chemically-sensitive investigations of a material's nanostructure, but its data is only valid after answering the challenges presented by preparing and preserving a desired state in an atom probe sample. This is important for all experiments, but some answering some materials science questions require significantly more isolation from the atmosphere or from room temperature. In collaboration with hardware suppliers, we have developed the necessary instrumentation and the UHV cryogenic transfer protocols.

Specimens are transferred using two Ferrovac VSN40S suitcases (now commercially available through Cameca) modified to accept standard Cameca atom probe pucks and can easily reach vacuums of 10^{-10} - 10^{-9} mbar and temperature of -184 °C with liquid N_2. Our two UV-laser-assisted atom probes (LEAP 5000XS/XR) are equipped with fast-docking suitcase stations (connection time ≈ 15-20 minutes) for direct transfers into the buffer chamber of the LEAP. Sample pucks are fitted with an insulating PEEK and a pre-cooled "piggyback" puck within the LEAP to ensure that the transfer from the suitcase into the analysis chamber is both rapid and with very limited undesired warming.

Three other platforms were or are being made to dock with the suitcases. Our xenon-plasma focused-ion beam microscope (FEI Helios) provides us with a unique capability for high-throughput APT sample preparation and, equipped with a solid-state cryo-cooled puck stage, the sample can be milled with less contamination and beam damage. For controlled processing of air-sensitive materials and hydrogen gas charging, we developed an environmental reaction chamber (Microscopy Improvements/Cameca) and separate glovebox (Sylatec).

The impact of these protocols will be discussed with reference to various projects at MPIE, including projects considering H/D-charging of various metals, hydrides for energy storage, preparation of hydrated samples and biological samples.

We wish to acknowledge the Max Planck Society and Deutsch Forschungsgemeinschaft for their ongoing funding support.