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Atom Probe Tomography with Extreme-Ultraviolet Light

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Abstract: We have adapted an atom probe tomograph (APT) with an extreme ultraviolet (EUV) source. The EUV photon energies open a direct photoionization pathway unavailable in conventional near-UV APT. © 2019 The Author(s) **OCIS codes:** (180.0180) Microscopy, (160.0160) Materials

Laser-assisted atom probe tomography (LAPT) is a powerful tool for materials characterization due to its combination of high spatial resolution and analytical sensitivity. In idealized terms, LAPT is a method that deconstructs a specimen one atom at a time, counts and identifies the individual atoms that comprised the specimen, and generates an atom-by-atom 3D "reconstruction" of the specimen [1-3].

Fig.1 shows a schematic representation of conventional LAPT. Briefly, a sample material is shaped into a sharp specimen "tip" with an apex radius ranging from 10-50 nm. The tip is then held at cryogenic temperatures (20-100 K) and electrically biased with a DC standing voltage (SV) under ultra-high vacuum conditions. The SV is carefully chosen to be below the threshold for field ion evaporation. Due to the high SV and the small radius at the tip's apex, the electric field strength at the vacuum/tip interface is in the order of tens of V/nm. In state-of-the-art conventional atom probe a pulsed, focused laser (typical wavelength 355 nm or 532 nm) is used to induce thermal transients that trigger field ion evaporation from the tip. These ions are then accelerated towards the detector where their times-of-flight (TOF) and impact positions are recorded. The identity of the ions can be inferred from the TOF data and their origination location can be extracted with back-projection algorithms. The accumulated dataset of TOF and origination position is then used to numerically generate a 3D "reconstruction" of the specimen.

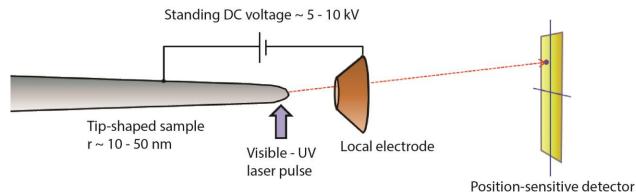


Fig. 1. Schematic representation of conventional atom probe tomography

The fact that field ion evaporation in LAPT is a thermally-triggered process is problematic as many materials are not uniformly heated by the near UV or visible pulsed laser and field evaporation may persist after laser excitation has passed. This results in "thermal tails" that increase background and can complicate identification and resolution of low-concentration species.

Here we present a different approach to laser-assisted APT, where instead of using a near-UV laser for inducing a thermal transient, we use an extreme-ultraviolet (EUV) coherent light source to photoionize the atoms at the tip's apex. In our apparatus we use a commercial Ti:Sapphire laser system and EUV source to generate 30-nm (42 eV) light through the high-harmonic generation process. The EUV photons are then steered and focused in a custom-built vacuum beamline until they reach a commercial APT chamber. The use of EUV photons, with an energy range of 10 - 100 eV, for APT opens the potential for an athermal field ionization pathway. To our knowledge, this process has not been previously observed, but it has been anticipated and described in a recent patent [4,5].

Fig. 2 presents some of our initial TOF mass spectra results on a fused silica (SiO₂) tip. The peak assignments were very similar to the ones obtained with conventional LAPT with the silicon peak (doubly ionized) appearing at

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14 daltons (Da). The peaks at 16 Da were assigned to single ionized atomic oxygen. Other peaks present in the spectrum are O_2^+ (32 Da), SiO^+ (44 Da), and SiO_2^+ (60 Da). The peaks at 1 and 2 Da are typically assigned to H^+ and H_2^+ respectively.

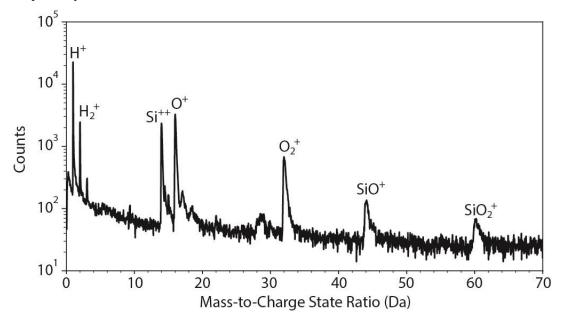


Fig. 2. Mass spectra for SiO₂ from EUV-assisted atom probe tomography.

Our results show a decrease in the peak height for some molecular clusters and a reduction of the thermal tails and widths of the mass spectra peaks when compared to LAPT. Additionally, the EUV-assisted APT measurements obtained the correct specimen composition, something that is not always possible in conventional LAPT [6].

We will also present EUV-APT on more technologically relevant semiconductor samples, such as gallium nitride (GaN). In general, our results show the same deductions meaning correct composition measurements, narrower peaks, and a reduced signal from molecular clusters.

In summary, we have adapted an atom probe tomograph with an ultrafast, EUV source in place of a pulsed, visible-UV laser. Initial results on fused silica and gallium nitride show that the EUV-equipped system will produce mass spectra with fewer complex ions, narrower peaks, and lower background when compared to conventional LAPT performed on the same samples. Our initial results suggest that the photoionization pathway provided by the EUV light, may be superior to the strictly thermal mechanism that is generally ascribed to field evaporation of ions in conventional LAPT.

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