Understanding field evaporation sequences by in-situ correlative microscopy and simulation

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Background

Atom Probe Tomography (APT) provides detailed information on a material's microstructure and its chemical composition. This technique enables access to the 3D position and chemical nature of each atom in a sample. The process involves evaporating the atoms using an electric field and reconstructing a 3D model of a needle-shaped specimen. However, the resolution of APT can be degraded for heterogeneous materials by artifacts potentially associated with the reconstruction methods. Furthermore, the geometric model of the hemisphere on a truncated cone used for APT reconstructions relies on assumptions about variations in the tip curvature along the evaporation sequences. These fluctuations depend on variations in the evaporation field, which are governed by the distribution of areas of higher or lower evaporation field in the sample. This is for instance illustrated in Figure 1.

Tracking the shape of the APT tip during evaporation can provide valuable information about the tip radius, which can help improve the reconstruction algorithm, mapping the electric field around the tip can also indicate areas of higher or lower electric field. In-situ correlative electron microscopy, achieved by performing APT experiments in a STEM can consequently help understanding the evaporation sequences.

Methods

4D-STEM [1] experiments were carried out on a polarized Al-Fe APT tip subjected to 1 kV. A CheeTah Timepix3 pixelated detector was used to record a diffraction pattern for each pixel as the tip was scanned. To mitigate dynamic diffraction effects, a precession angle of 0.7 degrees was used. Data analysis was performed using the Libertem [2] software. For simulating the field evaporation process, we utilized the Robin-Rolland [3] algorithm that calculates the electric charge on each atom and predicts evaporation events by identifying atoms that reach a critical evaporation field threshold.

Results

Our experiments showed variations in electrostatic fields around a polarized Al-Fe tip, subjected to a voltage of 1 kV. This variation is shown in Figure 2. The analysis shows a constant electrostatic field inside the tip, indicated by the blue color. A high field was observed near the apex of the tip, indicating a high probability of evaporation, confirmed by the results of simulation, represented in Figure 3.

Conclusion

In this study, we have developed an approach that makes it possible to follow the shape of an APT tip during evaporation, by performing APT experiments in a (S)TEM. In situ correlative microscopy was achieved by developing a sample holder capable of applying a voltage of up to 8 kV. In addition, 4D-STEM experiments made it possible to visualize the electrostatic field on a polarized APT tip. These results could help improve the accuracy and 3D reconstruction of atom probe data. The integration of numerical modeling can be used to predict material behavior during APT analysis and to confirm experimental results.

Graphic:

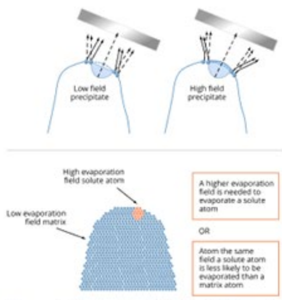

Figure 1: Illustration of effects of high and low field precipitate on the ion trajectories.

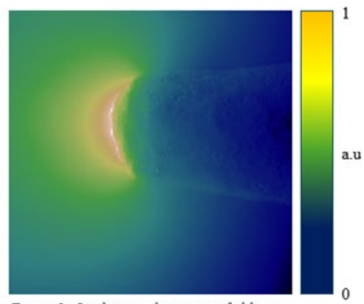

Figure 2: Qualitative electrostatic field map of a polarized Al-Fe sample, with an applied voltage of 1 kV.

Figure 3: Evaporation sequences of a simulated Al-Fe tip with a high field precipitate.

Keywords:

APT, (S)TEM, In-situ correlative microscopy

Reference:

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