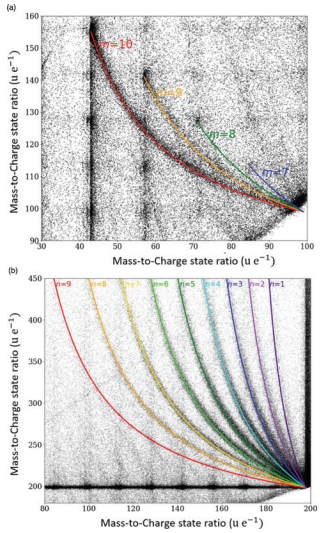
Highlights from Microscopy AND Microanalysis

Biological Applications

Frozen *n*-Tetradecane Investigated by Cryo-Atom Probe Tomography Levels by K Meng, TM Schwarz, EM Weikum, P Stender, and G Schmitz, *Microsc Microanal* | https://doi.org/10.1017/S1431927621012541X.

Atom probe tomography (APT) is well established for metallic or semiconducting nanostructured systems and is unique due to its chemical and spatial analysis. In recent years, there have been several efforts to take this analysis method a step further by using transfer shuttle systems to analyze soft matter materials. However, very limited knowledge is available on the evaporation and fragmentation behavior of frozen liquids in APT. Biological and organic soft materials are primarily hydrocarbon chains. In this work, we investigated the fundamental evaporation and fragmentation behavior of simple alkane chains (*n*-tetradecane). Based on multi-hit events and the following correlation plots, more detailed information about the evaporation behavior and the decay of molecules into smaller fragments in the region near the tip were studied. Multiple different dissociation tracks, of the whole *n*-tetradecane molecule in its excited state and the subsequent decay into either two singly charged molecules or one charged and one neutral molecule, could be observed (Figure), and the dissociation zone in the low field region could be calculated. Based on the intensity distribution along the dissociation track, most of the decay occurs into similarly sized fragments after a potential drop of 70%, corresponding to the 60 µm distance of the dissociation zone from the tip surface.

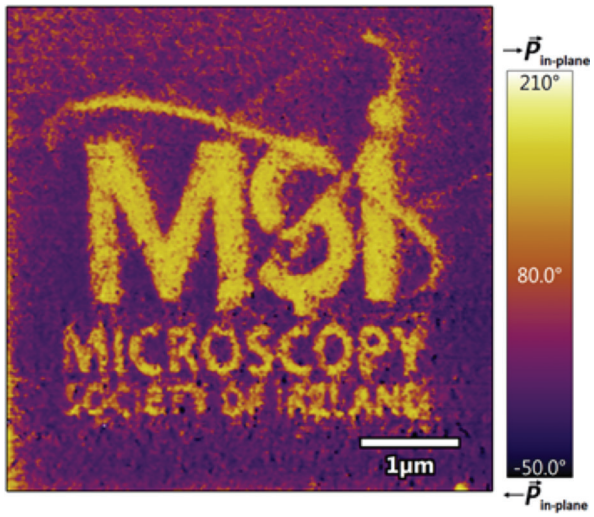


Areas of the correlation plot with periodically ordered dissociation tracks. In (a), the dissociation of the starting molecule $C_{14}H_{30}^{2+}$ into $CH_3(CH_2)_m^+$ and $CH_3(CH_2)_{12-m}^+$ with $m = 7–10$. In (b), the neutral dissociation of the origin tetradecane molecule $C_{14}H_{30}^+$ into neutral species of $CH_3(CH_2)_{n-1}CH_3$ and a charged species of $(CH_2)_{13-n}^+$ with $n = 1–9$.

Materials Applications

Probing Ferroelectric Behavior in Sub-10 nm Bismuth-Rich Aurivillius Films by Piezoresponse Force Microscopy by L Keeney, L Colfer, and M Schmidt, *Microsc Microanal* | https://doi.org/10.1017/S1431927621013726.

Sub-10 nm ferroelectric and multi-ferroic materials are attracting increased scientific and technological interest, owing to their exciting physical phenomena and prospects in miniaturized electronic devices, neuromorphic computing, and ultra-compact data storage. The $Bi_6Ti_{2.9}Fe_{1.5}Mn_{0.6}O_{18}$ (B6TFMO) Aurivillius system is a rare example of a multi-ferroic that operates at room temperature. Since the formation of impurity phases can lead to microstructural resistance to ferroelectric domain wall motion and thus limit its use, herein we use 49% bismuth excess to counteract bismuth volatility during sub-10 nm growth and reduce the volume fraction of secondary phase impurities from 2.95–3.97 vol.% down to 0.02–0.31 vol.%. Piezoresponse force microscopy (PFM) enables local ferroelectric investigations of 8 nm B6TFMO and reveals that the orientation of the ferroelectric polarization can be switched. Arrays can be "written" and "read" to express states permitting anti-parallel information storage (Figure). In-plane ferroelectric switching in ultra-thin B6TFMO demonstrates its potential for use in technologically competitive data storage devices based on in-plane tunnel junctions, which would not be hindered by competing depolarization fields upon scaling down to ultra-thin dimensions.



In-plane PFM lithography image of 8 nm B6TFMO on $SrTiO_3$(100) where the PFM tip was instructed to perform ferroelectric patterning of the MSI (Microscopy Society of Ireland) logo using negative (–7 V_{DC}) and positive (+7 V_{DC}) biases.

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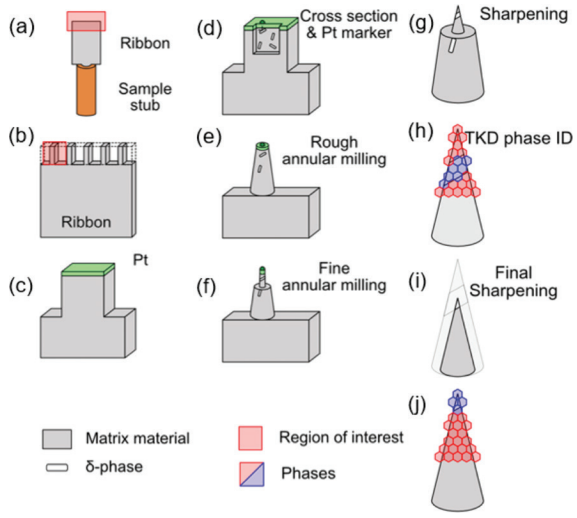
Techniques

Correlative Approach for Atom Probe Sample Preparation of Interfaces Using Plasma Focused Ion Beam Without Lift-Out
by VV Rielli, F Theska, and S Primig, *Microsc Microanal* | https://doi.org/10.1017/S1431927621000349.

Site-specific atom probe tomography (APT) requires sample preparation with nanofabrication techniques such as focused ion beam (FIB). Lift-out is the most common procedure, however, recent plasma FIBs (PFIB) enable higher milling rates and higher volume material removal. Here, we propose a site-specific technique where APT tips are PFIB-milled directly, without lift-out (Figure). In this method, after manufacturing posts on a thin ribbon-like sample, cross-section milling allows the visualization of the region of interest (ROI). Annular milling aligned with the ROI is performed, and transmission Kikuchi diffraction (TKD) facilitates phase identification and positioning the ROI close to the apex of the tip. We showcase this method for the scarcely explored interphase boundary between γ/δ in a Ni-based superalloy. In addition to avoiding artifacts, this method is faster and more reliable than common lift-out approaches, as the interface is precisely positioned along the tip length. It also generates stronger tips, less susceptible to fracture during the APT experiment, due to the absence of weak points such as Pt welds.



Schematics of the correlative approach for the preparation of APT tips via PFIB without lift-out. (a) Ribbon on a Cu sample stub; (b) posts on the top end of the ribbon after first material removal; (c) Pt deposition on top of a post; (d) cross-section milling for the visualization of δ-phase and pinpoint with a Pt marker; (e) rough annular milling; (f) fine annular milling; (g) sharpening of tip with γ(red)/δ(blue) interface; (h) identification of phases through TKD; (i) final sharpening; and (j) δ-phase on the apex of the tip.