Hydrogen Embrittlement Studies

APT Measurement of Hydrogen Traps with Room Temperature Transfer

High-strength steels are prone to hydrogen embrittlement. To mitigate this, microstructural features like carbides, which trap mobile H, can be introduced. To investigate these trapping sites, atom probe tomography (APT) offers near-atomic spatial resolution and high sensitivity for all elements. However, quantitative measurement of H is challenging since residual H_2 gas in the ultra-high vacuum chamber contributes to the mass-spectrum. A common work-around is the use of the heavier isotope deuterium, which is introduced into the sample via cathodic charging, gas charging or implantation. So far, the use of cryogenic transfer has been emphasized, to prevent H or D from diffusing and escaping the samples.

Jakob et al. [1] demonstrate in their work that cryogenic transfer is not necessarily needed if the binding of H at the trap sites is strong enough. They investigated the trapping character of nano-scale Cr-carbides in a co-precipitation steel. This alloy forms ß-NiAl particles and Cr-carbides during aging. The precipitates are finely distributed and hence offer abundant trapping sites as shown in Figure 1a.

Electrolytically polished specimens were measured with and without cathodic charging at -2.2V against a Pt counter-electrode in a solution of 0.1 M NaOH in D_2O. The specimens are moved right after charging through the load-lock and buffer chamber to the cooled sample stage in the analysis chamber, with room temperature handling of about 15 min. The measurement with a distinct peak at 2 Da in the mass-spectrum (Figure 1b) is evidence that D is retained even after a short period at room temperature. Furthermore, measurement parameters, namely pulse frequency, were varied to show the dependence of artificial H on the time between evaporation events in contrast to D, which is connected to the carbide content within the specimen. The increase of the D concentration near the Cr-carbides indicates that the interface is the main trapping site as shown by the proxigram profile in Figure 2.

The successful observation of H/D trapping within the alloy's microstructure without cryogenic transfer paves the way for more efficient and feasible experiments. This investigation not only provides insights into hydrogen behavior within materials but also offers a more practical approach for researchers to study hydrogen trapping mechanisms using APT without the logistical challenges of cryogenic handling.

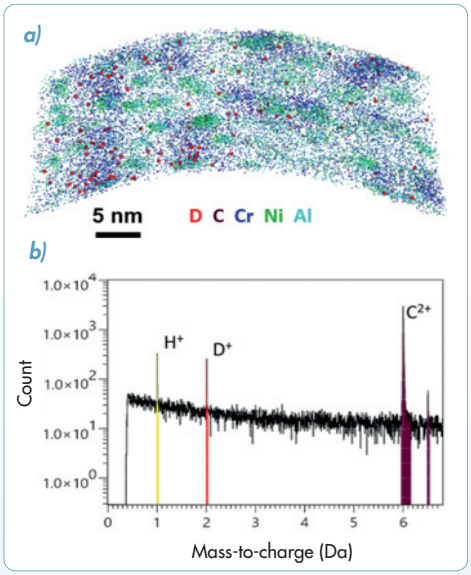


Figure 1:
a) 5 nm thin slice of the reconstruction of a D charged specimen showing precipitation forming elements as well as D;
b) Selected segment of the mass-spectrum showing the distinct D+ peak at 2 Da.

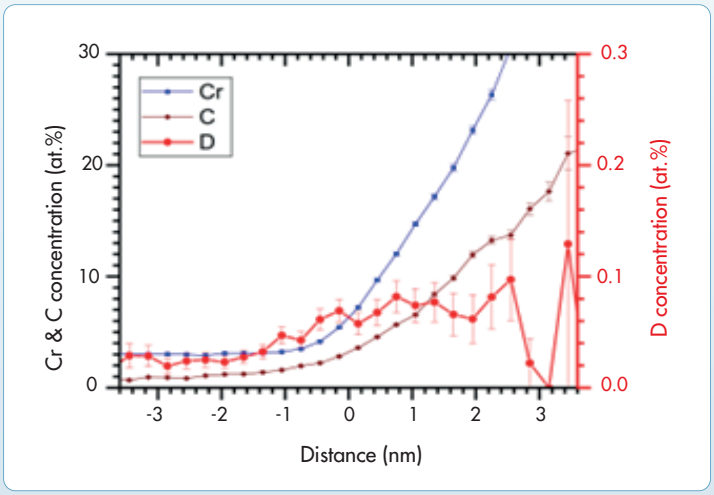


Figure 2:
Proxigram of 7 at.% Cr iso-concentration surfaces showing elevated D concentration in the vicinity of Cr-carbides.

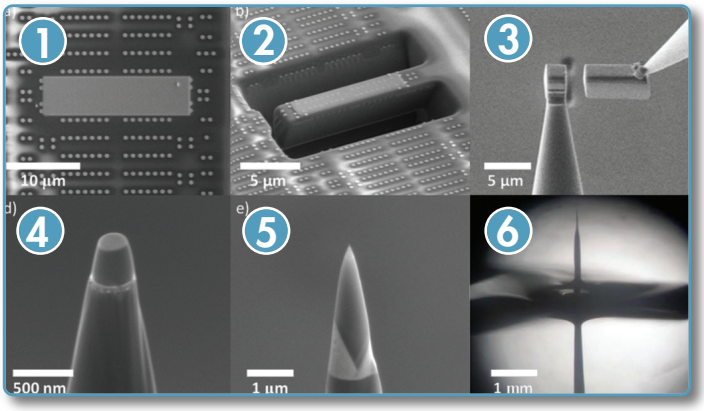
[1] *Jakob S. et al., Characterization of hydrogen traps in a co-precipitation steel investigated by atom probe experiments without cryogenic transfer, Scripta Mater. 243, 2024, 115963. https://doi.org/10.1016/j.scriptamat.2023.115963*

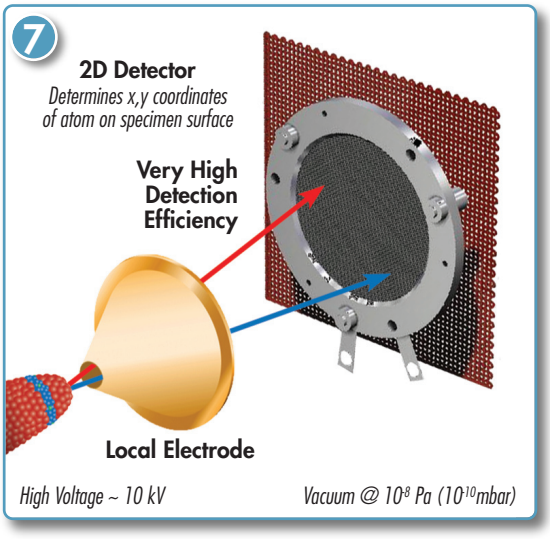
Three Steps to 3D Nanoscale Analysis

An Introduction to Atom Probe Tomography

Step 1: Specimen Preparation

An atom probe specimen usually has a nanoscale region of interest (ROI) requiring both 3D compositional imaging and analysis. The sample is formed into a needle shape containing the ROI. Common APT specimen preparation methods using electropolishing or a Focused Ion Beam system (FIB) are very similar to TEM methods except instead of forming a thin sheet, a needle shaped sample is desired. At the right, standard FIB liftout and mounting of a specimen (figures 1 through 3) and then sharpening the sample with the ROI left at the very apex (4 and 5). In 6, a wire geometry sample is being electropolished.





Step 2: Data Collection

An atom probe produces images by field evaporating atoms from a needle-shaped specimen and projecting the resultant ions onto a detector 7.

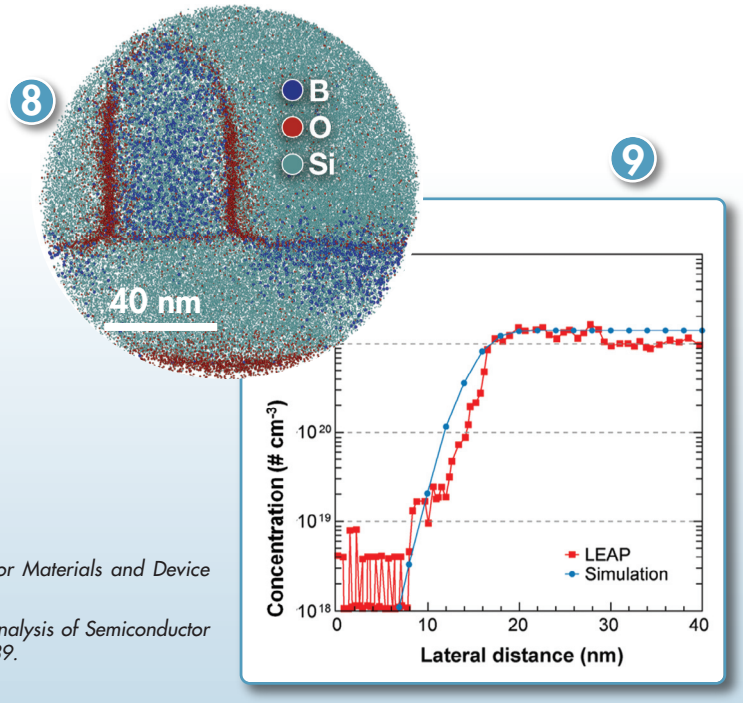
A high magnification results from the ~ 80nm tip being projected onto an 80mm detector resulting in a magnification of approximately 10^6.

An atom probe identifies atoms by their mass-to-charge-state ratio *(m/n)* using time-of-flight mass spectrometry. Charge state, *n*, is typically 1 to 3.

The specimen is held at approximately 50K to reduce surface diffusion during the experiment. The high electric field results in 100% ionization and the high speed detector is capable of measuring up to 80% of the collected ions, independent of ion mass.

Step 3: Data Visualization and Analysis

Examples of data output are illustrated by a slice of a 3D atom map of a transistor† 8, and a dopant composition profile‡ 9. The image shows the positions of individual atoms (oxygen is red and boron is blue) in the transistor with subnanometer resolution. From the reconstructed data set many types of useful analyses are possible. These include 3D visualization, 2D atom mapping 8, 1D depth profiling and line scanning 9, as well as mass spectra and compositional analysis from user-selected volumes.



† *Lauhon, L. J. et al, MRS Bulletin "Atom Probe Tomography of Semiconductor Materials and Device Structures" 34(10) (2009) 738.*

‡ *Moore, J. S.; Jones, K. S.; Kennel, H.; Corcoran, S., Ultramicroscopy "3-D Analysis of Semiconductor Dopant Distributions in a Patterned Structure using LEAP" (2008), 108, 536–539.*

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