Nuclear Applications of Atom Probe Tomography

Nanoscale Features Providing Radiation Damage Resistance

Application Note #APT-04

The stability of nanostructured ferritic alloys (NFAs) under high temperature creep and high-dose radiation damage has been attributed to the trapping of vacancies at various microstructural features, thereby minimizing or even preventing solute diffusion. The distribution, chemistry, and number density of these nanoscale precipitates both in the interior of individual grains and those on the grain boundaries are key to understanding and improving the alloy's stability.

However, unambiguously detecting and characterizing solute clusters in solid solutions has been challenging using standard microscopies because they contain just a few atoms and because the are in a matrix of metal of similar composition. Even in modern LEAP systems, due to the relatively high levels of the solutes in the matrix and the low number of atoms in the smallest of these clusters (fewer than 10), this type of analysis is incredibly challenging. Detecting them or even determining the detection limit for such structures is difficult.

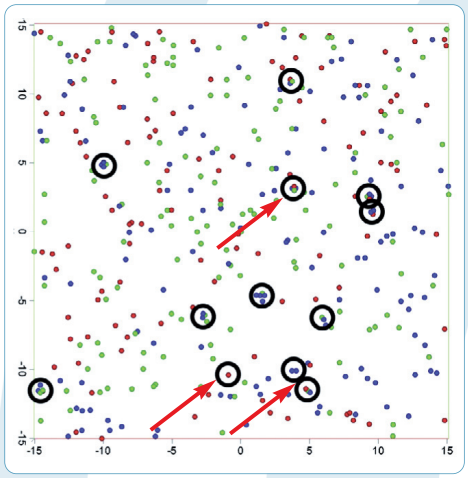
Simulated APT data with detection efficiencies (DE) of 40 and 80% (nominally those of the LEAP 4000X HR and LEAP 5000 XS, respectively) were generated for a 14YWT alloy containing small precipitates and solute clusters. The volumes generated contain clusters with a nominal radii of 0.25 and 0.5 nm (i.e., corresponding to solutes, and the size of the smallest nanocluster observed in the 14YWT alloy). As the same seed was used for the random number generator, the precipitates were located in the same positions in each simulated dataset. The simulations show that the 80% DE data found 90% of the small clusters, but the 40% DE found only 47% of them.

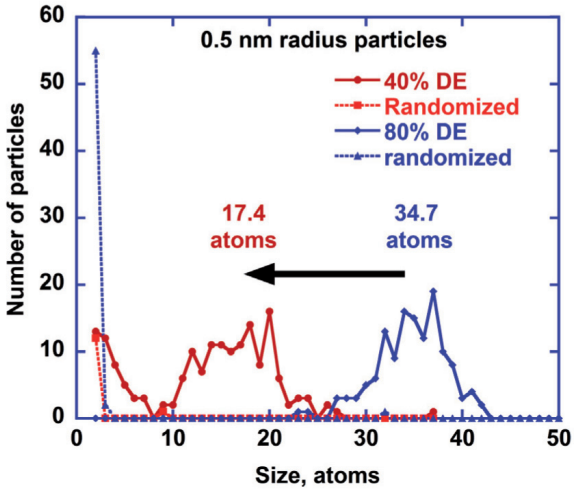
Friction stir welded samples of 14YWT were prepared for APT analysis using standard sample preparation. This alloy, even in the heat affected zone of the friction stir weld, is expected to have the defect trapping nanoscale features which give the alloy its high resistance to embrittlement, even in high temperature, high radiation environments. The samples were then analyzed using a LEAP 5000 XS system.

Solute clusters were found at a number density of 2.2 ± 1.1 x 10^{24} m-3 and contained between 2 and 9 Ti, Y, and O atoms. Knowledge that the nanoscale clusters that strengthen the alloy are still present after manufacturing steps like welding are important to understanding what the performance of the material will be in challenging environments.

Simulated APT datasets from standard and advanced detection systems with up to 80% detection efficiency demonstrate the advantages of analysis of alloys like 14YWT with very small features.

Comparing simulations of data collected with 80% efficiency versus 40% efficiency, 33% more clusters are found with the higher efficiency data.





This plot demonstrates that with lower detection efficiency (here 40% versus 80%), the measured particle distribution moves to lower sizes and overlaps the random solute clusters. With a higher detection efficiency, one can choose friends of friends cluster search parameters that can uniquely find the real precipitates while ignoring the randomly clustered atoms.

Adapted from M.K. Miller et al. Detection and quantification of solute clusters in a nanostructured ferritic alloy. Journal of Nuclear Materials (2015).

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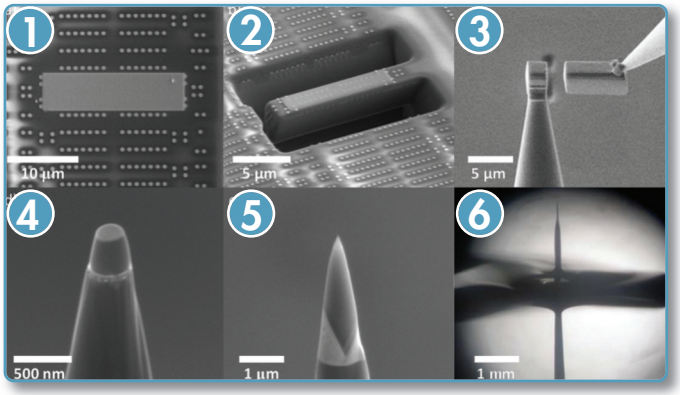
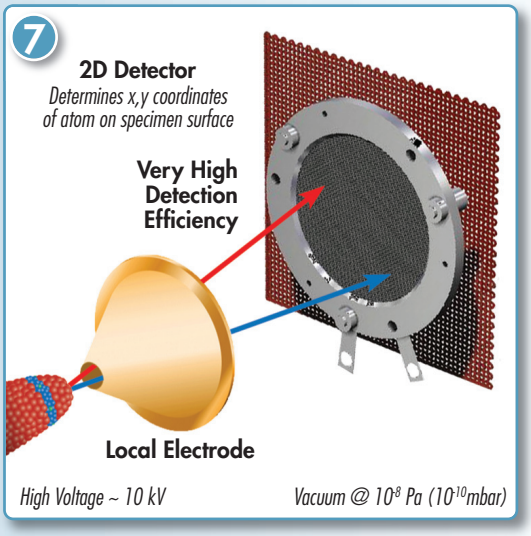
Three Steps to 3D Nanoscale Analysis
An Introduction to Atom Probe Tomography

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.

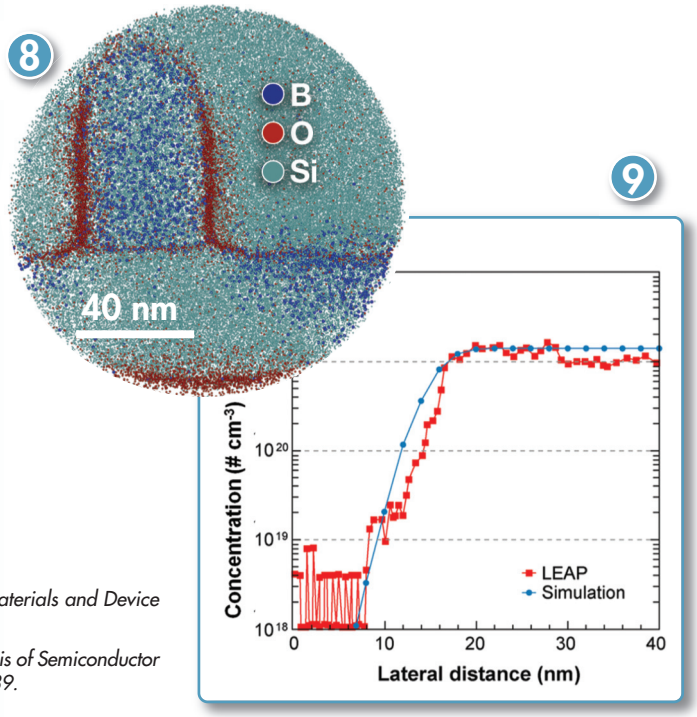
A high magnification results from the ~ 80nm tip being projected onto an 80mm detector resulting in a magnification of approximately 10^6.

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.*