Nanocluster Analysis
APT for Nuclear Structural Materials

Application Note #APT-14

Nanoscale elemental data obtained by atom probe tomography (APT) can help understand the chemical changes that materials undergo when exposed to irradiation. Understanding the nanoscale changes can enable alloy modification that can suppress the ductile to brittle transition in high radiation environments. CAMECA's Integrated Visualization and Analysis software (IVAS™) has built-in cluster search tools to help users analyze atom probe data and can provide quantitative cluster search analysis.

The maximum separation method is one of the most commonly applied algorithms for cluster identification [1]. This method is based on the separation distance between solute atoms to their nearest neighbors. The algorithm defines clustered atoms if they are separated by less than a critical distance of d_{max}, and contain more than a threshold of solute atoms (N_{min}).

Prior to irradiation, commercial purity 304 stainless steel exhibits a uniform solid solution. Cu clusters, Ni_3Si precipitation, and P segregation to dislocations defects are commonly seen features as irradiation-induced structural modification is responsible for hardening and embrittlement. Figure 1 is a nearest neighbor distribution of Cu atoms of this alloy after receiving a proton irradiation dose of 10 displacements per atom [2]. The data clearly exhibits two populations which consists of two Gaussian distributions from Cu atoms in the clusters and the matrix, respectively. The proper d_{max} was determined as the crossing point of the two distributions. The minimum cluster size, N_{min} , is subsequently determined using a cluster size distribution.

Figure 2 shows a Cu ion map and algorithmically defined clusters. The quantitative cluster analysis result shows Cu clusters are estimated to be 1.5 ± 0.2 nm in size based on the number of Cu atoms in clusters in conjunction with the atomic volume and density of Cu, 1.3 ± 0.2 x10^{24} in number density and 0.11 ± 0.01% in volume fraction. This information makes the prediction of mechanical properties as a function of irradiation damage possible, and can help in the development of nuclear materials that retain their strength under irradiation damage and thus not only extend the lifetime of nuclear plants but also improve their safety.

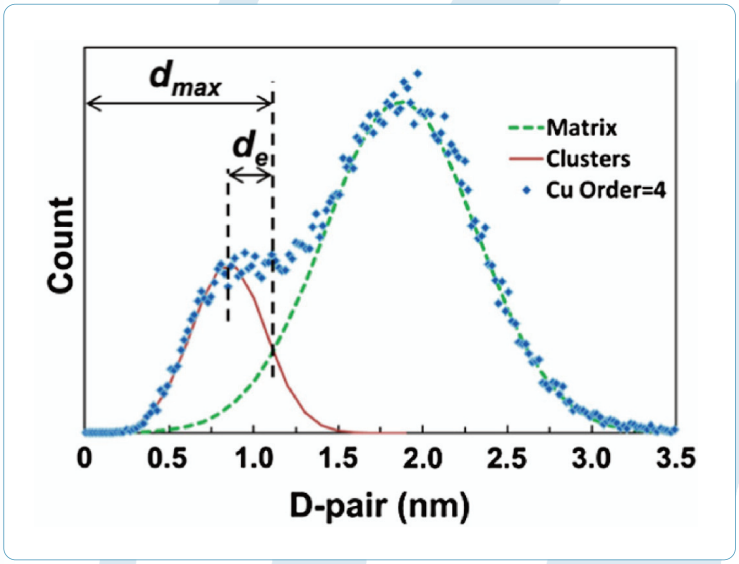
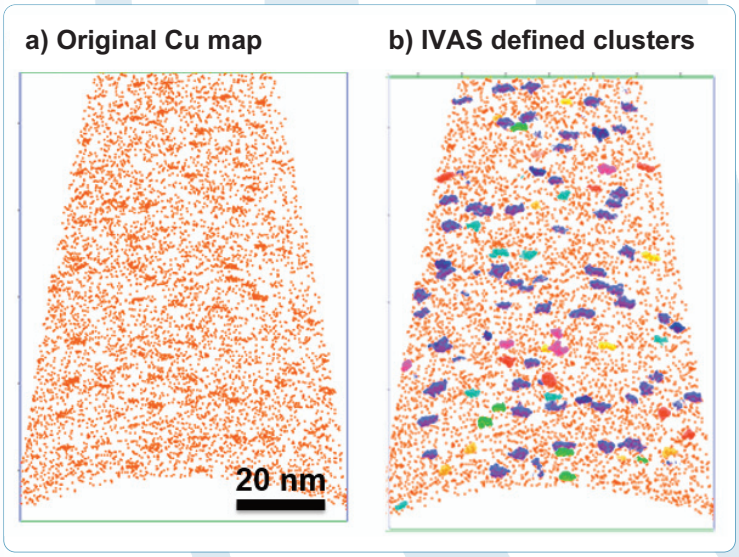


Figure 1
Cu-Cu nearest neighbor distribution with Gaussian peak deconvolution overlay.

Figure 2
a) 5 nm slice atom distribution map showing high density Cu clusters introduced by proton irradiation.
b) Defined clusters using the Maximum Separation Algorithm that is implemented in IVAS.



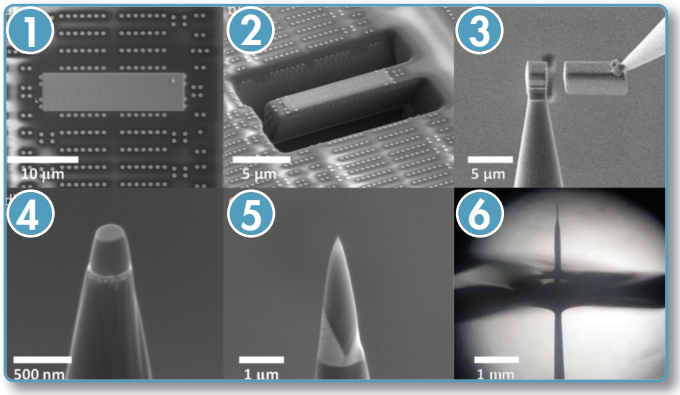
[1] Hyde, J. M. et al. "Microstructual processes in irradiated materials." MRS 2000 Fall Meeting Symposium, pp. 27-29. 2001.

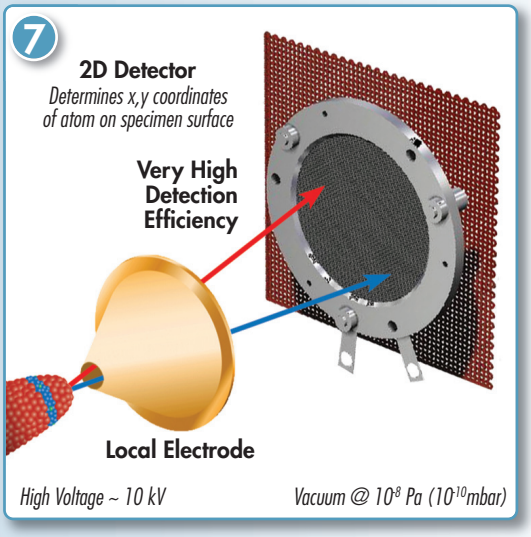
[2] Chen, Yimeng et al. "Quantitative atom probe tomography characterization of microstructures in a proton irradiated 304 stainless steel." Journal of Nuclear Materials 451, no. 1-3 (2014): 130-136.

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.

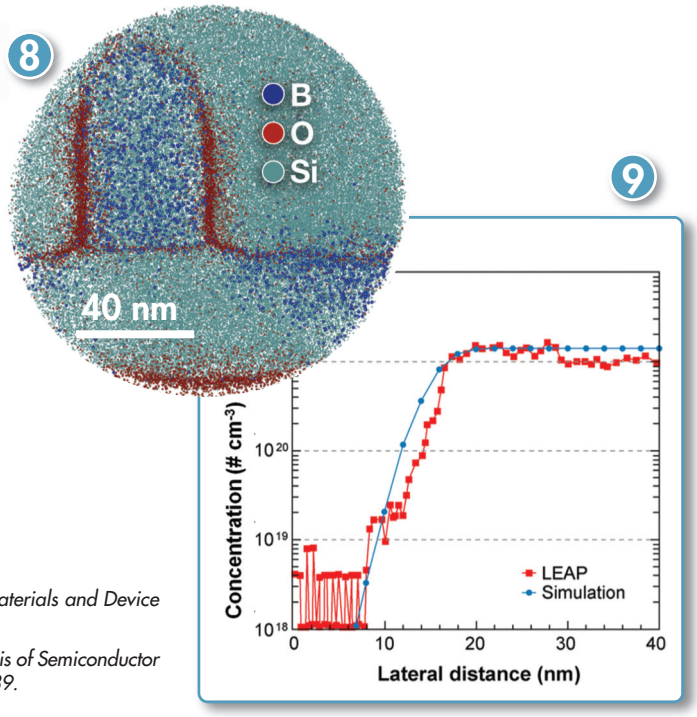
A high magnification results from the ~ 80nm tip being projected onto an 80mm detector resulting in a magnification of approximately 10^6.

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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