Expanding Atom Probe Tomography Applications

LEAP® Analysis of Dielectric Nanoparticles

Application Note #APT-08

Understanding the role of nanostructures in highly engineered, advanced materials like rare-earth doped silica glass fibers is key to understanding and improving their performance. Fiber optics are essential for telecommunications and medical applications, but the role of the doped dielectric nanoparticles (DNPs) in these optical materials is far from optimized due to the lack of information about their composition and growth mechanisms.

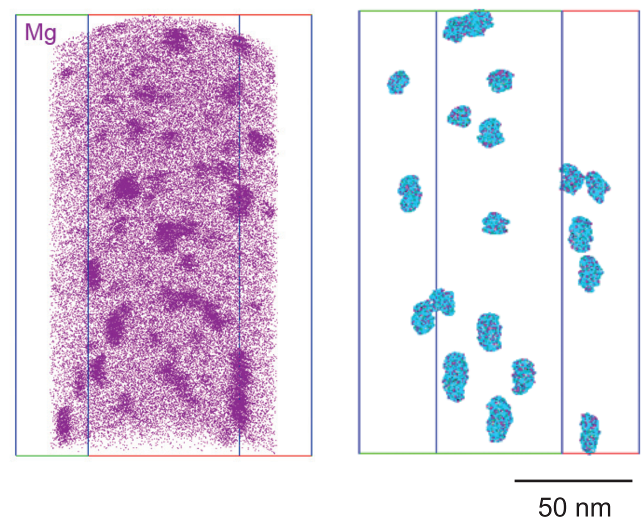
Local Electrode Atom Probe (LEAP) has recently been used in conjunction with other microscopies to further understand the size, composition, as well as growth and density of the DNPs. TEM and Electron Probe Micro Analysis were used to locate the center of the fiber core, where the nanoparticles were formed. These regions were then analyzed in laser pulsed mode with a LEAP system. Samples were prepared with a Focused Ion Beam SEM using standard APT liftout techniques similar to those used in TEM sample preparation.

Although the TEM was able to show the presence and general shape of DNPs ranging from 5nm to 200nm, atom probe tomography is the only technique able to determine the 3D particle density, composition, and intermixing with the matrix. While researchers and scientists still heavily rely on simulated data from nucleation and growth theories to tailor their materials processing steps, recent LEAP analyses paved the way for a better understanding of the fiber optics spectroscopic properties. Chemical composition variations in sub-10nm DNPs have led to the observation of a time plateau at the early stage of nucleation followed by an increase of the concentration as a function of the DNPs size.

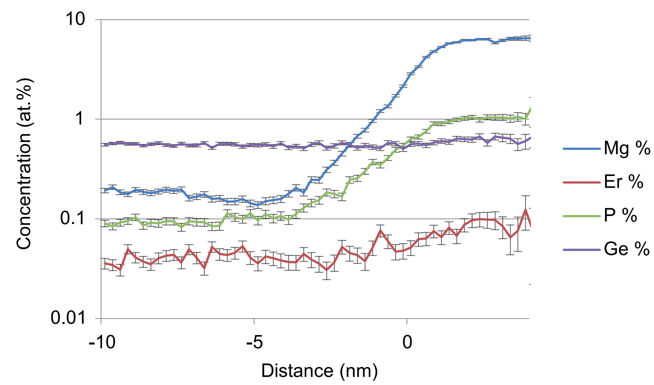
These experimental results provided by the 3D subnanometer elemental resolution of APT shed light on the fundamental science dominated by the classical nucleation theory model, and serve as a more reliable guide for the design of luminescent ion-doped materials.

Examples of advanced materials studied with APT:

- Light & superalloys
- Bulk metallic glasses
- High entropy alloys
- Nuclear materials
- Carbon-based materials
- Oxides and ceramics
- Magnetic materials
- Energy capture & storage
- Advanced glasses
- Biominerals
- Geochemistry
- Compound semiconductors
- Optoelectronics



DNPs revealed in fiber core using 3 at% Mg isosurfaces. Correlation between POx and Mg mappings in agreement with NanoSIMS, EPMA and TEM results.



Proximity histograms can be used to get either particle specific, or averaged concentration profiles measured from the particle edge. This analysis shows a 100-fold uptake in Mg and a 10-fold uptake in P in the Er rich particle.

Adapted from H. Francois-Saint-Cyr et al., Correlative Compositional Analysis of Fiber Optic Nanoparticles, microsc. Microanal. 20 (3), 2014

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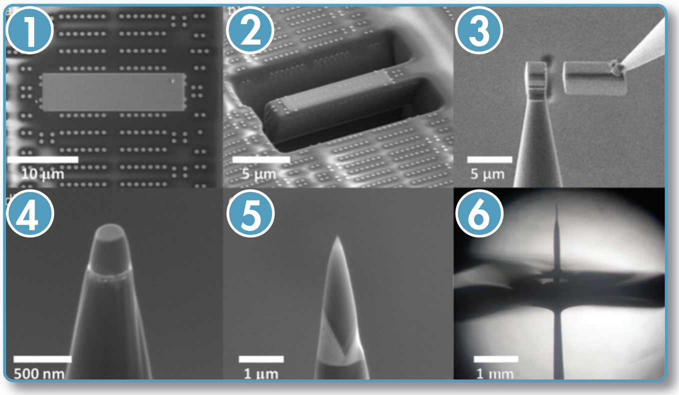
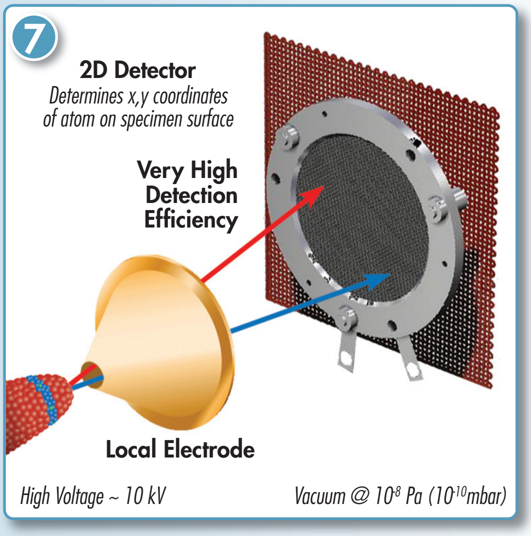
AMETEK® MATERIALS ANALYSIS DIVISION

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.





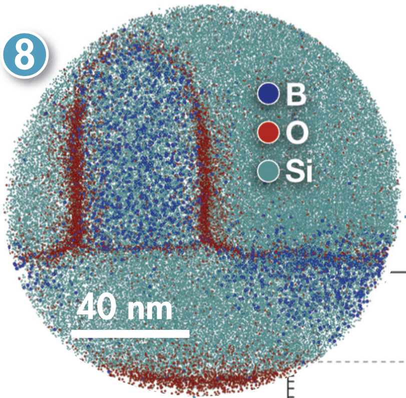
Step 2: Data Collection

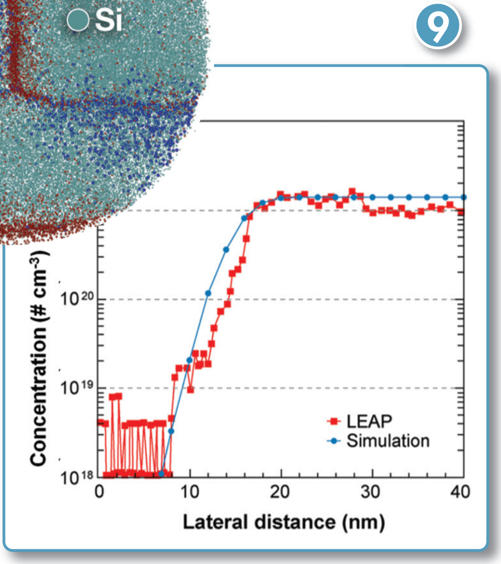
An atom probe produces images by field evaporating atoms from a needle-shaped specimen and projecting the resultant ions onto a detector 7.

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.*