

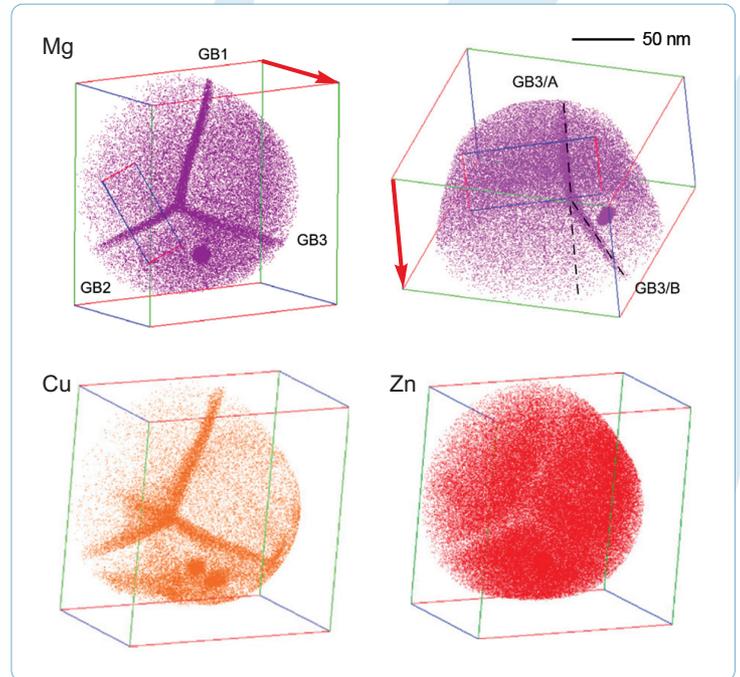
A better understanding of the structure and chemistry of grain boundaries is essential for developing a thorough knowledge of materials and their performance. However, due to the challenges in subnanometer 3D quantification, little research has been conducted to reveal the precise chemical information at grain boundaries and the role this chemistry plays in the response to processing conditions in ultra fine grained light alloys.

Attaining detailed information on grain boundary chemistry has long been a very challenging task for material scientists. Fortunately, the use of a three-dimensional atom probe provides unique information, which is sufficiently powerful to yield quantitative 3D subnanometer chemical information in and around the grain boundaries allowing a window into understanding how segregation from processing conditions affects material performance.

The samples were processed by equal channel angular pressing (ECAP) in the as-received condition at a temperature of 473K for different numbers of passes. Characterization by APT was performed in a local electrode atom probe (LEAP).

Results showed that Mg and Cu segregate strongly at grain boundaries. The relative intensities of the Mg and Cu segregation levels vary from boundary to boundary. Some grain boundaries have a higher Mg excess than Cu excess, but others were observed with higher Cu excess. The increase in disorientation angle of GBs in 4-pass ECAP samples appears to have links with higher level segregation of Mg. However, there is no clear dependence on Cu segregation levels, demonstrating the complexity of segregation of multiple solute elements at GBs. The mean excesses of Mg and Cu measured from 4-pass ECAP samples are lower than the values measured from grain boundaries of a sample processed by 8-pass ECAP. This suggests that the increase in the number passes of ECAP processing at 200 C increases the solute segregation levels. The increasing number of ECAP passes has been found to lead to further refinement of grains in the alloy.

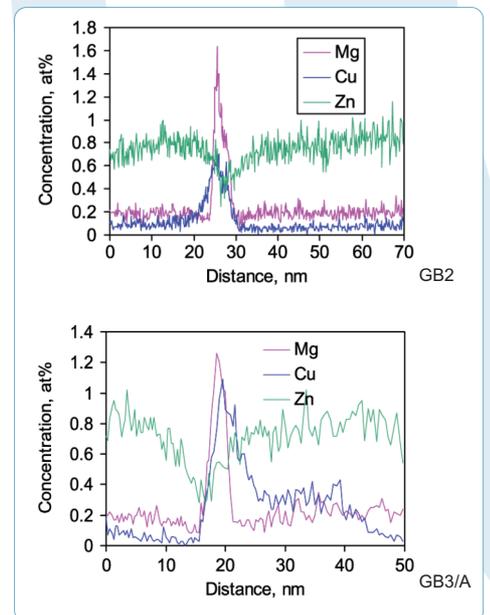
Continued advances in alloy processing can be made faster and more accurately with the understanding of these effects on 3D solute distribution. Atom Probe Tomography is ideally suited for this analysis.



Top: Rotated views of the same 3D Mg maps from the aluminum alloy demonstrating the 3D nature of APT Data. Selected regions were chosen across GB1 and GB2 and the concentration profiles are shown below.

Bottom: Cu and Zn maps with the same view of the top left Mg map.

Concentration profiles across GB1 and GB2 (noted above/top) demonstrate the differences in segregation of Mg, Cu and Zn across the grain boundaries. Additionally, trace levels of Cr, Mn, and Ti were observed, but were randomly distributed throughout the entire analyzed volume.



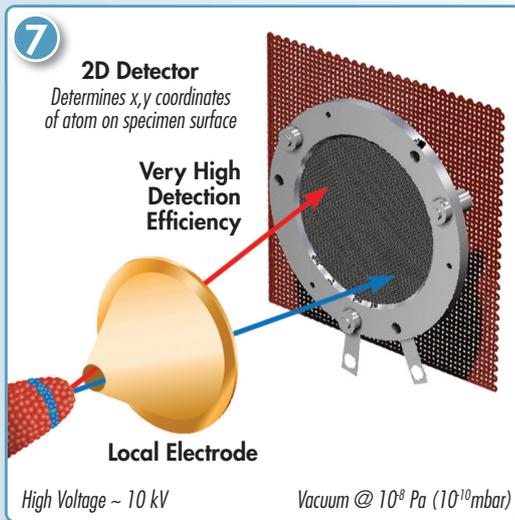
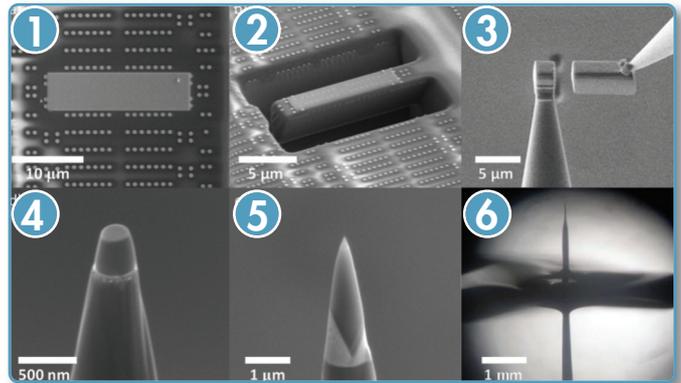
Adapted from G. Sha et al. Segregation of solute elements at grain boundaries in an ultrafine grained Al-Zn-Mg-Cu alloy' Ultramicroscopy, 2011, 111:500-5.

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.

