Atom-Probe Tomography – Different Analysis Tools for Three-Dimensional Atomic-Resolution Data

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Introduction

Like no other microscopy technique, atom-probe tomography (APT) requires detailed data analysis algorithms specific to the knowledge desired, as the data are both complex due to their three-dimensional nature and can only be collected in a digital format. With recent increases in speed and field of view available in contemporary instruments like the Imago Scientific Instruments LEAP™ microscopes, these challenges and significant benefits are exacerbated.[1] In practice, 'data collection' in APT, as understood in complementary techniques like scanning electron microscopy (SEM) or transmission electron microscopy (TEM), does not even begin until after the atom-probe experiment is over and the microscopist leaves the laboratory. The sample is prepared into the appropriate needle-shaped geometry, field evaporated atom by atom, and the 'experiment' part of the specimen analysis is over as soon as the ions are detected and stored in a digital file. Previous articles have addressed sample preparation and atom-probe operation and these topics will not be addressed here.[2,3] Once a three-dimensional image of the samples is recreated digitally, data collection to get the desired knowledge can actually begin. This article will review three data analysis techniques used to identify and quantify features of interest in an ion-implanted silicon wafer; radial distribution functions, average one-dimensional concentration profiles, and isoconcentration surfaces.

Samples

The samples were created by ion implantation of 50 keV arsenic to 2×10^{15} atoms/cm^2 into <100> silicon. The samples were coated with poly silicon and then received different heat treatments. The first sample (Type A) was annealed at 600°C for 30 minutes, the second sample (Type B) had an additional spike anneal at 1000°C for 30 seconds. The introduction of dopants via ion implantation usually results in the creation of undesired defects. During thermal treatments required to activate the dopants, typical in processing during the creation of computer chips, the defects can and grow into larger structures. The two anneal conditions demonstrate how a higher temperature spike-anneal can result in more complex defect geometries that could affect semiconductor device performance. The morphologies of these defects and their growth during annealing have been discussed in more detail separately.[4] Samples were prepared in an FEI NOVA™ dual-beam FIB and the data were collected on a LEAP 3000X Si™ system in laser-pulsed mode. The data were reconstructed with Imago's IVAS™ visualization and analysis software. The three-dimensional reconstructions of samples analyzed from each condition are shown in Figure 1. The silicon, oxygen and arsenic atoms are displayed, but only a small fraction of each is displayed for clarity.

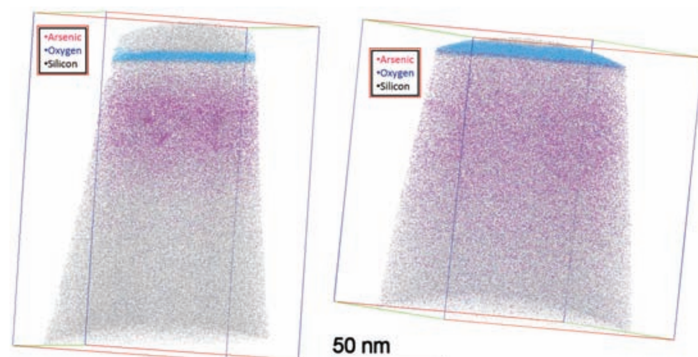

Figure 1: 3D reconstructions (atom maps) of samples analyzed from Type A [left] and Type B [right] anneals. Atoms are displayed as dots, Si[grey], As[Purple], and O[blue]. Only a small fraction of Silicon ions are displayed for clarity. Gross movement of the As dopants due to the high-temperature spike anneal both closer to the original silicon surface (decorated by the native oxide) and deeper into the silicon is clear.

Radial Distribution Analysis

The most basic analysis that can be done on three-dimensional (3D) data is a radial distribution function (RDF). The RDF for 3D atomic data determines the average local concentration variations for a species atom by atom. For example, atoms that do not cluster relative to other atoms in a material would have a flat concentration gradient on average away from the atom type of interest. This technique is extremely sensitive to clustering as it provides an average local radial concentration profile of all of the atoms of a particular type in the dataset. If implanted dopants tended to cluster, the average concentration would peak at the average center and drop off to the bulk concentration at the characteristic distance of the clustering. For the species of interest in this study, the implanted arsenic, it is clear from this simple radially symmetric analysis that the dopants cluster in both the type A and type B samples and that the characteristic distance and intensity of the clustering changes during the second spike anneal as shown in Figure 2. Although this is a calculation that is averaged over the entire volume of the sample and is insensitive to 3D variations, it is a calculation that can be used as a process monitor, or as a screening function to decide if more detailed analysis is required.

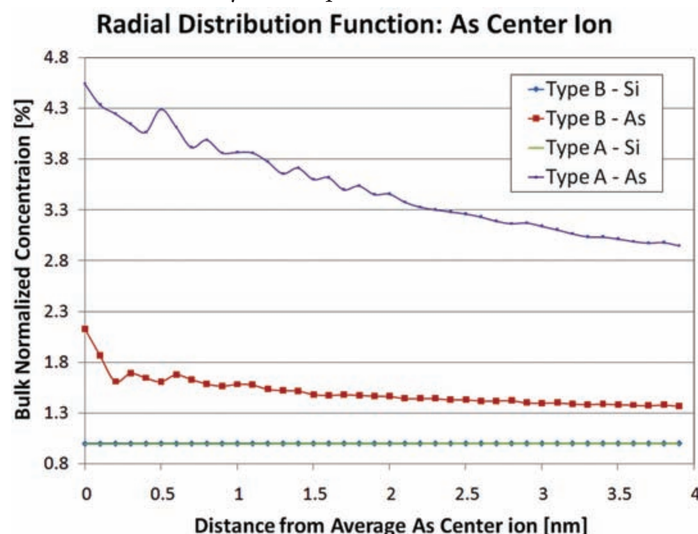

Figure 2: RDF plots of Type A and Type B samples showing the clear difference the spike anneal has on arsenic clustering in Type B. Ions that do not tend to cluster (like the silicon matrix in this case) have a flat bulk normalized concentration profile near 1.0.

One-Dimensional Concentration Analysis

The most straightforward analysis and perhaps the most used analysis with APT data is a one-dimensional (1D) concentration

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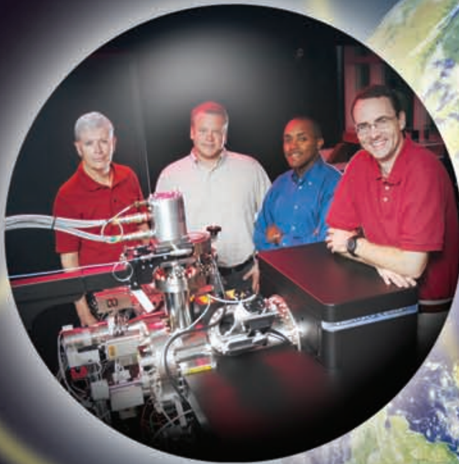

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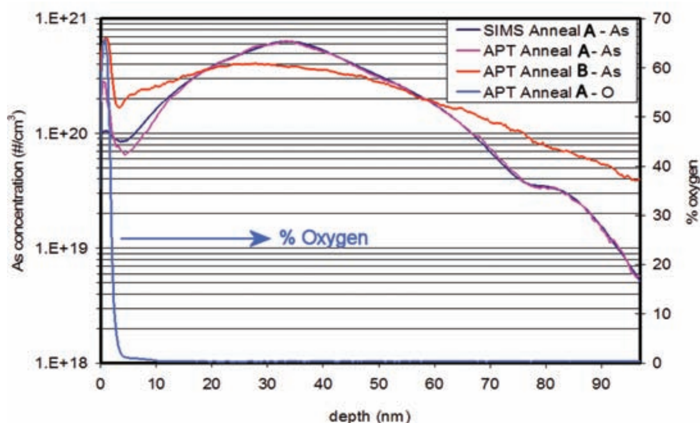

Figure 3: 1D APT concentration profiles from Type A and Type B anneals as compared to SIMS 1D SIMS profile.

analysis. In the simplest implementation, a 1D concentration profile can be calculated for the whole dataset from top to bottom. The result is similar to a secondary ion mass spectroscopy (SIMS) profile, but with higher spatial resolution. Figure 3 shows the comparison of a SIMS profile of As and O for sample type A, and the comparison APT data. In APT data, a region of interest, often right-circular cylindrical in geometry, can be arbitrarily chosen and the concentration as a function of distance can be calculated from one end to the other, averaged over the cross section of the cylinder. In Figure 4 sample type A, nanometer-scale local variation in the As concentration is visible from the atom map. A five nanometer diameter and 60 nm long cylindrical region of interest was chosen through the region of the dataset with the visible concentration variation. The concentration of As was calculated along the cylinder. Figure 4 shows the strong variation in As concentration from less than 0.5 at% to as high as 4 at% through the selected cylindrical region of interest. One-dimensional analysis can of course be calculated with an arbitrarily shaped region of interest, and can even be calculated with respect to a surface defined by an isoconcentration or isodensity level. This implementation of 1D concentration data is called a proximity histogram and is a technique that is very sensitive to detecting segregation to a phase or grain boundary.

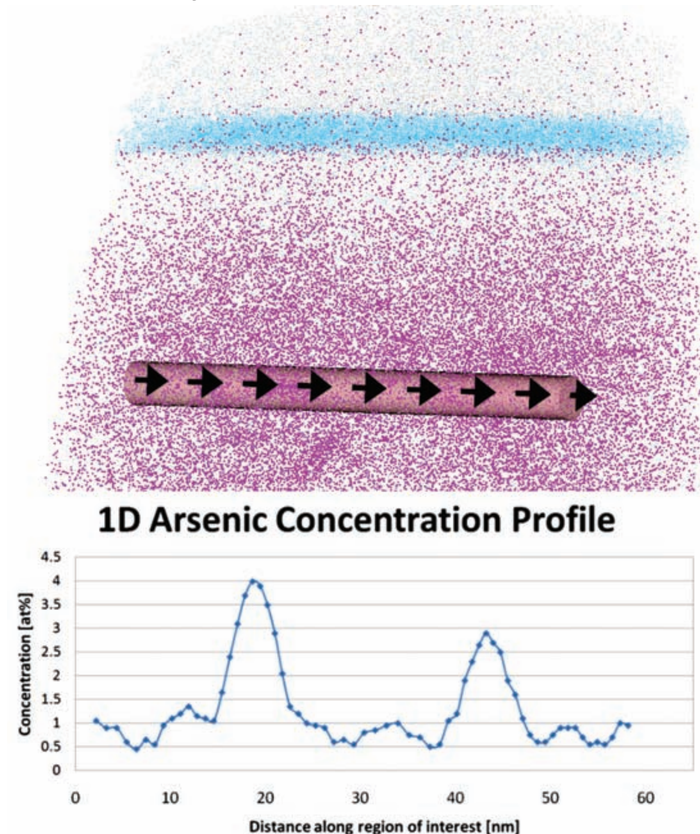

Figure 4: Lateral variation of As concentration as measured in the selected-region-of-interest cylinder in the 3D atom map.

Isoconcentration surfaces

One of the most powerful aspects of data analysis from atom-probe tomography is the ability to identify and analyze 3D nanoscale buried interfaces like grain boundaries and phase boundaries. These boundaries can be defined by 3D, element-specific concentrations or density variations, and can be used to calculate surface areas, enclosed volume, surface roughness, segregation levels, geometry and more. They can also be used to highlight subtle 3D features that might otherwise be missed. The surfaces are calculated by determining local concentrations on a 3D grid. Various grid parameters can be used to maximize spatial or compositional sensitivity. Of course, as this is atomic-scale data, the smaller the volume pixel, or voxel, that is used, the lower the statistical relevance. For example, a typical 1 nm^3 volume of a crystalline material will have between 30 and 100 atoms. In order to make a concentration calculation on a smaller voxel size there is a significant trade-off on statistical certainty inherent in the small volume analyzed. Typically, the concentration data are smoothed by a 3D Gaussian function to provide more statistical relevance and higher sensitivity for features on the distance scale of interest. The downside of spatial smoothing is that sharp spatial features become duller. Figure 5 shows surfaces defined by 10 at% oxygen and by ~3 at% arsenic for both the type A and type B samples. The clustering of the arsenic becomes very evident, and the incredible differences between the two samples caused by the 30-second spike anneal to 1000°C is particularly striking. The clustering, both spheroidal in type A and annular loop shaped in type B is due to a phenomenon known as a Cottrell Atmosphere Segregation where impurity atoms segregate to regions of high strain surrounding defects like dislocations, which results in a more stable, energetically favored, configuration. It is clear that the isoconcentration surfaces in Figure 5 highlight features not readily visible in Figure 1. Not only are isosurfaces able to highlight regions of interest in an appealing way, they give a quantitative way to measure the size of features in the 3D dataset. The observation of loop-shaped features in this analysis was not expected, but the application of isoconcentration surfaces immediately highlighted them. These features were subsequently observed and confirmed with TEM and EDS analysis[4].

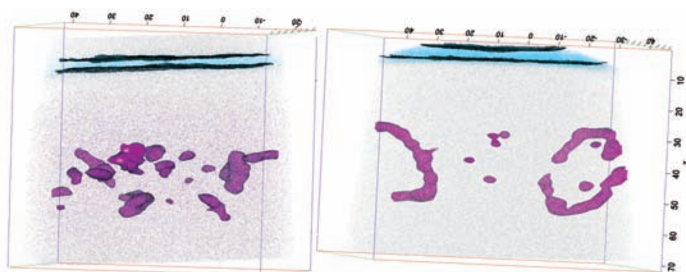

Figure 5: Oxygen[blue] and arsenic[purple] isoconcentration surfaces. Only a small fraction of Si[grey] and O[blue] atoms are shown for clarity. Clear differences in the type A anneal [left] and the type B anneal[right] are evident.

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Conclusion

A few of the many techniques used to quantify features in 3D atom-probe tomography data have been presented. The particular techniques used are specific to an application and desired information. The data analysis for APT is pursued away from the microscope itself and new analyses can be continued long after the experimental activities are complete due to the fully digital nature of the 3D atomic-scale data. New analysis techniques continue to be developed as the APT application space expands beyond the metallic alloys studied historically. Applications to materials with low electrical conductivity have recently become routine due to the introduction of a commercially available laser-assisted atom probe, such as the Imago LEAP 3000X Si.™ The type of analysis is limited only by the imagination of the microscopist and the computing power available. ■

Acknowledgments

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References

1. Kelly, T. F. and M. K. Miller (2007). "Invited Review Article: Atom Probe Tomography." *Review of Scientific Instruments* 78: 031101-1 to 031101-20.
2. Thompson, K., D. J. Lawrence, et al. (2007). "In-Situ Site-Specific Specimen Preparation for Atom Probe Tomography." *Ultramicroscopy* 107: 131-139.
3. Alvis, R. and T. F. Kelly (2008). "Atom-Probe Microscopy LEAPs the Chasm to Mainstream Applications." *Microscopy Today* 16(5).
4. Thompson, K., P. L. Flaitz, et al. (2007). "Imaging of Arsenic Cottrell Atmospheres Around Silicon Defects by Three-Dimensional Atom Probe Tomography." *Science* 317: 1370-1374.

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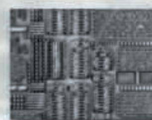

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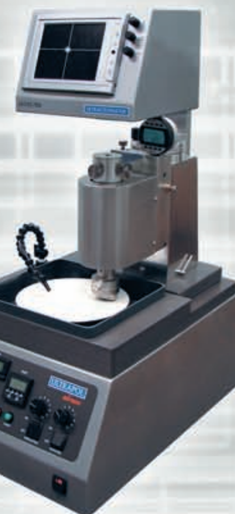

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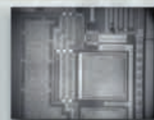

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