Interlaboratory Study: Laser-assisted Atom Probe Tomography (APT) of a Phosphorous-Doped Silicon Specimen

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Atom probe tomography (APT) is a powerful technique for the characterization of composition of materials and their three-dimensional structure down to the atomic-scale. As with any complex technique, the practice of APT is as important as the design of the instrument, and must be studied and standardized where necessary. Keeping in mind the ultimate goal of APT development, that of standards-less, absolute quantitative measurement of atomic-scale composition in three dimensions, we must as a field investigate and address a number of challenges: the large variety of materials systems and instrument configurations; the variations in sample preparation and the operating space of the instrument; and variations in reconstruction and analysis conditions.

A number of standards and standard-of-practice efforts have been made in recent years[1,2]; for this most recent effort, we chose to focus on a scientifically- and commercially-relevant semiconductor system, analyzed by laser-assisted APT, which would be used as a benchmark to compare the practice of APT at multiple different analysis facilities. To this end, an interlaboratory study of a straightforward, well-characterized Phosphorous-doped Silicon specimen has been performed, allowing us to determine the spread in measurements of a single well-characterized semiconductor sample analyzed by Atom Probe Tomography *as currently practiced* at world-class analysis facilities

Chemical Vapor Deposition (CVD)-grown epitaxial silicon doped with Phosphorous, deposited on a commercial silicon substrate, was chosen as the base material for this study. Following characterization of the base sample by Secondary-Ion Mass Spectrometry (SIMS) and Transmission Electron Microscopy (TEM) for composition and morphology, respectively, pieces of the material were sent to a number of APT-equipped facilities, including academic, commercial, and national laboratories. These facilities were asked to perform laser-assisted APT on the specimen, and provide raw data, details of sample preparation, and reconstructions and analyses, following their internal standard procedures. Datasets were then anonymized and collated for study.

Both raw data and the submitted analyses have been compared, particularly with regard to absolute concentration and composition profile of the Phosphorous dopant. Sample preparation, instrument operating conditions, and reconstruction parameters are correlated with results. The spread in results is discussed, and possible causes of variation and areas of potential improvement in the general standard-of-practice highlighted. We conclude that as currently practiced, APT does not consistently yield an accurate measurement of Phosphorous concentration in Silicon within the uncertainty of the measurement unless great care is taken in the selection of all tunable analysis conditions, making a condition-search experimental series a requirement for samples of unknown Phosphorous concentration and spatial distribution for the time being.

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References

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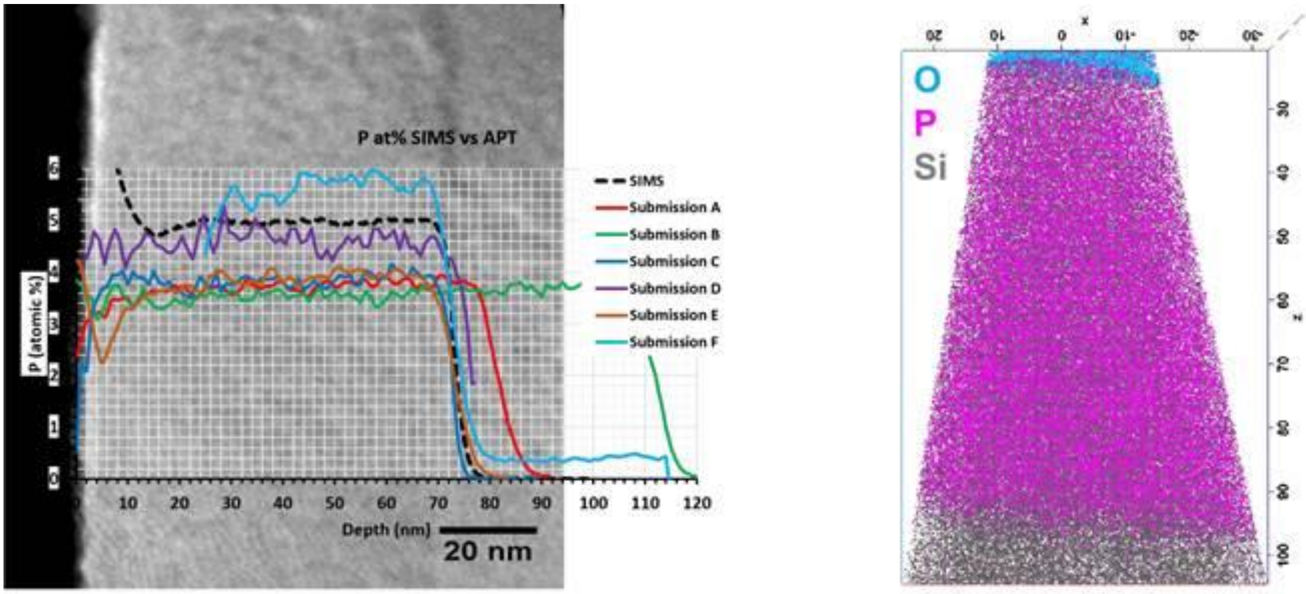

Figure 1. Results from the Interlaboratory Study, superimposed on specimen pre-characterization by BFTEM and SIMS. Left, BFTEM data with surface of specimen and dark Pt protective capping material at left, and interface between CVD-grown doped epi-Si and substrate visible at the right. Superimposed are P concentration profiles from SIMS and extracted from the received datasets, showing spread in simple 1D concentration. Right, a sample APT reconstruction showing O, P, and Si atoms with native oxide and epilayer-substrate clearly resolved.