

Dopant and Thin Film Analysis at Sub-Å Equivalent Thickness

We describe a new technique for non-destructive, quantitative measurements of dopants with equivalent thicknesses below 1 Angstroms, as well as high-fidelity measurements of thin films, using beam sizes of approximately 10-15 μ m, suitable for test pads smaller than 40 μ m.

This white paper will review semiconductor FEOL applications of the AttoMap XRF microscope.



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Dopant and Thin Film Analysis with the **AttoMap™ Series XRF Microscopes**

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Introduction: Requirements for rapid, non-destructive inspection of trace-level dopants and contamination are continuously driven by technological advances in electronics and materials. One example is modern 3D transistors (finFETs and emerging nanowire-based transistors), which are non-planar and can incorporate single-digit nanometer high-K dielectric insulators in place of SiO₂ gate oxides. These new 3D geometries, dimensions, and compositions introduce major challenges for existing measurement approaches¹⁻². This challenge is further compounded by the need to perform these complex measurements on ever-shrinking test structures (e.g., 40-50 µm pads).

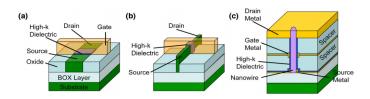


Figure 1: Semiconductor MOSFET designs: flat dielectric layers such as in traditional 2D designs (shown in a) are now moving to complex 3D structures in 3D FinFETs (b) and proposed vertical nanowire designs (c), resulting in new analytical challenges. A Moore and L Shi, "Emerging challenges and materials for thermal management of electronics." Materials Today 2014.

Current Mineralogical Approaches: SIMS and TEM

Secondary Ion Mass spectrometry (SIMS) has been a widely used analytical technique, in which a focused ion beam sputters the surface of a specimen, forming secondary ions that are analyzed for composition. However, new devices and materials introduce substantial challenges in its use, including quantification inaccuracies due to sputtering rate variations caused by factors such as non-planar structures¹ and impurities in high-k gate hafnium dielectrics³. Additionally, the acquisition times required for accurate analysis present a bottleneck, typically taking ~30 minutes per test pad point.

To address these problems, Transmission electron microscopy (TEM) is employed. TEM measures the transmission of electrons through a sample, and as a result, requires the preparation of an ultrathin lamella (<100 nm) for the region of interest. However, TEM is labor-intensive and low throughput,

and sample preparation can remove or destroy features of interest.

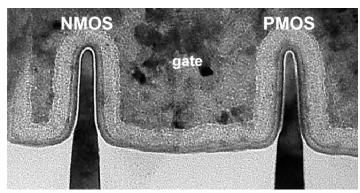


Figure 2: Current approaches to measure thin films are SIMS or TEM sectioning, both which are low-throughput and destructive. Shown above is a TEM image of a 16-nm finFET. D James, "Moore's Law Continues into the 1x-nm Era." 21st Itnl Conf on Ion Implanation Technology 2016.

Novel Approach with Sigray AttoMap XRF Microscope

Sigray has developed the AttoMap microXRF system using patented breakthroughs in x-ray source and x-ray optic technologies. The system achieves sensitivies orders of magnitude higher than electron-based techniques for elements ranging from metals such as hafnium (Hf) to organics such as boron (B) and carbon (C). Thin film and dopant sensitivities can reach down sub-Angstroms equivalent thicknesses, or $\sim 8 \times 10^{12} \, \text{atoms/cm}^2$ within a 10-15 µm spot size. The absolute concentration of high-K dielectrics with thickness of 1-2 nm can be measured within two minutes with 1% repeatability.

AttoMap features multiple innovations specifically developed for semiconductor use, including:

- A patented multi-target x-ray source that allows users to optimize fluorescence signals of interest and detect trace elements at the sub-ppm level
- 300mm wafer stage with detent mechanisms for wafer rotational positioning
- 3. Spot sizes **from 5 to 20 µm** suited for small test patterns
- 4. **Pattern recognition**: Integrated high resolution camera

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for automated test pattern recognition, enabling unsupervised recipe-based acquisition of points of interest in Sigray's software.

- Advanced software tools: A suite of tools, including an easy-to-use GUI interface, neural network peak fitting algorithms developed in collaboration with a major semiconductor manufacturer (trained on semiconductorspecific samples), and editable Jupyter python notebooks.
- 6. **Vacuum enclosure** in the AttoMap-310 for low-Z (e.g., B, C, O, N) dopant and contaminant analysis

In this applications note, we used an AttoMap-200 ambient system on third-party prepared samples of thin films on Si substrates to validate its capabilities and measure its lower limit of detection (LLD). Its multi-target x-ray source enabled the selection of different x-ray targets to optimize the x-ray fluorescence signal for various thin films of interest. Excellent linearity in signals (r^2 =0.9999) was established for Ni, Co, and HfO of 5, 10, and 20 Angstroms. As shown in the figures, the results for Co demostrate that, as clearly seen in Table 1, AttoMap achieved unprecedented lower limits of detection (LLDs) for Co of 0.03 Angstroms.

Thin Film	Source Target	Lower Limit of Detection with 99.7% Confidence
Со	Moly (k-a: 17.4 keV)	0.27 Angstroms
Со	Copper (k-a:8 keV)	0.03 Angstroms
Ni	Moly (k-a: 17.4 keV)	0.31 Angstroms

Table 1: Lower Limits of Detection (LLD) with 3-sigma Confidence at 400s showing excellent sensitivity far below 1 Angstrom. Moreover, as can be seen from the Co thin film rows (Rows 1 and 2), choice of **x-ray source target matters**: Co has a ~10X better LDL using a copper x-ray source target than a molybdenum target. This is why AttoMap's patented multitarget x-ray source is important.

Spectra of 5 Angstrom Co film with Peak Fitting

Figure 3: Co Thin Film Spectra: Spectra of 5 Angstrom thick Co (brown peak) peak fitted from background using Sigray's software.

Co Thickness (in Angstroms) vs Counts/Second

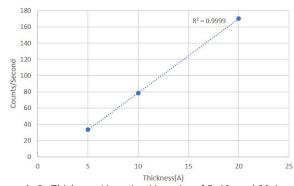


Figure 4: Co Thickness Linearity: Linearity of 5, 10, and 20 Angstrom Co films showing a r^2 linear regression of 0.9999. Counts/s shown are at a "flat" geometry; a 20X increase in counts/s can be achieved at higher angles.

Summary

Sigray's AttoMap provides a non-destructive, ultra-high sensitivity approach for quantifying thin film thicknesses and dopant concentrations down to sub-Å sensitivity for semiconductor applications. The performance is enabled by Sigray's patented high-brightness x-ray source and x-ray optics, which provide superior throughput, unprecedented sensitivity, and spot sizes suitable for small test pads.

- 1. Warlo, M., et al. (2019). Automated Quantitative Mineralogy Optimized for Simultaneous Detection of (Precious/Critical) Rare Metals and Base Metals in A Production-Focused Environment. Minerals, 9(7), 440.
- 2. Zhou, W., et al. (2006). Fundamentals of scanning electron microscopy (SEM). In Scanning Microscopy for Nanotechnology (pp. 1-40). Springer, New York, NY.
- 3. Prencipe, I., et al. (2015). Energy dispersive x-ray spectroscopy for nanostructured thin film density evaluation. Science and Technology of Advanced Materials, 16(2), 025007.
- 4. Nikonow, W. and Rammlmair, D. (2017). Automated mineralogy based on micro-energy-dispersive X-ray fluorescence microscopy (µ-EDXRF) [...]. Geoscientific Instrumentation, Methods and Data Systems, 6(2), 429-437.

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