Dopant and Thin Film Analysis at Sub-Å Equivalent Thickness

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ABSTRACT | We describe a new technique for non-destructive, quantitative measurements of dopants with equivalent thicknesses below 1 Angstrom and high-fidelity measurements of thin films, and with spatial resolutions of 10 μm. The need for such a system is driven by advances in materials science and semiconductor, such as 3D finFETs. Sigray's AttoMap MicroXRF provides ultra-high sensitivity at high throughput for measurements well below sub-angstrom concentrations for elements such as Hf, Ni, Co, and has demonstrated <1% repeatability for 1-2 nm thicknesses measured within 2 minutes.

INTRODUCTION

Fast, non-destructive, and quantitative compositional analysis of trace level dopants and nanostructures is a major capability demand among the semiconductor and nanotechnology research community. The need for such analysis is driven by technological advances in electronics and materials, such as modern 3D finFET transistors, which are non-planar and typically incorporate single-digit nanometer high-K dielectric insulators in place of SiO_2 gate oxides. These 3D new geometries, dimensions, and compositions introduce major challenges for existing measurement approaches[1-2], which must also achieve the required high performance on smaller test structures (e.g. 50 μm pads) of transistor arrays.

Current Approaches: SIMS and TEM

Secondary Ion Mass (SIMS) spectrometry has been the workhorse analytical technique, in which a focused ion beam sputters the surface of a specimen, forming secondary ions that are analyzed for composition. However, the advent of new devices and materials can introduce substantial challenges in its use, including quantification inaccuracies because of sputtering rate variations, which can be due to factors such as non-planar structures [1] and impurities in high-k gate hafnium dielectrics [3]. In addition, the acquisition times required for accurate analysis is a bottleneck, typically taking ~30 minutes per test pad point.

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

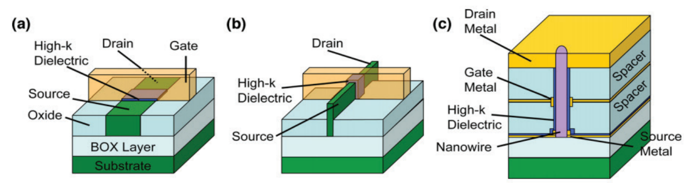

Figure 1. Semiconductor MOSFET designs: flat dielectric layers such as in traditional 2D designs (shown in a) are now moving to complex 3D structures in 3D FinFETs (b) and proposed vertical nanowire designs (c), resulting in new analytical challenges. A Moore and L Shi, "Emerging challenges and materials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

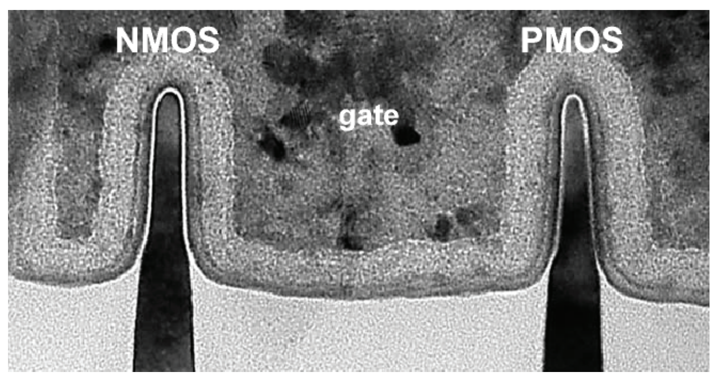

Figure 2. Current approaches to measure thin films are SIMS or TEM sectioning, both which are low-throughput and destructive. Shown above is a TEM image of a 16-nm finFET. D James, "Moore's Law Continues into the 1x-nm Era." 21st Itnl Conference on Ion Implanation Technology 2016.

A NOVEL APPROACH

Sigray AttoMap Patented MicroXRF System

Sigray, through patented breakthroughs in x-ray source and x-ray optic technologies, has developed the AttoMap microXRF system with sub-femtogram sensitivities. Relative concentration can be provided with a high degree of accuracy without standards, and absolute concentration of high-k dielectrics of **1-2 nm thicknesses have been measured within 2 minutes with 1% repeatability**. Moreover, the system is non-destructive, with high spatial resolution of 10 μm, making it ideal as a complementary upstream technique to SIMS and TEM for identifying regions of interest for follow-on characterization.

EXPERIMENT SUMMARY

The AttoMap was used on third party prepared samples of thin films on Si substrates to validate its capabilities and to 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 different thin films of interest. This capability is unique to the AttoMap and, as can be seen from Table 1 for Co, enables optimal lower limits of detection (LLDs).

Moreover, quantitative linearity was established with thin films (e.g. Ni, Co, HfO) of varying thicknesses: 5, 10, and 20 Angstroms.

SUMMARY

Sigray's AttoMap provides a non-destructive, ultra-high sensitivity approach for quantifying thin film thicknesses and dopant concentrations. Its patented high brightness x-ray source and x-ray optics enable excellent throughput and sensitivity, and moreover, due to its multi-target x-ray source design, has optimal performance for most elements-of-interest. The system can be used for single layers (as discussed) or even multiple elements and multi-layers (microXRF provides simultaneous detection of all elements).

Because of its high spatial resolution, the AttoMap can provide rapid (seconds to minutes) region-of-interest identification for follow-on analysis with complementary approaches such as SIMS and TEM.

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

Table 1. Lower Limits of Detection with 3-sigma Confidence at 400s: LDLs of well below sub-angstrom can be obtained with Sigray's AttoMap non-destructively. Moreover, as can be seen from the Co thin film rows, choice of x-ray source target matters: Cu has a ~10X better LDL than a Mo target. This is why AttoMap uses a patented multi-target x-ray source.

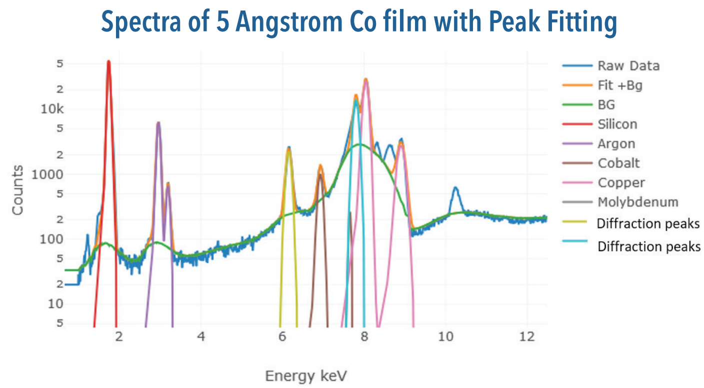

Figure 3. Co Thin Film Spectra: Spectra of 5 Angstrom thick Co (brown peak) peak fitted from background using Sigray's software.

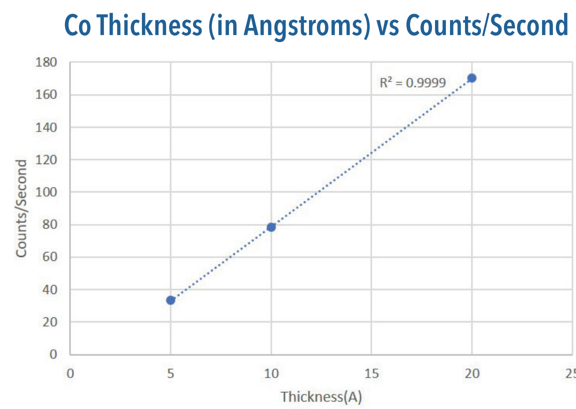

Figure 4. Co Thickness Linearity: Linearity of 5, 10, and 20 Angstrom Co films showing a r^2 linear regression of 0.9999. Counts/s shown are at a "flat" geometry; a 20X increase in counts/s can be achieved at higher angles.

References

1. AA Budrevich and W Vandervost. "Chapter 5: SIMS Analysis on the Transistor Scale: Probing Composition and Dopants in Nonplanar, Confined 3D Volumes," Metrology and Diagnostic Techniques for Nanoelectronics. Eds: Z Ma and DG Seiler (2017) Pan Stanford Publishing Pte. Ltd.
2. J Bennett, et al. "SIMS depth profiling of advanced gate dielectric materials," Applied Surface Science 203 (2003).
3. T Hasegawa, S Akahori. "High reliable quantification analysis of impurities in high-k gate dielectrics by SIMS," Special Issue on the Depth Profiling of Ultra Thin Films 28:11 (2007): 638-641.

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