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In-Line Compositional and Thickness Metrology Using XPS for Ultra-Thin Dielectric Films

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Abstract. 65 nm and 45 nm silicon devices will utilize compositionally critical processes for gate dielectrics, capacitor dielectrics, gate and capacitor electrodes, and ultra shallow junction layers. For example, small changes in nitrogen composition have been correlated with unacceptable shifts in electrical properties of devices with SiOxNy gate dielectrics. Present optically-based metrology technologies for such applications are reaching limits for precise thickness measurements and do not provide direct and adequately precise compositional information. As a result, mature analytical techniques, such as x-ray photoelectron spectroscopy (XPS), are now being transitioned to in-line production metrology usage.

We discuss the application of XPS optimized for 200/300 mm production to compositional and thickness metrology of SiOxNy and high k gate dielectrics, high k capacitor dielectrics, and new electrode materials. The development of optimized hardware, robust data analysis algorithms and high throughput, fully automated operation has led to production implementation of XPS in advanced logic applications. The precise correlation of plasma nitridation metrology data with electrical device parameters has proven valuable in detecting process drifts early in the process flow, without the need to prepare devices through the first metal layer for testing. High density maps of film thickness and composition have enabled optimization of oxidation, nitridation and post-nitridation anneal processes for SiOxNy film production for 90 nm, 65 nm and below. High precision compositional and thickness metrology data for high-k gate and capacitor dielectrics is also presented

Keywords: gate dielectrics, capacitor dielectrics, 300 mm, metrology

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INTRODUCTION

65 nm and 45 nm silicon devices will utilize compositionally critical processes for gate dielectrics, capacitor dielectrics, gate and capacitor electrodes, and ultra shallow junction layers. For example, at the 90nm node, small changes in nitrogen composition have been correlated with unacceptable shifts in electrical properties of devices with SiOxNy gate dielectrics, requiring the new use of compositional metrology to monitor nitrogen dose [1]. The precise correlation of plasma nitridation metrology data with electrical device parameters for SiON, an example of which is shown in Figure 1, has proven valuable in detecting process drifts early in the process flow, without the need to fabricate devices through the first metal layer for testing. High-k dielectric films are being implemented for memory capacitor applications, but the implementation of high-k gate dielectrics in advanced logic has proven an ongoing integration challenge and will be pushed to the 45nm node at least. Thus SiO_xN_y gate dielectrics likely will be widely used through the 45nm node, with possible changes in N concentration and distribution, raising further compositional metrology requirements.

Traditional optically-based metrology technologies for such applications are reaching limits for precise thickness measurements and do not provide direct and adequately precise compositional information. As a result, mature laboratory analytical techniques, such as x-ray photoelectron spectroscopy (XPS) and wavelength dispersive x-ray spectroscopy (WDS), are now being transitioned to in-line production metrology usage. XPS has emerged as the leading technology for ultra-thin

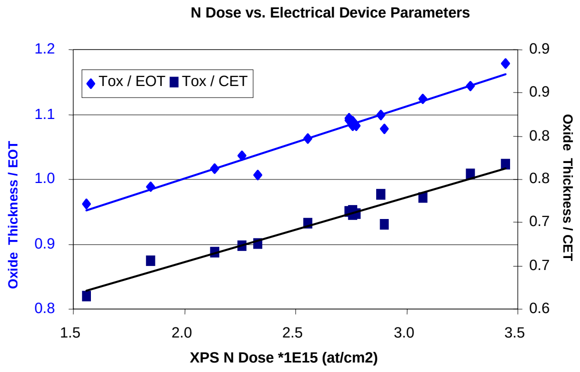


FIGURE 1. Correlation of SiO_xN_y gate dielectric electrical properties with N dose measured by XPS.

dielectrics, based on widespread use for 90nm and 65nm SiO_xN_y advanced logic metrology.

We will discuss the optimization required to transition XPS from laboratory analysis work to production metrology use. Examples of the value of XPS composition and thickness metrology data will be discussed for SiOxNy and high-k gate dielectrics, capacitor dielectrics, and related electrode materials.

OPTIMIZED XPS FOR SILICON DEVICE METROLOGY

XPS consists of x-rays going into the surface of a material, characteristic photoelectrons being ejected, and detection and analysis of photoelectron energy and signal intensity, as illustrated in Figure 2 [2]. Several aspects of XPS make it particularly well-suited for composition and thickness measurements for ultra-thin dielectric films. The escape depth of the photoelectrons ejected by the incident x-rays is limited to about 10nm, enabling XPS to provide accurate measurements for ultra-thin films down to a single monolayer [2]. Measurement of the photoelectron energy and intensity provides direct composition information. Further, the photoelectrons emerge from outer electron shells, providing chemical state information that allows identification of species among different molecules, for example, differentiating silicon among bulk Si, SiOx, and SiNx. Thus XPS can provide direct compositional information that optical techniques cannot. The spectral output of the photoelectron collection system provides for the detection of multiple elements in a single measurement without the need to change hardware, in contrast to WDS-based tools. This allows for identification of unknowns if needed, for example contaminants. Furthermore, XPS' relatively low energy incident x-rays do not cause the sample charging and film damage issues encountered for dielectric films with electron beam metrology technologies.

Traditional XPS tools are optimized for R&D laboratory applications, for which high energy resolution is key and repeatability of data and production worthiness are not design priorities [2]. A highly qualified tool expert operates the tool, collects spectra and interprets the results, a process which can take days to turn around. Long data collection times due to relatively low brightness x-rays, add to this. Further, lab XPS tools operate at ultra-high vacuum conditions, requiring long pump-downs after maintenance and also relatively expensive consumable components such as metal gaskets.

For production metrology, an XPS tool must be capable of repeatable, automated operation by non-experts such as fab operators, with very quick turn around for near-line or in-line operation. A fully automated algorithm approach to meet these requirements is illustrated in figure 3, for which data analysis is automated and desired output is precisely provided in real-time. Optimizations which can be implemented in the algorithms may include intensity extraction and materials parameters. Atomic compositions can be determined by extracted peak intensities, adjusted for appropriate atomic sensitivity factors. Use of intensity ratios and robust intensity extraction methods can enable high precision and throughput. Single and multilayer dose and thickness algorithms utilize attenuation theory and are relatively simple compared to optical tool algorithms, since XPS raw data contains high amounts of compositional and thickness information.

To achieve production metrology requirements of tight (<1%) precision, high reliability and good throughput, many hardware design optimizations can be made to XPS. Increasing the flux of the x-ray

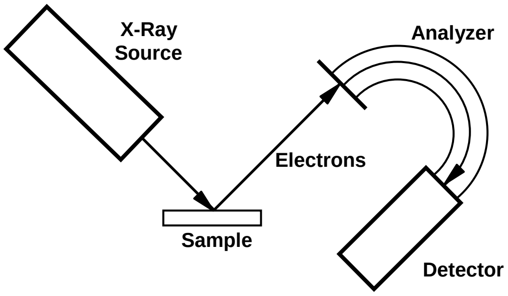


FIGURE 2. Basic XPS hardware schematic.

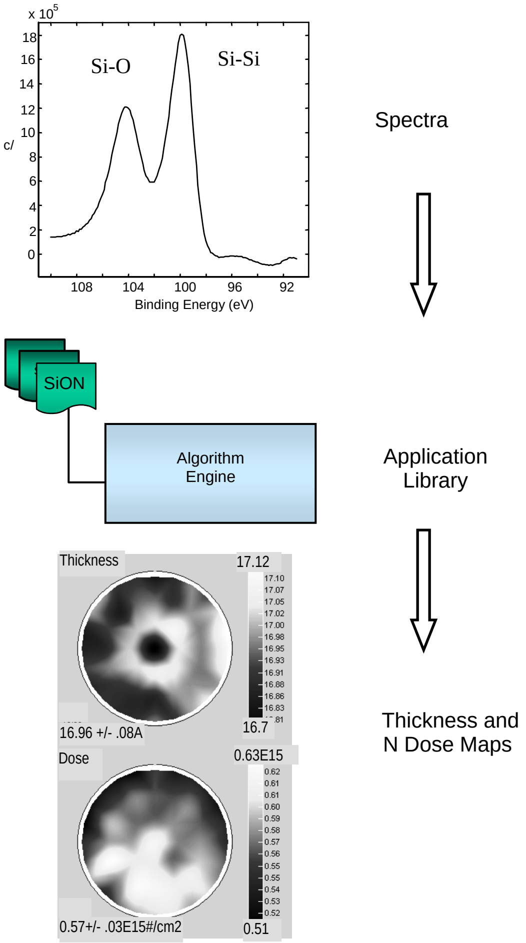


FIGURE 3. Fully automated XPS algorithm approach for the examples SiO_xN_y thickness and N dose.

source can increase the photoelectron signal, as can increasing the acceptance area for the analysis. The pass energy can be optimized for precision versus resolution. Using moderate high vacuum rather than UHV simplifies hardware and greatly reduces pump-down times. Eliminating the sample stage and using the same robot for both wafer transport and analysis eliminates wafer placement and simplifies hardware. Also eliminating unneeded R&D hardware, such as destructive ion sputter sources, simplifies the tool.

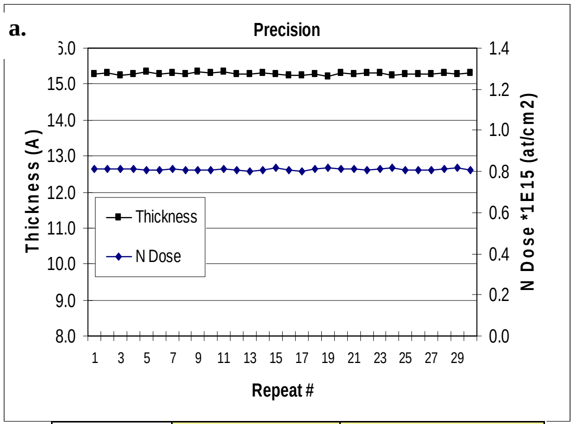
SiO_xN_y Gate Dielectrics

The maps in Figure 3 are 49 point maps on 300mm wafers. The SiO_xN_y thickness data in Figure 3 were obtained by measuring the attenuation of photoelectrons from the silicon substrate after they pass through the overlying SiO_xN_y film. As the SiO_xN_y film increases in thickness, fewer Si-Si photoelectrons make it through the film. By comparing the Si-O peak intensity to that of the Si-Si peak, the thickness of the film can be determined with a fully automated algorithm, as described above. The average thickness of the film in Figure 3 is 1.696nm with a uniformity of 0.008 nm. Dose is then determined using the nitrogen composition data, with an average value of 0.57E15 at/cm2 for the film in Figure 3. Note that the uniformity of the dose data (5.3%,1σ) in Figure 3 is significantly different from that of the thickness data (0.48%,1σ), a result which could not easily be observed with optical thickness measurements.

Such high density uniformity maps of film thickness and dose have enabled optimization of plasma nitridation and post-nitridation anneal processes and in-line metrology for SiOxNy film production for 90nm and 65nm. The strong correlation of XPS-measured N dose and thickness with equivalent oxide thickness (EOT), as illustrated in Figure 1, has enabled tight SiOxNy process control in production. Precision (repeatability) data for low N dose SiOxNy dose measurements are shown in Figure 4. Precision near 0.5% RSD are possible for low N dose, with an average 0.810E15 at/cm2, and near 0.2% RSD for thickness. Such precision capability can provide tight process monitoring and control.

High k Gate and Capacitor Dielectrics

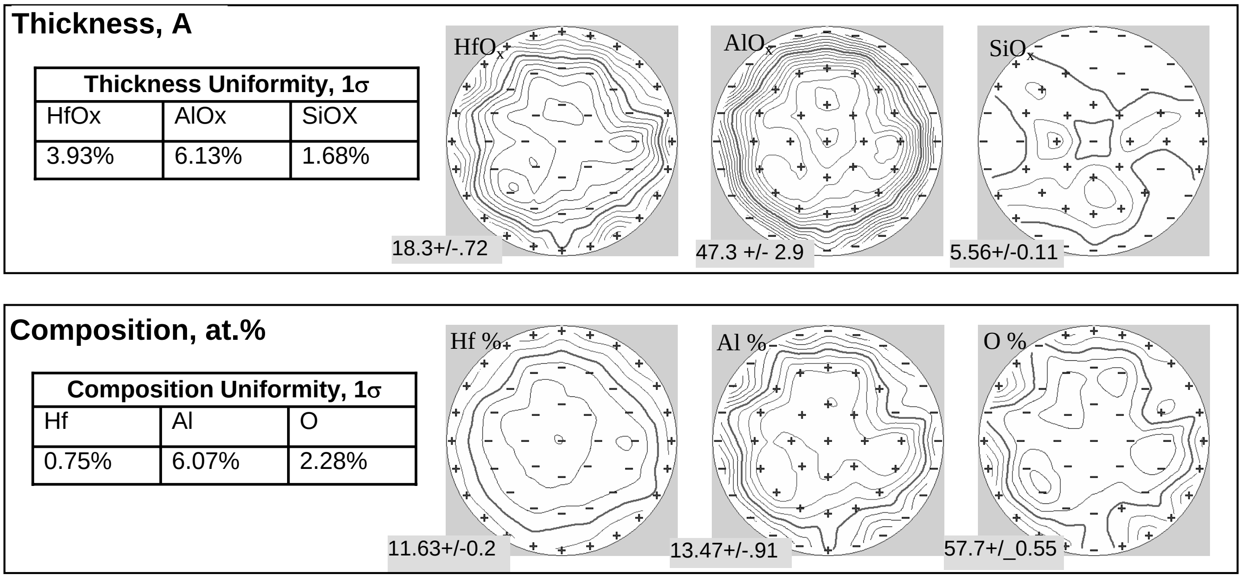
HfOx-containing dielectric films deposited by atomic layer deposition (ALD), particularly ternary or quaternary materials, have proven to be a compositional control challenge, not the idealized



b.

	Thickness, A	Dose, 1E15#/cm2
Ave	15.288	0.810
1 σ	0.032	0.004
RSD, %	0.208	0.529

FIGURE 4. a)XPS precision data for SiOxNy thickness and N dose and b) resultant statistics.



Thickness Uniformity, 1σ		
HfOx	AlOx	SiOX
3.93%	6.13%	1.68%

Composition Uniformity, 1σ		
Hf	Al	O
0.75%	6.07%	2.28%

FIGURE 5. XPS thickness and composition uniformity for three-layer, ~60 A HfOx/AlOx/SiOx capacitor stack.

layer-by-layer process originally envisioned. The final composition and electrical properties have been shown to depend on the starting surface condition and the layer growth sequence, plus other factors [3]. Visibility to the process uniformity across the wafer and to the compositional repeatability of the processes is key since the stoichiometry is critical to determining capacitor or transistor performance.

Various combinations and stacks of HfOx and AlOx are being considered for next-generation DRAM capacitors. With optimized algorithms, thickness and composition information can be obtained with XPS for bi-layer and multi-layer stacks, provided the total thickness is about 10 nm or less. Figure 5 illustrates the ability of XPS to provide thickness and composition uniformity for an ALD three-layer stack of HfOx/AlOx/SiOx, with a total thickness of about 6 nm. In production, monitoring the uniformity of the relative ratios of Hf to Al or O will be critical for optimal yield.

For ALD HfOx or HfSiOx gate dielectrics an interfacial layer of SiOx is required, but this layer needs to be as thin as possible to minimize impact on the EOT and leakage [3]. Further, if the optimal sub-oxide is not formed, the electrical characteristics can be degraded. Figure 6a shows how XPS thickness metrology can be used to determine the thicknesses of the high-k and sub oxide layers simultaneously. The composition of each layer is also provided in the same measurement. Figure 6b shows also how ALD layer thickness depends on the number of deposition cycles and the starting surface wet chemical conditioning. The number of ALD cycles is normalized to the maximum number run. Optical thickness metrology tools would have great difficulty in measuring the thickness and composition of the underlying oxide film.

Electrode Materials

Used in conjunction with high k gate or capacitor dielectrics, various new electrode materials are being investigated. For some DRAM architectures titanium nitride is being used. XPS can be used to determine the thickness and Ti/N composition uniformity in the film, and also can identify residual contaminants remaining from the deposition precursors. For example, Figure 7a shows a representative uniformity map for Cl in TiN on Si, while Figure 7b shows the correlation between resistivity of the TiN and Cl measured by XPS. The two symbol types represent two different sets of measurements. These data show that a 1% variation in residual chlorine in TiN can significantly impact the electrode resistivity.

CONCLUSION

Compositionally critical processes are being implemented with increasing frequency for 65nm and 45nm silicon technology nodes, and XPS has moved from laboratory analysis to production metrology to fulfill the process control needs for

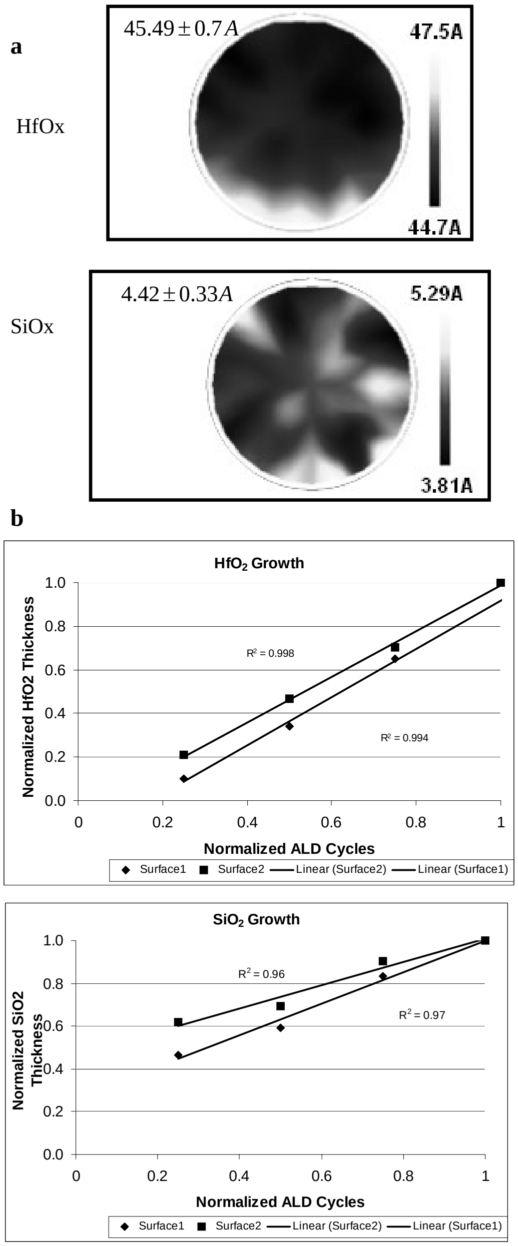


FIGURE 6. a) XPS thickness uniformity maps for HfOx/SiOx stack and b) thickness of HfOx and SiOx ALD layer growth as a function of ALD cycles and surface wet chemical preparation type.

ultra-thin dielectric films. In addition to the applications covered above, XPS compositional and/or thickness metrology also has applicability to surface conditioning, ultra shallow junctions, SiGe:B strain layers, BEOL barrier layers, and other materials.

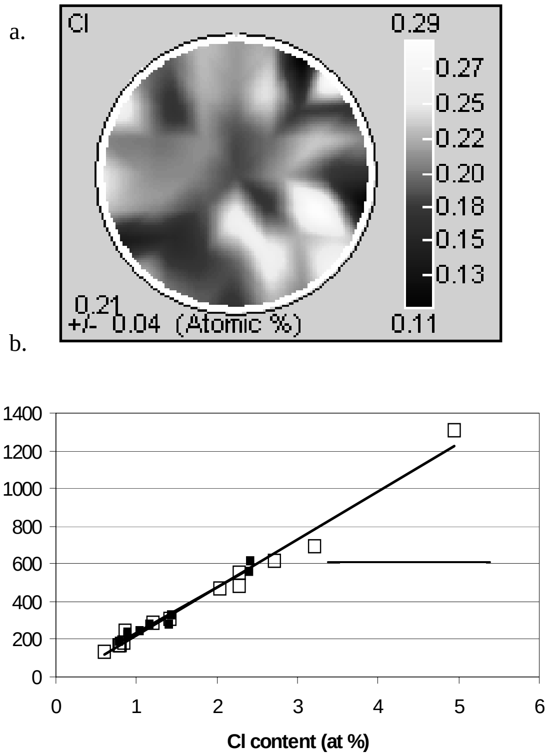


FIGURE 7. a) Compositional uniformity map for Cl impurity in TiN and b) correlation of TiN resistivity with Cl content.

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