1892
doi:10.1017/S1431927610059738

Microsc. Microanal. 16 (Suppl 2), 2010

Structure and composition of metal-doped HfO_2 gate oxides in CMOS devices studied by high resolution STEM and EELS

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Extreme scaling of CMOS devices has led to a number of challenges for characterization and analysis at high spatial resolution. Along with continued shrinking of feature size, such as channel length, contact size and gate oxide thickness, there is a concomitant deployment of new materials such as high dielectric constant (K) gate oxides, metal gate electrodes, and strained or high mobility channels. Future generation structures will likely include migration to 3D architectures. The focus of this study is a model gate stack which is comprised of a TiN metal-gate electrode, a metal doped HfO_2 high-K dielectric and a very thin lower-K interfacial SiO_2 layer on Si. Metallic dopants in high-K dielectrics, such as Al, La, Dy, or Ba, are often used to modify device threshold voltage (V_t) and/or increase K. In this study a thin (<1nm) Ba capping layer is deposited onto HfO_2 (<2.5nm) which following an anneal leads to a large flat-band voltage-shift of -750mV relative to a Ba-free stack desirable for low-V_t n-channel transistors, and an equivalent oxide thickness of 9Å. Adding dopants to HfO_2 for scaling is associated with increasing the dielectric constant of the SiO_2 or the stabilization of higher-K crystalline phases of HfO_2 . [1,2]

The chief challenge for microanalysis for this system is the detection of metallic dopants without significant radiation damage. Empirically, a 3Å-30pA probe (typical for a conventional FEG STEM) leads to visible damage after ~5s suggesting a critical dose of ~2nC/nm^2 at 200kV. This implies exposure times of 0.16s should be possible for 1Å-100pA Cs-corrected probes. The dependence on kV and dose rate on the loss of chemical species during the irradiation has not yet been fully studied. For a 20nm thick sample the number of Ba atoms at 5at.% in HfO_2 is ~6 in a 3Å probe and <1 in a 1Å probe. The detection sensitivity by EELS for 5at.%Ba in HfO_2 is estimated using "EELS Advisor". Using the 3Å-30pA probe, detection requires an exposure time of ~2s. Evaluation of the maximum dose necessary to detect dopants when the sampled volume contains on average less than 1 requires only that single atom sensitivity is achieved. For the 1Å-100pA probe this is accomplished with an 80ms exposure time, i.e. a factor of 2 less than would generate damage.

FIG1 shows an HREM image of the Ba-doped HfO_2 and the interface layer. Diffractogram analysis of several HfO_2 grains yields lattice spacings consistent with the higher-K polymorphs (cubic, orthorhombic or tetragonal) rather than the common monoclinic structure. An EELS line profile analysis recorded using an FEI F20 STEM is shown in FIG2. The max Ba/O ratio measured is 0.07±0.02 which is equivalent to 5 at % assuming either an HfO_2 or SiO_2 matrix. SNR of the extracted raw line profile is 12, consistent with "EELS advisor" value of 11 for 5at% Ba.

To understand the V_t shift mechanism requires high resolution dopant distribution across the gate stack. A series of line profiles were recorded using the 1Å C_s-corrected NION SuperSTEM1 operating at 100kV. In this case we observe the Ba distribution being weighted towards the IL with relatively little remaining at the TiN/HfO_2 interface Images before and after the line-profile indicate modest damage. It is observed as a slight shoulder in the intensity profile in SiO_2 layer. To extract the fractional amount of dopant in the IL and High K we assess the influence of elastic scattering on the signal strength. Acquisition of a low-loss spectrum profiles indicates that correction factor is less than 5% in this example. We find here that 30% of the total Ba is located in the IL layer and extends up to the Si interface.

To further improve detection sensitivity various noise filtering methods were explored. The most successful in this application was 4 component data reconstruction from principal component analysis, improving SNR by a factor of 2x (FIG 4). The success of PCA depends critically on the energy range, data pre-treatment and the number of components used for reconstruction. Other methods such as median filtering in either spectral or energy domain, multiple-linear-least-squares fitting of standards to the power-law-background-subtracted data or 2nd difference filtered data marginally improve SNR.

<u>References</u>

[1] M.M. Frank et al. Microelectron. Eng. 86, 1603-1608 (2009).
[2] H. Jagannathan et al ECS Trans. V19, p. 253 (2009)
[3] Work performed by the Research Alliance Teams at various IBM Research and Development Facilities

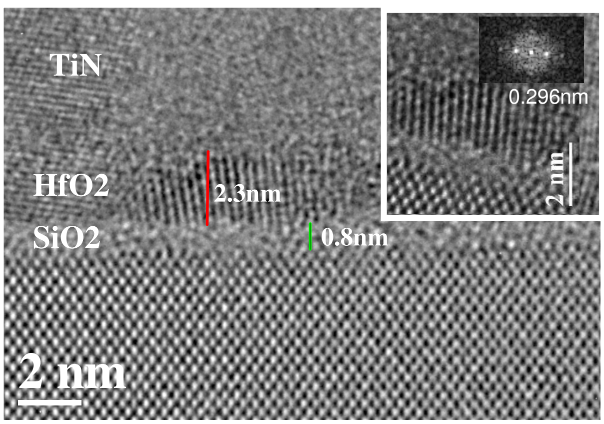

FIG. 1. Ba-doped HfO_2 gate oxide. SiO_2 interface layer is 8Å. Diffractograms of HfO_2 indicate the phase is predominantly tetragonal or cubic rather than monoclinic.

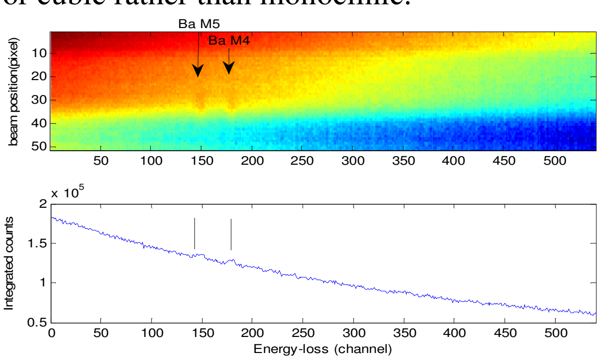

FIG. 2. EELS line spectrum (5s/pixel) showing Ba M_{45} edge (781eV) recorded of High-K stack using FEI F20 STEM. The max SNR is 11 for a 25eV window.

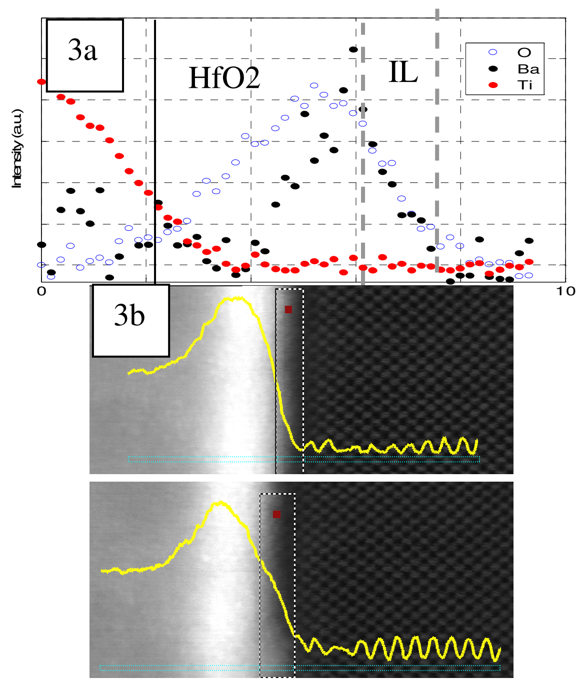

FIG 3 a) EELS profile showing Ba diffusion through HfO_2 and into SiO_2 IL (b) HAADF image before (top) and after (bottom) line profile. Shoulder on line profile suggests modest damage to IL.

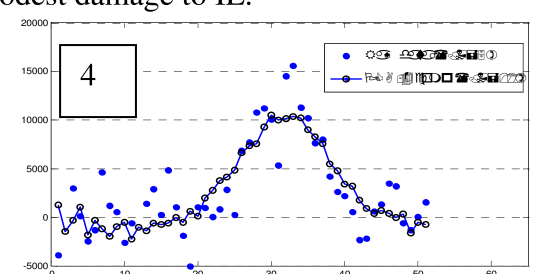

FIG 4.(right) PCA reconstruction using 4 components improves SNR by 2x

https://doi.org/10.1017/S1431927610059738 Published online by Cambridge University Press