Physical characterization of thin HfO_2 layers by the combined analysis with complementary techniques

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New dielectrics with higher dielectric constant, referred to as "high-k materials", are expected to replace soon the silicon oxide as gate dielectric in the electronic devices. Several candidate materials and deposition processes are currently under investigation. Both for the development of these processes and for future process control, the optimization of the physical analysis procedures for these materials is necessary.

The important physical parameters that need to be characterized are the layer thickness and interface or surface roughness, the material density, the composition and possible contaminants, the crystallinity, the bandgap, and the thickness and nature of the interfacial layer between the high-k material and the silicon. No single technique will be able to provide all these characteristics and therefore the combined effort by complementary techniques is necessary as discussed in this paper.

HfO_2 layers are deposited by Atomic Layer Deposition (ALD) and by Metal Organic Chemical Vapour Deposition (MOCVD) on a 1 nm oxide grown in-situ by rapid thermal oxidation (RTO) on 200 mm (100) silicon wafers. The thickness of the HfO_2 layers is varied in the range of 0-100 nm. Some wafers are annealed in a nitrogen ambient at 700ºC after the deposition. The crystallographic structure, density, roughness and interlayer properties of the latter samples might be changed so that a wide variety of sample characteristics can be investigated with the total set of wafers.

The samples are analyzed by spectroscopic ellipsometry, X-ray reflectometry (XRR), Angular resolved and single X-ray photo-electron spectroscopy (ARXPS, XPS), attenuated total reflection (ATR) infrared spectroscopy, time-of-flight secondary ion mass spectroscopy (TOF-SIMS), high resolution elastic recoil detection (H-ERD), Rutherford backscattering spectrometry (RBS), X-ray diffraction (XRD) and transmission electron microscopy (TEM).

It will be shown that by the combination of the information deduced from the different analysis techniques for the thickness, chemical and structural characterization of the layers, the measurement methodologies for the different techniques can be optimized.

Most techniques, except TEM and XRR, rely directly or indirectly on the material density for the calculation of the layer thickness. Using the bulk density for HfO_2 leads to a wide spread of the thickness obtained with the different techniques (Fig. 1). The density derived from the absolute thickness as determined by TEM or the one obtained from XRR is lower than the bulk density and gives better correspondence between the different results (Fig. 2). More results will be discussed in the paper.

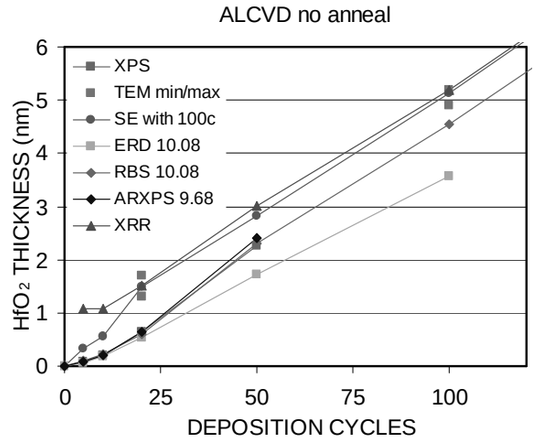

Fig. 1 : HfO_2 thickness versus number of deposition cycles as analyzed by TEM, SE using optical data derived for the 100 cycles sample, ERD and RBS using the bulk density of monoclinic or tetragonal HfO_2 and by XRR fitting both the thickness and the density. For the low cycle numbers incubation is observed. The XRR for the thinnest layers seems rather unreliable.

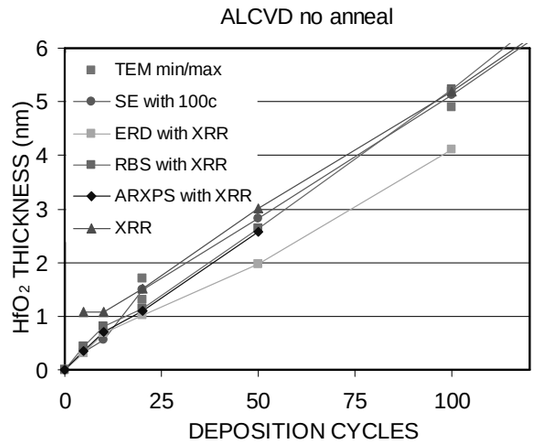

Fig. 2 : The same data using the densities as obtained by the XRR results for each sample. For the thicker layers the correspondence between SE, TEM, RBS, ARXPS and XRR is now excellent, while for the thinner layers the incubation effect is compensated by the much lower density obtained by XRR for these layers.