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Thickness mapping of high- κ dielectrics at the nanoscale

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Contact potential difference measurements by Kelvin probe force microscopy (KPFM) together with corona charging was applied for contactless equivalent oxide thickness (EOT) mapping with nanometer range lateral resolution. Characterization was performed under ambient conditions and compared to a conventional macroscale method based on Kelvin probe measurements. The presented method is directly applicable for dielectrics with homogeneous thicknesses. For other dielectric layers, the implemented laboratory setup suffers from imprecise KPFM sample positioning. If such samples have microscopically non-flat surfaces a proposed data evaluation procedure overcomes that issue which is demonstrated for nanoscale EOT maps of HfO_2 and HfSi_xO_y samples. © 2014 AIP Publishing LLC. [<http://dx.doi.org/10.1063/1.4863947>]

For process development or control of SiO_2 based amorphous dielectrics, it is sufficient to record thickness maps at the macroscale (wafer scale) in a contactless manner with a lateral resolution of millimeters. Ellipsometry or interferometry is most widely used for this purpose. However, to get additional information about charges in the oxide or about interface state densities, methods are used which employ corona charging together with contact potential difference (CPD) measurements between the sample's surface and its backside. The idea behind such characterization is explained by Edelman *et al.*¹ The equivalent oxide thickness (EOT) is evaluated by charging the sample at least twice with known densities of corona generated positive (H_3O^+) or negative (CO^{-3}) ions² and measuring the resulting change in CPD with a scanning Kelvin probe (SKP). If the sample is in accumulation throughout this procedure, EOT can be easily calculated from the corresponding differences in deposited charge density (ΔQ) and CPD (ΔCPD) (see Eq. (1); C_{Is} is the dielectric capacitance, ϵ_{SiO_2} and ϵ_0 are the relative permittivity of SiO_2 and the vacuum permittivity, respectively)²

$$\text{EOT} = \frac{\epsilon_{\text{SiO}_2} \epsilon_0}{C_{\text{Is}}} = \frac{\epsilon_{\text{SiO}_2} \epsilon_0 \Delta \text{CPD}}{\Delta Q}. \quad (1)$$

However, today, high- κ insulators are widely used as gate dielectrics in semiconductor devices. In contrast to SiO_2 , depending on deposition temperature, post-deposition annealing condition, film thickness, etc., high- κ layers are often nanocrystalline. Because of the formation of grains and grain boundaries which may lead to increased surface roughness, dielectric thickness fluctuations might occur, too. The lateral dimensions of such crystallites are rather small (typically some tens of nanometers). Even the best SKPs, however, reach only several microns as a maximum lateral resolution.³ Thus, a CPD measurement method with a

significantly higher lateral resolution is required to detect thickness variations at the nanoscale.

Recently, using Kelvin probe force microscopy (KPFM), charge distribution within a single molecule has been imaged⁴ which proves the very high resolution of KPFM's surface potential measurements. It is an offspring of non-contact atomic force microscopy (AFM) where each scan line is scanned twice: In tapping mode to measure topography (height) and in lift mode to obtain CPD. During lift mode, an oscillating drive voltage is applied, not to the piezo as in tapping mode but to the tip itself whose DC voltage is adjusted by a feedback loop to nullify the tip-sample Coulomb force.⁵ In such case, the adjusted tip's DC voltage is equal to the CPD. Besides, Lägél *et al.*⁵ have already observed that only insignificant amount of charge is lost due to slightly tapping the sample's surface with an AFM tip. Also, they have compared CPD measurements performed with both, SKP in ambient and KPFM in N_2 environment on differently corona charged SiO_2 samples, and found a nearly perfect agreement, meaning that KPFM can be used in conjunction with a corona charging technique to properly detect CPD changes due to the corona charge.⁵ Therefore, KPFM instead of SKP should be applicable for the thickness mapping of high- κ dielectrics with nanoscale lateral resolution which will be analyzed in this work.

As the used KPFM setup is operating in ambient rather than N_2 environment, first, samples with laterally homogeneous SiO_2 stripes are prepared to prove the agreement between macro- and nanoscale CPD measurements for the given setup, and, thus, that the given approach is capable to measure dielectric thicknesses. For this purpose, two 150 mm p -type Si wafers were thermally oxidized with an initial SiO_2 thickness of 20 nm. Subsequently, the samples (in the following referred to as SiO_2A and SiO_2B , respectively) were partially etched to produce SiO_2 stripes of different thickness (i.e., approximately 5, 10, 15, and 20 nm as confirmed by ellipsometry). Second, three nanocrystalline high- κ samples were prepared to investigate the lateral resolution of the method.

As the first high- κ sample, a 10 nm HfO_2 layer was grown by 75 cycles of atomic layer deposition at 300 °C on a

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150 mm *p*-type Si wafer covered with ~ 1 nm natural oxide layer. Subsequently, the wafer was rapid thermally annealed (RTA) in an O_2 environment at 900°C for 15 s.

The other two high- κ samples were prepared by growing a 4 nm SiO_2 layer using dry thermal oxidation on a 150 mm *p*-type Si wafer. On top of it, a 5 nm $HfSi_xO_y$ layer with slightly higher thickness towards the edge of the wafer was deposited by metal organic chemical vapor deposition. The wafer was then annealed by RTA at 900°C in O_2 for 10 s. Finally, the wafer was broken into halves, and one half was further annealed at 450°C in forming gas for 30 min. The latter will be referred to as “RTA + FGA” and the former as “RTA.” For sample “RTA,” measurements were performed close to the center of the wafer whereas for sample “RTA + FGA,” a measurement position closer to the wafer edge was chosen (i.e., higher thickness compared to sample “RTA”).

The procedure for comparing macro- and nanoscale CPD measurements, as well as estimated thicknesses, is summarized as follows: First, the sample was brought into deep accumulation by uniformly charging its surface with a linear corona charger of a macroscale tool (Semilab WT-2500). Then, the CPD values at the spots of interest (and, if relevant, their change with time) were measured with the same tool using SKP (8 mm diameter Kelvin probe). Immediately after, the sample was transferred to the AFM laboratory and within the spot area of the macroscopic tool, CPD was measured at the nanoscale with KPFM using a lift height of 10 nm and a tip drive amplitude of 1 V (Bruker Dimension Icon in ambient conditions, Nanoscope V controller, PtIr coated EFM probes for SiO_2 and SSS-NCH probes for high- κ samples, both NANOSensorsTM). Then, the sample was transferred back to the macroscale tool, and CPD was measured again to detect any charge leakage during KPFM and sample transfer, in which case leakage could be corrected for in the thickness determination procedure presented later. The sample was charged again with a known charge density, and the

FIG. 1. Comparison of macro- and nanoscale CPD measurements for samples SiO_2A and SiO_2B with stripes of different thicknesses t_{ox} for different amounts of corona deposited charge (symbols). Lines correspond to linear regressions for each data set (regression coefficients of 1.0 each).

TABLE I. Comparison of average SKP and KPFM EOTs for the SiO_2 samples together with the difference of the KPFM EOTs with respect to the SKP EOTs.

Sample	Thickness (nm)			Difference (%)
	Stripe	SKP	KPFM	
SiO_2A	5	5.3	5.8	9.4
	10	10.0	10.7	7.0
	15	14.9	15.6	4.7
	20	20.9	22.0	5.3
SiO_2B	5	5.1	5.1	0
	10	10.8	10.4	3.7
	15	15.8	14.9	5.7
	20	20.8	20.1	3.4

above described measurements were performed (i.e., SKP and KPFM CPD measurements). This cycle was repeated 2–3 times and EOT was obtained from the slope of the linear fit of the dependence of CPD on corona charge.¹

The comparison of SKP and KPFM CPD measurements for the SiO_2 samples is provided in Fig. 1. A very good correlation between macro- and nanoscale CPD values can be observed for both samples and for all thicknesses. Note that CPD is a relative not absolute quantity and the regression offsets for both samples are not equal because the Kelvin probe of the macroscale tool and the KPFM tips used for each sample had different surface potentials.

The average EOTs for the SiO_2 samples as evaluated from the described measurements are given in Table I. It can be seen that the absolute relative differences are mostly low (3%–6%), with a maximum being 9.4% for the 5 nm stripe of sample SiO_2A . The differences are caused by the fluctuating surface potential of the KPFM tip⁵ and charge leakage.

Next, it was aimed at determining thickness maps with nanoscale lateral resolution for high- κ samples. From the corresponding KPFM measurements, a strong correlation

FIG. 2. Height and CPD map of sample HfO_2 after first positive charging with 83.3 nC/cm^2 together with the corresponding evaluated EOT map. Please note the different scale for height and EOT, respectively.

between topography and CPD was observed: Smaller heights correspond to less negative CPD values and larger heights correspond to more negative CPD values (see Figs. 2 and 3). Similar results have been detected by Ludeke and Gusev⁶ who did not explain their findings. However, for the SiO₂ samples, the likewise observed correlation between height and CPD is obviously due to a direct correlation between measured height and thickness. This should also hold for the high- κ samples. Such hypothesis might be supported by the observation that in contrast to accumulation conditions, which were used so far, no correlation between height and CPD could be observed for depletion conditions. There, CPD is mainly governed by the contribution of space charges and trapped charges in the dielectric or at the interface and not by the dielectric capacitance (and, thus, dielectric thickness).

It should be noted that if corona charging could be performed at the KPFM tool *in-situ* without moving the sample, samples with laterally inhomogeneous thicknesses could be simply characterized as described for the SiO₂ samples. Due to the required realignment of the sample during the described procedure, however, it is practically impossible to measure exactly the same nanoscopic area with the KPFM. To overcome this problem, the corona-Kelvin method was adapted to nanoscale. The adaptation assumes linearity between height and CPD and that the layer properties in general do not change laterally substantially within micrometers (experimentally determined AFM positioning accuracy is $\pm 1 \mu\text{m}$). A scheme of the adapted method is provided in Fig. 3 with actual data for sample HfSi_xO_y “RTA + FGA” and is explained in the following.

FIG. 3. Scheme of the adapted method using measured data for sample HfSi_xO_y “RTA + FGA.”

KPFM measurements are performed at the same microscopic (but slightly different nanoscopic) spot after each corona charging ΔQ (step 1 in Fig. 3). Because height h and CPD are measured by separately scanning the same line, a small drift might occur between topography and CPD map. The measured image pairs are corrected for it so that height peaks coincide precisely with CPD troughs and vice versa. Also, the CPD images are corrected for leakage by an amount which was determined by measuring the decay of CPD for approximately 90 s right after charging with the macroscopic tool and extrapolating the change in CPD due to leakage for the KPFM measurement date (step 2). Then, CPD vs. height relation is extracted for each measurement. Simple linear regression cannot be used here, because least squares method does not appropriately filter out any outliers and, therefore, provides an incorrect slope. Instead, CPD vs. height covariance matrices are calculated for each deposited charge density and the longer axes of the corresponding ellipses (first principal components)⁸ are regarded as filtered CPD vs. height regression slopes (step 3). These slopes were extracted for the height and CPD image pairs for each charge density are used to recalculate the CPD maps, corresponding to each of the surface charge depositions for every measured height map (in the example, for each height map three CPD maps are recalculated, step 4). Step 3 and step 4 are explained in more detail in Ref. 9. Finally, EOT for each pixel is calculated from a linear regression of the CPDs at that pixel vs. deposited charge density (step 5, exemplarily given for the points with smallest and largest thickness) to evaluate the EOT maps for sample HfSi_xO_y “RTA + FGA” for each measurement (step 6).

According to the presented scheme, EOT maps for the other high- κ samples could be also evaluated. Exemplarily, Fig. 2 shows results for sample HfO_2 after charging the sample with 88.3 nC/cm^2 (initial accumulation conditions were obtained for charging with -596 nC/cm^2). The noise visible in the CPD image is due to measurements being performed in ambient conditions. By taking the first principal component⁸ as the slope for recalculating the CPD images, the influence of such noise is minimized. Table II summarizes the evaluated average EOTs for the high- κ samples which show a very good correlation between the results from both methods. The high average EOT of sample HfSi_xO_y “RTA + FGA” compared to sample HfSi_xO_y “RTA” results from the inhomogeneous thickness of the grown HfSi_xO_y layer.

Obviously, the presented adapted method has limitations. The first one arises from the assumption of a linear relation between CPD and height. The thickness variations must have

a noticeable signature in topography (see, e.g., topography maps Figs. 2 and 3) which is typical for most high- κ layers: Samples with smooth dielectric layer/substrate interface and rough surface, as well as those with both of them being rough could be properly evaluated. On the other hand, thickness variations would be impossible to evaluate for samples with flat surfaces. The majority of identical height points and noisy CPD data (low signal fluctuation to noise ratio) would cause very steep filtered CPD/height regression slopes and consequently result in an incorrect evaluated thickness.

The second limitation comes from using the corona charging, as the ions deposited are actually contaminants. At the nanoscale, this is of high importance because the ions are of comparable size to the features and care must be taken when choosing the charge density. If it is too large, the ions cover the valleys of surface and, therefore, topography as well as CPD features can no longer be observed. Directly charging the surface with an AFM tip could possibly overcome this issue and has been already performed and thoroughly studied by Mesquida.⁷ Very low charge density, on the other hand, causes only a negligible change in filtered CPD/height regression slope which would bring the recalculated CPD maps too close together on a CPD scale and would result in significantly high thickness errors. These regression slopes should decrease monotonically when negatively charging and vice versa. This can be easily monitored after a new measurement has been performed and, in case of a slope change being, e.g., too small, the sample could be further charged. Also, having measured image pairs for more charging steps or having measured more times for the same deposited charge density would improve the final results.

Summarizing, average EOT values for SiO_2 and high- κ samples were evaluated in a contactless manner using corona-Kelvin metrology with SKP and KPFM applying a proposed evaluation procedure. EOT values from both methods were found to be in close agreement. Furthermore, thickness maps with nanoscale lateral resolution of annealed HfO_2 and HfSi_xO_y samples could be determined in ambient conditions without a need for a very precise sample positioning and were exemplarily presented. Thus, it is proven that a macroscale thickness mapping method was adapted to nanoscale. Finally, method's limitations were discussed and possible workarounds provided.

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TABLE II. Comparison of average SKP and KPFM EOTs together with the difference of the KPFM EOTs with respect to the SKP EOTs.

Sample	Thickness (nm)		Difference (%)
	SKP	KPFM	
HfO_2	5.3	5.2	1.9
HfSi_xO_y RTA	5.7	5.7	0
HfSi_xO_y RTA + FGA	8.5	8.7	2.4

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