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Laterally resolved electrical characterisation of high-*K* oxides with non-contact Atomic Force Microscopy

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Abstract

Fixed oxide charges in atomic layer deposited (ALD) Al_2O_3 on hydrogen-terminated Si were observed by ultra-high vacuum (UHV) non-contact AFM with a conductive tip. Comparison of bias-dependent images with a spherical tip model showed the importance of the tip image in the substrate for a quantitative understanding of the image contrast. The Contact Potential Difference and differential capacitance were probed by means of bias modulation and lock-in detection at the 1st and 2nd harmonic, respectively. A difference in topography-capacitance cross-correlation was found between Al_2O_3 deposited on hydrogen-terminated Si and thermal SiO_2, which can be attributed to substrate-inhibited versus non-inhibited deposition.

Keywords: ALD; AFM; Kelvin Probe Force Microscopy; Differential Capacitance Imaging; substrate-inhibited ALD

1. Introduction

Further downscaling of the gate dielectric in complementary metal-oxide-semiconductor (CMOS) technology will soon require high-*K* gate dielectrics as replacement for SiO_2 [1]. The electrical quality of high-*K* gate dielectrics and their interfaces with the underlying silicon substrate are still poor compared to thermal silicon oxide. This fact hinders the integration of these oxides in CMOS technology. Scanning Probe techniques allow electrical characterisation of thin oxide films up to nanometer scale lateral resolution without the need of additional processing steps beyond the growth of the oxide.

In this work ALD deposited thin Al_2O_3 films were investigated by means of Kelvin Probe and differential capacitance imaging with UHV non-contact AFM [2,3]. Deposition of metal oxides on hydrogen-terminated silicon is often complicated by so-called substrate-inhibition, caused by a lack of reactive sites on the starting surface [4]. This results in a lower

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growth rate in the first cycles of the deposition compared to the equilibrium growth rate that is achieved when the metal oxide fully covers the substrate. We show that this substrate-inhibition can be visualised by linking the electrical and morphological scanning probe measurements.

2. Sample preparation and experimental details

Aluminium oxide films were deposited on *p*-type Si (001) samples (boron doped, $N_A = 10^{15}$ cm^{-3}) in a home-built Atomic Layer Deposition system from trimethylaluminium and water vapour at a growth temperature of 300 °C. Before deposition samples were cleaned in fuming and boiling nitric acid (HNO_3 100 % and 69 %, respectively) and the native SiO_2 was removed by a 1 % HF dip. X-ray Photoelectron Spectroscopy measurements confirmed that the layers consisted of stoichiometric Al_2O_3 and the growth rate of our ALD process on thermal SiO_2 was determined to be 0.089 nm per cycle in line with [4]. Al_2O_3 deposition was also carried out on a thermally grown SiO_2 film of 2.2 nm thickness. XPS was used to determine the deposited amount of aluminium oxide during the first cycles of the growth. The growth rate on hydrogen-terminated Si was significantly reduced with respect to deposition on thermal SiO_2, the latter showing an equivalent growth rate compared to deposition on chemical oxide. This confirms that the ALD is substrate-inhibited on hydrogen-terminated Si, but not on thermal SiO_2 [4].

Non-contact AFM measurements were performed with an RHK UHV 450 beam-deflection AFM operated in UHV combined with a Nanosurf EasyPLL FM demodulator. Conductive cantilevers with a nominal resonance frequency of 75 kHz were used to probe topography and electrostatic interactions. Kelvin Probe measurements were carried out by modulating the sample bias with a 1 kHz, 0.5 V RMS sine wave and detecting the electrostatic force gradient by feeding the demodulated PLL output in a lock-in amplifier tuned to the bias modulation frequency. For Contact Potential Difference (CPD) measurements a feedback loop was used to adjust the DC bias in order to minimise the Kelvin signal [5]. CPD measurements were also obtained by tip height vs. voltage spectroscopy, (i.e. finding the voltage at which minimal total force occurs,) in order to exclude modulation artefacts [6]. Differential capacitance images were obtained by tuning the lock-in to the second harmonic of the bias modulation. The electrostatic signal at this frequency is proportional to the second derivative of the capacitance with respect to the surface normal $\partial^2 C/\partial z^2$.

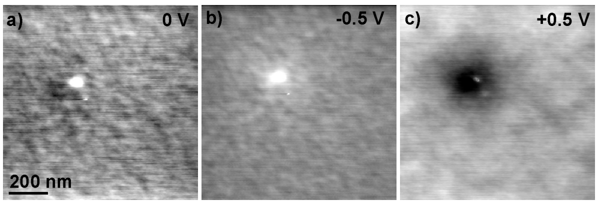

Fig. 1. NC-AFM images of 2.5 nm Al_2O_3 on Si obtained with a conductive tip in UHV. The large protrusion in image a) taken at zero bias results from an oxide charge. Application of a sample bias of –0.5 V (b) or +0.5 V (c) modifies the contrast.

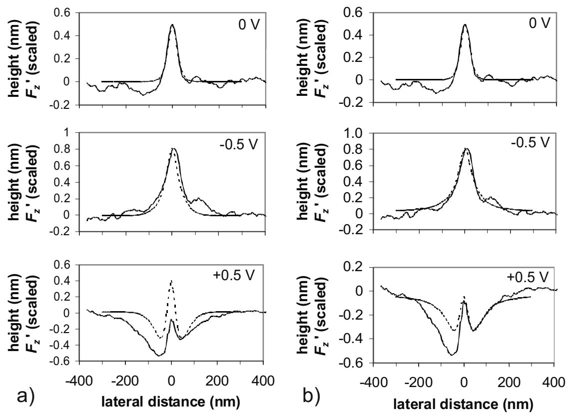

Fig. 2. Cross-sections through the centre of the oxide charge in bias-dependent images from Fig. 1 (solid lines) compared with calculations (dashed lines) of the electrostatic force gradient with a spherical tip model without incorporation of the tip image in the substrate (a) and with substrate image (b).

3. Fixed oxide charge imaging

Oxide charges appear in AFM images as large apparent protrusions on the surface (see Fig. 1), resulting from the electrostatic interaction between the oxide charge and its image charge in the tip [2]. Application of a bias voltage between tip and sample changes the overall electrical interaction and thus the contrast of the charge in the topography image. Fig. 2