Preliminary report of experiment SI995 (block allocation for SI-995, SI-996 and HS-2385)
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Introduction

The current CMOS gate dielectrics are approaching their limits. Fast shrinking of the transistor size has forced the channel length and gate dielectric thickness to also decrease rapidly. The thickness of the conventional SiO_2 has become so thin that the leakage current arising from electron tunneling is becoming a major problem for low power circuit applications. Many materials with a higher dielectric constant k are currently being investigated to replace SiO_2 (k=3.9) as gate dielectric. A higher-k dielectric can achieve an equivalent electrical thickness with a physically thicker film that will reduce the leakage current. For a high-k material with, for example, a k-value of 20 the physical thickness may still be 50Å to obtain an equivalent (silicon)oxide thickness of 10Å. Hf-based oxides are thought to be suitable candidates as dielectric. In particular hafnium silicate, $HfSi_xO_y$, is thought to be more easily integrated within the current transistor design.

Experimental

$HfSi_xO_y$ and HfO_2 samples were deposited on (100) Si using two different deposition tools to compare the deposition processes. In the first tool, three different compositions of hafnium silicate samples in a thickness range of 10Å-100Å were deposited. Some of the pure hafnium oxide and silicate samples received a post deposition anneal (PDA). In the second tool, the hafnium oxide samples were deposited at different deposition temperatures (second tool).

Ex-situ and in-situ measurements were made on the samples using the 4+2 diffractometer of ESRF beamline ID01. The ex-situ measurements were made using a setup that enclosed the samples by a helium purged capton cone. The in-situ measurements were made to study the temperature dependence using a small chamber with a Beryllium dome at pressure below 2E-5 mbar. Grazing incidence diffraction (GID), X-ray reflectivity (XRR), and grazing incidence small angle X-ray scattering (GISAXS) were performed.

Results

The as-deposited hafnium silicate samples were found to be amorphous for all compositions and thicknesses. After a PDA, the data indicates that the silicate samples started to become crystalline. Pure HfO_2 was already crystalline at the lower temperature PDA. To crystallize the hafnium silicate films, the samples were annealed in the sample environment for 8 minutes at >900°C. Similar to the crystalline HfO_2 samples, the films were found to be un-textured, poly crystalline, and mainly of monoclinic phase (partly orthorhombic). For higher annealing conditions, the orthorhombic phase tends to disappear. Using Scherrer's equation, the grain sizes were found to be of the order of the film thickness.

Spectra for very thin samples (10Å and 20Å) were successfully obtained using the beamline which are normally not measurable using conventional X-ray analytical tools. Some interesting

results obtained on these very thin samples were that the out-of-plane and in-plane results (GID) start to be different. It seems that these films have a preferred orientation without being textured. The very narrow peaks (Figure 1) indicate rather large grain sizes (around 200Å). For the 50Å and 100Å thick films, this large difference between in- and out-of-planed diffraction was not observed. TEM analyses are ongoing to investigate it. Knowing that the thickness in devices is only around 20-30Å, the obtained data is very useful to explain electrical results. At this stage the GISAXS analysis is in progress but the first impression is that there is no strong spatial correlation of composition variation.

HfO_2 films deposited at different temperature gave some results that seem to be in contradiction with results obtained before by ATR and TEM. Currently the ATR and TEM measurements are being repeated. It might be related to the different film thickness used during the ESRF experiment (which is thinner to be realistic for devices).

The in-situ measurements were not as successful as the ex-situ ones. Since the total sample height is only 0.5 mm, the beam was found to be blocked by the housing of the Beryllium dome (the height of the heater plate is not high enough for our type of samples). Connecting a second sample plate to increase the height of the heater caused temperature control problems at the temperatures of interest (900-1000°C) and the set-up needed to be changed back.

Acknowledgement

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Publication results

An abstract has been submitted for the MRS spring meeting 2005 entitled "Phase transformation of HfO_2 thin films grown by liquid pulsed MOCVD". It is based on results obtained during the SI995 experiment and it will be co-authored by e.g. H. Metzger and C. Mocuta.

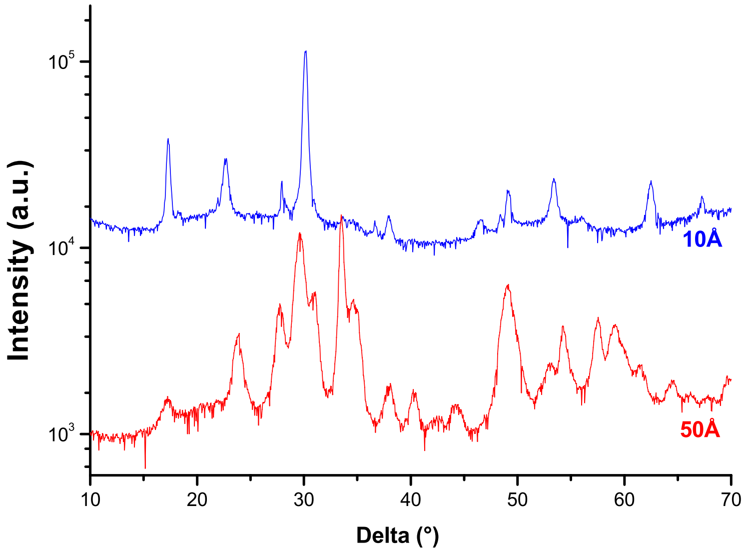

Figure 1. GID of a 10Å and 50Å thick $HfSi_xO_y$ film.