HfO₂/Al₂O₃ Nanolaminate on Si_{0.7}Ge_{0.3} (100) Surface by Thermal Atomic **Layer Deposition**

Iljo Kwak^{a,b}, Kasra Sardashti^{a,b}, Maximilian S. Clemons^{a,c}, Scott T. Ueda^a, Bernd Fruhberger^d, Serge Oktyabrsky^e, Andrew C. Kummel^a

^a Department of Chemistry and Biochemistry, University of California, San Diego,

^b Materials Science and Engineering Program, University of California, San Diego,

^c Department of Electrical and Computer Engineering, University of California, San Diego

^d California Institute for Telecommunications and Information Technology, University of California San Diego

^e SUNY Polytechnic Institute, Colleges of Nanoscale Science and Engineering, Albany,

NY

To integrate Silicon-Germanium (SiGe) into future CMOS devices, it is essential to deposit very thin high-k dielectrics on SiGe surfaces with low density interfacial defects. In this study, Al₂O₃/HfO₂ nanolaminate (HfO₂ layers incorporated with Al₂O₃ monolayers) gate stacks were deposited by atomic layer deposition (ALD) using HfCl₄ and H₂O precursors. Electrical properties of the interfaces were quantified by capacitance-voltage (C-V) spectroscopy. Interfaces of nanolaminate stacks were found to have 2x smaller density of interface traps (D_{it}) than pure HfO₂ gate stacks. Cross sectional TEM with Energy-dispersive X-ray spectroscopy (EDS) showed that an SiOx rich interlayer was formed between the nanolaminate and the $Si_{0.7}Ge_{0.3}(001)$ substrate. The SiOx interlayer contains almost no Ge indicating that the HfCl₄/TMA nanolaminate deposition reduced the GeOx in the interface. Furthermore, the SiGe surface was enriched in Ge from 30% to ~70% consistent with the HfCl₄/TMA nanolaminate process reducing and redepositing Ge on the SiGe surface.

INTRODUCTION

Silicon-Germanium (SiGe) alloys have shown a great promise as the channel material for fin field effect transistors (FinFETs) and nanowire field effect transistors (NW-FETs) due to tunability of their carrier mobilities and band gaps by variation in Ge content and tensile/compressive stresses (1), (2), (3), (4), (5), (6). In addition, SiGe alloys are expected to be more easily integrated into the existing Si CMOS fabrication technologies than III-V semiconductor or pure Ge due to rather facile growth of SiGe alloys on Si substrates (7), (8),(9),(10). However, to ensure full integration of SiGe into the future CMOS

technology, it is imperative to develop robust methods to deposit thin high-k dielectrics on SiGe surfaces with low leakage and high interface quality (11),(12),(13),(14),(15).

One of the challenges in realizing low-defect interfaces between high-k dielectrics and SiGe is to control the Ge reactions during the oxide deposition and post deposition annealing steps (16),(17),(18),(19). The presence of Ge at the high-k/SiGe interface could lead to Ge sub-oxide (GeO_x) formation which can readily diffuse within the high-k oxides (20). In addition, since GeO and GeO₂ are volatile and unstable in the presence of Ge, they can pose reliability issues for devices (21). Therefore, it is essential to chemically passivate the interfaces to minimize Ge concentration at the high-k/SiGe interfaces.

Various approaches have been tested for limiting the Ge–O bond formation at the interface including sulfur passivation (18) and plasma nitridation (22). However, these approaches were only successful for pure Al_2O_3 or Al_2O_3/HfO_2 bilayers with a 1-2 nm thick Al_2O_3 interfacial layer. Additionally, $Al_2O_3/HfO_2/Al_2O_3$ tri-layers (or sandwich) have been deposited on strained p-SiGe by ALD at 300°C, where a 0.7nm thick layer of mixed Al_2O_3 -SiOx was observed at the interface (23) In that report, Al_2O_3 was employed as a spacer between HfO_2 and SiGe to prevent interfacial reactions (i.e. Ge out-diffusion) during the routine post deposition annealing of the devices. However, despite low gate leakage as a function of gate bias, the C-V curves showed high fixed and interface trap charges as well as false inversion (23).

In this report, electrical characteristics of the nanolaminates and laminates/SiGe interfaces were compared by capacitance-voltage (C-V) and current-voltage (I-V) spectroscopy measurements on MOS capacitors (MOSCAPs). Al_2O_3 -HfO₂ nanolaminates were prepared by periodic incorporation of Al_2O_3 monolayers in between HfO₂ multilayers from HfCl₄ based ALD. For comparison, Al_2O_3 -HfO₂ laminates were prepared by deposition of single monolayers or multilayer of Al_2O_3 on the bottom and the top of the HfO₂. The nanolaminate gate oxides enabled formation of low-defect oxide/SiGe interfaces. High resolution TEM-EDS (transmission electron microscopy-energy dispersive x-ray spectroscopy) shows that the nanolaminates form a nearly pure SiOx interlayer between the oxide and SiGe substrate.

EXPERIMENTAL DETAILS

A 12nm thick p-type $Si_{0.7}Ge_{0.3}(100)$ with doping level of $2x10^{18}$ cm⁻³ (Applied Materials) was grown epitaxial on p-type Si(100) by molecular beam epitaxy (MBE). Prior to ALD, SiGe native oxides were removed by cyclic HF clean using 2% HF solution and DI water at 25°C, ending with HF dip (24). For surface passivation, HF-treated samples were immersed in 25% (NH₄)₂S solution at 25 °C for 15 min followed by 30 s of DI H₂O rinse. After surface clean, the samples were transferred to the ALD chamber with less than 2 min of air exposure. HfO₂ ALD was performed at 300 °C in a commercial continuous cross-flow reactor (Beneq TFS-200) with Ar as the carrier gas. The chamber base pressure during the ALD process was about 1.7 torr. HfO₂ was deposited by consecutive cycles of 500 ms of HfCl₄ and 500 ms of H₂O. After each HfCl₄ and H₂O pulse, a 6 s long Ar purge was employed. For Al₂O₃ deposition in the cross-flow reactor, 20 cycles of 45 ms TMA pre-pulses were followed by consecutive cycles of 200 ms of TMA and 50 ms of H₂O. Similar to the HfO₂ recipe, after each TMA and H₂O

pulse, a 6s Ar purge was employed. $Al_2O_3 - HfO_2$ nanolaminate structures were prepared by applying the above-mentioned HfO_2 and Al_2O_3 recipes in two different orders: i) 4 x [9 cycles of $HfO_2 + 1$ cycle of Al_2O_3]. ii) 12 x [3 cycles of $HfO_2 + 1$ cycle of Al_2O_3]. Note for the nanolaminates, no TMA prepulsing was employed for each Al₂O₃ cycle. After ALD, 50 nm thick Ni gates were deposited by thermal evaporation. 100nm thick Al back gate electrode deposition was followed by DC magnetron sputtering on the back sides of the SiGe substrates. After completion of fabrication, MOSCAPs were annealed in forming gas (5% H₂, 95% N₂) at 300 °C for 15 min.

Capacitance-Voltage (C-V) spectroscopy of the MOSCAPs was performed using an Agilent B-1500 semiconductor analyzer, with AC modulation amplitude of 30 mV, and with a gate bias range of -2 to 2 V, at multiple frequencies from 2 KHz to 1 MHz. Current-voltage (I-V) spectroscopy measurements was also carried out in the bias range from -2V to 2V. Gate leakage vs. gate bias was measured in the same bias range.

RESULTS AND DISCUSSION

The effects of the Al_2O_3/HfO_2 nanolaminate on the electrical properties of $HfO_2/SiGe$ interfaces were determined by variable frequency C-V spectroscopy. Figure 1a-1c displays the C–V results measured from 2 KHz to 1 MHz for HfO₂/SiGe and Al₂O₃/HfO₂ nanolaminate MOSCAPs. Densities of interface traps (D_{it}) were calculated using the conductance method (25). Figure 1a displays the C-V characteristics of a MOSCAP with 50 cycles of HfO₂ deposited using HfCl₄ and H₂O as the precursors. As shown in Fig 1b and 1c, use of nanolaminate structures resulted in 29% (Nanolaminate with 12 cycles of 3:1) and 39% (Nanolaminate with 5 cycles of 9:1 HfO₂: Al₂O₃) reduction in the density of interface traps relative to the pure HfO₂, at the expense of less than 0.2 μ F/cm² drop in the accumulation capacitance. Note these nanolaminates have no direct Al₂O₃ deposition on the SiGe interface but instead have only HfO₂ deposition on SiGe. This improvement of D_{it} is hypothesized to be due to incorporated Al₂O₃ as a Ge diffusion barrier, a H₂O diffusion barrier or Ni gate metal protection layer; however, as shown by Kavrik et al, it is possible that the TMA may diffuse to the interface during deposition (26). To better quantify the effect of TMA dosing, the values of Cox/Dit were compared; the unit of this approximate metric was defined as $\mu F \times eV \times 10^{-12}$. For the 9:1 and 3:1 HfO₂: Al₂O₃ nanolaminates, the Cox/Dit values were increased by 17% and 48% respectively compared to the pure HfO₂ device. Figure 1d-1F displays the I-V spectroscopy of the pure HfO₂/SiGe and Al₂O₃/HfO₂ nanolaminate MOSCAPs. Compared to 50 cycles of HfO₂, nanolaminate samples resulted in more than 3 orders of magnitude lower leakage current which is consistent with less Ge diffusion into the nanolaminate or less diffusion of Ni during gate metallization.



Figure 1. Capacitance – voltage characteristics for high-k/SiGe MOSCAPs with HfCl₄ precursor: (a) 50 cycles of pure HfO₂ ALD (b) Nanolaminate with 12 cycles of $3:1 \text{ HfO}_2:\text{Al}_2\text{O}_3$; (c) Nanolaminate with 5 cycles of $9:1 \text{ HfO}_2:\text{Al}_2\text{O}_3$. D_{it} values in eV⁻¹.cm⁻² were calculated by the conductance method. Current – voltage spectroscopy for high-k/SiGe MOSCAPs with HfCl₄ precursor: (d) 50 cycles of pure HfO₂ ALD (e) Nanolaminate with 12 cycles of $3:1 \text{ HfO}_2:\text{Al}_2\text{O}_3$; (f) Nanolaminate with 5 cycles of $9:1 \text{ HfO}_2:\text{Al}_2\text{O}_3$

In addition to being incorporated into the nanolaminate structure, Al₂O₃ can be used as an interlayer or a cap. The Al₂O₃ interlayer is hypothesized to prevent Ge out-diffusion by forming a Si-O-Al interface, while Al₂O₃ cap layer is used to protect top surface of the oxide from damage induced by the gate electrode deposition process (this includes ambient oxidation prior to gate metal deposition and Ni diffusion during gate metal deposition) (18). For thin HfO₂ layers, the chemistry of the Al₂O₃ cap may be more complicated since TMA might diffuse through the nanolaminate to the interface as shown by Kavrik et al. (26). Al₂O₃ directly deposited on SiGe may reduce native GeOx to Ge (27). Figure 2 displays the C-V characteristics of HfO₂/SiGe MOSCAPs with Al₂O₃ cap/interlayer. To determine the effect of the cap/interlayer layers, 3 different structures were studied: i) bilayer with Al₂O₃ interlayer: 1 or 5 cycles Al₂O₃ followed by 49 or 45 cycles of HfO₂ (Fig. 2a and 2d); ii) bilayer with Al₂O₃ cap layer: 49 or 45 cycles of HfO₂ followed by 1 or 5 cycles Al₂O₃ (Fig. 2b and 2e); iii) Al₂O₃/HfO₂/Al₂O₃ trilayer: 1 or 5 cycles Al₂O₃ followed by 48 or 40 cycles of HfO₂ and 1 or 5 cycles Al₂O₃ (Fig. 2c and 2f).

A thicker Al_2O_3 layer both as a cap and interlayer resulted in lower D_{it} . The trilayer structure with 5 cycles of Al_2O_3 has D_{it} levels even below the nanolaminates but with reduced Cmax compared to the nanolaminate; the EOT of the trilayer structure with 5 cycles of Al_2O_3 estimated by curve fitting of CV is about 1.9 nm. Compared to the pure

HfO₂/SiGe (Fig 1a), adding a single monolayer of Al₂O₃ either on top (Fig 2a), on the bottom (Fig 2b), or both on the top and bottom (Fig 2c) had no significant effect on the D_{it}. However, adding 5 cycles of Al₂O₃ either on the bottom (Fig 2d), on the top (Fig 2e) or both on the top and bottom caused a significant drop in D_{it} but also C_{ox}. C_{ox}/D_{it} values were also improved by adding 5 layers of Al₂O₃ either on the bottom of HfO₂ to the top of HfO₂ consistent with Al₂O₃ acting either as a diffusion barrier or for GeOx reduction.



Figure 2. MOSCAP Capacitance – voltage characteristics for bilayer and trilayer oxides on deposited SiGe using HfCl₄ precursor: (a) Bilayer with 1 cycle of Al_2O_3 followed by 49 cycles of HfO₂; (b) Bilayer with 49 cycles of HfO₂ followed by 1 cycle of Al_2O_3 ; (c) Trilayer with 1 cycle of Al_2O_3 followed by 49 cycles of HfO₂ and 1 cycle of Al_2O_3 ; (d) Bilayer with 5 cycle of Al_2O_3 followed by 45 cycles of HfO₂; (e) Bilayer with 45 cycles of HfO₂ followed by 5 cycles of Al_2O_3 ; (f) Trilayer with 5 cycle of Al_2O_3 followed by 40 cycles of HfO₂ and 5 cycle of Al_2O_3 . All samples were passivated by wet sulfur clean prior to ALD. D_{it} values in eV^{-1} .cm⁻² were calculated by conductance method.

Effect of Al₂O₃ cap and interlayer on the nanolaminate structures was also evaluated by adding 5 cycles of Al₂O₃ to the top or bottom of 9:1 and 3:1 HfO₂ : Al₂O₃ structures. Figure 3 shows the C-V characteristics of nanolaminates with and without Al₂O₃ caps/interlayers. For the 9:1 HfO₂ : Al₂O₃ nanolaminate (Fig. 3a), addition of 5 cycles of Al₂O₃ either as an interlayer (Fig. 3b) or a cap (Fig 3c) resulted in lower D_{it} levels and lower C_{ox}. Comparing the C_{ox}/D_{it} values of the 9:1 HfO₂ : Al₂O₃ nanolaminates (C_{ox}/D_{it} = 0.89 (Fig 3a) with 5 cycles Al₂O₃ interlayers or caps, both the Al₂O₃ interlayer and cap showed an improvement (C_{ox}/D_{it} = 0.94 (Fig. 3b) and 0.98 (Fig. 3c)). The data is consistent with the Al₂O₃ in the nanolaminate lowering Dit by reducing diffusion of H₂O (from the ALD process) through the gate oxide or reduction of GeOx at the interface; since the Al₂O₃ at the interface is most efficient in reducing the Dit, the GeOx reduction by TMA probably plays a role in the Dit reduction. The role of Al₂O₃ acting as a diffusion barrier or TMA acting as a reducing agent instead of Al₂O₃ directly passivating SiGe is consistent with the study on the 3:1 HfO₂ : Al₂O₃ nanolaminate. For the 3:1 HfO₂ : Al₂O₃ nanolaminate (Fig 3d), addition of 5 cycles of Al₂O₃ as a capping layer (Fig 3f) only modestly changed the D_{it} and the C_{ox}/D_{it} value only slightly increased (C_{ox}/D_{it} = 0.70 (fig 3d) vs 0.72 (fig3f)). However, the device with 5 cycles of Al₂O₃ interlayer (Fig 3e) had a very low D_{it} of 1.43 x 10¹² eV⁻¹.cm⁻², which is remarkably low for nanolaminate structures with EOT of less than 2nm. Furthermore, the C_{ox}/D_{it} value of the device (C_{ox}/D_{it} = 1.12 (Fig 3e) was increased by nearly 60% compared to the nanolaminate only device (C_{ox}/D_{it} = 0.70 (Fig 3d).



Figure 3. MOSCAPs Capacitance – voltage characteristics for Nanolamnate with Al_2O_3 Caps and Interlayers with HfCl₄ precursor: (a) Nanolaminate with 5 cycles of 9:1 HfO₂:Al₂O₃; (b) 5 cycles of Al₂O₃ interlayer followed by nanolaminate with 5 cycles of 9:1 HfO₂:Al₂O₃ followed by (IL); (c) Nanolaminate with 5 cycles of 9:1 HfO₂:Al₂O₃ followed by (IL); (c) Nanolaminate with 12 cycles of 3:1 HfO₂:Al₂O₃; (e) 5 cycles of Al₂O₃ cap; (d) Nanolaminate with 12 cycles of 3:1 HfO₂:Al₂O₃; (e) 5 cycles of Al₂O₃ interlayer followed by nanolaminate with 12 cycles of 3:1 HfO₂:Al₂O₃ plus 5 cycles of Al₂O₃ interlayer (IL); (f) Nanolaminate with 12 cycles of 3:1 HfO₂:Al₂O₃ followed by 5 cycles of Al₂O₃ interlayer (IL); (f) Nanolaminate with 12 cycles of 3:1 HfO₂:Al₂O₃ followed by 5 cycles of Al₂O₃ interlayer (IL); (f) Nanolaminate with 12 cycles of 3:1 HfO₂:Al₂O₃ followed by 5 cycles of Al₂O₃ interlayer (IL); (f) Nanolaminate with 12 cycles of 3:1 HfO₂:Al₂O₃ followed by 5 cycles of Al₂O₃ cap;. D_{it} values in eV⁻¹.cm⁻² were calculated by conductance method.

Cross sectional scanning TEM with energy-dispersive x-ray spectroscopy (STEM-EDX) was performed (Fig. 4a-d) on a 5 cycles of 9:1 HfO₂: Al₂O₃ nanolaminate sample to determine the thickness of the interlayer between nanolaminate and SiGe. The TEM clearly shows an ~0.8nm thick interlayer of low atomic number between the AlHfOx and the SiGe (Fig 4a) meaning the nanolaminate does not directly bond to SiGe. EDS composition analysis (Fig. 4b and 4c) of the sub 1 nm interlayer shows that it consists of SiO₂ and contains almost no Ge nor Hf. The interlayer could be formed during the ALD or FGA process. Figure 4d is an EDX lines can across the interface of 9:1 Hf:Al

nanolaminate after FGA. The oxygen signal (green line) in the SiGe region was attributed to the thin substrate having surface oxidation when it was exposed to the air between FIB cross sectioning and TEM analysis. The 5 nm thick nanolaminate layer consisting of HfAlOx was formed on SiOx. The annealing process which creates the pure SiO₂ layer also induces Ge enrichment of the SiGe substrate surface (blue arrow in Fig 4d). The Ge/Si atomic weight percent ratio next to the interlayer is 2 times greater than in the bulk SiGe. The data is consistent that in the presence of the NL, the annealing of SiGeOx results in formation of a SiO₂ interlayer and reabsorption of Ge into the SiGe substrate: Si_xGe_yO_z \rightarrow SiO₂ + Ge. This is consistent with an ALD cleanup process in which TMA reduces GeOx to Ge and the Al₂O₃ in the nanolaminate reduces Ge and GeOx diffusion, so Ge is concentrated on the SiGe substrate (28), (29).



Figure 4: STEM-EDX of 5 cycles of 9:1 HfO₂: Al₂O₃ nanolaminate (a) STEM-EDX characterization of ~10% Al₂O₃ and ~90% HfO₂/ Si_{0.7}Ge_{0.3}(001). Blue box shows a region of interlayer for EDX analysis revealing a Ge-free SiOx interlayer. The orange box is a control region in the SiGe. (b) The EDX spectra of the interlayer is in blue while the spectra on the SiGe control region is in orange. Note the absence of Ge in the interlayer. (c) EDX analysis of SiOx layer region (blue box). Note that the oxide composition is SiO₂. (d) EDX line scan across the interface of 9:1 Hf:Al nanolaminate.

CONCLUSION

Al₂O₃/HfO₂ nanolaminate by thermal ALD has been applied to Si_{0.7}Ge_{0.3}(100) surface at 300 °C. Compared to pure HfO₂, use of the nanolaminate structures effectively reduced the density of interface traps more than 30% at the expense of a small drop in the accumulation capacitance. In addition, at least 3 orders of magnitude lower leakage currents were achieved compared to pure HfO₂ layer. Effects of Al₂O₃ layer as an interlayer and a capping layer were also studied. Comparison of deposition of Al₂O₃ between HfO₂ and SiGe (interlayer) vs on top of HfO₂ (cap) shows the lower D_{it} was achieved when the Al₂O₃ is in the interlayer instead of the capping layer. The data is consistent with Al₂O₃ acting as a diffusion barrier to Ge diffusion for the substrate or TMA clean-up effect reducing Ge oxides. While the nanolaminates do not have a distinct Al₂O₃ layer, the data is consistent with the incorporation of Al into HfO₂ improving the diffusion barrier properties thereby lowering the D_{it}. Cross sectional scanning TEM showed formation of SiOx interlayer between the AlHfOx and the SiGe. EDX spectra of the interlayer revealed that the SiOx layer contains almost no Ge showing the nanolaminate effectively prevents interfacial GeOx formation.

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