# EXPERIMENTAL INVESTIGATION OF ULTRA-LOW TEMPERATURE LA<sub>2</sub>O<sub>3</sub>/HFO<sub>2</sub> BI-LAYER DIPOLE-FIRST PROCESS USING PVD METHOD FOR ADVANCED IC TECHNOLOGY

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### ABSTRACT

In this paper, a La<sub>2</sub>O<sub>3</sub>/HfO<sub>2</sub> bi-layer dipole-first (DF) process is proposed and investigated by ultra-low temperature PVD dielectric laminates to achieve lower gate effective work function (EWF) for monolithic 3D-IC (M3D) application. The impacts of ultra-low temperature La-dipole on EWF modulation and interfacial properties are comprehensively investigated. It is found that the flat-band voltage (V<sub>FB</sub>) negatively shifts 60 mV with sub-1nm La<sub>2</sub>O<sub>3</sub> thickness, which provides an effective way to meet the require of Si conduction band-edge EWF modulation. Furthermore, the electron trap/detrap densities (Not) and interfacial trap densities (Dit) are suppressed by La<sub>2</sub>O<sub>3</sub>/HfO<sub>2</sub> bi-layer DF process to improve device performance. These results exhibit a promising bi-layer DF process in low thermal integration for advanced IC technology.

Keywords—Dipole-first; fine range  $V_{FB}$  modulation; low temperature; magnetron sputtering; La-dipole; La<sub>2</sub>O<sub>3</sub>

## **INTRODUCTION**

To meet the needs of high performance and low consumption requirements of transistors in high integration density circuits like monolithic 3D-IC (M3D), a low thermal budget and volume-less multiple threshold voltage (multi-Vt) technique is in need [1-2]. Dipole engineering is a promising approach for threshold voltage fine/wide modulation with less space using, and La<sub>2</sub>O<sub>3</sub> is the mainstream flat-band voltage  $(V_{FB})$  shifter for NMOSFETs as it provides Si conduction band-edge effective work function (EWF) by La-dipole formation [3]. For La-dipole formation, depositing La<sub>2</sub>O<sub>3</sub> directly on SiO<sub>2</sub> IL as dipole-first (DF) process and depositing La<sub>2</sub>O<sub>3</sub> on HfO<sub>2</sub> high-k layer with drive-in anneal as dipole-last (DL) process is the commonly utilized method. However, the wide range  $V_{FB}$  negative modulation obtained by DF process is superfluous for the need of fine range Vt modulation, and thermal budget in DL process for fine range V<sub>FB</sub> negative modulation is too high to be compatible with M3D application [3-5].

In this paper, we have experimentally investigated a



Figure 1: Key flow and schematic of the ultra-low temperature process gate stack. The thermal budget of HKMG structure is at room temperature.

La<sub>2</sub>O<sub>3</sub>/HfO<sub>2</sub> bi-layer dipole-first (DF) approach via ultra-low temperature PVD process. Less than 100 mV  $V_{FB}$  fine range negative modulation was achieved and precisely controlled by sub-1nm ultra-thin La<sub>2</sub>O<sub>3</sub> thickness. Meantime, the electron trap/detrap densities (Not) and interfacial trap densities (Dit) were suppressed by La<sub>2</sub>O<sub>3</sub>/HfO<sub>2</sub> bi-layer DF process to improve device performance.

### **EXPERIMENT AND FABRICATION**

Fig.1 shows the key flow and schematic of high-kmetal gate (HKMG) structure with La2O3/HfO2 laminated dielectric used for this study. First, an n-type Si (100) wafer with resistivity of 2-5  $\Omega$  cm was used as the substrate. After etching native oxide on wafer by buffered oxide etchant (BOE), a 1.3 nm SiO<sub>2</sub> interface layer (IL) was grown by in-situ steam generation (ISSG) process. For the  $La_2O_3/HfO_2$  laminated dielectric high-k layer, a room temperature magnetron sputtering process was utilized. 5 Å and 10 Å La<sub>2</sub>O<sub>3</sub> was respectively deposited on IL firstly. To keep the physical thickness of high-klaminated layer at 3 nm, 25 Å and 20 Å HfO<sub>2</sub> layer was sputtered on La<sub>2</sub>O<sub>3</sub> respectively. As a comparison, the high-k dielectric of reference sample is a pure 3-nm HfO<sub>2</sub> layer without La<sub>2</sub>O<sub>3</sub>. Then, a 2-nm TiN was sputtered on La<sub>2</sub>O<sub>3</sub>/HfO<sub>2</sub> laminated dielectric high-k layer as the metal gate. At last, a 100-nm gap-filling metal of Pt was sputtered. The TiN and Pt sputtering process temperature is at room temperature. No post deposition annealing



Figure 2: C-V curves of different thickness La-dipole devices and reference device via room temperature PVD.



Figure 3: Flat-band voltage and EOT value variations of devices with PVD  $La_2O_3$  in a 3-nm high-k stack.

(PDA) process and forming gas annealing (FGA) process were utilized. For device testing, the capacitance-voltage (C-V) characteristics were measured by Keithley 4200 semiconductor parameter analyzer in the air ambient at room temperature.

#### **RESULTS AND DISCUSSIONS**

The C-V curves of devices with different thickness of PVD  $La_2O_3$  are showed in Fig.2. The C-V curves were measured at 1 MHz frequency from inversion region to accumulation region. As a comparison to the reference device, the device with PVD  $La_2O_3$  in high-*k* stack shows an obvious negative shift on C-V curves. With the thickness of  $La_2O_3$  increasing from 5 Å to 10 Å, the C-V curve keeps negative shifting. The extracted V<sub>FB</sub> value from C-V curves are presented in Fig.3. V<sub>FB</sub> value of devices with PVD  $La_2O_3$  thickness of 0 Å, 5 Å, 10 Å is -0.04 V, -0.08 V, and -0.10 V, respectively. A maximum



Figure 4: The variation of (a) voltage hysteresis and (b) Not of devices with PVD  $La_2O_3$  in a 3-nm high-k stack.

of 60 mV  $V_{FB}$  negative shift was obtained by 10 Å PVD  $La_2O_3$  in high-*k* stack, which can be seen as a fine range  $V_{FB}$  modulation. The trend of  $V_{FB}$  negative shifting with the thickness of PVD  $La_2O_3$  increasing is basically linear, which shows that the precise fine range  $V_{FB}$  negative modulation is achieved by changing PVD La-dipole layer thickness under 1 nm.

The extracted equivalent oxide thickness (EOT) value from C-V curves is also showed in Fig.3. The EOT value of devices with PVD La<sub>2</sub>O<sub>3</sub> thickness of 0 Å, 5 Å, 10 Å is 1.21 nm, 2.10 nm, and 2.58 nm, respectively. As a comparison to the reference device, an around 1-nm EOT penalty was obtained by sputtered La<sub>2</sub>O<sub>3</sub> in high-*k* stack. The reason for EOT increasing can be considered as the low-*k* LaSiOx formation at La<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> interface [6]. Although the thermal budget of HKMG sputtering is ultra-low, La<sub>2</sub>O<sub>3</sub> is easily to react with SiO<sub>2</sub> [6], especially a sufficient 1.3-nm SiO<sub>2</sub> IL is presented. According to our prior work [7], EOT value and interface charge density (Nss) would cause small range of V<sub>FB</sub> shifts. Nevertheless, the dominant origin of V<sub>FB</sub> shifts in this experiment is La-dipole in high-*k* layer.

Considering the defect affected by La<sub>2</sub>O<sub>3</sub> in gate stack bulk and at interface, the trap/detrap electrons density (Not) and interface trap density (Dit) were investigated by the same C-V test method as our prior work [7]. Fig.4(a) shows the V<sub>FB</sub> hysteresis value extracted from dual-sweep C-V curves of devices with different thickness of PVD La<sub>2</sub>O<sub>3</sub> in gate stack. With the La<sub>2</sub>O<sub>3</sub> thickness increasing from 0 Å to 10 Å, the V<sub>FB</sub> hysteresis is decreased from 56.6 mV to 50.0 mV. In Fig.4(b), the Not value calculated by V<sub>FB</sub> hysteresis is decreased from 75.51 × 10<sup>10</sup> cm<sup>-2</sup> to 34.92 × 10<sup>10</sup> cm<sup>-2</sup> at the same time, indicating that 53.8 % trap/detrap electron density reducing is achieved by La<sub>2</sub>O<sub>3</sub> in gate stack. This result can be considered both by oxygen vacancy passivation effect of La<sub>2</sub>O<sub>3</sub> and thickness of HfO<sub>2</sub> with more oxygen vacancies decreasing [8].



Figure 5: The variation of (a)  $Gp/\omega$  with frequency at different gate voltage of device with 5 Å PVD  $La_2O_3$  and (b) Dit values.

For the interface quality, the conductance method with multi-frequency C-V measurement from 1 KHz to 1 MHz was applied, and the measured parallel conductance/frequency (Gp/ $\omega$ ) curve at different gate voltage is showed in Fig.5(a). Fig.5(b) presents the calculated Dit value of devices with different thickness of PVD La<sub>2</sub>O<sub>3</sub> in gate stack. With the La<sub>2</sub>O<sub>3</sub> thickness increasing from 0 Å to 10 Å, the Dit is decreased from  $1.54 \times 10^{13} \text{ eV}^{-1} \text{ cm}^{-2}$  to  $1.15 \times 10^{13} \text{ eV}^{-1} \text{ cm}^{-2}$ , indicating an obvious interface trap passivation effect by La<sub>2</sub>O<sub>3</sub> in gate stack [9]. Both Not and Dit decreasing exhibits an effective ability on defect suppression in gate stack bulk and at interface by ultra-low temperature magnetron sputtering La<sub>2</sub>O<sub>3</sub> in gate stack.

#### CONCLUSION

In this paper, a  $La_2O_3/HfO_2$  bi-layer DF process is proposed and comprehensively investigated by room temperature magnetron sputtering dielectric laminates. A 60 mV V<sub>FB</sub> fine range negative modulation is achieved by 10 Å PVD La<sub>2</sub>O<sub>3</sub>, and can be precisely controlled by La<sub>2</sub>O<sub>3</sub> thickness in sub-1nm. Furthermore, the Not and Dit are suppressed by La<sub>2</sub>O<sub>3</sub>/HfO<sub>2</sub> bi-layer DF process to improve interface trap and electron trap/detrap density. These results exhibit a promising bi-layer DF process in low thermal integration for advanced IC technology.

#### ACKNOWLEDGEMENTS

This work is supported by Beijing Natural Science Foundation program (Grant No. 4224096), Strategic Priority Research Program of the Chinese Academy of Sciences (Grant No. XDA0330302), and Beijing nova program (Grant No. Z201100006820084). The authors would like to thank all staff at the Integrated Circuit Advanced Process Center, IMECAS, for their support in the fabrication on 8-inch CMOS pilot line.

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