

# Al<sub>2</sub>O<sub>3</sub>/β-Ga<sub>2</sub>O<sub>3</sub>(-201) Interface Improvement Through Piranha Pretreatment and Postdeposition Annealing

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**Abstract**—In this letter, we report on the improvement of atomic layer deposited (ALD) Al<sub>2</sub>O<sub>3</sub>/β-Ga<sub>2</sub>O<sub>3</sub> (-201) interface quality through piranha pretreatment and postdeposition annealing (PDA). The high quality interface is verified via the temperature dependent capacitance–voltage (*C–V*) and photo-assisted (deep UV) *C–V* measurements, considering its ultra wide bandgap of 4.8 eV for β-Ga<sub>2</sub>O<sub>3</sub>. A low *C–V* hysteresis of 0.1 V from the measurement frequency of 1 kHz to 1 MHz is obtained, compared with the hysteresis of 0.45 V without piranha optimization. An average interface trap density (*D<sub>it</sub>*) of  $2.3 \times 10^{11} \text{ cm}^{-2} \cdot \text{eV}^{-1}$  is extracted from the photo *C–V* measurements. Piranha pretreatments and PDA turn out to be an effective way to improve the ALD Al<sub>2</sub>O<sub>3</sub>/β-Ga<sub>2</sub>O<sub>3</sub> (-201) interface for future high quality Ga<sub>2</sub>O<sub>3</sub> metal–oxide–semiconductor field-effect transistors.

**Index Terms**—β-Ga<sub>2</sub>O<sub>3</sub>, interface, *D<sub>it</sub>*, hysteresis, ALD Al<sub>2</sub>O<sub>3</sub>, piranha, annealing.

## I. INTRODUCTION

VERY recently, β-Ga<sub>2</sub>O<sub>3</sub> has been considered as a promising candidate for the next generation power electronics due to its ultra wide bandgap (*E<sub>g</sub>*) of 4.8 eV compared with SiC and GaN with bandgap of 3.2 and 3.4 eV, respectively [1]–[8]. Its 4.8 eV bandgap enables the β-Ga<sub>2</sub>O<sub>3</sub> to have a theoretical breakdown field (*E<sub>c</sub>*) of 8 MV/cm. Even at such early development stage, a high *E<sub>c</sub>* of 3.8 MV/cm has already been achieved, which exceeds the *E<sub>c</sub>* of GaN and SiC [9]. Combined with the 100 cm<sup>2</sup>/V·s room temperature electron mobility (*μ*), β-Ga<sub>2</sub>O<sub>3</sub> possesses a high Baliga’s figure of merit of 3444, defined as  $\epsilon\mu E_c^3$  [10]. In addition to its excellent material property, potential cost effective large size substrate can be realized through Czochralski method [11], [12]. However, unlike the GaN HEMT and MOSHEMT with buried channels, β-Ga<sub>2</sub>O<sub>3</sub> can only form a depletion-mode MOSFET so far. Therefore, gate dielectric/oxide interface plays an important role in forming high performance MOSFETs. In general, high-*k* dielectric, high conduction band offset, and high oxide/β-Ga<sub>2</sub>O<sub>3</sub>

interface quality are major concerns in terms of obtaining high performance device characteristics [13], [14].

There are several methods to determine the MOS interface trap densities (*D<sub>it</sub>*), such as Terman method, hi/lo frequency (*f*) method and AC conductance method [15], [16]. However, some inherent limitations exist when applying those methods to β-Ga<sub>2</sub>O<sub>3</sub> with 4.8 eV bandgap [17]. For instance, inaccurate estimation of the doping concentration and oxide capacitance and the negligence of deep energy level traps lead to underestimation of *D<sub>it</sub>* by comparing the ideal *C–V* curve with high *f* *C–V* curve. Hi/lo *f* method leads to significantly underestimate the *D<sub>it</sub>* also since it takes an extremely long time to generate holes in n-Ga<sub>2</sub>O<sub>3</sub> so that low *f* criteria is not satisfied. AC conductance method can only detect shallow energy level (0.2–0.6 eV) *D<sub>it</sub>* due to the limited *f* and temperature (*T*) ranges to detect the traps deeply inside the bandgap. In addition, the Ga<sub>2</sub>O<sub>3</sub> trap capture cross-section *σ* is still undetermined yet, which makes the trap energy level in the bandgap less accurate. Photo-assisted *C–V* takes advantage of the ultraviolet (UV) illumination to generate electron-hole pairs in the wide bandgap materials, ensuring that all the traps in the bandgap can respond during the measurement [18], [19]. By comparing the dark high *f* *C–V* curve with the post-UV *C–V* curve, an overall average *D<sub>it</sub>* can be obtained from the shift of the two curves.

## II. DEVICE FABRICATION AND MEASUREMENT

As received 2 inch Sn-doped β-Ga<sub>2</sub>O<sub>3</sub> (-201) was first diced into 6 mm by 6 mm small pieces. The diced samples have been treated with acetone, methanol and isopropanol solvent clean for 30 minutes. Before loading into ASM F-120 ALD chamber, 6 pieces of samples were first pretreated by piranha (98% H<sub>2</sub>SO<sub>4</sub>: 30% H<sub>2</sub>O<sub>2</sub> = 3 : 1) for 1 min. and DI water rinse. 15 nm of Al<sub>2</sub>O<sub>3</sub> was then deposited by ALD at 250 °C with tri-methyl-aluminum (TMA) and H<sub>2</sub>O as precursors. 2 pieces were then annealed in Jepelec rapid thermal annealing (RTA) furnace for 2 minutes under O<sub>2</sub> and N<sub>2</sub> atmosphere at 500 °C. 3 samples were annealed for 1 minute under O<sub>2</sub> atmosphere at elevated temperatures of 600, 700 and 800 °C, respectively. As a comparison purpose, 3 samples without piranha pretreatment were also deposited with 15 nm of Al<sub>2</sub>O<sub>3</sub> at 250 °C, and 2 of them were annealed under the same conditions as the 2 samples with piranha pretreatment above. Serial MOS capacitors (MOSCAPs) were then fabricated with photo

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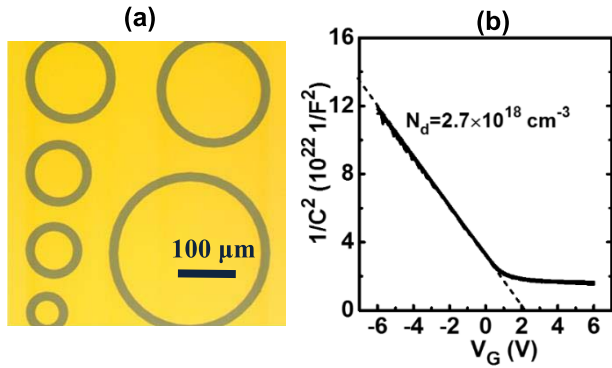


Fig. 1. (a) Top view of  $\text{Al}_2\text{O}_3/\beta\text{-Ga}_2\text{O}_3$  MOS serial capacitors and (b)  $1/C^2$  characteristic as a function of  $V_G$  with  $N_d = 2.7 \times 10^{18} \text{ cm}^{-3}$ .

lithography followed by Ti/Au (30/70 nm) lift-off process. A high precision HP4284 LCR meter was used for the capacitance measurement. Deep UV light with wavelength of 300 nm was used as the light source of the photo-assisted C-V measurements. The fabricated MOSCAPs were shown in Fig.1 (a), and the doping concentration ( $N_d$ ) of the  $\beta\text{-Ga}_2\text{O}_3$  is extracted to be  $2.7 \times 10^{18} \text{ cm}^{-3}$  from the slope of  $1/C^2$  [20]:

$$N_d = \frac{2}{q\epsilon_0\epsilon_s A^2 \frac{d(\frac{1}{C^2})}{dV}}$$

where the gate electrode area  $A$  is  $1.96 \times 10^{-5} \text{ cm}^2$ , and  $\epsilon_0$  and  $\epsilon_s$  of 9.5 are vacuum permittivity and relative dielectric constant of  $\beta\text{-Ga}_2\text{O}_3$ .

### III. RESULTS AND DISCUSSION

Fig. 2 shows the  $f$ -dependent C-V hysteresis measurements of two optimized samples with and without piranha pretreatments, respectively. The measurements start from depletion to accumulation and then sweep back to depletion. The existence of the interface and bulk traps leads to a shift of the flat-band voltage ( $V_{\text{FB}}$ ) due to the trapping and de-trapping at the bi-directional sweeps. The sweep rate for the C-V hysteresis measurement is 1.0 second per 0.1 V step and the forward to reverse sweep hold time is 10 seconds. With piranha pretreatment, the hysteresis is 0.1 V, while on the other hand the hysteresis is 0.45 V without piranha pretreatment. The trapped electrons or detectable interface trap quantity ( $Q_{\text{it}}$ ) can be roughly estimated through the  $V_{\text{FB}}$  shift by using the equation:  $Q_{\text{it}} = C_{\text{ox}} \times \Delta V/q$ , where  $C_{\text{ox}} = 0.5 \mu\text{F}/\text{cm}^2$  and  $\Delta V$  are the oxide capacitance and  $V_{\text{FB}}$  difference, respectively. The  $Q_{\text{it}}$  is reduced from  $1.4 \times 10^{12} \text{ cm}^{-2}$  without piranha treatment to  $3.2 \times 10^{11} \text{ cm}^{-2}$  after treatment. The piranha pretreatment is found to be useful in smoothing the sample surface and removing carbon-based organic contamination [21]. The root mean square (RMS) surface roughness after piranha is 0.17 nm, while the sample without piranha pretreatment is 0.26 nm, as shown in Fig.2 (c) and (d).

Fig.3 summarizes the post deposition annealing (PDA) influences on the piranha pretreated samples. Both room temperature and high temperature ( $T = 150 \text{ }^\circ\text{C}$ ) C-V curves are presented. Deep depletion behavior is observed due to the wide bandgap of  $\text{Ga}_2\text{O}_3$  so that inversion layer is not formed

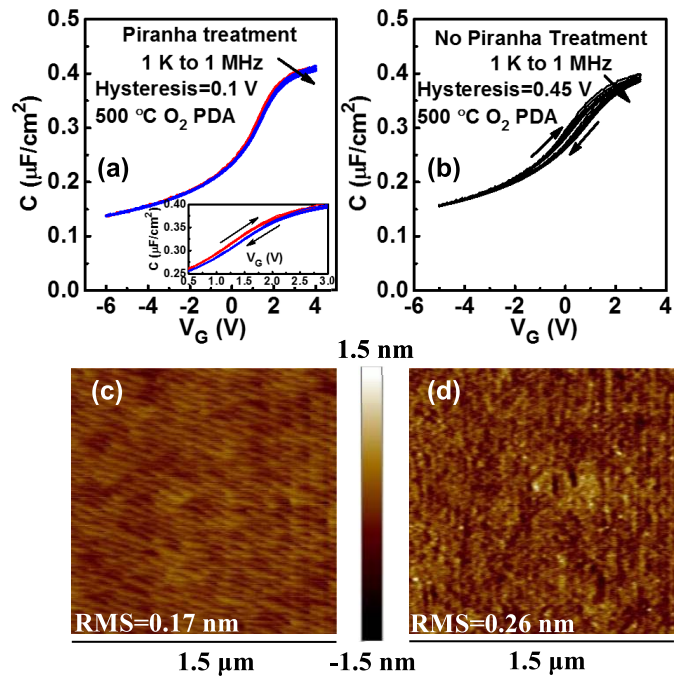


Fig. 2. C-V hysteresis measurement of 2 samples with  $500 \text{ }^\circ\text{C}$   $\text{O}_2$  annealing, (a) with piranha pretreatment and (b) without piranha pretreatment. Fig.2 (a) inset shows the zoomed in view of the C-V hysteresis measurement. (c) and (d) are atomic force microscopy images of  $\beta\text{-Ga}_2\text{O}_3$  with and without piranha treatment, respectively.

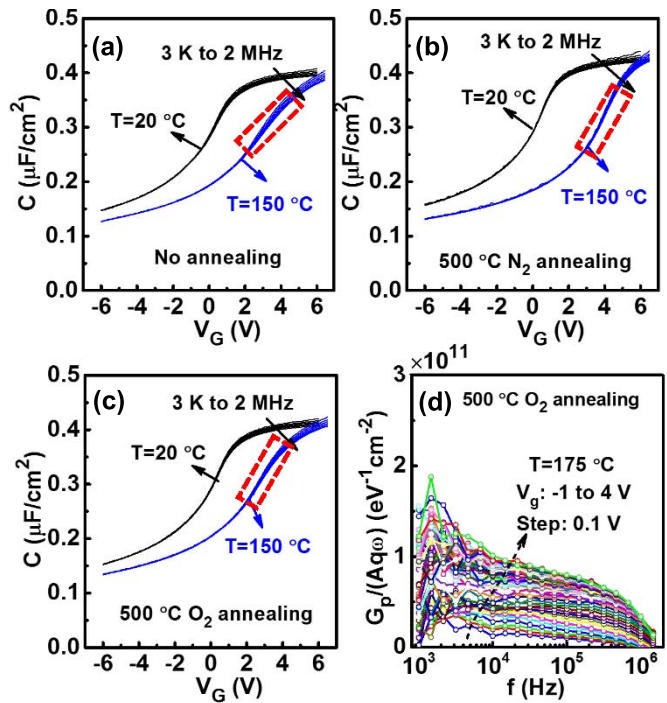


Fig. 3.  $f$ -dependent C-V measurements of different PDA MOS capacitors: (a) No annealing, (b)  $500 \text{ }^\circ\text{C}$   $\text{N}_2$  annealing and (c)  $500 \text{ }^\circ\text{C}$   $\text{O}_2$  annealing at  $T = 20 \text{ }^\circ\text{C}$  and  $T = 150 \text{ }^\circ\text{C}$  and (d) Extracted  $G_p/Aq\omega$  at  $T = 175 \text{ }^\circ\text{C}$  from AC conductance method. There is no  $G_p/\omega$  peak at such  $f$  and  $T$ . The red rectangles in (a), (b) and (c) highlight different  $f$ -dispersion under different PDAs. (c) has less annealing temperature dependent  $V_{\text{FB}}$  shift of 2.5 V compared to 3.5 V shift in (b).

at 3 kHz-2 MHz. At room temperature, there is no significant difference between unannealed and annealed samples with no obvious stretch out near depletion and slight frequency

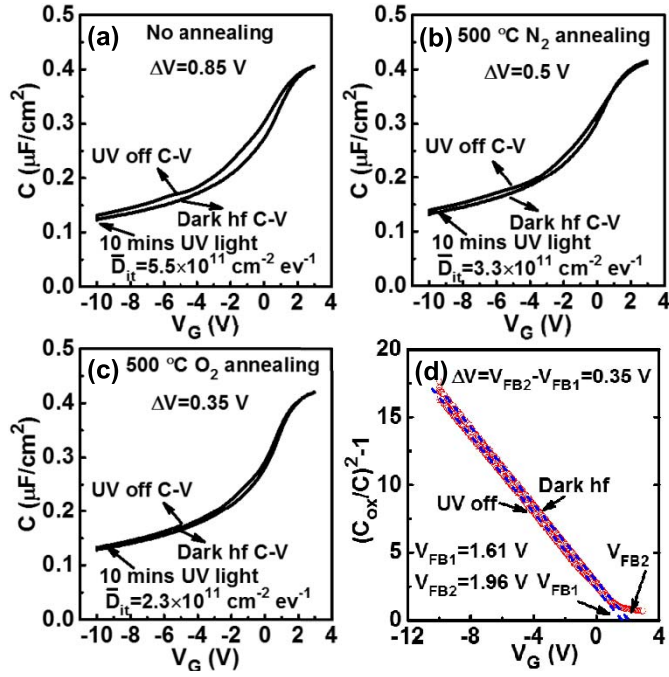


Fig. 4. Photo-assisted C-V measurements of different PDA MOS capacitors: (a) No annealing, (b) 500 °C N<sub>2</sub> annealing, (c) 500 °C N<sub>2</sub> annealing and (d)  $(C_{ox}/C)^2 - 1$  as a function of  $V_G$  to extract the  $V_{FB}$  of sample (c).

dispersion at accumulation. Compared with room temperature C-V curves, there is a right shift of high temperature C-V curves, showing the existence of negative charge at the Al<sub>2</sub>O<sub>3</sub>/Ga<sub>2</sub>O<sub>3</sub> interface. Similar right shift at high temperatures is also observed by Wong *et al.* [2]. Room temperature C-V measurement at accumulation region allow border traps capturing electrons and represent themselves as “negative” fixed charges, which leads the flatband voltage shifted to left compared to the ones measured at high temperatures [14]. One big issue for β-Ga<sub>2</sub>O<sub>3</sub> is its low thermal conductivity from room temperature to elevated temperatures, which degrades the device performances, such as electron mobility. The large flatband or  $V_T$  shifts are observed at elevated temperatures. At high temperatures, unannealed MOSCAP has higher frequency dispersion compared with annealed ones, as highlighted by the red rectangles, indicating that PDA can help to further increase the interface quality. Moreover, AC conductance method is also used to extract the  $D_{it}$  of the MOSCAP. The  $V_G$  is limited to the depletion of the MOSCAP. No obvious normalized conductance ( $G_p/Aq\omega$ ) peaks are observed even at  $T = 175$  °C. It is likely that even at this high temperature of 175 °C, the conductance method cannot detect deep trap energy levels in our experiment, since Ga<sub>2</sub>O<sub>3</sub> has a wide bandgap of 4.8 eV.

Because of the wide bandgap of Ga<sub>2</sub>O<sub>3</sub>, photo-assisted C-V method becomes a reliable approach to evaluate the MOS interface and extract the average  $D_{it}$ . The measurements started with biasing  $V_G$  at accumulation for 10 s to make sure traps are filled with electrons, and then sweeping  $V_G$  from deep depletion to accumulation at high  $f = 1$  MHz in dark. Then,  $V_G$  is kept biased at deep depletion with  $V_G = -10$  V and deep UV light is on for 10 minutes to generate electron-hole

pairs and forcing generated holes to move to the Al<sub>2</sub>O<sub>3</sub>/Ga<sub>2</sub>O<sub>3</sub> interface. After turning off the UV light and then sweeping the C-V from depletion to accumulation again in dark, a UV off C-V curve is obtained. UV generated holes recombine with those trapped electrons so that after UV exposure the interface is “donors” dominated compared to “acceptors” dominated interface without UV illumination. Therefore, a left shift of the C-V curves is observed and this  $V_{FB}$  shift can be translated into the average  $D_{it}$  by the equation:

$$D_{it} = \frac{C_{ox} \times \Delta V}{q \times E_g}$$

The  $V_{FB}$  is determined through the extrapolated line of the  $C_{ox}^2/C^2 - 1 \sim V_G$  at  $C_{ox}^2/C^2 - 1 = 0$ , as shown in Fig. 4 (d) [22]. Fig.4 (a)-(c) describes the originally measured photo-assisted C-V comparisons with dark high frequency C-V curves. The gate leakage currents at both sweeps are limited to 10  $\mu A/cm^2$ . Both the 500 °C N<sub>2</sub> and O<sub>2</sub> PDA can help to minimize the average  $D_{it}$ , which confirms that PDA is needed to optimize the Al<sub>2</sub>O<sub>3</sub>/β-Ga<sub>2</sub>O<sub>3</sub> interface. The improved interface quality after O<sub>2</sub> annealing is related to the compensation of oxygen vacancies [23]. The achieved minimal average  $D_{it}$  is  $2.3 \times 10^{11} \text{ cm}^{-2} \text{ eV}^{-1}$  with 0.35 V  $V_{FB}$  shift. Increased annealing temperature at 600, 700 and 800 °C starts to degrade the interface in our experiments. Both our work and the work from Zeng *et al.* [13] show a decent interface trap density less than  $10^{12} \text{ cm}^{-2} \text{ eV}^{-1}$ , which is favorable for device applications. Compared to Ref.[13], we have taken a further step to optimize the interface through piranha pretreatment and PDA, which might be useful to further improve β-Ga<sub>2</sub>O<sub>3</sub> MOS interface for device applications.

#### IV. CONCLUSION

We have investigated the piranha pretreatment and PDA effects on Al<sub>2</sub>O<sub>3</sub>/β-Ga<sub>2</sub>O<sub>3</sub> (-201) interface through frequency and temperature-dependent and photo-assisted C-V measurements. Low C-V hysteresis of 0.1 V, reduced high-temperature frequency dispersion and reduced  $V_{FB}$  shifts are benefited from the improved interface quality. Finally, a low average  $D_{it}$  of  $2.3 \times 10^{11} \text{ cm}^{-2} \text{ eV}^{-1}$  is achieved through photo-assisted C-V measurements. Piranha pretreatment and PDA process are demonstrated to effectively improve the Al<sub>2</sub>O<sub>3</sub>/β-Ga<sub>2</sub>O<sub>3</sub> interface, making the Al<sub>2</sub>O<sub>3</sub>/β-Ga<sub>2</sub>O<sub>3</sub> (-201) MOS structure possible for future power electronics.

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