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To cite this article: Mai Hattori et al 2016 Jpn. J. Appl. Phys. 55 1202B6

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Epitaxial growth and electric properties of γ -Al₂O₃(110) films on β -Ga₂O₃(010) substrates

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Received April 28, 2016; revised May 24, 2016; accepted June 5, 2016; published online October 21, 2016

Epitaxial growth and electrical properties of γ -Al₂O₃ films on β -Ga₂O₃(010) substrates were investigated regarding the prospect of a gate oxide in a β -Ga₂O₃-based MOSFET. The γ -Al₂O₃ films grew along the [110] direction and inherited the oxygen sublattice from β -Ga₂O₃ resulting in the unique in-plane epitaxial relationship of γ -Al₂O₃[10] || β -Ga₂O₃(001]. We found that the γ -Al₂O₃ layer had a band gap of 7.0 eV and a type-I band alignment with β -Ga₂O₃ with conduction- and valence-band offsets of 1.9 and 0.5 eV, respectively. A relatively high trap density ($\cong 2 \times 10^{12}$ cm⁻² eV⁻¹) was found from the voltage shift of photoassisted capacitance–voltage curves measured for a Au/ γ -Al₂O₃/ β -Ga₂O₃ MOS capacitor. These results indicate good structural and electric properties and some limitations hindering the better understanding of the role of the gate dielectrics (a γ -Al₂O₃ interface layer naturally crystallized from amorphous Al₂O₃) in the β -Ga₂O₃ MOSFET. (\bigcirc 2016 The Japan Society of Applied Physics

1. Introduction

Recently, β -Ga₂O₃ has been attracting attention as a candidate for future power electronics applications.^{1–3)} Owing to its wide band gap $(E_g = 4.4-4.6 \text{ eV})^{4-7}$ the breakdown electric field is expected to be as high as 8 MV/cm, which would lead to more than three times larger Baliga's figure of merit than those of 4H-SiC and GaN.⁸⁾ This is a tremendous advantage over its rivals from the viewpoint of practical use. Another advantage of β -Ga₂O₃ is the compatibility with current fabrication processes of semiconductor devices. Large and high-quality single crystals (≥ 2 in. in diameter) can be synthesized by melt growth methods such as Czochralski,⁹⁾ floating-zone,¹⁰⁾ and edge-defined film-fed growth.¹¹⁾ Consequently, high-quality β -Ga₂O₃ homoepitaxial films can be grown by molecular beam epitaxy^{12,13)} and halide vapor phase epitaxy.¹⁴⁾ Moreover, the electron density of the epitaxial films can be tuned by impurity levels by controlling the dose of ion implantation¹⁵⁾ as well as the source materials.^{2,13)} Using these and other conventional semiconductor-fabrication processes, metal-oxide-semiconductor field-effect transistors (MOSFETs) with a breakdown electric field as high as 755 V have been demonstrated.¹⁶⁾

The quality of the interface between a gate dielectric and β -Ga₂O₃ is a critical factor for the performance of β -Ga₂O₃based MOSFETs. In the aforementioned MOSFETs, atomiclayer-deposited amorphous-Al₂O₃ layers are used as gate dielectrics, and transistor channels are formed at the β -Ga₂O₃(010) surface.^{16,17)} By transmission electron microscopy, it has recently been revealed that a metastable γ -Al₂O₃ layer with nanometer thickness exists at the interface.¹⁸⁾ Interestingly, the density of interface trap states correlates with the thickness of the γ -Al₂O₃ layer. This finding suggests that a metastable γ -Al₂O₃ layer formed in contact with the β -Ga₂O₃ surface plays an important role in the device performance. However, the crystallization takes place naturally during device processing, and in such a case, the individual roles of γ -Al₂O₃ and amorphous-Al₂O₃ will be discussed only empirically. Moreover, the details of the crystal structure and physical properties of the γ -Al₂O₃/ β -Ga₂O₃ interface remain unclear. The preparation and characterization of a single-phase γ -Al₂O₃ epitaxial film on β -Ga₂O₃ is a direct approach to obtaining useful information for modeling this interface.

In this study, we fabricated γ -Al₂O₃ epitaxial films on β -Ga₂O₃(010) substrates by pulsed-laser deposition (PLD) and characterized the epitaxial relationship and the band alignment at the interface. We also investigated photoassisted capacitance–voltage (*C*–*V*) characteristics for MOS capacitors with contact metals.

The Al₂O₃ films were grown on unintentionally doped n-type β -Ga₂O₃(010) substrates by oxygen-radical-assisted PLD.¹⁹⁾ An Al₂O₃ ceramic target (99.99% purity) was ablated by focused KrF excimer laser pulses at a repetition rate of 10 Hz and a fluence of 0.3 J cm⁻². The ablated species were deposited on substrates heated at 500 °C and the oxygen radicals were supplied from a RF plasma cell operated at a RF power of 300 W and an O₂ gas flow rate of 0.70 sccm. The typical growth rate was 0.7–0.9 nm min⁻¹.

2. Experimental methods

The techniques and methods used to characterize the samples are as follows. The film thickness and epitaxial structure were evaluated using X-ray reflection and X-ray diffraction (XRD), respectively, with Cu K α_1 radiation (1.540562 Å). X-ray photoemission spectroscopy (XPS) measurements were performed using a VG-Scienta R3000 analyzer with a monochromatized Al K α X-ray source (1486.6 eV). The C 1s line of the adventitious carbon contamination (284.8 eV) was used to calibrate the binding energy. All spectra were acquired at room temperature with a total energy resolution of 400 meV. The E_g values were estimated by reflection electron energy loss spectroscopy (REELS) at a primary energy of 1 keV as well as from the O 1s loss structure in the XPS spectra. The photoassisted *C–V* measurement was conducted at a frequency of 1 MHz with the illumination



Gallium oxide and related semiconductors



Fig. 1. (Color online) (a) Out-of-plane XRD pattern of the γ -Al₂O₃ film grown on β -Ga₂O₃ substrate. S indicates a reflection from the sample stage. (b) Reciprocal space map around γ -Al₂O₃ 444 and β -Ga₂O₃ 420 diffraction spots. The coordinates of Q_X and Q_Z are orthogonal to the (100) and (010) surfaces of β -Ga₂O₃, respectively. A cross indicates γ -Al₂O₃ 444, corresponding to lattice parameters of bulk. (c) ϕ -scan profiles of asymmetric reflections from the γ -Al₂O₃ film and the β -Ga₂O₃ substrate.

of a Xe lamp (500 W) through a deep-UV optical fiber. The irradiated photon energies (up to 6.2 eV) were sufficient to excite the trapped electrons existing in the entire gap of β -Ga₂O₃. To excite all the trapped electrons, light illumination was continued until the photocapacitance saturated (the duration was about 2 min). For this measurement, ohmic contact was prepared prior to the γ -Al₂O₃ growth. A piece of indium metal put on the back of the β -Ga₂O₃ substrates was partially transferred to an ohmic contact layer through rapid thermal annealing in N₂ at 1000 °C for 2 min.²⁰⁾ After the growth, 10-nm-thick transparent Au electrodes with a diameter of 200 µm were vacuum-deposited through a stencil metal mask. A reference *C*–*V* curve was taken for a Schottky barrier diode fabricated by the same procedures. All the experiments were carried out at room temperature.

3. Results and discussion

Figure 1(a) shows the out-of-plane XRD pattern of a 28-nmthick Al₂O₃ film grown on the β -Ga₂O₃(010) substrate. Only the 440 reflection of γ -Al₂O₃ was observed, indicating that the metastable γ -Al₂O₃ phase was formed under a constraint from the surface lattice of β -Ga₂O₃(010). The full width at half maximum of the ω -rocking curve of this peak was 0.16°. Figure 1(b) shows a reciprocal space map of the sample, in which the coordinates of Q_X and Q_Z are orthogonal to the (100) and (010) surfaces of β -Ga₂O₃, respectively. The diffraction spots from the 420 reflection of β -Ga₂O₃ and the 444 reflection of γ -Al₂O₃ were clearly observed. Taking



Fig. 2. (Color online) Schematic drawings of oxygen atoms on γ -Al₂O₃(110) and β -Ga₂O₃(010), representing the observed in-plane epitaxial relationship. Double-headed arrows indicate spacings between each atomic row and d_{a^*} and d_c are their averages along a^* and c of β -Ga₂O₃, respectively.

this finding and the out-of-plane epitaxial relationship of γ -Al₂O₃(110) || β -Ga₂O₃(010) into account, the in-plane epitaxial relationship is γ -Al₂O₃[$\bar{1}$ 10] || β -Ga₂O₃[001]. Furthermore, these two diffractions from γ -Al₂O₃ 444 and β -Ga₂O₃ 420 exhibited similar ϕ -scan profiles with a twofold rotational symmetry [see Fig. 1(c)]. This result indicates that a single-crystalline γ -Al₂O₃ film was epitaxially grown on the β -Ga₂O₃(010) substrate.

Despite different symmetries of the unit cells (cubic γ -Al₂O₃²¹⁾ and monoclinic β -Ga₂O₃²²⁾), no other rotational domain was found, and thus the film consisted of only a single domain. This unique epitaxial relationship can be understood by taking the arrangements of oxygen sublattices at the film/substrate interface into account. We note that the crystal structures of γ -Al₂O₃ (as well as γ -Ga₂O₃) and β -Ga₂O₃ resemble each other and both are derived from a spinel-type structure (the former is often referred to as the defect spinel-type structure).²³⁾ Both of them have cubicclose-packed oxygen sublattices and cations occupy tetrahedral and octahedral sites. Figure 2 illustrates the observed epitaxial relationship with an emphasis on the oxygen sublattices on γ -Al₂O₃(110) and β -Ga₂O₃(010). Apparently, they are almost identical to each other. The mismatches of appropriate length in oxygen sublattices (L_{a^*} and L_c along the two orthogonal directions of a^* and c of β -Ga₂O₃, respectively) can be defined by the following equations using lattice parameters of γ -Al₂O₃ (a' = 7.911 Å)²¹⁾ and β -Ga₂O₃ (a =12.23, b = 3.04, c = 5.80 Å, and $\beta = 103.7^{\circ}$).²²⁾

$$L_{a^*} = \frac{d_{a^*}^{\text{Al}_2\text{O}_3} - d_{a^*}^{\text{Ga}_2\text{O}_3}}{d_{a^*}^{\text{Ga}_2\text{O}_3}} = \frac{\frac{\sqrt{2}}{4}a' - \frac{1}{2}c}{\frac{1}{2}c}$$

$$\approx -0.036$$

$$L_c = \frac{d_c^{\text{Al}_2\text{O}_3} - d_c^{\text{Ga}_2\text{O}_3}}{d_c^{\text{Ga}_2\text{O}_3}} = \frac{\frac{1}{2}a' - \frac{1}{3}a\sin\beta}{\frac{1}{3}a\sin\beta}$$

$$\approx -0.0013$$

Here, $d_{a^*}^{Al_2O_3}$, $d_c^{Al_2O_3}$, $d_{a^*}^{Ga_2O_3}$, and $d_c^{Ga_2O_3}$ are average spacings between atomic rows for γ -Al₂O₃ and β -Ga₂O₃ along the two directions of a^* and c of β -Ga₂O₃ (see Fig. 2 for details). We note a very small mismatch along the c-direction, which reduces the interface energy and promotes the oxygen



Fig. 3. (Color online) XPS spectra of (a) Ga $3p_{3/2}$ and a valence-band edge for the β -Ga₂O₃ substrate, (b) Al 2p and a valence-band edge for the 22-nm-thick γ -Al₂O₃ film, and (c) Ga $3p_{3/2}$ and Al 2p for the 2-nm-thick γ -Al₂O₃ film on the β -Ga₂O₃ substrate.

sublattices to connect across the interface with preferential orientation.

The band alignment at γ -Al₂O₃/ β -Ga₂O₃ was investigated by a standard procedure using XPS.²⁴) We used three samples, i.e., a pristine β -Ga₂O₃ substrate, a 22-nm-thick γ -Al₂O₃ film, and a 2-nm-thick γ -Al₂O₃ film grown on β -Ga₂O₃ substrates, to analyze signals independently generated from β -Ga₂O₃, γ -Al₂O₃, and their interface, respectively.

We first determined a valence-band offset (ΔE_V) at the γ -Al₂O₃/ β -Ga₂O₃ interface. Figures 3(a)–3(c) show XPS spectra for the three samples. In these spectra, peak energies of core levels were determined by fitting to a Gaussian function, while valence band maximums (VBMs) were estimated by the linear extrapolation of the valence-band edge. By comparing energy differences between the core levels and the VBMs for three spectra, ΔE_V can be extracted as

$$\Delta E_{\rm V} = (E_{\rm Ga3p_{3/2}}^{\rm Ga_2O_3} - E_{\rm VBM}^{\rm Ga_2O_3}) - (E_{\rm Al2p}^{\rm Al_2O_3} - E_{\rm VBM}^{\rm Al_2O_3}) - (E_{\rm Ga3p_{3/2}}^{\rm Al_2O_3} - E_{\rm Al2p}^{\rm Al_2O_3})$$
$$= (E_{\rm Ga3p_{3/2}}^{\rm Interface} - E_{\rm Al2p}^{\rm Interface})$$
$$\cong 0.5 \, {\rm eV},$$

where $E_{\text{Ga2}O_3}^{\text{Ga2}O_3}$ and $E_{\text{VBM}}^{\text{Ga2}O_3}$ are the peak energies of Ga $3p_{3/2}$ and VBM for β -Ga₂O₃, respectively, $E_{\text{Al2}O}^{\text{Al2}O_3}$ and $E_{\text{VBM}}^{\text{Al2}O_3}$ are the peak energies of Al 2p and VBM for γ -Al₂O₃, respectively, and $E_{\text{Ga3}p_{3/2}}^{\text{Interface}}$ are the peak energies of Ga $3p_{3/2}$ and Al 2p at the interface, respectively.



Fig. 4. (Color online) XPS O 1s loss spectra for (a) the β -Ga₂O₃ substrate and (b) the 22-nm-thick γ -Al₂O₃ film, respectively. (c) and (d) are REELS spectra for the same samples as (a) and (b), respectively.

Next, we evaluated $E_{\rm g}$ to determine the conduction-band offset ($\Delta E_{\rm C}$). We obtained two sets of XPS and REELS spectra to independently determine the E_g of β -Ga₂O₃ and γ -Al₂O₃. Figures 4(a) and 4(b) show XPS spectra near O 1s core levels for the β -Ga₂O₃ substrate and 22-nm-thick γ -Al₂O₃ film, respectively. In these spectra, the energy difference between the onset of the loss spectrum and the peak energy of the core level corresponds to E_g ; E_g values for β -Ga₂O₃ ($E_g^{Ga_2O_3}$) and γ -Al₂O₃ ($E_g^{Al_2O_3}$) were determined to be 4.6 and 7.0 eV, respectively. We paid attention to the poor signal-to-noise (S/N) ratios, which possibly cause statistical error for an estimate of E_{g} , in both XPS spectra. Therefore, E_{g} was also evaluated by REELS, a technique often employed to evaluate the E_g of gate dielectrics.²⁵⁾ Figures 4(c) and 4(d) show REELS spectra of the β -Ga₂O₃ substrate and γ -Al₂O₃ film, respectively. The energy difference between a primary peak and the onset energy of the loss spectrum corresponds to the minimum energy of the electronic transition from the valence band to the conduction band, that is, E_{g} . In this case, S/N ratios of the spectra were sufficiently large to analyze characteristic energies. $E_{a}^{Ga_2O_3}$ and $E_{a}^{Al_2O_3}$ were also determined to be 4.6 and 7.0 eV, respectively, which coincide with the values determined from XPS spectra. The obtained $E_{g}^{Ga_{2}O_{3}}$ and $E_{\sigma}^{Al_2O_3}$ showed good agreement with the reported values of 4.4–4.6^{4–7)} and 7.1 eV,²⁶⁾ respectively. Having determined $\Delta E_{\rm V}$, $E_{\rm g}^{\rm Ga_2O_3}$, and $E_{\rm g}^{\rm Al_2O_3}$, we calculated $\Delta E_{\rm C}$ using

$$\Delta E_{\rm C} = (E_{\rm g}^{\rm Al_2O_3}) - (E_{\rm g}^{\rm Ga_2O_3}) - \Delta E_{\rm V}$$

$$\approx 1.9 \,\text{eV}.$$

The band alignment at the γ -Al₂O₃/ β -Ga₂O₃ interface was thereby revealed to be type-I with $\Delta E_{\rm C} = 1.9 \,\text{eV}$ and $\Delta E_{\rm V} = 0.5 \,\text{eV}$, which are comparable to those at the amorphous-



Fig. 5. (Color online) Photoassisted *C*–*V* curves measured at 1 MHz for (a) Au/γ -Al₂O₃/ β -Ga₂O₃ MOS capacitor and (b) Au/β -Ga₂O₃ Schottky barrier diode. Insets show schematic cross sections of the sample structures.

Al₂O₃/ β -Ga₂O₃ interface [$\Delta E_{\rm C} = (1.5-1.6) \pm 0.2 \,\text{eV}, \, \Delta E_{\rm V} = 0.7 \pm 0.2 \,\text{eV}$].²⁷⁾

Finally, we describe the electrical properties of the γ -Al₂O₃/ β -Ga₂O₃ interface. Figure 5(a) shows a photoassisted C-V curve measured for a Au/ γ -Al₂O₃/ β -Ga₂O₃ MOS capacitor. First, the carrier accumulation and deep depletion behaviors were clearly observed at positive and negative biases, respectively, which are common features of wide-band-gap-semiconductor-based MOS capacitors. From the saturation value at a positive bias, which corresponds to the capacitance of the γ -Al₂O₃ layer (C_{ox}), the relative dielectric constant of γ -Al₂O₃ was calculated to be 6.9 ± 0.2. This value is comparable to the reported value of 7.²⁸⁾ Next, we compared two curves measured with voltage swept down voltage 5 to -15 V in the dark and up to 5 V in the dark after all the trapped electrons were eliminated under illumination at -15 V. In this case, charging and discharging of the average trap states appear in a voltage shift (ΔV) .²⁹⁾ Considering the relatively small $\Delta V \sim 1 V$ observed for the Schottky barrier diodes [see Fig. 5(b)], the $\Delta V \sim 8 V$ for the MOS capacitors is attributed not to the bulk β -Ga₂O₃ but rather to the γ -Al₂O₃/ β -Ga₂O₃ interface and/or γ -Al₂O₃. Assuming that the majority of trap states exists at the interface, as is the case for SiO₂/SiC²⁹⁾ and AlN/GaN,³⁰⁾ the average density of trap states (D_{trap}) can be estimated using²⁹⁾

$$D_{\text{trap}} = \frac{C_{\text{ox}}\Delta V}{qE_{\text{g}}^{\text{Ga}_2\text{O}_3}}$$
$$\cong 2 \times 10^{12} \text{ cm}^{-2} \text{ eV}^{-1},$$

where *q* is the electron charge. The estimated D_{trap} is much larger than those of SiO₂/SiC ($1.42 \times 10^{11} \text{ cm}^{-2} \text{ eV}^{-1}$)²⁹⁾ and AlN/GaN (9.5×10^{10} – $1.4 \times 10^{11} \text{ cm}^{-2} \text{ eV}^{-1}$).³⁰⁾ The relatively large D_{trap} for γ -Al₂O₃/ β -Ga₂O₃ may be attributed to different crystal structures (namely, relatively large mismatch along the *a**-axis) and/or surface damage due to high-energy precursors during PLD.

4. Conclusions

We fabricated single-phase crystalline γ -Al₂O₃ epitaxial films on β -Ga₂O₃(010) substrates to investigate the electric properties of the γ -Al₂O₃/ β -Ga₂O₃ interface as a model of β -Ga₂O₃ MOSFETs. The epitaxial relationship between γ -Al₂O₃ and β -Ga₂O₃ was clarified, where we observed similar arrangements of oxygen sublattices between γ -Al₂O₃(110) and β -Ga₂O₃(010) surfaces. The ΔE_V and ΔE_C of the γ -Al₂O₃/ β -Ga₂O₃ interface were revealed to be comparable to those of the amorphous-Al₂O₃/ β -Ga₂O₃ interface. Despite good structural and electric properties, trap states ($\cong 2 \times 10^{12} \text{ cm}^{-2} \text{ eV}^{-1}$) were detected by photoassisted *C*-*V* measurement. Whether this behavior is intrinsic to the present epitaxial structure or arises from the fabrication process employed in this study remains an open question.

Acknowledgments

The authors thank M. Tada of Center for Advanced Materials Analysis for assistance with REELS, and M. Higashiwaki and T. Kamimura of National Institute of Information and Communications Technology for valuable discussions. This work was supported by a Grant-in-Aid for Scientific Research (No. 26709020) from the Japan Society for the Promotion of Science Foundation, MEXT, the Element Strategy Initiative Project to Form Core Research Center, and a research grant from the Murata Science Foundation.

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