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Realization of Conductive n-Type Doped α -Ga₂O₃ on *m*-Plane Sapphire Grown by a Two-Step Pulsed Laser Deposition Process

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Structural and electrical properties of undoped and doped α -Ga₂O₃ thin films grown by pulsed laser deposition on *m*-plane sapphire in a two-step process are presented. A buffer layer of undoped α -Ga₂O₃ is introduced below the electrically active thin film to improve the crystal quality and enable the stabilization of the α -phase at lower substrate temperatures for sufficient dopant incorporation. Donor doping of the active layers with tin, germanium, and silicon, respectively, is realized below a critical substrate temperature of 600 °C. Depth-resolved X-ray photoelectron spectroscopy measurements on tin-doped samples reveal a lower amount of tin in the bulk thin film compared to the surface and a lower tin incorporation for higher substrate temperatures, indicating desorption or float-up processes that determine the dopant incorporation. Electron mobilities as high as 17 cm² V⁻¹ s⁻¹ (at 1.4×10^{19} cm⁻³) and 37 cm² V⁻¹ s⁻¹ (at 3.7×10^{18} cm⁻³) are achieved for tin- and germanium doping, respectively. Further, a narrow window of suitable annealing temperature from 680 to 700 K for obtaining ohmic Ti/Al/Au layer stacks is identified. For higher annealing temperatures, a deterioration of the electrical properties of the thin films is observed suggesting the need for developing low temperature contacting procedures for α -Ga₂O₃-based devices.

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1. Introduction

The wide bandgap material Ga₂O₃ is, due to its large optical band gap of 4.6-5.3 eV,^[1] of special interest for use in high-power transistors,^[2,3] transparent conductive thin films,^[4] and solar blind photodetectors.^[2] Especially the thermodynamically stable β -polymorph of Ga₂O₃ has been extensively studied due to the availability of large single-crystalline wafers for homoepitaxial devices and a large theoretical electric breakdown field of 8 MV cm^{-1} .^[2,3] However, up to now, only devices with a breakdown field as high as 5.5 MV cm⁻¹ have been demonstrated.^[5,6] Another auspicious polymorph of Ga2O3 is the corundum-structured α -phase, which has lower thermodynamic stability compared to the β - and κ -polymorphs of gallium oxide, but exhibits the highest bandgap of all Ga2O3 phases of about 5.3 eV.^[1] Similar to the β -phase of Ga₂O₃, it can be used in highpower electronics with even higher

expected electric breakdown fields due to the increased bandgap.^[7,8] Another advantage of this phase is the availability of cost effective, isostructural α -Al₂O₃ substrates providing rather low lattice mismatch. Numerous reports exist on the deposition of α -Ga₂O₃ by various deposition techniques such as mist chemical vapor deposition (CVD),^[9–11] molecular beam epitaxy (MBE),^[12] and pulsed laser deposition (PLD).^[13–15]

The deposition of α -Ga₂O₃ by PLD has only recently been described. Homoepitaxially grown $\alpha - (Al_{1-x}Ga_x)_2O_3$ with aluminum contents 0 < x < 0.08 deposited by PLD were described in 2018 by Lorenz et al.^[13] In 2020, Hassa et al. presented results on the deposition of $\alpha - (Al_{1-x}Ga_x)_2O_3$ by PLD in the entire composition range.^[1,15]

Grundmann et al. performed calculations and experimental work on the stress–strain relation of $\alpha - (Al_{1-x}Ga_x)_2O_3$ grown by PLD.^[14,16–18] However, up to now, no reports on doped and conductive α -Ga₂O₃ deposited by PLD exist to the best knowledge of the authors.

Typically, a low dislocation density in α -Ga₂O₃ thin films on sapphire is required to achieve high electron mobilities.^[19] Different approaches have been reported to decrease the dislocation density and therewith improve the structural and

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electrical thin-film properties. An epitaxial lateral overgrowth approach^[20–22] as well as buffer layers have been employed. $\alpha - (Al_xGa_{1-x})_2O_3^{[23-25]}$ buffer layers with a compositional gradient have been successfully used to improve the thin-film properties of α -Ga₂O₃ thin films. A second approach is the use of buffer layers with a homogeneous composition. $\alpha - (Al_xGa_{1-x})_2O_3$,^[23,26] annealed Sn-doped $\alpha - Ga_2O_3$,^[27] or unintentionally doped α -Ga₂O₃^[28] has been reported as homogeneous buffer layers.

Using *m*-plane sapphire substrates, electron mobilities as high as 51 and 65 cm² V⁻¹ s⁻¹ have been reported for Si-doped and Sn-doped α -Ga₂O₃ thin films, respectively.^[19,28] The electron mobility of α -Ga₂O₃ thin films grown on c-plane sapphire is significantly lower.^[29]

Following the approach presented by Akaiwa et al. in 2020 for mist-CVD,^[28] we present undoped and doped α -Ga₂O₃ thin films deposited by PLD on *m*-plane sapphire substrates. To enable a deposition of the thin films at lower substrate temperatures, an insulating, nominally undoped α -Ga₂O₃ buffer layer was deposited first. The structural and electrical properties of the thin films for doping with tin, germanium, and silicon are demonstrated in dependence on the substrate temperature during the deposition.

Finally, the electrical properties of thin films upon annealing are investigated in detail for tin-doped Ga_2O_3 and the implications for α -Ga₂O₃-based devices are discussed.

2. Experimental Results

Previous experiments in our work group have shown that the α -polymorph forms for very low deposition pressures of 3×10^{-4} mbar O₂ and high substrate temperatures of 700 °C only.^[1] However, lower substrate temperatures are desired to achieve conductive thin films as Uchida et al. and Akaiwa et al. have shown. There, annealing of tin-doped α -Ga₂O₃ thin films led to an insulating behavior. This is either due to an evaporation of tin from the thin film, a migration of tin to dislocations, or changes in its charge state from Sn⁴⁺ to Sn²⁺, where the latter acts as acceptor leading to compensation instead of n-type doping.^[27,30] We assume high substrate temperature inhibits the incorporation of the dopant into the crystal lattice, especially for tin doping. Further, a deposition at high substrate temperatures may lead to an evaporation or float up of the dopant, as it has been reported in the case of tin for κ -Ga₂O₃.^[31] It was, therefore, our aim to enable the deposition of our thin films at lower substrate temperatures. However, deposition of Ga₂O₃ by PLD at lower substrate temperatures leads to the formation of the β -phase.^[1,31]

We use a buffer layer of α -Ga₂O₃ as seed layer to expand the growth window of the alpha phase toward lower substrate temperatures $T_{\rm S}$. To investigate the growth of α -Ga₂O₃ grown at lower substrate temperatures and to determine the optimal thickness of the buffer layer, we followed the proposition of Akaiwa et al.^[28]. Nominally undoped buffer layers with different thicknesses of 75 to 450 nm were deposited under optimal growth condition of $T_{\rm s} = 700$ °C on *m*-plane sapphire. Subsequently, a top-layer Ga₂O₃thin film with a thickness of \approx 250 nm was in situ deposited at a lower substrate temperature



Figure 1. $2\theta - \omega$ X-ray diffractogram for three different buffer layer thicknesses between 75 and 450 nm of a nominally undoped buffer layer deposited at $T_S = 700$ °C and a ≈ 200 nm thick undoped top layer deposited at $T_S = 610$ °C. On the right hand side, the full width at half maximum (FWHM) obtained from omega scans in dependence on the buffer layer thickness is depicted.

of $T_{\rm s} = 610$ °C. In **Figure 1**, the X-ray diffraction (XRD) patterns for three thin films with different thicknesses of the buffer layer are depicted. A splitting of the (3 0 0) α -Ga₂O₃ peak into the k_{α_1} and k_{α_2} peak is visible. The 2θ full width half maximum (FWHM) of the (3 0 0) α -Ga₂O₃ reflection, obtained from omega scans, decreases from a value of 0.526°–0.498° for buffer thicknesses of 450 and 75 nm, respectively. It was therefore possible to achieve high-quality α -Ga₂O₃ thin films at a lower substrate temperature, using this approach.

A buffer layer grown at 700 $^\circ \rm C$ with a thickness of 75 nm was used for all further experiments.

Using these results, doped thin films were in situ deposited on undoped α -Ga₂O₃ buffer layers. In Figure 2a, XRD diffractograms for doped samples deposited at a substrate temperature of $T_s = 540$ °C are shown. Silicon- and tin-doped thin films were deposited at temperatures of 420 - 580 °C. Germanium-doped thin films were deposited at temperatures of 510-580 °C (see Figure S1, Supporting Information). For all samples, a $K_{\alpha 1}/K_{\alpha 2}$ splitting of the (3 0 0) reflection of α -Ga₂O₃ is visible and very small 2θ FWHM of 0.09° (Ge-doping, $T_s = 510$ °C), 0.11° (Sn-doping, $T_{\rm S} = 580$ °C), and 0.12° (Si-doping, $T_{\rm S} = 540$ °C) were obtained. The FWHM increased with decreasing substrate temperature (compare Figure 2b). In Figure 2c, the FWHM obtained from omega scans is depicted for tin-doped thin films in dependence on the growth temperature. The values range between 1500" and 2000", which is comparable to the values obtained by Akaiwa et al. for tin-doped α -Ga₂O₃ thin films.^[28] The surface crystallinity of the doped layers was investigated by reflection high-energy electron diffraction (RHEED) measurements (see Figure S2, Supporting Information, for exemplary RHEED images). For germanium and silicon doping, the doped layer showed no in-plane orientation for $T_{\rm S} \leq 510$ °C. For the Sn-doped top layer in-plane orientation was observed for substrate temperatures as low as 470 °C. It has to be noted that the formation of the additional phases





Figure 2. $2\theta - \omega$ X-ray diffraction (XRD) pattern for silicon-, tin-, and germanium-doped Ga₂O₃ thin films deposited at a substrate temperature of 540 °C. a) All doped thin films were deposited on 75 nm thick, undoped α -Ga₂O₃, b) 2θ FWHM for the three different dopants in dependence on the substrate temperature during thin-film growth, and c) FWHM obtained from omega scans for tin-doped thin films.

was suppressed for all three dopants even at temperatures below 600 $^\circ\text{C}.$

The Sn-doped thin films were \approx 500–900 nm thick with increasing thickness for decreasing $T_{\rm S}$, probably caused by the increased formation of volatile gallium suboxides at higher substrate temperatures and low oxygen partial pressure.^[32,33] The thickness of the Si-doped thin films was \approx 480 nm independent of the deposition temperature. Thicknesses of 650 and 550 nm were obtained for the Ge-doped thin films for deposition temperatures of 510 and 540 °C, respectively.

As previously shown for $\kappa - Ga_2O_3$:Sn, tin is not homogeneously incorporated in thin films grown by PLD, therefore depth-resolved X-ray photoelectron spectroscopy (XPS) measurements were used to investigate if this is also the case for α -Ga₂O₃. The measurements were performed to investigate the influence of the substrate temperature on the doping level of two tin-doped samples. The doped layers were deposited at 470 and 580 °C. In **Figure 3**, the depth-resolved elemental composition is depicted.

For both thin films, a higher tin content on the surface of the sample is evident compared to the bulk thin film. This corresponds well to the findings of Kneiß et al., who observed a similar float up of tin in PLD grown $\kappa - Ga_2O_3$.^[31] The tin content on the



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Figure 3. Elemental composition as function of the sputtering time for an active layer deposited at 580 $^\circ$ C (diamonds) and 470 $^\circ$ C (circles determined by XPS).

thin film surface is ≈ 1 at%, which corresponds very well to the tin concentration in the ceramic PLD target.

The tin content inside the thin film is significantly higher for a deposition temperature of 470 °C (0.11 at%), compared to 580 °C (0.02 at%). This corresponds very well to previous reports, where annealing of the thin films at different temperatures led to similar decrease of the tin content.^[27,30]

All thin films were characterized electrically. In **Figure 4**a, the resistivity in dependence on the substrate temperature during the deposition of the active layer is shown for the three dopants.

The lowest resistivity is achieved for tin doping, where it is as low as $\rho = 2.7 \times 10^{-2} \,\Omega$ cm for a substrate temperature of $T_{\rm S} = 540$ °C. For higher and lower substrate temperatures, the resistivity of the tin-doped thin films increases and for temperatures $T_{\rm S} > 580$ °C, the resistivity exceeded 10⁶ Ω cm and could not be measured with our setup. The germanium-doped thin film exhibits a very similar resistivity of $\rho = 4.4 \times 10^{-4} \,\Omega$ cm at $T_{\rm S} = 540$ °C. Similar as for the tin-doped thin film, the resistivity at $T_{\rm S} = 580$ °C has been found to exceed 10⁶ Ω cm and could not be measured with our setup.

The silicon-doped thin films exhibit an overall higher resistivity than the tin and germanium-doped $\alpha - \text{Ga}_2\text{O}_3$ thin films. The smallest resistivity of $\rho = 2.5 \times 10^{-1} \,\Omega \,\text{cm}$ was obtained for $T_{\rm S} = 470 \,^{\circ}\text{C}$. The free carrier density and electron mobility could only be determined for $T_{\rm S} = 470 \,^{\circ}\text{C}$ for the silicon-doped thin films, where they were $4.8 \times 10^{18} \,\text{cm}^{-3}$ and $4 \,\text{cm}^2 \,\text{V}^{-1} \,\text{s}^{-1}$, respectively. For all other deposition temperatures, the Hall constant switched sign during the measurement indicating that the actual Hall constant is below the noise level of the setup used. This is likely due to an electron mobility below $1 \,\text{cm}^2 \,\text{V}^{-1} \,\text{s}^{-1}$.

The higher resistivity compared to the tin- and germaniumdoped thin films could be due to the much lower dopant concentration within this target. However, using targets with higher silicon contents led to the growth of nonconductive layers only.

The free carrier density of the tin-doped thin films increased with increasing substrate temperature up to 550 °C, even though the incorporated tin content decreased, compare Figure 4b. The free carrier density varied between 1×10^{18} and 1.4×10^{19} cm⁻³. The increase of the carrier density with increasing substrate

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Figure 4. a) Resistivity ρ , b) free carrier density *n*, and c) electron mobility μ determined by Hall-effect measurements at room temperature for Si (circles), Sn (squares), and Ge (diamonds) doping in dependence on the substrate temperature during the deposition. The noncolored region denotes the growth temperature regime leading to in-plane-oriented thin films.

temperature could therefore be due to an improved crystal quality and consequently a lower density of trap-like defects or a change in valence. The conductivity of the active layer is undoubtedly induced by the dopants as undoped α -Ga₂O₃ thin films deposited at similar conditions on the buffer layer did not exhibit a measurable electrical current flow. The here found carrier density is very close to the yet highest reported carrier density of 2×10^{19} cm⁻³ for tin-doped α -Ga₂O₃ thin films.^[28] Using the free carrier density *n* obtained by Hall-effect measurements and the dopant concentration $N_{\rm D}$ obtained from the XPS measurements, we approximated the doping efficiency $r_{\rm Dop}$ as

$$r_{\rm Dop} = \frac{n}{N_{\rm D}} \tag{1}$$

This yields a comparatively low doping efficiency of 1.7×10^{-2} and 5.8×10^{-4} for the thin films grown at 580 and 470 °C, respectively. In Figure 4c, the electron mobility is depicted in dependence on the substrate temperature during the deposition. The highest electron mobility of $37 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ at a carrier density of $3.7 \times 10^{18} \text{ cm}^{-3}$ is achieved for a germanium-doped thin film deposited at $T_{\rm S} = 540$ °C. The silicon-doped thin film has an electron mobility of $4 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. For the tin-doped thin films, the highest electron mobility of $17 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ is measured for the thin film deposited at $T_{\rm S} = 540$ °C. The electron mobility decreases for higher and lower substrate temperatures. The decrease for lower substrate temperatures can be attributed to the reduced crystal quality, whereas the decrease for higher substrate temperatures is not yet understood.

As Uchida et al. and Akaiwa et al. described, tin-doped α -Ga₂O₃ thin films become insulating upon annealing.^[27,30] However, a certain annealing is required to achieve ohmic Ti/Al/Au contacts.^[34,35] For β -Ga₂O₃, Peterson et al. showed that upon annealing of the Ti/Au contact, a thin defective Ga₂O₃ layer forms at the interface and that the titanium contact is partially

oxidized.^[34] To gauge the influence of thermal annealing on the contact and electrical properties of a Sn-doped α -Ga₂O₃ thin film, we performed temperature-dependent Hall-effect measurements. Therefore, we in situ annealed the samples in the setup at a certain temperature. The resistivity, free carrier density and electron mobility were measured and the ohmic behavior of the contacts was probed by measuring the differential resistance. In **Figure 5**a, the resistivity measured at 360 K of a tin-doped thin film (grown at 510 °C) after each annealing step is depicted. The thermal annealing in our Hall-effect setup was performed in vacuum.

The high-temperature Hall-effect measurement will be referred to as annealing in the following. Up to an annealing temperature of 680 K, the Ti/Al/Au contacts exhibited non-ohmic behavior, as confirmed by the measurement of the voltage-dependent differential resistance. For temperatures above 680 K, the contacts became ohmic. A strong increase of the resistivity of the thin film is observed for annealing temperatures above 700 K. This is also seen in a sharp decrease of the free carrier density and a decrease of the electron mobility (compare Figure 5b,c). The electron mobility drops from a value of $37 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ in the as-deposited state to $8 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ for an annealing at 780 K. The strong decrease of the mobility, even for an annealing at 680 K stresses, the need for room-temperaturedeposited ohmic contacts that do not require annealing. The free carrier density drops from 2.1×10^{19} cm⁻³ (as deposited), which is higher than the so far highest reported value, to $1.2 \times 10^{18} \text{ cm}^{-3}$ (annealed at 780 K). Unfortunately, it was not possible to measure at even higher temperatures, as 780 K is the limit of the used device.

Uchida et al. used XPS measurements to investigate as-deposited and thermally annealed tin-doped $\alpha - Ga_2O_3$ thin films. They showed that thermal annealing at 500 °C in nitrogen atmosphere leads to an evaporation of tin from the thin film or the formation of Sn²⁺ from Sn⁴⁺.^[30] Therefore, it is reasonable to





Figure 5. a) Resistivity ρ , b) free carrier density *n*, and c) electron mobility μ of a tin-doped thin film measured at 360 K after a high-temperature Hall measurement at the denoted temperature. The gray line marks the transition from non-ohmic to ohmic contact behavior.

assume that the same effect occurs during the annealing of the here presented thin film. The transition from Sn⁴⁺ to Sn²⁺ and/or the evaporation of tin from the thin film can also explain the reduced free carrier density and increased resistivity for annealing at higher temperatures.^[30]

3. Conclusion

We have presented conductive, doped α -Ga₂O₃ thin films deposited by PLD in a two-step growth process. A doping with silicon, tin, and germanium was feasible and the structural and electrical properties of the thin films were presented. Higher temperaturegrown α -Ga₂O₃ buffer layers were employed to enable the deposition of doped thin films at lower substrate temperatures. A depth-resolved elemental investigation of tin-doped thin films deposited at different substrate temperatures by XPS was used to validate a temperature-dependent incorporation of tin into the thin films. Highly conductive thin films with resistivities as low as $2.7\times 10^{-2}\Omega\,\text{cm}$ for tin doping were achieved. The highest electron mobility of $17 \text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ at $1.4 \times 10^{19} \text{cm}^{-3}$ and $37 \text{cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ at $3.7 \times 10^{18} \text{cm}^{-3}$ were achieved for a doping with tin and germanium, respectively. We confirmed the increase of the electrical resistance of tin-doped α -Ga₂O₃ upon thermal annealing. We found a narrow annealing temperature window to obtain ohmic contacts before the drastic deterioration of the electrical properties of the doped thin films sets in. Additionally, the onset of a mobility decrease already starts at significantly lower annealing temperatures. These findings suggest the need to investigate low-temperature processes, for example, shallow implantation, to obtain ohmic contacts regarding the application of α -Ga₂O₃ as active layer in optoelectronic devices.

4. Experimental Section

All thin films were deposited by PLD employing a KrF excimer laser with a wavelength of 248 nm. The ceramic targets consisted of pressed and

sintered Ga₂O₃ powder by Alpha Aeser with a purity of 99.999%. For doped thin films, SnO₂ (purity 99.99%), GeO₂ (purity 99.999%), and SiO₂ (purity 99.999%) were mixed with the Ga₂O₃ powder and subsequently pressed and sintered under normal atmosphere for 72 h at 1300 °C. The dopant concentrations in the targets were 1 wt% SnO₂, 2 wt% Ga₂O₃, and 0.1 wt% SiO₂. These doping concentrations were chosen in preliminary experiments to yield high crystallinity and low resistivity of the thin films. Especially higher silicon percentages in the target led to thin films with high resistivity and absent in-plane orientation.

The incident laser energy density on the target surface was chosen to be approximately \approx 2.6 J cm⁻² and the pulse frequency was 10 Hz. All thin films were deposited on 10 × 10 mm² single-side polished *m*-plane sapphire substrates by CrysTec. An oxygen atmosphere with a pressure of 3 × 10⁻⁴ mbar was maintained during the deposition for all thin films.

The buffer layer of undoped α -Ga₂O₃ was deposited at a substrate temperature T_S of 700 °C, followed by the in situ deposition of the doped active layer at a lower substrate temperature. Between buffer and active layer growth, a 30 min waiting step was implemented to enable a cooling down of the sample. The substrate temperature for the deposition of the doped layer was varied between 470 and 580 °C.

The thickness of the thin films was determined by spectroscopic ellipsometry. XRD measurements were employed to investigate the crystallographic structure using a Philips X'Pert diffractometer. The in-plane crystallinity of the top layer was confirmed by ex situ RHEED measurements. Surface roughness and structure were determined by atomic force microscopy (AFM) measurements for selected samples in a Park Systems AFM. The XPS measurements were performed at the Humboldt Universität zu Berlin with a JEOL JPS-9030 setup with an achromatic magnesium source. Argon was used as sputter gas ($p_{\rm Ar} = 3 \times 10^{-4}$ mbar) for depth profiling. A low acceleration voltage of 300 V and ion current of 3.5 mA were used to avoid sputter damage. The sensitivity factors were determined by measuring pure Ga₂O₃ and SnO₂ reference samples and assuming perfect stoichiometry.

For the electrical characterization, Ti/Al/Au contacts with a thickness of 30/30/30 nm were evaporated on the sample corners. An annealing in nitrogen atmosphere at a temperature of 410 °C for 30 min was used to ensure ohmic behavior of the contacts. All thin films were characterized electrically by Hall effect and resistivity measurements in van der Pauw geometry using a home-built setup.^[36–38] The ohmic contacts on the thin-film corners were small (3–4 mm² per contact) compared to the sample size (100 mm²). The resistivity and Hall-effect measurements were performed in the dark using a magnetic field of 0.43 T.



Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

alpha gallium oxide, doped α -Ga₂O₃, tin doping

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