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Silicon Doping of Epitaxial Layers of Gallium Oxide by MOCVD

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Abstract

The article considers homoepitaxy of beta gallium oxide layers doped with silicon grown by metal organic chemical vapor deposition (MOCVD). Epitaxial growth was carried out on substrates of iron doped gallium oxide. Epitaxial layers at different rates of silicon doping from a diluted monosilane and at different temperatures were obtained. The crystal quality of epitaxial layers was analyzed, as well as mobility of electrons and conductivity were measured. The possibility of controlling the electrical properties (such as electron mobility and conductivity) of homoepitaxial gallium oxide layers doped with silicon during growth by the MOCVD method was demonstrated.

Keywords: Gallium oxide; MOCVD; Homoepitaxy; Silicone doping; Electron mobility; Conductivity

1. INTRODUCTION

The most stable β-phase of gallium oxide has the unique properties, such as a large band gap (4.9 eV [1]) and high values of the breakdown field (up to 8 MV/cm according to calculations [2], 5.3 MV/cm in the device [1]), as well as relatively high electron mobility (up to 196 $cm^2/(V \cdot s)$ [3]) and radiation resistance [4].

These facts confirm the significance of using this material in the production of power electronic devices and ultraviolet (UV) sensors. Recently, β-Ga_2O_3 has attracted significant attention for its potential use in various applications, including an UV solar radiation detector, gas sensor, photocatalyst and high-power electronic devices [5–9].

Another advantage of β-Ga_2O_3 is the ability to intentionally introduce *n*-type dopants, using a variety of dopant materials, such as Si [10], Sn [11], Ge [12]. Among these dopants, silicon is regarded as a mostly used one to improve the electrical conductivity of β-Ga_2O_3 layers. In addition, doping of gallium oxide with silicon allows to achieve a specific concentration of electrons, which is necessary for using the material in the production of electronic devices based on gallium oxide [13].

The manufacture of high-quality semiconductor devices, including power electronics devices, requires epitaxial layers with a minimum number of defects, which will primarily be determined by the choice of the substrate material. It is possible to use different substrate materials for epitaxy of various polytypes of gallium oxide. For example, α-Ga_2O_3 and κ-Ga_2O_3 are often grown on sapphire substrates [14,15] and it has been reported about the successful growth of α-Ga_2O_3 layers on a diamond substrate [16]. However, the most effective way to obtain layers with a low defect rate is to use homoepitaxy, which implies matching the substrate material and the epitaxial layer. Thus, another advantage of gallium oxide can be used: the possibility of producing substrates from bulk crystals grown by relatively cheap methods of pulling from the melt, for example, the Czochralski [17] and Stepanov [18] methods.

The process of growing a layer of β-Ga_2O_3 using epitaxy has been explored through various techniques, includ-

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ing molecular beam epitaxy (MBE) [19–21], metalorganic chemical vapor deposition (MOCVD) [22–24], halide vapor-phase epitaxy (HVPE) [25–28] and low-pressure chemical vapor deposition (LPCVD) [29–31]. Each method comes with its own unique challenges. For example, when using the MBE method to grow β-Ga_2O_3, it has proven difficult to precisely control the *n*-type doping level, especially at low dopant concentrations. HVPE and LPCVD epitaxial methods are well suited for producing relatively thick films for vertical power devices, but these methods are not ideal for growing complex heterostructures or for precisely controlling epitaxial layers in the nanometer range. MOCVD is the most widely used method for industrial fabrication of semiconductor devices today. However, there is still a need to improve the quality of β-Ga_2O_3-based epitaxial heterostructures. A detailed understanding of the mechanisms that affect the characteristics of β-Ga_2O_3/β-Ga_2O_3 and β-$(Al_xGa_{1-x})_2O_3$/β-Ga_2O_3 epitaxial layers and structures remains an unsolved problem [32]. Thus, based on the characteristics of the methods, the MOCVD process was chosen in our study.

2. METHODS

Bulk crystals of β-Ga_2O_3 were grown using the Czochralski method in the Nika-3 growth installation (FSUE EZAN, Russia). The melt was prepared in the iridium crucible, which has a height of 26 mm and a diameter of 40 mm. The crucible was placed in a thermal zone made of zirconium dioxide. Induction heating was used for melting. The raw material for the melt was Ga_2O_3 powder with a purity of 99.999%. Iron oxide powder (Fe_2O_3) of 99.99% purity (LLC Lanhit, Russia) was added to the charge for alloying. The addition of iron allows for the production of semi-insulating substrates, which makes it possible to measure the electrical properties of the epitaxial layers. The mass fraction of iron in the charge was 0.011%. The growth was carried out at a temperature of about 1850 °C and a pressure of 1.4 bar in a mixture of gases (Ar + O_2), the oxygen content in the growth atmosphere was about 5 vol.%. Previously grown gallium oxide crystals were used as seeds. The rate of pulling of the crystal was 0.15 mm/min.

The classical approach in the manufacture of bulk crystal substrates requires cutting, as well as mechanical and chemical polishing followed by cleaning. The nature of the cleavage of β-Ga_2O_3 crystals makes it possible to use a different method of manufacturing substrates, which consists in delaminating the bulk crystal along the cleavage plane (100) [33]. Moreover, using this method of obtaining substrates makes it possible to obtain samples with a sufficiently smooth surface without the need for additional processing. In this way, the substrates were obtained in the form of plane-parallel plates about 10×15 mm in size and 0.5 mm thick.

Epitaxial growth of β-Ga_2O_3 layers doped with silicon was carried out on β-Ga_2O_3:Fe substrates using MOCVD method at the Epiquip VP-50 facility, upgraded for the growth of oxides. The installation has a horizontal reactor with induction heating. A diluted monosilane (200 ppm) was used for silicon doping.

The carrier gas (nitrogen) flow was 4.5 slm (liters per minute under standard conditions), the oxygen flow was 1 slm, the trimethylgallium flow was 21 micromol/min, and the initial SiH_4 flow was 6.7 nmol/min. Epitaxial growth of the β-Ga_2O_3:Si layer was conducted for 60 minutes. The approximate growth rate was 1100 nm/h, which was estimated using a reflectometry system with a He-Ne laser. Four structures were grown at a fixed temperature of 980 °C with varying concentrations of silicon by changing the initial flow of silane (with a decrease in the flow by 2 and 5 times, and a 2-fold increase in the flow). The other 4 structures were grown with a standard silane flow (6.7 nmol/min), but at different temperatures of 1020 °C, 1050 °C, 1060 °C, and 1100 °C, respectively. A DRON-8 X-ray diffractometer (NPO Burevestnik, Russia) was used to analyze the phase composition of structures and their crystalline quality. Images of chip structures were obtained using a scanning electron microscope (Tescan MIRA-3, Czech Republic). The SE detector was used, the accelerating voltage was 2 kV, with the working distance of 7 mm. Gold plating was not used in this study.

Measurements of electron mobility the conductivity of structures were performed at the BIO-RAD HL5200 Hall effect measurement system by the four-point probe van der Pauw method.

3. RESULTS AND DISCUSSION

The X-ray diffraction 2θ-ω scans obtained from the set of samples with different silane fluxes are shown in Fig 1. These curves confirm the presence of only the β-phase of

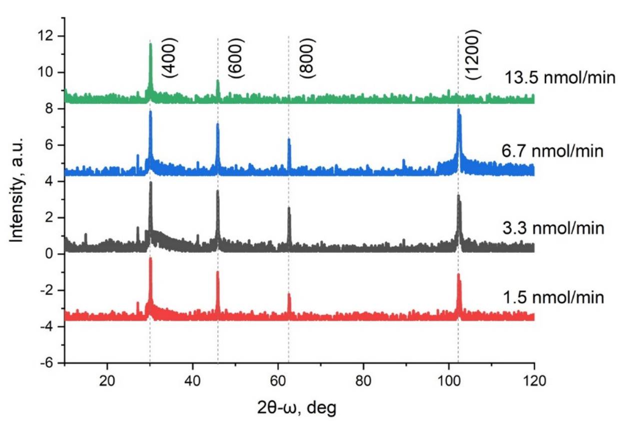

Fig. 1. X-ray diffraction 2θ-ω scans of β-Ga_2O_3:Si samples grown at different silane fluxes.

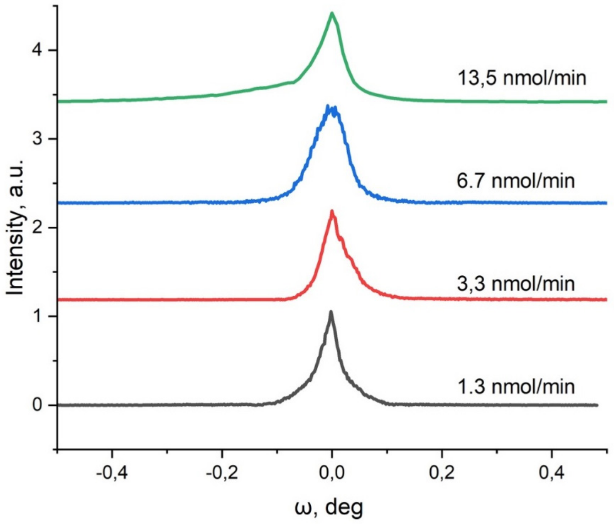

Fig. 2. The rocking curves of the β-Ga_2O_3:Si samples grown at different silane fluxes.

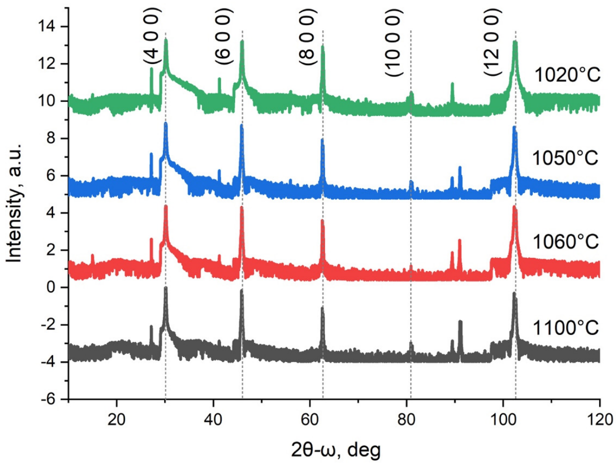

Fig. 3. X-ray diffraction 2θ-ω scans of β-Ga_2O_3:Si samples grown at different growth temperatures.

gallium oxide in all epitaxial structures, which is expected for the homoepitaxy process.

Based on the rocking curves for the same structures in Fig. 2, it can be said that the minimal silane flux (1.3 nmol/min) provides the best crystal quality of the structure. On the contrary, an increase in silicon concentration leads to increase in the full width at half maximum (FWHM) from 0.031 to 0.056 of a rocking curve. This is caused by an increase in the number of defects and a deterioration in the crystal quality. For example, authors of Ref. [34] reports that the properties of thin films of gallium oxide grown on (100) substrates are significantly affected by the formation of planar defects, such as packaging defects and twins, which result from the formation of two-dimensional islands with two different orientations rotated 180° in the (100) plane.

An increase in temperature intensified the entry of silicon, which worsened the quality of the layers. The X-ray diffraction rocking curves of the samples grown at different growth temperatures show no significant differences of the quality of these layers. Therefore, their graphs are not given in this work. The similar 2θ-ω curves of the samples grown at different temperatures (see Fig. 3) confirm the presence of only the β-phase of gallium oxide in all structures. Unfortunately, two structures were unsuitable for further measurements (a structure with a silane flux of 3.3 nmol/min and a structure with a growth temperature of 1100 °C). Thus, in the further part of the study, only 6 samples will be considered (obtained under different growth temperatures of 1020 °C, 1050 °C, 1060 °C, respectively and different silane fluxes: 1.3, 6.7, 13.3 nmol/min).

The images of the chip samples were obtained using a scanning electron microscope (Fig. 4) and the thickness of the layers of structures was determined. The thicknesses of the layers were in the range of 0.9–1.0 μm which are consistent with the results of measurements of the epitaxial growth rate using a reflectometry system. An increase in the growth temperature also had no effect on the growth rate, at least in this temperature range. In addition, based on the images of surface of the sample (see Fig. 5a), it is possible to see the presence of a non-planar growth type. In our experiments, the plane (100) should promote layer-by-layer growth, but due to the lack of misorientation of the substrate (usually by 0.2–0.3° relative to the normal to the growth surface), the density of seed steps on the initial substrate is small and flat areas without steps predominate on the surface of the substrate, which led to island-like layer growth.

Fig. 5 shows the dependence of the electron mobility and the conductivity of three structures with different silane flows. It is easy to see that an increase in the silane flow (and, as a consequence, an increase in the concentration of silicon in the layers) leads to a decrease in the electron mobility. This may be due to scattering by electrons on Si atoms (which are interstitial impurities in gallium oxide) and may also be due to the presence of additional defects in the layer, which is consistent with the data of the X-ray rocking curves.

It is known that dopants and other defects such as interstitials and vacancies may act as scattering centers for electrons which will degrade the mobility. In a recent study [35], it was reported that the mobility of electrons in homoepitaxial films of β-Ga_2O_3 is constrained by the high density of edge dislocations and crystal defects caused by high levels of doping. The nature of the dependencies is comparable with those reported in study [36] in which a decrease in electron mobility with the increase in Si doping concentration suggests the domination of ionized impurity scattering.

The dependence of conductivity (Fig. 6) on the doping level is approximately linear, which is associated with an

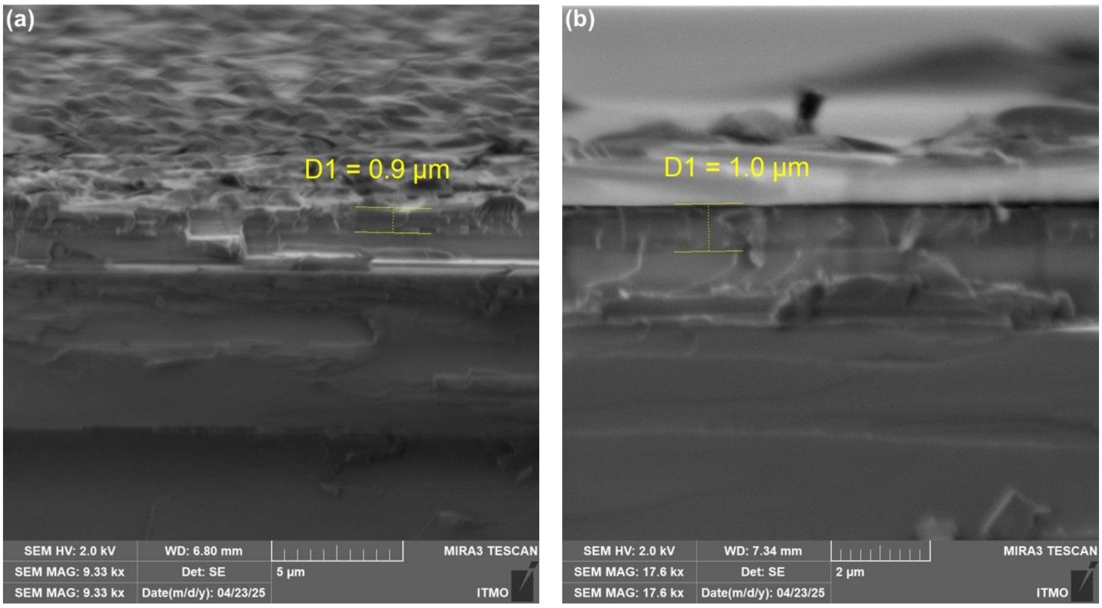

Fig. 4. Images of layers on the chip of structures obtained using a scanning electron microscope: (a) sample with a silane flux of 6.7 nmol/min, a layer of β-Ga_2O_3:Si is about 0.9 μm thick; (b) sample with a 3.3 nmol/min silane flux, a β-Ga_2O_3:Si layer is about 1.0 μm thick.

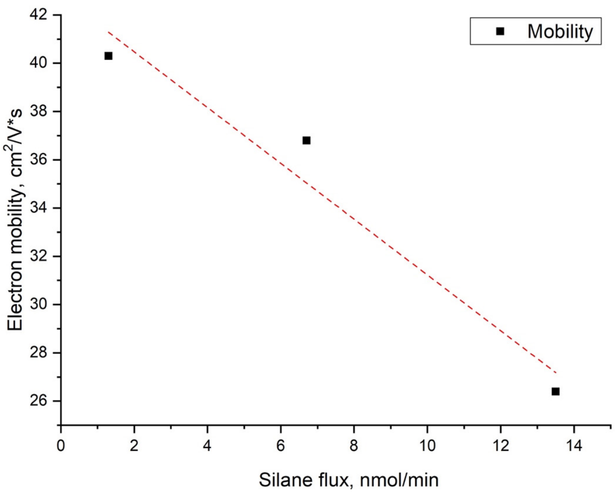

Fig. 5. Dependence of the electron mobility of β-Ga_2O_3:Si layers grown at fixed temperature 980 °C and different silane fluxes on the silane flux.

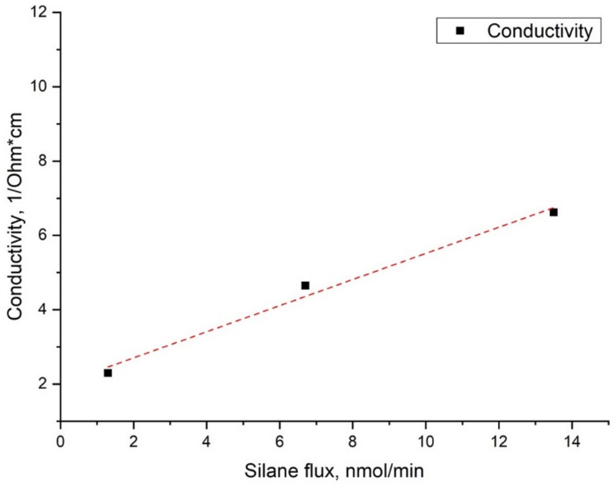

Fig. 6. Dependence of the conductivity of β-Ga_2O_3:Si layers grown at fixed temperature 980 °C and different silane fluxes on the silane flux.

increase in the silicon concentration in the layer, and is consistent with the results of the work [37].

Fig. 7 shows measurements of the conductivity of three structures grown at different growth temperatures. We assume that an increase in temperature leads to the incorporation of larger number of silicon atoms from the silane flux into the epitaxial layer. For example, in work [38] it is reported that a higher growth temperature can contribute to a greater probability of Si entering the epitaxial layer, which further reduces the resistivity of Ga_2O_3. Due to these factors, the concentration of silicon in the layer and its conductivity increase.

The dependence of electron mobility on the growth temperature of the epitaxial layers is shown in Fig. 8. In the given temperature range, a linear increase in mobility is observed with increasing temperature. We suppose that the main mechanism for limiting electron mobility is its scattering by charged point defects, for example, on vacancies [39]. In this case, as the temperature rises, the silicon content in the layer increases and, as a consequence, the electron concentration increases. In turn, the growth in the electron concentration leads to the screening of charged point defects (scattering centers), to a decrease in scattering and to an increase in mobility.

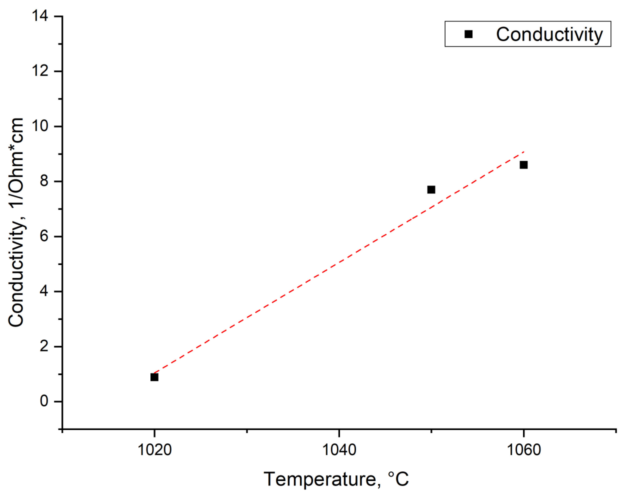

Fig. 7. Dependence of the conductivity of β-Ga_2O_3:Si layers on growth temperature.

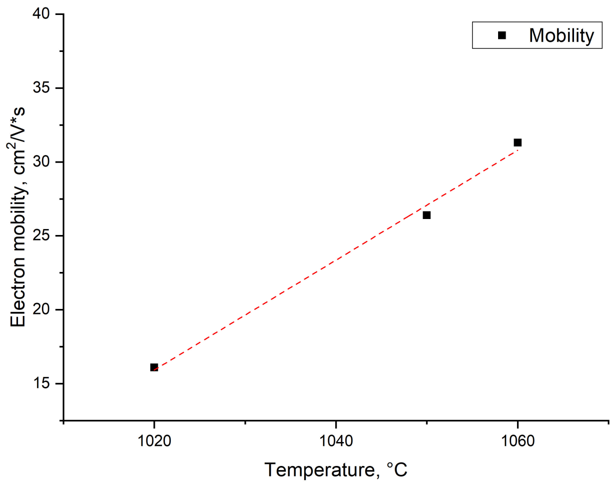

Fig. 8. Dependence of the electron mobility of β-Ga_2O_3:Si layers on different growth temperature.

4. CONCLUSIONS

The paper describes the possibility of homoepitaxial growth of gallium beta oxide layers doped with silicon and a change in their electrical properties depending on the dopant flow and the growth temperature. An increase in the silane flux leads to reduction of the mobility of charge carriers in the layer and deterioration in crystalline quality with an increase of its conductivity. An increase in the growth temperature leads to an increase of the conductivity of structures and the mobility of charge carriers in them with increased crystalline quality.

Thus, the possibility of β-Ga_2O_3 layer growth by MOCVD with control of conductivity and mobility of electrons depending on the growth parameters (temperature and silane flux) has been shown. This result will be crucial for creating efficient power optoelectronic components with a high breakdown field and minimal resistance.

A special feature of this work is the growth of gallium oxide layers on the (100) plane of the substrate, while most papers consider other growth planes, such as (010) [13,23,40] and (001) plane [34]. The effect of doping at high growth temperatures is considered, as opposed to other works, where the epitaxial growth temperature on the (100) plane is fixed and is less than 980 °C, for example, 740 °C [40], 800–900 °C [41], 825 °C [42].

The data obtained in the work will form the basis for future technology for the production of device structures and microelectronic devices based on gallium oxide for promising power electronics devices, such as field-effect transistors and Schottky diodes.

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Легирование кремнием эпитаксиальных слоев оксида галлия методом осаждения металлорганических соединений из газовой фазы

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Аннотация. В работе рассмотрены особенности легирования кремнием эпитаксиальных слоев оксида галлия в процессе гомоэпитаксии. На легированных железом подложках β-Ga_2O_3 методом осаждения металлорганических соединений из газовой фазы (MOCVD) выращены слои β-Ga_2O_3 легированные кремнием из раствора моносилана при различных потоках силана и температурах роста. Проанализировано кристаллическое качество структур, получены данные подвижности носителей и проводимости слоев в зависимости от параметров легирования.

Ключевые слова: оксид галлия; МОГФЭ; гомоэпитаксия; легирование; подвижность электронов; проводимость