Activation energy of silicon diffusion in gallium oxide: roles of the mediating defects charge states and phase modification

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Silicon (Si) is an efficient *n*-type dopant in gallium oxide (Ga₂O₃) - an ultra-wide bandgap semiconductor promising in a number of applications. However, in spite of the technological importance for the device fabrication, the activation energy for Si diffusion in Ga₂O₃ is missing in literature. In the present work, we do such measurement in ion implanted monoclinic β -Ga₂O₃ samples employing anneals in air ambient, also admitting the influence of the potential ion beam induced phase modifications on diffusion. Importantly, we show that Si diffusion in β -Ga₂O₃ fits with the concentration dependent diffusion model, involving neutral and single negatively charged point defects to mediate the process; so that we assumed gallium vacancies in the corresponding charge states to assist Si diffusion in β -Ga₂O₃ with the activation energies of 3.2±0.3 eV and 5.4±0.4 eV, respectively. Moreover, we also found that a pre-existing phase modified surface layer efficiently suppressed Si diffusion in β -Ga₂O₃ for temperatures up to 1000 °C.

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The dopant redistribution control is of paramount importance in modern semiconductors, motivated by enhanced requirements for the integration and scaling down of the device components. Thus, understanding of the diffusion mechanisms and documenting the diffusion energetics is crucial for predictive device processing. Meanwhile, even in elemental semiconductors, such as Si [1] and Ge [2], dopant diffusion exhibits a complex behavior, since it depends on the intrinsic defect balance governing the process; specifically if modifying the defect generation occurring at the surface [3]. The situation becomes even more complicated in compound semiconductors, with defects occurring in different sublattices [4]. This holds, in particular, for gallium oxide (Ga_2O_3) - an ultra-wide bandgap semiconductor, emerging as a promising platform for the next generation power electronics, deep UV applications, etc. [5].

Ga_2O_3 can be crystallized in several polymorphs, with monoclinic phase (β - Ga_2O_3) being most stable under normal conditions and, therefore, attracting much of interest. It was demonstrated previously, that *n*-type doping of Ga_2O_3 can be accomplished by such impurities as Si, Ge, and Sn forming shallow donors on substitutional Ga sites or by F and Cl substituting oxygen [6-9]. Notably, under Ga-rich condition the Si incorporation into Ga sublattice occurs most efficiently comparing with other common donors [9]. Indeed, Si doping was realized by ion implantation to fabricate low resistance ohmic contacts [10] and MOSFET channels [11,12].

However, despite the technological importance of Si for Ga_2O_3 -based device fabrication, the literature data on Si diffusion in Ga_2O_3 are not fully systematic; in particular, the activation energy has not yet been reported, resulting in predominantly qualitative conclusions [10,13,14]. Indeed, Sasaki *et al.* [10] studied Si ion implantation into (010) β - Ga_2O_3 and observed that Si atoms start to diffuse after 1100 °C anneals in

N₂ atmosphere, while Si piles-up near the projected range (R_p). Meanwhile, Tadjer *et al.* [13] studied Si diffusion in (-201) β -Ga₂O₃ in O₂ ambient and did not detected Si accumulation at the R_p for the temperatures ≤ 1150 °C. Sharma *et al.* [14] have also confirmed a strong influence of the annealing ambient on Si diffusion in (-201) β -Ga₂O₃, demonstrating that the Si redistribution is significantly suppressed in N₂ atmosphere as compared to that in O₂. In the present paper, we measure the activation energy of Si diffusion in β -Ga₂O₃ and show that Si diffusion in β -Ga₂O₃ is mediated by neutral and single negatively charged gallium vacancies. Notably, we also demonstrate that a pre-existing phase modified surface layer can be used for the suppression of the Si diffusion in β -Ga₂O₃.

In the present study we used Sn-doped (-201) β -Ga₂O₃ single crystals (Tamura Corp.) containing $\sim 7 \times 10^{17}$ Sn/cm³ homogeneously distributed throughout the samples. The samples were implanted with 1 MeV ²⁸Si⁺ ions to an ion dose of 1×10^{15} cm⁻². Notably, some selected samples, before the Si implants, were pre-implanted with 400 keV ⁵⁸Ni⁺ ions to 2×10^{14} cm⁻² in order to form a pre-existing phase-modified layer in the region between 1 MeV Si implantation peak and the sample surface [15]. All implants were performed at room temperature, maintaining 7° off-angle orientation from a normal direction to minimize the channeling. Further, the samples were isochronically annealed at 900–1200 °C for 30 min in air using a tube furnace.

The concentration versus depth profiles of Si and Ni atoms were measured with secondary ion mass spectrometry (SIMS) using Cameca IMS 7f microanalyzer. The measurements were performed with the 10 keV O₂⁺ primary beam rastered over the 150×150 μ m² area and only the central part of the crater was used to collect the SIMS signal. During the measurements, an electron gun was used to minimize charging

effects. The SIMS intensity-to-concentration calibration was performed using the as-implanted samples. The depth conversion of the recorded profiles was performed by measuring the sputtered crater depth and assuming a constant erosion rate.

Selected samples were analyzed with 2theta X-ray diffraction (XRD) measurements using the Bruker AXS D8 Discover diffractometer with Cu $K_{\alpha 1}$ radiation in the locked-coupled mode. The XRD measurements were performed in order to check the phase status of the implanted samples in the context of the possible ion-beam-induced phase transformations [15]. Notably, the Si implants were insufficient to induce the phase transformation due to the lighter ion mass, enabling interesting comparison with Ni+Si implants characterized with the pre-existing phase modified surface layer.

Fig. 1 summarizes the Si diffusion data in β -Ga₂O₃ implanted with 1 MeV Si ions including (a) the SIMS profiles / simulations and (b) the corresponding Arrhenius plot. As seen from Fig. 1(a), the as-implanted Si profile exhibits a Gaussian shape with the peak located at ~ 800 nm, i.e. corresponding well to the theoretically predicted R_p value obtained with the SRIM code simulation [16], see the dashed line in Fig. 1(a). The background Si concentration, $\sim 2 \times 10^{17}$ Si/cm³, corresponds to the unintentional Si contamination in the samples. No measurable diffusion was detected at ≤ 900 °C; however, at ≥ 1000 °C prominent diffusion was observed. Specifically at 1000 °C, distinct shoulders form on both sides of the implantation peak, while a fraction of the Si atoms remains at the peak region. Further anneals up to 1150 °C, lead to a strong Si diffusion into the bulk, still maintaining a fraction of Si atoms in the peak until it is significantly reduces at 1200 °C. Notably, starting from 1050 °C, there is Si accumulation near the surface; however excluded from the further analysis for clarity, since the essential part of the data were collected much deeper in the sample .

In order to simulate Si diffusion we use the second Fick's law with the concentration depended effective diffusion constant (D_{eff}), so that the Si concentration (C_{Si}) is given by:

$$\frac{\partial C_{Si}}{\partial t} = \frac{\partial}{\partial x} \left(D_{eff} \frac{\partial C_{Si}}{\partial x} \right) \quad (1)$$

In its turn, D_{eff} can be expressed using Fair's diffusion model [17], taking into account different charge states of the point defects assisting Si diffusion in form of:

$$D_{eff} = h \left[D_{Si}^0 + D_{Si}^{1-} \frac{n}{n_i} + D_{Si}^{2-} \left(\frac{n}{n_i} \right)^2 + \dots \right] \quad (2)$$

where D_{Si}^k corresponds to the diffusivities of Si atoms assisted by the point defects in the charge state k , while n and n_i are the free and intrinsic carrier concentrations, respectively. The parameter h is the field enhancement factor described as:

$$h = 1 + \frac{n}{2n_i} \left[\left(\frac{n}{2n_i} \right)^2 + 1 \right]^{-1/2} \quad (3)$$

We assume the full ionization of the implanted impurity, so that we set $n = C_{Si} + C_{Sn}$, where C_{Sn} is the concentration of Sn atoms in the samples. Further, in order to calculate the n_i we use Varshni model of the temperature dependence of the energy bandgap with the parameters taken from [18] and the values of the electron and hole effective masses equal to $0.28m_e$ and $0.4m_e$, respectively, in accordance with Ref. [9]. The results of the simulation with Eqs. 1-3 are shown in Fig. 1(a) by the solid lines [19] and it is seen that the model adequately describes the experimental data, specifically deeper in the sample – which is the region of interest for this work. Importantly, the best fits between the experimental and the simulated profiles were obtained only if applying the first two terms in Eq. (2). To illustrate this fact we included Fig. S1 in the supplementary materials. Thus, it can be concluded that only neutral and single

negatively charged point defects assist Si diffusion in β -Ga₂O₃. The prime candidate is the Ga vacancy (V_{Ga}) taking into account that V_{Ga} exhibits four different charge states (0, 1-, 2- and 3-) and its formation energy decreases in *n*-type material [20]. Further decrease of the V_{Ga} formation energy can occur near the surface due to O-rich conditions [20]. Thus, an additional argument in favor of the V_{Ga} -assisted mechanism for Si diffusion is in its enhancement in oxygen-rich ambient as compared to N₂ (Ref. 14). Notably, the triple negatively charged V_{Ga} has the lowest formation energy in *n*-type β -Ga₂O₃ according to the density functional calculations [20]. However, the exact position of the Fermi level at the diffusion temperatures is actually not known and it can be potentially lowered due to the acceptor behavior of V_{Ga} 's; in some reports resulting even to the *n*- to *p*-type conductivity conversion in β -Ga₂O₃ while measuring at high temperatures [21]. Thus, it is realistic that V_{Ga} 's in other than 3- charge states may mediate Si diffusion in β -Ga₂O₃ at high temperatures. Additionally, in the context of the discussion above, we believe that the interstitial part of the diffusion is negligible comparing to the mediation by vacancies.

Using the fits in Fig. 1(a), we performed the Arrhenius analysis for D_{Si}^0 and D_{Si}^{1-} in Fig. 1(b). It is seen that D_{Si}^0 exhibits the activation energy of $E_a^0 = 3.2 \pm 0.3$ eV and the pre-factor of $D_0^0 = 0.1$ valid throughout the whole temperature range. D_{Si}^{1-} demonstrates a linear dependence through the whole temperature range studied too, with the activation energy $E_a^{1-} = 5.4 \pm 0.4$ eV and the pre-factor $D_0^{1-} = 1.6 \times 10^2$. Thus, the data in Fig. 1 provide the first systematic analysis of the Si diffusion in β -Ga₂O₃, including the determination of the V_{Ga} charge states mediating the diffusion and the estimates for the corresponding activation energies. In the context of literature [10,13,14], since both neutral and singly negatively charged V_{Ga} were found to mediate the diffusion, it is not surprising that the annealing atmospheres affect the diffusion, because of different V_{Ga} balance at the surface [20].

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Importantly, the change in the conditions for the V_{Ga} generation at the surface may be potentially induced not only by different atmospheres, but also by modifying the surface. Indeed, recently, ion-beam-induced phase transformations of β -Ga₂O₃ to κ -phase were reported [15,22]. In particular, Azarov *et al.* [15] have demonstrated that a certain threshold in the density of the radiation damage may be reached to enable the β -to- κ phase transformation. In relation to the present Si diffusion study, the conditions for the Si implants (as shown in Fig. 1) were selected to be under this threshold as compared with the heavier ion mass Ni, Ga, Ge, or Au implants, known to result into the β -to- κ phase transformation in the near surface volume [15,22]. In the present work, in order to modify the Si diffusion conditions we fabricated pre-existing κ -phase surface layer on the top of the bulk β -Ga₂O₃ wafer where Si diffused. As an illustration, Fig. 2 compares typical examples of the 2 θ scans of the as-grown, Si, Ni and Ni+Si implanted samples around a prominent (-402) reflection characteristics of (-201) β -Ga₂O₃. Following the interpretations from literature [15] (see also Fig. S2 in Supplementary materials) we conclude that only samples implanted with Ni or Ni+Si exhibit signatures characteristic to the κ -phase. Meanwhile, the Si implants lead to the formation of broad shoulders at the low-angle side of the (-402) peak in Fig. 2 attributed to the strain accumulation.

Fig. 3 summarizes (a) Si and (b) Ni diffusion data in Ni+Si implanted samples, while panel (c) compares the Arrhenius analysis of Si diffusion with and without pre-existing surface modified layer. As seen from Fig. 3(a) as well as the insets into Fig. 3(b), the 1000 °C anneal does not lead to any noticeable Si redistribution in contrast to the data in Fig. 1. The observed suppression of Si diffusion in the Si+Ni implanted samples could be attributed to the lowering of the V_{Ga} concentration. We assume that the κ -phase layer formed in the near surface region can trap V_{Ga} migrating from the

surface. However, κ -phase is metastable and recrystallizes back to β -Ga₂O₃ rapidly upon annealing [15,22], so that it affects the diffusion process only at lower temperatures. Notably Ni atoms are much more thermally stable as compared to Si and very little Ni diffusion was detected for the temperature range studied, see Fig. 3(b). The insets in the panel 3(b) compare the Si concentration depth profiles near the Si projected range in the samples with and without the pre-existing phase, confirming the observations discussed above. Notably, a certain fraction of the implanted Si atoms remains in the peaks throughout the anneals.

The Si diffusion data in Fig. 3(a) were fitted to the simulations in accordance with Eqs. 1-3, so that Fig. 3(c) plots the energetics of the Si diffusion in all samples studied in this work. It is seen that the diffusion in the samples with pre-existing surface modified phase layer, exhibits two stage behavior with lower diffusion activation at ≤ 1050 °C and practically the same activation as that in pure β -Ga₂O₃ for higher temperatures.

Conventionally, in addition to the binding energy, the impurity diffusion activation energy is comprised of the formation and migration energies of the defect mediating the process, i.e. V_{Ga} for the present case. Neglecting the binding energy and accounting for the recently reported V_{Ga} migration energy of ~ 0.8 eV [23], from the data in Fig. 3(c) the upper limit of the V_{Ga} formation energy can be estimated as ~ 2.4 or ~ 4.6 eV; in agreement with the theoretically predicted values for V_{Ga} in β -Ga₂O₃ under O-rich conditions and expected Fermi level position [20,24].

In conclusion, Si diffusion in (-201) β -Ga₂O₃ single crystals have been studied in the temperature range of 900-1200 °C in air ambient. The diffusion of Si has been simulated using the Fair's diffusion model, confirming the neutral and singly negatively charged point defects to assist the diffusion process; we assumed it to be gallium

vacancy in the corresponding charge states. The activation energies of 3.2 ± 0.3 eV and 5.4 ± 0.4 eV were obtained for the diffusion via neutral and singly negatively charged defects, respectively. Furthermore, we demonstrate that κ -phase layer formed near the surface by heavy ion mass implants can be used for effective suppression of Si diffusion at the temperatures up to 1000 °C.

Supplementary Material

See Supplementary Material for additional details of the simulations and XRD peak identification in the as-implanted samples.

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AUTHOR DECLARATIONS

Conflict of interest

The authors have no conflicts to disclose.

DATA AVAILABILITY

The data that supports the findings of this study are available within the article and supplementary materials.

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Fig. 1 (a) Si concentration vs depth profiles, as measured by SIMS, in (-201) $\beta\text{-Ga}_2\text{O}_3$ implanted with Si ions and annealed as indicated in the legend. The solid lines represent the diffusion simulations, while the dashed line is the SRIM [16] prediction of the as-implanted Si profile. (b) the Arrhenius plot summarizing the diffusion simulations.

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Fig. 2 XRD 2theta scans across (-402) reflection of the (-201) β - Ga_2O_3 samples implanted with Ni, Si as well as co-implanted with Ni+Si ions as indicated in the legend. The XRD peak at $\sim 37.95^\circ$ is related to the sample holder.

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Fig. 3 (a) Si concentration vs depth profiles in (-201) $\beta\text{-Ga}_2\text{O}_3$ with pre-existing phase modified surface layer implanted with 1 MeV Si ions and annealed as indicated in the legend. The solid lines represent the diffusion simulations. (b) Corresponding Ni concentration vs depth profiles. The insets in panel (b) compare the fractions of Si atoms remaining in the peaks for the Si and Si+Ni implanted samples. (c) the Arrhenius plot summarizing the diffusion simulations.

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