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ABSTRACT

Highly resistive undoped p-type gallium oxide samples were subjected to cumulative proton irradiation with energies ranging from 25 to 70 keV and doses in the 1.6×10^{14}–3.6×10^{14} cm^{-2} range. Proton irradiation resulted in up to a factor of 2 reduction of minority electron diffusion length in the samples for temperatures between ~300 and 400 K. Electron injection into the samples under test using a scanning electron microscope beam leads to pronounced elongation of diffusion length beyond the pre-irradiation values, thus demonstrating stable (days after injection) recovery of adverse radiation impact on minority carrier transport. The activation energy of 91 meV estimated from the temperature dependent diffusion length vs electron injection duration experiments is likely related to the local potential barrier height for native defects associated with the phenomenon of interest.

The main limiting factor in Ga_2O_3 technology is related to difficulties of p-type conductivity realization.[1–4] Previous studies indicate that holes in β-Ga_2O_3 have characteristics of low dispersion, high effective mass, and high density of states. This results in formation of weak polarons (or localized holes), which are trapped by lattice distortions[5–9] and hindered p-type conductivity. Contradicting these claims, Ref. 10 has experimentally shown a possibility of low p-type conductivity in Ga_2O_3. It is additionally reported that the self-trapping nature of holes practically disappears at temperatures above 90–120 K.[3] Given the current state-of-the-art and ever mounting interest in p-Ga_2O_3, one can reasonably claim that a robust p-type Ga_2O_3 electrical conductivity will be possible in the foreseeable future in addition to the use of p–n heterojunctions.[11]

In Ga_2O_3, once bipolar devices become available, minority carrier transport (diffusion length) will be of primary importance. The minority carrier diffusion length defines performance of bipolar devices such as p–n junction diodes, bipolar transistors, and p-i-n detectors.[12] One of the major issues in the current ZnO and GaN device technology is the low diffusion length of minority carriers, which is partially due to dislocation scattering.[13,14] It has been previously shown that in p-type GaN and ZnO, low energy electron injection, either with an electron beam of a scanning electron microscope (SEM) or forward bias, results in a significant increase in the minority carrier (electron) diffusion length.[13,15–18] Similar effects have been very recently observed in n-type Ga_2O_3 subjected to low energy electron beam injection.[19–21]

This paper studies minority carrier transport (electron diffusion length) in p-Ga_2O_3 highly resistive samples subjected to proton irradiation following injection from a SEM electron beam. The below-presented results demonstrate feasibility for a significant diffusion length increase beyond the initial pre-irradiated values, thus paving the road toward solid-state electron injection in the future Ga_2O_3 bipolar devices.

Undoped β-Ga_2O_3 samples, analyzed in this study, were grown in a RF-heated horizontal metalorganic chemical vapor deposition (MOCVD) reactor. Trimethylgallium (TMGa) and 5.5 N pure oxygen were used as gallium and oxygen sources, respectively. Argon was used as the carrier gas (cf. Ref. 4). The β-Ga_2O_3 layer was grown on a c-oriented sapphire substrate using the Ga/O ratio and growth

temperature as 1.4×10^{-4} and 775 °C, respectively. The total reactor pressures of 30–38 Torr and variable growth rates (gallium and oxygen precursor fluxes) were used to have different native defect (V_{Ga} and V_o) concentrations in the Ga_2O_3 films, leading to different values of hole concentrations. The epitaxial layer thickness was ~450 nm. X-ray diffraction revealed highly textured films of gallium oxide in the β-Ga_2O_3 phase with monoclinic space group ($C2/m$) symmetry.

Ohmic contacts for electrical characterization were prepared by silver paint at the four corners of the sample. Hall effect measurements were performed in a Van der Pauw configuration in the 500–850 K temperature range for magnetic fields perpendicular to the film plane varying from −1.6 to 1.6 T using a high impedance high temperature custom-designed measurement setup. Resistivities at highest measured T = 850 K were found in the range from 1.2×10^3 to 1.3×10^4 Ω cm. Hall effect measurements demonstrated the positive sign for majority carriers in both samples, thus confirming the p-type conductivity. The free hole concentrations and mobilities at 850 K were estimated in the 10^{13}–5.6×10^{14} cm^{-3} range and the 8–16 cm^2 V^{-1} s^{-1} range, respectively. More details could be found elsewhere.[22]

Minority carrier diffusion length (L) measurements were carried out using the electron beam-induced current (EBIC) technique *in situ* in a Phillips XL-30 scanning electron microscope (SEM). Details on EBIC techniques could be found in Refs. 13, 14, and 22. The measurements were carried out in the temperature range of 294–404 K using a temperature-controlled stage integrated into the SEM. For EBIC measurements, the electron beam energy was kept at 10 keV, corresponding to ~0.1 nA absorbed current and ~350 nm electron range (penetration depth) in the material. The EBIC line-scans for diffusion length extraction were carried out in a planar configuration using Ni/Au (20 nm/80 nm) asymmetrical pseudo-Schottky contacts created on the film with lithography/liftoff techniques. Single line-scan takes approximately 12 s, which is sufficient for extraction of the minority carrier diffusion length value. To perform electron injection in the region of EBIC measurements, line-scans were not interrupted for the total time of up to ~350 s (corresponding to the primary excitation electron charge density of 42.8 pC/μm^3). The values of diffusion length were extracted intermittently. The EBIC signal was amplified with a Stanford Research Systems SR 570 low-noise current amplifier, digitized with Keithley DMM 2000, and controlled by a PC (personal computer) using a home-made software.

It should be noted that the primary excitation SEM electron beam serves for generation of non-equilibrium electron–hole pairs in the material due to the band-to-band (the valence band to the conduction band) transition of excited electrons. The primary excitation electrons do not accumulate in the material since the sample is grounded, thus preserving the sample's electroneutrality.

The experimentally reported value for radiation ionization energy consumed per electron–hole pair creation is 15.6 eV for β-Ga_2O_3.[23] Based on the injected charge density of 42.8 pC/μm^3 and accounting for ~600 electron–hole pairs generated in this volume (10 000 eV/15.6 eV), a non-equilibrium electron–hole pair density of ~10^{23} cm^{-3} is obtained for the experimental conditions of this work. Therefore, the minority carrier diffusion length, reported here, pertains to the non-equilibrium electrons with concentrations significantly exceeding the Hall effect equilibrium majority carrier (hole) concentration. It should be stressed, however, that in the vicinity of the electron beam (during the measurements), the amount of non-equilibrium majority and minority carriers is equal (while both significantly exceed the equilibrium Hall majority carrier concentration at given temperature), thus avoiding a high injection level regime.

The values for minority carrier diffusion length obtained in the samples under test and previously reported in Ref. 22 and in this work exceed by a factor of 3 those presented in Ref. 24 for minority holes in n-type Ga_2O_3 under the same temperatures. The diffusion length of minority carriers is primarily dictated by the carrier mobility (larger for electrons in p-type materials as compared to holes in n-type materials), which is, in turn, related to the diffusion coefficient.[16] This fact serves as an additional proof for p-type electrical conductivity of the samples under test close to room temperature.

Cathodoluminescence (CL) measurements were carried out under 10 kV accelerating voltage using Gatan MonoCL2 attached to the SEM. Spectra were recorded with a Hamamatsu photomultiplier tube sensitive in the 150–850 nm range and a single grating monochromator (blazed at 1200 lines/mm).

Ga_2O_3 samples were irradiated with protons having a dose/energy sequence to create a near-uniform hydrogen concentration around 10^{19} cm^{-3}. This consisted of 25 keV/1.6×10^{14} cm^{-2}, 50 keV/1.7×10^{14} cm^{-2}, and 70 keV/3.6×10^{14} cm^{-2}. EBIC and CL measurements were carried out before and after proton irradiation, and the results were compared.

The temperature dependence of L before and after proton irradiation is shown in Fig. 1. L decreased with increasing temperature with room temperature values being at 1040 and 635 nm. Relatively large values of L are partially due to the very low majority carrier concentration. Within the current temperature range of measurements, it is likely that the origin of L decrease is due to phonon scattering.[22]

The activation energy for temperature dependence of L is given by[13,22]

$$L(T) = L_0 \exp\left(\frac{\Delta E_{A,T}}{2kT}\right). \quad (1)$$

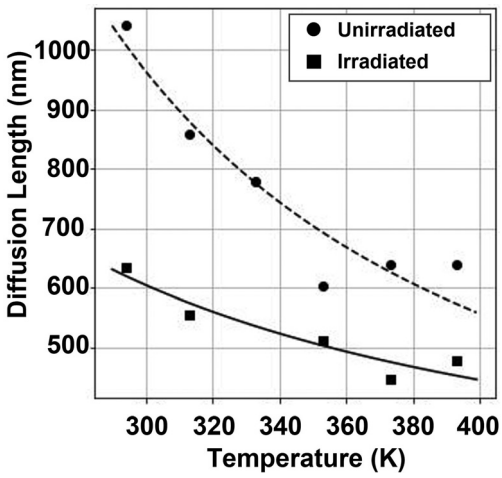

FIG. 1. Temperature dependence of the diffusion length in the structure before and after irradiation with high energy protons (25 keV 1.6×10^{14} cm^{-2} + 50 keV, 1.7×10^{14} cm^{-2} + 70 keV, 3.6×10^{14} cm^{-2}).

Here, L_0 is a scaling constant, $\Delta E_{A,T}$ is the thermal activation energy, k is the Boltzmann constant, and T is the temperature. The activation energy pertaining to the reduction of L with temperature is estimated at 76 meV after irradiation (113 meV pre-irradiation). A detailed discussion regarding the origin of $\Delta E_{L,T}$ is presented in Ref. 22.

Figure 2 presents results of the electron injection experiments carried out at varying temperatures. The minority electron diffusion length exhibits a linear increase with duration of electron injection before saturation occurs (not shown in Fig. 2). The linear increase in L with electron injection duration was previously observed in p-GaN,[16] p-ZnO,[13] unintentionally doped GaN,[23] and n-Ga_2O_3.[20] The minority carrier diffusion length increase in Fig. 2 is characterized by the rate R (dL/dt, where t is the duration of electron injection), which drops from 1.6 nm/s at room temperature to about 0.9 nm/s at 120 °C.

The effect of temperature on rate R is described by[23]

$$R(T) = R_0 \exp\left(\frac{\Delta E_{A,T}}{2kT}\right) \exp\left(\frac{\Delta E_{A,I}}{kT}\right). \quad (2)$$

Here, R_0 is a scaling constant and $\Delta E_{A,I}$ is the electron injection effect activation energy. Equation (2) can be used to find the activation energy of an injection-induced component for the increase in L from the Arrhenius plot in the inset of Fig. 3. The latter figure shows a decrease in R with increasing temperature. The slope of the Arrhenius plot is $\Delta E_{A,I} + 0.5\ \Delta E_{A,T}$, from which $\Delta E_{A,I} \sim 91$ meV is obtained. $\Delta E_{A,I}$ is associated with the mechanism responsible for the elongation of L with injected charges. The latter effect is likely associated with the gallium vacancy (V_{Ga}), which is a pervasive point defect in undoped Ga_2O_3.

A detailed mechanism for the electron injection-induced L increase is presented in Ref. 25 for undoped GaN. This mechanism is also applicable to p-Ga_2O_3, and its key points are outlined as follows:

- A non-equilibrium electron, generated by a primary scanning electron microscope beam gets trapped by gallium vacancies, which are deep acceptors in Ga_2O_3.[25] Because of its energetic position in Ga_2O_3 forbidden gap and fairly large concentration in the material ($\sim 10^{18}$ cm^{-3}), a pronounced number of V_{Ga} remains in the neutral state, thus acting as a meta-stable electron trap. Trapping non-equilibrium electrons on the V_{Ga}-levels prevent recombination of the conduction band electrons through these levels. This leads to an increase of lifetime, τ, for a non-equilibrium electron in the conduction band and, as a result, to an increase of L [$L = (D\tau)^{1/2}$, where D is the carrier diffusivity].
- The V_{Ga}-level containing a trapped electron becomes again available for recombination of a non-equilibrium conduction band electron as this level captures a hole. Capturing a hole means a transition of the trapped electron to the valence band. The rate of this transition increases with increasing temperature, and one notes the existence of the activation energy, preventing the immediate hole capture by negatively charged gallium vacancy. This activation energy is $\Delta E_{A,I}$, experimentally estimated at 91 meV.
- As the rate of hole capture on the V_{Ga}-level increases, the conduction band electrons have more chance for recombination on this level. This results in shorter non-equilibrium minority electron lifetime and a slower rate for L increase at higher temperatures as seen in Fig. 2.
- Trapping non-equilibrium electrons only occur on neutral V_{Ga}-levels, which do not affect the electrical conductivity type in the sample under test. P-type conductivity is dictated by negatively ionized V_{Ga} acceptors, which are unable to trap additional electrons.

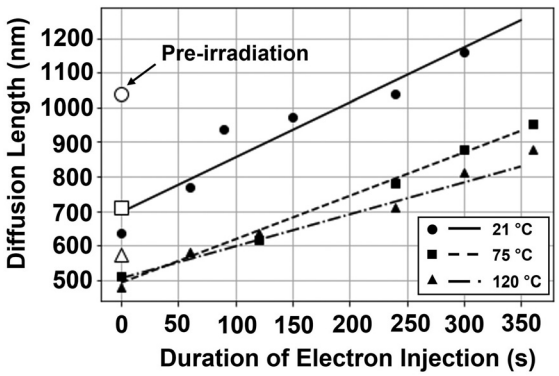

FIG. 2. The change in the diffusion length as a function of duration of electron injection for temperatures in the 294–393 K range. Pre-irradiation diffusion length values (zero injected charge) are shown in open circle, square, and triangle for 21, 75, and 120 °C, respectively. The activation energy $\Delta E_{A,I}$, obtained from L dependence on electron injection duration at varying temperatures, is estimated at 91 meV using Eq. (2).

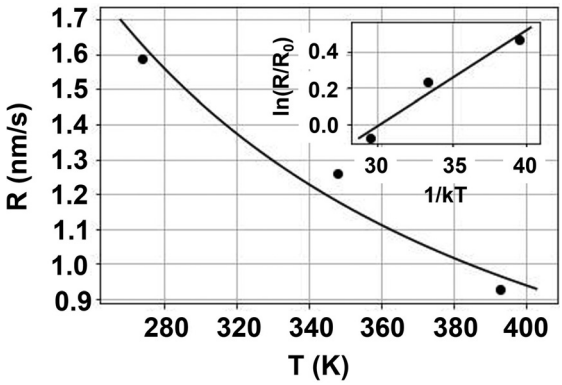

FIG. 3. Temperature dependence of the rate (R) for L change with duration of electron injection. Inset: Arrhenius plot of Eq. (2) for calculation of activation energy for the effect of electron injection.

The results in Fig. 2 demonstrate that the adverse impact of proton irradiation can be fully recovered due to electron injection, and the diffusion length in the irradiated material can increase even beyond the pre-irradiation values at respective temperatures. Stability of electron injection-induced increase in the diffusion length was studied at room temperature after stopping the electron injection (up to $\sim$ 350 s total time at each temperature). It has been found that the increased diffusion length stays unchanged for at least several days.

Figure 4 demonstrates normalized room temperature CL spectra before and after proton irradiation. Detailed studies of p-Ga_2O_3 optical properties prior to irradiation were recently presented in Ref. 22. Narrowing of the CL spectrum after proton irradiation in Fig. 4 is

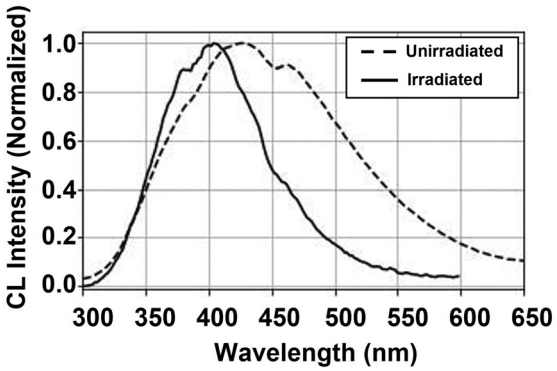

FIG. 4. Normalized room-temperature cathodoluminescence spectrum before and after proton irradiation. A slight blue shift of the irradiation peak with smaller full width at half-maxima was observed after irradiation.

likely related to a variety of complexes, which is created between V_{Ga} and implanted hydrogen. These complexes may reduce strain-induced broadening of the CL peaks.[26] CL measurements after electron injection into proton-irradiated Ga_2O_3 did not reveal any additional changes in the spectrum (both in terms of shape and intensity), indicating that the injection-induced increase in non-equilibrium carrier lifetime is mostly non-radiative in nature.

In summary, electron injection from the SEM beam was employed to "heal" the adverse impact of proton irradiation on minority carrier transport in p-type Ga_2O_3. The obtained results demonstrate that reduction in the diffusion length due to irradiation could be fully recovered, and L can even exceed the pre-irradiation values. The effect was ascribed to non-equilibrium electron trapping on native defects (V_{Ga}) and consequent increase in minority carrier (electron) lifetime in the conduction band of Ga_2O_3.

Once Ga_2O_3 p–n junctions become available, a solid-state electron injection due to forward-bias (injection of electrons from the n-side of the p–n junction into the p-side due to applying a negative voltage to the n-type material) will be attempted, thus paving the road toward purely electrical (athermal) mitigation of radiation-induced defects in bipolar devices. This type of defect mitigation does not require high temperature annealing or changes in technological processes, which are both costly. Additionally, based on the results obtained so far, the process of electron injection requires only seconds, while the induced improvements of minority carrier transport are stable for days.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Sushrut Modak: Formal analysis (lead); Methodology (lead); Software (lead); Visualization (lead). **Alfons Schulte:** Writing – review & editing (equal). **Corinne Sartel:** Methodology (equal); Writing – review & editing (supporting). **Vincent Sallet:** Methodology (equal); Writing – review & editing (supporting). **Yves Dumont:** Funding acquisition (supporting); Methodology (equal); Writing – review & editing (supporting). **Ekaterine Chikoidze:** Methodology (equal); Writing – original draft (equal); Writing – review & editing (supporting). **Xinyi Xia:** Methodology (supporting). **Fan Ren:** Methodology (supporting); Writing – review & editing (supporting). **Stephen J. Pearton:** Funding acquisition (supporting); Methodology (equal); Writing – review & editing (lead). **Arie Ruzin:** Writing – review & editing (equal). **Leonid Chernyak:** Conceptualization (lead); Data curation (equal); Investigation (equal); Methodology (equal); Supervision (lead); Writing – original draft (lead); Writing – review & editing (lead).

DATA AVAILABILITY

The data that support the findings of this study are available within the article.

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