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X-ray spectrometry investigation of electrical isolation in GaN

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Electrical isolation of *n*-type GaN epilayers bombarded with MeV light ions is studied by energy dispersive x-ray spectrometry (EDS). We show that the maximum bremsstrahlung x-ray energy (the Duane–Hunt limit) can be used to monitor the isolation process in GaN. This method allows the dose region above the threshold dose for isolation to be conveniently studied, whereas the application of conventional (low-voltage) electrical techniques in this dose range with large sheet resistances of the material ($\geq 10^{11} \Omega/\text{sq}$) is often impossible due to comparable parasitic resistances of the experimental setup. A correlation of EDS and resistance measurements of GaN strongly suggests that the magnitude of sample charging scales with the number of ion-beam-produced deep electron traps which are empty at equilibrium. The results presented demonstrate the utility of EDS as a powerful and simple technique to study electrical isolation in wide band-gap semiconductors. © 2002 American Institute of Physics.

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Ion bombardment is an important technological tool for electrical isolation of compound semiconductors.¹ Under specific conditions, ion irradiation can render the material highly resistive due to the formation of defect-related deep carrier traps. The isolation process can often be characterized by the so-called threshold dose (D_{th}), which is the ion dose necessary to produce an effective concentration of deep level traps comparable to the initial concentration of free carriers. After ion irradiation to D_{th} , material resistivity is close to its maximum value. This threshold dose depends on both material properties and implant conditions [such as ion energy, mass, implantation temperature, and (possibly) beam flux].² For effective and thermally stable electrical isolation of a particular semiconductor device structure, it is important to study the dose range close to and above D_{th} . Indeed, the optimum ion dose is usually above D_{th} to ensure that electrical isolation is thermally stable during subsequent low-temperature annealing.

Electrical isolation by ion irradiation has recently been applied for GaN, a technologically important wide band-gap semiconductor.^{2–7} In particular, it has been shown that GaN thin films can be rendered highly resistive by MeV light-ion irradiation² when projected ion ranges are significantly larger than film thickness, and depth profiles of ion-beam-generated

atomic displacements are essentially uniform within GaN epilayers. However, previous studies² have shown that isolation measurements of GaN for ion doses above D_{th} are often difficult due to very large values of sheet resistance (R_s , $\geq 10^{11} \Omega/\text{sq}$) in this dose range. This arises for a material with a large band gap (~ 3.4 eV for GaN), and, hence, very poor intrinsic conductivity, since resistances to be measured are of the same order of magnitude as typical parasitic resistances of the experimental setup.

In this paper, we present a simple (contactless) method to study electrical isolation in GaN for ion doses above D_{th} when the application of conventional electrical techniques is often impossible. The method is based on the fact that GaN rendered highly resistive by ion irradiation can exhibit strong charging effects during imaging in the secondary electron microscope (SEM).⁸ A number of different techniques have previously been applied to study (undesirable) charging effects in insulators during SEM imaging, such as the mirror method and methods based on energy shifts in Auger lines or in high-energy cutoff of the x-ray bremsstrahlung [the so-called Duane–Hunt limit (DHL)].⁹ In the present study, the electrical isolation process in GaN is monitored by measuring the DHL, which reflects electron trapping at deep defect levels produced by ion bombardment. Hence, this study demonstrates that charging phenomena, which are often undesirable during imaging in the electron microscope, can be

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FIG. 1. EDS spectra acquired from GaN (wafer A) irradiated at room temperature with 1.8 MeV He ions with a beam flux of $\sim 6 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ to different doses. Ion doses (in 10^{14} cm^{-2}) are indicated in the legend.

used to conveniently monitor electrical isolation in wide band-gap semiconductors, such as GaN.

The $\sim 2\text{-}\mu\text{m}$ -thick wurtzite n -type GaN epilayers used in this study were grown on c -plane sapphire substrates by metalorganic chemical vapor deposition (MOCVD) in a rotating disk reactor at Ledex Corporation. Wafers with room-temperature free electron concentrations of $\sim 1 \times 10^{16}$ (nominally undoped wafers A and B) and $\sim 3 \times 10^{17} \text{ cm}^{-3}$ (Si-doped wafer C) were used. Samples were irradiated at room temperature with 1.8 MeV $^4\text{He}^+$ ions with a beam flux of $\sim 6 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$ using the ANU 2 MV Van de Graaff accelerator or with 6.6 MeV $^{12}\text{C}^+$ ions with a beam flux of $\sim 6.4 \times 10^{10} \text{ cm}^{-2} \text{ s}^{-1}$ using an ANU 1.7 MV tandem accelerator (NEC, 5SDH-4). Ion energies were chosen to place the damage peak deep in the sapphire substrate, beyond the GaN layer. In this case, the distribution of ion-beam-generated atomic displacements is essentially uniform throughout the GaN film. During bombardment, samples were tilted by 7° off the surface normal direction to minimize channeling. Selected samples were also subjected to conventional furnace post-ion-irradiation annealing for 1 h at temperatures up to 700°C in a nitrogen ambient at atmospheric pressure.

After ion irradiation, samples were studied by SEM and energy dispersive x-ray spectrometry (EDS). Electron images and EDS spectra were obtained using a Philips XL30 environmental SEM equipped with an EDAX DX-4 EDS system. Imaging conditions (high-vacuum mode, electron energy = 10 keV, working distance = 10 mm, scan size = $70 \times 50 \mu\text{m}^2$, and scan rate = 10 frames/s) were kept constant during acquisition of the EDS data shown below. The electron beam current and x-ray spectrum acquisition time (200 live seconds per spectrum) were selected so as to attain satisfactory signal-to-noise ratios in x-ray data (counting rate ≈ 2000 c.p.s.) and were also kept constant.

It has previously been shown⁸ that GaN rendered highly resistive by MeV light-ion irradiation exhibits strong electric field assisted secondary electron emission (i.e., charging effects). Our present results show that the strength of such charging effects (monitored by EDS measurements) depends on implant and post-ion-irradiation conditions. Below, we first present results of EDS characterization of GaN irradiated with MeV light ions and, then, show a correlation of EDS data with resistance measurements.

FIG. 2. (a) DHL shift as a function of ion dose for 1.8 MeV He ion bombardment of GaN (wafer A) at room temperature with a beam flux of $\sim 6 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$. (b) DHL shift as a function of annealing temperature for samples (wafer B) bombarded at room temperature with 1.8 MeV He ions to a dose of $\sim 4.8 \times 10^{15} \text{ cm}^{-2}$ with a beam flux of $\sim 6 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$. Postirradiation annealing was performed for 1 h in a nitrogen atmosphere.

Figure 1 shows typical EDS spectra acquired from GaN irradiated with 1.8 MeV He ions to different doses. It is seen from Fig. 1 that, for the as-grown sample, the DHL is ~ 10 keV, the electron energy used to acquire the spectra. Conversely, spectra acquired from implanted samples clearly exhibit DHL values less than 10 keV. This decrease in the DHL results from the slowing down of the primary electrons, before they enter the sample, by an electric field above the sample.¹⁰ Such an electric field consists of two distinct components, produced during electron irradiation, as a result of (i) secondary electron emission and (ii) electrons injected and trapped in the material.¹¹ A decrease in the DHL indicates a net negative surface potential generated by trapped electrons.^{12,13}

Figure 2(a) illustrates the ion dose dependence of the DHL shift (i.e., primary electron energy minus the DHL) in GaN samples irradiated at room temperature with 1.8 MeV He ions. It is seen from Fig. 2(a) that, with increasing ion dose, the DHL shift rapidly increases after a dose of $\sim 10^{13} \text{ cm}^{-2}$, reaches the maximum, and then exhibits a decrease with further ion irradiation. Shown in Fig. 2(b) is the DHL shift as a function of annealing temperature for GaN samples bombarded at room temperature with 1.8 MeV He ions to a dose of $\sim 4.8 \times 10^{15} \text{ cm}^{-2}$, which is larger than the dose corresponding to the maximum of the DHL shift. Figure 2(b) reveals that, with increasing temperature of post-ion-irradiation annealing, the DHL shift first increases and then exhibits a decrease.

FIG. 3. Dose dependences of sheet resistance (the right axis, taken from Ref. 2) and DHL shift (the left axis) for irradiation of GaN (wafer C) at room temperature with 6.6 MeV C ions with a beam flux of $\sim 6.4 \times 10^{10} \text{ cm}^{-2} \text{ s}^{-1}$.

These main features of dose and annealing temperature dependences of the DHL shift illustrated in Fig. 2 are typical for ion dose and annealing temperature curves of R_s for a number of semiconductors.¹ For example, with increasing ion dose, R_s of GaN exhibits a fast nonlinear increase due to the trapping of carriers at ion-beam-produced defects together with damage-induced degradation of carrier mobility.² With further increasing ion dose, R_s is expected to decrease due to the onset of hopping conduction with increasing density of ion-beam-produced defects.¹ In addition, for ion doses above D_{th} , post-ion-irradiation annealing [Fig. 2(b)] results in, first, an increase in R_s (due to a partial annealing of ion-beam-produced defects, resulting in a reduction in hopping conduction) followed by a decrease in R_s (due to a further annealing of deep traps responsible for electrical isolation).

The above comparison of data from Fig. 2 with (expected) ion dose and annealing temperature dependences of R_s suggests that the DHL shift reflects the magnitude of R_s of GaN. The correlation between R_s and the DHL shift is quantified in Fig. 3, which shows ion dose dependences of these two parameters for GaN bombarded at room temperature with 6.6 MeV C ions. It is seen from Fig. 3 that a fast increase in the DHL shift occurs for ion doses above D_{th} (i.e., $\geq 10^{13} \text{ cm}^{-2}$ in this case).¹⁴ This gives a compelling argument that, for ion doses above D_{th} and below the onset of pronounced hopping condition, the DHL shift scales with the number of ion-beam-produced electron traps which are empty at equilibrium. Indeed, during electrical isolation of *n*-type GaN, all deep traps produced by ion bombardment to doses below D_{th} are filled with electrons supplied by shallow donors. Figure 3 shows that the DHL shift exhibits a fast increase only for ion doses above D_{th} when all free carriers supplied by shallow donors are trapped at ion-beam-produced deep levels. At such doses, the chemical potential moves toward the middle of the band gap, and some deep traps are empty (at equilibrium).¹⁵ These empty deep levels trap electrons injected into the material during SEM imaging, resulting in strong charging effects accompanied by a shift in the DHL. With further increasing ion dose, the number of deep traps which are empty at equilibrium also increases,

resulting in a corresponding increase in the DHL shift. Figures 2(a) and 3 reveal a decrease in the DHL shift with further ion irradiation ($\geq 10^{14} \text{ cm}^{-2}$). This decrease can be attributed to hopping conduction.

The above results demonstrate that simple DHL measurements give an ideal opportunity to study electrical isolation in GaN for ion doses above D_{th} . However, it should be noted that the magnitude of DHL shifts caused by charging cannot be always directly proportional to the surface potential of the charged sample. Such nonlinearities may result from artifacts in EDS spectra, as has been discussed in Ref. 16. However, where necessary, high precision quantitative surface potential measurements can be obtained using other techniques such as secondary or Auger electron spectroscopy.¹⁶

In conclusion, we have shown that EDS measurements can be used to study the process of electrical isolation in GaN. In particular, this method allows the dose region above the threshold dose for isolation to be conveniently studied, whereas the application of conventional electrical techniques is often impossible. In addition, such contactless EDS measurements allow the whole semiconductor wafer to be studied without cutting and give an ideal opportunity to study electrical isolation in some cases when the fabrication of ohmic contacts is not feasible. Results suggest that the DHL shift reflects the number of ion-beam-produced deep electron traps which are empty at equilibrium. This simple technique may prove useful in studies of electrical isolation in other wide band-gap semiconductors.

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¹⁵In our scenario, ion-beam-induced formation of defects with shallow levels is neglected. Although this assumption may not be strictly correct, Fig. 3 indicates that the introduction rate of deep levels, trapping free carriers, is larger than the rate of creation of defects with shallow levels. Indeed, Fig. 3 shows that R_s monotonically increases with increasing ion dose.

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