

Increase in oxide hole trap density associated with nitrogen incorporation at the SiO₂/SiC interface

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Nitrogen incorporation at the SiO₂/SiC interface via high temperature nitric oxide annealing leads to the passivation of electrically active interface defects, yielding improved inversion mobility in the semiconductor. However, we find that such nitrated oxides can possess a larger density of hole traps than as-grown oxides, which is detrimental to the reliability of devices (e.g., can lead to large threshold voltage instabilities and to accelerated failure). Three different charge injection techniques are used to characterize this phenomenon in metal–oxide–semiconductor structures: x-ray irradiation, internal photoemission and Fowler–Nordheim tunneling. Some nitrogen-based atomic configurations that could act as hole traps in nitrated SiO₂ are discussed based on first-principles density functional calculations. © 2008 American Institute of Physics. [DOI: [10.1063/1.2940736](https://doi.org/10.1063/1.2940736)]

I. INTRODUCTION

Silicon carbide has received considerable attention for its potential use in high-power devices due to its high achievable blocking voltages and good thermal conductivity, making it superior to Si for a wide range of such applications. It is also preferred over other wide band gap semiconductors because of its unique ability to grow a thermal SiO₂ gate oxide. A significant stepping stone toward the feasibility of SiC metal–oxide–semiconductor field-effect-transistors (MOSFETs) has been the incorporation of nitrogen in the gate dielectric via post-oxidation anneals which yields a tenfold reduction of electrically active defects at the complex SiO₂/4H-SiC interface and a significant increase in channel mobility. Although the atomic-scale mechanisms remain unclear, this effect has been attributed to the removal of threefold coordinated excess carbon and to the passivation of sub-oxide bonds.^{1–8}

Following this improvement, it is important to study the impact of nitrogen on the reliability of the gate oxide and of the SiO₂/SiC interface. In particular, oxide charge buildup during device operation, yielding threshold voltage instabilities in MOSFETs and ultimately inducing oxide breakdown, is a major concern inherent to the properties of the wide band gap semiconductor. Indeed, the band offsets of SiC with respect to SiO₂ are smaller than in the case of silicon which leads to more efficient charge injection into the oxide under similar operation conditions.^{9,41} Moreover, it has been shown

that hole emission from SiC is mediated by some states in the oxide band gap resulting in an even lower effective energy barrier.¹⁰ Furthermore, the use of power devices at higher fields and temperatures increases the tunneling and thermal emission probabilities of carriers not only from the semiconductor but from the gate metal as well.^{11,41} The characterization of the impact of injected charges on the oxide and on the SiO₂/SiC interface after nitrogen incorporation is therefore crucial to the establishment of SiC MOSFETs as commercial devices. Ideally, this should be understood at the atomic level and in terms of nitrogen bonding configurations.

We have recently shown that electron injection into SiC gate oxides subjected to high temperature nitric oxide (NO) annealing does not result in the generation of interface states.¹² This explains results previously observed by other groups which reported the reduced negative charge buildup in nitrated oxides when compared to as-grown oxides under such conditions.^{13–15}

In this paper, we compare the behavior of NO-annealed and as-grown oxides exposed to holes injected via three different techniques. We find that nitrogen incorporation can be detrimental to the reliability of oxide-based devices. The nature of the enhanced net positive charge buildup in nitrated samples we observe following x-ray irradiation¹⁶ is interpreted as promoted hole trapping based on results from metal–oxide–semiconductor structures subjected to internal photoemission and Fowler–Nordheim tunneling. The total deposited charge and the amount of generated interface states are deduced from capacitance-voltage (CV) measurements. Charge annealing results in the neutralization of the trapped charge and of the generated (near-) interface states. Defects responsible for such behavior are considered and the role of nitrogen is discussed in light of first-principles calculations.

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II. EXPERIMENTAL AND THEORETICAL METHODS

For our experiments, 35 to 60 nm thick oxides were grown at 1150 °C in flowing oxygen on the (0001) Si-face of RCA cleaned *n*- and *p*-type 4H-SiC wafers (doping $\approx 5 \times 10^{15} \text{ cm}^{-3}$) obtained from Cree, Inc. The samples were then annealed for 30 min in argon at the same temperature. Some were subsequently exposed to NO for 2 h at 1175 °C.

X-ray irradiation was performed using a 10 keV source while a positive bias ($E \approx 1.5 \text{ MV/cm}$) was maintained on the dc sputtered 200 nm thick Mo/Au metal gate stacks. Under these conditions, electron-hole (e-h) pairs are induced in the oxide by secondary photoelectrons from the metal and the semiconductor, and by direct interaction of x-ray photons with SiO₂. Accordingly, 1 Mrad (SiO₂) is estimated to generate about 10^{14} e-h pairs in the oxide, the majority of which (about 70%) do not recombine as they are effectively separated by the electric field. A similar oxide field ($E \approx 1.5 \text{ MV/cm}$) was used for the devices subjected, under N₂ ambient, to 10 eV [vacuum ultraviolet (VUV)] photons emitted by a Kr resonant discharge lamp through a MgF₂ window; those samples had semitransparent (15 nm thick) evaporated Au electrodes. To selectively inject holes in a third set of devices, we took advantage of the small band offset between the 4H-SiC and SiO₂ valence bands ($\approx 2.9 \text{ eV}$ versus 4.7 eV in the case of Si) and of the reduced effective barrier yielding the onset of Fowler–Nordheim hole tunneling observed in *p*-type samples at approximately -5.5 MV/cm .^{10,17} Here we used a low intensity ultraviolet (UV) light ($< 3.5 \text{ eV}$) to generate an inversion layer, source of tunneling holes, in the *n*-type capacitors under negative gate bias. Those samples had thin Al ($< 30 \text{ nm}$) evaporated gate contacts.

Two methods were used to neutralize the trapped positive charge: isochronal annealing and electron injection. Isochronal annealing was performed under positive bias ($E \approx 1.5 \text{ MV/cm}$) using 10 min steps at temperatures between 25 and 175 °C. Electrons were injected at low fields ($|E| < 3 \text{ MV/cm}$) using the focused radiation of a 100 W mercury lamp to emit free carriers from the negatively biased semitransparent metal gate.¹² The large attractive Coulomb neutralization cross section of trapped holes in SiO₂ allowed for a limited electron dose, assuring no significant background trapping of negative charge.¹⁸

All CV measurements were performed at room temperature using a Keithley model 82 or a HP 4275A LCR meter. The change in effective charge was deduced from shifts in the flatband voltages (V_{fb}) of the CV curves taken from accumulation to deep depletion.¹² The density of interface states (D_{it}), between 0.2 and 0.6 eV from the SiC band edges, was monitored through simultaneous quasistatic (0.5 V/s) and high-frequency (100 kHz) CV.¹⁹ The hysteresis of the high-frequency curves were used to detect energetically deep interface states and slow border states as a low intensity UV-light pulse was used to form an inversion layer prior to the sweep toward accumulation.²⁰

First-principles calculations were performed by applying density-functional theory, with the pseudopotential method,^{21–23} to supercells representing a SiO₂/4H-SiC

interface,¹ or amorphous SiO₂.²⁴ The exchange-correlation effects were treated with the generalized gradient-corrected exchange-correlation functionals given by Perdew and co-workers.²⁵ The Vanderbilt ultrasoft pseudopotentials were adopted. A plane-wave energy cutoff of 396 eV and the Γ point in the Brillouin zone were used for the calculations. Relaxation of the atomic structures was performed for each configuration via a conjugate-gradient technique using the total energy and the Hellmann–Feynman forces on the atoms.²¹ While all the atoms in the supercells representing amorphous SiO₂ and defects were fully relaxed, only the SiO₂ layer and the top four SiC layers for the structures involving the SiO₂/4H-SiC interface were relaxed. The equilibrium configurations were assumed when the forces on the atoms were smaller than 0.05 eV/Å.

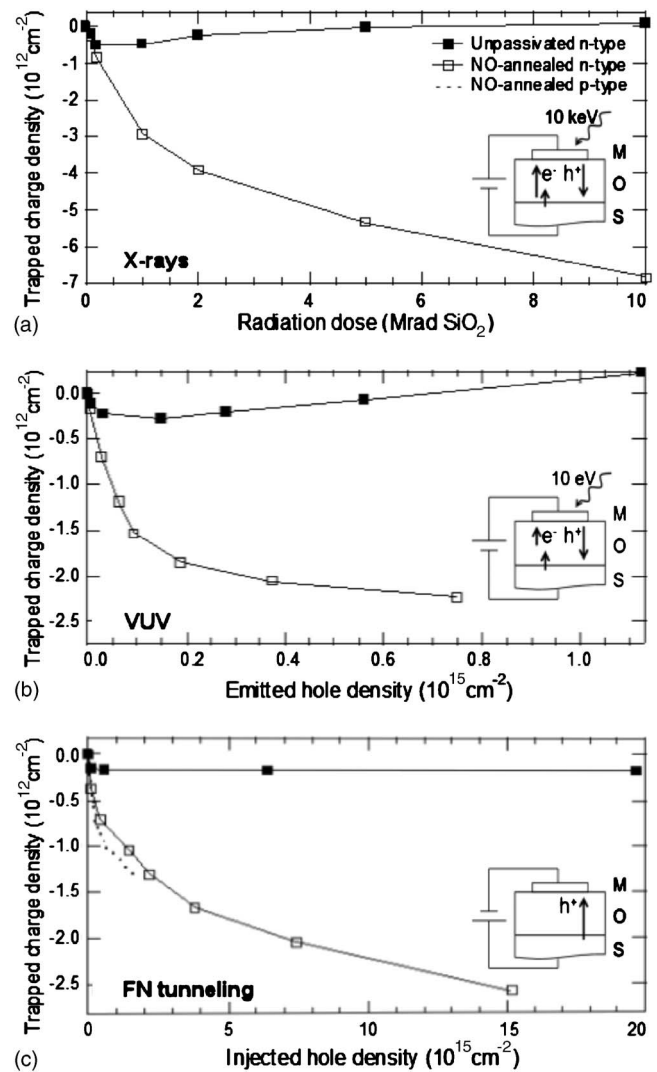


FIG. 1. Effective charge density trapped upon carrier injection by three different techniques: (a) 10 keV x-rays under positive gate bias, $E \approx 1.5 \text{ MV/cm}$, (b) 10 eV VUV photons under positive gate bias, $E \approx 1.5 \text{ MV/cm}$, and (c) Fowler–Nordheim tunneling under negative gate bias, $E \approx -6 \text{ MV/cm}$. The trapped charge density is calculated as $\Delta V_{fb} C_{ox} / q$, where C_{ox} is the oxide capacitance and q is the elementary charge; negative values imply net positive charge trapping.

III. RESULTS

A. Hole Injection

The effective trapped charge density resulting from the different injection techniques is shown in Fig. 1. It is clear that in all cases the nitrided samples exhibit a larger net positive trapping. The turn-around behavior observed in as-oxidized samples [Figs. 1(a) and 1(b)] reveals the presence of background electron trapping which yields charge compensation. Indeed, the trapping of negative charge in the bulk of the oxide, and/or the generation of interface acceptor states, can counter-balance the positive charge deposited by holes. Therefore, the sources of electrons must be identified and the background trapping must be quantified in order to highlight the response of the oxide relative to holes alone.

X-rays generate electron-hole pairs throughout the oxide.²⁶ The positive bias drives the holes to the semiconductor interface as electrons are swept toward the metal. Therefore, some negative charge trapping should be expected. Post-irradiation (10 Mrad SiO₂) isochronal anneals lead to the neutralization of the positive charge.¹⁶ The remaining negative charge due to background effects has been estimated to be approximately 2×10^{12} cm⁻² in as-oxidized samples and 9×10^{11} cm⁻² in nitrided samples. *Kr lamp 10 eV photons* are mostly absorbed in a thin oxide layer beneath the metal (SiO₂ skin depth at this energy is approximately 10 nm),^{27,28} which implies that most electrons finding their way to the SiO₂/SiC interface are the ones tunneling from the *n*-type semiconductor in accumulation.²⁹ This background effect is quantified by biasing the samples in the dark; this yields the trapping of at most 2×10^{11} electrons/cm² in all samples within the time frame of the experiments. *Fowler–Nordheim tunneling* is performed with a negative bias on the gate ($E \approx -6$ MV/cm). The resulting field is insufficient to inject electrons from the metal,¹⁰ or to induce hot carriers in the oxide.³⁰ Moreover, the absence of a turn-around in the unpassivated sample [Fig. 1(c)] confirms that there are no compensating electrons.

We conclude that in the case of x-rays and 10 eV photons, the amount of positive trapped charge in as-oxidized

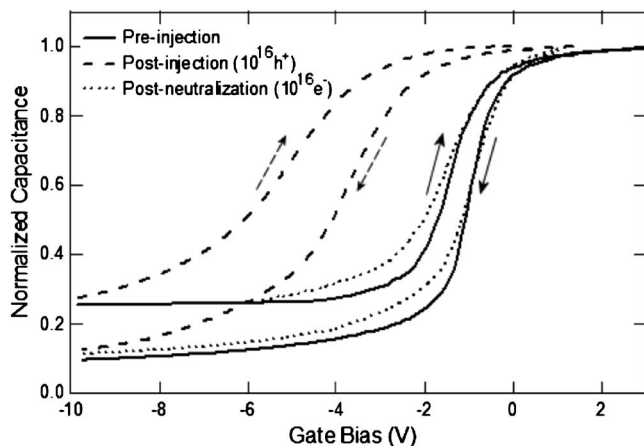


FIG. 2. Evolution of the photo-CV hysteresis of a NO-annealed *n*-type sample. A low intensity UV pulse was applied at -10 V, prior to the sweep toward accumulation, in order to form an inversion layer and empty deep/slow states.

samples is of the same order as the negative one and that there is no background effect due to electrons in the samples subject to Fowler–Nordheim tunneling. In all cases, the net positive trapped charge in nitrided capacitors by far exceeds the compensating negative charge which indeed means that the NO-annealed oxides trap holes more efficiently. Also, from the evolution of the CV stretch-out,¹⁶ and of the width of the photo-CV hysteresis (Fig. 2), we observe that states are generated as well. Since no such increase in the D_{it} between 0.2 and 0.6 eV from the semiconductor band edges could be detected, it is inferred that mostly deep states and/or border states are generated. Note that these induced states also lead to larger flatband voltage shifts in the nitrided *p*-type sample shown in Fig. 1(c).

B. Charge annealing

In order to understand the nature of the excess positive charge in nitrided samples, we performed post-injection charge annealing. Isochronal annealing leads to the removal of positive charges via hole emission.^{31,32} The emission probability at each temperature $p_{em}^*[T]$ is calculated from flatband voltage shifts of NO-annealed capacitors,

$$p_{em}^*[T] = \frac{Q_{trap}^*[T - \Delta T] - Q_{trap}^*[T]}{Q_{trap}^*[T - \Delta T]}, \quad (1)$$

where $Q_{trap}^*[T] = Q_{trap}[T, \Delta T, \Delta t]$ is the remaining positive trapped charge measured after each temperature step ΔT (i.e., 25 °C) of duration Δt (i.e., 10 min). Assuming that the charge annealing occurs predominantly via thermal emission of holes (even at room temperature, no significant neutraliza-

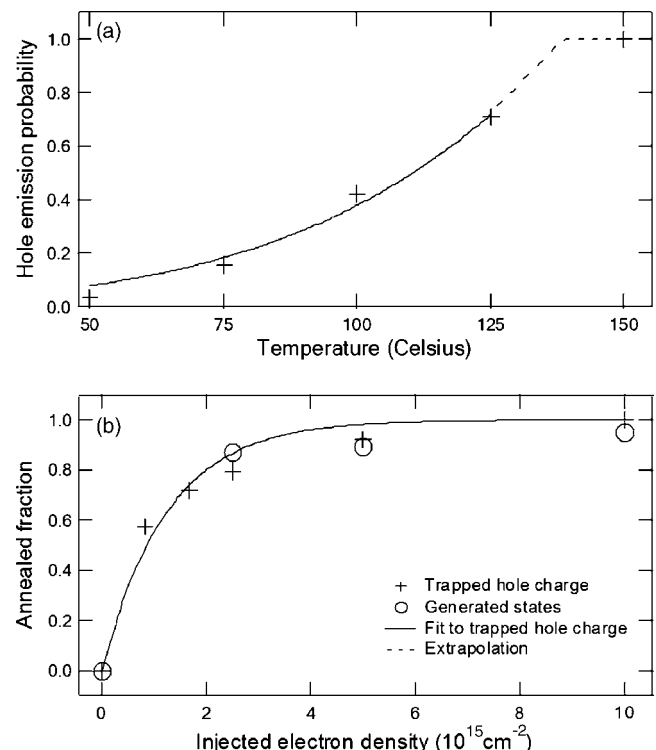


FIG. 3. Positive charge annealing in NO-treated capacitors. (a) Probability of hole emission as a function of temperature deduced from flatband recovery during isochronal annealing. (b) Fraction of the positive charge and of the generated states annealed as a function of the injected electron density.

tion by electrons tunneling from the semiconductor into positively charged states was detected within Δt ,

$$p_{\text{em}}^*[T] \propto \Delta t e_{\text{th}}[T] \propto \Delta t T^2 e^{-E_a/k_B T}, \quad (2)$$

where e_{th} is the thermal emission rate at equilibrium and k_B is the Boltzmann's constant. It yields an activation energy E_a of approximately 0.3 eV [Fig. 3(a)]. If holes emit only into the oxide valence band, this energy corresponds to an effective trap energy level relative to the valence band edge of SiO_2 . This suggests that at least some of the trap states induced by nitrogen incorporation could have energy levels located within the valence band of SiC. Also, we note that positive charge neutralization is completed at 150 °C, so that the use of devices around (or above) that temperature might prevent the observed charge buildup.

Another way to remove the positive charge accumulated after hole injection is to inject electrons. The dose dependence of the annealed charge fraction F_a in nitrated samples, exposed to photoinjected electrons ($|E| < 3$ MV/cm), is shown in Fig. 3(b);

$$F_a[t] = 1 - \frac{Q_{\text{trap}}[t]}{Q_{\text{trap}}[t=0]}, \quad (3)$$

where $Q_{\text{trap}}[t]$ is the remaining positive trapped charge after injecting the oxide with electrons for a time t . Equation (3) can be used to estimate the neutralization cross-section σ , assuming a single-process rate equation (field-induced detrapping was not observed at 1.5 MV/cm);

$$F_a[t] \equiv (1 - e^{-Jt\sigma}), \quad (4)$$

where J is the current density of electrons. We find the value of σ to be of the order of 8×10^{-16} cm², close to the measured range for hole annihilation cross sections in SiO_2 on Si.¹⁸

Interestingly, we observed that the amount of generated switching states calculated from the photo-CV hysteresis,³³ is also reduced upon charge removal. This is seen in Fig. 2 where the width and the position of the hysteresis after electron-induced annealing are very close to their pre-injection values. Moreover, the annealing rate of the generated switching states seems to match the one of the positive trapped charge [Fig. 3(b)]. These observations suggest a correlation between the density of trapped charge and (near-) interface states generation upon hole injection.

IV. DISCUSSION

The origin of hole trapping in as-oxidized SiO_2 on SiC has been previously discussed in terms of E' centers,³⁴ which are partly responsible for positive charge buildup in SiO_2/Si systems.³⁵ Also, high oxidation temperatures increase the amount of oxygen vacancies (E' precursors) in gate oxides on Si.³⁶ It is therefore important to establish if NO annealing, performed at a slightly higher temperature than oxidation (1175 °C versus 1150 °C), increases the density of oxygen vacancies in the dielectric. Electron-paramagnetic-resonance (EPR) experiments, complementing this work, indicate that NO annealing of SiO_2 grown on SiC does not lead to a

higher density of E' precursors, as evidenced by the E' signals measured in as-grown and NO-treated oxides after x-ray irradiation.

We conclude that the enhanced hole trapping in nitrated oxides on SiC does not originate in the formation of oxygen vacancies. Instead, we consider the direct relationship between nitrogen incorporation and the increase in hole trap density resulting from its possible bonding configurations. We have previously shown that NO annealing leads to a nitrogen profile peaking at the SiO_2/SiC interface and extending at most 1.5 nm into the oxide.^{37–39} The nitrogen density after the NO process is typically between 10^{14} and 10^{15} atoms/cm². Since the positive trapped charge after hole injection is found to be stable against the formation of an electron accumulation layer, the N-related traps are thought to be located within the first few monolayers of the oxide.

Theory suggests that the passivation of the interface is achieved in part by nitrogen substitution of threefold coordinated atoms (C interstitials, or Si atoms in long suboxide bonds), which removes the dangling bond states in the upper part of the SiC band gap.¹ It also results in doubly occupied levels that are located below the SiC valence band edge, but within the SiO_2 band gap. In addition, a threefold nitrogen atom has a singly occupied lone pair state, which is located in the lower part of the SiC band gap. This state could act as a trap. After capturing a hole, it becomes empty and its energy moves down toward the SiO_2 valence band edge, as does the doubly occupied state. Depending on the nature of the defects and on their distance from the interface, the relaxation, and/or the electric field associated with the capture of a hole, can affect the measured D_{it} .

In particular, two defect configurations involving N in the oxide are considered (Fig. 4). They may be formed when N or NO replaces interstitial C or CO. The one in Fig. 4(a) can also be formed when a Si–Si suboxide bond is inserted by NO. Interestingly, these configurations can result from N or NO insertion in an ideal stoichiometric oxide as well, in which case nitrogen generates states (including hole traps) within the SiO_2 band gap previously free of defect levels.

This implies that the large density of hole traps could be due to the formation of a SiO_xN_y transition layer resulting from the incorporation in SiO_2 of nitrogen atoms that are not necessarily involved in passivating the interface. It is therefore important to note that the enhanced positive charge trap-

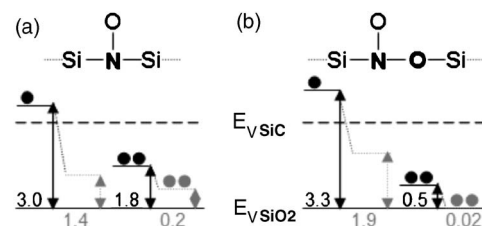


FIG. 4. Configurations resulting from N and NO incorporation in SiO_2 . The energy of the corresponding levels, are noted in electron-volts. Before trapping (labeled in black), they each have a singly occupied level and a doubly occupied level. The capture of a hole leads to a lowering of the energy states (labeled in gray). The calculated energy levels are adjusted using the experimental value ≈ 2.9 eV for the valence band offset between 4H-SiC and amorphous SiO_2 .

ping reported here after NO annealing could have the same origin as the one observed at the nitrated SiO₂/Si interface.⁴⁰ The role of nitrogen in oxide-based SiC devices remains, however, peculiar as its presence is required precisely at the interface in order to reduce the D_{it} and increase the mobility.

In an effort to optimize the effect of the nitridation, we have studied the evolution of the interface state density and of the hole trap density as a function of the amount of nitrogen incorporated.⁴¹ Results of that study will be published in a separate paper.

V. CONCLUSIONS

We have shown that the incorporation of nitrogen in dry oxides grown on SiC via high temperature NO annealing leads to a density of hole traps larger than the one measured in oxides annealed in argon. A model that correlates this increase with the bonding of nitrogen atoms in the interfacial layer is proposed. First-principle calculations suggest that the resulting nitrogen lone pairs of electrons can act as hole traps. For device applications, it is important to optimize N incorporation in order to avoid excess nitrogen content over and above that required for defect passivation at the SiO₂/SiC interface, so that both interface state density and charge buildup are reduced.

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