In-cascade ionization effects on defect production in 3C silicon carbide*

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ABSTRACT
Understanding how energy deposited in electronic and atomic subsystems may affect defect dynamics is a long-standing fundamental challenge in materials research. The coupling of displacement cascades and in-cascade ionization-induced annealing are investigated in silicon carbide (SiC). A delayed damage accumulation under ion irradiation is revealed with a linear dependence as a function of both increasing ionization and increasing ratio of electronic to nuclear energy deposition. An in-cascade healing mechanism is suggested with a low threshold value of electronic energy loss (∼1.0 keV nm⁻¹). The in-cascade ionization effects must be considered in predicting radiation performance of SiC.

IMPACT STATEMENT
A considerable impact of ionization-induced in-cascade defect annealing is presented. A surprisingly low threshold of electronic energy loss is revealed.

1. Introduction
A long-standing fundamental challenge in materials research is to understand how energy is dissipated in electronic and atomic subsystems in irradiated materials, and how related non-equilibrium processes may affect defect dynamics and microstructure evolution [1]. SiC is an important semiconductor material [2–4]. Besides its excellent mechanical properties [5,6], SiC shows remarkable thermal stability and mechanical strength at elevated temperatures [7]. Its wide band gap, high break down voltage and excellent thermal conductivity make SiC an ideal candidate for electronic applications in harsh environments [8–10]. SiC, especially its 3C polytype, is also considered as an important nuclear material. A relatively small neutron displacement cross-section [11] and a low critical temperature for amorphization [12] make SiC a promising structural material, such as a coating layer in TRISO (tristructural-isotropic) fuel particles [13]. For both electronic and nuclear applications, radiation damage from ion implantation processes or due to nuclear collisions are inevitable. Intense energy transfer in collisions leads to atomic displacements and accumulation of defects [14,15], amorphization [16], volume swelling [17] and changes in other physical properties. Moreover, ionization due to electronic energy deposition to the electronic subsystem occurs simultaneously. The ionization effects are, however, less understood, due to the inelastic interactions, non-equilibrium processes and material-dependent behavior.

Some early works on ionizing ion irradiation have focused on swift heavy ions (SHIs) [18–20]. For the SHIs, the ion energies are more than a few hundred MeV, and electronic stopping powers are more than 20 keV nm⁻¹ [18,19,21]. Defect annealing and damage
recovery are attributed to melting within the thermal spikes and epitaxial recrystallization. Recent work has drawn attention to ionization effects at a lower energy regime [1]. Ionizing irradiation with ions in the intermediate energy regime (\(\sim\) tens of MeV and electronic stopping of 5–10 keV nm\(^{-1}\)) has been shown to anneal pre-existing radiation-induced defects. This healing mechanism could allow tailoring of operating conditions and materials design to promote the repair of the damage caused by radiation exposure in nuclear reactors and space environments, and therefore increase the lifetime of materials. This ionization-induced defect annealing can be utilized to heal defects created by doping during fabrication of advanced electronic materials. However, unlike SHIs, for which the ionization due to electronic stopping is dominant, in the intermediate or lower energy regime, the electronic and nuclear stopping powers can be comparable and vary as a function of ion velocity. Most experimental investigations in this regime have focused on damage accumulation at the damage maximum [14,22–24], where ionization effects are mainly ignored.

In this work, ionization effects are studied using MeV Si ion irradiations in single crystal 3C-SiC. The general concept is derived from the basic nature of ion–solid interactions for MeV heavy ions, for which the electronic energy loss and the ratio between electronic and nuclear stopping powers (\(r_{Se/Sn}\)) decrease as the ions penetrate from the surface into the bulk. By comparing the behavior of damage accumulation at different depths as a function of the local dose, the role of ionization effects is revealed. Advancing the understanding of in-cascade ionization effects on defect accumulation in SiC will promote improved predictive modeling of the response of materials to energetic ions used in fabricating electronic devices and to the high radiation environments associated with space exploration and nuclear power generation.

### 2. Experimental details

Single crystal SiC wafers used in this study were obtained from NOVA Electronic Materials. The (001) oriented 3C-SiC films were grown on a silicon substrate. The thickness of the SiC film is 3.8 \(\mu\)m, and the strain due to lattice mismatch is negligible in our region of interest (surface to 1.5 \(\mu\)m depth). Ion irradiations and Rutherford backscattering spectrometry in channeling geometry (RBS/C) were performed at the UT-ORNL Ion Beam Materials Laboratory (IBML) [25] using a 3 MV Tandem accelerator and ion beam analysis capabilities. Two groups of Si irradiations were performed: (1) 1.5 MeV Si\(^{+}\) ion irradiation at 7\(^{\circ}\) off the surface normal direction to avoid channeling effects in the single crystals and (2) 5.0 MeV Si\(^{2+}\) ion irradiation at 60\(^{\circ}\) off the surface normal to create a shallow damage profile that is comparable with the damage profile resulting from the 1.5 MeV Si ion irradiation. The charge states of the Si ions will not affect the ion–solid interactions because the ions reach charge-state equilibration within an extremely short time in the solid target [26] and the energy losses are independent of the initial change state [27]. Ion fluences from 9.0 \(\times\) 10\(^{13}\) to 1.2 \(\times\) 10\(^{16}\) cm\(^{-2}\) were employed to create damage profiles from slightly disordered to fully amorphous. The ion fluxes were 1.4 \(\times\) 10\(^{12}\) and 3.8 \(\times\) 10\(^{12}\) cm\(^{-2}\) s\(^{-1}\) for the 5.0 and 1.5 MeV ion irradiations, respectively. RBS/C measurements employing 3.5 MeV He ions were performed in situ to obtain the damage profile for each ion fluence. All the ion irradiations and RBS/C measurements were at room temperature in a high vacuum better than 2.0 \(\times\) 10\(^{-7}\) Torr. During the ion irradiations and RBS/C measurements, a temperature increase due to beam heating was negligible (< 10 K). The depth profiles of local displacement dose (displacements per atom or dpa) were predicted using the SRIM 2008 code [28] in full cascade mode with a density of 3.21 g cm\(^{-3}\). The threshold displacement energies used were 35 eV for Si and 20 eV for C in SRIM predictions [29].

### 3. Results

The impact of ionization effects on defect production and damage accumulation during the simultaneous deposition of irradiation energy to atomic nuclei and electrons in SiC are investigated using 5.0 and 1.5 MeV Si ions (Table 1). Energy deposition to the atomic (\(dE/dx_{\text{nucl}}\)) and electronic (\(dE/dx_{\text{ele}}\)) subsystems and damage profiles are predicted by the SRIM code, where the electronic and nuclear stopping powers are less than 4.0 and 0.40 keV nm\(^{-1}\), respectively. The coupled effects are investigated with a relatively low-density energy deposition, but high \(r_{Se/Sn}\).

The results in Figure 1 show the SRIM-predicted displacement damage (right axis) and ionization energy (left axis) deposited from both ions and energetic recoils, as well as the electronic and nuclear stopping powers (left axis). For the 5.0 MeV irradiation at 60\(^{\circ}\), the energy deposition shown in Figure 1(a) is projected perpendicular to the surface; however, the actual stopping powers in the SiC target deposit energy along the ion penetration path. Since the ion path length (60\(^{\circ}\) off the surface normal) is twice as long as the projected ion range, half of the predicted energy loss values (per unit depth) in Figure 1 were included as the actual stopping powers shown in Table 1. It is known that the surface may act as a defect sink, but the active range is not expected to be more than
Table 1. SRIM-predicted energy deposition.

| Depth [nm] | 5.0 MeV Si at 60° |  |  | 1.5 MeV Si at 7° |  |  |
|-----------|-------------------|------|------|-----------------|------|------|
|           | \(\frac{dE}{dx_{\text{ele}}}\) (keV nm\(^{-1}\)) | \(\frac{dE}{dx_{\text{nucl}}}\) (keV nm\(^{-1}\)) | Ratio | \(\frac{dE}{dx_{\text{ele}}}\) (keV nm\(^{-1}\)) | \(\frac{dE}{dx_{\text{nucl}}}\) (keV nm\(^{-1}\)) | Ratio |
| 200       | 3.54              | 0.041 | 87.0 | 1.84            | 0.109 | 17.0 |
| 300       | 3.25              | 0.052 | 62.8 | 1.62            | 0.126 | 12.8 |
| 400       | 2.91              | 0.067 | 43.4 | 1.41            | 0.149 | 9.4  |
| 500       | 2.51              | 0.088 | 28.5 | 1.21            | 0.178 | 6.8  |
| 600       | 2.04              | 0.117 | 17.4 | 1.04            | 0.217 | 4.8  |
| 700       | 1.56              | 0.155 | 10.0 | 0.90            | 0.268 | 3.3  |
| 800       | 1.11              | 0.199 | 5.6  | 0.75            | 0.331 | 2.3  |
| Peak      | 0.41              | 0.202 | 2.0  | 0.25            | 0.297 | 0.85 |

Notes: Silicon ions with 5.0 MeV (left) and 1.5 MeV (right) at different depths: electronic stopping powers \(\frac{dE}{dx_{\text{ele}}}\), nuclear stopping powers \(\frac{dE}{dx_{\text{nucl}}}\) and the corresponding ratio \(\frac{r_{\text{Se}}}{r_{\text{Sn}}}\) of \(\frac{dE}{dx_{\text{ele}}}\) to \(\frac{dE}{dx_{\text{nucl}}}\). For the 60° irradiation, the cited values are the actual stopping powers per unit path length (not per unit depth), since the path length is twice the predicted project range perpendicular to the surface, as shown in Figure 1(a).

Figure 1. Energy deposition and damage dose in SiC under Si ion irradiations: (a) 5 MeV Si irradiation at 60° off surface normal direction; and (b) 1.5 MeV Si with an incident angle of 7°, where the left axis of (a) and (b) is electronic stopping power (dots), nuclear stopping power (dash dots) and total ionization (solid lines) profiles, and the right axis is the SRIM-predicted displacement dose (dpa), shown as dashed lines, for a fluence of \(1.0 \times 10^{14} \text{ cm}^{-2}\). The measured relative disorder levels for (c) 5.0 MeV Si and (d) 1.5 MeV Si irradiation to different fluences is determined using an iterative procedure, respectively.

For both irradiation conditions, the displacement damage peaks are located close to a depth of 990 nm. The damage accumulation behavior under both irradiation conditions should be comparable, with similar effects, if any, of the surface.

The disorder profiles resulting from 5.0 to 1.5 MeV Si ion irradiations at different fluences are calculated by an iterative procedure [32] and shown in Figure 1(c,d), respectively. The damage accumulation as a function of local dose is plotted at different depths with different \(r_{\text{Se}}/r_{\text{Sn}}\) and summarized in Figure 2(a–d). Ionization has its maximum at the surface (Figure 1(a,b)), which is 7.9 and 2.3 keV nm\(^{-1}\) for 5.0 and 1.5 MeV Si ions, respectively. The energy of the ions decreases along their path length, with \(\frac{dE}{dx_{\text{ele}}}\) gradually decreasing with depth and \(\frac{dE}{dx_{\text{nucl}}}\) increasing until the damage peak region at \(\sim 990\) nm. As a result, \(r_{\text{Se}}/r_{\text{Sn}}\) decreases considerably from \(\sim 87\) and 17 to 2.0 and 0.85 for 5.0 and 1.5 MeV Si ions (Table 2), respectively. Inspection of the experimentally determined radiation damage profiles shown in Figure 1(c,d) reveals a similar shape as the SRIM-predicted displacement damage for the low-fluence samples before the completely amorphous state is achieved. With further irradiation, the disorder level increases in width. Under both Si irradiation conditions, complete amorphization
Figure 2. Disorder fraction as the function of displacement dose: (a) at the damage peak for 1.5 and 5.0 MeV Si irradiations and at shallower depths for (b) 5.0 MeV Si irradiation at 60° off surface normal direction and (c) 1.5 MeV Si irradiations at 7°. For convenience, the electronic stopping power (keV nm\(^{-1}\)) and the corresponding values of \(r_{\text{Se}}/r_{\text{Sn}}\) (Table 1) are included for each depth. Also included in (b) and (c) is the curve fit to the damage accumulation (dashed line) at the damage peak from (a). The critical doses for amorphization are determined from the damage accumulation, as shown in (d) for different depths.

Table 2. Delayed damage accumulation.

| Depth (nm) | \(r_{\text{Se}}/r_{\text{Sn}}\) | 0.10 | 0.15 | 0.20 | 0.10 | 0.15 | 0.20 |
|-----------|-----------------|-----|-----|-----|-----|-----|-----|
|           |                 |     |     |     |     |     |     |
| 200       | 87.0            | 0.129, 54% | 0.179, 53% | N/A          | 17.0 | 0.181, 71% | 0.236, 70% | 0.284, 69% |
| 400       | 43.4            | 0.178, 74% | 0.244, 73% | 0.300, 70% | 9.4  | 0.233, 78% | 0.307, 79% | 0.368, 80% |
| Peak      | 2.0             | 0.241 | 0.336 | 0.426 | 0.85 | 0.241 | 0.336 | 0.426 |

Notes: The relative disorder induced by silicon ions with 5.0 MeV (left) and 1.5 MeV (right) at different 200 nm, 400 nm, and the damage peak region at \(\sim 990\) nm. The disorder level is given at 0.10, 0.15 and 0.20 dpa. The disorder at 200 and 400 nm relative to the peak disorder at each given dose are shown as percentage for easy comparison. The corresponding curves are shown in Figure 2(b, c).

(disorder level = 1.0), as determined at the damage peak, occurs at an ion fluence of \(\sim 1.1 \times 10^{15}\) cm\(^{-2}\) (\(\sim 0.6\) dpa), as shown in Figure 1(c,d) and more clearly in Figure 2(a). In the damage peak region, the ion energy deposition to the atomic subsystem is comparable to (5.0 MeV Si), or higher (1.5 MeV Si) than that of, the electronic subsystem (Table 1). Since the energy deposition processes in the damage peak region are similar under both irradiation conditions, a similar damage response, as demonstrated in Figure 2(a), is expected. The slight deviation of the data points from the trend line is within the experimental uncertainty. At ion fluences above \(1.1 \times 10^{15}\) cm\(^{-2}\), the buried amorphous layer expands towards both the surface and deeper into the bulk. A broad buried amorphous layer, over 500 nm in width, is formed under irradiation with 5.0 MeV Si ions to a fluence of \(1.9 \times 10^{15}\) cm\(^{-2}\). In the case of irradiation with 1.5 MeV Si ions to a fluence of \(1.2 \times 10^{16}\) cm\(^{-2}\), an amorphous layer over 700 nm in width is created, with the disorder level exceeding 0.8 at the surface.

To better predict SiC performance under irradiation and evaluate the effects of ionization, defect evolution and damage accumulation under different energy deposition conditions (different \(dE/dx_{\text{ele}}, dE/dx_{\text{nucl}}\) and \(r_{\text{Se}}/r_{\text{Sn}}\)) are examined. The effect of varying \(r_{\text{Se}}/r_{\text{Sn}}\) is determined through analyzing the disorder level at different depths. As shown in Table 1, electronic stopping is the primary process for energy deposition at the intermediate region from 200 to 600 nm. For these large \(r_{\text{Se}}/r_{\text{Sn}}\) values, energy
loss is dominated by ionization processes. With increasing depth, nuclear energy loss increases and the \( r_{\text{Se}/\text{Sn}} \) values become relatively small, as the atomic displacement processes become more dominant. The damage accumulation behavior at different depths, or for different values of \( r_{\text{Se}/\text{Sn}} \), is shown in Figure 2(b,c) as a function of local dose for the 5.0 and 1.5 MeV Si ion irradiation, respectively. For clarity purposes, the corresponding values of \( dE/dx_{\text{ele}} \) (keV nm\(^{-1}\)) and \( r_{\text{Se}/\text{Sn}} \) at each depth are shown. The result determined from the damage peak region, with \( r_{\text{Se}/\text{Sn}} \leq 2.0 \), is also included as a reference and shown as a dashed line. The role of ionization can be evaluated by comparing the trend of damage accumulation.

Evaluation of the damage accumulation at the damage peak region (\( \sim 990 \) nm) with respect to the intermediate depths from 200 to 500 nm is shown in Figure 2(b,c), and a much lower disorder level is observed at the shallower depths. Under the 5.0 MeV Si ion irradiation at depths of 200 and 400 nm, where \( r_{\text{Se}/\text{Sn}} \) is 87 and 43, respectively, the disorder level is only about 53–54% or 70–74% (Table 2) relative to the damage peak region. Similar damage reduction is observed under the 1.5 MeV Si irradiation at depths of 200 and 400 nm, where \( r_{\text{Se}/\text{Sn}} \) is 17 and 10, respectively, and the disorder level is 69–71% and 78–80%, respectively, relative to the peak region. It is worth noting that lower relative disorder is observed under the 5.0 MeV Si irradiation, compared to the 1.5 MeV results. This reduction is attributed to a more effective in-cascade ionization-induced annealing under irradiation with 5.0 MeV Si ions. As shown in Table 1, in the intermediate region (200–600 nm), \( dE/dx_{\text{ele}} \) is about twice as large in the case of 5.0 MeV Si than for 1.5 MeV Si, while the corresponding \( dE/dx_{\text{nucl}} \) is about half. Therefore, \( r_{\text{Se}/\text{Sn}} \) (5.0 MeV) is about 3.6–5.1 times larger than \( r_{\text{Se}/\text{Sn}} \) (1.5 MeV). At the same depth of 200 or 400 nm, the lower disorder under 5.0 MeV Si ion irradiation cannot be attributed to surface effects. The clearly slower damage accumulation observed in the intermediate region, when compared with the damage peak region, under 5.0 MeV Si irradiation reveals the impact of ionization effects and distinctively coupled in-cascade defect annihilation. The observed difference in damage accumulation is attributed to in-cascade ionization-induced recovery from the higher \( dE/dx_{\text{ele}} \).

Additional evidence for in-cascade ionization-induced defect annihilation is the significant increase of the amorphization dose, shown in Figure 2(d), for 1.5 MeV Si irradiation. The critical dose for amorphization is determined to be \( > 1.2, 1.0 \) and 0.7 dpa at 200, 600 and 950 nm, respectively. Delayed damage accumulation is also demonstrated in the results shown in Figure 2(b,c), where all the trend lines at different depths are delayed when compared with the disorder accumulation at the damage peak. The delay in damage accumulation is attributed to more effective defect annihilation, resulting from both simultaneous in-cascade recovery (coupled energy deposition in both subsystems) and from ionization-induced recovery of previously created irradiation-induced defects that occurs continuous along the ion path for electronic energy loss above the threshold of 1.4 keV nm\(^{-1}\) determined previously [1].

For the 1.5 MeV Si irradiation, the trend of shifting to higher dose to reach the same level of disorder at shallower depths clearly indicates more effective defect annealing, resulting from the increase of \( r_{\text{Se}/\text{Sn}} \) from \( \sim 0.85 \) to 17 and the decrease of energy deposition to the atomic subsystem (0.426–0.108 keV nm\(^{-1}\)), as indicated in Table 1. While ionization-induced annealing of pre-existing damage is active above 1.4 keV nm\(^{-1}\) [1], ionization-induced annealing process is also observed at 400, 500 and 600 nm, as shown in Figure 2(c,d), where the electronic energy deposition is 1.41, 1.21 and 1.04 keV nm\(^{-1}\), respectively. This suggests that in-cascade ionization-induced annealing has a lower electronic energy loss threshold at \( \sim 1.0 \) keV nm\(^{-1}\), less than the threshold of 1.4 keV nm\(^{-1}\) determined in a separate effects study [1].

In-cascade ionization-induced defect annihilation is further demonstrated in Figure 3 from the increasing critical dose for amorphization with the increasing ratio of electronic to nuclear energy loss. Under Si irradiations at room temperature \( (T_{\text{RT}}) \), the thermally induced recovery is insignificant, the critical dose, \( D \), for amorphization has a general expression that [33,34]:

\[
D = D_0/[1 - (\sigma_r/\sigma_d) \exp(-E_{\text{int}}/kT_{\text{RT}})],
\]

**Figure 3.** The linear dependence of the inverse of the critical amorphization dose \((1/D)\) on the ratio of ionization to dpa rate. The critical dose is determined at a relative disorder level of 0.97.
where $D_0$ is the amorphization dose at 0 K or at a temperature where flux effects are negligible, $E_{irr}$ is the activation energy for the irradiation-induced recovery, $\sigma_r$ and $\sigma_d$ are the irradiation-induced recovery cross-section and the damage cross-section, respectively. For the 1.5 MeV Si irradiation, the critical dose, $D$, can be estimated from the disorder profiles at different depths. To reduce the analysis error, the critical dose $D$ is estimated at a disorder level of 0.97. Based on Equation (1), $1/D$ should be linearly proportional to $\sigma_r/\sigma_d$, and as shown in Figure 3, $1/D$ exhibits a linear dependence on the ratio of ionization to dpa rate. These results suggest that ionization-induced recovery and displacement damage production through elastic collisions are the competing mechanisms responsible for the observed delayed damage accumulation shown in Figure 2.

### 4. Discussion

Normally when materials are bombarded with energetic particles, such as ions or neutrons, the materials are damaged at the atomic scale. The accumulation of radiation defects results in structure and property degradation. Recent work of Zhang et al. [1] has demonstrated that pre-existing defects in SiC can be healed by exposure to a beam of highly energized charged particles, due to the energy transferred to the target electrons that causes a highly localized thermal spike that promotes healing. The present work has further revealed that in-cascade ionization effects are also effective in suppressing or delaying damage accumulation. The experimentally determined threshold for the in-cascade ionization-induced annealing is an electronic energy loss of $\sim 1.0$ keV nm$^{-1}$, which is slightly less than the threshold of 1.4 keV nm$^{-1}$ determined from the previous separate effects study [1].

The exposure of SiC to energetic ions, even with energies of a few MeV, can cause local heating along the ion path and thermally instability of the potential energy landscape within the cascades. Such non-equilibrium processes can result in a significant reduction in the formation of stable defects from cascade events, as well as structural disorder at the microscopic and atomic scales. Both ionization-induced healing of pre-existing defects from our previous study [1] and suppression of the defect production within cascade events in this work draw attention to the fact that energy transferred to electrons in SiC by energetic ions via ionization can effectively heal defects and restore structural order. Because SiC is a critical material for nuclear applications and power electronics for space applications, ionization-induced self-healing may contribute to increased radiation tolerance in extreme radiation environments.

### 5. Conclusion

In-cascade defect, production and annealing have been studied in single crystal 3C-SiC using MeV Si ion irradiations. This work has demonstrated that, in the MeV energy regime, damage accumulation is effectively delayed as the ratio of electronic to nuclear stopping, $r_{Se}/s_{Nuc}$ increases. The critical dose for amorphization increases as a function of ionization to dpa rate. For in-cascade annealing, a threshold value of ionization is estimated to be surprisingly low at $\sim 1.0$ keV nm$^{-1}$, which is in the region of interest for routinely utilized ion implantation doping and ion irradiation processes. Such a significant healing mechanism must be considered in evaluating the response of SiC to ion implantation processes for the electronic devices and to the high radiation environments in space exploration and nuclear applications.

### Disclosure statement

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