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# Transmutation of the Crystalline Structure of $\beta$-SiC Nanowires to an Amorphous Structure Through Cu Ion Shelling

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The amorphous structural study of silicon carbide nanowires (SiC-NWs) has drawn strenuous attention in recent years due to their worthwhile properties for wide applications, chiefly in optoelectronics. The facile transformation of crystalline SiC-NWs to amorphous defective SiC-NWs is a challenging task for their broad-scale applications. Herein, we report a fantastic strategy (by applying a 5UDH Pelletron accelerator, located at the National Centre for Physics, Islamabad, Pakistan) for Cu ion implantation (fixed at 10 MeV) on the crystalline SiC-NWs to incorporate them into an amorphous structure. For the defects study, various dose rates of $Cu^{+}$ ion ranging from $5 \times 10^{15}$ ions/cm$^2$ to $5 \times 10^{16}$ ions/cm$^2$ were bombarded on SiC-NWs, and a complete transmutation to the amorphous structure of SiC-NWs under a shelling dose of $8 \times 10^{16}$ ions/cm$^2$ was observed. This work will provide a better avenue for the structural deformation blueprints of the next-generation nanomaterials. Amorphous structural transformation is explained by collision cascade effects phenomena.

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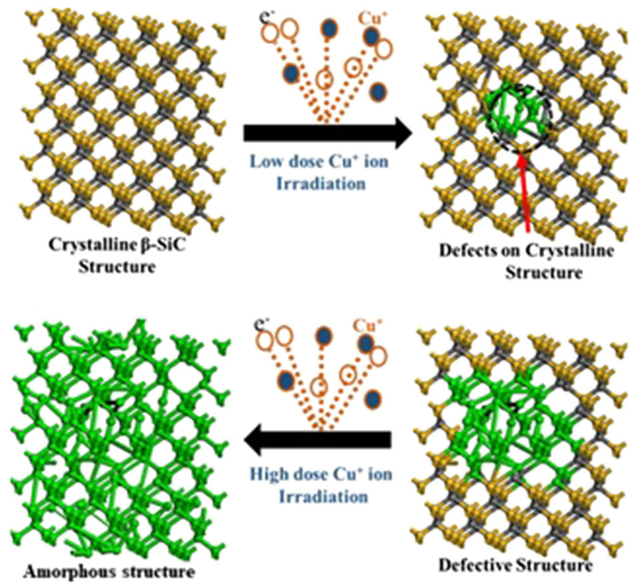


**Key words:** SiC nanowires, Cu ion irradiation, structural transformation, amorphization

## INTRODUCTION

One-dimensional (1D) nanomaterials have garnered intense interest for use in optoelectronic devices because of their inherent unique physical properties.[1,2] Among the myriad 1D nanomaterials, silicon carbide nanowires (SiC-NWs) have attracted much attention due to their wide bandgap, chemical inertness, high thermal conductivity, high breakdown field, high field emission, high electron mobility and biocompatibility.[3–5] Owing to these outstanding properties, SiC-NWs have been employed extensively in many applications such as field emission displays, nanosensors, nanoscale electronic devices and optoelectronic devices.[2,6–8] Recently, amorphous nanomaterials, especially nanowires, have become the subject of intensive research, due to their microstructural properties.[9] Consequently, there is an interest in exploring the techniques of their fabrication and characterization.[10]

There are many methods by which amorphous nanowires have been prepared, including electrodeposition, thermal vapor transfer, chemical vapor deposition (CVD), thermal heating of target materials mixed with a catalyst, and ion beam irradiation.[10–20] Among these fabrication techniques, ion beam-induced amorphization is important owing to its precise control and reproducibility.[10] This method can be useful for most crystalline NWs to transform them into amorphous NWs. However, the fabrication of amorphous SiC-NWs is still a major challenge. Transmutation of crystalline to amorphous SiC-NWs through ion beam shelling is tricky, and to our knowledge, only a few studies have been reported in the literature. The fabrication of amorphous SiC NWs by ion bombardment was demonstrated by Liu et al. using an approach that is different from the one presented in the current work.[21] The amorphization of SiC by ion bombardment of nanotubes (NTs) was demonstrated by Taguchi et al.[22] Jiang et al. obtained full amorphization of Au ion-induced 3C-SiC induced by high ion flux due to sluggish migration of point defects.[23] Bockstedte et al. used density functional theory for the ab initio study of intrinsic defects of 3C-SiC for their carbon-split interstitials and silicon interstitials.[24]

We are creating a database of ion irradiation-induced NWs, NTs and nanoparticles (NPs). We have also synthesized NPs such as AgNWs, CuNWs, NiNWs and ZnNws, and want to further expand the study to SiC-NWs, which have not been explored using an ion beam. This database will be useful for scientists to design NW-based devices for harsh (radiation) environments.

In this work, we have successfully demonstrated the fabrication of amorphous silicon carbide nanowires (a-SiC-NWs) by ion beam shelling. In this technique, SiC-NWs have been irradiated with copper (Cu) ions of different doses (as described in Fig. 1), keeping constant the parameters of irradiation current, energy and substrate temperature, which leads to the transmutation of a crystalline phase to an amorphous. The formulated amorphous SiC-NWs may find application for future smart optoelectronic devices.

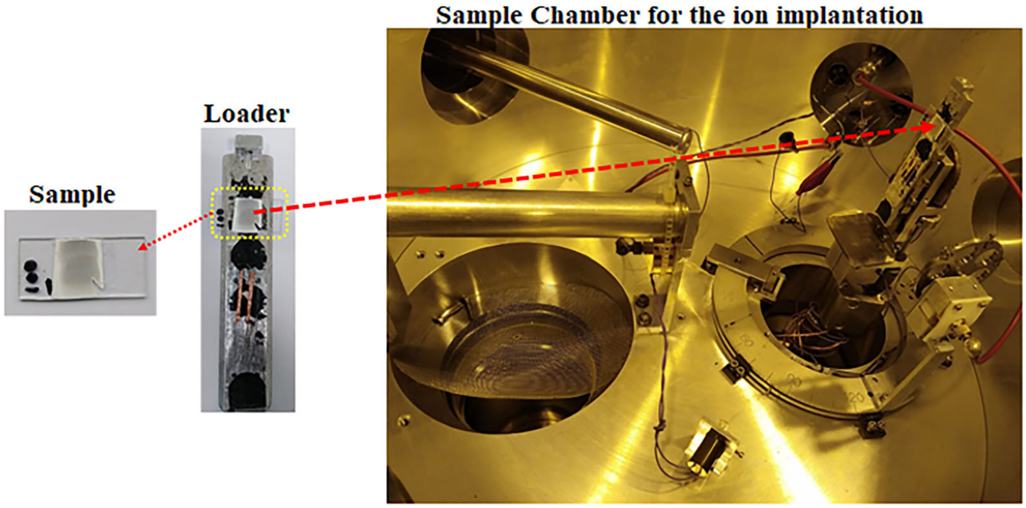


Fig. 1. Schematic illustration of Cu ion implantation in SiC-NWs: (a) sample, (b) sample loader, (c) chamber.

**Table I. Simulation results of Cu ion interaction with SiC at energy of 10 MeV and incident angle of 90°**

| Total ions | Layer width (Å) | Total transmitted ions | Vacancies/ion | Energy loss in ions (%) | | Energy loss in recoil (%) | |
|---|---|---|---|---|---|---|---|
| 1000 | 6000 | 1000 | 931.6 | 96.75 | 0.03 | 1.85 | 1.3 |

## MATERIALS AND METHOD

The crystalline SiC-NWs were procured from ACS Material, LLC (product ID: NWSH0101). SiC-NWs had a diameter between 100 nm and 600 nm, and their length was in the micrometer range. SiC-NWs were dispersed directly on a copper grid attached to a piece of glass slide for a direct ion irradiation experiment. The experiment was designed by simulation using Stopping and Range of ions in Matter (SRIM) software[25] and the simulation results of Cu ion interactions with SiC are shown in Table I. The energy of Cu ions was fixed at 10 MeV in order to permit Cu ions through the SiC-NWs instead of implantation, whereas the current of Cu ions was also fixed at 50 nA to avoid ion beam-induced crystallization due to local heating along the ion track.[26] The prepared samples were then irradiated with a Cu ion beam at various fluences ranging from $5 \times 10^{15}$ ions/cm$^2$ to $5 \times 10^{16}$ ions/cm$^2$ in a 5UDH-Pelletron accelerator under room temperature. The sample was placed on a sample holder and then transferred to the chamber before irradiation with different fluences, as described in Fig. 1.

### Characterization Techniques

The structure and morphology of un-irradiated and Cu ion-irradiated SiC-NWs were analyzed through powder X-ray diffraction (PXRD) spectroscopy at room temperature, employing a Shimadzu XRD-6000 diffractometer using a Cu$K_\alpha$(0.154 nm) radiation source in a $2\theta$ range of 20°–70°, high-resolution transmission electron microscopy (HRTEM, JEOL JEM-2010) with an accelerating voltage of 200 kV and PXRD using an X'Pert PRO 3040/60 with a Cu$K_\alpha$ radiation source ($\lambda$ = 0.154 nm) from a generator operating at 40 kV and 40 mA.

## RESULTS AND DISCUSSION

### XRD Analysis

The XRD spectrum analysis of un-irradiated SiC-NWs (Fig. 2a) shows two dominant peaks indexed to (111) and (220) that match well with the face-centered cubic (FCC) structure of 3C-SiC, also called β-SiC, with JCPDS card no. 29-1129; $a$ = 4.359 Å. The relative intensity and narrow width of the peaks show that SiC-NWs are crystalline in nature, which confirmed the results of selected area electron diffraction (SAED) and TEM. The XRD spectrum of SiC-NWs after Cu ion beam irradiation with a dose of $5 \times 10^{15}$ ions/cm$^2$ (as shown Fig. 2b) exhibited a width broadening in the dominant (111) and (220) peaks. This change can be attributed to attrition in a long-range crystalline structure, or as a result of the formation of disordered pockets within the structure. Furthermore, the reduction in the intensity of the XRD peaks also shows withering in the

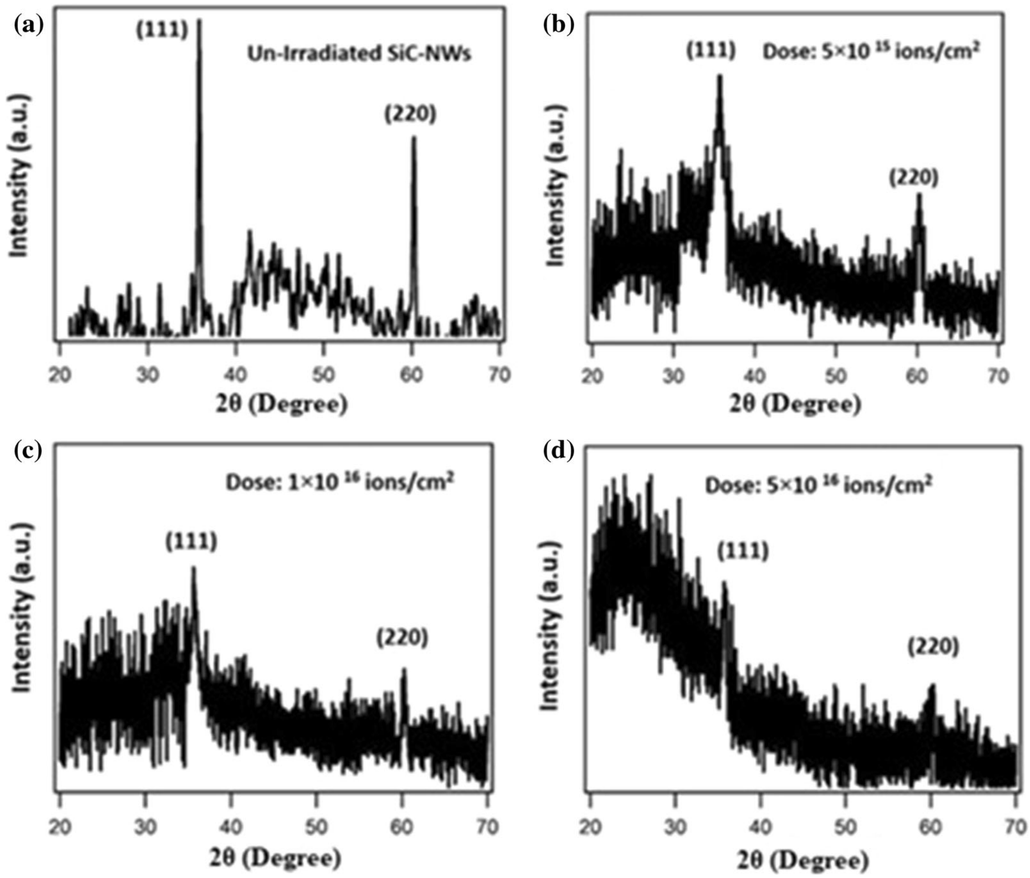


Fig. 2. PXRD analysis of (a) un-irradiated crystalline SiC, (b, c) partially crystalline SiC-NWs irradiated by Cu ion beam at doses of $5 \times 10^{15}$ ions/cm$^2$ and $1 \times 10^{16}$ ions/cm$^2$, respectively, and (d) amorphous SiC-NWs irradiated by Cu ion beam at a dose of $5 \times 10^{16}$ ions/cm$^2$.

crystallinity of SiC-NWs. As the Cu ion beam irradiation dose increases to $1 \times 10^{16}$ ions/cm$^2$, the crystallinity of SiC-NWs decreases, as shown in Fig. 2c. A stark decrease in peak intensities together with peak broadening showed drastic damage to the crystalline structure and the growth of amorphous zones. A further increase in the irradiation dose up to $5 \times 10^{16}$ ions/cm$^2$ leads to a complete transformation of the SiC-NW crystal structure into an amorphous structure, as shown in Fig. 2d. The XRD results are in consistent agreement with SAED and HRTEM results.

## HRTEM Analysis

The TEM, HRTEM and SAED images of SiC-NWs were analyzed and are presented in Fig. 3. The results of un-irradiated SiC-NWs in Fig. 3a (i) show clearly that SiC-NWs have uniform morphology. The growth direction along the (111) plane, as shown in Fig. 3a (ii), with a distance of 0.26 nm separating the adjacent planes is evident by selected-area electron diffraction (SAED) as shown in Fig. 3a (iii). Figure 3a (iv), which is the analysis of the degree of lattice distances and crystallinity, also shows the existence of sharp peaks, indicating a good crystallinity. Figure 3b (i) shows the TEM, HRTEM and SAED images of SiC-NWs bombarded by a $Cu^+$ beam at a dose of $5 \times 10^{15}$ ions/cm$^2$. It can be seen that as a result of ion beam bombardment, point defects such as interstitials and vacancies are created on the SiC-NWs, as shown in Fig. 3b (iii), whereas the SAED image in Fig. 3b (ii) portrays a marked decrease in crystallinity due to a collision cascade effect. Although no change in lattice distances was noted, as seen in Fig. 3b (iv), the broadening of peak width exhibits a shift toward low ordering in the crystalline structure. As the dose of $Cu^+$ beam is increased to $1 \times 10^{16}$ ions/cm$^2$, the amorphous structure progressively grows with a rise in Cu ion fluence, as shown in Fig. 3c (i). The amorphous phase becomes dominant in the structure of SiC-NWs. A few crystal planes still exist, as shown in the SAED results [Fig. 3c (ii)], and the appearance of point defects in the crystalline SiC-NWs structure is pronounced, as seen in Fig. 3c (iii), due to the formation of interstitial and vacancy loops. Further, the lattice analysis indicates that the amorphous structure has become larger and that the crystallinity is reduced, as observed in Fig. 3c (iv). Upon a further increase in the irradiation dose up to $5 \times 10^{16}$ ions/cm$^2$, an amorphous structure grows completely, leading to the transformation of SiC-NWs into the amorphous structure.

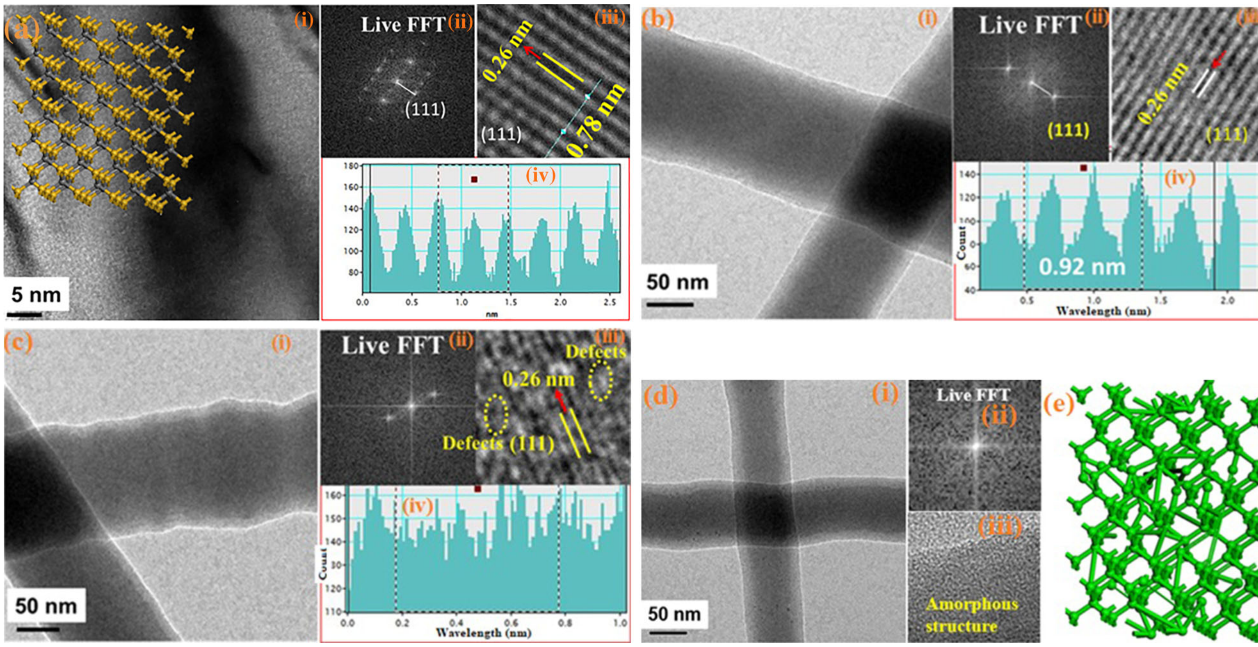


Fig. 3. TEM, HRTEM and SAED analysis of (a) un-irradiated crystalline SiC, (b, c) partially crystalline SiC-NWs irradiated by a Cu ion beam at dosages of 5 × $10^{15}$ ions/$cm^2$ and 1 × $10^{16}$ ions/$cm^2$, respectively, (d) amorphous SiC-NWs irradiated by a Cu ion beam at a dose of 5 × $10^{16}$ ions/$cm^2$, and (e) atomic distribution in the amorphous structure of SiC-NWs.

HRTEM and SAED results, as shown in Fig. 3d (i, ii & iii), confirmed the transformation to amorphous SiC-NWs. It is evident that such changes are attributable to the interstitials and/or presence of vacancies in the structure.[17] Since the ion beam heating was minimized by keeping the current at a low level (≃50 nA), no change in the morphology of the SiC-NWs was observed. It is important to note that ion beam current plays a significant role in the transition of crystalline SiC-NWs into relatively defective NWs without changing their morphology. Similar results were observed for Ag-NWs at a high radiation dose and under high C ion current.[27]

## Amorphization Process of SiC-NWs

Ion beam irradiation-induced phase transformation is a nonequilibrium process wherein thermodynamically stable NW structures can be transformed into a meta-stable structure.[28,29] In this work, an ion beam is utilized as an option to fabricate a new class of nanostructured materials such as amorphous nanowires. In the phase transformation process under the ion beam, the collision cascade effect is dominated by heavy ions, such as Cu ion beam irradiation on SiC-NWs. The Cu ions transferred their kinetic energy to target Si and C atoms in the SiC-NWs. As a result, target atoms gain kinetic energy and cause displacement from crystal planes, creating point defects such as interstitials and/or vacancy defects due to knock-on collision cascade effects.[15,30] This causes instability in the SiC-NWs structure, leading to phase transformation from a crystalline into an amorphous phase. The schematic diagram of the amorphization

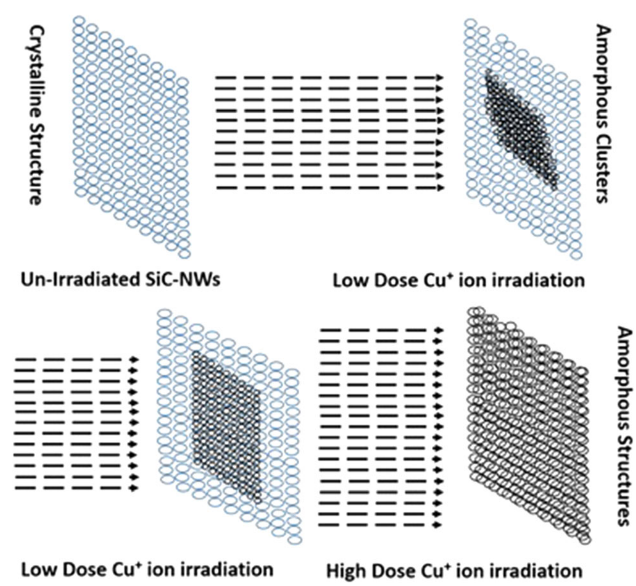


Fig. 4. Amorphization process of SiC-NWs.

process of SiC-NWs is shown in Fig. 4. At a low ion irradiation dose, interstitials and vacancy loops are created. These defects and loops are agglomerated to form amorphous zones at medium doses. Finally, at a higher dose, the whole crystalline SiC-NWs are transformed into SiC-NWs. To transform SiC-NWs into amorphous NWs, the ion irradiation current is kept as low as possible; otherwise, an electron beam-induced heating phenomenon will dominate, causing the crystal structure to be maintained.[31] Similarly, welding of Ag-NWs using high-current ion beam irradiation caused ion beam-induced local heating, but the crystal structure of Ag-NWs was found stable.[32,33] In our previous work, where experiments were done on proton-irradiated ZnO-NWs and Ag-NWs under $He^+$ ions, we observed similar transformations from a crystalline to an amorphous phase.[15,18] It is envisaged that similar experiments can be performed with other materials to examine their crystalline-to-amorphous phase transformation for future applications.

## CONCLUSION

Amorphization of SiC-NWs was successfully achieved under Cu ion beam irradiation. Keeping the Cu ion irradiation energy at 10 MeV, current at 50 nA and substrate temperature (room) constant, it can be concluded that the point defects leading to the amorphization of crystalline nanowires are kinetically dependent on the irradiation doses. At a sufficiently high dose, the crystalline SiC-NWs completely transformed into the amorphous phase. It is noteworthy that, like other ion beam irradiations, a Cu ion beam at low current is very suitable for SiC-NWs amorphization. High current leads to the slicing and cutting of NWs. The amorphization mechanism of SiC-NWs can be explained using collision cascade effects.

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## CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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