

Radiation-induced defect dynamics in two-dimensional/three-dimensional systems: A dimensionality advantage preserved within patterned graphene–SiC heterostructures

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2D/3D heterostructures provide a powerful platform for uncovering how reduced dimensionality governs radiation response. While two-dimensional (2D) materials such as graphene, transition metal dichalcogenides, and MXenes have demonstrated notable structural resilience to radiation, fundamental differences in their radiation response compared with bulk three-dimensional (3D) crystals remain largely unverified. Here, we fabricate patterned *a*-Al₂O₃/graphene/H-intercalated SiC(0001) heterostructures and expose them to fast neutrons (1–2 MeV) across fluences from 1.9×10^{17} to 6.5×10^{18} n/cm². The results reveal a striking disparity: despite the 3D environment, graphene accumulates damage 69–273 times more slowly than SiC, with its resilience further enhanced at high fluences due to early saturation, soft limited to only 0.0064% at 1.3×10^{19} n/cm². This behavior is attributed to a two-channel defect accumulation model in 2D materials, involving interfacial evolution that dominates the early-stage response when the interface is chemically active, and direct lattice damage from atomic displacements, which persists but remains comparatively weak. These findings establish 2D lattices as fundamentally more radiation-resistant than 3D crystals and demonstrate that their advantages persist in realistic device architectures. Harnessing this resilience offers orders-of-magnitude improvements in structural stability for technologies operating under extreme irradiation, including fusion and fission reactors, particle accelerators, and aerospace systems.

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I. INTRODUCTION

Two-dimensional (2D) materials have emerged as a powerful platform for exploring fundamental physics. Since the discovery of graphene and the unexpected phenomena arising from its reduced dimensionality—such as massless Dirac fermions [1] and the anomalous quantum Hall effect [2]—significant progress has been made. It is now well established that systems confined to fewer than three dimensions can exhibit profoundly different physical behavior from their bulk counterparts. This realization has driven advances in several emerging fields, including spintronics [3–5], topological superconductivity [6–8], quantum computing [9–11], and strongly correlated systems [12–14], where the unique electronic and quantum

coherence properties of 2D materials and their heterostructures unlock new technological possibilities [15–17].

However, despite significant research efforts, many fundamental properties associated with reduced dimensionality remain insufficiently understood. One critical yet underexplored area is the interaction between high-energy particle radiation and low-dimensional materials. At the atomic scale, irradiation perturbs the crystal lattice, leading to the formation and accumulation of defects, which ultimately compromise long-range structural order [18–20]. In conventional three-dimensional (3D) bulk crystals, radiation-induced atomic displacements create primary defects and trigger complex collision cascades, substantially accelerating defect accumulation [21]. Even materials traditionally regarded as highly radiation-resistant [22–24], such as silicon carbide (SiC) [25–40], gallium nitride (GaN) [41–47], gallium oxide (Ga₂O₃) [48–53], and diamond [29,54–59]—all used in nuclear instrumentation and sensing applications—are susceptible to such effects.

Since the likelihood of collisions between radiation particles and lattice atoms increases with the amount of material traversed, and because collision cascades generally require a minimum material thickness to fully develop,

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a fundamental question emerges: *Does reducing a material's dimensionality fundamentally alter its response to radiation?*

Two-dimensional (2D) layered materials, such as monolayer graphene [60], hexagonal boron nitride (h-BN) [61], and MXenes [62], have attracted significant interest due to their exceptional mechanical, electronic, and thermal properties [63–66], making them promising candidates for extreme-environment applications. Beyond these intrinsic advantages, their atomically thin structure could, in principle, render them largely transparent to irradiation by reducing the probability of primary collision events and limiting the propagation of collision cascades, potentially mitigating defect accumulation and structural degradation. These properties could have profound implications for high-performance applications in aerospace [67–69], nuclear fusion [70,71], and particle accelerator technologies [72], where radiation exposure imposes stringent constraints on the operational lifetime and reliability of critical components, such as electronic systems and detectors [73–75].

However, this hypothesis remains largely unverified, with no definitive evidence demonstrating that 2D materials exhibit reduced structural degradation compared with well-established radiation-resistant materials such as SiC and GaN. Moreover, even if such a novel interaction regime between high-energy particle radiation and low-dimensional matter were feasible, a critical question arises: Would these advantages persist in practical implementations? Since 2D materials are typically integrated with 3D substrates in real-world applications [76–78], it remains unclear whether their radiation resilience would be preserved in 2D/3D systems, or whether radiation-induced damage would instead be distributed uniformly across both the 2D and 3D components.

Addressing these questions is crucial not only for assessing the potential of 2D materials in extreme radiation environments but also for advancing the fundamental understanding of high-energy particle–matter interactions and irradiation-induced damage mechanisms.

In this work, we employ a specifically designed experimental framework to investigate the role of reduced dimensionality in defect accumulation. By characterizing structural damage in 2D/3D graphene–SiC heterostructures subjected to varying fast-neutron fluences, we demonstrate that graphene layers largely retain their structural integrity. While some radiation-induced defects are observed, the relative defect concentration in graphene remains significantly lower than that in the SiC substrate. Notably, this disparity increases from a factor of 69 to 273 as the radiation fluence escalates, revealing a pronounced qualitative advantage of 2D materials over their 3D counterparts.

Crucially, we establish that irradiation-induced disorder in graphene saturates at a soft limit of just 0.0064% under a

neutron fluence of 1.3×10^{19} n/cm², representing an unprecedentedly low defect concentration and saturation threshold compared with 3D materials. These findings provide compelling evidence for the superior radiation tolerance of 2D materials and underscore their potential for next-generation radiation-resistant technologies.

II. NEUTRON–MATTER INTERACTIONS

A systematic investigation of radiation damage in 2D versus 3D materials is best conducted using 2D/3D heterostructures, as this configuration ensures identical exposure conditions—including spectral distribution, fluence, and temperature—while enabling a direct evaluation of whether the intrinsic radiation resistance of 2D materials is retained in practical, bulk-supported systems. However, this approach imposes specific requirements on the nature of the radiation itself: the experimental probe must isolate defect dynamics arising solely from material dimensionality, provide sufficient penetration depth, and ensure unbiased energy deposition throughout the heterostructure.

Neutrons, unlike charged particles such as protons, electrons, or heavy ions, interact exclusively through nuclear collisions and carry no electric charge. This allows them to penetrate deeply and uniformly into materials, including buried interfaces, without being influenced by electromagnetic fields [see Figs. 1(a) and 1(b)]. Such volumetric interaction makes neutron irradiation particularly well suited for assessing the structural resilience of both 2D and 3D components within a shared heterostructure. Moreover, the absence of Coulomb interactions minimizes extrinsic effects such as surface charging and electronic excitation, isolating the primary damage mechanism to atomic displacements initiated by primary knock-on atoms. This provides a clean and direct assessment of structural degradation across materials of differing dimensionalities. The fast-neutron energy spectrum employed in this study naturally exceeds the displacement thresholds of atoms in both graphene and SiC, ensuring effective and comparable defect generation within each material.

III. 2D/3D HETEROSTRUCTURE

Given the use of fast neutrons as the primary mechanism for inducing radiation damage, the heterostructure composition must be carefully engineered to support postirradiation characterization and to meet the stringent experimental constraints of nuclear reactor environments [79].

For these reasons, we utilize a patterned heterostructure comprising *a*-Al₂O₃/bilayer graphene/H-intercalated SiC(0001) (*a*GHSiC) (see Fig. 2 for structural and schematic representations and Appendix A 1 for fabrication details). This platform is designed to leverage the distinct physical properties of each constituent material and enables a direct comparison of the structural resilience of 2D and 3D

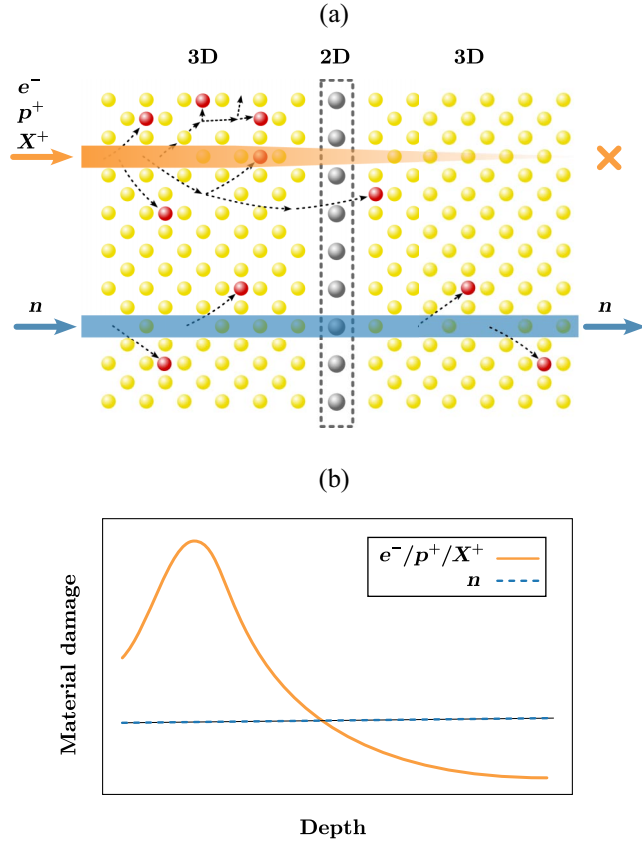


FIG. 1. (a) Schematic illustration of electron, proton, and ion (orange) versus neutron (blue) radiation propagation through a 2D/3D heterostructure, highlighting their respective interactions with the crystal lattice. (b) Simplified plot illustrating the difference in damage accumulation behavior in materials subjected to charged versus neutral particle radiation. Exact damage and penetration profiles will vary depending on fluence, energy spectrum, and material properties.

systems under neutron irradiation. Moreover, the atomic composition of the heterostructure permits a relatively short radioactive decay cooling period (see Appendix A 2), facilitating efficient postirradiation analysis.

Graphene, as a prototypical 2D material [1,2], exhibits exceptional physical properties and, owing to its reduced dimensionality, may demonstrate enhanced radiation resistance relative to its 3D counterparts. In contrast, SiC serves as an ideal benchmark material, given its high atomic displacement threshold and well-established performance in extreme radiation environments [80–82]. The amorphous alumina $a\text{-Al}_2\text{O}_3$ layer acts as an effective passivation barrier [83,84], isolating the underlying materials from ambient influences [85], pinning the double-polarization effect [86], and ensuring that observed material evolution is driven solely by neutron irradiation. However, while this passivation layer permits optical assessment of both graphene and SiC [87], it also limits direct access to interface-level characterization.

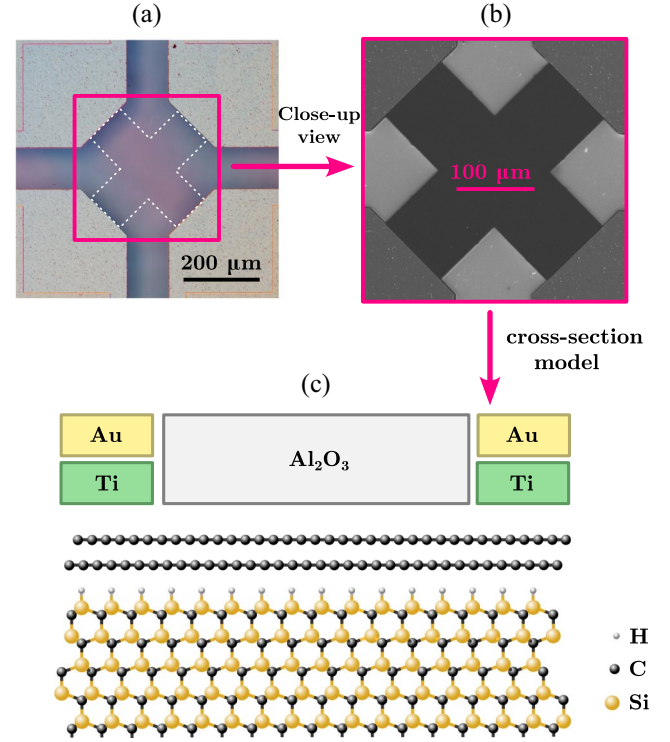


FIG. 2. Top-view images of the $a\text{GHSiC}$ heterostructure obtained using (a) Nomarski interference contrast optical microscopy and (b) scanning electron microscopy. (c) Schematic side-view illustration of the heterostructure architecture.

To address this limitation, hydrogen intercalation is introduced at the interface. This intercalation chemically decouples the graphene from the SiC substrate, while the intrinsic spontaneous polarization of SiC induces charge accumulation at the hydrogen interlayer, resulting in hole (p -type) doping of the graphene [88]. Upon hydrogen removal, this effect is reversed, leading to electron (n -type) doping [89]. This tunable doping response provides an indirect yet effective means of probing the evolution of the 2D/3D interface under neutron irradiation, even in the absence of direct physical access [71].

To ensure chemical decoupling of the upper layer from the SiC substrate, bilayer graphene is employed, with the lower layer serving as a buffer in the event of significant intercalation loss [88].

Additional details on the constituent materials and structural rationale of the heterostructure are provided in Appendix A.

IV. RADIATION-INDUCED EVOLUTION OF THE 2D/3D HETEROSTRUCTURE

To investigate the radiation-induced evolution of the 2D/3D system, we first employ high-resolution transmission electron microscopy (HR-TEM) to analyze cross-sectional images of the heterostructure interfaces before and after exposure to varying neutron fluences

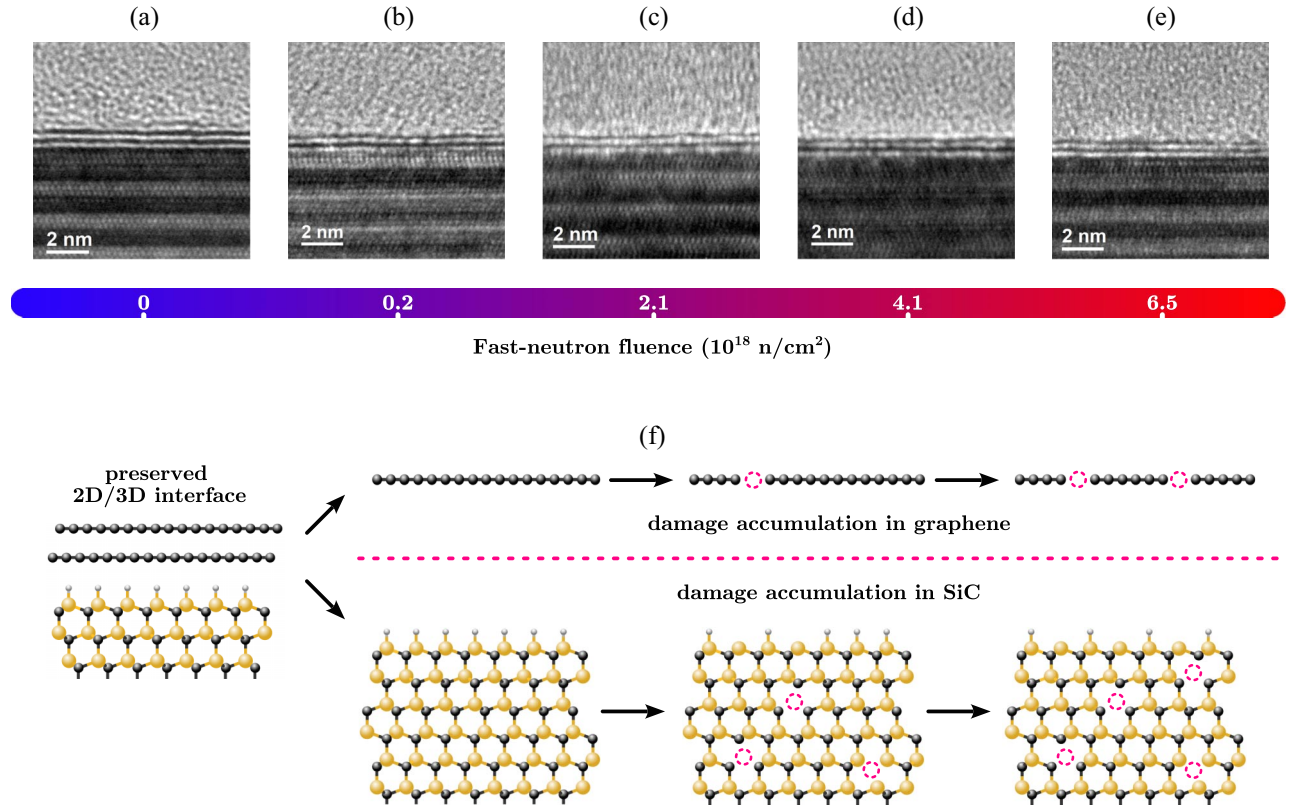


FIG. 3. (a)–(e) Representative HR-TEM cross-sectional images of the 2D/3D interface in *a*GHSiC heterostructures subjected to progressively increasing levels of neutron radiation. Each image corresponds to a distinct sample exposed to a different level of neutron irradiation. (f) Schematic illustration depicting the independent accumulation of radiation-induced damage to the crystal structures of graphene and SiC. Vacancy-type defects are used to illustrate increasing defect density for clarity.

(1.9×10^{17} to $6.5 \times 10^{18} \text{ n/cm}^2$, see Appendix B 1 for details on the irradiation campaign, and Appendix B 2 for the irradiation geometry).

Prior to irradiation [see Fig. 3(a)], HR-TEM imaging reveals amorphous Al_2O_3 in the upper portion of the image, crystalline SiC in the lower portion, and well-defined graphene layers between (dark horizontal stripes) [60]. The presence of sharp, atomically thin interfaces in the reference sample confirms the expected structural integrity of the heterostructure before irradiation. These preirradiation features serve as a benchmark for comparison with postirradiated interfaces, which, to our knowledge, represent the first HR-TEM imaging of neutron-irradiated graphene [see Figs. 3(b)–3(e)].

The results reveal that *a*- Al_2O_3 remains amorphous, SiC retains its crystalline structure, and graphene is preserved at the interface across all fluences examined. This reveals that the low-dimensional character of 2D materials can be retained at a 2D/3D interface, even after extreme neutron-radiation exposure. Furthermore, the presence of graphene at the interface indicates negligible diffusion between *a*- Al_2O_3 and SiC [90], as substantial intermixing would likely disrupt its structural integrity. This, in turn, implies that lattice damage in graphene and SiC can be treated

independently. Consequently, radiation-induced defects are expected to accumulate separately within the crystal structure of each material [see Fig. 3(f)].

However, while comparisons with the preirradiated heterostructure suggest some degree of structural damage—evidenced by slightly less defined features at the postirradiated interfaces—it should be emphasized that the presented images correspond to different cross sections of distinct samples. This limitation arises from the availability of the neutron source within the nuclear infrastructure (see the experimental setup of the fast-neutron irradiation campaign in Appendix B 1). Therefore, in this case, HR-TEM alone is insufficient for a meaningful defect analysis. Sections V and VI present Raman spectroscopy measurements targeting graphene and SiC independently, providing a more comprehensive qualitative and quantitative assessment of radiation-induced structural changes.

V. EFFECTS OF HIGH-ENERGY PARTICLE RADIATION IN 2D MATTER

The characteristic honeycomb lattice of graphene gives rise to several Raman-active vibrational modes, which can

be used not only to confirm its presence in heterostructures but also to provide statistical insight into its structural integrity and electrical properties. In graphene, the G and 2D bands are the primary Raman modes; both are intrinsically active and do not require defects for activation. The G band corresponds to the in-plane stretching vibrations of sp^2 -bonded carbon atoms, reflecting the fundamental planar structure of graphene. The 2D band arises from a second-order double-resonance process involving zone-boundary phonons and is associated with ring-breathing vibrations in the sp^2 carbon networks [91]. In contrast, the D and D' bands are defect-activated; they become Raman-active only in the presence of structural disorder. The intensities of these peaks are highly sensitive to defect concentration, making them reliable indicators of irradiation-induced or fabrication-related damage in graphene [92].

Figure 4(a) presents the Raman spectra of graphene subjected to varying levels of neutron radiation, averaged over 1681-point maps collected from $20\text{-}\mu\text{m} \times 20\text{-}\mu\text{m}$ areas to ensure statistical reliability (see Appendix B 4

for details). Upon inspection, the spectra consistently display the characteristic vibrational modes of graphene, confirming its persistent presence at the heterostructure interface across all neutron fluences. These results are consistent with the initial structural assessment based on the HR-TEM imaging.

However, the systematic evolution of the spectra with increasing neutron fluence (Φ) clearly indicates the emergence of radiation-induced structural changes. In the preirradiated reference sample, only the G and 2D bands are visible, with no measurable D or D' peaks—a spectral signature of high-quality, defect-free epitaxial graphene grown on SiC(0001). Consequently, the preirradiated graphene can be regarded as effectively pristine, and any defect-related spectral features observed after irradiation are attributed exclusively to neutron-induced damage.

Three primary spectral changes are observed upon irradiation [see Fig. 4(a)]:

- (1) The D band, an early indicator of point defects, appears at the lowest neutron fluence and increases

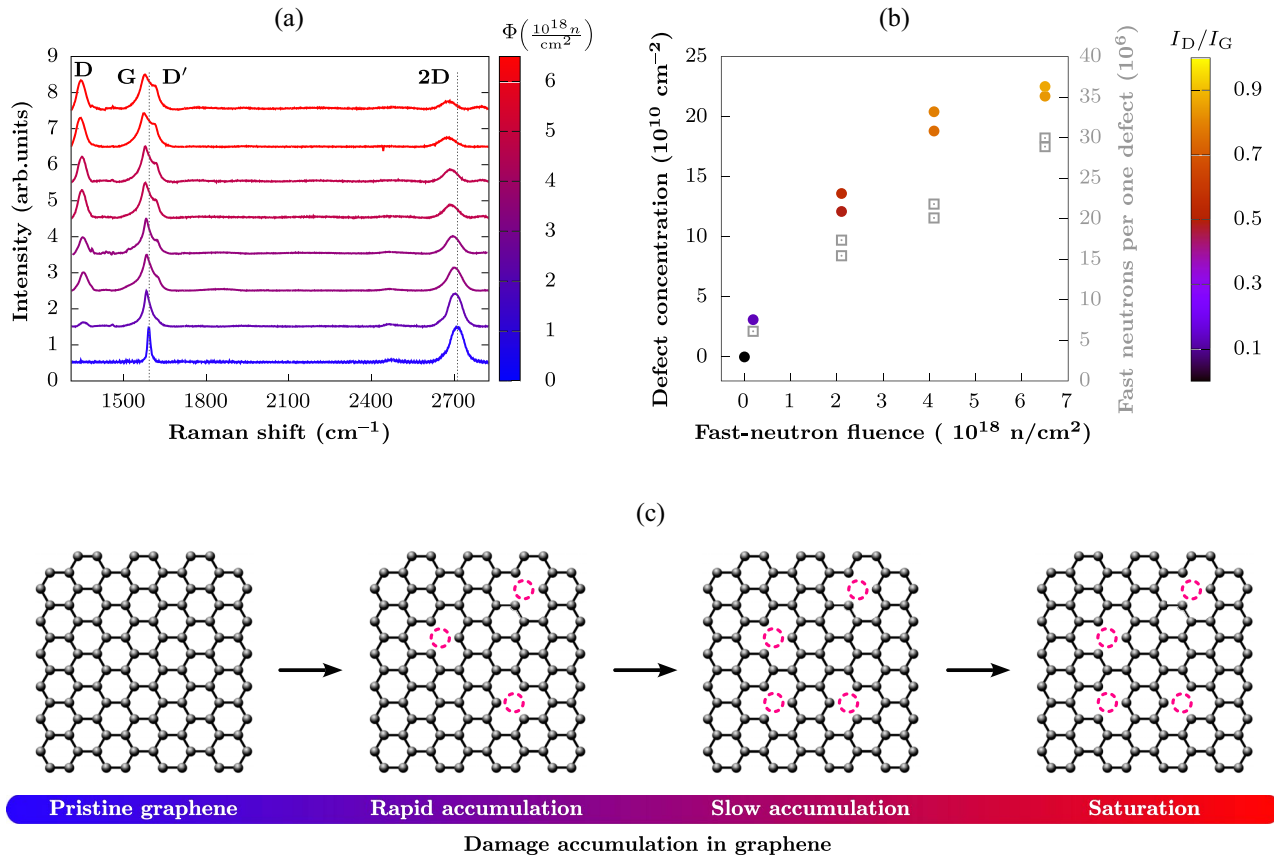


FIG. 4. (a) Averaged Raman spectra of graphene sheets in $a\text{GHSiC}$ heterostructures subjected to varying levels of neutron radiation. For details on the evolution of graphene's Raman modes, see Appendix C 1. (b) Evolution of defect concentration in graphene as a function of neutron fluence, along with the corresponding ratio of fast neutrons per generated defect (see Appendix C 3 for methodological details). (c) Schematic illustration depicting the independent accumulation of radiation-induced damage in graphene crystal structures. Vacancy-type defects are used to illustrate increasing defect density for clarity, although a range of defect types may be present.

in intensity with further exposure, reflecting the progressive accumulation of damage.

- (2) At higher fluences, the D' band becomes discernible, indicating increased defect accumulation.
- (3) The G and 2D bands exhibit reduced intensity and spectral shifts, consistent with the presence of defects and irradiation-induced strain (see Appendixes C 1 and C 2).

Notably, the changes observed in the postirradiation spectra remain confined to the evolution of intrinsic graphene modes, with no emergence of features indicative of amorphization. This suggests that graphene retains its crystalline nature even under significant neutron exposure, underscoring its exceptional resistance to amorphous phase formation.

To further contextualize the effects of neutron radiation, we quantify the defect density in graphene using the I_D/I_G ratio—an effective Raman-based approach for graphene [93,94]—see Appendix C 3.

Figure 4(b) presents the defect density as a function of the fast-neutron fluence, with the color scale representing the corresponding I_D/I_G ratio. The right Y axis shows the average number of fast neutrons required to generate a single defect, offering an alternative perspective on graphene's radiation tolerance.

The results reveal a systematic accumulation of defects, with defect densities ranging from 3×10^{10} to $2.2 \times 10^{11} \text{ cm}^{-2}$. Despite this increase, the overall damage remains minimal, corresponding to less than 0.006% of carbon atoms—irrespective of fluence. Notably, the accumulation trend exhibits clear signs of saturation, as the rate of new defect formation decreases with increasing neutron exposure. This is particularly evident in the rising average number of neutrons per defect, which increases from approximately 5×10^6 at low fluences to 3×10^7 at higher fluences. These findings demonstrate that graphene is highly transparent to neutron radiation and exhibits a low defect-generation cross section, indicating an early onset of saturation in irradiation effects.

VI. EFFECTS OF HIGH-ENERGY PARTICLE RADIATION IN 3D MATTER

The crystal structure of SiC consists of tetrahedrally coordinated silicon and carbon atoms, where each silicon atom is bonded to four carbon atoms—and vice versa—via sp^3 hybridization. This well-defined lattice gives rise to several Raman-active vibrational modes that can be used to assess the material's structural integrity, particularly under conditions that may induce disorder [95]. The stacking sequence of these tetrahedral units determines the specific SiC polytype.

Across all polytypes, the first-order Raman spectrum of SiC is primarily characterized by two fundamental zone-center optical phonon modes: the transverse optical (TO) and longitudinal optical (LO) modes [96]. The TO mode

corresponds to atomic vibrations perpendicular to the crystallographic c axis, while the LO mode corresponds to vibrations parallel to the c axis. In hexagonal polytypes such as 6H-SiC, additional complexity arises from crystal anisotropy and noncubic stacking, which result in intrinsic polarization along the c axis. This polarization activates an additional planar optical (PO) mode, which is absent in the cubic 3C-SiC polytype. The presence, evolution, and eventual degradation of these modes under irradiation provide valuable insight into changes in lattice symmetry, strain, and defect concentration.

Figure 5(a) presents the averaged Raman spectra of the SiC component within the heterostructures exposed to varying levels of neutron radiation. The spectra exhibit the persistent presence of the characteristic PO, TO, and LO phonon modes across all fluences, indicating that the SiC lattice remains predominantly crystalline. The sustained visibility of these modes suggests a high degree of structural integrity, even under substantial neutron exposure, consistent with observations from initial TEM analysis.

However, with increasing fast-neutron fluence, the Raman spectra begin to show signs of radiation-induced degradation. New Raman-active features emerge in spectral regions associated with homonuclear and heteronuclear bond vibrations, including: (i) Si—Si bonds between 180 and 550 cm^{-1} , (ii) Si—C bonds between 580 and 950 cm^{-1} , and (iii) C—C bonds between 1080 and 1200 cm^{-1} [97–101].

Additionally, a distinct spectral shoulder appears between the TO and LO modes, indicative of local disorder within the SiC lattice. These effects are attributed to the formation of antisite defects, interstitial clusters, and other displacement-induced defects, which disrupt long-range order and facilitate the formation of homonuclear bonding networks [99,102].

A progressive increase in the integrated area of the Raman spectra with increasing fluence further suggests the gradual emergence of an amorphous phase within the SiC matrix.

To quantify the magnitude of this structural degradation, we analyze the fluence-dependent shift in the LO phonon mode, which serves as a robust proxy for estimating the defect concentration in SiC (see Appendix C 4 for methodological details). The extracted defect densities are shown in Fig. 5(b).

The results indicate a systematic accumulation of defects in SiC, with concentrations ranging from 5.3×10^{19} to $1.6 \times 10^{21} \text{ cm}^{-3}$. Even at the highest fluence studied, this corresponds to less than 2% lattice damage, indicating partial preservation of crystallinity. The accumulation trend appears approximately linear across the measured range, with no evidence of saturation—consistent with the expectation that saturation effects emerge only at substantially higher fluences [103].

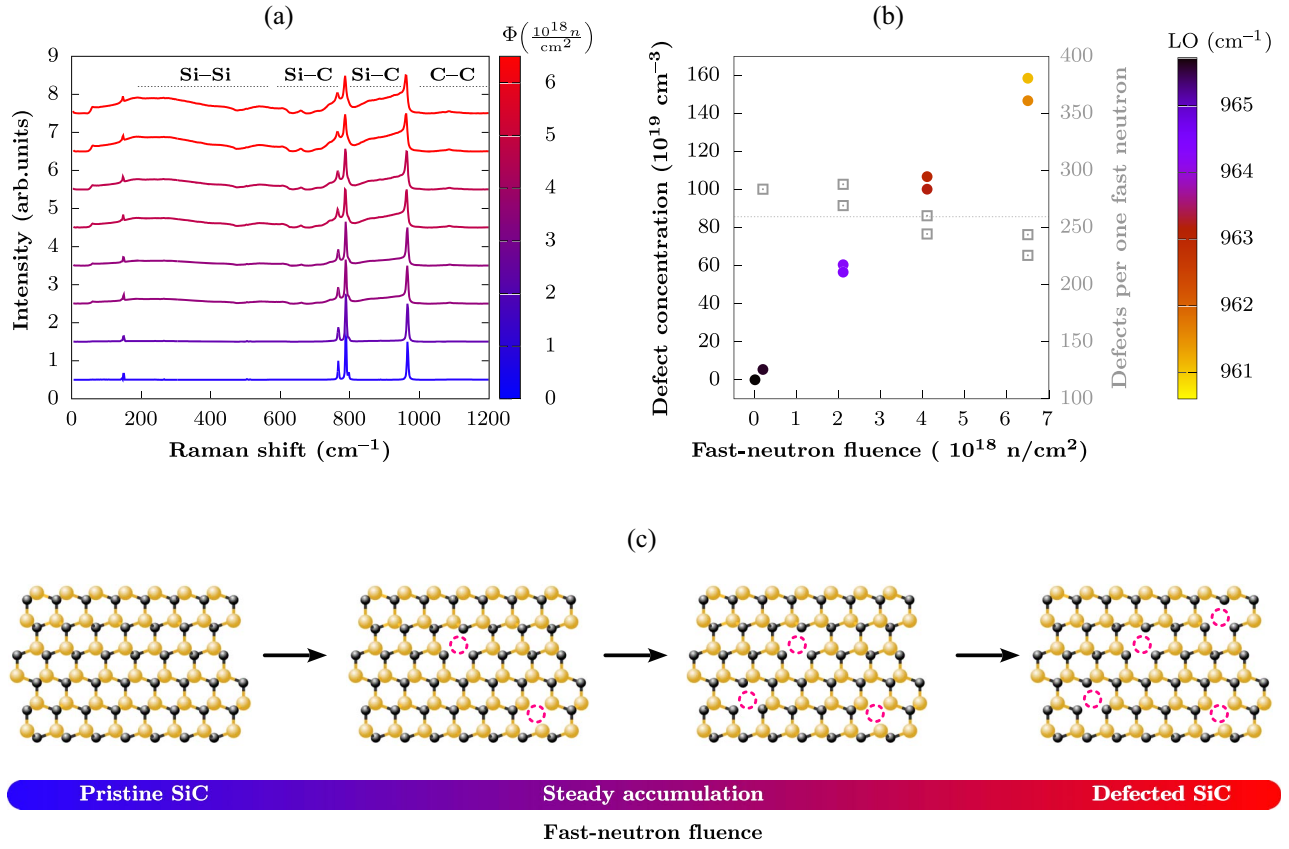


FIG. 5. (a) Averaged Raman spectra of SiC substrates in *a*GHSiC heterostructures subjected to varying levels of neutron radiation. (b) Evolution of defect concentration in SiC as a function of neutron fluence, along with the corresponding number of defects generated per fast neutron (see Appendix C 4 for methodological details). (c) Schematic illustration depicting the independent accumulation of radiation-induced damage in SiC crystal structures. Vacancy-type defects are used to illustrate increasing defect density for clarity, although a range of defect types may be present.

Thus, within the investigated regime, defect density scales proportionally with neutron fluence, maintaining a relatively near-constant fast-neutron-to-defect conversion ratio. Specifically, each fast neutron statistically generates approximately 259 defects in SiC, underscoring its high susceptibility to displacement cascades.

VII. 2D VERSUS 3D

The contrast in radiation response between graphene and SiC is striking. In graphene, it takes millions to tens of millions of fast neutrons to produce a single lattice defect, whereas in bulk SiC, a single fast neutron statistically generates approximately 259 defects. At first glance, this disparity appears to indicate that 2D materials accumulate radiation-induced defects at a much slower rate. While this conclusion is supported by the absolute defect concentrations shown in Figs. 4(b) and 5(b), it can be misleading. The reported values are normalized per unit surface area for graphene and per unit volume for SiC, reflecting their respective dimensionalities. Consequently, the total number of atoms differs substantially between the systems: bulk SiC contains

approximately 4.8×10^{22} atoms/ cm^3 [104], whereas graphene contains about 3.8×10^{15} atoms/ cm^2 [105]. Thus, an equivalent number of defects corresponds to a much smaller atomic fraction in a 3D lattice than in a 2D one.

Consequently, to meaningfully compare defect accumulation in 2D and 3D materials, it is essential to consider relative defect concentrations—defined as the ratio of generated defects to the total number of atomic sites. As shown in Fig. 6(a), the results confirm that graphene exhibits superior structural resilience under neutron irradiation. Specifically, the relative structural damage in graphene ranges from approximately 0.0008% to 0.0057% across the studied fluence range, while in bulk SiC the corresponding damage spans from 0.05% to nearly 2%. These findings indicate that graphene not only accumulates fewer absolute defects but also preserves a significantly higher degree of structural integrity under equivalent irradiation conditions.

Moreover, the disparity in radiation response between graphene and SiC is fluence-dependent, with the divergence becoming increasingly pronounced at higher fluences [see Fig. 6(b)]. Specifically, graphene accumulates structural damage approximately 69 to 273 times more

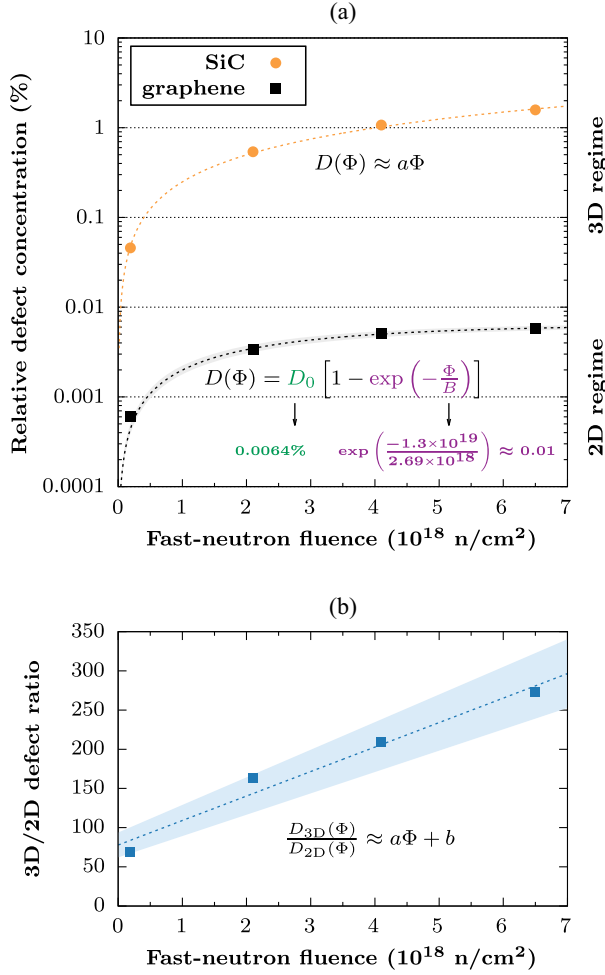


FIG. 6. (a) Relative defect concentrations in graphene sheets and SiC substrates subjected to varying levels of neutron irradiation. Right-hand side labels indicate the distinct differences in damage accumulation between the 2D and 3D materials. (b) Ratio of 3D to 2D defects (SiC to graphene) as a function of neutron fluence, highlighting the contrast in radiation response between the two material systems.

slowly than SiC. This pronounced contrast not only highlights a fundamental qualitative difference in the radiation tolerance of 2D versus 3D materials, but also suggests the presence of interfacial mechanisms within the 2D/3D heterostructure that may further modulate defect formation and evolution.

To contextualize the radiation response of graphene, the experimental data are fitted using a saturation model that captures the canonical evolution of radiation-induced structural damage—characterized by an initial rapid accumulation followed by saturation at higher fluences [20]. The relationship between structural damage (D) and neutron fluence is described by

$$D(\Phi) = D_0 \left[1 - \exp\left(-\frac{\Phi}{B}\right) \right], \quad (1)$$

where D_0 denotes the saturation damage level, and B is a scaling factor for the saturation fluence threshold.

When applied to the experimental data, the saturation model yields an excellent fit, with an R^2 value of 0.9994, providing a clear description of the saturation behavior of graphene within the heterostructure. The model predicts that the observed damage mode in graphene reaches a saturation level of approximately 0.0064%, with over 99% of this level attained at a neutron fluence of approximately 1.3×10^{19} n/cm². This reflects both a relatively low saturation threshold and a modest damage ceiling, further reinforcing the exceptional radiation resilience of graphene. However, it also reveals a notably low absolute damage threshold compared with typical 3D materials.

By contrast, under the same irradiation conditions, SiC exhibits damage exceeding 2% and shows no signs of saturation—projected to occur only at fluences approaching 10^{21} n/cm² [103]. This stark disparity underscores a fundamental difference in both the accumulation dynamics and the saturation behavior of radiation damage in 2D versus 3D systems.

VIII. INTERFACIAL EVOLUTION IN 2D/3D HETEROSTRUCTURES

The results presented thus far confirm the structural integrity of the heterostructure and its individual 2D and 3D components under neutron irradiation, while clearly revealing the markedly greater radiation resilience of graphene compared with SiC. This observation not only underscores the fundamental advantage of 2D materials over their 3D counterparts in neutron-rich environments but also demonstrates that this resilience can be preserved within practical 2D/3D architectures. Nevertheless, a critical question remains: *Is this dimensional advantage preserved only against particle showers originating from adjacent bulk materials, or do additional interaction pathways exist through which substrate damage can influence the 2D materials?*

Although displacement cascades are expected to occur universally across all heterostructure systems—and, as demonstrated by our findings, are insufficient to suppress the enhanced resilience of 2D materials—the presence of a 2D/3D interface introduces additional complexity. Unlike bulk regions, the interface may undergo distinct structural or electronic evolution under irradiation, potentially leading to localized modifications that do not reflect the behavior of the individual constituent materials. This raises the possibility that interface-specific mechanisms may play a pivotal role in shaping the overall radiation response of 2D materials within heterostructures.

Accordingly, it becomes essential to determine whether the radiation resilience of graphene is preserved even as the substrate surface evolves, or whether it relies on the integrity of an undisturbed 2D/3D interface. To address this, the *a*GHSiC heterostructure system has been

deliberately designed and engineered to enable targeted probing of interface-specific radiation effects, while ensuring that graphene remains chemically decoupled and isolated from external environmental influences.

A key element of the heterostructure design is the hydrogen intercalation layer, which is commonly employed to chemically decouple epitaxial graphene from the SiC(0001) substrate [88]. This intercalation process induces *p*-type doping in the graphene layer, driven by the spontaneous polarization inherent to the specific SiC polytype used [106]. Spontaneous polarization arises in dielectric crystals where aligned dipoles are oriented along the surface normal. While periodic boundary conditions in the bulk enforce a vanishing net electric field across the unit cell, this constraint breaks down at surfaces, where the lack of translational symmetry permits dipole accumulation and the emergence of a nonzero, uncompensated polarization field. This surface polarization drives charge accumulation, with the intercalated hydrogen layer acting as an effective acceptor [see Fig. 7(a)]. As a result, electron transfer from the graphene is promoted, leading to

consistent, polytype-dependent *p*-type doping on the order of 10^{12} e/cm² [106].

Consequently, the carrier concentration in graphene is highly sensitive to the degree of hydrogen intercalation, which represents the most vulnerable and radiation-sensitive component of the system. This susceptibility arises from the relatively weak single H—Si bonds in the intercalation layer, which have a dissociation energy of 3.2 eV (as determined by density functional theory, DFT), in contrast to the stronger *sp*²-bonded carbon atoms in graphene (20.6 eV) and the *sp*³-bonded Si and C atoms in the SiC substrate (19.3 eV). As a result, hydrogen atoms are the most easily displaced in radiation-induced collisions and require the least local heating to spontaneously desorb [88].

The loss of intercalated hydrogen reduces the charge accumulation capacity of the acceptor layer, thereby diminishing *p*-type doping in the graphene [see Fig. 7(b)]. In regions where hydrogen is sufficiently depleted, the chemical decoupling between graphene and the SiC substrate is lifted [see Fig. 7(c)], re-enabling local

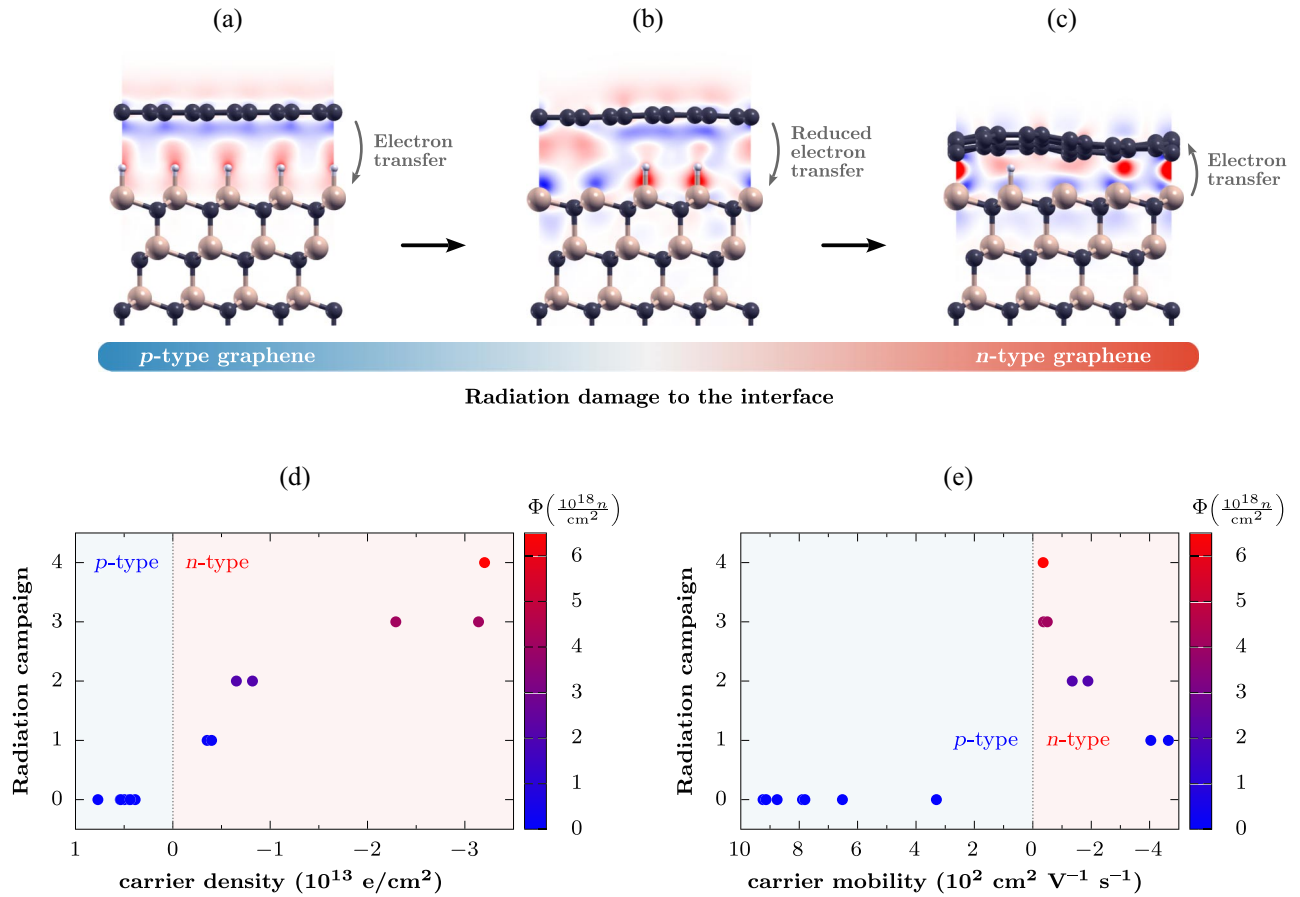


FIG. 7. (a)–(c) Schematic illustration depicting the evolution of charge transfer at the graphene–SiC interface relative to the level of hydrogen intercalation obtained at DFT level of theory. The blue and red regions represent electronic density depletion and accumulation, respectively, arising from the graphene–substrate interaction. (d) Carrier density and (e) mobility of graphene sheets in *a*GHSiC heterostructures as a function of neutron fluence.

electronic coupling between the two materials. This renewed interaction perturbs the graphene lattice and facilitates electron transfer from the 6H-SiC substrate into the graphene layer, resulting in a reversion to *n*-type doping, typically on the order of $10^{13} - e/\text{cm}^2$ [107].

These doping levels exceed the irradiation-induced defect densities in graphene by at least 2 orders of magnitude, establishing sheet–substrate interactions as the dominant factor governing the overall carrier concentration. Accordingly, changes in the electrical properties of graphene serve as a direct and sensitive probe of the 2D/3D interface evolution under neutron irradiation [71].

Figures 7(d) and 7(e) show the charge carrier concentration and mobility, respectively, as determined from Hall-effect measurements. To emphasize the effects of irradiation on the electrical properties, both panels are divided into two regions corresponding to conductivity type, as identified through Hall analysis under single-carrier transport conditions. Notably, the electrical characteristics of the preirradiated samples consistently fall within the *p*-type region, in agreement with the dual-polarization model associated with the SiC substrate and the *a*-Al₂O₃ passivation layer [86,106]. The measured hole concentrations range from $+4$ to $+8 \times 10^{12} \text{ cm}^{-2}$, with mobilities on the order of $10^2 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$. These values confirm a high hydrogen content at the 2D/3D interface in the *a*GHSiC heterostructures prior to irradiation [88,106,107], consistent with the intended fabrication design.

Upon exposure to fast neutrons, however, this doping behavior changes significantly. Although electrical parameters evolve gradually with increasing neutron fluence, a key observation is that all samples transition to *n*-type conductivity regardless of total exposure, as shown in Fig. 7(d). This indicates that even low levels of neutron exposure affect the hydrogen-terminated SiC surface. The progressively stronger *n*-type doping at higher fluences suggests continued depletion of interfacial hydrogen [89,107]. Interestingly, the data also reveal a diminishing radiation effect at higher fluences (see Appendix C5 for a detailed summary).

This saturation trend is mirrored in the carrier mobility evolution [Fig. 7(e)], where mobility initially decreases sharply with increasing fluence but levels off near $4 \times 10^{18} \text{ n/cm}^2$. The overall mobility loss is consistent with the depletion of hydrogen intercalation, as the resulting exposure of the SiC surface enables local graphene–substrate coupling [see Fig. 7(c)]. This coupling introduces interfacial strain in addition to primary dislocation damage within the graphene lattice. These findings are further supported by Raman spectral analysis, which reveals radiation-induced increases in tensile strain within the graphene sheets (Appendix C1 and C2).

Collectively, these insights into the 2D/3D interface indicate that the dimensional advantage in radiation

resilience of 2D materials is retained in heterostructures, despite particle showers from adjacent bulk materials and interfacial evolution leading to chemical coupling with the substrate.

IX. ORIGIN OF DIVERGENT DEFECT DYNAMICS IN 2D/3D HETEROSTRUCTURES

In this investigation, we explored whether the potential benefits to the structural resilience of freestanding 2D materials, arising from their reduced dimensionality, can be preserved in practical heterostructure environments. Specifically, we examined whether the absence of collision cascades within graphene is effectively offset by cascades originating in adjacent 3D materials when the 2D layer is encapsulated, given the relatively small number of atoms in a 2D sheet compared with bulk materials. Our findings demonstrate that this is not the case—graphene retains a markedly lower damage accumulation rate even under these conditions.

However, to what extent might this observation be influenced by factors not intrinsic to the dimensionality of graphene itself?

A. Assessment of extrinsic factors

One potential concern is the irradiation geometry. In beamlike irradiation environments, directional effects may bias damage accumulation, particularly in asymmetric heterostructures, as collision cascades preferentially propagate along the incident direction [108–110]. Under such conditions, the relative orientation of the layers could influence the extent of damage transferred across the structure. However, the irradiation campaigns in this study were conducted in a reactor-based environment characterized by a broad angular neutron distribution (see Appendix B2 for details on the radiation environment). Consequently, collision cascades are initiated throughout the volume without a well-defined preferential direction, and the observed differences cannot be attributed to irradiation geometry.

Another possible mechanism is *in situ* defect annealing. Graphene has been reported to exhibit effective self-healing of radiation-induced defects at elevated temperatures (typically above approximately 600°C) [111]. However, the present experiments were conducted at significantly lower temperatures (approximately 130°C during irradiation and lower during cooldown), and no postirradiation annealing was performed. Therefore, thermally activated healing processes cannot account for the reduced defect accumulation observed in graphene.

Finally, we consider whether defect morphology could bias the analysis. In particular, one might hypothesize that irradiation induces large-scale perforation (nanopore formation), which could be partially undetected in Raman-based measurements. While controlled nanopore

formation in graphene is achievable, it requires carefully engineered conditions, including low-energy ion irradiation to generate initial vacancies, followed by subthreshold electron-beam-induced removal of atoms selectively at defect edges [112]. In contrast, fast neutrons transfer hundreds of keV to primary knock-on atoms, producing stochastic and spatially distributed damage throughout the heterostructure. Under such conditions, dislocation damage in graphene is expected to be spatially homogeneous rather than localized at preexisting defect sites. Moreover, large-scale structural discontinuities would result in reduced Raman signal intensity and instability in electrical measurements, neither of which is observed. We therefore exclude nanopore formation as a viable explanation.

B. Dimensionality-driven defect dynamics

Having excluded extrinsic factors, the divergent defect dynamics must originate from intrinsic material properties, most notably dimensionality.

In bulk materials, defect accumulation proceeds through cascade overlap and defect recombination, leading to gradual saturation. In graphene, however, the experimentally observed saturation occurs at an exceptionally low defect density (approximately 0.0064%), which cannot be explained by conventional bulklike accumulation kinetics. We attribute this behavior to the dominant role of interfacial processes in 2D/3D heterostructures. Unlike bulk systems, where surfaces constitute a negligible fraction of the material, a 2D layer is entirely interfacial. Consequently, irradiation-induced modifications at the interface directly affect the entire material.

In the graphene/SiC system, neutron irradiation leads to the progressive depletion of hydrogen intercalation at the interface. Since hydrogen intercalation governs both the electronic decoupling of graphene from the substrate and its doping state, its removal promotes rehybridization and partial chemical coupling between graphene and SiC. This process alters the electronic properties (e.g., transition from *p*-type to *n*-type behavior) and introduces disorder within the graphene sheet without direct atomic displacements arising from collision events.

Notably, this interfacial modification is inherently self-limiting: once the available intercalated hydrogen is depleted, further evolution saturates. This provides a natural explanation for the apparent early saturation of defect density, despite the relatively low level of structural disorder in the graphene lattice, and frames this behavior as a soft limit. Furthermore, this interpretation indicates that, even under significant neutron irradiation, direct displacement damage in graphene remains minimal.

C. Two-channel damage model

The results therefore indicate that defect accumulation in a 2D material within a 2D/3D heterostructure proceeds via two distinct channels:

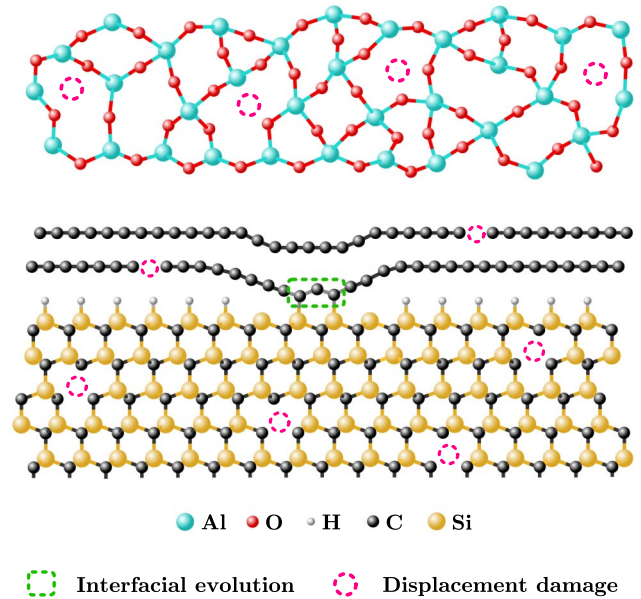


FIG. 8. Schematic illustration depicting two-channel model of damage accumulation in graphene within the α GHSiC heterostructure. Vacancy-type defects are used to indicate displacement damage, although a range of defect types may be present.

- (1) Interfacial evolution (highlighted by the dashed rectangle in Fig. 8), which dominates when the interface is chemically active and governs the apparent defect dynamics of the 2D layer during the initial stages of irradiation (negligible in 3D crystals);
- (2) Direct lattice damage (highlighted by the dashed circles in Fig. 8), arising from atomic displacements within the 2D material itself, which persists throughout irradiation but remains comparatively weak (dominant in 3D crystals).

The first mechanism is negligible in bulk materials but becomes dominant in 2D systems due to their inherently high surface-to-volume ratio. Once interfacial processes saturate, further defect accumulation is governed by the intrinsically slower direct damage channel.

D. Implications for radiation tolerance engineering

These findings demonstrate that graphene retains substantially slower defect accumulation than its 3D counterpart even in a realistic heterostructure environment, where cascade injection from the interfacing materials is possible. Moreover, the present system represents a conservative case, as the interface was intentionally susceptible to radiation-induced modification.

This suggests a clear pathway for further enhancing radiation tolerance: engineering chemically inert and structurally stable interfaces. Optimization of substrate composition, surface termination, and interfacial chemistry can suppress the dominant interfacial damage channel,

thereby more effectively preserving the intrinsic radiation resilience of the 2D material.

X. CONCLUSION

We have presented unequivocal evidence that not only can 2D materials persist at the interface of 2D/3D heterostructures under extreme fast-neutron fluence, but, notably, they exhibit fundamentally superior radiation resilience—an advantage retained even when integrated within bulk-supported frameworks.

Using HR-TEM imaging, we consistently observed the presence of 2D structures at the interface of α -Al₂O₃/bilayer graphene/H-intercalated SiC(0001) across multiple heterostructures subjected to neutron fluences ranging from 1.9×10^{17} to 6.5×10^{18} n/cm².

Employing micro-Raman spectroscopy, these 2D structures were unambiguously identified as graphene. Notably, even under intense radiation, damage to the graphene remained minimal, with defect densities ranging from 3×10^{10} to 2.2×10^{11} cm⁻², corresponding to less than 0.006% of carbon atoms at the upper damage bound. These findings highlight the exceptional transparency of graphene to neutrons, with, on average, only a single lattice defect formed per millions to tens of millions of fast neutrons due to the rapid saturation of radiation-induced damage.

Critically, our model predicts a soft saturation limit for structural disorder in graphene of only 0.0064% at a neutron fluence of 1.3×10^{19} n/cm², highlighting an exceptionally low defect concentration and saturation threshold relative to conventional 3D materials. Under the same irradiation conditions, the SiC substrate accumulates damage in a near-linear fashion, reaching approximately 1.6% relative damage at 6.5×10^{18} n/cm². As a result, graphene is revealed to be 69–273 times more resilient than SiC under equivalent fluences, with the disparity becoming increasingly pronounced at higher fluences.

To investigate the evolution of the 2D/3D interface under irradiation, we combined Hall-effect measurements with DFT-based insights. Leveraging the spontaneous polarization mechanism responsible for graphene doping on SiC substrates, the observed transition from *p*-type to *n*-type conductivity in graphene directly reflects a progressive depletion of intercalated hydrogen at the interface. These results demonstrate that the exceptional radiation resilience of 2D materials reported here can be sustained within 2D/3D heterostructures—even when the supporting substrate is significantly less resilient than the 2D layer itself.

The results support a two-channel model of defect accumulation in a 2D material within a 2D/3D heterostructure, comprising (i) interfacial evolution, which dominates when the interface is chemically active and governs the early-stage response of the 2D layer, and (ii) direct lattice damage from atomic displacements within the 2D material, which persists but remains

comparatively weak. While the former is negligible in bulk systems, it becomes dominant in 2D materials due to their inherently high surface-to-volume ratio. Once interfacial processes saturate, defect accumulation proceeds via the intrinsically slower direct damage channel.

Taken together, our findings establish a qualitative and quantitative advantage of 2D materials over their 3D counterparts in radiation resilience that goes beyond theoretical predictions or best-case scenarios. Rather, we reveal orders-of-magnitude improvements realized in practical setups, with further potential to harness the intrinsic resilience of 2D materials through heterostructure designs incorporating more radiation-tolerant interfaces or optimized decoupling strategies.

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DATA AVAILABILITY

The data supporting the findings of this study are openly available in Ref. [113].

APPENDIX A: MATERIALS

1. 2D/3D heterostructure fabrication

The aGHSiC is a four-terminal, equal-arm *van der Pauw* element comprising *in situ* hydrogenated, semi-insulating, vanadium-compensated, nominally on-axis 6H-SiC(0001), quasi-free-standing epitaxial chemical vapor deposition graphene [60,114], and a 100-nm-thick, atomic-layer-deposited, amorphous Al₂O₃ passivation [86,87,115] synthesized from trimethylaluminum and deionized water at 770 K. The double-cladded structure is part of the GET[®] brand material and device portfolio [116].

TABLE I. Summary of key radionuclides produced in the α GHSiC heterostructure under fast-neutron irradiation, along with their corresponding half-lives and commentary on their role in the radioactive decay cooling process of the system.

Radionuclide	Half-life	Notes
^{28}Al	2.2 min ^a	Short-lived β and γ emitter; the primary radionuclide generated in Al_2O_3 under a fast-neutronspectrum. It is formed via the $^{27}\text{Al}(n, \gamma)^{28}\text{Al}$ reaction, involving the most abundant aluminumisotope (approximately equal to 100%) [117].
^{27}Mg	9.5 min ^b	Short-lived β and γ emitter; a fast-neutron-induced radionuclide in Al_2O_3 with an energythreshold of approximately 4 MeV. It is formed via the $^{27}\text{Al}(n, p)^{27}\text{Mg}$ reaction, involving the most abundantaluminum isotope (approximately equal to 100%) [117].
^{31}Si	2.6 h ^c	Short-lived β and γ emitter; the main radionuclide produced in SiC under a fast-neutron spectrum.It is formed via the $^{30}\text{Si}(n, \gamma)^{31}\text{Si}$ reaction, involving a stable silicon isotope (3.1%) [118].
^{48}Sc	43.7 h ^d	Short-lived β and γ emitter; the main radionuclide produced in Ti contacts under a fast-neutronspectrum. It is formed via the $^{48}\text{Ti}(n, p)^{48}\text{Sc}$ reaction, involving the predominant stable titaniumisotope (74% abundance) [119].
^{198}Au	2.7 d ^e	Short-lived β and γ emitter; the primary radionuclide produced in Au contacts under a fast-neutron spectrum. It is formed via the $^{197}\text{Au}(n, \gamma)^{198}\text{Au}$ reaction, involving the most abundantgold isotope (approximately equal to 100%) [120].
^{32}P	14.3 d ^f	Short-lived β emitter; a minority radionuclide produced in SiC under a fast neutron spectrum. Itis formed via the $^{30}\text{Si}(n, \gamma)^{31}\text{Si}(\beta)^{31}\text{P}(n, \gamma)^{32}\text{P}$ reactions [118].
^{46}Sc	83.8 d ^g	Short-lived β and γ emitter; a minority radionuclide produced in Ti contacts under a fast-neutronspectrum. It is formed via the $^{46}\text{Ti}(n, p)^{46}\text{Sc}$ reaction, involving a stable titanium isotope (8.25%abundance) [119].
^{14}C	5.7 ka ^h	Long-lived β emitter; however, as it is produced via neutron capture by ^{13}C —which constitutesonly approximately 1% of natural carbon—it remains a minor radionuclide in graphene + SiC under fast-neutronirradiation [118].
^{26}Al	718 ka ^h	Long-lived β and γ emitter; however, its production requires high-energy neutrons (>14 MeV) via $^{27}\text{Al}(n, 2n)^{26}\text{Al}$ reaction, and is therefore not efficiently generated in Al_2O_3 and neutron SiC under a typical fast- spectrum [118,121].

^aRef. [122]

^bRef. [123]

^cRef. [124]

^dRef. [125]

^eRef. [126]

^fRef. [127]

^gRef. [128]

^hRef. [129]

2. Radioactive decay cooling period

The primary radionuclides produced in α GHSiC under fast-neutron irradiation are summarized in Table I.

The half-lives and elemental composition of the radionuclides produced in α GHSiC under fast-neutron irradiation have resulted in a radioactive decay cooling period ranging from several weeks to a few months, depending on the total neutron fluence accumulated by the samples. However, it should be emphasized that this cooling period could have been significantly extended in the presence of initial isotopic contamination [118].

3. Heterostructure composition rationale

a. α - Al_2O_3 —a resilient nanoceramic passivator decoupling from the environment

α - Al_2O_3 serves as an effective passivation layer [87,115], pinning the double-polarization effect [86], and isolating the underlying materials from environmental influences while offering exceptional radiation resistance. It has been extensively studied in nuclear applications and

is widely recognized as a structurally robust material capable of withstanding extreme radiation exposure. Early studies confirmed that irradiation damage does not impose significant limitations on the use of aluminum oxide in reactor environments [130,131]. More recent investigations on Al_2O_3 nanoceramics have demonstrated remarkable radiation endurance [132], further supporting the applicability of our 100-nm-thick layer in nuclear environments. Beyond its intrinsic radiation stability, α - Al_2O_3 provides excellent HR-TEM contrast against SiC [60], enabling effective structural characterization at the heterostructure interface.

b. Graphene—an exemplary 2D material with intrinsic radiation resistance

Graphene, the prototypical two-dimensional material, offers a promising pathway for mitigating radiation-induced damage due to its unique atomic structure. As a monolayer of graphite—a material already known for its radiation resistance and exceptional stability under neutron

irradiation [133]—graphene inherits key structural characteristics while benefiting from reduced dimensionality, which may further enhance its resilience to neutron exposure. Indeed, several studies have reported that graphene sheets retain their structural integrity under harsh neutron irradiation conditions [71,134–136]. Beyond its promising radiation stability, graphene benefits from mature fabrication technologies, making it the most technologically advanced two-dimensional material currently available [66]. Furthermore, its exceptional electronic properties and thermal stability broaden its applicability in radiation-hardened electronic systems [137].

c. Hydrogen—2D/3D interface probing

Hydrogen termination of Si-faced SiC(0001) is employed to enable effective probing of 2D/3D interface evolution while remaining compatible with the 100-nm-thick passivation layer required to isolate the interface from environmental influences. This intercalation functions as an effective acceptor layer, driven by the spontaneous polarization of the SiC substrate [106]. The resulting polarization promotes electron transfer from the graphene layer, thereby inducing *p*-type doping, with the doping level dependent on the specific hexagonal SiC polytype [138].

Consequently, the carrier concentration in graphene is highly sensitive to the degree of hydrogen intercalation, which represents the most vulnerable and radiation-sensitive component of the system [136]. This vulnerability arises from hydrogen's high neutron scattering cross section and its strong effectiveness in neutron thermalization; neutron scattering studies confirm that hydrogen enhances sensitivity to radiation-induced changes [139]. Moreover, hydrogen atoms are the most weakly bonded species in the heterostructure, forming single H—Si bonds in contrast to the more robust *sp*²-bonded carbon atoms in graphene and *sp*³-bonded Si and C atoms in the SiC substrate. As a result, hydrogen atoms are the most readily displaced during radiation-induced collisions and require the least local heating to spontaneously desorb [88].

Thus, the incorporation of hydrogen intercalation ensures a radiation-sensitive 2D/3D interface, enabling the investigation of how robust the enhanced radiation resilience of 2D materials remains within heterostructures—and whether it may be compromised, or even lost, under specific conditions. This design also allows interface evolution to be probed directly through electrical characterization.

d. SiC—benchmarking against the best radiation-resistant 3D materials

SiC is among the most radiation-hardened materials used in extreme environments [140]. As a wide-bandgap semiconductor with high atomic displacement energy, it is the material of choice for nuclear sensing [30], avionics [141], and space applications [142]. Its intrinsic radiation tolerance significantly surpasses that of conventional silicon-based

electronics [143], positioning it as a leading candidate for next-generation radiation-hardened devices [144–146]. Accordingly, SiC serves as a benchmark for assessing radiation-induced damage in graphene, providing a stringent and well-established standard for comparison.

APPENDIX B: EXPERIMENTAL SETUP

1. Fast-neutron irradiation campaign

Neutron irradiation was conducted at the MARIA research nuclear reactor, operated by the National Centre for Nuclear Research in Otwock, Poland [147]. As in typical light-water reactors, the neutron energy spectrum exhibits a continuous distribution: at lower energies (meV), it follows a Maxwellian distribution resulting from thermal equilibrium with the moderator, while at higher energies (MeV), it reflects a fission-derived spectrum governed by the kinetics of the fission process.

For this study, irradiation was performed in an out-of-core facility specifically designed to suppress low-energy (thermal) neutrons, reducing their fluence rate by more than 2 orders of magnitude. As a result, the ratio of fast to thermal neutron fluence rates in this setup is significantly elevated compared with conventional reactor conditions [148]. The resulting spectrum is dominated by neutrons in the energy range of 0.5–3.0 MeV, with a peak between 1 and 2 MeV. This configuration provides an effective approximation of neutron environments generated by deuterium-deuterium fusion reactions [149].

The irradiation campaign comprised four individual irradiation runs, each lasting between 166 and 997 hours and conducted at different irradiation positions to accumulate a range of fast-neutron fluences. Each run included two *a*GHSiC samples, and a detailed summary of the irradiation conditions is provided in Table II.

2. MARIA research nuclear reactor and the irradiation process

The MARIA reactor is a channels-in-pool type research reactor featuring a compact core composed of pressurized fuel channels surrounded by beryllium moderator blocks and a graphite reflector, all immersed in a light-water pool that serves both as coolant and shielding [147]. The fuel channels and moderating structures are arranged in a

TABLE II. Summary of irradiation processes conducted as part of the radiation campaign.

Campaign	Fluence (10^{17} n/cm ²)	Samples
Φ_1	$1.9 \pm 6\%$	$1_A, 1_B^a$
Φ_2	$20.9 \pm 10\%$	$2_A, 2_B$
Φ_3	$41.3 \pm 10\%$	$3_A, 3_B$
Φ_4	$64.9 \pm 10\%$	$4_A, 4_B^b$

^aDamaged, not used in Raman analysis.

^bDamaged contact, not used in Hall effect analysis.

concentric, quasiconical geometry, resulting in a spatially distributed neutron source rather than a collimated emitter.

While MARIA is equipped with horizontal beam channels that provide well-defined, quasiparallel neutron beams for scattering and imaging applications, these facilities deliver neutron fluxes several orders of magnitude lower than those required for materials irradiation studies. Consequently, such beamlines were not suitable for the present work, where high fast-neutron fluence and the lack of directional bias were the primary requirements.

Instead, irradiation was conducted in dedicated out-of-core experimental facilities located within the reactor pool, in proximity to the core and reflector regions. In these positions, the neutron field arises from the superposition of fission neutrons emitted from multiple fuel elements, further modified by scattering, moderation, and reflection in water, beryllium, and graphite. As a result, the neutron flux exhibits a *cosinelike* spatial distribution and is inherently multidirectional rather than beamlike.

This configuration results in an irradiation environment that, at the scale of the samples, can be approximated as a *neutron gas*, with neutrons incident over a wide angular distribution. Such conditions are characteristic of reactor-based irradiation facilities and fundamentally differ from the unidirectional exposure typical of extracted neutron beams. Within experimental uncertainty, *no single preferential irradiation direction* exists in the employed configuration. The adopted setup is therefore well suited for achieving high integrated fast-neutron fluence while providing a realistic and unbiased irradiation environment for materials testing [147].

3. High-resolution transmission electron microscopy

High-resolution (HR) cross-sectional imaging of the α GHSiC system was performed with the JEOL JEM-2100 transmission electron microscope (TEM) operating at 200 kV. Thin lamellae for the TEM observations were prepared with the focused ion beam technique using the FEI Helios NanoLab 600 Dual Beam Microscope equipped with the Omniprobe lift-out system [60].

4. Micro-Raman analysis

The analysis of graphene and SiC-related modes was carried out with a Renishaw InVia Raman spectrometer equipped with the 532-nm (2.33-eV) line of an Nd:YAG laser. The laser beam was directed onto the samples via a 100 $\times$ objective lens and focused on the SiC surface. Three Raman maps were collected for the presented experiment. The first map 20 $\mu\text{m} \times 20 \mu\text{m}$ 0.5 μm -step 1681-point in the middle of the graphene mesa, in the Raman shift range (1200 – 2800 cm^{-1}). The second map, reference map, 20 $\mu\text{m} \times 20 \mu\text{m}$ 0.5 μm -step 1681-point was measured outside the graphene mesa within the same spectral range, but without visible Raman modes of

graphene. The third Raman map 20 $\mu\text{m} \times 20 \mu\text{m}$ 1 μm -step 441-point was also collected outside the graphene mesa, in the range of 0 – 1200 cm^{-1} , for the analysis of the SiC substrate. Following irradiation, the same three maps were acquired for each structure. To extract the graphene Raman modes, the SiC reference map measured after irradiation was subtracted, as the Raman spectrum of SiC exhibits substantial changes upon irradiation.

5. Hall effect analysis

The density, mobility, and type of charge carriers in α GHSiC were determined both before and after irradiation using a 1 mA direct current bias. This was conducted with a LINSEIS HCS-1 automated Hall effect measurement system, which operates under the van der Pauw formula and utilizes a single-carrier transport model. Therefore, hole and electron carrier concentrations in graphene are not monitored independently; instead, the measured electrical parameters reflect their combined contribution. In single-carrier transport regimes, the measurements primarily represent the majority carrier concentration, with a minor modulation from residual minority carrier effects [71]. The measurements were conducted at room temperature with a 0.65 T permanent magnet.

6. DFT

The *in silico* modeling in this study was conducted within the framework of DFT using a plane-wave basis set and ultrasoft pseudopotentials, as implemented in the QUANTUM ESPRESSO package [150–152]. The pseudopotentials employed incorporated scalar-relativistic effects and nonlinear core corrections. Exchange–correlation interactions were treated using the generalized gradient approximation with the Perdew–Burke–Ernzerhof functional [153,154]. To account for van der Waals (vdW) interactions, Grimme’s D3 dispersion correction was applied [155,156]. Structural optimizations were carried out by relaxing all atomic positions until the residual forces on each atom were below $10^{-4} \text{Ry au}^{-1}$ and the total energy change was less than 10^{-5}Ry . The surface model consisted of periodic slabs built from a 4×4 SiC(0001) substrate, with a 5×5 graphene layer adsorbed on one side to simulate surface coverage.

APPENDIX C: SUPPLEMENTARY DATA

1. Micro-Raman analysis of spectral shifts in graphene

A detailed analysis of the position and full width at half maximum (FWHM) of the Raman modes is presented in Fig. 9.

With increasing fast-neutron fluence, the G and 2D Raman modes exhibit spectral broadening, attributed to a rise in defect density [93,157]. Irradiation also induces a redshift in both modes. The absence of peak splitting, combined with the concurrent redshift of the G and 2D

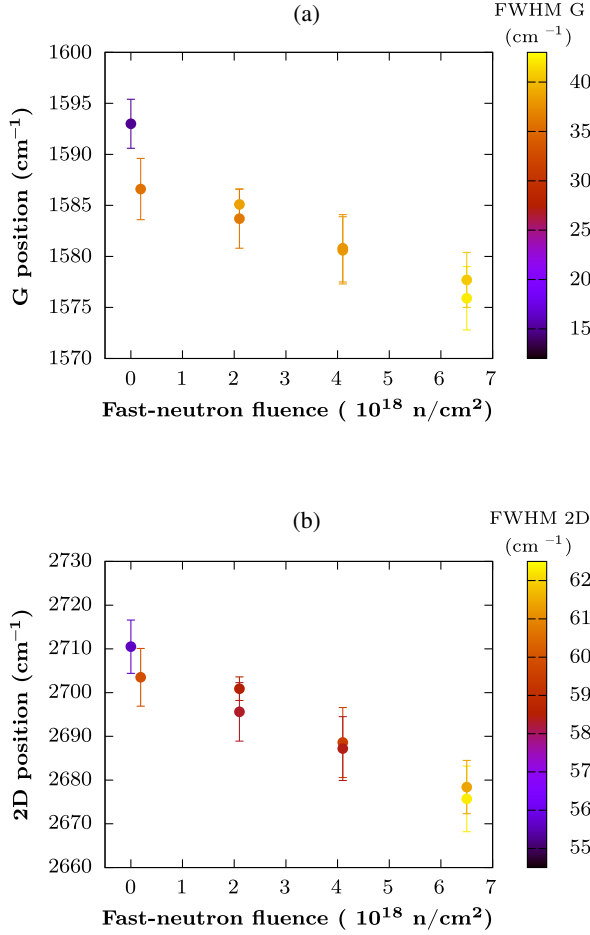


FIG. 9. Positions of the (a) G and (b) 2D Raman modes for all samples. The fill color of the points indicates the corresponding width of the G and 2D Raman modes.

modes, is indicative of biaxial strain [158]. Accordingly, the pronounced redshift and increased FWHM observed in both modes following fast-neutron exposure suggest a reduction in hole doping levels [159,160].

2. Micro-Raman analysis over Lee's projection

Figure 10 presents the 1681-point $20\text{--}\mu\text{m} \times 20\text{--}\mu\text{m}$ Raman maps of each sample's 2D and G mode positions. We adjusted *Lee's* space to the laser wavelength (532 nm) compared with the original research designed for transferred graphene on a SiO₂/Si substrate [158]. Data clouds above the neutral point indicate compressive deformation, and below this point indicate tensile deformation [158,161]. Graphene epitaxy occurs at 1873 K. Upon cooling to room temperature, the SiC substrate contracts, inducing compressive strain in the graphene layer. Following neutron irradiation, the SiC substrate begins to swell, causing the compressive stresses in graphene to release. The data indicate that at higher neutron fluences, tensile stresses develop.

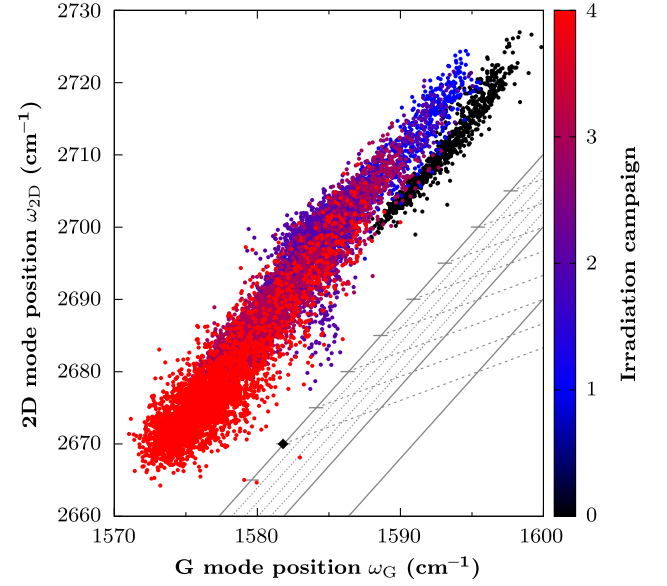


FIG. 10. Data cloud of G and 2D band positions derived from 1681-point Raman maps ($20\text{--}\mu\text{m} \times 20\text{--}\mu\text{m}$) of heterostructures exposed to varying levels of neutron irradiation. The points are plotted against Lee's half-plane, which defines the limits for p-doped single-layer micromechanically exfoliated graphene transferred onto a SiO₂/Si substrate. The black diamond represents undoped and unstrained graphene on SiO₂/Si.

The data points of most samples are scattered along a line with a slope of 2.2. This slope corresponds to the ratio of strain-induced shifts of the Raman G and 2D modes [158]. Table III summarizes the slope of a straight line around which the experimental data points lie and the associated Pearson coefficient. They indicate that as a result of neutron irradiation, in addition to changes in strain, free carrier concentration also shifts.

3. Micro-Raman-based defect approximation in graphene

This study employs a well-established micro-Raman spectroscopic approach to approximate the defect density in graphene. The method relies on the intensity ratio of the

TABLE III. Slope and Pearson's coefficient for the data points of the G and 2D band positions as a function of fast-neutron fluence.

Samples	Campaign	Slope	Pearson's r
Ref.	Not applicable	2.4	0.96
1 _A	Φ_1	2.0	0.95
2 _A	Φ_2	1.4	0.75
2 _B	Φ_2	2.0	0.87
3 _A	Φ_3	2.3	0.93
3 _B	Φ_3	2.1	0.94
4 _B	Φ_4	2.2	0.91
4 _B	Φ_4	1.7	0.79

disorder-induced D mode to the G mode, which provides a quantitative measure of defect density [93,94]. The relationship is given by

$$\frac{I_D}{I_G} = C_A \frac{r_A^2 - r_S^2}{r_A^2 - 2r_S^2} \left[e^{-\pi r_S^2/L_D^2} - e^{-\pi(r_A^2 - r_S^2)/L_D^2} \right]. \quad (C1)$$

Here, C_A represents the maximum attainable value of I_D/I_G , corresponding to the idealized case in which the D band is activated throughout the sample while preserving the hexagonal sp^2 carbon lattice. The parameter C_A decreases with increasing laser excitation energy [93,94]; accordingly, we express it as

$$C_A = 160 \times E_L^{-4}, \quad (C2)$$

where E_L is the laser energy. In our calculations, we use the excitation wavelength of 532 nm ($E_L = 2.33$ eV) employed in the experiment. Therefore, the value of C_A is 5.43.

The parameter L_D denotes the average distance between defects. The quantity r_S defines the radius of the structurally disordered region created by a defect, whereas r_A corresponds to the radius of the surrounding area in which D-band scattering is activated while the sp^2 hexagonal lattice remains preserved. In the original work by Lucchese *et al.* [94], values of $r_S = 1$ nm and $r_A = 3$ nm were estimated based on scanning tunneling microscopy observations of highly oriented pyrolytic graphite subjected to 90-eV Ar^+ ion bombardment.

For the purposes of the present study, we adopt the same values of r_S and r_A for neutron-irradiated graphene. This approximation is justified by the generally comparable spatial extent of lattice distortions induced by different types of high-energy particle irradiation in graphene [162–164].

Once L_D is obtained from the above expression, the defect density n_D can be calculated as

$$n_D (\text{cm}^{-2}) = \frac{10^{14}}{\pi L_D^2}. \quad (C3)$$

We note that the micro-Raman-based defect approximation outlined above is valid in the low-defect-density regime, where $L_D \gtrsim 10$ nm. At higher defect densities, when L_D approaches r_S , the ratio I_D/I_G becomes sensitive not only to the activated area, weighted by the parameter C_A , but also to the structurally disordered region, weighted by an additional parameter C_S . In this regime, a second contribution must be added to Eq. (C1), taking the form $C_S[1 - \exp(-\pi r_S^2/L_D^2)]$ [94].

4. Micro-Raman-based defect approximation in SiC

Establishing a quantitative link between the phonon-mode shifts and defect concentration in SiC is essential for

interpreting irradiation-induced structural modifications using Raman spectroscopy. Previous study by Wendler *et al.* [95] investigated defect evolution in neutron-irradiated SiC through a combination of Rutherford back-scattering spectrometry, optical absorption, and Raman spectroscopy. Their results established a robust, three-way correlation between irradiation fluence, LO-mode position, and defect concentration. This demonstrated that the irradiation-induced LO shift is a strong and predictable indicator of defect density, thereby enabling a micro-Raman-based approximation of the defect content in SiC.

However, neutron sources used in different irradiation campaigns typically exhibit distinct neutron energy spectra, which can introduce discrepancies in defect formation rates if not properly accounted for. To assess the potential influence of these spectral differences, we superimposed our experimental results for the LO phonon mode position in 6H-SiC as a function of fast-neutron fluence onto the data reported by Wendler *et al.* The agreement between the two datasets is excellent, indicating that differences in neutron spectral distribution have no significant impact on the LO-mode behavior within the investigated fluence range.

Consequently, we adopt the defect concentration scaling established by Wendler *et al.* and directly transpose their fluence-dependent relationship to our results (see Fig. 11). The combined dataset reveals that, within the fluence range of 10^{17} to 10^{19} n/cm², the LO-mode shift—used here as a proxy for the relative defect concentration in SiC—follows a quadratic decay model described by

$$D(\omega_{\text{LO}}) = a(\omega_{\text{LO}}^0 - \omega_{\text{LO}})^2 + b(\omega_{\text{LO}}^0 - \omega_{\text{LO}}), \quad (C4)$$

where ω_{LO}^0 and ω_{LO} denote the LO phonon mode positions before and after irradiation, respectively. The

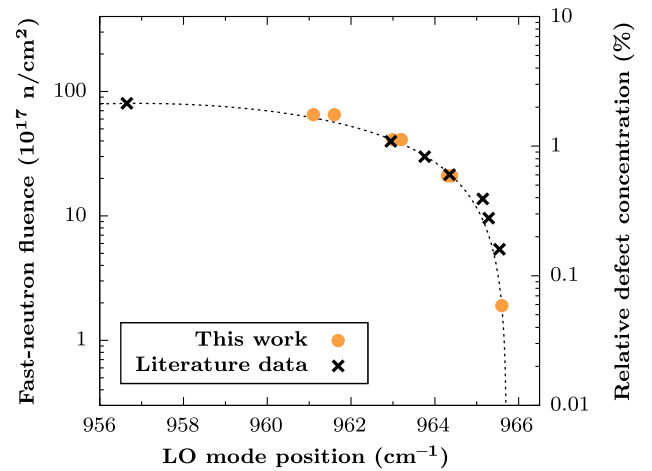


FIG. 11. Defect evolution in neutron-irradiated SiC, relative to the position of its LO phonon mode. Literature data points are obtained from Ref. [95].

empirical fitting parameters are $a = 0.0122 \text{ cm}^2$ and $b = 0.3269 \text{ cm}$.

This correlation provides a consistent framework for estimating defect densities in irradiated SiC using Raman spectroscopy, extending the applicability of the Wendler *et al.* model to fast-neutron environments relevant to the present study.

5. The impact of increasing neutron fluence on the electrical parameters of the system

The extent of radiation-induced changes in electrical parameters is presented in Fig. 12(a). Neutron irradiation with progressively increasing fluence levels (corresponding to campaign steps Φ_1 - Φ_4 , as summarized in Table II) resulted in average changes in measured carrier density (Δn) of 24%, 42%, 404%, and 618%, respectively—regardless of the change in conductivity type. These shifts were accompanied by a progressive decline in charge

carrier mobility ($\Delta\mu$), with average reductions of 39%, 80%, 91%, and 96%, respectively, relative to the preirradiation values.

Using the changes observed after irradiation step Φ_1 as reference, we assess the impact of increasing the neutron fluence by factors of approximately 10 (Φ_2/Φ_1), 20 (Φ_3/Φ_1), and 30 times (Φ_4/Φ_1), as shown in Fig. 12(b). Notably, the linear increase in neutron fluence does not result in a proportional degradation of the system's electrical properties. Beyond the fluence level reached in campaign step Φ_3 , further increases in exposure do not induce significant additional changes. This behavior indicates a saturation effect, wherein radiation-induced alterations stabilize, with carrier density and mobility approaching asymptotic values of approximately $-3 \times 10^{13} \text{ e/cm}^2$ and $-40 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$, respectively.

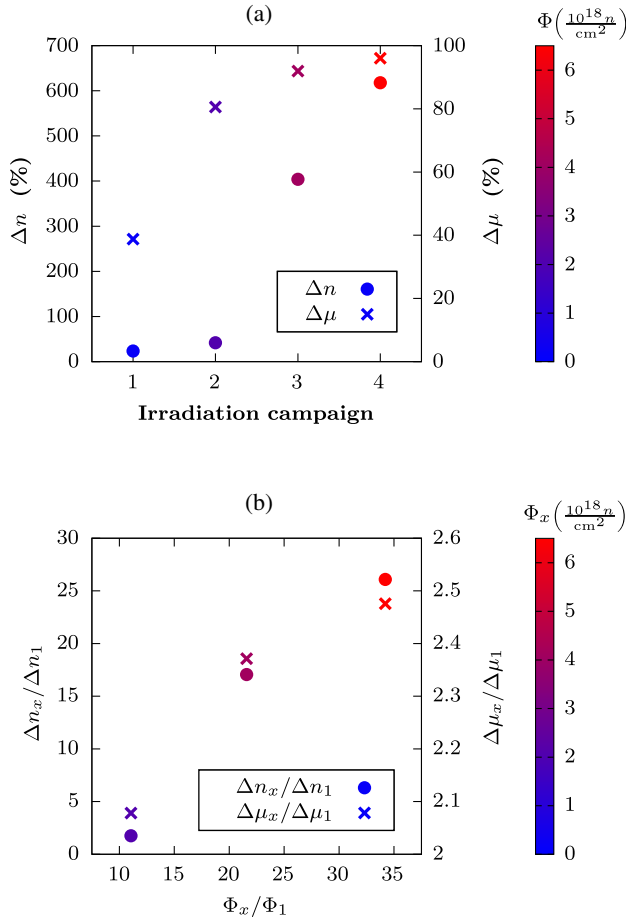


FIG. 12. (a) Radiation-induced changes in electrical parameters of heterostructures, relative to their preirradiated values. (b) Progressive saturation of radiation-induced effects on the electrical characteristics of the irradiated heterostructures, illustrated in relation to the effects induced by the initial process of the radiation campaign (Φ_1).

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