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Effect of low dose neutron irradiation on the microstructure of 6H-SiC single crystals

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Abstract

The irradiation response of pure and nitrogen-doped 6H-SiC single crystal was investigated after low-fluence neutron irradiation at 30~60°C. Internal Friction, X-Ray Diffraction, Raman spectroscopy and Transmission Electron Microscopy were used to study the microstructural changes. Neutron irradiation induced a significant increase, the internal friction, which show different behaviors with the increasing temperature, indicating a competition of two opponent contributions from different types of defects. The doped nitrogen atoms have resulted in minor lattice shrinkage because of smaller atomic radius. Neutron irradiation has induced obvious volume swelling of (2.4-3.4)%, which is slightly larger for nitrogen-doped 6H-SiC. Raman spectra showed significant changes in peak intensity, shift, and broadening, correlating with the formation of irradiation-induced defects. The total disorder, derived from Raman spectra, increased with irradiation dose and reached saturation, with nitrogendoped samples showing higher disorder than pure ones. These findings highlight the influence of nitrogen doping on the microstructural response of 6H-SiC to neutron irradiation, offering insights into its potential applications in nuclear environments.

Full Text

Preamble

Effect of Low-Dose Neutron Irradiation on the Microstructure of 6H-SiC Single Crystals

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Abstract

The irradiation response of pure and nitrogen-doped 6H-SiC single crystals was investigated following low-fluence neutron irradiation at 30–60°C. We used internal friction (IF), X-ray diffraction (XRD), Raman spectroscopy, and transmission electron microscopy (TEM) to characterize the microstructural changes. Neutron irradiation induced a significant increase in internal friction, which exhibited different temperature-dependent behaviors, indicating competition between two opposing contributions from distinct defect types. The nitrogen dopants caused minor lattice shrinkage due to their smaller atomic radius. Neutron irradiation produced obvious volume swelling of 2.4–3.4%, which was slightly greater in nitrogen-doped 6H-SiC. Raman spectra showed significant changes in peak intensity, shift, and broadening that correlated with irradiation-induced defect formation. The total disorder, derived from Raman spectra, increased with irradiation dose and reached saturation, with nitrogen-doped samples displaying higher disorder than pure ones. These findings highlight the influence of nitrogen doping on the microstructural response of 6H-SiC to neutron irradiation, offering insights for potential applications in nuclear environments.

Keywords: 6H-SiC, neutron irradiation, swelling, internal friction, XRD, Raman

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1. Introduction

Silicon carbide (SiC) is highly attractive for nuclear applications due to its superior high-temperature mechanical properties, high thermal conductivity, low thermal expansion coefficient, low neutron absorption cross-section, and minimal production of long-lived radioisotopes under neutron irradiation [1][2][3]. Consequently, significant research has focused on SiC-based materials for potential use in structural components and accident-tolerant fuel cladding within the nuclear energy sector [4][5]. Additionally, neutron-irradiated SiC, particularly at relatively low fluences, can serve as a temperature monitor in research reactors and for aerospace applications [6][7][8][9][10].

For these applications, high-energy neutrons create atomic displacements that induce high concentrations of defects, causing substantial microstructural changes that alter the material’ s mechanical, thermal, and electrical properties—factors crucial for performance in extreme conditions [11]. Extensive studies have elucidated the mechanisms of irradiation-induced swelling [12][13][14][15][16][17], concluding that swelling occurs primarily through the accumulation of radiation-induced defects, including point defects, small interstitial clusters, large dislocation loops, and vacancy clusters [18]. At moderate fluences and low irradiation temperatures, SiC swelling is dominated by point defects [14][15], whose stability can be affected by polytype and impurities [19]. While nitrogen doping has become a routine method to improve SiC' s electronic properties, the synergistic effects of nitrogen doping and neutron irradiation-induced defects on 6H-SiC single crystal microstructure remain understudied [20][21]. Furthermore, limited application of techniques such as internal friction (IF), which has proven valuable for detecting irradiation-induced microstructural changes, has hindered investigation of SiC' s irradiation response [22][23][24].

In this work, we conducted a comparative study of pure and nitrogen-doped 6H-SiC single crystals to reveal microstructural changes after low-fluence neutron irradiation at low temperature. Using internal friction (IF), X-ray diffraction (XRD), Raman spectroscopy, and transmission electron microscopy (TEM), we aimed to deepen understanding of irradiation-induced microstructural evolution and compare the effects of nitrogen doping on these changes. These findings provide valuable insights for optimizing SiC-based materials for nuclear applications and enhancing their performance under irradiation.

2. Experimental

The samples consisted of bulk pure 6H-SiC (denoted as 6H-SI, electrical resistivity: \$ 1×10^{5}\$ $\Omega \cdot$cm) and nitrogen-doped 6H-SiC (denoted as 6H-N, doping concentration: 10^{19} ions/cm^3, electrical resistivity: 0.015–0.028 $\Omega \cdot$cm). All single crystal wafers were oriented on the (0001) plane with dimensions of 40 mm length, 3 mm width, and 0.30–0.42 mm thickness. Samples were irradiated under fast neutron fluxes (E $>$ 0.1 MeV) of 0.80×10^{14} and 0.91×10^{14} n/cm$^2 \cdot$s, achieving fast neutron fluences of approximately 2.31×10^{20} and 2.44×10^{20} n/cm^2, respectively, using the China Mianyang Research Reactor (CMRR). A perforated capsule minimized heating effects by allowing maximum water coolant flow during irradiation, as described elsewhere [25]. The irradiation temperature was estimated to be 30–60°C based on neutronics and thermal-hydraulic calculations. The neutron irradiation parameters for all samples are summarized in Table 1 .

X-ray diffraction (XRD) measurements were performed using a conventional DX-2700BH diffractometer (Haoyuan Instrument Co., China) with a Cu-Kα source operating at 40 kV and 30 mA. Spectra were recorded for the (00012) Bragg reflection plane from 73.5° to 76° with an angular step size of 0.004° and a counting time of 1 s per step.

Raman spectroscopic investigation was conducted using a micro-Raman spectrometer (WITec, alpha300 R). All Raman spectra were excited with a 532 nm laser at approximately 2 mW intensity to avoid sample heating.

Internal friction measurements were performed using an inverted torsion pendulum (developed by the Institute of Solid State Physics, HFIPS, Chinese Academy of Sciences) operating in free-decay mode with a strain amplitude of 80×10^{-6} and frequencies around 1 Hz. The internal friction coefficient, Q^{-1}, was determined from the free-decay signal. Spectra were measured from room temperature to 400°C at a heating rate of 1°C/min.

Microstructure was characterized using a transmission electron microscope (TEM, FEI, Themis Z). TEM samples were prepared using conventional focused ion beam (FIB, Thermo Scientific, Scios 2) lift-out techniques.

3.1 Internal Friction

Figure 1 presents the temperature-dependent internal friction of virgin and irradiated 6H-SI and 6H-N single crystals. No detectable relaxation peaks were observed in any samples. For unirradiated samples, internal friction was relatively low and increased slowly with temperature for both 6H-SI and 6H-N, which is typical for single-crystal SiC and similar to sapphire behavior [26]. In the virgin 6H-N sample, an upward shift occurred after temperatures reached 300-350°C. At low interstitial-solute concentrations, the relaxation strength of internal friction at a given temperature depends on the nature, positions, and concentration of interstitial atoms [27]. At the testing frequency of 1 Hz, nitrogen-related Snoek peaks have been observed in various materials, including around 300 K for Fe, 562 K for Nb, and 615 K for Ta [28]. In this work, the increased internal friction in the virgin 6H-N sample at higher temperatures is attributed to the evolution of doped nitrogen atoms, with the possible nitrogen-related Snoek peak expected to appear at temperatures above the tested maximum of 673 K.

For both 6H-SI and 6H-N samples, internal friction increased significantly after neutron irradiation. At room temperature, internal friction for virgin samples was less than 0.5×10^{-3}, but it rose substantially to approximately 2×10^{-3} for all irradiated samples. A distinct plateau appeared in the internal friction curves between approximately 150°C and 230°C for all irradiated samples, with internal friction rising rapidly outside this temperature range. The primary contributor to internal friction in SiC is defect relaxation [29], and the formation of numerous radiation-induced defects leads to the observed increase. The nature of radiation-induced defects in SiC is multifaceted, and their recovery behavior at elevated temperatures varies significantly depending on defect type [15][18][4][14], resulting in complex temperature dependence of internal friction. The plateau in Figure 1 suggests concurrent contributions from two competing defect types to internal friction between approximately 150°C and 230°C. This competition warrants further investigation, as it may provide insights into the thermal recovery of specific defects under irradiation.

Figure 2 shows the relative elastic moduli of virgin and irradiated 6H-SI and

6H-N single crystals. Neutron irradiation caused significant modulus increases for both materials, with higher doses producing higher moduli. This can be attributed to enhanced deformation resistance caused by accumulated radiation-induced defects, whose strain fields impede dislocation motion.

3.2 Irradiation-Induced Swelling

To characterize neutron irradiation-induced lattice swelling, XRD measurements recorded patterns near the (00012) reflection plane, as shown in Figure 3. Small peaks accompanying the main peaks correspond to Kα_2 spectra. Compared to 6H-SI, diffraction peaks of 6H-N shift to higher angles under identical irradiation conditions, indicating lattice shrinkage attributed to nitrogen' s smaller atomic radius (65 pm) compared to Si (110 pm) and C (70 pm). For both 6H-SI and 6H-N, neutron irradiation caused obvious peak shifts to lower angles. According to Bragg's law ($2d \sin\theta = n\lambda$), this shift indicates interplanar spacing expansion, signifying lattice swelling induced by neutron irradiation [16], with the extent of swelling increasing with irradiation dose.

The XRD data were quantitatively analyzed using Jade software, with results listed in Table 2 . Volume swelling was calculated assuming isotropic lattice swelling in 6H-SiC, as demonstrated in previous studies [21]. The results indicate volume swelling of 2.4-3.4% following low-dose neutron irradiation at low temperature, consistent with Kuryachiy et al., who observed 2.5-3% lattice volume expansion in 3C-SiC after a neutron fluence of $\sim 2 \times 10^{20}$ n/cm^2 (E > 0.18 MeV) at $\sim$ 100°C [30]. Notably, volume swelling in 6H-N samples exceeded that in 6H-SI samples under identical irradiation parameters, suggesting that nitrogen doping may enhance swelling behavior. This indicates that neutron-irradiated 6H-N could be more suitable for temperature-sensing applications due to its higher swelling response.

3.3 Irradiation-Induced Microstructural Changes

Raman spectroscopy examined neutron irradiation effects on 6H-SiC crystalline structure, with spectra shown in Figure 4 [Figure 4: see original paper]. For both 6H-SI and 6H-N samples, relative Raman intensity decreased exponentially after irradiation, a direct result of increased optical absorption in the damaged layer where defects accumulate [31]. Theoretical and experimental studies indicate that Raman-active modes in 6H-SiC include 5A$_1$, 5E$_1$, and 6E$_2$ [32]. For virgin 6H-SI samples in the 200-2000 cm^{-1} range, three first-order Si-C vibration peaks were detected at 773, 795, and 972 cm^{-1}, corresponding to E$_2$(TO), E$_2$(TO), and A$_1$(LO) modes, respectively [20][33]. In contrast, the virgin 6H-N sample exhibited an additional first-order peak at 505 cm^{-1} (A$_1$(LA) mode) and two second-order peaks in the 1500-1800 cm^{-1} range. The asymmetric peak at 505 cm^{-1} can be deconvoluted into two smaller peaks (486 cm^{-1} and 507 cm^{-1}) attributed to valley-orbit transitions at nitrogen donors on two inequivalent cubic sites [34].

Figure 4 shows significant changes in peak intensity, shift, and broadening in neutron-irradiated samples compared to virgin ones, indicative of irradiation-induced defect formation [35]. As discussed by Chen et al. [20], Raman analysis is highly sensitive to antisite defects and interstitial atoms. Obvious blueshift of the A_1(LO) peak occurred after neutron irradiation due to radiation-induced lattice strain [20][36], consistent with the substantial lattice swelling discussed in Section 3.2.

To quantitatively assess irradiation-induced damage from Raman spectra, total disorder (1 - A_{norm}) was calculated based on Menzel et al.' s definition, where A_{norm} corresponds to the total area A under characteristic Raman lines normalized to the crystalline material value A_{cryst} (i.e., A_{norm} = A/A_{cryst}) [36]. Available ion and neutron irradiation data from literature [20][31] are included in Figure 5. The calculated total disorder for 6H-N samples follows a trend similar to ion-irradiated SiC at both room temperature and 400°C, increasing with irradiation dose and reaching saturation at comparable doses. In contrast, 6H-SI samples exhibit much lower total disorder at equivalent doses, suggesting that nitrogen doping significantly impacts defect structure, as changes in nitrogen concentration can dramatically alter the position and shape of the A_1(LO) phonon, leading to higher total disorder [37].

4. Conclusions

This study investigated microstructural evolution in pure and nitrogen-doped 6H-SiC after low-fluence neutron irradiation at 30–60°C. The results demonstrate that neutron irradiation significantly alters internal friction, lattice swelling, and mechanical properties of SiC. The internal friction of both pure and nitrogen-doped samples increased markedly post-irradiation, with a characteristic plateau observed between 150°C and 230°C indicative of competing contributions from different defect types. Additionally, neutron irradiation caused substantial increases in the elastic modulus of both 6H-SI and 6H-N samples, with higher irradiation doses correlating with higher moduli, suggesting that radiation-induced defect accumulation leads to increased material stiffness consistent with radiation hardening. XRD revealed that neutron irradiation induced lattice swelling in both pure and nitrogen-doped 6H-SiC, with volume expansions ranging from 2.4% to 3.4%. Notably, swelling was more pronounced in nitrogen-doped SiC, attributable to enhanced defect formation due to nitrogen doping. Raman spectroscopy confirmed that irradiation induced extensive defect formation, evidenced by changes in peak intensity, shift, and broadening, with nitrogen-doped samples exhibiting higher total disorder compared to pure SiC. These findings provide valuable insights into SiC irradiation behavior, particularly the influence of nitrogen doping on defect formation, lattice swelling, and material properties under neutron irradiation.

CRediT Authorship Contribution Statement

Songbao Zhang: Writing -original draft, Methodology, Investigation
Zhanfeng Yan: Methodology, Investigation
Hao Wang: Methodology, Investigation
Jiting Tian: Investigation
Wei Zhou: Investigation, Funding acquisition
Yuchi Cui: Methodology, Investigation
Jian Zheng: Writing -review & editing, Methodology, Investigation, Conceptualization
Zhe Chen: Supervision, Resources, Conceptualization

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data Availability

Data will be made available on request.

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