Quantification of irradiation defects in beta-silicon carbide using Raman spectroscopy^{a)}

T. Koyanagi,^{1, b)} M. J. Lance,¹ and Y. Katoh,¹

¹*Oak Ridge National Laboratory, Oak Ridge, Tennessee 37831, USA*

Raman spectra from polycrystalline beta-silicon carbide (SiC) were collected following neutron irradiation at 380–1180°C to 0.011–1.87 displacement per atom. The longitudinal optical (LO) peak shifted to a lower frequency and broadened as a result of the irradiation. The changes observed in the LO phonon line shape and position in neutron-irradiated SiC are explained by a combination of changes in the lattice constant and Young's modulus, and the phonon confinement effect. The phonon confinement model reasonably estimates the defect-defect distance in the irradiated SiC, which is consistent with results from previous experimental studies and simulations.

I. INTRODUCTION

Silicon carbide (SiC) has been extensively investigated as a high temperature material for use in nuclear core structures¹. Core materials are exposed to extreme environments, including high temperatures and neutrons during operation, which results in significant changes in the thermo-physical and mechanical properties¹. The changes are due to irradiation-induced defects including Frenkel pairs, antisite defects, and clusters of interstitials and vacancies. Transmission electron microscopy (TEM) has been systematically applied to understand radiation temperature and neutron dose dependencies on the microstructural evolution of SiC². However, the correlation between microstructures and macroscopic properties, such as volumetric swelling and irradiation creep, is still poorly understood, as TEM-visible defects account for only a small fraction of the changes in those properties³. This suggests the importance of evaluating atomistic defects, that cannot be observed by conventional TEM techniques because of the resolution limit (typically ~1 nm in TEM dark field imaging). Atomistic defects in irradiated SiC have been successfully evaluated in detail using various techniques, including positron annihilation spectroscopy⁴, electron paramagnetic resonance⁵, photoluminescence⁶, deep-level transient spectroscopy⁷ and Raman spectroscopy^{8,9,10,11}. We propose a new approach to characterize irradiation defects in SiC using Raman spectroscopy to provide a better understanding of irradiation effects on the microstructure of SiC.

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^{b)} Corresponding author. Electronic mail: koyanagit@ornl.gov

Tiong et al.¹² proposed an approach to estimate the sizes of undamaged regions in irradiated semiconductors using Raman spectroscopy. In that study, the analysis interpreted the Raman spectra of ion-irradiated GaAs using the phonon confinement model¹³. We interpret the longitudinal optical (LO) phonon line shape and position of the Raman spectra of neutron-irradiated SiC using the phonon confinement model to quantify the radiation damage. This analysis considers the irradiation-induced changes in the modulus and lattice constant and the phonon confinement effects. The approach to analyzing the LO phonon line in this study is different from the approach used in previous studies^{8, 9, 10, 11}. The interests in the previous Raman spectroscopy studies, such as radiation-induced amorphization and analysis of Si-Si and C-C homonuclear bands, are beyond the focus of this study.

II. EXPERIMENTAL METHODS

Polycrystalline, chemical vapor deposited (CVD) β -SiC (high resistivity grade, Dow Chemical Co., Marlborough, Massachusetts) was used in this investigation. The material purity was $\geq 99.9995\%$. The unirradiated material contained preexisting defects such as grain boundaries, twin boundaries, and stacking faults. The average coherent grain size of ~ 200 nm was confirmed by X-ray diffraction (XRD) and TEM, and nearly isotropic grain growth was observed¹⁴. Neutron irradiations were conducted in the High Flux Isotope Reactor at Oak Ridge National Laboratory. The irradiation temperature and neutron fluence ranged from 380 to 1180°C and 0.011 to 1.87×10^{25} n/m² [$E > 0.1$ MeV], respectively. These fluences correspond with nominal doses of 0.01 to 1.87 displacement per atom (dpa) in SiC. The irradiation conditions are summarized in TABLE I. The irradiation temperatures were determined by post-irradiation measurement of the dimensional changes of the SiC specimens upon annealing, using a dilatometer¹⁵.

Raman spectra of the SiC specimens were recorded at room temperature with a DilorXY800 (Horiba Scientific, Edison, NJ) Raman microprobe using an Innova 308C Ar⁺ ion laser (Coherent Laser Group, Santa Clara, CA) at 515 nm and 50 mW output power. The laser was focused using a 50 $\times$ objective to a spot roughly 2 μ m in diameter. Calibration of the spectra was conducted using a peak from the argon laser at 116.2 cm⁻¹. Five spectra were collected and averaged for each specimen, which enabled us to evaluate ~ 500 coherent grains. Since the pre-existing defects were not changed by irradiation³ and the defect density was much smaller than the irradiation-defect density, the preexisting defects could be ignored during the analysis of the Raman spectra.

Other physical property and microstructural observations were also conducted to aid in analysis. The volumetric swelling of the specimens was calculated based on the length change of the specimen measured using a conventional micrometer, assuming isotropic swelling in all directions. The original specimen length was ~ 40 mm, and the accuracy of the evaluation of the volumetric swelling was $\pm 0.08\%$. The microstructure of a selected specimen was examined by TEM. The TEM foil

was prepared using a focused ion beam (FEI, Versa 3D DualBeam) operated at 16 and 30 kV for rough milling and 2 and 5 kV for final thinning. TEM observation was conducted using a JEOL JEM2100F operated at 200 kV. The lattice constants were obtained using high-energy XRD in another work¹⁶.

TABLE I. Summary of irradiation conditions and properties of SiC materials. Parentheses indicate the standard deviation. The error in the determination of the lattice constant and volumetric swelling was less than 0.00008 Å and $\pm 0.08\%$, respectively.

Irradiation temperature [°C]	Neutron dose	Volumetric swelling [%]	Lattice constant [Å] ¹⁶	Estimated correlation length [nm]
Unirradiated	0	0	4.36075	Not applicable
380	0.011 dpa	0.48	4.36481	2.7 (0.5)
380	0.11 dpa	1.50	4.38131	0.6 (0.0)
540	0.011 dpa	0.39	4.36302	2.0 (0.3)
540	0.11 dpa	1.12	4.37448	0.8 (0.0)
790	0.11 dpa	0.34	4.36650	2.5 (0.5)
760	1.4 dpa	0.76	4.36800	0.8 (0.1)
1180	1.9 dpa	0.28	4.36221	15 (5)

III. RESULTS AND DISCUSSION

A. ANALYSIS OF RAMAN SPECTRA

The recorded Raman spectra of the unirradiated and irradiated specimens are shown in FIG. 1. The peaks of the transverse optical (TO) and LO phonon lines of β -SiC appear at around 800 and 950 cm^{-1} , respectively. Additional broad bands at ~ 200 , ~ 520 , ~ 1400 and $\sim 1600 \text{ cm}^{-1}$ were observed only in irradiated specimens. Those peaks are considered to be related to vibrations of Si-Si (~ 200 and $\sim 520 \text{ cm}^{-1}$) and C-C (~ 1400 and $\sim 1600 \text{ cm}^{-1}$) bonds⁹. In all cases, the peak intensity of the TO and LO phonon lines decreased following irradiation. In addition, there was a significant peak shift to lower frequencies and broadening of the TO and LO phonon peaks, depending on the irradiation conditions. These qualitative changes in the Raman spectra were also reported in previous work^{8, 9, 10, 11}. Several factors can contribute to changes in the Raman peak position and/or linewidth of the TO and LO peaks. These include changes in the lattice constant and phonon confinement caused by the reduction of undisturbed regions due to defects.

Irradiation defects in SiC are known to cause lattice expansion (elastic strain)². The effect of elastic strain on phonon Raman bands of β -SiC were studied by Olego et al.¹⁷, who found a linear correlation between Raman peak positions and elastic strain. For irradiated SiC, the peak shift resulting from elastic strain will change as the crystallite size decreases

because more defects will increase the strain-induced peak shift, which is related to the anharmonicity of the atomic bonding. Since the lattice expansion of the irradiated SiC was almost isotropic, we assume that the change in lattice spacing caused by applied stress and irradiation defects had the same effect on the peak shift. This assumption is reasonable based on the comparison of calculated and experimental TO lines shown below. The strain dependence of frequency (ω) of the TO and LO mode against the relative lattice constant can be given (in units of cm^{-1}) by¹⁷

$$\omega(TO) = 797 - 3734 \frac{E_{unir}}{E_{irr}} \frac{(a_{irr} - a_{unir})}{a_{unir}}, \quad \omega(LO) = 973 - 4532 \frac{E_{unir}}{E_{irr}} \frac{(a_{irr} - a_{unir})}{a_{unir}}, \quad (1)$$

where E and a are the Young's modulus and lattice constant, respectively. The subscripts *unir* and *irr* indicate properties of unirradiated and irradiated materials, respectively. The lattice constant was obtained from XRD¹⁶ (Table I). The change in modulus by radiation is given by²:

$$E_{irr} = E_{unir}(1 - 6.974 S) \quad (T_{irr} < \sim 1000^\circ\text{C}), \quad (2)$$

where S is volumetric swelling and T_{irr} is the irradiation temperature. As mentioned previously, the swelling was determined from the length change of the specimen (Table I). The reduction in the Young's modulus was up to 10% in this study, and the change in Young's modulus was negligible beyond the irradiation temperature of 1000°C . Using Equations (1) and (2), we calculated and then compared the peak position resulting from the change in the lattice constant with the observed peak shifts in FIG. 2. The calculated change in the lattice constant contributed to peak shifts of the TO and LO lines of up to 21 and 25 cm^{-1} , respectively. The peak shift of the TO phonon lines due to irradiation defects is fully explained by Equation (1). However, Eq. (1) does not explain the peak shift of the LO lines. The disagreement between the experimental and calculated shift becomes more significant for a larger lattice expansion. The remainder of the LO peak shift can be explained by phonon confinement effects due to irradiation defects, as will be shown later. The phonon confinement effect on the TO line is expected to be minimal owing to the phonon dispersion relation of β -SiC¹⁸.

The downshifting and asymmetric broadening of the Raman lines due to the nanostructures of the materials has been reported for various semiconductors¹⁹. Richter et al.¹³ reported a quantitative explanation of the Raman spectra of microcrystalline silicon using a phonon-confinement model, which was also applied to ion-irradiated GaAs¹² and MoS₂²⁰ materials. In an infinite crystal, only phonons from the center of the Brillouin zone ($q \sim 0$) are involved in first-order Raman scattering because of wave vector selection rules; but in a finite crystal, phonons can be confined in space by grain boundaries or defects. This results in uncertainty in the phonon momentum, and allows for phonons with $q \neq 0$ to contribute to the Raman spectrum. In the phonon confinement model the contribution of modes to the Raman spectrum is calculated by assuming a Gaussian distribution function of phonon states in q space. The q -dependent weight factor $C(q)$ can be written as¹³:

$$|C(q)|^2 = \exp\left(-\frac{q^2 L^2}{4}\right), \quad (3)$$

where q is expressed in units of $2\pi/a$, where a is the lattice constant, and L is the diameter of the correlation region. This weight factor was used for an ion-irradiated material^{12, 20} and recognized as an appropriate value²¹. Assuming uniform distribution of a spherical undamaged region in the irradiated material, the first-order Raman spectrum at frequency w , $I(w)$, can be written as integrating these contributions over the complete Brillouin zone¹³:

$$I(w) = \int \frac{|C(q)|^2 d^3q}{[w - w(q)]^2 + (\frac{\Gamma_0}{2})^2}, \quad (4)$$

where $w(q)$ is the phonon dispersion curve and Γ_0 is the natural linewidth of the zone-center optical phonon in the bulk (~ 3.0 cm^{-1} in this study). The dispersion $w(q)$ of the LO phonon in β -SiC is approximated as¹²: $w(q) \sim A + B \cos(\pi q)$, where $A = 903$ cm^{-1} and $B = 70$ cm^{-1} . This equation is shown to reasonably describe the dispersion of the LO phonon in β -SiC along the $\langle 110 \rangle$ direction¹⁸.

FIGURE 3(a) shows calculated Raman spectra of confined LO phonon lines in β -SiC based on Eq. (4). Both the asymmetric broadening of the line shape and the shift of the peak position toward the lower-frequency side became significant when the diameter of the correlation region was less than ~ 5 nm. This means that the phonon confinement model enabled an accurate evaluation of the size of the undistorted area only for highly damaged SiC. We will show later that the correlation length was less than 5 nm in most cases of this study, so the use of the phonon confinement model is an appropriate approach to quantifying the radiation damage. The Raman spectra of irradiated SiC can be calculated by the summation of Eqs. (1) and (4), which is shown for the experimental Raman spectra for low-dose (FIG. 3 (b)) and high-dose (FIG. 3 (c)) irradiations. The Gaussian peaks used for fitting are known as the Raman bands of disordered or amorphous SiC with peak positions of ~ 940 cm^{-1} for the low-dose case and ~ 870 cm^{-1} for the high dose case⁹. The Gaussian peak position tends to decrease as the lattice expansion increases, which might be useful as an indication of radiation damage. The experimental LO phonon line was fitted with the calculated LO line based on the peak position. For of the material irradiated at a low-dose, the fitting was straightforward because of the relatively sharp line shape. In the case of high-dose irradiation, there may have been certain variations in the fitting due to the broad LO line. However, the variation of the correlation length was small (typically less than 0.5 nm) because the calculated line shape and position were highly sensitive to small changes in the correlation lengths. The combined effects of the phonon confinement and change in the lattice constant explain the experimentally observed LO peak shifts up to 49 cm^{-1} and the LO peak broadening for all cases. The estimated correlation lengths are summarized in Table 1. Except for the case of irradiation at 1180 $^\circ\text{C}$, the correlation lengths were less than 4 nm.

B. VALIDATION OF ANALYSIS OF THE RAMAN SPECTRA

Validation of the analysis of the Raman spectra was carried out using TEM. A weak beam dark field micrograph (the inserted image in FIG. 4) shows high-density defects in SiC irradiated at 380 °C to 0.11 dpa, which are known to be interstitial clusters with an unidentified habit plane and Burgers vector due to the small size³. The average defect diameter and the density were ~1.5 nm and $\sim 1 \times 10^{24} \text{ m}^{-3}$, respectively. The average distance between the defects, i.e., average radius from a defect to the surrounding defects, was estimated to be ~7 nm, assuming the defect size was much smaller than the defect distance. This estimated defect distance was one order of magnitude larger than the value obtained from the Raman spectra (0.6 nm) if it is assumed that the correlation length in the phonon confinement model corresponds to the average defect distance. This is likely because TME could not capture the point defects.

FIGURE 4 shows the defect distances in irradiated SiC evaluated by different methods. The definition of defect distance for Raman is the correlation length, and the spacing between defects is used as the TEM defect distance. Additional TEM results from Katoh et al.²² are also included for comparison. A third experimental approach to evaluating the defect density is to analyze temperature-dependent thermal conductivity^{23, 24}. This method is sensitive to any defects causing phonon scattering. The defect mean free path, from the thermal conductivity results, is used as the average defect distance in FIG. 4. Crocombette et al.²⁵ conducted a simulation study of the thermal resistance of point defects and estimated the vacancy concentration in irradiated SiC. Note that these previous studies also investigated neutron-irradiated, high-purity, polycrystalline CVD β -SiC. Defect distances were observed that are similar to the results from this study and the historical thermal conductivity and simulation studies. The simulation work did not consider interstitial defects, but the agreement between this work and the simulations indicates that the phonon analysis of Raman spectra can capture significant numbers of the atomistic defects that cannot be observed in TEM. A similar defect distance was also found from the Raman spectra, and the thermal conductivity analysis provided additional validation of this method of Raman spectra analysis. The increasing defect distance with higher irradiation temperature can be explained by defect recovery processes, i.e., the annihilation of Frenkel pairs, which was enhanced with increasing temperature². The significant increase in the defect distance from ~800 to 1180 °C is attributed to the vacancy mobility above ~1000 °C in SiC². In addition, the defect distance tended to decrease with increasing swelling and neutron dose (up to 1.4 dpa) (TABLE I), which is quite reasonable. In summary, the correlation length obtained from Raman spectroscopy is consistent with the previous non-TEM experimental and simulation studies. Although Fano interference as a result of electron-phonon interactions can cause a low-lying frequency mode in Raman spectra, as observed in silicon²⁶, this phenomenon was not observed in the LO phonon line of

SiC²⁷. LO-phonon-plasmon coupling (LOPC) is another possible cause of a peak shift to a lower frequency and peak asymmetry. However, the LOPC mode caused peak asymmetry only at higher frequencies in both n- and p-type beta SiC^{28, 29}, which is not consistent with our observations. In addition, transmutation elements such as P, Mg, and Be produced during neutron irradiation are believed to have an ignorable effect on Raman spectra because of their limited amounts³⁰. Moreover, a high-density stacking fault is another possibility as a cause the asymmetric LO peak broadening in beta SiC³¹. However, this type of defect was not dominant in the irradiated SiC^{2, 3}. Therefore, the irradiation effect on the LO line is explained by the phonon confinement effect and lattice strain.

IV. CONCLUSIONS

This study proposes and provides preliminary validation of an experimental method to evaluate and quantify defects invisible to conventional TEM. The shift and peak broadening of the LO phonon line in the Raman spectra of SiC resulting from neutron irradiation, is interpreted as a combination of lattice strain and phonon confinement. The phonon confinement model enabled an estimation of the size of the undistorted area, i.e., the defect distance in highly disordered SiC due to the sensitivity to atomic-scale correlation lengths. The estimated defect distances were <3 nm and ~15 nm following irradiation at 380–790 °C and 1180 °C, respectively. The results are consistent with the results of previous experimental and simulation studies.

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FIG. 1 Raman spectra of unirradiated and irradiated SiC. Dotted lines indicate the peak positions of TO or LO phonon lines of the unirradiated material.

FIG. 2 Comparison of measured and calculated peak positions of SiC TO and LO phonon lines. The calculated values are based on results from equations (1).

FIG. 3 (a) Calculated Raman spectra of confined LO phonon lines of β -SiC with various size of correlation diameters. Raman spectra of irradiated SiC at 540°C to (b) 0.011 dpa and (c) 0.11 dpa are compared with the calculated spectra considering changes in lattice constant and the phonon confinement effect. The experimental spectra are in good agreement with the sum of the calculated spectra and a Gaussian peak known as the Raman band of amorphous or disordered SiC.

FIG. 4 Estimated defect distance calculated from Raman spectra plotted against irradiation temperature. Defect distances obtained by TEM, analyzing thermal conductivity, and simulation are also plotted. The number in red indicates neutron fluence for the Raman result (unit: dpa). The TEM micrograph inserted was taken by weak beam dark field imaging for SiC irradiated at 380°C to 0.11dpa. Beam direction (B) and g vector are indicated near the image.

Graphical Abstract

(B) Calculated,
Correlation length 2nm

Measured and calculated Raman spectra of irradiated SiC