Validity of Bragg's rule for heavy-ion stopping in silicon carbide

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The stopping powers for O, Al, Cr, Mn, Co, and Cu in a self-supporting SiC film have been measured in transmission geometry over a continuous range of energies using a time-of-flight elastic recoil detection analysis system. These stopping data, along with the stopping data in Si and C obtained using the same ions and measurement technique, are used to assess the validity of the Bragg additivity rule for stopping powers in SiC over a range of ions and energies. Within experimental uncertainties ($\pm 4\%$), the results indicate that Bragg's rule is valid in SiC for the ion species and energy regions studied. The measured stopping powers in C, Si, and SiC are also compared with the stopping power predictions of the two most recent versions of the SRIM (stopping and range of ions in matter) codes. While both versions of SRIM show varying degrees of agreement with the measured stopping data, there are significant deviations of the SRIM predictions for some ions and energy regions.

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I. INTRODUCTION

Knowledge of heavy-ion energy loss in matter is of fundamental and practical importance for rapidly expanding applications in ion-beam-based materials analysis, materials modification, device fabrication, implantation technology, nuclear physics, radiation damage, and radiation therapy. For over a century, the stopping of energetic ions in matter has been a subject of great experimental and theoretical interest. From an experimental aspect, most of the stopping studies deal with idealized situations: elemental targets.[1–15] Due to experimental difficulties in preparing and handling compound targets for energy-loss measurements, heavy-ion stopping data in compounds are very limited.[16–20] Most studies have concentrated on the analysis of light ion stopping (H, He, and Li) in compounds composed of light elements. For stopping in a compound, Bragg's rule, which states that the stopping cross sections for individual target elements are additive, is commonly used to determine the stopping power in a compound. The quantitative validity of Bragg's rule for heavy ions in compounds is critical to applications involving ion implantation and irradiation effects in compounds, as well as to ion-beam-based materials analysis techniques, such as Rutherford backscattering spectrometry (RBS), elastic recoil detection analysis (ERDA), particle-induced x-ray emission (PIXE), and nuclear reaction analysis (NRA).

Silicon carbide (SiC) has remarkable physical, chemical, and electronic properties that make it very attractive for high-power, high-temperature, and high-frequency applications in the semiconductor industry.[21–23] Due to exceptional nuclear and mechanical properties,[24] SiC has also been proposed for structural components in harsh nuclear environments, such as in fusion reactors[25] and as cladding material for gas-cooled fission reactor.[26] Accurate values for heavy-ion stopping power in SiC are essential for successful implementation of ion-implantation doping techniques in SiC device fabrication, as well as to performance predictions for SiC-based components and devices in high-radiation environments.

The present paper employs a recently developed approach,[8] which takes advantage of the continuous energy spectra of recoils produced by a high-energy heavy-projectile beam, to determine the transmitted energy loss of recoils in a stopping medium using the time-of-flight (TOF) data from a TOF elastic recoil detection analysis (TOF-ERDA) setup. This approach eliminates much of the error resulting from pulse-height defects that are associated with the Si detector[27,28] and improves the accuracy of stopping power measurements. Validation of the approach has been confirmed in studies of stopping powers for a number of heavy ions in elemental targets of C, Al, and Au.[8,9] In this study, the stopping powers for O, Al, Cr, Mn, Co, and Cu in a self-supporting SiC film are determined. The measured stopping values in SiC are compared with the Bragg additive results calculated from stopping data for C and Si films, which are measured using the same ions and experimental approach. The measured stopping values are also compared with the predictions of the SRIM (stopping and range of ions in matter) code.[29,30]

II. EXPERIMENTAL AND ANALYSIS PROCEDURES

The self-supporting $3C$-SiC films used in this study were fabricated by FLX Micro (Solon, Ohio). Silicon carbide films, approximately 215 nm thick, were deposited on 1.5 cm$\times$1.5 cm silicon substrates, and the silicon was subsequently etched away over a 1 cm$\times$1 cm area, which yielded 1 cm$\times$1 cm$\times$215 nm self-supporting SiC films. The thickness of the film used in this study was determined based on the energy loss of α particles in the film over the energy region from 120 to 600 keV/nucleon, using the same experimental arrangement and the previously measured stopping powers of α particles in SiC.[31] The quality and thickness of the SiC film after completion of the measurements were also characterized by scanning electron microscopy (SEM) and transmission electron microscopy (TEM), and the thickness measurements were consistent with the results obtained from the energy loss of α particles. Based on these measurements, the thickness of the SiC film used in these measurements was 215 ± 6 nm, which corresponds to 69 μg cm^{-2} when the the-

oretical density of 3.21 $g\,cm^{-3}$ for SiC is assumed.

An iodine beam ($^{127}I^{10+}$) with energy of either 44 or 47 MeV was obtained from the Uppsala 5 MV NEC Tandem accelerator and used as the incident projectile beam to produce energetic recoils. The energetic recoils, with a continuous range of energies from a few tens to a few hundreds keV per nucleon, were scattered from elemental bulk targets of Al, Cr, Mn, Co, and Cu into the TOF-ERDA system. A thick SiO_2 film deposited on a Si substrate was used to produce O recoils. The time-of-flight–energy (TOF-E) telescope consisted of two carbon-foil time detectors separated by a 437.5 mm flight length and followed by an ORTEC Si detector. The SiC film was mounted on a push rod, which was reproducibly moved into and out of the recoil path between the second time detector and the Si detector. A more detailed description of the experimental configuration is provided elsewhere.[8,9]

The particle energies (keV) impinging (E_1) and exiting (E_2) the SiC film are measured using the TOF data that have the same response in the Si detector, with and without the stopping film, respectively. The Si detector is used to tag identical energies, with and without the stopping film present, and to screen out the extraneous components. By calibrating the Si detector for each channel over the whole measured energy region, this approach, which takes advantage of the continuous energy spectra, eliminates much of the error associated with pulse-height defects and improves the accuracy of stopping power measurements.[8] The mean stopping power $-\overline{dE/dx}$ ($MeV\,mg^{-1}\,cm^2$) at the mean particle energy $\overline{E}$ (keV/nucleon) for the particles passing through the SiC film is obtained by scaling the energy loss in the film to the film thickness, Δx ($\mu g\,cm^{-2}$), and is well described over the range of energies by a sixth-order polynomial. The expressions for $-\overline{dE/dx}$ and $\overline{E}$ are given by

$$-\frac{\overline{dE}}{dx}=\frac{\overline{(E_1-\Delta E_{foil_{in}})-(E_2-\Delta E_{foil_{out}})}}{\Delta x}=\sum_{i=0}^{6}\kappa_i\overline{E^i} \tag{1}$$

and

$$\overline{E}=\frac{\overline{(E_1+E_2-\Delta E_{foil_{in}}-\Delta E_{foil_{out}})}}{2A}, \tag{2}$$

where κ_i are the polynomial coefficients, and A is the mass number of the particle. The parameters $\Delta E_{foil_{in}}$ and $\Delta E_{foil_{out}}$ are the energy loss of the particles in the carbon foil of the second time detector with and without the stopping film present. These small energy corrections (a few percent of the particle energy) are taken to be the product of the carbon foil thickness (7 $\mu g\,cm^{-2}$) and the stopping powers of the corresponding ions in carbon, which were recently measured using this same procedure.[9] It is worth noting that there is a very low probability that particles passing through the SiC foil will undergo some nuclear stopping and be scattered into the limited solid angle of the Si detector. However, those particles that do undergo nuclear stopping and are registered in the Si detector will be excluded from the analysis by the mass window selected.[31] Thus, the effect of low-probability, nuclear scattering events is negligible. The uncertainty in the film thickness determination, including the thickness variations, surface roughness, and uncertainty in density, is about 3.5%; consequently, the experimental uncertainty of the measured stopping power in this study is less than 4%.

III. BRAGG'S RULE

Stopping power, also known as stopping force,[32] is defined as the rate of energy loss per unit path length, $-\mathrm{d}E/\mathrm{d}x$, by an ion in the target, where the path length can be given in units of length (cm) or areal mass density ($\mu g\,cm^{-2}$). The stopping cross section,[33] ε, is a measure of the mean ion energy loss per atom in each atomic layer and is independent of the mass density ρ ($g\,cm^{-3}$). The conventional unit for stopping cross sections is $eV/(10^{15}\,atoms\,cm^{-2})$, where $10^{15}\,atoms\,cm^{-2}$ is approximately the thickness of one monolayer. If N is the atomic density ($atoms\,cm^{-3}$), the relationship between stopping cross section and stopping power ($MeV\,mg^{-1}\,cm^2$) is given by the expression

$$\varepsilon=\frac{1}{N}\rho\left(-\frac{\mathrm{d}E}{\mathrm{d}x}\right). \tag{3}$$

Particles lose energy either through encounters with electrons or with nuclei of individual atoms. Assuming that each target atom acts independently in the energy loss process, the stopping cross section for a multielement target is simply the sum of the individual elemental cross sections multiplied by the atomic fraction of each element in the compound target.[34] Thus, for a compound A_mB_n, where m and n are the atomic fractions of elements A and B, the stopping cross section $\varepsilon^{A_mB_n}$ for the compound is given by the relationship

$$\varepsilon^{A_mB_n}=m\varepsilon^A+n\varepsilon^B, \tag{4}$$

where ε^A and ε^B are the stopping cross sections for a given ion and energy in elemental targets A and B, and $m+n$ is normalized to unity. This concept was first presented by Bragg and Kleeman[35] in 1905 and is known as Bragg's rule.[16,36]

The accuracy of Bragg's rule is generally limited because the energy loss to electrons in a material depends on the detailed orbital and excitation structure within the material.[37] Any differences in electron behavior between elemental materials and compounds could cause Bragg's rule to become inaccurate.[36] Furthermore, both the chemical and physical states of the medium have been observed to have an effect on the energy loss.[38,39] While the validity of Bragg's rule in SiC has been previously confirmed for 2 MeV ^{4}He ions in SiC,[16] stopping results for ^{4}He at other energies and for heavy ions in SiC have not been reported. The present paper provides a comprehensive study of the validity of the Bragg additivity relationship for heavy ions in the compound SiC over a continuous range of energies.

TABLE I. Polynomial fit parameters for the stopping data over the corresponding energy regions.

Element	O	Al	Cr	Mn	Co	Cu
E_{min} (keV/nucleon)	87	55	37	45	37	40
E_{max} (keV/nucleon)	523	428	330	331	322	295
κ_0	2.2652	5.9599×10^{-1}	-1.8699×10^{-1}	2.4322	-2.0082	4.6793×10^{-2}
κ_1	6.2667×10^{-2}	1.5104×10^{-1}	2.6213×10^{-1}	1.5097×10^{-1}	3.6735×10^{-1}	3.0572×10^{-1}
κ_2	-2.5110×10^{-4}	-1.0658×10^{-3}	-2.1898×10^{-3}	-1.1787×10^{-4}	-4.5007×10^{-3}	-3.6270×10^{-3}
κ_3	4.4898×10^{-7}	5.0858×10^{-6}	1.3557×10^{-5}	-4.1811×10^{-6}	3.8582×10^{-5}	3.3999×10^{-5}
κ_4	-3.0185×10^{-10}	-1.4683×10^{-8}	-5.2177×10^{-8}	2.6920×10^{-8}	-1.8679×10^{-7}	-1.7986×10^{-7}
κ_5	-1.3456×10^{-20}	2.2433×10^{-11}	1.0918×10^{-10}	-6.6697×10^{-11}	4.6075×10^{-10}	4.7766×10^{-10}
κ_6	6.0364×10^{-24}	-1.3848×10^{-14}	-9.4735×10^{-14}	5.8903×10^{-14}	-4.4966×10^{-13}	-4.9765×10^{-13}

IV. RESULTS

The stopping powers of O, Al, Cr, Mn, Co, and Cu in SiC have been measured over the energy range from a few tens to a few hundreds keV/nucleon. The results are summarized in Figs. 1–6, using the fitted trend lines from a sixth-order polynomial regression [Eq. (1)] to represent the experimental data from this study, as described in more detail previously.[8,9] Because no previous experimental data in SiC are available in the literature over the energy regimes of this study, the fitting parameters and the corresponding energy regions are listed in Table I for convenient implementation into other applications. Since the available stopping data from the literature[40] for heavy ions in Si and C are rather scattered and do not always agree within the stated uncertainties, the same experimental approach and analysis method used for SiC have been employed to determine the stopping powers over similar energy regions for amorphous Si films in the present study and previously for amorphous C films.[8,9] These stopping power data for Si and C are used to examine the validity of Bragg's rule. The uncertainties in the Si and C stopping measurement are 4% and 3%, respectively, which are mainly attributed to the thickness determination of the stopping films. Because the stopping powers for ions in C, Si, and SiC (Figs. 1–6) are obtained using the same approaches in the same laboratory, systematic errors in the data are, to a large extent, canceled, which makes these stopping data particularly suitable for investigations of possible deviations from Bragg's rule. In Figs. 1–6, the Bragg stopping powers in SiC, based on applying Bragg's rule to the measured Si and C stopping data, are also included in the plots for direct comparison to the measured stopping powers and to evaluate the validity of the Bragg additivity relationship. Due to the propagation of errors in the Si and C stopping measurements, the uncertainty of the additive Bragg stopping values is 5%.

The computer code SRIM (Refs. 29 and 30) is widely used for calculating stopping powers and ranges of ions in matter. In the past few years, improvements in SRIM have focused on the stopping of relativistic light ions with energies above 1 MeV/nucleon. In 1998, improvements to SRIM were made to account for the Barkas effect and the theoretical stopping of Li ions.[30] More recent efforts have emphasized the stopping of heavy ions at lower energies. In the newly released version SRIM-2003, the average accuracy of the stopping powers is stated to be about 5% overall and 6% for heavy ions. The stopping powers predicted by the two most recent versions SRIM-2003 and SRIM-2000 (Refs. 29 and 30) are also included in Figs. 1–6 for comparison to the measured stopping powers in C, Si, and SiC.

V. DISCUSSIONS

A. Stopping power of O ions

As shown in Fig. 1(a), the measured stopping data for O ions in SiC are in good agreement with SRIM predictions, and a slight improvement of SRIM-2003 over SRIM-2000 is evident at energies lower than 150 keV/nucleon. Good agree-

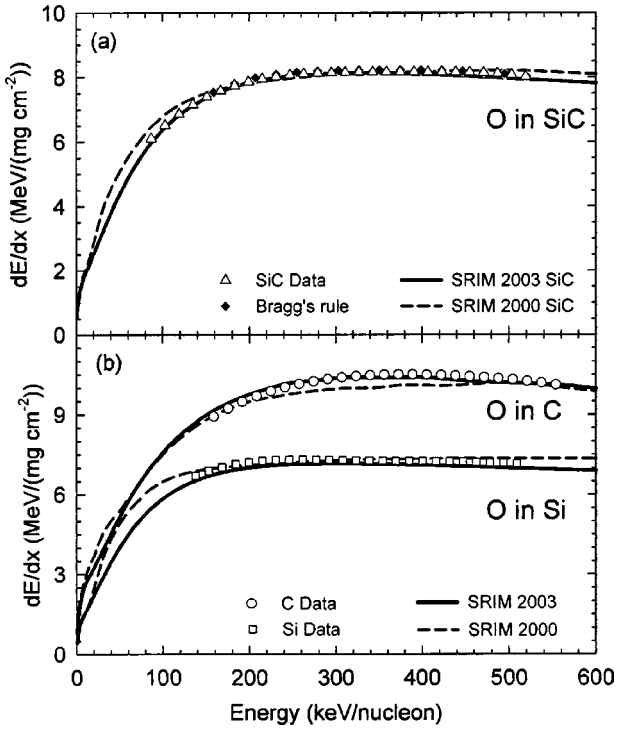

FIG. 1. Measured and predicted (SRIM) stopping powers for O ions in (a) SiC, along with Bragg values, and (b) C and Si.

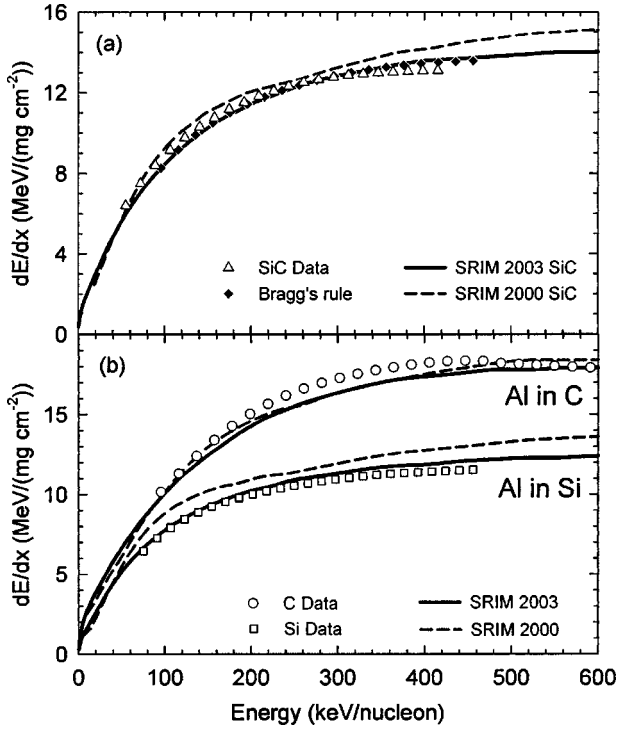

FIG. 2. Measured and predicted (SRIM) stopping powers for Al ions in (a) SiC, along with Bragg values, and (b) C and Si.

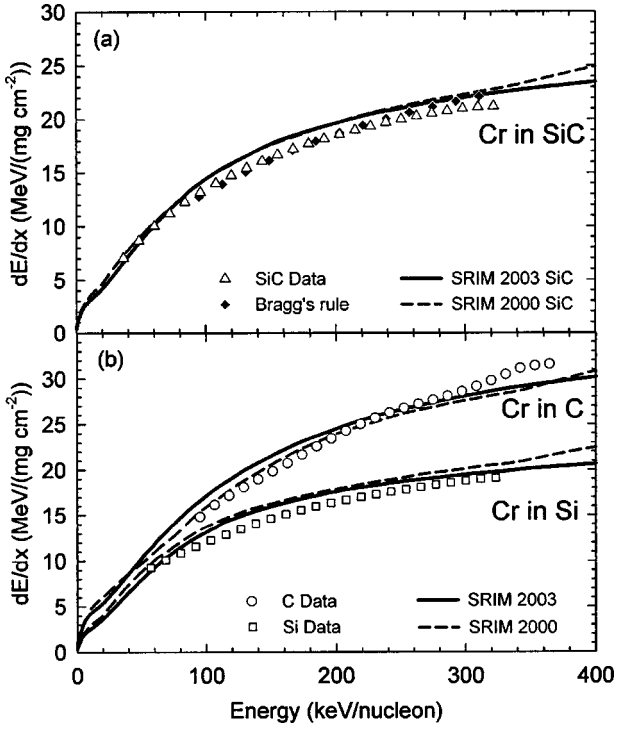

FIG. 3. Measured and predicted (SRIM) stopping powers for Cr ions in (a) SiC, along with Bragg values, and (b) C and Si.

ment of the SRIM-2003 predictions with the experimental data for O ions in C is observed in Fig. 1(b). Both SRIM versions predict similar stopping values for O ions in Si, over the energy range of this study, that are generally consistent with the measured Si stopping data.

The predicted values based on Bragg's rule using the C and Si stopping data overlaps the measured stopping powers in SiC, as shown in Fig. 1(a). The maximum deviation of the Bragg values from the measured stopping data is less than 1%, which is well within the experimental and calculated uncertainties. In stopping studies of light ions in various compounds, several authors[17,36,39–50] have stated that deviations from Bragg's rule appear to be greatest at the stopping peak and disappear at energies above 2.5 MeV/nucleon. As shown in Fig. 1(a), the excellent agreement between the directly measured SiC stopping data and the values predicted by the Bragg additivity law indicate that such deviations are less than 1% in SiC.

B. Stopping power of Al ions

As shown in Fig. 2(a), the measured stopping data for Al ions in SiC are consistent with the predictions from both SRIM versions at lower energies. At higher energies, the SRIM-2003 predictions are in better agreement (within 3%) with the measured data, and SRIM-2000 predicts a higher stopping force at higher energies. For the stopping data in Si shown in Fig. 2(b), SRIM-2003 predicts lower stopping values and is in better agreement with the measured values. Small changes between the two SRIM versions is found for the stopping data in C, as shown in Fig. 2(b), where a higher stopping force is measured in the energy region from 200 keV/nucleon to 450 keV/nucleon.

Although overestimation and underestimation of SRIM-2003 predictions are observed in C and Si, respectively, the SRIM-2003 predictions for SiC overlap with the calculated Bragg values. The measured stopping powers for Al ions in SiC are in good agreement with the Bragg additivity results (within 3%). While a slight difference in energy dependence is observed between the measured stopping power and that calculated from Bragg's rule, the difference is within the experimental and calculated uncertainties.

C. Stopping power of Cr ions

Over the energy region used for Cr ions, there is no significant difference between the SRIM-2003 and SRIM-2000 predictions for Cr stopping in SiC, as shown in Fig. 3(a), and the measured stopping data indicate that the SRIM predictions overestimate the stopping values in SiC by up to 7%. In Fig. 3(b), the measured stopping data for Cr ions in C are lower than the SRIM-2003 predictions and closer to the SRIM-2000 predicted values at lower energies; however, the measured data indicate a tendency for a faster increase of stopping force with increasing particle energy. In the case of Cr stopping in Si, the predicted values from both SRIM versions are higher than the measured data over most of the energy region studied. The Bragg stopping values in SiC, calculated from the experimental stopping data for Cr ions in C and Si, are within 3% of the measured stopping values in SiC, as shown in Fig. 3(a).

D. Stopping power of Mn and Co ions

In the case of Mn stopping in C, as shown in Fig. 4(b), the SRIM-2000 predictions are in better agreement with the mea-

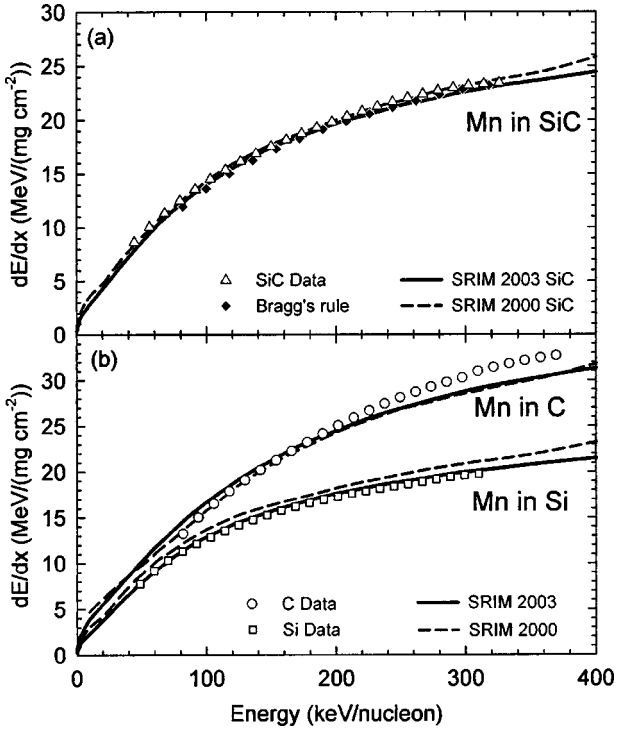

FIG. 4. Measured and predicted (SRIM) stopping powers for Mn ions in (a) SiC, along with Bragg values, and (b) C and Si.

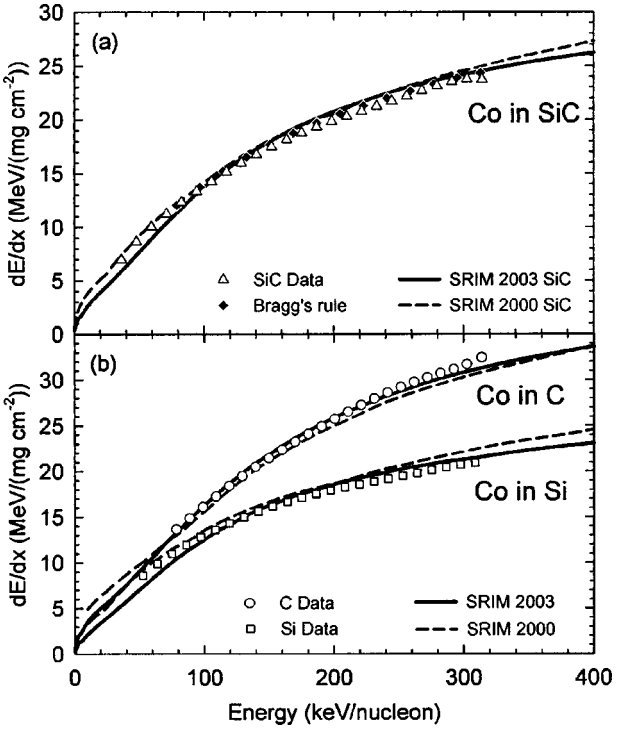

FIG. 5. Measured and predicted (SRIM) stopping powers for Co ions in (a) SiC, along with Bragg values, and (b) C and Si.

sured data than those of SRIM-2003 at lower energies; above energies of 200 keV/nucleon, the SRIM predictions overlap with each other and underestimate the measured values. As compared with SRIM-2000, the stopping values predicted by SRIM-2003 for Mn ions in Si are lower and in better agreement with the experimental data. Despite the underestimation of SRIM predictions for Mn ions in C at higher energies, the measured stopping data for Mn ions in SiC are consistent with the predictions of both SRIM versions within the experimental and SRIM uncertainties, as shown in Fig. 4(a). Bragg's additivity law predicts slightly lower stopping values than the measured stopping powers in SiC. However, the differences ($<4\%$) are less than the combined uncertainties of the stopping measurement and the uncertainty from the Bragg results.

The Co stopping powers exhibit behavior similar to that of Mn. The experimental data and the predicted values from both Bragg's rule and SRIM are shown in Fig. 5. In the energy region between $\sim$100 and 250 keV/nucleon, there is no significant difference between the two SRIM versions in SiC, Si, and C, and the measured data are consistent with the SRIM values. At higher energies ($>$250 keV/nucleon), the experimental data indicate that SRIM-2003 provides better predictions for Co ions in C, Si, and SiC. At energies below 100 keV nucleon, the experimental data for Co ions in Si agree well with the SRIM-2000 version, while the SRIM-2003 underestimates the stopping force by over 10%. In the case of Co ions in SiC, the measured stopping powers lie between the two SRIM predictions. As with the other ions, there is good agreement (within 2%) between the measured stopping powers in SiC and the Bragg values.

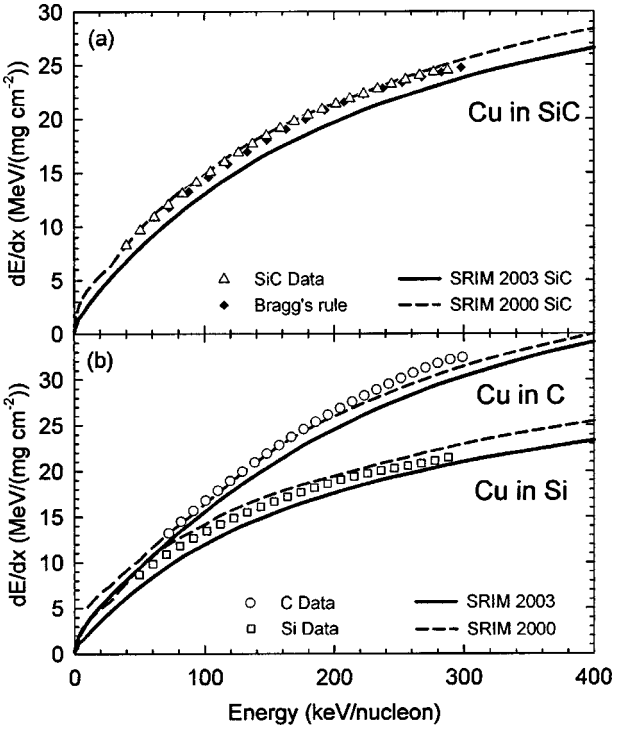

FIG. 6. Measured and predicted (SRIM) stopping powers for Cu ions in (a) SiC, along with Bragg values, and (b) C and Si.

E. Stopping power of Cu ions

Experimental stopping results for Cu ions in SiC, Si, and C and the corresponding predicted stopping values from both SRIM and Bragg's rule are shown in Fig. 6. The measured stopping powers in C are in reasonable agreement with the SRIM-2000 predictions; however, the measured stopping powers in Si lie between the SRIM-2000 and -2003 predictions. In the case of SiC, SRIM-2003 predicts lower stopping values over the entire energy region studied, and both the measured SiC data and the calculated values from Bragg's rule are in good agreement with each other (within 2%) and with the predictions of SRIM-2000. In comparison with the measured SiC stopping data, SRIM-2003 underestimates the stopping force by up to 15% at low energies and by ~4% at higher energies.

F. Validity of Bragg's rule

Because of the different chemical bonding states between constituent elements that are present in compounds and the detailed orbital and excitation structure of the stopping medium, the validity of Bragg's rule has been questioned for years.[36–52] Breakdowns in Bragg's rule by 10% or more have been observed for H, ^{4}He, and Li ions in gases, liquids, and solids.[41–43] Intensive experimental investigations of hydrocarbons[43–47] have also shown that Bragg's rule can lead to enormous discrepancies with experimental data. Compared with hydrocarbons, it has been stated that deviations from Bragg's rule disappear in compounds with heavier elements.[16,36,39] However, large deviations from Bragg's rule have been reported around the stopping maximum in Al_2O_3 and SiO_2,[48,49] which have been attributed to strong chemical effects. Since the maximum ion-target interaction occurs at the stopping peak, where the valence electrons of an atom and the plasma electrons of a solid dominate the stopping process, the effect of chemical binding is strong, which leads to the greatest deviation from Bragg's rule around the stopping peak region.[37,38] Theoretical efforts have attempted to understand the underlying causes of the breakdown. Binary stopping theory[51] has been recently developed to predict the electronic stopping of heavy ions in matter. Incorporating screening, relativistic correction, shell corrections, and projectile excitation and ionization, the binary theory successfully predicts electronic stopping in elemental targets over a wide energy range. Systematic deviations from Bragg's rule are observed when the binary theory is used to predict antiproton stopping in LiF.[52]

In the experimental studies of Si_xC_{1-x}:H ternary compounds,[17,50] the measured stopping cross sections for H and He indicate that the effects of Si-C chemical bonding limit the validity of the additivity law at energies near the stopping cross section maximum. In addition, C-H bonding effects are important over the whole energy range in these compounds, while the influence of Si-H bonds is small in these ternary compounds. In the present study, the validity of Bragg's rule in SiC has been investigated for heavy ions. As discussed above, all deviations from Bragg's rule are well within the experimental uncertainties (±4%). Thus, Bragg's rule appears to be valid to within 4% in SiC for these ions and energy ranges. It is worth noting that the particles in the present study are heavy particles, and the breakdown of Bragg's rule is usually observed for H or He ions.[17,41–50] However, Bragg's rule has been shown to be valid for 2.0 MeV ^{4}He in SiC.[16] Moreover, a recent study of He stopping in SiC (Ref. 31) has also shown that no breakdown in Bragg's rule is found over the energy region from 500 keV to 2.4 MeV, within the limits of the experimental uncertainty (4%). Thus, there appears to be no significant influence of Si-C chemical bonding effects[17,50] in SiC. One of the reasons may be attributed to the high-energy transfers from the particles to the target electrons (~80 eV to few hundreds eV) relative to the very small (few eV) binding energies.

VI. CONCLUSIONS

The stopping powers for heavy ions in the compound SiC have been measured over a continuous range of energies, and the data are described by a sixth-order polynomial for easy implementation in other applications. The measured stopping values in SiC are compared with those predicted by a linear combination of the measured stopping values in Si and C using Bragg's rule. The deviations of the Bragg values from the measured stopping powers in SiC are less than 4%, which are well within the experimental (4%) and Bragg additive (5%) uncertainties in this study. Thus, the validity of Bragg's rule in SiC has been confirmed for all the ions and energy regions considered.

The predictions of SRIM-2000 and SRIM-2003 show varying degrees of agreement with the measured Si and C data. In the case of SiC, both SRIM versions predict stopping powers that are in good agreement with the measured results for O, Mn, and Co ions in SiC. The SRIM-2003 predictions are in better agreement with the measured data for Al stopping in SiC. For Cr stopping in SiC over the energy region from 100 to 250 keV/nucleon, both SRIM versions underestimate the stopping power by up to 7%. In the case of Cu ions in SiC, the SRIM-2000 predictions are consistent with the measured stopping data, and SRIM-2003 overestimates the stopping power by up to 15% at ~100 keV/nucleon, but by only ~4% at 250 keV/nucleon.

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