Magnesium behavior and structural defects in Mg^+ ion implanted silicon carbide

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ABSTRACT

As a candidate material for fusion reactor applications, silicon carbide (SiC) undergoes transmutation reactions under high-energy neutron irradiation with magnesium as the major metallic transmutant; the others include aluminum, beryllium and phosphorus in addition to helium and hydrogen gaseous species. The impact of these transmutants on SiC structural stability is currently unknown. This study uses ion implantation to introduce Mg into SiC. Multiaxial ion-channeling analysis of the as-produced damage state indicates a lower dechanneling yield observed along the $\langle 100 \rangle$ axis. The microstructure of the annealed sample was examined using high-resolution scanning transmission electron microscopy. The results show a high concentration of likely non-faulted tetrahedral voids and possible stacking fault tetrahedra near the damage peak. In addition to lattice distortion, dislocations and intrinsic and extrinsic stacking faults are also observed. Magnesium in 3C-SiC prefers to substitute for Si and it forms precipitates of cubic Mg_2Si and tetragonal MgC_2 . The diffusion coefficient of Mg in 3C-SiC single crystal at 1573 K has been determined to be $3.8 \pm 0.4 \times 10^{-19} \text{ m}^2/\text{sec}$.

1. Introduction

Due to their outstanding mechanical, thermal and chemical properties and other attractive features, silicon carbide composites (SiC/SiC) are candidates for structural material [1] and flow channel insert applications [2] in recent fusion power plant conceptual designs. First-wall/blanket materials will be subjected to high-energy neutron irradiation in the fusion environment, resulting in elemental transmutations and atomic displacements. Recent calculations by Sawan, *et al.* [3] predict that at a high-energy neutron dose of ~ 100 dpa (displacements per atom), there is ~ 0.5 at.% Mg generated in SiC among other transmutants that include 0.15 at.% Al, 0.2 at.% Be, 0.01 at.% P, 2.2 at.% He and 0.84 at.% H through (n,α) , (n,p) and $(n,\beta\gamma)$ nuclear reactions. The impact on the structural stability and thermo-mechanical properties in the presence of Mg impurities and irradiation-induced defects in SiC is currently unknown, but potentially could limit the service lifetime. Possible material degradation needs to be investigated and fully evaluated before informed decisions can be made regarding uses of SiC in fusion energy applications. Irradiation of SiC with a fusion relevant neutron source would be the most desirable way to study microstructural evolution under fusion conditions, but such a neutron source is not currently available. In addition, production of a meaningful fraction of Mg and other elements via transmutation reactions in SiC is currently not feasible utilizing fission reactors because neutron energies are not high enough to cause these reactions. We have employed Mg^+ ion-implantation to introduce Mg into SiC and have examined the microstructure and implanted Mg. Post-implantation thermal annealing has been applied to study damage recovery and Mg diffusion in the implanted SiC. Reported here are the

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data for defect generation and recovery, microstructures, and Mg precipitation and diffusion in as-implanted and annealed SiC. The data will provide a useful basis for modeling and simulation that can advance our understanding of the physical and chemical processes involved.

2. Experimental procedure

Monocrystalline 3C-SiC (001) films on Si, 6H-SiC (0001) wafers, and polycrystalline CVD 3C-SiC disks have been used in this study. 3C-SiC single crystal was chosen because it has the same cubic phase as the candidate SiC/SiC composite and allows for a quantitative study of implantation defects in the crystal structure. CVD 3C-SiC has abundant grain-boundaries that may provide faster paths for Mg diffusion. X-ray diffraction (XRD) analysis shows that the sample has a single cubic phase with an average grain size of ~33 nm. The 6H-SiC wafer obtained from Cree Inc. has very high crystalline quality and is suitable for thermal annealing studies without the constraint imposed by the melting point (1687 K) of Si substrates. Ion implantation of the SiC samples was performed 7° off the surface normal (to minimize or avoid ion-channeling effects in single crystals) with 200 keV Mg⁺ ions to fluences of up to 3.2×10^{16} ions/cm² at 773 K and of 9.6×10^{16} ions/cm² at 673 K. The implantation temperatures were chosen to be above the critical temperature (~550 K for Au²⁺ ion irradiation [4]) to avoid full amorphization in SiC. During ion implantation, surface exfoliation in some areas of the 3C-SiC films (~2 μm thick) on Si occurred at 773 K, but this was not the case at the lower temperature (673 K). The maximum ion fluence (9.6×10^{16} Mg⁺/cm²) applied in this study corresponds to a dose of ~53 dpa and ~6 at.% Mg at the depths of 220 and 280 nm (peak maxima), respectively, according to SRIM simulation [5]. The estimation of the absolute dose could be in error according to a recent report [6], but this will not affect the major data interpretation in this study.

Multiaxial ion channeling along the <100>, <110> and <111> axes based on 2 MeV He⁺ Rutherford backscattering spectrometry (RBS/C) at a scattering angle of 150° was used to analyze the lattice disorder on the Si sublattice in the as-implanted 3C-SiC. Time-of-flight secondary ion mass spectrometry (ToF-SIMS) was applied to obtain the Mg depth profile with 2 keV O₂⁺ ions as a sputtering beam and 25 keV Bi⁺ ions as an analyzing beam. The mass resolution power ($M/\Delta M = 10^3 - 10^4$) of the ToF-SIMS system is high enough to resolve ¹²C₂ (24.0000 u) from ²⁴Mg (23.9850 u) peak. The depth scale for monocrystalline 6H-SiC with a smooth surface was calibrated by measuring the crater depth with a Veeco Dektak 150 stylus profilometer, while that for polycrystalline CVD 3C-SiC samples was determined based on the same sputtering rate in units of nm/(nA·sec) from 6H-SiC. The accuracy for the depth measurement is within a few percent.

The implanted samples were cut into smaller pieces for different thermal treatments. The 6H-SiC and CVD 3C-SiC samples were isochronally (2 hrs) annealed up to 1673 K in flowing Ar gas. All of the annealing performed for this study had a ramp-up time of 2 hrs and a ramp-down rate of 2 °C/min to minimize surface oxidation of the SiC samples and to allow sufficient time for physical/chemical processes to proceed without abrupt quenching. RBS/C analysis was performed to study damage recovery in 6H-SiC, while ToF-SIMS was used to measure diffusion behavior of Mg in all the SiC samples. Isothermal annealing was also conducted for a 3C-SiC single crystal at 1573 K from 30 min to a cumulative time of 720 min. In addition to Ar gas flow, the sample was placed inside a chimney of Zr metal foil that effectively reduced the oxygen partial pressure near the SiC sample. ToF-SIMS was done to measure the Mg depth profile after each anneal. For comparison of defect recovery and Mg behavior, one additional sample was annealed at 1573 K for an uninterrupted time of 720 min. The center region of the annealed sample was found to be covered with flakes of Zr oxide, which were removed using acetone prior to RBS/C and ToF-SIMS measurements. Irremovable layers of Zr oxide were also present near the sample edges. ToF-SIMS measurements for Mg depth profiles in both the center and edge areas were conducted. RBS/C was also used to analyze the lattice disorder in the center area, but the edge area was too small to be probed. The 3C-SiC sample annealed at 1573 K for 720 min in one of the edge areas and

the as-implanted 3C-SiC sample were used for specimen preparation using a focused ion beam (FIB). They have been examined using an FEI Titan 80-300 Cs-corrected scanning electron transmission microscope (STEM) and a new JOEL ARM 200cF Cs-corrected TEM/STEM with an enhanced energy dispersive spectroscopy (EDS) system that was equipped with a silicon drift detector collecting x-rays from samples at a solid angle of up to 0.98 steradians from a detection area of 100 mm². EDS analysis in selected areas and elemental mapping with automatic sample drift corrections were performed.

3. Results and discussion

3.1. Defect accumulation and recovery

2 MeV He⁺ ion RBS/C experiments along the <100>, <110> and <111> axes in (001)-oriented 3C-SiC irradiated at 773 K to 3.2×10^{16} Mg⁺/cm² have been performed and typical <100>-aligned and random spectra are shown in Fig. 1. According to Fig. 1, the minimum yield for the Si sublattice in the unirradiated 3C-SiC is estimated to be 3.6%, indicating that the unirradiated 3C-SiC in the near-surface region of interest has high crystalline quality. The random and channeling spectra from an unirradiated area respectively define the upper and lower levels of the backscattering yields from the completely amorphized SiC material and essentially defect-free SiC single crystal. The depth scale for Si, shown on the top axis of Fig. 1, is converted based on the SRIM-2003 database of random stopping powers for both incoming and outgoing paths. The conversion also adopted a surface energy approximation [7]. There is an apparent damage peak in the irradiated sample. Because RBS does not resolve Mg from Si in this study, the damage peak also includes a Mg contribution. However, the scattering yield from the implanted Mg is small compared to that from displaced Si atoms. As expected, the irradiated SiC is not fully amorphized at the elevated irradiation temperature (773 K), which is above the critical temperature for amorphization. Also expected is that the interaction between the irradiation induced point defects not only enhances simultaneous defect recovery, but also leads to formation of extended defects in the as-implanted SiC, as will be discussed below.

The relative disorder on the Si sublattice is obtained from the RBS/C spectra (Fig. 1) using an iterative procedure [8,9] that is based on dechanneling from the actual disorder present. This procedure extracts the probability of close encounters between the energetic particles and the displaced atoms, which is directly proportional to the defect concentration. The depth profiles of the relative disorder on the Si sublattice in the irradiated 3C-SiC observed along the <100>, <110>, and <111> axes are shown in Fig. 2. The solid lines in the figure are drawn to aid the eye. In the near-surface region, lattice disorder cannot be accurately determined due to the influence of the peak from the surface-atom scattering. From Fig. 2, the damage peak maximum is located at a depth of ~250 nm. The disorder levels on the Si sublattice observed along the <110> and <111> axes are comparable, but they are noticeably higher than along the <100> axis. The data suggest that in addition to randomly distributed Si atoms, there are preferred interstitial defect configurations that are well aligned with the <100> axis. These may include Si-Si interstitial splits along the <100> axis possibly formed during ion implantation. Those aligned Si atoms are effectively shielded by the <100> atomic columns, leading to a reduction in the dechanneling yield. Orientation dependence of Si disorder in 3C-SiC [10] and 6H-SiC [11] irradiated at low and room temperatures was also previously observed. A recent first-principles calculation [12] on Mg-incorporated 3C-SiC reveals that although Si-Mg <100> and C-Mg <100> configurations are unstable, the energetically favored interstitial site of Mg is at the tetrahedral center of C, with the Mg and Si interstitials aligned with the <100> axis. In addition, Si-Si <100> interstitial splits or dumbbells are stable with defect formation energy of 9.48 eV.

Fig. 3 shows 2-hr isochronal recovery of defects in 6H-SiC implanted at 773 K to 2.5×10^{16} Mg⁺/cm² and annealed up to 1573 K, as observed along the <0001> axis. In general, the recovery behavior of 6H-SiC is similar to that of 3C-SiC [10] as the local tetrahedral structure in all SiC polymorphs is identical.

During isochronal annealing, there is observable recovery of defects at 1073 K, where the damage peak becomes lower and narrower. In contrast, the depth profile of the implanted Mg in the annealed sample does not show a noticeable change (see below). Significant recovery of defects was not observed from 1073 to 1273 K. Further annealing at 1473 K leads to an apparent recovery, followed by a more gradual recovery at 1573 K, where the damage peak appears to be broader. The recovery stage between 773 and 1073 K may be mainly a result of dissociation of small interstitial clusters and/or annihilation of point defects due to migrating interstitials. In addition, local lattice relaxation below 1273 K [13] might also contribute to the reduction in the dechanneling yields. The higher-temperature stage between 1273 and 1573 K could be attributed to clustering of vacancies and formation of extended defects. Vacancies become mobile in SiC above ~1273 K, which is evidenced by void swelling [14-17].

3.2. Microstructures

A low-resolution bright-field TEM image (from the ARM 200cF) showing a cross-sectional view of (001)-oriented 3C-SiC film on a Si substrate irradiated with 200 keV Mg⁺ to 9.6×10^{16} ions/cm² at 673 K is given in Fig. 4. There is a clear damage band (dark contrast) evident in the near surface region due to the ion irradiation. The depth scale of the buried band is consistent with the ion-channeling results (Fig. 2). The unirradiated region exhibits {111} stacking faults that originate from the Si (001) and 3C-SiC (001) interface [18] that has a lattice mismatch of ~20% [$a_{\text{SiC}} = 0.436$ nm and $a_{\text{Si}} = 0.543$ nm]. As observed previously, 3C-SiC films on Si are highly faulted on {111} planes with the fault density decreasing quickly away from the interface [19]. The most common defects in the CVD epitaxial 3C-SiC films on Si are {111} stacking faults, micro-twins and anti-phase boundaries. The film thickness in this study is ~2 μ m and the defect concentration is relatively small in the surface region of interest, as indicated by Fig. 4 and the ion-channeling data in Fig. 1.

The high-angle annular dark field (HAADF) STEM micrographs (from the Titan 80-300) in Fig. 5 show microstructures at two different magnifications along the zone axis [110] of the as-irradiated 3C-SiC (Figs. 5a and 5b) and post-annealed at 1573 K for 12 hours in an Ar environment (Figs. 5c and 5d). The image contrast originates from various contributions, including enhanced electron scattering from atomic displacements and lattice strain, presence of Mg and vacancies in atomic columns, and change in the atomic density of possible second-phase precipitates. The irradiated lattice structure near the damage peak (~53 dpa) in the as-irradiated sample exhibits strain contrast and pockets of lattice disorder of a few nanometers in size without evidence of full amorphization. Those types of lattice disorder may be associated with what have been often called black spot defects. The microstructure is slightly different from that produced by Si²⁺ ion irradiation in 3C-SiC to 10 dpa at 1073 K, where the defects are mainly dislocation loops and black spots [20]. The higher irradiation temperature might promote interstitial clustering during ion irradiation. In the sample annealed at 1573 K, stacking faults on the {111} planes are formed, as shown in Fig. 5c. Similar microstructures of Frank faulted loops were observed by Katoh, *et al.* [20] in 3C-SiC irradiated with Si²⁺ ions at 1673 K to 30 dpa. In addition, some dark triangles appear in the microstructure, which could be tetrahedral voids as will be discussed below. Fig. 5d shows a zoomed view of the dark triangles whose sides are parallel to the {111} planes. These triangles are aligned along one direction.

Fig. 6a shows an inverse fast Fourier transformation (IFFT) of a HAADF STEM image (from the ARM 200cF), taken near the damage peak, with a dark triangle in the center of the image for the same irradiated and annealed 3C-SiC as shown in Fig. 5. Identification of the defect nature was attempted under TEM mode, but the result was not conclusive due to the fact that the defect concentration was very high, leading to severe overlapping of the defect features from different depths of the specimen, in addition to the implanted Mg present in the microstructure. The dark triangle is likely to be a tetrahedral void with 4 non-faulted sides on {111} planes and 6 dislocation-free edges along <110> directions based mainly on two observations: the triangles are dark in contrast from HAADF imaging and they are unidirectionally aligned, as seen from Figs. 5c, 5d and 8a from which Fig. 6a was cropped. Similar spatially oriented non-faulted tetrahedral voids have been observed [21] in neutron irradiated 3C-SiC at 1733 K. Normally, one

would expect the opposite orientation of the triangular voids to be also present due to equal probabilities for all $\{111\}$ planes. Kondo *et al.* [21] have attributed the unidirectional distribution to the different surface energies between the Si- and C-terminated polar surfaces. In contrast to the small size (~ 1.2 nm) of the voids created in 3C-SiC with neutron irradiation to a fluence of 5.3×10^{21} n/cm² (~ 5 dpa) at 1573 K [22], the suspected voids formed here at the same temperature are larger (~ 4 nm in size) near the damage peak of the Mg⁺ ion irradiated 3C-SiC (to ~ 53 dpa), as shown in Figs. 6a and 8a. This can be understood because the average void size in the growth regime generally increases with increasing dose (or vacancy concentration) and irradiation temperature (or vacancy mobility). Compared to a more uniform distribution of vacancies from neutron irradiation, ion irradiation produces a high concentration of vacancies in a narrow depth range. For high-purity polycrystalline 3C-SiC irradiated with Si²⁺ ions to 10 dpa, cavities were found [20] to be absent at the irradiation temperature of 1073 K, few at 1273 K and abundant at 1673 K. Although the annealing temperature (1573 K) in this study is near the low end for vacancies to be mobile in SiC [14-17], it appears that formation of well-defined tetrahedral voids occurs during post-implantation annealing of the high-dose Mg⁺ ion irradiated 3C-SiC. To date, there are no other post-irradiation annealing data reported on formation and growth of tetrahedral voids in ion-irradiated 3C-SiC.

In addition, the brighter triangle in the lower right corner of Fig. 6a and more in Fig. 8a resemble those observed for energetically favored stacking fault tetrahedra (SFTs) in low stacking fault energy face centered cubic (fcc) metals and alloys [23,24]. To the authors' best knowledge, there have been no reports of the formation of SFTs in neutron or ion irradiated 3C-SiC. However, there is a similarity between 3C-SiC and fcc alloys in the formation and evolution of defects at similar homologous temperatures [20]. Nucleation and growth of SFTs in fcc metals (such as Cu) and alloys can readily occur during irradiation with fast neutrons or energetic ions [23,25,26]. Post-irradiation thermal annealing of neutron irradiated Cu caused an increase in the SFT size and a decrease in the SFT density [27]. In fact, an SFT represents one of the most common types of vacancy-clustered defects in fcc metals and alloys. In the low angle annular dark-field (LAADF) STEM mode [24], SFTs can be imaged with an enhanced contrast due to dechanneling-enhanced electron scattering. An SFT has 4 faces of $\{111\}$ stacking faults and 6 edges of $\langle 110 \rangle$ stair-rod dislocations. Explanation for the formation of the structure is provided by the Silcox-Hirsch mechanism [28] where an intrinsic Frank dislocation loop is transformed via most energetically favorable Shockley partial-dislocation reactions [29-31] to produce stair-rod dislocations $1/6\langle 110 \rangle$. Once nucleated, SFTs can grow in a supersaturation of vacancies by the climb of jog lines on the $\{111\}$ planes [32]. In general, stable SFTs are favored to form in materials with a low stacking fault energy and a high shear modulus [23]. The density and size of SFTs depend on the level of vacancy supersaturation [27]. 3C-SiC has relatively low stacking fault energy of 0.1 mJ/m² [33] and is expected to have a relatively high shear modulus (Young's modulus for 3C-SiC: 448 GPa). More studies are needed to determine whether or under what irradiation conditions (dose, temperature, etc.) and/or post thermal annealing parameters stable SFTs in 3C-SiC might form. From Fig. 6a, lattice distortion on the $\{111\}$ planes is also observed near the central void that could effectively release the local lattice stress.

Fig. 6b shows bright spots from 3C-SiC ($2\bar{2}0$), $(1\bar{1}1)$, $(\bar{1}11)$, and (002) planes in the fast Fourier transformation (FFT) pattern from a $[110]$ cross-sectional HAADF STEM micrograph (also see Fig. 8b). By filtering the corresponding spots with IFFT, the strain and atomic displacement maps for each plane are obtained, as shown in Figs. 6c, 6d, 6e, and 6f, where the dislocation cores and lattice strain are exhibited in darker contrast. Abundant lattice distortion in the form of bent planes is visible from all directions. There are also dislocations (extra half planes), intrinsic and extrinsic stacking faults, as indicated by arrows. The extra half planes observed on the $(2\bar{2}0)$ plane in Fig. 6c are the cores of perfect edge dislocations, as are normally expected in fcc metals and alloys. In general, the energetically favored $1/2\langle 110 \rangle$ dislocations glide on $\{111\}$ planes via splitting into two glissile Shockley partial dislocations, such as $1/2[110] \rightarrow 1/6[211] + 1/6[12\bar{1}]$. Stacking faults on $\{111\}$ planes are created in a band between the two partial dislocations. Both the extra half planes on $(1\bar{1}1)$ in Fig. 6d and the intrinsic stacking fault on $(\bar{1}11)$ in Fig. 6e are the sessile Frank partial dislocations with Burgers vectors $1/3\langle 111 \rangle$, often

observed in fcc metals and alloys. The extra half planes denoted in Fig. 6d are formed by annihilating a part of a close-packed $\{111\}$ layer of atoms. However, these edge dislocations can move by climb by absorbing mobile point defects. The intrinsic stacking fault in Fig. 6e is the core of a vacancy Frank loop, probably formed by the collapse of a platelet of vacancies during post-irradiation thermal annealing; the platelet of vacancies was created from a local supersaturation of vacancies produced by displacement cascades during ion implantation. Both types of stacking faults in Figs. 6d and 6e may be removed through reaction with a Shockley partial dislocation, such as $1/6[11\bar{2}] + 1/3[111] \rightarrow 1/2[110]$.

Emergence of intrinsic and extrinsic stacking faults on the (002) plane in Fig. 6f is a surprise. The intrinsic and extrinsic stacking faults are a segment of absent and extra planes usually formed by condensation of vacancies and self-interstitials, respectively, on close-packed layers because of their low stacking fault energies. As discussed above, to minimize dislocation energy, two perfect dislocations $1/2[01\bar{1}]$ and $1/2[101]$ can split into Shockley partial dislocations [29]: $1/2[01\bar{1}] \rightarrow 1/6(\bar{1}2\bar{1}) + 1/6[11\bar{2}]$ and $1/2[101] \rightarrow 1/6[2\bar{1}\bar{1}] + 1/6[112]$, and two of the leading partial dislocations may combine at the intersection to form sessile Lomer-Cottrell stair-rod dislocation $1/6\langle 110 \rangle$ via reaction: $1/6(\bar{1}2\bar{1}) + 1/6[2\bar{1}\bar{1}] \rightarrow 1/6[110]$. Alternatively, $1/2[110]$ can dissociate to a Lomer-Cottrell dislocation and two Shockley partial dislocations: $1/2[110] \rightarrow 1/6[110] + 1/6[112] + 1/6[11\bar{2}]$. The intrinsic stacking faults in Fig. 6f might be associated with Lomer-Cottrell dislocations on $\{002\}$ planes. Since stair-rod dislocations are the edges of SFTs, existence of the intrinsic stacking faults is consistent with the possible formation of SFTs in the Mg^+ ion implanted and post-irradiation annealed 3C-SiC, as discussed above. The extrinsic stacking faults observed in Fig. 6f might be associated with local aggregation/precipitation of implanted Mg atoms (see below).

3.3. Mg mapping and precipitation

Fig. 7a shows a low-resolution cross-sectional HAADF STEM image (from the ARM 200cF) for the same irradiated and annealed 3C-SiC as for Figs. 5 and 6 in the near surface region with a scan length of 500 nm. The scan starts near the sample surface. EDS maps for Si, Mg and C over the same region are shown in Figs. 7b, 7c and 7d, respectively. Integrated EDS counts over the region for each element of interest are plotted as a function of the length and are shown in Fig. 7e. As expected, the Mg concentration shows a Gaussian distribution. Interestingly, this same region has a similar profile showing Si depletion while the C distribution appears to be rather uniform. This behavior may suggest that Mg prefers to substitute for Si in the 3C-SiC lattice. A recent first-principles calculation [12] has predicted that Mg at a Si vacant site has a lower formation energy and is more stable than other types of defects. It should be noted that high-resolution EDS mapping generally indicates that there is a change in the relative concentration of Mg, from $\sim 5.5 \text{ Mg}\%$ in a dark area to $4.0 \text{ Mg}\%$ in a brighter area. It remains to be further investigated whether the internal surfaces of the tetrahedral voids have absorbed some Mg atoms.

The same IFFT image as that for Fig. 6a is shown in Fig. 8a without cropping the image. In addition to the expected bright spots from 3C-SiC planes, there are 6 darker spots that are not from cubic SiC, as shown in Fig. 8b. These spots belong to two separate groups of patterns that are labeled with circles and squares and their d-spacings are determined to be 0.3623 and 0.5070 nm, respectively. From all known phases between Mg, Si and C based on the x-ray diffraction database of PDF-4+ (version 2013), the best d-spacing matches suggest that the circled spots are from cubic Mg_2Si [Fm-3m (225), PDF#00-035-0773] with (111) d-spacing of 0.36685 nm and the squared spots from tetragonal MgC_2 [P42/mmc (131), PDF#00-003-0748] with (001) d-spacing of 0.502 nm. The differences between the measured values and the database in both cases are small, $\sim 1\%$, and well within experimental error. Mg in metallic form or other available compound phases in the SiC matrix are not found. Comparison with the known electron diffraction patterns of fcc crystals suggests that Mg_2Si is (110)-oriented, implying that $\text{Mg}_2\text{Si}(110)//3\text{C-SiC}(110)$. The crystal structure of tetragonal MgC_2 was reported by two sources [34,35] to have a similar lattice constant c . The more recent report [35] suggests that MgC_2 has a body-centered tetragonal structure, while the earlier [34] excluded the possibility of face-centered tetragonal lattice. Fig. 8b shows

only 2 diffraction spots from $\{001\}\text{MgC}_2$. It is not certain whether $\{010\}$ or $\{110\}$ spots are also present, yet unresolvable from the bright line in the center due to sample drift. In principle, the MgC_2 orientation can be $\langle 100 \rangle$ or $\langle hk0 \rangle$ with h and k being equal to or greater than 1. Because strong $\{110\}$ diffractions from $\langle 100 \rangle$ -oriented MgC_2 are not visible off the center line and high indices are not probable, the MgC_2 orientation could be $\langle 110 \rangle$, implying that $\text{MgC}_2(110)//3\text{C-SiC}(110)$. Determination of the complete orientation relationships of Mg_2Si and MgC_2 with 3C-SiC requires further studies. However, based on the PDF-4+ database, the minimum lattice mismatch for the lowest possible indices would suggest $\text{Mg}_2\text{Si}(1\bar{1}0)//3\text{C-SiC}(001)$ and $\text{MgC}_2(1\bar{1}2)//3\text{C-SiC}(001)$ with mismatches of 2.9% and 3.3%, respectively, which could be the orientations of the two precipitates within the 3C-SiC lattice.

To locate the precipitates, IFFT operations for the precipitates filtered from specific spots in the FFT pattern in Fig. 8b were conducted. The resulting spot-filtered images for Mg_2Si and MgC_2 are shown in Figs. 8c and 8d, respectively. The brighter areas in Figs. 8c and 8d show where the precipitates are in the microstructure. The two largest second-phase precipitates (~ 5 nm in size) are overlapping in the images and are likely located at different depths in the TEM specimen. It should be noted that because the image intensity is approximately proportional to $Z^{1.7}$ (Z : atomic number) [36], the Mg_2Si precipitate cannot be well resolved from the matrix 3C-SiC in the HAADF image (Fig. 8a) as the former phase has a similar Z and atomic density to the latter (4.863 at./cm³ in Mg_2Si vs. 4.821 Si/cm³ in SiC). However, the atomic density for Mg in MgC_2 is smaller (2.582 Mg/cm³) and a darker contrast would be expected for the precipitate if the specimen thickness is comparable to the precipitate size and other contrast contributors (e.g., lattice strain) are not dominant in the local area. While these conditions might not be perfectly met, there is a relatively darker contrast at the location of the largest MgC_2 precipitate in Fig. 8a, which is consistent with the smaller Mg density.

It should be also noted that the MgC_2 phase is converted to Mg_2C_3 at 673 – 873 K and starts to decompose to elemental Mg and C at 873 K; the decomposition is nearly instantaneous at 1073 K [37]:

Likewise, the Mg_2Si phase decomposes completely at 1073 K [38]:

Second-phase precipitates MgC_2 and Mg_2Si , if any, which were formed in the sample during Mg^+ ion implantation at 673 K would be decomposed during thermal annealing at 1573 K. The observed precipitates in the annealed sample have then nucleated and grown after the thermal annealing step.

3.4. Mg diffusion

The implanted Mg in 6H-SiC wafers, CVD 3C-SiC disks and 3C-SiC films was analyzed using ToF-SIMS. The same analysis was used after each 2-hr isochronal annealing in a sequence up to 1673 K. The peak-area-normalized depth profiles of the implanted Mg in the as-implanted and annealed 6H-SiC and CVD 3C-SiC are shown in Fig. 9. The normalization follows:

$$I_{\text{Mg}}(i) = 100I_{\text{Mg}}(i)/[w\Sigma_{\text{Mg}}(j)] \quad (3)$$

where $I_{\text{Mg}}(i)$ is the Mg intensity at channel number i and w is the channel width. This normalization procedure does not alter FWHM of the peak, but allows for all the ToF-SIMS data measured under different conditions (dwelling time of sputtering beam, beam current, etc.) to be plotted in one figure for comparison. The corresponding peak widths for both 6H-SiC and CVD 3C-SiC are given in Table 1, including half of the FWHM towards the surface (on the left-hand side), the other half towards the bulk, and the entire FWHM. Here, we assume that Mg diffusion is different in the two directions (lattice

disorder towards the surface and mainly pristine crystal towards the bulk) and we ignore any peak shift effects, if any, by repositioning the peak maxima to the original depth (250 nm).

The Mg profile in the as-implanted 6H-SiC in Fig. 9a has a FWHM of 122 nm. Annealing at higher temperatures up to 1473 K does not lead to a significant change in the peak width. The data suggest that the implanted Mg in 6H-SiC is essentially immobile up to high temperature (1473 K). This behavior is similar to Ag in crystalline and amorphized SiC at elevated temperatures [39]. Further isochronal annealing at 1573 for 2 hrs results in observable Mg diffusion to both the surface and bulk, as shown in Fig. 9a, which is again somewhat similar to the Ag behavior in 6H-SiC at elevated temperatures [40]. There is no further Mg diffusion observed at 1673 K. The Mg diffusion is still insignificant with an increase in FWHM from 126 nm at 1473 K to 149 nm at 1673 K (Table 1). The Mg profile in 6H-SiC remains somewhat Gaussian up to 1673 K in this study.

Compared to its monocrystalline 6H-SiC counterpart, the Mg peak width in as-implanted polycrystalline CVD 3C-SiC below 1473 K is systematically larger by 20-30%, as shown in Fig. 9b and Table 1. This peak broadening could be attributed to diffusion-enhancing impurities or excess volume at the grain boundaries that could provide a faster Mg diffusion path at elevated temperatures. Similar interpretations have been proposed for Ag diffusion in CVD SiC [41-43]. Additional contributions from ToF-SIMS measurements due to the surface roughness of the polycrystalline samples cannot be ruled out. The data in Fig. 9b suggest that there might be some modest Mg diffusion starting at 1473 K. More rapid Mg diffusion occurs with an increase in FWHM from 168 nm at 1473 K to 322 nm at 1673 K (Table 1). Furthermore, the Mg atoms tend to diffuse deeper into the polycrystalline host, making its depth profile highly skewed towards the bulk. The half FWHM of the Mg profile at the greater depth is 138 nm vs. 106 nm for the other half towards the surface in CVD 3C-SiC at 1573 K. Similar behavior was also observed for Ag in CVD SiC [40]. This preferred Mg inward diffusion into the undamaged crystal is not yet fully understood, but could be associated with implantation induced effects in the near-surface region through a slower process of Mg trapping and release mechanism [41] or SiC surface oxidation [observed for CVD 3C-SiC at 1573 and 1673 K (data not shown)] that could serve as a possible diffusion barrier. It should be noted that shifts in peak positions in both 6H-SiC and CVD 3C-SiC are observed and all the peaks except for that in CVD 3C-SiC at 1673 K are repositioned to 250 nm. The peak shifts could originate from various contributors, including surface sublimation and oxidation of SiC at the high temperatures.

To determine the diffusion coefficient of Mg in single crystal 3C-SiC, isothermal annealing at 1573 K was conducted from 30 to 720 min. The selected temperature is based on consideration of the observed Mg diffusion at 1573 K (Fig. 9) and the limiting melting point of the substrate Si (1687 K). The results from the isothermal annealing experiment are given in Table 1 and shown in Fig. 10 (solid lines). Mg diffusion is observed at 180 min, below which it is not noticeable. There is significant Mg diffusion into the bulk at 720 min. In addition, an uninterrupted annealing at 1573 K for 720 min was also performed for comparison. A nearly complete recovery of the implantation damage from RBS/C analysis was observed in the center area, where a great majority of the Mg atoms diffuse out of the sample (data not shown). The Mg depth profile in an edge area that had an irremovable layer of Zr oxide (~400 nm thick) formed during the annealing (see section 2 for details) is included in Table 1 and Fig. 10 (dashed line), where depth correction has been made. This annealed sample was used for STEM examinations in this study. The two Mg distributions at 720 min are different in shape, but their HWHM values are comparable. It should be noted that a very small amount of Zr diffused into SiC during the annealing process (data not shown). Some of the Mg peaks have been repositioned and diffusion is assumed to account for the peak broadening. Vacuum annealing of the same implanted samples at 1573 K was also performed at the base pressure of 10^{-5} Torr. When the sample is face down with 3C-SiC film in contact with a Ta metal support, annealing for 180 min leads to a similar Mg diffusion to that at 180 min shown in Fig. 10. However, out-diffusion and complete release of Mg from 3C-SiC occur at 1573 K when the sample is face up in vacuum for only 90 min. Si sublimation is also expected under the annealing conditions because the Si vapor pressure is on the order of 10^{-2} Torr at 1573 K [44].

For a Gaussian distribution $C(x,t) = K[\pi Dt]^{-1/2} \exp(-x^2/4Dt)$ in the dilute limit, the solution to Fick's equation leads to [45]:

$$[W(t)]^2 = 4Dt \ln 2 + [W(0)]^2 \quad (4)$$

where $W(t)$ is the FWHM of the peak at time t . The diffusion coefficient D can be determined through a linear fit to the data in an Arrhenius plot. While experimental profiles are not exactly Gaussian, the Mg peaks in Fig. 10 are assumed to consist of two independent Gaussian portions for the convenience of data analysis. The left-hand side towards the surface has implantation damage decorated with some diffused Zr atoms and the right-hand side is largely a pristine crystal towards the bulk. The peak widths have been determined from Fig. 10 and are given in Table 1. An Arrhenius plot of the data is shown in Fig. 11. Linear fits to the data lead to the diffusion coefficients of Mg in 3C-SiC at 1573 K:

$$\begin{aligned} D_S &= (1.8 \pm 0.1) \times 10^{-19} \text{ m}^2/\text{sec} \text{ (towards surface),} \\ D_B &= (3.8 \pm 0.4) \times 10^{-19} \text{ m}^2/\text{sec} \text{ (towards bulk), and} \\ D_A &= (2.8 \pm 0.2) \times 10^{-19} \text{ m}^2/\text{sec} \text{ (average).} \end{aligned} \quad (5)$$

D_S and D_B are obtained through doubling the half FWHM on the left and right of the peak, respectively, while D_A is obtained from the FWHM of the peak. They have the same order of magnitude, but D_B may be most representative for Mg diffusion in unirradiated 3C-SiC bulk. Compared to Ag in 6H-SiC at 1573 K ($< 10^{-21} \text{ m}^2/\text{sec}$) [40], the D_B value for Mg in 3C-SiC is at least 2 orders of magnitude higher.

4. Conclusions

Monocrystalline 3C-SiC films on Si, 6H-SiC wafers and polycrystalline CVD 3C-SiC disks were implanted with 200 keV Mg^+ ions at 673 and 773 K. Multiaxial RBS/C analysis suggests that there are preferred defect configurations of Si interstitials that are well aligned with the $\langle 100 \rangle$ axis in the as-implanted 3C-SiC at 773 K. The microstructure in the as-implanted sample features pockets of disordered regions of a few nanometers in size without evidence of full amorphization. Two recovery stages were observed between 773 and 1073 K and between 1273 and 1473 K, suggested to be due to migrating interstitial and vacancy processes, respectively, that lead to annihilation of point defects. The annealed 3C-SiC at 1573 K appears to contain a unidirectional distribution of non-faulted tetrahedral voids of a few nanometers in size with possible formation of stacking fault tetrahedra; however, these observations could not be completely established in the current study. There are bent planes, perfect edge dislocations, Frank partial dislocations, possible Lomer-Cottrell stair-rod dislocations, and intrinsic and extrinsic stacking faults in the microstructure. EDS mapping indicates that a higher Mg concentration is correlated to Si deficiency, suggesting that Mg tends to substitute for the Si sublattice in 3C-SiC, consistent with first-principles calculations. There is evidence that precipitates of cubic Mg_2Si and tetragonal MgC_2 are formed in the annealed microstructure of Mg^+ ion implanted and post-irradiation annealed 3C-SiC. Mg diffusion in SiC occurs at 1573 K; more rapid diffusion proceeds in polycrystalline CVD 3C-SiC at that temperature. Mg out-diffusion is strongly suppressed by a surface layer of Zr oxide or in contact with Ta metal. Vacuum annealing of the implanted 3C-SiC at 1573 K for only 90 min results in out-diffusion and a nearly complete Mg release from the SiC sample. A representative diffusion coefficient of Mg in bulk 3C-SiC has been determined to be $(3.8 \pm 0.4) \times 10^{-19} \text{ m}^2/\text{sec}$ at 1573 K.

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Table 1

ToF-SIMS peak widths of Mg in implanted and annealed SiC.

Impl/Ann T (K)	6H-SiC wafer (nm)			CVD 3C-SiC disk (nm)			Ann t (min)	3C-SiC film (nm)		
	Surf	Bulk	FWHM	Surf	Bulk	FWHM		Surf	Bulk	FWHM
2 hr Isochronal							Isothermal at 1573 K			
773 (I)	61	61	122	80	79	159	0	81	71	152
1073 (A)	64	64	128	79	77	156	30	81	71	152
1273 (A)	64	63	127	76	75	151	90	81	71	152
1373 (A)	61	62	123	75	75	150	180	89	82	171
1473 (A)	62	64	126	86	82	168	720	109	127	236
1573 (A)	75	76	151	106	138	244	Uninterrupted at 1573 K			
1673 (A)	75	74	149	163	159	322	720	105	134	239

Note: 1. Implantation was performed using 200 keV Mg⁺ ions at 773 K. 2. The uninterrupted annealing had a Zr oxide layer on top of 3C-SiC film. 3. Surf and Bulk represent the half peak widths towards the surface and bulk, respectively.

Figure Captions

Fig. 1. 2 MeV He⁺ RBS/C spectra along the <100> axis in 3C-SiC irradiated with 200 keV Mg⁺ to 3.2×10^{16} ions/cm² at 773 K. Also included are random and channeling spectra from an unirradiated area.

Fig. 2. Depth profiles of disorder on the Si sublattice in 3C-SiC irradiated with 200 keV Mg⁺ to 3.2×10^{16} ions/cm² at 773 K, as observed along the <100>, <110> and <111> axes.

Fig. 3. Isochronal recovery of defects in 6H-SiC single crystal irradiated with 200 keV Mg⁺ to 2.5×10^{16} ions/cm² at 773 K, observed along the <0001> axis.

Fig. 4. Bright-field TEM micrograph at low resolution for cross-sectional view of 3C-SiC irradiated with 200 keV Mg⁺ to 9.6×10^{16} ions/cm² at 673 K.

Fig. 5. Cs-corrected HAADF STEM micrographs of (a) and (b) cross-sectional view of 3C-SiC irradiated with 200 keV Mg⁺ to 9.6×10^{16} ions/cm² at 673 K, and (c) and (d) post annealed at 1573 K for 12 hours in Ar.

Fig. 6. (a) IFFT of Cs-corrected HAADF STEM micrograph for cross-sectional view of 3C-SiC irradiated with 200 keV Mg⁺ to 9.6×10^{16} ions/cm² at 673 K and post annealed at 1573 K for 12 hours in Ar. (b) FFT of HAADF micrograph. (c), (d), (e) and (f) IFFT of (2 20), (1 11), (1 11) and (002) spots in (b), respectively.

Fig. 7. (a) Cs-corrected HAADF STEM micrograph of 3C-SiC irradiated with 200 keV Mg⁺ to 9.6×10^{16} ions/cm² at 673 K and post annealed at 1573 K for 12 hours in Ar. (b), (c) and (d) EDS maps of Si, Mg and C in the same depth region, and (e) depth profiles of Si, Mg and C from the intensity integration of the respective EDS maps.

Fig. 8. (a) IFFT of Cs-corrected HAADF STEM micrograph for cross-sectional view of 3C-SiC irradiated with 200 keV Mg⁺ to 9.6×10^{16} ions/cm² at 673 K and post annealed at 1573 K for 12 hours in Ar. (b) FFT of the micrograph in (a). (c) and (d) IFFT of precipitates Mg₂Si and MgC₂, respectively.

Fig. 9. ToF-SIMS depth profiles of Mg in (a) 6H-SiC single crystal and (b) CVD 3C-SiC poly crystal implanted with 200 keV Mg⁺ at 773 K and isochronally annealed at temperatures from 1073 to 1673 K for 2 hours at each temperature in Ar environment.

Fig. 10. ToF-SIMS depth profiles of Mg in 3C-SiC single crystal implanted with 200 keV Mg⁺ to 9.6×10^{16} ions/cm² at 673 K and isothermally annealed at 1573 K from 30 to 720 min in Ar environment. Also included is the data (dashed line) from the annealing (1573 K, 720 min in Ar) of another sample implanted under the same conditions.

Fig. 11. Arrhenius fits to the peak widths of the isothermal annealing data at 1573 K. Two separate diffusion regions (towards the surface and bulk) are considered, along with the total FWHM broadening.

Figure 1

Figure 2

Figure 3

Figure 4

Figure 5

Figure 6

Figure 7

Figure 8

Figure 9a

(b) **CVD 3C-SiC**, 200 keV Mg^+ , 773 K
 $3.2 \times 10^{16} \text{ Mg}^+/\text{cm}^2$
 Isochronal Annealing
 2 hrs. in flowing Ar

Normalized Mg Intensity

Depth (nm)

Legend:

- As-implanted
- Ann. 1073 K
- Ann. 1273 K
- Ann. 1373 K
- Ann. 1473 K
- Ann. 1573 K
- Ann. 1673 K

The graph shows the depth profile of Mg intensity for CVD 3C-SiC after 200 keV Mg^+ implantation at 773 K and subsequent isochronal annealing for 2 hours in flowing Ar. The y-axis represents the normalized Mg intensity from 0.0 to 0.8, and the x-axis represents the depth in nanometers from 0 to 800. The 'As-implanted' profile (black line) shows a sharp peak at approximately 250 nm with a maximum intensity of about 0.6. As the annealing temperature increases from 1073 K to 1673 K, the peak intensity decreases and the profile broadens. The 1673 K profile (orange line) is the broadest, with a peak intensity of approximately 0.28 and a significant tail extending to 800 nm.

Figure 10

Figure 11