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Atomically resolved microscopy of ion implantation induced dislocation loops in 4H-SiC

L. H. Karlsson¹, A. Hallén², J. Birch¹, L. Hultman¹ and P.O.Å. Persson^{1*}

¹ Thin Film Physics Division, Department of Physics Chemistry and Biology, Linköping University, SE-581 83 Linköping, Sweden

² KTH Royal Institute of Technology, School of ICT, SE-164 40 Kista, Sweden

*Corresponding author

e-mail: perpe@ifm.liu.se

phone: +46 (0)13 28 1248

Abstract

During high temperature electrical activation of ion-implanted dopant species in SiC, extrinsic dislocation loops are formed on the basal planes of the SiC lattice. Investigations have suggested Si-based loops are caused in accordance with the well-known +1 model. Herein we apply aberration corrected STEM to resolve the atomic structure of these loops. It is shown that the dislocation loops formed during annealing of Al-implanted SiC consist of an extra inserted Si-C bilayer of the (0001) polar sense, which upon insertion into the lattice causes a local extrinsic stacking fault. The +1 model thus needs to be expanded for binary systems.

Introduction

Silicon Carbide (SiC) is a material which is suitable for a large range of applications from nuclear materials to high power electronic devices working at high temperatures and frequencies. Additionally, it is radiation hard and resists corrosive environments which makes SiC a very resilient material. For electronic applications, ion implantation is an important method for doping the SiC structures.[1] The ion implantation is commonly conducted at elevated temperatures to reduce the lattice damage formed in the structure during the process, which, depending on ion mass, dose and dose rate, can entirely amorphize the SiC.[2] After implantation below amorphization levels, the ions typically occupy interstitial sites, where they are electrically inactive.[3] In order to subsequently activate the implanted dopant atoms electrically, the structure is subject to a high temperature annealing process. During this process, the interstitial ions assume substitutional lattice sites to a varying, but high, degree of success, and the damaged lattice structure is mostly restored.[4] However, structural imperfections are still found in the material. Given that the implanted dose has not exceeded an amorphization threshold, the remaining structural imperfections after annealing consist mainly of dislocation loops, residing on the SiC (0001) basal planes.[5-7]

The formation of these loops have been described in accordance with the +1 model,[8] whereby the activated implanted ion assumes the position of a native species and consequently leaves a (+1) native interstitial. Ostwald ripening of native interstitials during the high temperature annealing process eventually produces extrinsic dislocation loops. The kinetics, ion dependence and structural properties of these loops were investigated in previous reports.[5-7] Additionally, the atomic structure of these dislocation loops were also discussed by a number of authors, though no conclusive investigations were presented.[7,9] The fundamental interest in the atomic structure of these loops stem from the consequence that the +1 model and electrical activation to preferential

sites leaves single element interstitials. For example, Al substitutes for Si, which subjects Si interstitials to the ripening process.[4] Ripening of single element interstitials exclusively, poses a fundamentally interesting process in a binary material. For this reason it is of interest to investigate the structure of these dislocation loops by atomically resolved methods.

In recent time, the resolving power of the transmission electron microscope (TEM) has improved substantially as a consequence of the introduction of aberration correctors, which has brought the performance of the best microscopes significantly below the Ångström level. In the following report, we investigate, by atomic resolution imaging, the structure of dislocation loops in Al ion-implanted 4H-SiC.

Experimental details

The material used for implantation was low doped *n*-type epitaxial SiC layers, grown on commercially available SiC substrates of the 4H polytype. The Stopping and Range of Atoms in Matter (SRIM) software package was used to calculate the ion and damage distribution in the SiC. [10]

A density of 3.2 gcm^{-3} and threshold energy, E_d , for atomic displacements of 22 eV for C and 35 eV for Si,[11] and full damage cascades was used in the simulations. It is experimentally verified that about 5-10% of the simulated vacancies and interstitials avoid recombination during the implantation and form more stable defects.[1]

The SiC was implanted by 180 keV ^{27}Al ions with a dose of $1.3 \times 10^{14} \text{ cm}^{-2}$. The sample was implanted at an elevated temperature of 600 °C in order to minimize the implantation damage. No

amorphization is expected, nor was amorphization observed under these implantation conditions. For more information on the implantation conditions, see Ref. 5. The implanted sample was then subject to an annealing process which was performed in an inductively heated Epigress furnace in an Ar- containing atmosphere at 1900 °C for 1 h. A cross-sectional sample for (scanning) (S)TEM was prepared by mechanical thinning and polishing. Finally, low-angle (4°) Argon ion milling, in a Gatan PIPS ion milling system operated at 5 kV, was used to make the samples electron transparent. A final polishing stage using low-energy ions at 1.5 kV was applied to remove the amorphous surface layer formed in the previous stage. TEM and high resolution STEM imaging was performed in the Linköping double aberration corrected and monochromated Titan³ at 300 kV, using a beam current of less than 10 pA, at a beam convergence angle of 30.6 mrad, for a camera length of 115 cm and with a pixel dwell time of 30 ms in order to minimize beam damage.

Results and discussion

The calculated damage and ion distributions of as-implanted SiC are shown in Figure 1. As expected from the lower formation energy, the number of C vacancies is higher than the Si vacancies. Additionally, the total number of vacancies produced is significantly higher than the number of implanted Al ions. However, it is known that about 90% of the initially formed vacancies and interstitials recombine during implantation as a consequence of the elevated implantation temperature.[1] Figure 1 also shows that the damage distribution, i.e. produced vacancies, is more evenly distributed in comparison to the implanted Al ion profile. At a sample depth of 50 nm, the vacancy concentration is approximately half of the maximum, which occurs at ~170 nm into the sample, after which the damage level quickly drops. The ion distribution on the other hand exhibits a well-defined peak at ~220 nm into the sample with a FWHM of ~120 nm, shifted deeper into the sample compared to the broader peak of the damage distribution.

Looking at the cross sectional implanted and annealed sample in Figure 2, the microstructure is revealed. In the figure, dark and continuous bands are seen and these are predominantly present near the surface. These bands originate from varying diffraction conditions as a consequence of local crystal bending and are not related to the implantation. The dislocation loops formed during annealing are visible as an extended region of dark spots centered around 200 – 250 nm below the sample surface, with the majority of the larger spots just below 200 nm. Also it can be seen that there are essentially no dislocation loops between 0-100 nm depth, and very few deeper than 300 nm.

These observations of the defect distribution compares very well with the simulated ion distribution, indicating that the defects are related to the implanted ions as opposed to the damage caused by the implantation. As was shown previously, the dislocation loops are not caused by ripening of the implanted ions, since most of these are activated during the high temperature annealing process, but rather from ripening of the excess interstitials resulting from the +1 mechanism.

A more extensive investigation of the loops by atomically resolved STEM is shown in Figure 3. The hexagonal lattice period is interrupted in the middle of Figure 3a. It is found that the normal stacking sequence of 4H SiC (ABAC ABAC) is locally replaced by ABAC ACBAC ABAC, which seamlessly integrates the fault with the ambient lattice. This sequence requires one native Si-C bilayer to rotate 180° in the plane, and the inserted plane to initiate this rotation. Regretfully, it is not possible to determine which the inserted plane from these two is. Looking closely at the structure image it is possible to differentiate between the bright Si atoms and the slightly darker C

atoms. In some locations, the atomic structure of the Si-C dumbbell is clearly separated (the distance between Si and C in this projection is 1.09 Å), which e.g. can be used for polarity determination and is indicated in Figure 3a) (confirming that the studied wafer is (0001) oriented as opposed to (000-1)). For some of the dumbbells, the separation is more diffuse. This is caused by local strain which is particularly strong in the vicinity of the loops. However, where the dumbbell structure is not entirely resolved, the two atoms together form a pear-shaped feature, which also enables identification of the atomic structure. A schematic structure is overlaid in Figure 3b for clarification. As suggested by this model, the image shows that for each layer of Si atoms, there is an associated layer of C atoms, together forming the Si-C bilayers which constitutes the SiC. However, this observation also holds for each bilayer through the stacking fault, which concludes that the dislocation loop consist of a Si-C bilayer.

This observation is remarkable since it shows that although the dislocation loops are caused by ripening of excess Si atoms in the lattice, a number of C interstitials must also be available to form and stabilize this extrinsic bilayer. There are two potential sources for the C interstitials. I) In C vacancy-interstitial pairs which are formed during growth of the epitaxial layer and during post-implant annealing, and from II) implantation damage.

The formation energy for the C vacancy-interstitial pair was recently established to be 5.1 eV.[12] Hence, a large number of such pairs are formed during high the temperature processing conditions which the sample is subject to, both during growth and during annealing. While the number of generated pairs is still orders of magnitude lower than the peak ion concentration, the migration energy for C interstitials has been found to be about 1 eV (at least for positive and neutral charge states)[13], which enables the C interstitials to migrate several microns during the post-

implantation annealing process. It is therefore plausible that some of the C atoms present in the extrinsic Si-C bilayer are formed both during growth of the epitaxial layers and then later during the high temperature annealing to stabilize the bilayer.

It is further possible that the C interstitials are supplied from the remaining damage distribution. Although as much as 90 % of the damage is restored, as a consequence of the elevated temperature implantation, and that even more damage is healed during the high temperature annealing process, a large number of C interstitials will be present in the vicinity of the growing loops. Also these C interstitials are highly mobile during the post-implantation annealing process and exhibit a source for the growing dislocation loops.

Conclusions

By use of atomically resolved scanning transmission electron microscopy we have shown that the extrinsic dislocation loops formed in implanted and annealed SiC consist of an additional Si-C bilayer, causing a local extrinsic stacking fault. The Si interstitials required to form this bilayer stems from activation of the implanted Al ions according to the +1 model. Additionally, the C atoms which participate in the formation of the bilayer dislocation loop, are suggested to originate from both damage caused during implantation, but also from vacancy-interstitial pairs which form during the high temperature processes. The low migration energy barrier for C interstitials enable diffusion over large distances before the C interstitials are trapped by the growing dislocation loops.

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Figure text

Figure 1. Vacancy and ion concentrations as a function of depth. Note the different scales.

Figure 2. Cross sectional image of the implanted post annealed SiC sample, revealing the defect distribution with respect to depth.

Figure 3. Atomically resolved image of a dislocation loop in a) with indications of the inserted additional plane and of the rotated native plane. The indicated area in a) is further magnified in b) with atomic positions indicated. (White disk = Si; grey disk= C).