The Effects of Damage on Hydrogen-Implant-Induced Thin-Film Separation from Bulk Silicon Carbide

R. B. Gregory, T. A. Wetteroth, and S. R. Wilson
Motorola Inc, Tempe, AZ

O. W. Holland and D. K. Thomas
Oak Ridge National Laboratory, Oak Ridge, TN

Prepared by the
Oak Ridge National Laboratory
Oak Ridge, Tennessee 37831
managed by
LOCKHEED MARTIN ENERGY RESEARCH CORP.
for the
U.S. DEPARTMENT OF ENERGY
under contract DE-AC05-96OR22464

August 1999
ABSTRACT

Exfoliation of SiC by hydrogen implantation and subsequent annealing forms the basis for a thin-film separation process which, when combined with hydrophilic wafer bonding, can be exploited to produce silicon-carbide-on-insulator (SiCOI). SiC thin films produced by this process exhibit unacceptably high resistivity because defects generated by the implant neutralize electrical carriers. Separation occurs because of chemical interaction of hydrogen with dangling bonds within microvoids created by the implant, and physical stresses due to gas-pressure effects during post-implant anneal. Experimental results show that exfoliation of SiC is dependent upon the concentration of implanted hydrogen, but the damage generated by the implant approaches a point when exfoliation is, in fact, retarded. This is attributed to excessive damage at the projected range of the implant which inhibits physical processes of implant-induced cleaving. Damage is controlled independently of hydrogen dosage by elevating the temperature of the SiC during implant in order to promote dynamic annealing. The resulting decrease in damage is thought to promote growth of micro-cracks which form a continuous cleave. Channeled H+ implantation enhances the cleaving process while simultaneously minimizing residual damage within the separated film. It is shown that high-temperature irradiation and channeling each reduces the hydrogen fluence required to affect separation of a thin film and results in a lower concentration of defects. This increases the potential for producing SiCOI which is sufficiently free of defects and, thus, more easily electrically activated.

INTRODUCTION

Hydrogen implantation through an oxide film followed by hydrophilic wafer bonding and a thermal cycle is a process developed to cleave a thin film of silicon-on-insulator (SOI). The process has recently been applied to produce silicon carbide-on-insulator (SiCOI) films for possible use as a wide bandgap semiconductor in power rf and switching devices. SiC thin films separated from bulk material using this process have measured too resistive, a condition attributed to damage in the SiC thin film caused by the hydrogen implant itself. The experiments described in this work are motivated by the desire to understand the implant damage mechanisms in order to make the separation process more efficient and produce defect-free, low-resistivity SiC.

The problem is illustrated in Figure 1 which shows a schematic of the hydrogen-implant-induced separation process and a channeled RBS spectrum of SiCOI (~500 nm) produced by this process. Backscattered counts from Si in the SiCOI (integrated over channels 540-640) measure 824 greater than similarly measured counts from virgin SiC. Calculating density using the RBS data, one measures $1.27 \times 10^{20}$ displaced atoms/cm$^3$. These vacancies have the potential to cause
Figure 1. Schematic diagram of transferred and cleaved SiC (left) and RBS-channeling spectra of the transferred SiC film (right) ... H⁺ implant damage polished off before analysis.

the deactivation of electrical carriers in material with typical doping concentrations of $10^{17}$-$10^{18}$ atoms/cm³.

Previous work (with Si) shows that the process to cleave a thin film by hydrogen implantation followed by a thermal cycle is a combination of hydrogen chemistry and physical processes. The implant results in the formation of platelet-like microvoids which, during subsequent anneal, expand due to gas pressure of excess hydrogen. This links the microvoids into a continuous fracture, cleaving a thin film from the bulk wafer. The use of H⁺-implantation to affect transfer of a thin film from a bulk Si wafer was based upon observations of bubbling and exfoliation of implanted Si wafers after annealing.

The effects of ion-induced damage on the efficiency of the transfer process and its dependence on H⁺ dose are demonstrated by observing exfoliation of SiC following H⁺-implantation and anneal. Means to control damage independently of H dose are demonstrated with elevated-temperature and channeled implantation. It is proposed that channeled implantation generates less residual damage from the surface to at least half the projected range, $1/2R_p$, of the implant simply because crystalline axes of SiC are aligned with the H⁺ beam, decreasing the cross section for ion-solid collisions. The elevated temperature implants affect in-situ, dynamic annealing in order to control H⁺ implant damage in SiC.

**EXPERIMENTS and RESULTS**

**Random vs. Channeled H⁺-implantation**

Experiments to measure damage and exfoliation of SiC as a function of H dose were accomplished using bulk SiC samples, 4H polytype, supplied as research grade material by Cree Research. They were implanted with 60 keV H⁺ to doses ranging from $2.5 \times 10^{16}$ to $10.5 \times 10^{16}$ atoms/cm². Samples were tilted 7° from normal to affect random beam alignment. Additional samples were implanted with the H⁺ beam aligned to [1000] axes to affect channeled implants over the same dose range. Damage analyses were accomplished by Rutherford backscattering (RBS)-channeling using a 2.3 He⁺ ion beam aligned with [1000] axes normal to the surface of the
sample. Backscattered ions were detected at 160° relative to the incident beam using a solid state, surface barrier detector. Samples were then annealed in order to cause exfoliation of thin SiC from the bulk material. The amount of exfoliation was evaluated using optical microscopy.

Figure 2 shows RBS-channeled spectra for three of the samples from the set generated to evaluate the effects of implant damage on exfoliation for random-implanted H⁺. The spectra represent as-implanted samples at room- or ambient-temperature (RT). These spectra show that damage to the SiC at the projected range, \( R_p \), increases with the H⁺ implant dose. The scattering yields near 1/2 \( R_p \) are also progressively greater (as a function of dose) than the yield from the virgin reference sample indicating the presence of ion-induced, residual damage at this location in all the samples. This is possibly due to displaced atoms that either dechannel or directly backscatter the incident H⁺ ions. Analysis by positron annihilation spectroscopy (not shown) indicates the presence of open volume defects, the result of displaced atoms at concentrations below the sensitivity of RBS. Such defectivity may be responsible for deactivating intrinsic carriers in SiC as previously reported for similarly implanted material.⁶

Following RBS characterization, all samples were subjected to 950°C, 15-minute anneals, then optically imaged using a microscope with Nomarski contrast. Figure 3 shows a portion of a series of optical micrographs produced to observe exfoliation of SiC as a function of dose for 60 keV H⁺ implants done at RT. During the 950°C, 15-minute anneal, bubble formation occurs as the H⁺ dose approaches 4.5 \( \times 10^{16}\) cm⁻², as seen in the optical micrograph [Figure 3(a)]. (Samples implanted with small increments of dose between 2.5 \( \times 10^{16}\) and 4.5 \( \times 10^{16}\) cm⁻² revealed that the critical dose to produce exfoliation is very near 4.5 \( \times 10^{16}\) cm⁻².) Evidence for material removal or exfoliation of the 4.5 \( \times 10^{16}\) cm⁻² sample is clearly seen in Figure 3(a) by the appearance of broken bubbles. The amount of exfoliated surface material maximizes near a dose of 5.5 \( \times 10^{16}\) cm⁻² [Figure 3(b)], but at higher doses exfoliation decreases as seen in Figures 3(c) and (d), indicating a retrograde effect of the 60 keV H⁺ implant to doses greater than 5.5 \( \times 10^{16}\) cm⁻².

The information conveyed by the images in Figure 3 is represented graphically in Figure 4 which shows the percentage of area that exfoliates following the 950°C anneal. Two sets of data are graphed, one for the randomly implanted samples and one for channel-implanted samples. One sees that channeled implants shift the onset of exfoliation (as well as maximum exfoliation) to approximately 1 \( \times 10^{16}\) lower dose than the random implants. Furthermore, the maximum

![Figure 2. RBS-channeled spectra for Si in 60 keV H⁺-implanted SiC. Three dosages shown are 4.5 \( \times 10^{16}\), 6.5 \( \times 10^{16}\), and 10.5 \( \times 10^{16}\) cm⁻². Reference spectra include the aligned yield from nonimplanted (virgin) SiC and the randomized yield from an implanted sample.](image-url)
exfoliated area increases from 37% for the random implant (dosed $5.5 \times 10^{16}$/cm$^2$) to 69% for the channeled implant (dosed $4.5 \times 10^{16}$/cm$^2$). The rate of retrograde behavior of exfoliation appears the same for both random and channeled series.

SIMS depth profiles of hydrogen in random and channel-implanted samples are shown in Figure 5. Each of the samples was implanted with 60 keV H$^+$ to $2.0 \times 10^{16}$/cm$^2$, then annealed. The dose was held low enough to prevent exfoliation of the SiC during the anneal. The profiles show that the channeled implant has slightly greater range than the random implant. More significant, though, the retained hydrogen concentration measures almost three times greater for the channel-implanted sample.
Elevated Temperature Implantation

To learn the effects of elevated temperature implantation for controlling H⁺ implant damage, two sets of 4H-SiC samples were implanted with 60 keV H⁺. One set was implanted at room temperature to doses ranging from $3.25 \times 10^{16}$ to $4.5 \times 10^{16}$/cm². Samples of the second set were heated to 600°C during implant to doses ranging from $2.25 \times 10^{16}$ to $8.0 \times 10^{16}$/cm².

Figure 6 shows RBS-channeling spectra for two samples H⁺-implanted to a dose of $2.0 \times 10^{16}$/cm², one implanted at RT and the other with the temperature elevated to 600°C ("hot" implant). It is clear from comparing the scattering yields in the respective samples that the hot implant generated less damage at $R_p$ as well as 1/2 $R_p$. Also evident is a slightly greater $R_p$ for the hot implant. Optical micrographs for the series of hot implants (not shown) indicate the threshold dose for surface exfoliation of SiC during a 950°C anneal is $2.75 \times 10^{16}$/cm².

Optical micrographs shown in Figures 7(a) and (b) compare the surface morphology after annealing for hot and RT implants, respectively. The images show about the same degree of bubbling and exfoliation although the H⁺ implant dose for the RT implant is much higher, $4.5 \times 10^{16}$/cm², compared with $2.75 \times 10^{16}$/cm² for the 600°C implant. Previous work shows that the critical fluence for exfoliation decreases almost linearly with irradiation temperature.⁷

![Figure 6. RBS-channeling spectra comparing damage to SiC following 60 keV H⁺. 2 x 10¹⁶/cm² implants at R.T. and 600°C.](image)

![Figure 7. Nomarski optical micrographs of 60 keV H⁺-implanted SiC showing similar degress of bubbling and exfoliation for (a) 2.75 x 10¹⁶/cm² @ 600°C and (b) 4.5 x 10¹⁶/cm² @ RT.](image)

DISCUSSION and CONCLUSIONS

The dependence of surface exfoliation of SiC on H⁺ dose and the retrograde behavior of exfoliation as damage increases beyond a specific dose supports the following model. Both the hydrogen concentration and the lattice damage affect the degree of exfoliation. Both increase with implantation dose, but damage retards exfoliation. It is clear that more hydrogen available within the lattice will lead to more bubbling and exfoliation, but the role of damage in suppressing the effect is not obvious. As seen from previous work with H⁺-implanted Si, the formation of extended defects (i.e., platelets) is critical to the formation of microcracks within the lattice.⁵ These microcracks and their ability to expand and interconnect yield the large macroscopic regions within the lattice which become separated from the underlying substrate either during
exfoliation or thin-film transfer. One anticipates that substantial lattice damage may inhibit the
formation of such macroscopic regions by hindering or stopping the propagation of the
microcracks and thus preventing them from forming an interconnecting network.

The present work demonstrates the ability to control ion-induced damage independently from
the implant dose by elevating the temperature of a sample to 600°C during the implant in order
to dynamically anneal the SiC and potentially reduce the damage relative to an implant performed
at room temperature. The RBS data show a significant reduction in residual damage (from the
surface to 1/2R_p) for the hot implant. The optical micrographs indicate that implanting hot also
allows a substantial reduction in critical H dose needed for cleaving the thin SiC film, resulting
with further decrease in damage.

Channeling the H⁺ implant dramatically enhances the process of exfoliation. Measurements
of exfoliated area from optical images indicate more robust exfoliation with lower dose relative to
random implantation. The SIMS results suggest less out-diffusion of hydrogen during anneal, but
increased diffusion into the bulk (below R_p) in the channel-implanted sample. This in turn
suggests unique damage morphology at R_p which is not entirely understood.

It appears possible for damage in SiC to reach a concentration great enough to disrupt the
formation of a continuous network of cracks. This conclusion is supported when damage is
controlled independently of hydrogen concentration, either by elevating the temperature of the
SiC during implant, or by channeling the hydrogen, or, quite possibly both. Each of these
methods allows a reduction in critical H⁺ fluence required to affect separation of a thin film and,
therefore, may provide high-quality SiC01 material.

REFERENCES


7. R. B. Gregory, T. A. Wetteroth, S. R. Wilson, O. W. Holland, and D. K. Thomas, to be
published.