Nanosecond Pulsed Laser Processing of Ion Implanted Single Crystal Silicon Carbide Thin Layers

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Abstract

The attractiveness of single crystal SiC in a variety of high power, high voltage, and high temperature device applications such as electric vehicles and jet engines is counteracted by the very high cost of substrates. Precision cutting of multiple micrometre thick SiC layers and transferring them to lower cost substrates would drive the cost down and allow expanding the use of single crystal SiC. In this study, laser beam processing has been utilized to exfoliate thin layers from a surface of single crystal SiC that was prepared with hydrogen and boron ion implantation. The layer thickness of 1 μm has been achieved by ion implantation that formed voids and microcracks under the surface at a layer of 150 nm thick. High energy laser pulses provided the layer removal and its transfer to bonded Si substrate has been shown. Exfoliated surfaces and topography have been evaluated with Scanning Electron Microscopy. Furthermore, thermal modelling of pulse laser irradiation of implanted multi-layer SiC material has been conducted and temperature profiles are obtained at different peak pulse intensity settings to optimize exfoliation process parameters. It was found that laser exfoliation mechanism can be further improved by higher optical absorptance of defect rich layer obtained with boron ion implantation.

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1. Introduction

There is a growing demand for SiC devices in high power and high voltage applications (Zolper & Skowronski 2005), e.g. electric vehicles, high power radio frequency (RF) devices, high temperature sensors (Patil et al. 2010), radiation-resistant ultra-violet (UV) photo-detectors (Monroy et al. 2003) and ionizing radiation detectors (Ivanov et al. 2010), and robust and corrosion-resistant MEMS (Cimalla et al. 2007). There is also growing interest in SiC for mainstream devices (Dimitrijev 2006) where its unique combination of a large bandgap and surface passivation with a thermally grown SiO₂ offers a possibility of making devices with extremely low leakage in the off-state.

| Nomenclature | Description |
|--------------|-------------|
| A            | area of laser beam spot (cm²) |
| C            | specific heat coefficient (J g⁻¹ K⁻¹) |
| E            | pulse energy (mJ) |
| I₀           | initial intensity (Wcm⁻²) |
| I            | peak intensity (Wcm⁻²) |
| K            | thermal conductivity coefficient (W cm⁻¹ K⁻¹) |
| L            | thickness of the SiC layer (µm) |
| T            | Temperature (K) |
| Tₜ           | transmittance (%) |
| R            | reflectivity (-) |
| Rₚ           | depth of implantation (µm) |
| vₛₐₜ         | saturation electron velocity (cm sec⁻¹) |
| Vₜ           | electrical breakdown field (MV cm⁻¹) |
| nᵣ           | refractive index |
| t            | time (ns) |
| z            | distance from surface (cm) |
| α            | absorption coefficient (cm⁻¹) |
| ρ            | density (g cm⁻³) |
| λ            | laser beam wavelength (nm) |
| τ            | pulse duration (ns) |
| Φ            | laser energy density, fluence (J cm⁻²) |

1.1. Properties of single crystalline silicon carbide

High quality single crystalline silicon carbide, and in particular its 4H polytype, is an important electronic material, used in a variety of applications where silicon electronics is unable to satisfy the requirements. 4H-SiC has energy bandgap of 3.26 eV vs. 1.12 eV for Si, has a thermal conductivity coefficient of about 3.3 W cm⁻¹ K⁻¹, more than double that of Si, and the electrical breakdown field Vₜ >2.2 MV cm⁻¹, almost 10 times higher than that in Si (Monroy et al. 2003). The saturation electron velocity vₛₐₜ in 4H-SiC is at 2.2×10⁷ cm sec⁻¹ at least twice that of Si. Another attractive feature of SiC is that high quality thermal SiO₂ can be grown on its surface, the only compound semiconductor that has this attribute. These unique properties have led to the development of superior high voltage and high power devices in 4H-SiC, capable of withstanding several kV, and to handle high power levels at very elevated temperatures. Very high Vₜ allows making devices that are smaller than those made in silicon and with lower specific on-resistance, and this leads to power switching devices that are more compact, energy efficient, and operate at higher frequency than in Si. In addition, SiC circuits designed for high temperature operations are used in harsh environments at up to 600°C, at which temperature reverse junction leakage in Si devices renders them useless.
1.2 Exfoliation and layer splitting of single crystalline silicon carbide

The attractiveness of single crystalline SiC in a variety of device applications is counteracted by the very high cost of substrates. Being able to exfoliate multiple 1 to 10 μm thick layers from one standard thickness SiC wafer and bonding such layers to lower cost substrates, such as silicon or polycrystalline SiC, would drive the material cost down and allow expanding the use of single crystalline SiC. It is known that hydrogen ion (H⁺) implantation can form voids and microcracks under the surface at a depth corresponding roughly to the implantation range and at very high temperature in furnace heat treatment these defects may lead to exfoliation which is known as ion-beam cutting process (Di Ciocio et al. 1998). Nanometric cutting of single crystalline silicon by utilizing ion implantation modification is also demonstrated (Fang et al. 2011).

The conventional ion-beam cutting process and layer transfer utilizing hydrogen ion (H⁺) implantation followed by thermally induced exfoliation is shown in Fig. 1a. The steps involved in this process include: (i) hydrogen implantation into SiC wafer capped with a dielectric layer, e.g. thermally grown SiO₂ (H⁺ dose in the $5 \times 10^{16}$ ions cm$^{-2}$ range) when the depth of implantation is about 1 μm and hydrogen rich layer thickness is about 150 nm, (ii) bonding of SiC wafer to a “handle” wafer, (iii) heat treatment of the two bonded wafers at 800-950°C for exfoliation, and (iv) polishing-after splitting. This layer splitting approach requires high implantation doses and subsequent high temperature furnace annealing. The disadvantages of this approach includes high damage induced to the SiC substrate during H⁺ implantation process and lower processing times due to slow heat treatment processes typically in the order of hours. In this manufacturing process, heat treatment can be done in furnace or by using continuous wave (c.w.) laser heating. In the heat treatment with a c.w. laser case a lower initial ion implant dose can be used. Further heat treatment may follow laser heating/irradiation. Alternatively, ion-cutting process can be done by using laser irradiation. Therefore, in this study, pulsed laser exfoliation method has been proposed to cut and exfoliate thin SiC layers as shown in Fig. 1b. In this case, processing steps involved are: (i) hydrogen or boron ion implantation using into SiC wafer capped with a dielectric layer, e.g. thermally grown SiO₂ (lower ion dose in the $3 \times 10^{16}$ ions cm$^{-2}$ range) when the depth of implantation is about 1 μm and hydrogen or boron rich layer thickness is about 100-150 nm, (ii) bonding of SiC wafer to a “handle” wafer, (iii) laser processing with pulsed nanosecond (5-7 ns) visible (532 nm) irradiation, and (iv) possible polishing-after splitting.
1.3. Ion implantation of silicon carbide

It is reported that laser irradiation of 6H-SiC depicted some peculiarities on ablation mechanism due to liquid phase caused on crystal lattice and subsequent sublimation (Medvid & Lytvyn 2004). Laser ripple patterns on 4H-SiC and 6H-SiC wafers (Gupta et al. 2011).

Pulsed laser assisted exfoliation method utilizes a similar H\(^+\) implantation dose to furnace annealing or B\(^+\) implantation which is not typical in furnace annealing, but exfoliation occurs during laser irradiation or by application of very weak mechanical force at room temperature after the irradiation. In both methods, the implanted SiC sample is bonded to another “handle” substrate before exfoliation is done, and that there are layers of SiO\(_2\) on both surfaces that are bonded together.

In this study, the feasibility of exfoliating a thin crystalline layer of SiC from a surface of single crystalline 4H-SiC by using ion (H\(^+\) or B\(^+\)) implantation to define the layer thickness and high energy nanosecond laser pulses to cut a thin layer and its transfer to another substrate has been shown. It was also demonstrated that the mechanism of laser induced exfoliation is entirely different than the one that is responsible for exfoliation in a furnace. Exfoliation under isothermal conditions present in a furnace is only possible for hydrogen implanted layers (not possible for boron implanted layers) and is uniquely tied to the agglomeration of hydrogen into voids of nanoscale dimensions that lead to microcracks and eventually to layer separation from the main substrate. On the other hand, laser exfoliation occurs under approximately adiabatic conditions, with the fracture in a narrow laser heated zone occurring faster than conductive heat transfer out of this very small and momentarily very hot region.

Therefore, the main objective of this study is to investigate the mechanism of pulsed nanosecond laser based exfoliation and cutting of thin layers of single crystalline SiC for microelectronics applications. In addition, it is aimed for developing a thermal model to understand the rapid temperature rise in relation to pulsed laser processing parameters such as pulse energy and mechanism of transferring SiC thin layer to another substrate.
2. Experimental work

2.1. Specimen preparation

Three-inch-diameter polished single crystalline 4H-SiC wafers (polytype 4H with hexagonal closed packed atomic layers) were n-type doped (nitrogen introduced during crystal growth is the doping impurity) with resistivity of about 0.02 ohm cm. The wafers are intentionally miscut from the C plane by 4 degrees, as this facilitates the growth of epitaxial layers that are usually needed in power device applications. It should be noted here that in hexagonal SiC wafers the two sides of each wafer are not equivalent. Because of the way the carbon and silicon atoms are bonded, one side is always terminated with Si atoms (Si-face), while the other one is always covered with C atoms (C-face).

2.2. Ion implantation of SiC wafers

4H-SiC wafers were thermally oxidized to produce a thin layer of SiO\(_2\) on all surfaces after a standard cleaning procedure. The main reason was that fusion bonding of two semiconductor samples requires that both surfaces be coated with SiO\(_2\). Thermal oxide was grown in a furnace with flowing dry oxygen at 1150°C for 60 minutes which resulted in about 10 nm of SiO\(_2\) on the Si-face, and about 70 nm on the C-face as measured by ellipsometry. It should be noted that ion implantation was done into the Si-face, and most laser irradiations were carried out by shining light on the opposite, C-face.

Implantation parameters were selected similarly to the furnace heat treatment induced exfoliation dosages where the thickness of the removed film corresponds to the depth of implantation (\(R_p\)). At 180 keV acceleration voltage energized hydrogen ions penetrate about 1 \(\mu\)m into the SiC (\(R_p=1.07\mu\)m) under non-channelling conditions. A dose of 6\(\times\)10\(^{16}\) ions cm\(^{-2}\) for hydrogen ion (H\(^+\)) implantation was selected as this dose is very effective in conventional furnace based exfoliation. Some samples were implanted with boron ions (B\(^+\)) at 400 keV energy which provided a penetration of \(R_p=621\) nm into a 4H-SiC crystal and the width of the light absorbing zone of about 140 nm. A dose of 3\(\times\)10\(^{15}\) ions cm\(^{-2}\) was chosen which was expected to provide significant absorption of light at 532 nm wavelength.

2.3. Optical transmittance evaluation of implanted samples

Ion implantation into 4H-SiC crystals created damages in crystalline structure by displacing atoms of the matrix crystal from their original positions resulting in changes in the optical properties that are depended upon the implantation species, ion energy, ion dose, and implantation temperature. Each of these parameters affects the displacement of atoms. The transmittance spectra of a typical n-type unimplanted 4H-SiC wafer, H\(^+\) implanted wafer (6\(\times\)10\(^{16}\) ions cm\(^{-2}\) at 180 keV) and B\(^+\) implanted wafer (3\(\times\)10\(^{15}\) ions cm\(^{-2}\) at 400 keV) are shown in Fig. 2. While H\(^+\) implanted sample absorbs more light than unimplanted sample, B\(^+\) implanted sample absorbs even more light at 532 nm than the typical H\(^+\) implanted samples.
2.4. Laser processing experimental set-up

A high energy Q-switched Nd:YAG Gaussian-like laser beam with a fundamental wavelength of $\lambda=1064$ nm, a maximum pulse energy of $E=415$ mJ, pulse duration of $\tau=5-7$ ns, and $A=0.25$ cm² spot size (at $\lambda=1064$ nm) has been utilized in the experimental set-up. The pulse energy is monitored by using a Molectron EPM1000 with a Coherent J-50MB-YAG energy sensor to adjust optical attenuation to reduce or increase the power in conditioned optical beam at second and third harmonics. For experimenting at those wavelengths, second ($\lambda=532$ nm) and third ($\lambda=355$ nm) harmonic generating units for the Nd:YAG laser system have been utilized (see Fig. 3).

Initially, single pulse laser irradiation experiments on the Si-face (implanted side) and C-face of the ion implanted single crystalline SiC specimens ($10 \times 10$ mm²) have been conducted. It was found that ion implantation greatly increases absorption of light at a $\lambda=532$ nm wavelength and SiC is highly absorptive at 355 nm, which is above the optical bandgap, and irradiation at this wavelength may result in direct ablation and surface damage on the SiC specimen. Irradiation experiments were carried out at a $\lambda=532$ nm wavelength with individual pulses of $E=115, 140,$ and $170$ mJ energy and with a spot size area of $A=0.283$ cm² (at $\lambda=532$ nm) that provided about $\Phi=0.4, 0.5,$ and $0.6$ J cm⁻² energy density, respectively.
3. Thermal modelling of laser irradiation

A thermal model is proposed to compute temperature rise at different layers of implanted SiC wafer (see Fig. 4). A higher than normal temperature rise may result in damaging transferred SiC layer and can cause an undesirable change in crystalline structure of the film.

![Fig. 4. Configuration for H⁺ implanted SiC bonded with Si substrate. (SiC wafer thickness \( \approx 365.8 \mu m \) and SiO₂ layer thickness \( \approx 10 \text{ nm} \), H⁺ and B⁺ implanted layer thickness \( \approx 0.15 \text{ µm} \) and \( 0.14 \text{ µm} \).)](image)

In order to calculate the laser energy absorbed in each layer of the implanted SiC wafer, the absorption coefficients have to be determined. Transmittance expression for refractive index and absorption coefficients can be calculated using Eqs. (1) and (2). Optical transmission at operational wavelength of \( \lambda=532 \text{ nm} \) was utilized from the data given in Fig. 2.

\[
T_r = (1 - R)^2 e^{-\alpha z} / (1 - R^2 e^{-2\alpha z}) \quad (1)
\]
\[
R = (n_1 - n_2 / n_1 + n_2)^2 \quad (2)
\]

where, \( T_r \) is transmittance (\%), \( \alpha \) is absorption coefficient (cm\(^{-1}\)), \( R \) is reflectivity, \( z \) is the distance from surface (cm) and \( n_i \) is refractive index of layer \( i \). Based on the selected wavelength, absorption coefficients for SiC and implanted layer have been determined as given in Table 1. After obtaining the absorption coefficients, the laser peak intensity at each layer can be calculated by applying Beer’s law (Eq. 3) as given in Fig. 5.

\[
I(z) = (1 - R)I_0 e^{-\alpha z} \quad (3)
\]

where, \( I(z) \) = light intensity at depth \( z \) (W cm\(^{-2}\)) and \( I_0 \) = initial light intensity (W cm\(^{-2}\)).

| Absorption coefficient               | at \( \lambda=532 \text{ nm} \) |
|--------------------------------------|----------------------------------|
| 4H-SiC wafer                         | 19.6 cm\(^{-1}\)                |
| H⁺ implanted layer                   | 75,205 cm\(^{-1}\)              |
| B⁺ implanted layer                   | 566,470 cm\(^{-1}\)             |
| Si wafer                             | 7,850 cm\(^{-1}\)               |
| Reflectivity                         | at \( \lambda=532 \text{ nm} \) |
| Air to SiC                           | 0.2086                          |
| SiC to SiO₂                          | 0.072                           |
| SiO₂ to Si                            | 0.209                           |

Table 1. Optical properties of SiC, Si, and SiO₂.
For the temperature calculation, since the goal was to identify the maximum temperature within the SiC wafer, the temperature profile over the irradiated area was out of interest. Therefore, to save the computational time, the problem was simplified by solving it as one-dimensional heat diffusion. Also the following assumptions were made: (i) no heat consumption throughout the process, (ii) use of average beam intensity of a Gaussian beam, (iii) no reflection between implanted layer and SiC, (iv) absorption coefficient is independent of temperature, (v) neglect the heat transfer through SiO$_2$ layer, (vi) the implanted layer has the same thermal properties as SiC.

In order to formulate this thermal modelling problem, the general heat equation with external heat source term was applied as given with Eqs. (4) and (5).

\[
\rho C \left( \frac{\partial T}{\partial t} \right) = k \left( \frac{\partial^2 T}{\partial z^2} \right) + S(z, t) \quad (4)
\]
\[
S(t, z) = (1 - R)I(t)\alpha e^{-\alpha z} \quad (5)
\]

where $T$ is temperature (K), $I(t)$ is power intensity on the surface (W cm$^{-2}$), $z$ is distance from surface (cm), $t$ is time (s), $R$ is reflectivity, $\alpha$ is absorption coefficient (cm$^{-1}$) and SiC properties are (Brink et al. 2009) ; $\rho = 3.22$ density (g cm$^{-3}$), $k = 611/(T-115)$ thermal conductivity (W K$^{-1}$ cm$^{-1}$), $C=0.6+0.77/(222+T)$ specific heat coefficient (J g$^{-1}$ K$^{-1}$). The following condition is applied for solving Eq. (4).

\[
T(t = 0, z) = \frac{\partial T}{\partial z} (t, z = 0) = T(t, z = L) = 300 K \quad (6)
\]

3.1. Effect of energy density on predicted temperature profiles

The effect of laser energy density or fluence on predicted temperature profile on H$^+$ and B$^+$ implanted layers as well as on Si bonding layer has been investigated. The bonded wafer was treated as a combination of many thin layers in which each layer has a difference properties (optical and physical) based on the properties of materials including SiC, H$^+$ implanted SiC, SiO$_2$ and Si as shown in Fig. 4. The heat transfer equation was solved numerically by applying Crank–Nicolson method for solving partial differential equations and temperature rises at the irradiated surface, H$^+$ and B$^+$ implanted layer and at the Si surface of bonded SiC/Si wafers were obtained (Fig. 6).
4. Results on laser assisted exfoliation

Laser irradiation experiments have been done on two different specimen configurations; (i) free SiC and (ii) SiC bonded with Si wafer and clamped. Our typical configuration was to have laser light incident on the polished C-face of a SiC sample, traverse most of the 360 μm thickness of the sample with only a minor absorption loss, and be largely absorbed in the implantation-modified layer. Sudden heating of this thin subsurface layer, modelled through heat diffusion results in a temperature peak to 1636 K (at 0.6 J cm\(^{-2}\)), 1410 K (at 0.5 J cm\(^{-2}\)), 1054 K (at 0.4 J cm\(^{-2}\)) for H\(^+\) implanted layer and 2989 K (at 0.6 J cm\(^{-2}\)), 2368 K (at 0.5 J cm\(^{-2}\)), 1644 K (at 0.4 J cm\(^{-2}\)) for B\(^+\) implanted layer (see Fig. 6). On the Si bonding layer, peak temperature rises predicted were 737 K, 676 K, 575 K for H\(^+\) implanted wafer and 909 K, 768 K, 604 K for B\(^+\) implanted wafer. The sudden (a few nanoseconds time scale) release of energy modifies the properties of the sub-surface film (ion implanted layer) in several ways: (i) At high temperature, thermal vibrations reduce the density of point defects (displacements of Si and C atoms in the crystal lattice by the energetic implanted ions). This thermal annealing causes increased optical transmittance in the irradiated region after the sample cools down. Very large thermal gradients during and immediately after the conclusion of the laser pulse and the associated shear strains caused by the sudden thermal expansion of the absorbing sub-surface layer produce a shock wave; and (ii) hydrogen or boron agglomeration into voids occurs at the elevated temperature. Experiments show that single pulses in an energy range of 0.5 to 0.6 J cm\(^{-2}\) result in exfoliation of thin layers, consistent with the depth defined by ion implantation damage. Examples of the transferred film and of the surface from which a thin layer was removed are shown in Fig. 7. Because of somewhat non-uniform laser beam profile, only irregular fragments of SiC are removed from the substrate and captured by a secondary substrate (a silicon wafer). A thermally induced shock wave could cause laser exfoliation. Several experimental results point us in that direction. It is known that furnace annealing, regardless of temperature, duration, or the implant dose, will not produce exfoliation of a B\(^+\) implanted sample. This confirms that it is the high thermal gradient in a thin subsurface layer that is responsible for the laser induced exfoliation, a mechanism not related to the furnace-based process.

Fig. 6. Predicted temperature rise during single pulse laser exfoliation: (a) H\(^+\) implanted wafer; (b) B\(^+\) implanted wafer.
Since we did use a “flat top” laser intensity profile, where the central area of the irradiated spot would have a uniform energy density and then abruptly drop off at the edge, there is a transition region around each exfoliated region. In this transition region of the intermediate light intensity, there is some modification of the material properties but not enough to produce exfoliation. Instead laser induced heating produces partial annealing of the lattice point defects, which improves the optical transparency of the material. This bleaching effect complicates the effort to obtain large exfoliated areas by raster scanning the laser beam across the surface, with each consecutive pulse slightly overlapping the previous spot. Because of the reduced light absorption in the transition region, some areas are more difficult to exfoliate. Perfectly flat intensity profile in each laser spot would of course eliminate this problem. Alternatively, furnace annealing subsequent to scanned laser irradiation can be used to complete exfoliation in the regions where laser energy was insufficient.

In Fig. 8, we show a sample that was irradiated with a single laser pulse, which produced local exfoliation. Then this sample was heated in a furnace for 60 min at 900 °C. Three regions with different properties can be discerned. Next to the area where a thin film was completely removed by the laser pulse, there is a transition area that was affected by the heat from the laser. During furnace heat treatment, small blisters were created in this region. Further away from the laser spot, the material was not affected at all and in that area during the same furnace annealing large blisters formed and burst open producing flakes, some of which remain visible on the surface.

The reduced blister size in the intermediate area suggests that rapid heating with the laser caused partial loss of hydrogen by outdiffusion, which in turn reduced the blistering effect. Nevertheless, blistering occurs and can be taken advantage of by simply increasing the furnace heating cycle in order to complete the exfoliation in the regions between laser spots. It should be added here that Nomarski interference contrast optical micrographs, as well as SEM images, indicate the formation of microscopic surface corrugations or ripples within the flat surface of the craters left by laser induced exfoliation. An example of concentric ripples is shown in Fig. 9. Such ripples of different periodicities, sometimes but not always related to the laser light wavelength, have been observed in the majority of reports on laser processing of materials, regardless of the laser chosen and the exact nature of the solid target. Fabrication of large area foils suitable for electronic or mechanical devices will require addressing the removal or minimization of these surface non-uniformities.
5. Conclusions

In this study, exfoliation and cutting of thin single crystalline SiC layers has been achieved by using ion implantation to define the film thickness and high energy laser pulses to create a removal mechanism. It was found that the mechanism of laser-induced exfoliation is entirely different than the one that is responsible for exfoliation in a furnace. Exfoliation under isothermal conditions present in a furnace is only possible for hydrogen implanted layers. However, pulsed laser exfoliation occurs under approximately adiabatic conditions, with the fracture in a narrow laser heated zone occurring faster than conductive heat transfer out of this very small and momentarily very hot region.

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