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ABSTRACT

Microwave plasma oxidation under a relatively high pressure (6 kPa) region is developed to rapidly grow a high-quality SiO\(_2\) layer on 4H-SiC, based on a thermodynamic analysis of SiC oxidation. By optimizing the plasma power, an atomically flat interface is achieved, and the interface trap density is lower than that of standard 1300°C thermal-oxidized and 1350°C NO-annealed samples measured by various methods under multiple temperature conditions. Moreover, the oxide breakdown field is higher than 9.3 MV/cm, which is comparable to that of a sample produced by high-temperature thermal oxidation. Particularly, the results of electron energy loss spectroscopy show that the transition layer between 4H-SiC and SiO\(_2\) is lower than 2 nm, indicating that microwave plasma oxidation can greatly suppress the formation of interface defects. The results strongly demonstrate the effectiveness of high-pressure plasma oxidation for SiC.

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INTRODUCTION

Silicon carbide (SiC) is an attractive material currently used in areas requiring high temperature, high power, and high speed. It is related to its properties such as high thermal conductivity, wide bandgap, and high saturated electron drift speed. In addition, SiC is the wide bandgap semiconductor that can form SiO\(_2\) thermal-oxidized films for producing metal oxide semiconductor field effect transistors (MOSFETs). Compared to the oxidation temperature of Si, thermal oxidation of SiC requires a temperature increase of 200°C (i.e., ~1300°C), and the oxidation products and processes of SiC are more complicated. Such thermal oxidation features of SiC lead to more complex interface properties. The interface traps of SiC mainly include carbon clusters, near interface traps and dangling bonds. SiC, especially 4H-SiC transistors, suffers large interface traps, which causes a substantial degradation in device performance; for example, the SiC MOSFETs, through the thermal oxidation method, show a lower electron mobility (30–50 cm\(^2\)/V s) compared to the mobility of bulk SiC (600 cm\(^2\)/V s). To improve the interfacial characteristics of the SiO\(_2\)/SiC structure, extensive studies have been performed by optimizing oxidation and postoxidation annealing processes. Despite these studies, interface trap densities at the SiO\(_2\)/SiC interface are still more than one order of magnitude higher than those for SiO\(_2\)/Si interfaces.

Plasma oxidation, utilizing a highly activated oxygen plasma, is a low-temperature technique used to grow dielectric films on semiconductor surfaces. Early studies preliminarily revealed the equipment principles of plasma oxidation, namely, the in situ monitoring method of oxidation thickness, the relationship between oxidation rate and power/pressure/time, and the influence of external bias. Lucovsky et al. have reported that SiC can be oxidized by remote plasma-assisted oxidation. However, their method has limited applicability because of its extremely low oxidation rate. Satoh et al. reported a rapid plasma oxidation method of SiC with a relatively low pressure (100 mTorr) at a substrate temperature of 200°C. Recently, Kim and Cho reported that the direct plasma-assisted oxidation process can obtain a high quality SiO\(_2\) on SiC by controlling the generation of the SiO\(_x\)C\(_y\) layer. In previous studies by other groups, plasma oxidation of SiC experiments was carried out in a...
low pressure (<100 Pa) environment. In this paper, we developed a high-pressure (6 kPa) microwave plasma oxidation process for forming oxide films on 4H-SiC, which combined plasma oxidation with the thermodynamic conditions of SiC (details can be seen in the supplementary material), for rapidly forming a high-quality oxide layer with good interfacial properties.

**EXPERIMENTAL DETAILS**

The 4H-SiC (0001) wafers with approximately $8 \times 10^{15}$ cm$^{-3}$ doped n-type epitaxial layers were cleaned by using buffered oxide etch (BOE) and then oxidized in different ways. The plasma oxide was grown in a microwave chamber at room temperature and a pressure of 6 kPa for 8 min under a flow of pure (7N) O$_2$ gas and microwave powers of 700 W, 1000 W, and 1500 W. From the observation window, a plasma hemisphere was observed that touched the surface of SiC and was located at the center of the chamber [supplementary material, Fig. S1(b)]. As one reference group, the 1300° C thermal oxide was grown for 30 min in dry O$_2$ atmosphere. The thermal oxide was annealed in ambient NO at 1350° C for 2 h as another reference group. Transmission electron microscopy (TEM), X-ray photoelectron spectroscopy (XPS), and atomic force microscopy (AFM) were carried out to analyze the chemical components and physical structures of the oxide layer stacks (test details can be seen in the supplementary material). For the metal oxide semiconductor (MOS) capacitors, Al electrodes were deposited to form gate [diameter is approximately 213 μm for the capacitance-voltage (C–V) measurement and 112 μm for the time-zero dielectric breakdown (TZDB) measurement; thickness =400 nm] and backside ohmic contacts.

**RESULTS AND DISCUSSION**

Figure 1(a) shows the TEM image of the SiO$_2$/SiC structure after the 1000 W oxygen plasma exposure to n-SiC for 8 min. The thickness of the oxide layer is 25.6 nm and reflects that high-pressure plasma oxidation can achieve an oxidation rate of 3.2 nm/min, which is higher than the rate of the 1300° C thermal oxidation (2 nm/min). It is obvious that the SiO$_2$/SiC interface is clear and flat, and there is no obvious transition layer. Figure 1(b) shows the Si 2p and O 1s XPS spectra of 4H-SiC samples before and after plasma oxidation, and the spectra of the 1300° C thermal oxide sample are used for comparison. All spectra were performed on the upper surface of the oxide. Formation of the SiO$_2$ layer was confirmed with the peaks of the Si 2p spectra at the position of 104.5 eV and the O 1s spectra at 532.6 eV. It should be noted that the peak positions of the plasma-oxidized samples are the same as that of the 1300° C thermal-oxidized sample, eliminating the possible formation of other materials on 4H-SiC during the plasma-oxidation process. The roughness results and the typical AFM image of the 1000 W plasma oxidized sample after etching the SiO$_2$ layer are shown in Fig. 1(c). The surface of the 4H-SiC wafer and the thermal oxidized sample after etching the SiO$_2$ layer are also shown as references. A smooth surface [root-mean-square roughness (rms roughness) = 0.25 nm] achieved by 6 kPa plasma oxidation at 1000 W is slightly higher than that before oxidation (rms roughness = 0.24 nm), while it is lower than that after 1300° C thermal oxidation (rms roughness = 0.42 nm). Meanwhile, the interface of thermal oxidation shows the formation of a nanoisland, which can be explained by the formation of C clusters and/or silicon oxy carbides during oxidation. It is inferred that plasma oxidation may restrain the formation of these substances to obtain an atomically flat interface.

Figure 2(a) shows the bidirectional C–V curves of the MOS structure measured at different frequencies from 1 kHz to 1 MHz at a temperature of 300 K. From the inset of the figure, the hysteresis of the C–V curves, caused by the oxide traps, is well suppressed by plasma oxidation. In particular, the best result at approximately 50 mV is demonstrated by the oxidation at 1000 W. However, a large frequency dispersion is observed in the sample oxidized at 700 W, which is remarkably suppressed by increasing the power to 1000 W, while for the 1500 W sample, the frequency dispersion becomes slightly worse again. This indicates that the microwave power has an apparent influence on the interface trap densities ($D_{it}$). Then, $D_{it}$ of all the samples were quantitatively evaluated by the high (1 MHz)–low (quasistatic) method at 100 K, 200 K, and 300 K, as
FIG. 2. (a) Bidirectional capacitance-voltage characteristics (inset) of the metal-oxide-semiconductor capacitor fabricated by plasma oxidation with powers of 700, 1000, and 1500 W. The measurement was conducted at various frequencies from 1 kHz to 1 MHz at room temperature. The interface trap densities as a function of energy level below the conduction band were evaluated by the high (1 MHz)-low (quasistatic) method (dotted lines) or conductance method (yellow triangles) for the samples (plasma oxidation with different microwave powers, 1300 °C thermal oxidation, and 1350 °C NO annealing). Measurements were conducted at 100 K (b), 200 K (c), and 300 K (d).

shown in Figs. 2(b)–2(d), respectively. As a result, $D_{it}$ of the 1000 W sample is the lowest among all samples. $D_{it}$ of the 1000 W sample was also evaluated by the conductance method. The conductance peaks of the 1000 W sample were obtained at 100 K (supplementary material, Fig. S4), and $D_{it}$ measured by the conductance method was obtained with a value of $\sim 10^{11}$ cm$^{-2}$ eV$^{-1}$ at a position of 0.2 eV below the 4H-SiC conduction band edge, as shown in Fig. 2(b). The results of the conductance method are consistent with the high-low method. Therefore, a low $D_{it}$ can be obtained for 4H-SiC by this oxidation process. Compared to $D_{it}$ of other studies ($10^{11} - 10^{12}$ cm$^{-2}$ eV$^{-1}$ at 0.2 eV), the $D_{it}$ of the 1000 W sample is still very low (supplementary material, Fig. S12 and Table S1). The test details can be seen in the electrical analysis of the supplementary material. The oxide breakdown characteristics were obtained from time-zero dielectric breakdown (TZDB) measurements (supplementary material, Fig. S2). The breakdown electric field of the SiO$_2$ sample from the 1000 W microwave plasma is higher than 9.3 MV/cm, which is comparable to the high-temperature thermal-oxidized sample. These results reveal that this oxidation method can reduce the interface traps and the reliability of the oxide layer may not decrease compared with the thermal oxidation method.

To further evaluate the oxide quality at the interface and adjacent region, scanning transmission electron microscopy (STEM) measurement and electron energy-loss spectroscopy (EELS) measurement were performed at various depths. The core-loss spectra were measured in four regions: at 1 nm, 2 nm, and 4 nm far from the SiO$_2$/4H-SiC interface in the layer of the SiO$_2$ and at 2 nm far from the SiO$_2$/4H-SiC interface in the layer of the 4H-SiC, as shown in Fig. 3(a). Figure 3(b) shows Si$_{L2,3}$ edges of 700 W, 1000 W, and 1500 W samples. The Si$_{L2,3}$ edge onset of SiO$_2$ (bottom spectrum) is at 105 eV. Also, the Si$_{L2,3}$ edge onset shift to that of SiC (highest spectrum) is at about 101 eV with the decrease in oxidation state. The edge onset shift of the 1000 W sample is significantly lower than the 700 W and 1500 W samples at 1 nm from the interface, suggesting that the transition layer (related to SiO$_x$C$_y$) is minimized at 1000 W. It is reported that carbon in the transition layer strongly contributes to $D_{it}$. Therefore, by optimizing the microwave power at 1000 W, lower $D_{it}$ is expected. This is consistent with the $D_{it}$ result in Fig. 2. Moreover, the energy loss near-edge structures (ELNESs) of the measured C$_{K}$ signal provide insight into the configuration of the carbon atoms near the interface and within the SiC layer, as shown in Fig. 3(c). Compared with the spectra of the 700 W case, the ones for 1000 W and 1500 W are different at 2 nm and 4 nm far from the interface, while the spectra for all three samples are identical inside the substrate. Inside the SiC layer, the primary carbon component peak is measured at 292 eV, in close agreement with the previously reported energy of the $\sigma^*$ antibonding orbital. This result is indicative of sp$^3$ hybridized carbon and matches that of 4H-SiC. For the 700 W case, at 1 nm–4 nm from the interface, the presence of $\pi^*$ plateau suggests that sp$^2$ carbon hybridization is present, as shown in Fig. 3(c). It indicates that the transition layer thickness exceeds 4 nm. However, for the 1000 W case, the presence of the $\pi^*$ plateau is only limited in 1 nm–2 nm from the
interface. This is consistent with the results in Fig. 3(b). Therefore, it is verified from EELS that high quality SiO$_2$ with a very thin transition region can be obtained by microwave plasma oxidation with optimized power.

CONCLUSION

In this work, we developed a high-pressure microwave plasma oxidation process for forming oxide films on SiC. Oxide films formed by this method have an amorphous structure, with an atomically flat interface comparable to the initial substrate. $D_0$ of the MOS capacitors fabricated by this method shows a trade-off dependency with the microwave power, and $D_0$ as low as $1 \times 10^{11}$ cm$^{-2}$ eV$^{-1}$ at 0.2 eV below the conduction band of 4H-SiC was achieved by optimizing the power. At the same time, the highest oxide breakdown field can reach more than 9.3 MV/cm.

SUPPLEMENTARY MATERIAL

See the supplementary material for more results on high pressure plasma oxidation analysis, which has been discussed in detail along with the thermodynamic theory, optical emission spectrum (OES), and electrical and material analysis.

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