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Dry Ex Situ Cleaning Processes for $(0001)_{Si}$ 6H-SiC Surfaces

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A completely dry ex situ cleaning process based on UV/O_3 oxidation and HF vapor exposure for removal of residual C and oxide, respectively, from $(0001)_{Si}$ [the silicon-terminated surface of SiC] 6H-SiC surfaces to levels equivalent to or better than conventional wet chemical ex situ processing has been demonstrated. X-ray photoelectron spectroscopy (XPS) of surfaces exposed to UV-generated ozone revealed the formation of carbon and silicon oxides, as indicated by the broad Si-O Si 2p peak at 102.4 eV (full width at half-maximum = 2.1 eV) and a shift in the surface C 1s peak from 283.6 to 284.2 eV, respectively. Evidence for a reduction in the amount of surface C was shown by an increase in the ratio of the SiC C peak to the surface C peak from 0.8 to 2.7 after the UV/O_3 treatment. Removal of the UV/O_3 silicon oxide via exposure to the vapor from a 10:1 buffered HF solution was indicated by the absence (below the XPS detection limit) of the Si-O Si 2p peak at 102.4 eV. However, this last process results in a F-terminated surface.

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The continued enhancement in the electronic properties of SiC devices for high-power applications requires the continued reduction in the densities of line and planar defects.[1,2] Incomplete removal of contamination on surfaces of Si wafers results in an increase in the density of the aforementioned defects in homoepitaxial films from $<10^4/cm^2$ to $>10^{10}/cm^2$. [3-8] This correlates directly with a reduction in device yield.[6] Studies regarding Si_xGe_{1-x} alloy growth on (100)Si have shown that surface defects produced in the Si substrate by residual organic/C contamination act as the preferred sites for misfit dislocation generation.[9] These examples illustrate that surface preparation and cleaning should be equally important to the control of defects in films of SiC and the III-V nitrides grown on (0001) 6H-SiC.

Due to a limited number of studies concerned with ex situ SiC cleaning practices,[10-13] most SiC ex situ wet chemical processing has been based on processes specifically developed for and employed in Si technology.[13,14] Typically, these procedures have consisted of some variation of solvent degreasing, organic contaminant removal using RCA or Piranha cleans, and oxide removal using HF-based solutions.[10-14] It has been assumed that the SiC surfaces behave similarly to Si surfaces in these wet chemicals. In a previous study,[15] we have provided examples of where this assumption fails. The primary difficulty concerns the oxide removal from the SiC surfaces using an HF dip process. In Si technology, oxide removal with a dilute HF etch generates a hydrophobic, hydrogen-terminated surface, stable against oxidation in air for several hours.[16-21] However, we have shown[15] that SiC surfaces are inherently hydrophilic after oxide removal with HF due to a preference for OH termination. The hydrophilic surface allows H_2O and HF to become trapped in micropipes in the SiC wafer which can lead to large concentrations of oxygen and fluorine at the SiC-dielectric interface if not properly outgassed. To produce a hydrophobic surface, passivation or capping layers based on Si and/or fluorocarbons are required.[15]

An alternative to the use of passivation layers to form hydrophobic SiC surfaces would be to develop a completely dry cleaning process. In Si and GaAs technology, dry removal of C contaminants from surfaces using UV/O_3 oxidation has become an alternative to wet chemical processing.[22-30] In this process, UV radiation from a Hg lamp (specifically the 184.9 nm line) is used to photoexcite molecular oxygen (O_2) and generate ozone (O_3). [22] Additionally, the 253.7 nm line of Hg assists in removal of carbon contaminants, as it is adsorbed by most hydrocarbons and excites C–H and C–C bonds.[22] Removal of the UV/O_3 generated oxide is typically achieved by wet chemical processing and/or in situ thermal desorption.[25,26,28,30] However, Iyer et al.[31] have shown that the equilibrium vapor from an HF solution can be alternatively used to remove the oxide from a Si wafer via a dry process. Use of completely dry processing techniques eliminates the need for large quantities of expensive, high-purity chemicals while simultaneously reducing the costs for disposal of these toxic materials.[32-34]

In this research, we have demonstrated for the first time a completely dry cleaning process for $(0001)_{Si}$ 6H-SiC [the silicon-terminated surface of SiC] surfaces which is based on the combined use of UV/O_3 oxidation and HF vapor cleaning. This procedure is equivalent to or better than typical wet chemical processes in terms of residual surface C and oxide contamination levels, as measured by X-ray photoelectron spectroscopy (XPS). The combined UV/O_3-HF vapor treatment eliminates the need for a hydrophobic SiC surface and avoids the use of a deposited passivation layer.

Experimental

As-polished, on-axis, n-type (typically $N_d = 10^{18}/cm^3$) $(0001)_{Si}$ 6H-SiC wafers were used in these experiments. Selection of the wafers for examination was based on previous investigations which showed these surfaces to be terminated with a thin (5-10 Å) contamination layer of C–C, C–F, and Si–F bonded species.[15] Each wafer was first ultrasonically cleaned in the solvents of trichloroethylene, acetone, and methanol for 10 min each. The UV/O_3 exposures were made using a high-intensity Hg lamp positioned $\sim$1 cm from the SiC wafer. To increase the concentration of generated O_3 and, therefore, the oxidation rate, the UV/O_3 box was purged with 1 L/s O_2 during the UV exposure. Further details of this process have been described previously.[22,26] The HF vapor exposures were achieved by positioning the SiC wafer in ambient air $\sim$5 mm from a 10:1 buffered HF solution for times ranging 5-30 min. Condensation of HF on the SiC surface was not observed for the exposures.

Each wafer prepared using these procedures was mounted onto a Mo sample holder, loaded into the load lock of an integrated ultra-high-vacuum system, and subsequently analyzed via XPS, Auger electron spectroscopy (AES), and low-energy electron diffraction (LEED) techniques. Details of this system are given elsewhere.[34] The XPS analyses were performed using an Al anode ($h\nu$ = 1486.6 eV) at 20 mA and 12 kV. The AES spectra were obtained using a beam voltage of 3 keV and an emission current of 1 mA. The LEED was performed using rearview optics, a beam voltage of approximately 115 eV, and an emission current of 1 mA. Calibration of the XPS binding energy scale was performed by measuring the position of the Au $4f_{7/2}$ core level (from a $\sim$1 μm thick Au film) and shifting the spectra such that the peak position occurred at the accepted value of 83.98 eV.

Results

Solvents and UV/O_3.—Figure 1a and b shows the broad XPS spectra of the C 1s core level obtained from (a) a solvent-cleaned $(0001)_{Si}$ 6H-SiC surface followed by (b) a UV/O_3 oxidation treat-

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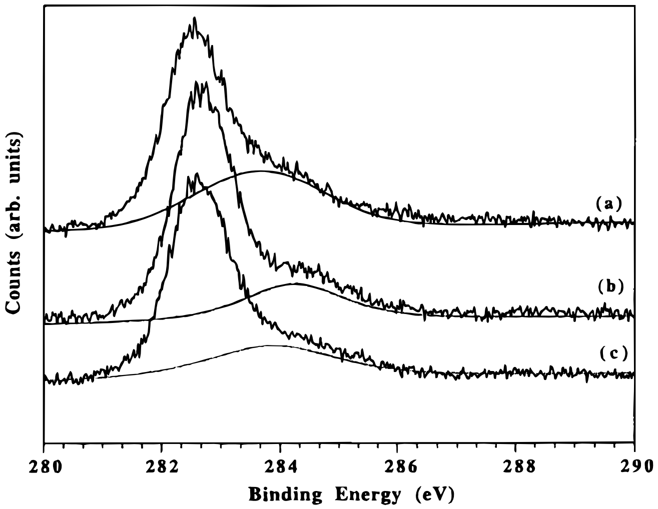

Figure 1. The XPS of the C 1s core level of the (0001)$_{\mathrm{Si}}$ 6H-SiC surface after (a) solvent cleaning, sequentially followed by (b) UV/O$_3$, and (c) HF vapor treatments.

Table I. The XPS core level binding energies (eV) from (0001)$_{\mathrm{Si}}$ 6H-SiC surfaces after various treatments.

Treatment	Si 2p (fwhm)	C 1s (fwhm)	O 1s (fwhm)	F 1s (fwhm)
Solvents	100.4, 1.5	282.5, 1.1 283.6, 2.6 286.0, 4.5	531.6, 2.3	685.4, 1.8 687.2, 2.7
UV/O$_3$	100.5, 1.4 102.4, 2.1	282.6, 1.1 284.2, 2.1	532.1, 2.4	685.9, 1.9
HF vapor	100.5, 1.5	282.6, 1.1 283.8, 2.8	531.8, 2.3	685.8, 1.9

Table II. The XPS core level intensity ratios from (0001)$_{\mathrm{Si}}$ 6H-SiC after various treatments (uncorrected for differences in sensitivity factors). The first ratio represents the C 1s peaks attributed to carbon in SiC and to adventitious or surface carbon.

Treatment	SiC C 1s/surface C 1s	Si 2p/O 1s	Si 2p/F 1s
Solvents	0.8	1.1	5.4
UV/O$_3$	2.7	0.3	10.6
HF Vapor	1.7	1.4	0.5

ment to form an ~20 Å thick oxide layer. Previous analysis of the spectrum[15] obtained after solvent cleaning revealed the presence of three C 1s peaks centered at 282.5, 283.6, and 286.0 eV. The most intense peak at 282.5 is associated with C bonded to Si in SiC. Based on the large full width at half-maximum (fwhm) values of 2.6 and 4.5 eV, the latter two peaks were, respectively, assigned to a mixture of C–C and C–H$_x$ (283.6 eV), and C–F$_x$ (286.0). [35-37] The presence of C–F$_x$ species was further supported by the XPS spectra of the F 1s core level presented in Fig. 2a that showed two F 1s peaks located at 685.4 and 687.2 eV. These peaks were assigned to Si–F [38-40] and C–F [35-37] bonding, respectively. The presence of a thin "fluorocarbon" contamination layer is also indicated by the inability to obtain a LEED pattern from these surfaces at beam energies (E_{p}) < 200 eV.

As a result of the 2 h UV/O$_3$ exposure, the C–F$_x$ C 1s peak disappeared and the C 1s peak associated with C–C and C–H$_x$ shifted from 283.6 to 284.2 eV (see Fig. 1b and Table I). The SiC C 1s peak showed an increase in intensity and shifted by only 0.1 eV to 282.6 eV. In addition, the ratio of the C 1s peak intensities associated with the SiC and the surface carbon (uncorrected for sensitivity factors) increased from 0.8 to 2.7 (see Table II). This result indicates that the UV/O$_3$ process removes the contamination layer via oxidation. The shift and reduction in the C–C and CH$_x$ C 1s peak is consistent with the formation of C–O bonding at the surface and removal of some surface carbon via desorption of CO and CO$_2$. [22,24,37] Removal of the contamination layer was also supported by the complete disappearance (below the XPS detection limit) of the F 1s peak at 687.2 eV after the UV/O$_3$ treatment, as shown in Fig. 2b. Only a slight trace of the lower binding energy F 1s peak was detected, and it was observed to be shifted by 0.5-685.9 eV (see Fig. 2b).

Evidence for the formation of silicon oxides on the SiC surface is shown in the XPS spectra of the Si 2p core level from the (0001)$_{\mathrm{Si}}$ 6H-SiC surface before and after UV/O$_3$ treatment (see Fig. 3a and b). As shown in Fig. 3a, a single Si 2p peak was detected before UV/O$_3$ oxidation. The line shape of this Si 2p peak is asymmetric, suggesting the possibility of a Si–O bonding peak on the higher binding energy (BE) side. Unfortunately, deconvolution of this peak was complicated by the fact that the Si 2p peak is an unresolved doublet (i.e., Si 2p$_{3/2,1/2}$) and fitting a second peak to this spectrum showed only a small peak at 102.2 eV with a fwhm more narrow than the substrate peak (1.1 vs. 1.4 eV). As such, it was not possible to conclusively detect a Si–O peak prior to the UV/O$_3$ exposure.

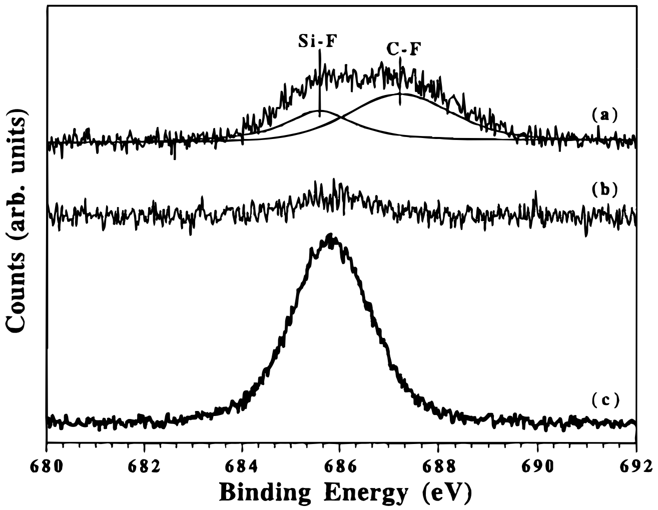

Figure 2. The XPS of the F 1s core level of the (0001)$_{\mathrm{Si}}$ 6H-SiC surface following (a) solvent cleaning, sequentially followed by (b) UV/O$_3$, and (c) HF vapor treatments.

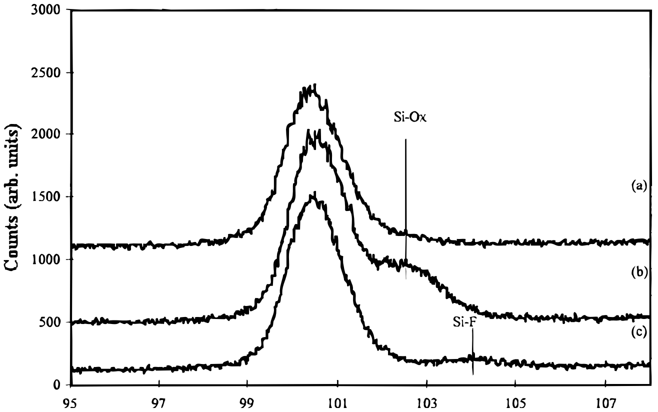

Figure 3. The XPS of the Si 2p core level of the (0001)$_{\mathrm{Si}}$ 6H-SiC surface after (a) solvent cleaning, sequentially followed by (b) UV/O$_3$, and (c) HF vapor treatments.

However, after the UV/O_3 exposure, a broad Si–O peak centered at 102.4 eV (fwhm 2.1 eV) was clearly observed, as shown in Fig. 3b. The width of the Si 2p peak at 102.4 eV indicates that the Si is bonded to oxygen in +2, +3, and +4 oxidation states (i.e., SiO_x).[17,41,42] The degree of attenuation of the Si–C Si 2p peak indicated the thickness of the SiO_x layer to be <20 Å.

HF vapor.—Removal of the thin silicon oxide layer obtained from the UV/O_3 exposure was achieved by a 30 min exposure of the SiC surface to a vapor from a 10:1 buffered HF solution. As shown in Fig. 3c, the higher binding energy Si 2p peak centered at 102.4 eV was not detectable after the exposure. However, in some cases, a small peak at ∼104 eV was detected and attributed to Si–F bonding at the SiC surface.[38-40]

The amount of surface C was monitored from the intensity ratio of the SiC C 1s to the surface carbon C 1s peaks. The ratio was observed to decrease from 2.7 to 1.7 (see Table II) after the HF vapor exposure, indicating an increase in the amount of surface C contamination. The surface C1s peak was likewise observed to shift to 283.8 eV and displayed a large increase in the fwhm from 2.1 to 2.8 eV (see Table I and Fig. 1c). However, the SiC/surface C intensity ratio of 1.7 after the HF vapor treatment was still much larger (i.e., less surface C) than the 0.8 value found after the solvent cleaning process.

Discussion

UV/O_3 oxidation.—The results presented in the previous section showed that exposure of $(0001)_{Si}$ 6H-SiC surfaces to ozone generated by a Hg UV lamp oxidized and removed adventitious and CF_x bonded C from the SiC surface. This resulted in an increase in the SiC/non-SiC carbon ratio from 0.8 to 2.7. This is in agreement with similar studies of UV/O_3 oxidation of Si and GaAs surfaces which have shown a reduction of C contaminants.[22-30] Some adventitious C or surface C remained on the SiC surface, even after a 2 h UV/O_3 treatment. Some of this C was likely due to recontamination of the surface during sample transfer and mounting in a laboratory ambient prior to insertion into vacuum. However, contamination levels of this magnitude are usually not observed from Si wafers cleaned in the same environment. Alternatively, the remaining surface C could be entrapped in the SiO_x layer and/or bonded to both Si and O at the SiC/SiO_x interface. Studies of Fominski et al.[24] and Baunack and Zehe[30] showed incomplete removal of C contaminants from Si surfaces using O_3 generated from a Hg lamp. The former group found it necessary to employ deeper UV radiation from a D_2 lamp and to immerse the wafer in an $O_2/NF_3/H_2$ gas mixture.

The shift in the position of the surface C 1s peak from 283.6 to 284.2 eV with UV/O_3 oxidation is consistent with the oxidation of C–C, CH_x, and CF_x bonds to form CO. For HF-dipped Si wafers, it has been previously determined that residual C contaminants with C 1s peak positions of 284.6, 286.3, and 288.4 eV are composed mostly of C–H_2, C–O, and O–C=O bonded C, respectively, which illustrates the trend to higher binding energies for C–O bonds.[43] Unfortunately, a direct comparison between the surface C 1s peak position and bonding configuration for both Si and SiC surfaces is complicated by the probable existence of 0.5-1.0 eV of band bending at the SiC surface due to surface Fermi level pinning.[10]

A comparison of the UV/O_3 oxidation treatment with other wet chemical processes shows the utility of the technique for removal of at least some of the noncarbidic carbon from the SiC surface. In a previous study,[15] we examined the efficacy of standard wet chemical treatments including RCA SC1 and Piranha etch for the removal of the C surface contamination observed in this study. Table III provides a direct comparison of the SiC C1s/surface C 1s and Si 2p/O 1s intensity ratios for each treatment. The UV/O_3 treatment resulted in the highest SiC C 1s/surface C 1s ratio. Afanas'ev et al.[44] have reported UV/O_3 oxidation to be a useful cleaning or preoxidation procedure prior to thermal oxide growth for p and n-SiC/SiO_2 metal-oxide semiconductor (MOS) structures. They observed that the UV/O_3 preoxidation treatment resulted in a reduction of defects (fast interface states) and a decrease in positive charge at the p-SiC/SiO_2 interface from $2 \times 10^{12}/cm^2$ to 6-8 $\times 10^{11}/cm^2$ relative to that obtained for RCA cleaned SiC samples. They suggested that the reduction in positive charge by the UV/O_3 treatment was due to the removal of C clusters (i.e., C–C bonding) that remain on the SiC surface after the growth of epitaxial layers and which are not removed by RCA cleaning or the thermal oxidation process. This suggestion is supported by our observation that UV/O_3 oxidation removes the non-SiC carbon (C–C, CH_x, and CF_x) from SiC surfaces or results in most of the C being in higher oxidation states.

Table III. Summary of SiC-C 1s/surface C 1s and Si/O intensity ratios from XPS data (uncorrected for differences in sensitivity factors).

Treatment	SiC C1s/surface C 1s	Si 2p/O 1s
Solvents	0.8	1.1
Piranha	1.1	0.9
RCA SC1	2.2	1.0
Aqua regia	1.2	1.2
UV/O_3	2.7	0.3
HF vapor	1.7	1.4

It is important to emphasize the ability of a room temperature UV/O_3 treatment to grow or to form thin SiO_x layers (<20 Å) on SiC. In a separate study,[15] we investigated the ability to oxidize SiC surfaces using other wet chemical treatments commonly employed to form passivating oxides on silicon. As shown in Table III, the Si 2p(Si–C)/O 1s intensity ratio of 0.3 resulting after a UV/O_3 exposure is much lower than the ∼1.0 ratio obtained after wet chemical treatments such as boiling aqua regia or RCA SC1. The Si 2p(Si–C)/O 1s intensity ratio after the RCA SC1 clean is not significantly different from the Si 2p(Si–C)/O 1s ratio of 1.1 observed for solvent-cleaned SiC surfaces. This observation is consistent with the recognized inability of these acids to etch SiC. Therefore, the ability of UV/O_3 to grow a thin (10-20 Å) passivating oxide is an added benefit over conventional wet chemical processing.

HF vapor.—Figure 3b and c shows that the vapor from an HF solution can be used to effectively remove thin silicon oxide layers from SiC surfaces. The latter figure also shows that the higher BE Si 2p peak at 102.4 eV is below the detection limits of the XPS system; however, some O remained on the SiC surface, as indicated by the presence of a small O 1s signal (see Table II) (probably in the form of suboxides or hydroxides of Si and C, i.e., C_3-Si-O(H) and Si_3-C-O). The data in Table III shows that the resulting Si/O XPS intensity ratio after the vapor treatment increased from 0.3 to 1.4. The Si/O intensity ratio of 1.4 compares well with the value of 1.3 obtained from a SiC surface after removal of a thermal oxide using a 10:1 HF dip.[15] These results indicate that HF vapor exposure is as effective as an HF dip in removing surface silicon oxides from SiC surfaces. However, the silicon oxide etch rates for HF vapor and an HF dip are substantially different. In the former, a 30 min exposure was required to remove only 10-20 Å of surface oxide resulting from a UV/O_3 treatment, whereas in the latter case, only 10 min were required to remove 1000 Å of thermal oxide. Correspondingly, the amount of F on the surface was also observed to increase significantly with the HF exposure (see Fig. 2c). Prior to the HF vapor treatment, the Si/F ratio was 10.6; after the exposure, this ratio decreased to 0.56 (see Table II). The binding energy of the F 1s was not observed to shift but remained centered at 685.9 eV, which suggests that F is bonded only to Si atoms at the surface.

Unfortunately, the SiC C 1s/surface C 1s intensity ratio was observed to decrease from 2.7 to 1.7 after the HF vapor treatment. Some of this increased surface carbon may be attributable to the ambient exposure during and after the vapor treatment. An in situ HF vapor exposure, however, could eliminate this recontamination. Takayuki et al.[25] has previously demonstrated the removal of native oxides on (001)Si using photoexcited fluorine gas. Our results suggest that an in situ HF vapor exposure should work as well.

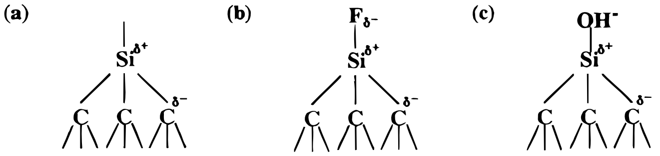

Figure 4. Schematic illustrating the mechanism for F and OH termination of $(0001)_{Si}$ 6H-SiC. (a) bare $(0001)_{Si}$ 6H-SiC surface with uncanceled dipole, (b) and (c) $(0001)_{Si}$ 6H-SiC surface dipole canceled by F^- and OH^-, respectively.

The HF vapor exposure also results in significant amounts of fluorine on the $(0001)_{Si}$ 6H-SiC surface. The observed F coverage following the HF vapor treatment was three to four times larger than that previously observed from $(0001)_{Si}$ 6H-SiC wafers dipped in 10:1 HF and blown dry (without a deionized water rinse).[15] The surface coverage approaches that of one-half to a full monolayer. As the peak position of the F 1s core level after the HF vapor treatment remains essentially unchanged at 685.9 eV (i.e., Si–F bonding), this suggests that the HF vapor treatment leaves a Si–F-terminated SiC surface.[38-40] These results are in contrast with those of Iyer et al.[31] for (100)Si in which no F was detected by XPS and hydrogen termination was confirmed by temperature-programmed desorption (TPD). However, in our previous study regarding HF wet chemical processing of SiC, [15] it was argued that OH^- termination would be preferred for $(0001)_{Si}$ 6H-SiC as opposed to hydrogen due to the polarity of the Si–C bond. Termination of the $(0001)_{Si}$ SiC surface with OH^- tends to cancel the dipole created by the Si–C bond, whereas termination with H does not, as shown schematically in Fig. 4. Similarly, F^- ions derived from the HF vapor could also cancel this dipole as the F^- ions are more readily available than OH^- ions. Therefore, F termination of SiC surfaces should be expected after HF vapor processes, as opposed to either H or OH^- termination.

Conclusions

A completely dry cleaning process which removes C contamination from $(0001)_{Si}$ 6H-SiC surfaces via UV/O_3 oxidation and removes surface oxides via HF vapor exposure has been demonstrated. This procedure has been found to be equivalent to or better than other standard wet chemical processes based on the residual levels of surface C and oxide contaminants. The HF vapor exposure resulted in a F-terminated SiC surface, in contrast to a H-terminated surface which occurs on Si. This process also resulted in residual adventitious C which was largely attributed to recontamination in the laboratory ambient. This effect may be eliminated by either in situ vapor phase cleaning or the use of well-controlled mini environments.

Acknowledgments

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