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Growth of aluminum oxide on silicon carbide with an atomically sharp interface

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The development of SiC wafers with properties suitable for electronic device fabrication is now well established commercially. A critical issue for developing metal-oxide-semiconductor field effect transistor devices of SiC is the choice of dielectric materials for surface passivation and insulating coatings. Although SiO₂ grown thermally on SiC is a possibility for the gate dielectric, this system has a number of problems related to the higher band gap of SiC, which energetically favors more interface states than for SiO_2 on Si, and the low dielectric constant of SiO_2 leading to $2.5 \times$ higher electric fields across the oxide than in the surface of SiC, and to a premature breakdown at the higher fields and higher temperatures that SiC devices are designed to operate under. As a replacement for SiO₂, amorphous Al₂O₃ thin film coatings have some strong advocates, both for *n*- and *p*-type SiC, due to the value of its band gap and the position of its band edges with respect to the band edges of the underlying semiconductor, a number of other material properties, and not the least due to the advances of the atomic-layer-deposition process. Exploring the fact that the chemical bonding of Al_2O_3 is the strongest among the oxides and therefore stronger than in SiO_2 , the authors have previously shown how to form an Al_2O_3 film on Si (111) and Si (100), by simply depositing a few atomic layers of Al on top of an ultrathin (0.8 nm) SiO₂ film previously grown on Si surfaces [Si (111) and Si (100)] and heating this system up to around 600 °C (all in ultrahigh vacuum). This converts all the SiO_2 into a uniform layer of Al_2O_3 with an atomically sharp interface between the Al_2O_3 and the Si surface. In the present work, the same procedures are applied to form Al₂O₃ on a SiC film grown on top of Si (111). The results indicate that a similar process, resulting in a uniform layer of 1-2 nm of Al₂O₃ with an atomically sharp Al₂O₃/SiC interface, also works in this case. © 2016 American Vacuum Society. [http://dx.doi.org/10.1116/1.4972774]

I. INTRODUCTION

The industrial use of SiC for semiconductor devices is today mostly based on epitaxial wafers of 4H-SiC on SiC. A challenge is the realization of SiC MOSFETs (Ref. 1) with sufficiently high electron mobilities. In the present work, thin films of 3C-SiC on Si are used because they are readily available with the present techniques, using the lowest growth temperatures, but the results obtained should be transferable to other types of SiC. There are, however, earlier reports about the use of 3C-SiC/Si MOSFETs at high temperatures.² In this reference, operation at up to 400 °C of this MOSFET was achieved. It was fabricated with an *n*-type SiC layer grown epitaxially on a Si substrate.

The aim of the present study was to investigate an easy and reproducible method for depositing a thin (1-2 nm) film of amorphous Al₂O₃ with an atomically sharp Al₂O₃/SiC interface on the surface of 3C-SiC on Si, by adopting procedures already proven successful for the similar process with Si (111) and Si (100). The advantages of the use of Al_2O_3 as a dielectric on SiC has been discussed and studied by Wolborski et al.³ and in Ref. 1, where the preferred method of deposition was the atomic layer deposition (ALD) method. For the use of this method, various pretreatments of the SiC surface are necessary (see Ref. 1) and some posttreatments like annealing at lower $(150 \,^{\circ}\text{C})^3$ or at relatively high temperatures $(800 \degree C)^1$ have also been tried to reduce the accumulation of negative charges in the oxide. However, some of these procedures may act to crystallize the Al_2O_3 , which would lower the barrier for charge tunneling through the oxide. To avoid some of the problems described in these

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Refs. 1 and 3 with impurities and initial surface cleaning, the present studies are conducted under ultrahigh vacuum (UHV) conditions, to minimize surface contamination problems. A simpler approach to the deposition of Al_2O_3 than with conventional ALD is also used, based on the advantages of the UHV environment. This environment, with proper analytical equipment installed, also allows for *in situ* photoemission with synchrotron radiation studies during the reactions, for optimized surface sensitivity and spectral resolution. The fresh SiC surfaces are produced in UHV shortly before the reaction steps described in the following.

Thin layers of epi-3C (polycrystalline)-SiC may be grown directly on a clean Si surface with a microwave-excited CH₄ plasma interacting with hot Si surfaces in UHV.⁴ In analogy with previous findings for Si, an ultrathin Si-oxide layer may be grown on top of the SiC surface, with a thickness of around 1 nm, by thermal oxidation. This reaction works as a self-limiting growth process, at a relatively low oxygen pressure, and at a temperature of 800 °C of the SiC/Si sample. On top of this layer, after cooling down to room temperature, 1 nm of Al is now deposited from a Al-wire suspended in a hot W-wire loop, with the thickness monitored by a quartz crystal microbalance. This system, with an Al layer on top of the SiO₂ is then quickly heated to $530 \,^{\circ}$ C and further up in temperature in small steps. To follow and characterize the processes occurring after all steps as described above, we monitored the surface-, interface-, and substrate properties using synchrotron radiation induced core level photoemission at ASTRID, Aarhus, Denmark.

We found a similar outcome for this procedure, as for Si,⁵ i.e., spectroscopic signs of an atomically sharp interface between Al_2O_3 and SiC. Several steps in this reaction scheme offer self-limiting behaviors, i.e., the growth of SiC on Si, the oxidation to create SiO₂, and the conversion into Al_2O_3 , which only needs a sufficient amount of Al to affect the total conversion of the SiO₂, while excess Al will leave the system at sufficiently elevated temperatures. Thus, all steps ultimately qualify as examples of self-limiting, genuine atomic layer deposition processes.

II. EXPERIMENT

In this work, there have been three different processes of interest, and experimentally, there has been a constructive interplay between suitable facilities.

The first topic is the possibility to form epitaxial 3C-SiC films on Si (111) and Si (100) surfaces using a beam of a microwave excited CH₄ plasma. Early results of this work have been described in Ref. 4, and the work has later been extended in Refs. 5 and 6. In summary, these studies have shown that the growth of 3C-SiC on Si (111) and Si (100) is possible at a relatively low substrate temperature (above 500 °C) from exposure to a CH₄ plasma excited with microwaves. The build-up of the SiC layer follows the same kinetics as for oxidation and nitridation of Si, as described by a Hill's function,⁶ implying initial 2D-island partial coverage of SiC_x entities and later a coalescence of the islands into a uniformly covering layer with SiC composition in the form

of covalently bonded SiC₄-units, and finally a vertical growth increasing the layer thickness. The optimal growth conditions for forming the polycrystalline epitaxial system with as few pin-holes as possible and otherwise uniform film thickness were established to be at a substrate temperature of 700 °C, and with suitable plasma parameters. A slightly better procedure consisted of varying the growth temperature from 500 °C in steps up to 900 °C, and varying the duration of the CH₄ plasma exposures correspondingly in order to increase the thickness of the SiC layer, as monitored with x-ray induced photoemission (XPS). The thickness of the resulting SiC layers of the present study, made at 700 °C, were estimated from the pit depths in the AFM scans of samples prepared in another UHV system⁶ to be around 3 nm, thus thick enough to completely mask the Si 2p component from the substrate at the conditions used for the studies with synchrotron radiation at the ASTRID facility.

The second topic studied was the thermal oxidation of SiC with neutral molecular oxygen. Previously, we have explored how to grow a 0.8 nm thick Si-oxide with a high quality and optimal interface (and MOS) properties [both on Si (111) and on Si (100) surfaces] on clean Si surfaces in UHV at 600-700 °C and relatively low oxygen pressures." This procedure is self-limiting, which means that the oxide formation process saturates after a certain oxygen exposure (around 600 L). [1 L (Langmuir) = 1.33×10^{-4} Pa × s.] It was recently shown that this method of oxide growth results in flat bands on the Si (111) surface,⁸ and later TEM studies have shown that the oxide is of uniform thickness across the surface (unpublished results). Similar studies of the oxidation of SiC have been carried out⁶ with the result that a selflimiting, approximately 1 nm thick oxide consisting of SiO₂, forms on SiC at 700-800 C, still under UHV background conditions (at 10⁻³Pa oxygen partial pressure), but at much higher total exposures of oxygen (6000 L) than for the Si surfaces. The morphology and uniformity of this system has not yet been studied, except what can be deduced about this from the photoemission studies.

The final topic of interest here, and the central part of this report, is the deposition of Al on top of the SiO₂/SiC/Si system and its conformal conversion to Al-oxide by a thermal treatment. This process has earlier been used for converting SiO₂ on Si to Al₂O₃ on Si, and the idea behind this was based on the almost a factor of two higher heat of formation for Al₂O₃ than for SiO₂, which was believed to be a driving force for the conversion. For these studies, we used a UHV system with surface analysis equipment at the ASTRID storage ring facilities at Aarhus University, Denmark. This gives access to the use of photoemission with high resolution and optimal surface sensitivity by being able to change the photon energy and the resolution/sensitivity of the spectrometer.

A. Experimental facilities

For the initial studies of the plasma processing and oxidation steps, a traditional UHV system (SPECS) installed in our laboratories in Odense, Denmark, was used with facilities for surface reaction studies and surface analyses with XPS, low energy electron diffraction (LEED), ultraviolet photoelectron spectroscopy, and Auger electron spectroscopy. A AFM in air, a scanning electron microscope, and x-ray diffraction facilities were also used.^{4,6} The sample holder was transferable with built in electrical connectors, which could enable us to heat Si by resistance heating, and the temperature was monitored with a spot photometric detector from outside the vacuum system. This detector was calibrated for Si and transmission through the windows. The Si crystals used in this study were cut from 1 mm thick *n*-type Si (111) wafers from TOPSIL, Denmark, with a doping corresponding to a 5 $\Omega \times$ cm resistivity, in the form of 2.5 by 1 cm rectangular pieces.

For the work at the synchrotron, the same type of Si samples, sample heating method, and photometer were used, but the UHV system at the beam line in this case had to be baked after inserting the sample. Thus, the Si sample also in this case had to be cleaned before the experiments started. This was done by ohmic heating of the sample to above 1000 °C, and the cleanliness was verified with LEED and recording of the Si 2p spectral region in photoemission. For measuring the changes in the Si environment with Si 2p core level photoemission, a photon energy of 130 eV was chosen. At this photon energy, the kinetic energies of the Si 2p spectral details are around 30 eV, which is at the minimum of the penetration depth of electrons emitted from Si.⁹ The spectral information for Si (in the Si 2p spectral range) is thus at a maximum sensitivity of the surface features with photoemitted electrons. The beam line was equipped with a spherical grating monochromator with three different gratings for different energy ranges. The spectrometer is a hemispherical SCIENTA analyzer with a radius of curvature of 20 cm and a position sensitive detector system at the exit. The spectrometer operates at a fixed pass energy of the hemispheres, which defines the spectrometer resolution, while the electron energy is scanned with a retarding lens system at the entrance to the spectrometer. The choice of photon energies for the different spectra, and the energy resolution, is explained later in the text. However, it should be mentioned here that as a result of choosing photon energies in the range where the kinetic energies of emitted electrons produce a high surface sensitivity, the electron distribution emitted from the sample is close to the secondary electron maximum. This gives an effect of a background in all spectra shown here, which increases linearly toward lower kinetic energies, or, equivalently (see later) to higher binding energies. The electron current in the storage ring of ASTRID typically varies between 140 and 180 mA, with a lifetime of over 30 h, allowing for extended experimentation without significant changes of the experimental conditions. (A new storage ring, ASTRID 2, is now operating with a continuous circulating electron current in the ring.)

III. EXPERIMENTAL DETAILS AND RESULTS

After cleaning of the Si (111) sample, the Si 2p spectrum marked Si (111) in Fig. 1 was obtained. Immediately after recording this spectrum, a thin layer of SiC was formed on



Fig. 1. (Color online) Normalized raw Si 2p spectra of a Si (111) surface (red dots), before and after microwave excited CH₄ plasma exposure [SiC/Si (111)], and after oxidation [SiO₂/SiC/Si (111)], at a photon energy of 130 eV. The spectra were normalized to the photon flux. The energy resolution in the spectra is 0.2 eV, using an analyzer pass energy of 20 eV and 50 μ m slits placed before and after the monochromator, defining the energy width of the synchrotron radiation incident on the sample. The spectra are sampled at 0.02 eV intervals. The Si (111) 2p_{3/2} peak is at 98.8 eV.

the surface by exposing the Si sample to a plasma of methane for 30 min at a temperature of 700 °C of the Si sample.

The methane plasma was excited with a microwave cavity placed around a glass tubing system with a pressure differential between the plasma source and the UHV system.⁴ This plasma system is focusing the plasma on the sample as shown in Ref. 4. The Si 2p region was afterward recorded at a photon energy of 130 eV. The results are shown in Fig. 1 as raw data [marked SiC/Si (111)] and analyzed in more detail in Fig. 2. The spectrometer measures the kinetic energy, E_{Kinetic} , of the photoemitted electrons. These energies are converted into binding energies with respect to the Fermi level, E_{Binding} , with the formula: $E_{\text{Binding}} = E_{\text{Photon}}$ $-E_{\text{Kinetic}} - \varphi_{\text{Sample}}$. The photon energy, E_{Photon} , is determined from the calibration of the monochromator, and the value of φ_{Sample} , given in the formula above as the work function of the sample, is actually treated as a parameter which is adjusted in the spectrometer calibration to give the correct binding energies, through corrections for the difference in work functions between the spectrometer and the sample, which are in galvanic contact. This correction is therefore necessary because the kinetic energies measured in the spectrometer are influenced by the work function of the spectrometer. In all the spectra shown in this work, this convention for the value of the binding energy is strictly followed without any corrections for shifts due to charging. Because the layers on top of Si are very thin, no charging is expected. In Figs. 1 and 2(a), the Si 2p spectrum of the SiC surface layer shows the complete coverage of the Si substrate by SiC. In this spectrum, it is possible to resolve two peaks with the Si 2p spin-orbit splitting of 0.60 eV. For the oxidized sample [Fig. 2(b)] the spin-orbit splitting cannot be resolved, but the spectral shape obtained with a fit of two peaks at the expected experimental resolution of 0.2 eV clearly indicates two peaks with nearly equal intensities, from Si-C at 101.2 eV and Si-O species at 102.7 eV, respectively, indicating that the oxide layer is thinner than the SiC layer. All fits are done using the CASAXPS software.¹⁰ The



FIG. 2. (Color online) Analysis of the components of the spectra of Fig. 1 in terms of Gaussian–Lorentzian peaks with a linear background. (a) The Si 2p spectrum of the SiC/Si (111) sample with Si spin-orbit decomposition (Si $2p_{3/2}$ at 101.2 eV; Si $2p_{1/2}$ at 101.8 eV); (b) The Si 2p spectrum of the SiO₂/SiC/Si (111) sample shows a presence of two peaks with nearly equal intensities, from Si-C (101.2 eV) and Si-O species (102.7 eV), respectively, also indicating that the oxide layer is thinner than the SiC layer. All fits are done using the CASAXPS software (Ref. 10).

binding energy observed for Si 2p (101.2 eV) in SiC closely matches reported values as in Ref. 11, of 101.3 eV.

A C 1s spectrum after the creation of the SiC layer with the plasma exposure is seen in Fig. 3. It was recorded with photons of 350 eV energy, to obtain kinetic energies in the range around 65 eV for optimal surface sensitivity and for optimal transmission of the monochromator. It shows two peaks at a rather low and noisy intensity, which is due to the low thickness of the SiC layer, the lower photoemission cross section (than for Si 2p at 130 eV photon energy) at this energy, and a low photon flux from the monochromator. In addition to these effects, the spectra show an increasing background toward lower kinetic energies (higher binding energies) in the spectra as explained above. The peak at 284.7 eV is assumed due to C-Si bonds while the peak at 286.0 eV is due to C-C bonds from a layer of unreacted C on top of the SiC. The value for the C-Si bonds in this case could seem to deviate somewhat from the value reported in Ref. 11 of 283.8 eV, while the value for the C-C bonds is identical to the value reported in Ref. 11. The difference for the C-Si bond energy may be due to a difference in the bonding at the surface of the 3C-SiC as compared to the 6H-SiC of Ref. 11, or due to the relatively noisy quality of the present data. It is therefore safe to assume that the C 1s

 $\begin{array}{c} 13 \times 10^{3} \\ 12 \\ 11 \\ 10 \\ 290 \\ 288 \\ 286 \\ 286 \\ 284 \\ 282 \\ 280 \\$

FIG. 3. (Color online) C 1s region of the SiC/Si (111) sample, recorded with a photon energy of 350 eV. The deconvoluted solid curves are a fit of two combined Gaussian–Lorentzian peaks to the experimental points (dots), with maxima at 284.7 eV (C-Si) and 286 eV (C-C). The energy resolution (FWHM) in these spectra is 0.5 eV (40 eV pass energy; 100 μ m slits).

peak observed here at 284.7 eV actually represents the C-Si bonds.

As the next step, the SiC/Si (111) system was exposed to oxygen inside the UHV system (at 2×10^{-4} Pa partial pressure). To monitor the reaction of oxygen O 2s spectra were recorded. For these spectra, a photon energy of 60 eV was used, again to render the kinetic energies around the O 2s emission optimally surface sensitive, at around 40 eV kinetic energy. With the beamline used, it was not possible to obtain a proper O 1s signal. The optimal temperature for the oxidation had earlier⁶ been determined to be around 800 °C. At this temperature, the saturating oxygen exposure was of the order of 6000 L, which is much higher than for the corresponding process for Si (111) or Si (100) (around 600 L).⁷ The O 2s spectrum of the sample is shown in Fig. 4 [SiO₂/SiC/Si (111)] after saturation of the oxygen signal from this procedure, i.e., from SiO₂ on top of SiC.

The Si 2p spectrum of this situation after the oxidation was shown in Figs. 1 and 2. The third O 2s spectrum included in Fig. 4 [Al/SiO₂/Si (111)] relates to the deposition of Al on top of the structure, and will be discussed later.

The C 1s spectrum (not shown) of the oxidized SiC surface contains a (C-Si) peak at 284.3 eV and a very weak and



FIG. 4. (Color online) Normalized O 2s spectra for three different samples fitted with Gaussian–Lorentzian lineshapes. The pass energy was 75 eV and the monochromator slits were 100 μ m wide, giving a FWHM resolution of 0.8 eV. The fitted peak energies are 24.1 eV [Al₂O₃/SiC/Si (111)], 24.2 eV [Al/SiO₂/SiC/Si (111)] and 24.9 eV [SiO₂/SiC/Si (111)].



FIG. 5. (Color online) Normalized raw Si 2p spectra after Al-deposition: Al on SiO₂/SiC/Si (111) (lower spectrum, lower spectrum), and after annealing of this sample (upper spectrum, upper spectrum): Al_2O_3 on SiC/Si (111), recorded at a photon energy of 130 eV. Further analyses of these spectra are shown in Fig. 8. The same resolution and sampling conditions were used as in Fig. 1.

broad peak around 286 eV, which might be due to C–O bonds in this case.

The Si 2p spectrum of SiC after oxidation [Fig. 1: SiO₂/ SiC/Si (111)] shows a broadening toward higher binding energies. In other studies of the oxidation of 4H-SiC, Si 2p and C 1s spectra have been reported with sharper structures relating to the different oxidation stages of Si through the oxide. These oxides were produced in furnaces at much higher temperatures and pressures.¹² The position of the Si-O peak in the present experiments coincides with the energies of the intermediate $(Si^{2+}-Si^{3+})$ oxidation states near the interface for thicker oxides with well-defined bulk structures (Si^{4+}) formed at a distance from the interface.¹² Thus, the thickness of the oxide layer produced here seems comparable to a fraction of the thickness of the SiC layer, probably of the order of 1 nm, judged roughly from the attenuation of the Si-C Si 2p signal and the intensity of the Si 2p signal of the Si-O features around 103 eV binding energy. The sample now consists of an approximately 1 nm thick layer of amorphous Si-oxide on top of a 3 nm layer of SiC, all on top of Si (111).

A thin layer of Al (1 nm, gauged by a quartz crystal monitor) is now deposited on this sample from a heated Alwire. The wire was suspended in a loop of W-wire, which



Fig. 6. (Color online) Normalized raw Al 2p spectra of Al deposited on SiO₂/SiC/Si (111) (black dots: with Al-Al indication) and after the sample has been annealed (red crosses: Al₂O₃; see text). The conditions for recording these spectra are the same as in Fig. 1.

was heated by passing a current through it. The results of this deposition for the Si and Al intensities are illustrated in Fig. 5 [Al on the SiO₂/SiC/Si (111)] for the raw Si 2p signal, and in Figs. 6 and 7 for the Al 2p signal. Both sets of spectra were recorded at 130 eV photon energy, with nearly the same surface sensitivity for the signals of Si and Al. The effect of the deposition of Al on the SiO₂/SiC/Si (111) sample is clearly seen in Fig. 8(a): When Al is deposited on the top surface with oxide, it releases elemental Si at the SiO₂ surface, and masks the underlying SiC and SiO₂. We assume that the liberated Si evaporates under the thermal treatments following, but it cannot be proved under the present experimental conditions. Figure 8(b) shows the result of the heat treatment of the system (see later).

The thickness of the Al layer has reduced the signal from the SiO₂ layer (see Fig. 1) by approximately a factor of 15 and now displays, along with the Si-Si peaks, two separate Si 2p peaks from SiC and SiO₂ with a weak Si 2p oxide $(Si^{2+} + Si^{3+})$ component at 103 eV, and the Si-C Si 2p component at 101 eV binding energies [Fig. 8(a)].

The Al 2p spectrum (Figs. 6 and 7) shows the metallic Al-Al (unreacted) component at 72.5 eV binding energy and a broad oxide (Al-O) spectrum centered at 75 eV binding energy. The Al 2p spectra are also recorded with 130 eV energy photons, and with similar parameters as for the Si 2p



Fig. 7. (Color online) Analyses of the spectra in Fig. 7. (a) Al on $SiO_2/SiC/Si$ (111); (b) $Al_2O_3/SiC/Si$ (111) (only Al_2O_3 visible). The fitting of the spectra shows individual components due to bonding differences. For the Al-Al part the Al 2p spin-orbit splitting is resolvable in (a), but this is not the case for the other components in (a) or (b) at the present data quality.



FIG. 8. (Color online) Analysis of the spectra in Fig. 5 by deconvolution. (a) Al on SiO₂/SiC/Si (111), un-normalized; (b) Al₂O₃/SiC/Si (111), after thermal reaction of the sample in (a). Fitted peaks at 99.3 (Si-Al) and 100.7 eV (Si-C).

spectra. Thus, Al atoms in direct contact with O atoms and Si atoms are observed from the deconvoluted spectra, in addition to Al in contact with other Al atoms. The carbon C 1s spectra in this case have too little intensity to detect any Al-C contacts.

The sample is now gradually heated at various temperatures above 560 °C, which was the temperature for the onset of the reaction between Al and SiO₂ on Si, and a number of times (in 1 min flashes) until the spectral changes had settled, which finally occurred at 800 °C for a 2 min exposure. The results are seen in Fig. 4 (for oxygen, O 2s) and Figs. 6 and 7 (for Al, Al 2p, crosses).

The spectra in Fig. 6 are also analyzed for details by fitting with Gaussian–Lorentzian peaks. The results are shown in Fig. 7.

These experiments and measurements indicate the complete conversion of all Al to Al-oxide (Al 2p) and a shift of the O 2s peak (from O in SiO₂ to O in Al₂O₃, see Fig. 4). The Si 2p spectra of the sample corresponding to this treatment are included in Figs. 5 and 8. The [Al₂O₃ on SiC/Si (111)] Si 2p spectrum in Fig. 8 and the corresponding fitted spectrum in Fig. 8(b) show evidence for the presence of Si-C bonds in SiC (peak at 100.7 eV) and Si-Al contacts (small bulge at 99.3 eV, which is not Si-Si bonds from the Si substrate, due to the total overlayer thickness), but no signs of Si-O bonds at the same position as in the Al on SiO₂/SiC/Si (111) Si 2p spectrum, recorded before the thermal conversion. The Al 2p spectra do not show signs of crystallization of Al₂O₃, when compared to earlier experiments,⁵ despite the relatively high final temperature for the conversion reaction. In terms of intensities, the data for O 2s indicate that all the oxygen from SiO₂ goes into Al₂O₃ (see Fig. 4). The shift of the Si-C Si 2p peak from 101.2 to 100.7 eV after the processing may be due to a shift of the SiC band edges at the oxide/SiC interface (band bending).

We therefore infer from all the results obtained in this work that the same mechanism as for the reaction of Al with SiO₂ on Si surfaces may be explored for the formation of an atomically sharp interface between Al₂O₃ and the 3C-SiC surface, based on the intermediate deposition of a sacrificial SiO₂ layer on SiC. The thin oxide films studied here are typically denominated as SiO₂ and Al₂O₃ in the text and figures, although previous studies have identified that the average coordination in these thin layers differs from typical bulk stoichiometry, due to the various stages or degrees of oxidation from the interface and upward into the layers.^{8,12} We have not measured the morphology of the present system, but for the SiO₂/Si interface with the self-limiting oxidation process, we have earlier obtained a uniform layer covering all the surface area, as observed with TEM studies (unpublished). It is therefore assumed that this structure will also not be significantly changed in the present case, after the reaction with Al.

IV. SUMMARY AND CONCLUSIONS

We have with this work demonstrated a very simple and reproducible procedure for the formation of a thin (approximately 1–2 nm thick) film of Al₂O₃ on SiC with an atomically sharp oxide/semiconductor interface. A sacrificial thin layer (about 1 nm) of SiO₂ was first formed on SiC upon which a suitable amount of Al was deposited afterward and then heated to complete a reaction in which all the SiO₂ gets converted to Al₂O₃. Most of this study was conducted with the core level photoemission excited with synchrotron radiation, for high resolution and optimal surface sensitivity. To comply with this method, very thin layers were used throughout, and the oxides remain amorphous. This has the effect that spectral details are less distinct, due to relatively low count rates, than in earlier reports for thicker oxides and crystalline SiC substrates and in experiments using conventional XPS and photoemission with synchrotron radiation. Still, based on the relatively extensive and combined spectral information obtained here and in previous studies with XPS, we find strong evidence for having obtained the objective, i.e., formation of an atomically sharp Al2O3/SiC interface, which should be interesting to test electrically. Thus, the adoption of this procedure, with a maximum reaction temperature of 800 °C, could be an alternative to conventional ALD without the same needs for pre- and postprocessing for depositing ultrathin Al₂O₃ layers on SiC as a high quality gate dielectric for future commercial planar SiC-MOS devices, and for other types and geometries of SiC-based devices.

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¹A. Hallén, M. Usman, S. Suvanam, C. Henkel, D. Martin, and M. K. Linnarson, IOP Conf. Ser. 56, 012007 (2014).

- ²Y. Kondo, T. Takahashi, K. Ishii, Y. Hayashi, E. Sakuma, S. Misawa, H. Daimon, M. Yamanaka, and S. Yoshida, Jpn. J. Appl. Phys. 26, 310 (1987). ³M. Wolborski, M. Bakowski, A. Ortiz, V. Pore, A. Schöner, M. Ritala, M.
- Leskelä, and A. Hallén, Microelectron. Reliab. 46, 743 (2006). ⁴R. Dhiman and P. Morgen, Thin Solid Films **536**, 130 (2013).
- ⁵P. Morgen, J. Drews, R. Dhiman, and P. Nielsen, Nanotechnological Basis for Advanced Sensors, NATO Science for Peace and Security Series-B: Physics and Biophysics Vol. 257, edited by J. P. Reitmayer et al. (Springer, Netherlands, 2011).
- ⁶E. R. Albrechtslund, "Growth of carbon containing structures on Si-surfaces (in Danish)," Master's thesis (University of Southern Denmark, 2012).
- ⁷P. Morgen, A. Bahari, and K. Pedersen, Functional Properties of Nanostructured Materials, NATO Science Series, II. Mathematics, Physics and Chemistry Vol. 223, edited by R. Kassing et al. (Springer, Netherlands, 2006), p. 229.
- ⁸A. G. Silva, K. Pedersen, Z. S. Li, and P. Morgen, Appl. Surf. Sci. 353,1208 (2015).
- ⁹M. P. Seah and W. A. Dench, Surf. Interface Anal. 1, 2 (1979).
- ¹⁰CasaXPS Ltd., Casa Software Ltd, 2016.
- ¹¹D. Ferrah, J. Penuelas, C. Bottela, G. Grenet, and A. Ouerghi, Surf. Sci. **615**, 47 (2013).
- ¹²H. Watanabe and T. Hosoi, "Fundamental aspects of silicon carbide oxidation," in Physics and Technology of Silicon Carbide Devices, edited by Y. Hijikata (InTech, Croatia, 2012), Chap. 9.