APP Applied Physics

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Citation: J. Appl. Phys. **113**, 044517 (2013); doi: 10.1063/1.4789924 View online: http://dx.doi.org/10.1063/1.4789924 View Table of Contents: http://jap.aip.org/resource/1/JAPIAU/v113/i4 Published by the American Institute of Physics.

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Systematic structural and chemical characterization of the transition layer at the interface of NO-annealed 4H-SiC/SiO₂ metal-oxide-semiconductor field-effect transistors

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(Received 8 January 2013; accepted 11 January 2013; published online 31 January 2013)

We present a systematic characterization of the transition layer at the 4*H*-SiC/SiO₂ interface as a function of nitric oxide (NO) post-annealing time, using high-resolution transmission electron microscopy for structural characterization and spatially resolved electron energy-loss spectroscopy for chemical analysis. We propose a systematic method for determining transition layer width by measuring the monotonic chemical shift of the Si- $L_{2,3}$ edge across the interface, and compare its efficacy to traditional measures from the literature, revealing the proposed method to be most reliable. A gradual shift in the Si- $L_{2,3}$ edge onset energy suggests mixed Si-C/Si-O bonding in the transition layer. We confirm an inverse relationship between NO-anneal time and transition layer width, which correlates with improved channel mobility, enhanced N density at the interface, and decreased interface trap density. No excess C was noted in the interfacial region. © 2013 American Institute of Physics. [http://dx.doi.org/10.1063/1.4789924]

I. INTRODUCTION

Silicon carbide (SiC) is an extremely promising material for high power and high temperature electronics due to its native SiO₂ oxide, wide band gap (3.23 eV), high thermal conductivity, and high bulk electron mobility.¹ Progress in developing 4*H*-SiC metal-oxide-semiconductor field effect transistors (MOSFETs) has been hampered by low carrier mobilities in the FET channel introduced by electrically active defects at the oxide interface. The energy of these defects is within the band gap of 4*H*-SiC,² leading to mobility reduction during inversion of the channel by field termination, carrier trapping, and Coulomb scattering.³ Possible defects include accumulated interfacial carbon,^{4,5} threefoldcoordinated O and C interstitials,⁶ Si vacancies,⁷ and dangling Si and C bonds.³ Distinct transition layers at the SiC/ SiO₂ interface have been associated with these defects.^{8,9}

In recent years, a number of groups have found that the incorporation of N at the interface between 4H-SiC and SiO₂ can improve the inversion mobility of SiC MOSFETs by reducing the density of electrically active defects.^{3,10,11} Biggerstaff *et al.*⁹ have also reported that the width of the transition layer observed between SiC and SiO₂ is inversely correlated with effective channel mobility in samples that have been post-annealed in nitric oxide (NO). There are no studies, however, that systematically characterize this transition layer as a function of post-annealing time, as is the primary goal of this study. Initial electron energy-loss spectroscopy (EELS) experiments suggested an excess of C in this transition region,^{4,8,9,12} although recent theoretical,⁵ X-ray photoelectron spectroscopy (XPS),¹³ medium-energy ion scattering (MEIS),¹⁴ and further EELS¹⁵ investigations have raised doubts about this possibility. The present work attempts to further resolve this question.

II. EXPERIMENTAL METHODS

The 150- μ m *n*-channel MOSFET devices investigated in this study were fabricated on the epitaxial layer (dopant density $\simeq 5 \times 10^{15}$ cm⁻³) deposited on the (0001) Si-face of 4° miscut 4*H*-SiC wafers available commercially from Cree, Inc. The samples were thermally oxidized in dry O₂ at 1150 °C, resulting in 55 nm oxides, and post-annealed in NO at 1175 °C for up to 240 min. Further sample and process details, as well as peak field-effect mobility, N density, and interface trap density measurements are available in Ref. 11.

The samples were analyzed using high-resolution transmission electron microscopy (HRTEM), Z-contrast highangle annular dark-field scanning TEM (HAADF-STEM), and EELS using a JEOL JEM 2100F TEM/STEM equipped with a Gatan Tridiem imaging filter (GIF). The Mo gate metalization was removed to facilitate TEM specimen preparation. Cross-section specimens were prepared from the region under the gate contact using an FEI Helios 650 or a Zeiss NVision 40 focused ion beam (FIB). The specimens' foil

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FIG. 1. $[1\bar{1}00]$ HRTEM image and electron diffraction pattern of the NO-annealed (60 min) 4*H*-SiC/SiO₂ interface. Contrast variations near the interface suggest a structurally evident transition region of a few nm, but it is difficult to quantitatively measure these HRTEM images.

normals were [1100], perpendicular to the [1120] miscut direction on the Cree SiC wafers, preventing the steps from artificially inflating the transition layer width (w_{TL}) when viewed in projection. A layer of Pt (or C, if not available) was deposited to minimize FIB ion damage to the SiC/oxide interface. After thinning, the specimens were polished in the FIB at low voltages to remove Ga contamination. Final "flag" specimens had thicknesses <80 nm with minimal ion damage.

EELS data were collected as spectrum images (SIs) at 200 kV in STEM mode, allowing for simultaneous HAADF-STEM imaging. Typical SIs were taken from a rectangular area extending approximately 20 nm into each side of the interface and 50+ nm along the interface. By summing spectra parallel to the interface, a very high signal/noise (S/N) ratio could be obtained with a short dwell time (typically <0.01 s). SIs were collected with a spot size of 0.5 nm and a dispersion of 0.3 eV/channel. Data from a few of the samples were collected with line SI under similar experimental conditions. Elemental quantification of the EELS data was performed using Hartree-Slater ionization cross sections and inverse power-law background fitting.¹⁶

III. RESULTS AND DISCUSSION

A typical HRTEM image of the SiC/oxide interface in the NO-annealed MOSFET devices is shown in Fig. 1. The atomic columns are clearly visible in the SiC region, and the images reveal a smooth interface free from obvious voids or structural defects. Transition regions narrower than those in prior works⁸ are observed on both sides of the interface, in better agreement with more recent studies.¹⁷ HRTEM analysis reveals minimal interfacial roughness in these NOannealed samples aside from that inherent from the miscut of the substrates (agreeing with Ref. 18). While a large degree of interfacial roughness could lead to a possible overestimation of w_{TL} , a quick calculation reveals that the small degree of roughness present in our samples will have a negligible effect. With a 4° miscut and a single atomic step of 0.2513 nm in the $\langle 0001 \rangle$ direction of 4*H*-SiC, the expected roughness perpendicular to the interface will be only 0.2507 nm. This is substantially lower than the w_{TL} values



FIG. 2. (a) Typical background-subtracted core-loss EELS spectra at three locations across the 4H-SiC/SiO₂ interface (60 min *NO-anneal*). Inset shows O-*K* edge. (b) HAADF-STEM image highlighting SI location. (c) SI across interface; each pixel contains a full core-loss EEL spectrum and these are summed parallel to the interface (as shown) to increase S/N ratio. Quantification allows calculation of relative composition maps of (d) Si, (e) C, and (f) O.

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FIG. 3. Overview of chemical shift w_{TL} measurement method. (a) Typical high energy-resolution view of the core-loss EELS spectra across the interface from SiC to SiO₂ (60 min *NO-anneal*). Only the Si- $L_{2,3}$ edge is shown. The two thick black spectra with peaks on the left and right depict the edge in fully SiC and SiO₂ regions, respectively. (b) Cropped HAADF-STEM survey image from Fig. 2(b). The solid green box shows the location of the SI; the dotted red box indicates the region from which the spectra in (a) were taken. (c)-(e) Three examples of the chemical shift as a function of position for unannealed, 15 min NO-anneal, and 240 min NO-annealed samples, respectively, showing clear monotonic shifts in each case. Chemical shift was measured by tracking the inflection point of the spectra in (a); w_{TL} is indicated by dashed lines.

found with every measurement method (as shown below), and thus interfacial roughness is not expected to artificially inflate the transition layer thickness. It remains difficult, however, to quantitatively measure the interface width from these HRTEM images alone, so chemical analysis is needed to fully describe the nature of the transition layer.

To perform this chemical analysis, and to quantify w_{TL} , the chemical shift of the Si- $L_{2,3}$ EELS edge onset energy was measured across the interface. This parameter reveals information about the local bonding state of Si throughout the transition region.¹⁹ The results from this measure were compared to the common EELS profiling methods that have been used in previous works. These involved performing EELS quantification across the interface and taking profiles of (i) the C/Si and O/Si atomic concentration ratios (normalized to 1:1 and 2:1, respectively).^{4,8,9} Using the tails of these ratios, (ii) the relative contributions to w_{TL} of C in SiO₂ and O in SiC were determined. These tails were measured from the point where the ratio profiles crossed to the point where the derivative of the C or O signal was zero. Using atomic ratios helps to eliminate systematic quantification errors due to possible thickness variations and diffraction contrast.²⁰ Finally, w_{TL} was measured by (iii) HAADF intensity profiles⁹ in order to reveal contrast from differences in atomic composition. w_{TL} was correlated with peak field-effect mobility of devices made from the same samples as reported in Ref. 11.

Fig. 2 depicts a typical EELS analysis. The HAADF-STEM image in (b) was used to locate an EELS SI, shown in (c). Typical SiC (A), SiO₂ (C), and interface (B) spectra are shown in (a). These spectra illustrate the chemical changes that occur across the interface, including a shift of the Si- $L_{2,3}$ edge onset from $\sim 100 \text{ eV}$ to $\sim 104 \text{ eV}$, as well as the emergence of the O-*K* edge (540 eV) and suppression of the C-*K* edge (283 eV) as the beam passes from SiC to SiO₂. These phenomena can be used to systematically determine w_{TL} at the interface.

A. Si-L_{2.3} chemical shift

The primary method used to determine w_{TL} was a measurement of the chemical shift of the Si- $L_{2,3}$ edge (Fig. 3). As the local bonding configuration of Si changes across the interface, the core-electron levels and conduction band edge shift with respect to the Fermi level.²¹ This leads to a chemical shift of the core-level EEL spectrum onset, representing a change in the local bonding of the element as the material transitions from semiconducting to insulating.¹⁹ Since this method probes Si bonding and depends on relative energy shifts, many sources of systematic error cancel, including thickness variations, sample contamination, and spectrometer energy shifts. This leads to very precise values for w_{TL} , as are shown in Fig. 4(b).

Figure 3 depicts the details of the chemical shift method used to measure w_{TL} . In Fig. 3(a), the Si- $L_{2,3}$ near-edge



FIG. 4. w_{TL} vs. NO-anneal time for all methods investigated. Methods in (a) were less reliable and had larger errors than those in (b). Errors are reported as the standard deviation of multiple measurements for each method. The chemical shift measure had the least error and proved most reliable.

structure is shown for SiC and SiO₂, as well as spectra from sequential points within the transition region. These spectra are separated spatially by $0.7 \,\mathrm{nm}$, giving a w_{TL} value of 6.3 nm for the 60 min. NO-anneal sample shown in the figure. The spectral changes in the transition region are primarily characterized by a shift of the edge onset to higher energy, as discussed above. Due to spectral noise, accurately determining the absolute edge onset energy can prove difficult, but the inflection point of the onset can be easily found. This point is a reliable means to determine edge onset position,²² and provides a convenient way to determine the boundaries of the transition layer. Plotting the energy of the inflection point as a function of position (as in Figs. 3(c)-3(e)) reveals a clear transition region that was uniform in character among all samples examined. The limits of w_{TL} were chosen as the points where the derivative of the profile was equal to zero (see further discussion of this point in Sec. III B).

Measuring w_{TL} with the Si- $L_{2,3}$ chemical shift method revealed a clear downward trend in w_{TL} with respect to NOannealing time (see Fig. 4(b)). The energy of the Si- $L_{2,3}$ edge for all samples increased from SiC to SiO₂ monotonically, indicating a gradual progression in the character of Si bonding across the interface. If a layer containing a unique configuration were present (such as Si-Si), it would manifest as an anomaly in the progression of the chemical shift. The absence of such anomalies suggests a mix of Si-C and Si-O bonding in the transition region.¹⁰ The narrowest w_{TL} was measured for the 240 min anneal at 5.3 nm, which is significantly larger than the "few atomic layers" as has been suggested by recent XPS measurements.¹³ The reasons for this discrepancy are not immediately obvious, but since this prior work did not include XPS depth profiles, it is difficult to make a direct comparison to the present results. Our results indicate w_{TL} values on the same order (a few nm) as a number of previous profiling methods, including secondary ion mass spectrometry (SIMS),¹⁸ XPS,²³ and EELS composition profiles.^{8,9,15,24,25} With the Si- $L_{2,3}$ edge shift method, we are observing direct evidence of a defined transition region in Si bonding character, and its width correlates with decreased mobility (see Sec. III D), suggesting that the local bond structure of Si has a pronounced effect on the electronic properties of SiC MOSFETs.

B. Atomic composition ratios and elemental "tails"

To further evaluate this chemical shift method, we compare it to measurement methods used in the literature. Standard quantification procedures¹⁶ were used to calculate the relative concentration of each element, using the relationship

$$\frac{N_a}{N_b} = \frac{I_a}{I_b} \cdot \frac{\sigma_b}{\sigma_a}.$$
 (1)

In this formula, N_i is the number of atoms contained in the analyzed volume, I_i is the integrated core-loss spectral intensity (with the background removed), and σ_i is the calculated partial ionization cross section for each element. This quantification is performed with a known collection angle and a constant integration range (β and Δ in Ref. 16, respectively). Quantifying each pixel of the SI yields relative composition maps (Figs. 2(d)–2(f)), which were divided to give atomic composition ratio maps (Fig. 5(a)). From these maps, a



FIG. 5. (a) O/Si composition ratio map calculated from Figs. 2(d) and 2(f); (b) profiles from one row of map (a); (c) profiles summed from all rows of map (a). Note the pronounced improvement in S/N ratio. (d) Derivative of profile was used to determine w_{TL} ; (e) comparison of chemically derived w_{TL} (in blue) with HRTEM from Fig. 1.

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FIG. 6. (a) HAADF-STEM image showing region from which the intensity profile (b) was taken (240 min *NO-anneal*). w_{TL} was defined as the width between the intensity peak and inflection point, as shown.

single row of the SI can be selected as a profile (b), and summed parallel to the interface, increasing the S/N ratio (c). The derivative of this summed profile (d) provides a convenient (if arbitrary) way to define w_{TL} . In this experiment, w_{TL} was defined as the region between the zeros of the first derivative of the profile. This choice is a conservative definition for the w_{TL} boundaries, but these points were found to be very reproducible between samples and have an immediate physical interpretation. Fig. 5(e) shows how w_{TL} , as determined by EELS, extends much farther than expected from the HRTEM images alone.

An excess C signal (with a sensitivity of 2.5% above the expected stoichiometric value) was not observed for any of the NO-annealed samples, agreeing with a number of recent works.^{5,14,15} We observed that w_{TL} decreases nonlinearly as the annealing time increases, suggesting a limit to the gains from the NO-anneal, as previously seen.²⁶ The O/Si ratio indicated a slightly larger contribution to w_{TL} than the C/Si ratio (Fig. 4(b)). Likewise, the O signal "tail" extended farther into SiC than the C signal "tail" into SiO₂ (see Fig. 4(a)). A few possibilities could explain this difference. During oxidation, any ejected C is very efficiently removed from the interface via formation of CO,⁶ leaving a small C concentration tail in the SiO₂. Also, the solubility of O in SiC is very low,⁶ meaning that even the limited incorporation of O in the SiC lattice will force O atoms farther from the interface. It is important to note that the C and O elemental "tail" measurements are not explicitly measures of w_{TL} , but rather of the relative contribution of each element. As such, the values reported in Fig. 4(a) cannot be directly compared with those from the chemical shift and ratio methods in Fig. 4(b).

C. HAADF-STEM imaging

Although previous work⁹ has characterized w_{TL} by identifying a transition in the intensity profile of HAADF-STEM images, this method proved difficult in the present investigation. Attempting to maintain consistency with Ref. 9, w_{TL} was defined as the distance between the inflection point and the maximum in the intensity profile of the SiC HAADF image (see Fig. 6(b)). While the HAADF results yielded w_{TL} values on the same order as the EELS (Fig. 4(a)), this method did not display a clear downward trend and appears subject to greater error from external factors such as experimental conditions, thickness variations due to preferential thinning of the oxide during FIB milling, or possible surface contamination. Additionally, since C was observed in its expected stoichiometric ratios, the bright region present in the SiC (see Fig. 6(a)) could not be correlated to excess C, as was reported by Zheleva *et al.*⁸ We could not reliably characterize w_{TL} using HAADF-STEM intensities, and thus the chemical shift method remains as the most appropriate means to do so.

D. Correlation with electrical measurements

Fig. 7 shows a clear correlation between w_{TL} (as measured from the Si- $L_{2,3}$ edge chemical shift) and peak fieldeffect mobility, and both characteristics improve with longer NO-anneal times. These results correlate further with decreased interface trap density and increased N density at the interface (as measured by SIMS in Ref. 11). The narrowing of w_{TL} observed with longer NO-anneal times suggests that in addition to passivating dangling bonds at the interface,¹⁰ the anneal process (including the incorporation of N) can help clean the interface by actively removing C and reducing extraneous bonding configurations, which has been



FIG. 7. Comparison of NO-anneal time, w_{TL} (as measured by chemical shift), and peak field-effect mobility, as measured in Ref. 11.

previously observed by XPS.²⁷ EELS results did not reveal any significant N-*K* edge signal at 401 eV near the interface, likely due to the very dilute concentrations and brief collection times used for our ~80 nm sample. Our results nevertheless suggest that the presence of N introduced by an NO-anneal significantly narrows the SiC/SiO₂ interface and enhances the MOSFET electrical properties.

IV. CONCLUSIONS

Aside from the HAADF intensity, all w_{TL} measurement methods reveal the same trend of decreasing w_{TL} with increased NO-anneal time. Each of these measurements probes a different physical phenomenon, and their agreement enhances the validity of our methods and results. This effect correlates very well with increased peak field-effect mobility, decreased interface trap density, and increased N density, as reported in Ref. 11.

The chemical shift method presented in Sec. III A was the most reliable means used for measuring w_{TL} . This technique had the least error and every sample analyzed exhibited the same gradual shift in edge onset energy, which is measured easily by the inflection point of the edge. This method directly probes changes in the electronic structure of the transition region and is thus particularly suited to the measurement of w_{TL} . Since the electrical performance of SiC MOSFETs is adversely affected by defects in the transition layer and is what needs improvement, we propose the chemical shift technique as the most appropriate measurement method.

In summary, our results reveal a clear transition layer at the 4H-SiC/SiO₂ interface of NO-annealed samples at different annealing times and suggest a nonlinear inverse relationship between transition layer width and NO-anneal time. We provide a clear, objective, and repeatable method using the Si- $L_{2,3}$ EELS edge shift to determine w_{TL} , and confirm the inverse linear relationship between w_{TL} and peak field-effect mobility observed by Biggerstaff et al.⁹ using a systematic sample set. The observed chemical shift of the Si- $L_{2,3}$ edge indicates a gradual modification of the local Si configuration near the interface. Future work should probe the nature of this change by investigating how the near edge structure of this edge compares with *ab initio* modeling and further XPS of the interface. This work will provide a clearer picture of stresses and charge transfer at the interface and help explain the origin of mobility-reducing traps. Finally, we did not observe excess C with a sensitivity of 2.5% above the expected stoichiometric ratio on either side of the interface of these NO-annealed Si-face samples.

ACKNOWLEDGMENTS

This research was performed in part at the NIST Center for Nanoscale Science and Technology. We would like to thank Dr. Joshua Schumacher and Mike Hernandez at NIST for their assistance in FIB TEM specimen preparation. We acknowledge the support of the Maryland NanoCenter and its NispLab. The NispLab is supported in part by the NSF as a MRSEC Shared Experimental Facility. This work was supported by the U.S. Army Research Laboratory under Contracts W911NF-11-2-0044 and W911NF-07-2-0046.

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