

## Composition of 4H-SiC/SiO<sub>2</sub> Interfaces by Electron Energy-Loss Spectroscopy

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The ability to easily form an SiO<sub>2</sub> dielectric layer by oxidation in oxygen or steam is important for potential SiC microelectronic devices. Our previous studies revealed the presence of excess carbon at the SiC-SiO<sub>2</sub> interface [1-3]. This excess C is believed to contribute to high interface-state densities and low channel mobilities. Post-oxidation annealing in NO has been used to improve the quality of the interface, possibly through reduction of excess interfacial C. The materials for this characterization study on the effects of NO annealing were C- and Si-faced 4H-SiC (0001) with a 5- $\mu\text{m}$  thick doped ( $8 \times 10^{15} \text{ cm}^{-3}$ ) n-type homoepitaxial layer. Following standard cleaning procedures, oxidation was performed in dry O<sub>2</sub> for 20 min at 1150°C. The temperature was then raised at 3°C/min to 1175°C in a carrier gas of Ar, the gas was changed to NO for 2 h, and then the temperature was reduced to 900°C at 5°C/min again in Ar. A thin layer of Cr was deposited at room temperature to protect the oxide surface and cross-section TEM specimens were prepared by conventional grinding and ion milling procedures. Elemental mapping by energy-filtered transmission electron microscopy (EFTEM), as used in related work [1-3], resulted in severe beam damage to the SiO<sub>2</sub> layer. Shrinkage could exceed 50% after recording core-loss image sets for Si, C, N, and O. Instead of characterization by EFTEM, electron energy-loss spectroscopy (EELS) spectrum lines were acquired at 200 kV with a Philips CM200-FEG operating in the scanning transmission electron microscopy (STEM) mode and equipped with a GIF operated in spectroscopy mode at 1 eV/pixel and an Emispec Vision acquisition system. Typically, a  $\sim 1.2 \text{ nm}$  (FWHM) probe of  $\sim 1.0 \text{ nA}$ , an incident beam-convergence semi-angle  $\alpha$  of  $\sim 7 \text{ mrad}$ , a collection semi-angle  $\beta$  of  $\sim 10 \text{ mrad}$ , 0.5 nm pixel spacing and 1 s/pixel acquisition were used. Analyses were performed  $\sim 3^\circ$  from  $\langle 11\bar{2}0 \rangle$  and  $\langle 10\bar{1}0 \rangle$  zones (to avoid strong diffraction effects) with the SiC-SiO<sub>2</sub> interface aligned accurately on edge.

In figure 1(a) the bright line at the SiC-SiO<sub>2</sub> interface is not Z-contrast but could be from a high concentration of defects (e.g., N atoms or vacancies) that locally distort the SiC lattice. N was consistently detected at the interface in NO-annealed C-face material [fig. 1(b,c)] but never detected in equivalent as-oxidized material [fig. 1(d,e)]. There was no obvious excess interfacial C for most regions of NO-annealed C-face samples [fig. 2(a)], but 6 of 20 analyzed regions did exhibit excess C [fig. 2(b)]. Excess C in C-face samples was not efficiently removed by NO annealing since similar results were obtained for samples not subjected to the NO anneal. N profiles ( $2.3 \pm 0.6 \text{ nm}$  FWHM) peak at the interface. The low N intensities away from the interface reflect the inaccuracy of the background fit; N is not detected away from the interface. For 20 regions in NO-annealed C-face samples the average interfacial N areal density  $A_N = 1.0 \pm 0.1 \times 10^{15} \text{ cm}^{-2}$  (equivalent to  $\sim 1$  monolayer). No excess C was detected in NO-annealed Si-face samples and for 13 regions where interfacial N was detected,  $A_N = 0.48 \pm 0.1 \times 10^{15} \text{ cm}^{-2}$  [fig. 3(a)]. In 15 other analyzed regions no N was detected [fig. 3(b)]. Overall for the Si-face samples  $A_N = 0.35 \pm 0.13 \times 10^{15} \text{ cm}^{-2}$  ( $\sim 0.3$  monolayers). Measurements of “total” N incorporation by <sup>15</sup>N (p, $\alpha$ ) <sup>12</sup>C nuclear reaction analysis (NRA) agree closely with the C- and Si-face interfacial N areal densities measured by EELS [4,5].

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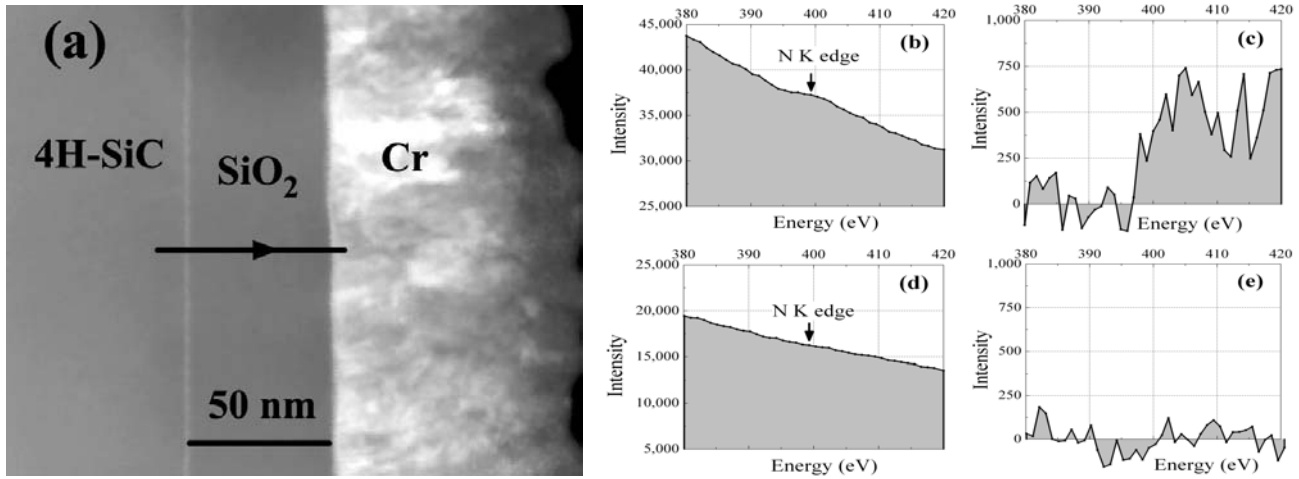


Fig. 1. (a-c) C-face oxidized and NO-annealed 4H-SiC. (a) HAADF (80-300 mrad) STEM image near [10 $\bar{1}$ 0]. N edge in interface spectrum (b) before and (c) after background subtraction. Equivalent data from interface in oxidized non NO-annealed material (d) before, (e) after background subtraction.

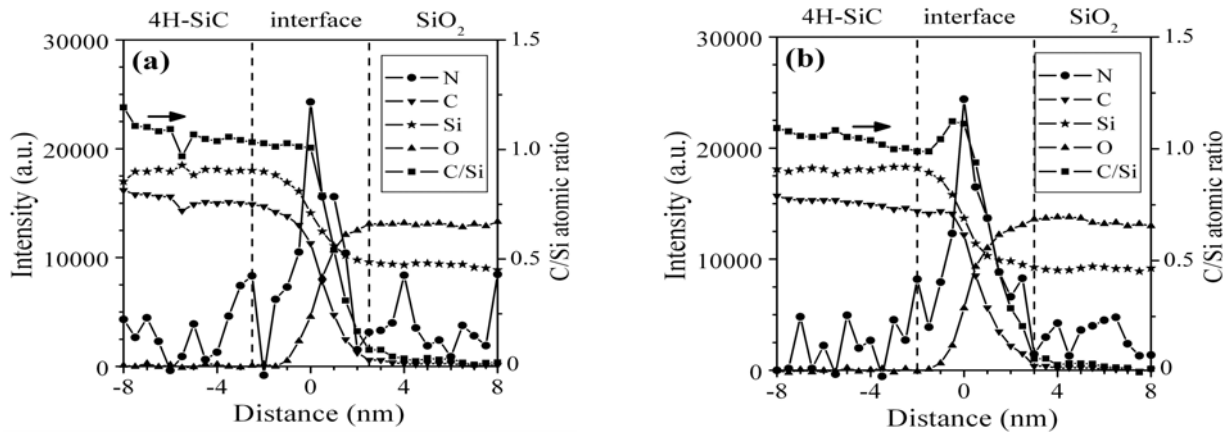


Fig. 2. Intensity profiles in NO-annealed C-face oxidized 4H-SiC. Si, C, O intensities scaled to N intensities (C/70, Si/700, O/20). (a) No obvious excess interfacial C (b) Data representative of regions with excess interfacial C (6 of 20).

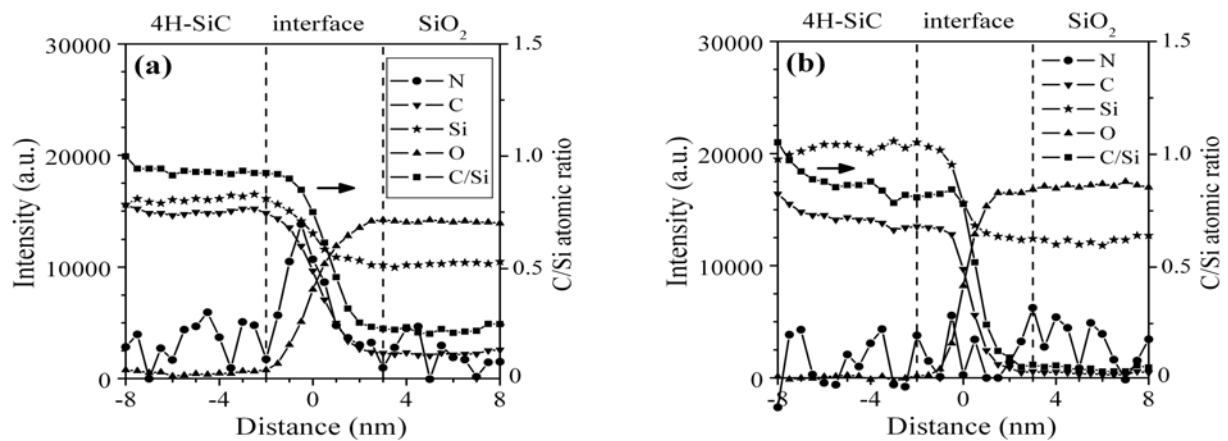


Fig. 3. Intensity profiles in NO-annealed Si-face oxidized 4H-SiC. No excess interfacial C detected. (a) Interfacial N detected in 13 of 28 analyzed regions. (b) N not detectable in 15 of 28 regions.