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# Schottky contacts on sulfurized silicon carbide (4H-SiC) surface <sup>EP</sup>

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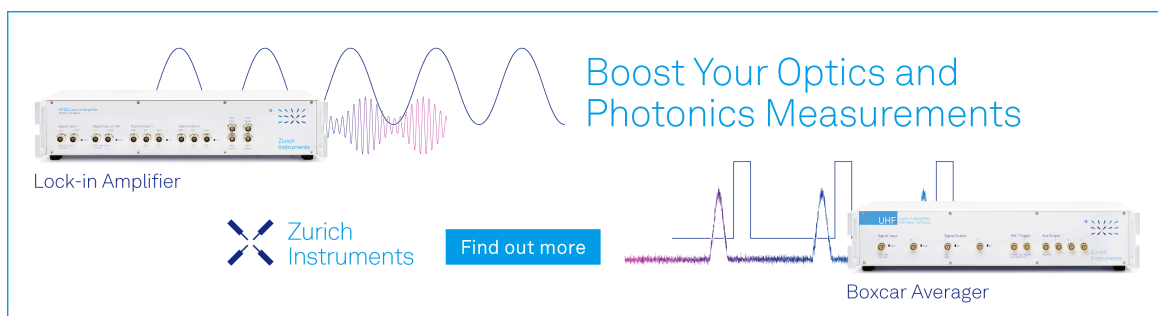
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## ABSTRACT

In this Letter, the effect of a sulfurization treatment carried out at 800 °C on silicon carbide (4H-SiC) surface was studied by detailed chemical, morphological, and electrical analyses. In particular, x-ray photoelectron spectroscopy confirmed sulfur (S) incorporation in the 4H-SiC surface at 800 °C, while atomic force microscopy showed that 4H-SiC surface topography is not affected by this process. Notably, an increase in the 4H-SiC electron affinity was revealed by Kelvin Probe Force Microscopy in the sulfurized sample with respect to the untreated surface. The electrical characterization of Ni/4H-SiC Schottky contacts fabricated on sulfurized 4H-SiC surfaces revealed a significant reduction ( $\sim 0.3$  eV) and a narrower distribution of the average Schottky barrier height with respect to the reference untreated sample. This effect was explained in terms of a Fermi level pinning effect induced by surface S incorporation.

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Silicon carbide (4H-SiC) is considered the semiconductor of choice for high efficiency power electronic devices, because of its wide bandgap, high breakdown electric field strength, and high thermal conductivity.<sup>1</sup> However, while in the last few decades, a variety of 4H-SiC devices have already reached the market, and there are still several technological issues to be addressed to better exploit the huge potential of this semiconductor.<sup>2</sup> In particular, understanding the mechanisms of current transport at metal/SiC interfaces is a continuously debated topic, due to the strong implications in practical devices, such as Schottky barrier diodes (SBDs).<sup>3,4</sup> Specifically, the control of the metal/semiconductor Schottky barrier height (SBH) is a crucial aspect required both for reducing the static power losses in SBDs<sup>5</sup> and to achieve an Ohmic behavior in non-alloyed contacts.<sup>4</sup>

The use of metals with different work functions is the common approach to tailor the SBH, owing to the linear dependence of the SBH on the metal work function in 4H-SiC, with a slope (S-parameter) of around 0.4–0.8.<sup>1,6,7</sup> However, Fermi-level-pinning (FLP) can reduce the effect of the metal work function on the SBH in 4H-SiC.<sup>8</sup> Hence, over the last decades alternative approaches have been proposed to tailor the SBH in 4H-SiC, by employing additional surface/interface

processes while using the well-established metallic contacts (e.g., Ti, Ni, Mo, etc.).

As an example, Skromme *et al.*<sup>9</sup> observed a pronounced Fermi level pinning in dry etched 4H-SiC surfaces with fluorine-based chemistry (CHF<sub>3</sub>/O<sub>2</sub> plasma), thus leading to a lowering of SBH, and more uniform current–voltage characteristics with acceptable leakage current. A similar effect has been observed using CF<sub>4</sub> plasma, while the barrier lowering could be completely recovered after appropriate annealing, leading to silicidation and consumption of a 4H-SiC surface layer.<sup>10</sup> More recently, the combination of Ar plasma treatments with rapid annealing has been proposed to control the reduction of the SBH in 4H-SiC.<sup>11,12</sup>

Alternatively, Triendl *et al.*<sup>7</sup> adopted the insertion of an ultrathin a-SiC:H interlayer below the metal to modify the SBH, using the strong FLP associated with the presence of the defect-rich amorphous layer.

Also, ion implantation has been proposed as a method to tailor the SBH on 4H-SiC, either by near-surface dopant deactivation<sup>13</sup> or by the creation of electrically active interface defects.<sup>14</sup> In this context, the local interface doping by ion-implantation can be a promising route to modify the properties of the SBH.

So far, a large number of atomic species have been considered for ion-implantation doping of SiC. Since more than two decades, it is known that *n*-type doping of 4H-SiC can be achieved by implantation of sulfur (S) ions,<sup>15</sup> which act as a deep donor in 4H-SiC.<sup>16</sup> This local doping technique has been recently applied for improving the performances of 4H-SiC transistors<sup>17,18</sup> or enhancing the carrier tunneling in Schottky contacts to 4H-SiC.<sup>19</sup>

In addition to ion implantation, which allows to introduce atoms at controlled depths inside 4H-SiC, also thermal treatments of SiC under a flux of S atoms have been recently considered. As an example, sulfurization at 700–800 °C of ultra-thin (1–2 nm) deposited molybdenum oxide (MoOx) films has been employed for the controlled synthesis of monolayer molybdenum disulfide (MoS<sub>2</sub>) on the 4H-SiC surface, and the demonstration of advanced heterojunction diodes.<sup>20</sup> More recently, Wolff *et al.*<sup>21</sup> demonstrated the successful intercalation of S at the interface between the carbon buffer layer and SiC(0001) during a sulfurization process at 850 °C, resulting in the formation of a quasi-free-standing graphene layer with the S atoms bonded to the topmost Si atoms of SiC. However, the chemical and electrical effects of the sulfurization process on the bare 4H-SiC surface remain unclear.

In this Letter, we report a detailed chemical, morphological and electrical investigation of 4H-SiC before and after sulfurization treatment at a temperature of 800 °C. Surface sensitive compositional analyses by x-ray photoelectron spectroscopy (XPS) confirmed S incorporation in the 4H-SiC surface. Morphological analyses by atomic force microscopy (AFM) showed that 4H-SiC surface topography is unaffected by this thermal process, whereas a significant increase in the 4H-SiC electron affinity ( $\sim 0.3$  V) was revealed by Kelvin Probe Force microscopy (KPFM). Finally, electrical characterization of a large number of Ni Schottky contacts fabricated on virgin and S-treated 4H-SiC surfaces revealed a significant reduction ( $\sim 0.3$  eV) of the average Schottky barrier height and its statistical dispersion, which was explained in term of a FLP effect induced by surface S incorporation.

Silicon carbide (4H-SiC) samples, consisting of a 6  $\mu\text{m}$  thick *n*-type epitaxial layer with a doping concentration of  $1 \times 10^{16} \text{ cm}^{-3}$ , grown onto heavily doped ( $5 \times 10^{18} \text{ cm}^{-3}$ ) substrates, were used in this study. In order to assess the impact of the sulfurization on the electrical properties of metal/4H-SiC contacts, Schottky diodes were fabricated. In particular, after cleaning of the sample surface, an Ohmic contact was formed on the sample back-side by sputtering 100 nm Ni films followed by a rapid annealing in N<sub>2</sub> at 950 °C. Then, the sample front side was exposed to the sulfurization process, carried out in a two heating zones tube furnace at atmospheric pressure. Sulfur powders were hosted in a crucible in the lower temperature zone at 225 °C, whereas the 4H-SiC sample was placed in the higher temperature zone at 800 °C. The sulfur vapors from the crucible were transported to the sample's surface by an Ar carrier gas at flow rate of 50 sccm. The sulfurization was carried out for 90 min, during this time 1 g of S was evaporated.

Thereafter, 100 nm thick Ni Schottky contacts were defined on the sample front-side by optical photolithography and liftoff, and subjected to a post metallization annealing at 400 °C. The active area of the diodes was  $10^{-4} \text{ cm}^2$ .

Current-voltage (I-V) measurements on the diodes were carried out in a probe station Karl Suss MicroTec equipped with a parameter

analyzer (HP4156B). The measurements were carried out both on sulfurized diodes, as well as on reference devices not subjected to the process.

In order to evaluate the impact of sulfurization on the 4H-SiC surface, x-ray photoelectron spectroscopy (XPS) analyses were performed by using Escalab Xi+ equipment by Thermo Fisher with a monochromatic Al K x-ray source (energy = 1486.6 eV). Comparative analyses on 4H-SiC samples not subjected to this process were carried out as a reference.

Kelvin Probe Force Microscopy (KPFM) measurements were carried out in PeakForce Tapping Mode Amplitude Modulation (AM)-KPFM with a Dimension Icon system by Bruker. Silicon tips with a triangular geometry, a nominal radius of 5 nm and a nominal spring constant of 0.8 N/m supported on a silicon nitride cantilever (Model PFQNE-AL) were employed in order to estimate the variation of the electron affinity of 4H-SiC induced by the sulfurization process, using patterned gold (Au) stripes as a reference.

First, XPS investigations were carried out to quantify the amount of sulfur bound to SiC lattice as a consequence of sulfurization. Figure 1 shows the comparison of the spectra acquired on a SiC reference sample and immediately after the sulfurization process at 800 °C. The presence of S in the surface or near surface region of 4H-SiC is clearly demonstrated by the S 2p core level peak. Here, the original S 2p doublet is smeared and gave a single, but widened, peak at 162.5 eV overlapped to the background. The curved shape of the background is given by the proximity of the plasmon satellites of the Si 2s. Interestingly, the binding energy of this peak is consistent with S atoms bonded to the topmost Si atoms of the SiC lattice, as recently reported in Ref. 21.

In particular, assuming that S atoms diffusion in SiC during the treatment at 800 °C is not deeper than 1 nm and all observed S is uniformly distributed within this thickness, a S concentration of 0.6 at. % could be estimated in this region.

The effects of the sulfurization process on the morphology and electronic properties of 4H-SiC surface were investigated by AFM and KPFM, as reported in Fig. 2.

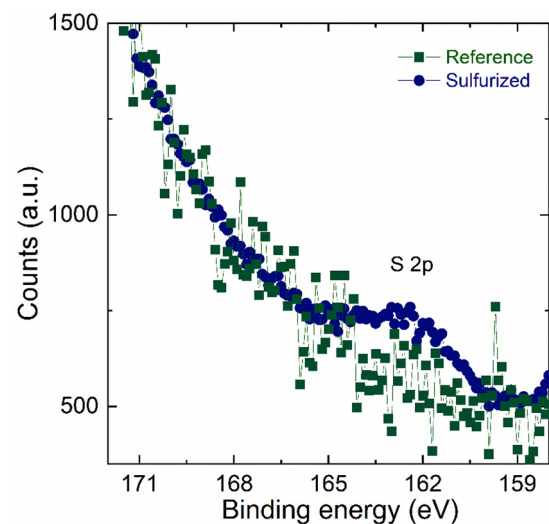
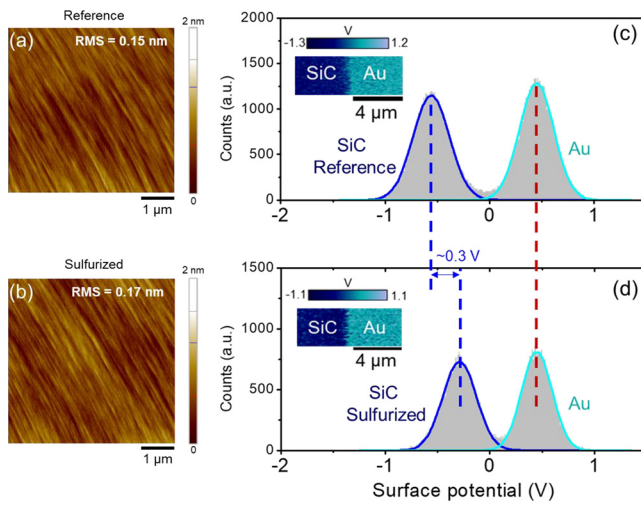


FIG. 1. XPS-spectra recorded on the reference 4H-SiC sample and after sulfurization at 800 °C.



**FIG. 2.** AFM morphology of 4H-SiC before (a) and after (b) the sulfurization process. Histograms of the KPFM surface potential values measured on 4H-SiC before (c) and after (d) the sulfurization process (blue profiles), compared to those measured on patterned Au strips, taken as reference (cyan profiles). Insets of (c) and (d) are the KPFM maps from which the surface potential histograms were extracted.

Specifically, Figs. 2(a) and 2(b) report the morphology of 4H-SiC before and after the sulfurization process, respectively, showing the typical steps associated with the  $4^\circ$ -off miscut angle. The samples' surface roughness was almost unaffected by this treatment, as deduced from the very similar root mean square (RMS) values obtained from the images acquired before ( $\sim 0.15$  nm) and after ( $\sim 0.17$  nm) the sulfurization. The effect of S atoms incorporation on the electrical properties of 4H-SiC surface was probed by surface potential mapping with KPFM, carried out both on the starting sample and after the sulfurization treatment. Patterned Au stripes were fabricated on the two samples and employed as a reference to evaluate the variation on the surface potential of the 4H-SiC after sulfurization process. Figures 2(c) and 2(d) show the histograms of surface potential values extracted from two KPFM maps measured in a region including an Au strip and 4H-SiC surface before and after sulfurization [insets of Figs. 2(c) and 2(d)]. These surface potential distributions were fitted by Gaussian

functions (cyan and blue profiles), with the peak surface potential values on Au and 4H-SiC indicated by red- and blue-dashed lines as a guide for the eye. Noteworthy, a significant increase ( $\sim 0.3$  V) of the surface potential difference between 4H-SiC and Au was observed after the sulfurization process, indicating an increase in 4H-SiC electron affinity.

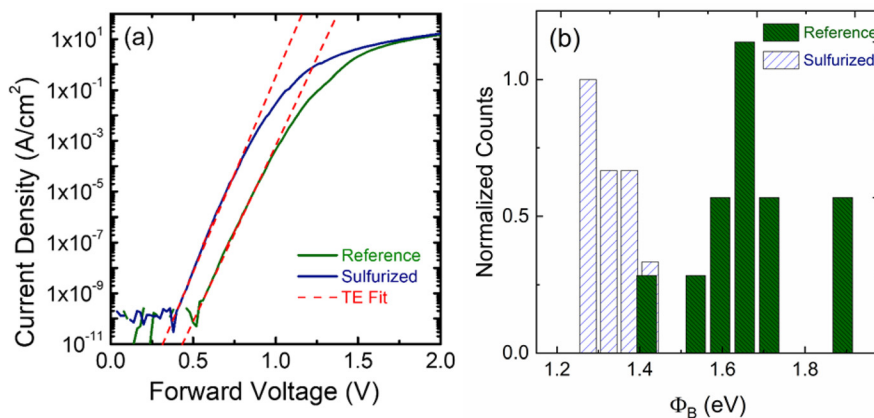
Then, Schottky diodes were fabricated on both the untreated and sulfurized sample, in order to get insights on the effect of this treatment on the electrical properties of metal/semiconductor interfaces.

Figure 3(a) shows the forward I-V curves of Ni/4H-SiC Schottky diodes fabricated on the surface of the reference sample and the sample subjected to the sulfurization process (sulfurized). The reported I-V curves are representative of the average behavior observed by measuring a set of equivalent diodes. As can be seen, the onset of the current conduction for the sulfurized diodes is lowered with respect to that of reference diodes. In particular, by a fit of the I-V curves in the linear region (superimposed dashed lines) using the thermionic emission model,<sup>22</sup> it was possible to extract the Schottky barrier height value  $\Phi_B$  and the ideality factor  $n$ .

The statistical distributions of the Schottky barrier height values determined by the fits of all the acquired I-V curves, are reported in Fig. 3(b). Interestingly, while the reference diode exhibits a Gaussian distribution of the SBH values with an average value of 1.58 eV and a FWHM of 0.11 eV, a much narrower and asymmetric distribution peaked at 1.32 eV is found in the sulfurized sample, i.e., about 0.26 eV lower than in the reference. This difference is in good agreement with the surface potential difference determined by KPM.

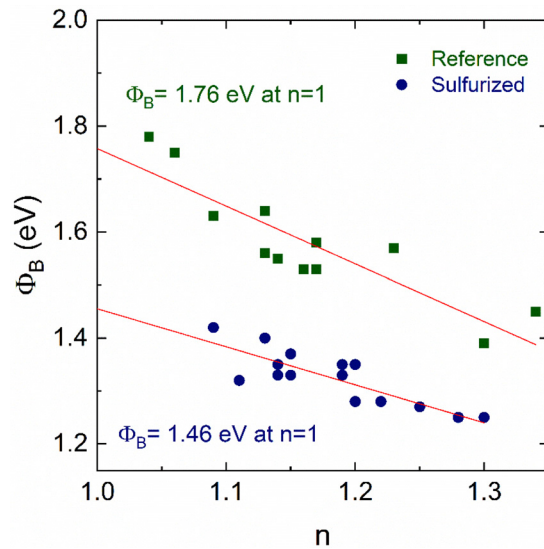
These experimental evidence indicates that the Schottky barrier lowering observed after the sulfurization of the surface is associated with a pinning of the Fermi level below the conduction band edge.

In order to get additional insights into the homogeneity of the barrier height, the experimental values of the SBH  $\Phi_B$  have been reported in Fig. 4 as a function of the ideality factor  $n$ , for both the reference and the sulfurized diodes. As can be seen, in both case a linear correlation between the ideality factor and the Schottky barrier height is found, which is an indication of the formation of an inhomogeneous barrier.<sup>9,23</sup> From the extrapolation at  $n = 1$  (ideal limit) it was possible to extract the ideal value of homogeneous barrier of 1.76 eV (reference) and 1.46 eV (sulfurized). It is worth noting that a higher slope of the plot is found in the reference contact with respect to the sulfurized one. This latter is an indication of an improved homogeneity of the



**FIG. 3.** (a) Representative forward I-V curves of Ni/4H-SiC Schottky diodes fabricated on an untreated surface (reference) and on a surface subjected to the sulfurization process at  $800^\circ\text{C}$  (sulfurized). The dashed lines represent the linear fits of the curves obtained by the thermionic emission model. (b) Statistical distributions of the Schottky barrier height values determined in the reference and for the sulfurized diodes.





**FIG. 4.** Plot of the Schottky barrier  $\Phi_B$  as a function of the corresponding ideality factor values  $n$  determined in the Ni/4H-SiC Schottky diodes fabricated on an untreated surface (reference) and on a surface subjected to the sulfurization process at 800 °C (sulfurized). The continuous lines are a linear fit of the experimental data, from which the ideal values of the Schottky barrier height can be extrapolated (at  $n = 1$ ).

barrier after sulfurization<sup>24</sup> and is in good agreement with the narrower distribution found by statistical measurements on Schottky diodes [see Fig. 3(b)].

In conclusion, the effect of a sulfurization treatment on Schottky contacts fabricated on 4H-SiC was studied by detailed chemical, morphological and electrical analyses.

In particular, the incorporation of sulfur observed by XPS did not produce any notable change in the 4H-SiC surface morphology. On the other hand, an increase in the 4H-SiC electron affinity was revealed by Kelvin probe force microscopy in the sulfurized sample. The electrical characterization of Ni/4H-SiC Schottky contacts fabricated on sulfurized 4H-SiC surfaces revealed a significant reduction (0.3 eV) of the Schottky barrier height with respect to the reference untreated sample, which is consistent with the observed variation of the electron affinity and could be explained by pinning of the Fermi level induced by surface S incorporation.

These results provide a better understanding of the electrical impact of S incorporation on SiC surfaces and can be particularly useful not only for 4H-SiC power device technology but also for integrating MoS<sub>2</sub> layered materials on SiC surfaces in advanced devices concepts.

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## AUTHOR DECLARATIONS

### Conflict of Interest

The authors have no conflicts to disclose.

### Author Contributions

**Fabrizio Roccaforte:** Conceptualization (equal); Data curation (equal); Funding acquisition (equal); Supervision (equal); Writing – original draft (equal); Writing – review & editing (equal). **Marilena Vivona:** Data curation (equal); Investigation (equal); Writing – review & editing (equal). **Salvatore Ethan Panasci:** Data curation (equal); Investigation (equal). **Giuseppe Greco:** Data curation (equal); Investigation (equal). **Patrick Fiorenza:** Data curation (equal); Investigation (equal). **Attila Sulyok:** Data curation (equal); Investigation (equal). **Antal Koós:** Data curation (equal); Investigation (equal). **Bela Pecz:** Funding acquisition (equal); Validation (equal); Writing – review & editing (equal). **Filippo Giannazzo:** Conceptualization (equal); Data curation (equal); Funding acquisition (equal); Investigation (equal); Writing – original draft (equal); Writing – review & editing (equal).

## DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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