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Low-Energy Muons as a Tool for a Depth-Resolved Analysis of the SiO₂/4H-SiC Interface

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Abstract. In this work, the potential of muon spin rotation (μ SR) with low-energy muons (LE- μ) for the investigation of oxidation-induced defects at the SiO₂/4H-SiC interface is explored. By using implantation energies for the muons in the keV range and comparing the fractions of muonium in different regions, the depth distribution of defects in the first 200 nm of the target material can be resolved. Defect profiles of interfaces with either deposited or thermally grown SiO₂ layers on 4H-SiC are compared. The results show an increased number of defects in the case of a thermal oxide, both on the oxide and on the SiC side of the interface, with a spatial extension of a few tens of nm.

Introduction

Thermal oxidation of silicon carbide (SiC) is known to generate a large density of interface defects (D_{it}), leading to threshold-voltage instabilities and low channel mobilities of SiC MOSFETs [1, 2]. While the energy position of electrically active traps has been intensely studied, their spatial position with respect to the SiO₂/SiC interface is less understood. Based on the electrical analysis of MOS capacitors using capacitance or conductance methods as well as photoemission (PE) or electron energy loss spectroscopy (EELS), these interface traps are assumed to reside in a sub-stoichiometric, defect-rich transition region on the oxide side of the interface. There is, however, a large spread of experimental data on the extension of this sub-stoichiometric region and reports range from several Å to tens of nm [3-6].

Muon spin rotation is an experimental technique that is based on the implantation of spin-polarized muons and is commonly employed to study magnetic fields inside the target material at the atomic scale [7, 8]. After a mean lifetime of 2.2 μ s, the muons decay into a positron e^+ and two neutrinos. By measuring the direction of the emitted positrons in a time-resolved fashion with detectors placed around the sample, conclusions on the interaction of the muons with the sample's local environment can be drawn. Although hydrogen or hydrogen-related impurities are by far the most studied type of semiconductor defects in μ SR, it was recently shown that this technique also allows the investigation of other defect types such as surface defects in quaternary solar cell materials [9].

In this work, we present first results where LE- μ SR is used to study the depth distribution of defects at the SiO₂/4H-SiC interface. The interface formation during two oxidation processes – thermal oxidation and SiO₂ deposition using a plasma-enhanced chemical vapor deposition (PECVD) tool are compared.

Working principle of μ SR

Positive muons are a decay product of pions, which are generated when high-energy protons (>500 MeV) collide with the nuclei of a production target (e.g. C or Be). They have a spin of 1/2, a

mass of $207 m_e$ (or $1/9 m_p$) and due to the parity violating nature of the pion decay, are spin-polarized.

A sketch of a typical μ SR setup is shown in Fig. 1. When the μ^+ are implanted into the sample, they quickly lose energy, adopt interstitial sites of the crystal lattice and thermalize without any significant loss of polarization. In the presence of a magnetic field, in our case applied perpendicular to the initial muon polarization, the spins of the implanted muons precess at the Larmor frequency $\omega_\mu = \gamma_\mu \cdot B$ ($\gamma_\mu \cdot B = 2\pi \cdot 136$ MHz/T). The field at the muon stopping site is then the vector sum of the applied field and any local field present in the sample. Once the muons decay with a mean lifetime of $\sim 2.2 \mu\text{s}$, the e^+ are preferentially emitted along the direction of the muon spin at the time of the decay and detected by the scintillator plates.

The implantation of μ^+ into insulators or semiconductors leads to the formation of a hydrogen-like bound state called Muonium [$\text{Mu}^0 = (\mu^+ e^-)$]. Mu^0 has a ~ 103 times larger muon precession frequency due to the hyperfine coupling between μ^+ and the electron spin, which allows to distinguish between diamagnetic (unbound μ^+) and paramagnetic (Mu^0) contributions. The μ^+ and Mu^0 decay symmetries A_D and A_{Mu} are determined by the amplitude of the corresponding precession signals and are proportional to the fraction of muons in a particular state.

Fig. 1. Schematic of a transverse field (TF) μ SR setup used for studying the $\text{SiO}_2/4\text{H-SiC}$ interface. In this sketch, the muons are entering from the left. The brown arrows indicate the spin of the muon, which is opposite to its flight direction.

As it was previously shown, the fraction of implanted muons forming Mu^0 changes in materials with large structural disorder [10-13]. Conventional μ SR techniques, however, use implantation energies in the MeV range that do not allow to measure shallow depth profiles with satisfying resolution. In order to extend μ SR to device-relevant thin film structures, low-energy muons with kinetic energies in the low keV range are required. Only recently, a LE- μ SR study, dedicated to the investigation of point defects in shallow crystal regions of Si and SiC was successfully demonstrated [14].

Experimental Details

For this study, both the epitaxially grown (0001) Si-face (n-type, $30 \mu\text{m}$, $N_D = 2.8 \times 10^{15} \text{ cm}^{-3}$) and the highly doped (000-1) C-face (n-type, $N_D \approx 1 \times 10^{19} \text{ cm}^{-3}$) of 4H-SiC wafers were used. All samples received either a PECVD-deposited SiO_2 with an oxide thickness of $t_{\text{ox}} = 105 \text{ nm}$ (Si-PECVD), or were thermally oxidized in O_2 ambient at 1050°C ($t_{\text{ox}} = 85 \text{ nm}$) (C-DRY, Si-

DRY+PECVD). The Si-face sample with a thermally grown oxide (Si-DRY+PECVD) received an additional PECVD-deposited SiO₂ layer to have comparable depths of the interface with respect to the surface. On another C-face sample with a dry oxide, the SiO₂ was stripped off again and a PECVD-SiO₂ ($t_{\text{ox}} = 95$ nm) was deposited instead (C-DRY-PECVD). A profilometer was used to determine the final oxide thickness while the oxide densities were extracted with an x-ray diffractometer. An overview of the measured samples is shown in Table 1.

Table 1: Sample overview. All samples used in the study were cut from the same 4H-SiC wafer.

Sample Name	Orientation	Oxidation Process	t_{ox} (nm)
Si-PECVD	(0001)	PECVD	105
C-DRY	(000-1)	thermal	90
Si-DRY+PECVD	(0001)	thermal + PECVD	30 + 70
C-DRY-PECVD	(000-1)	thermal, etch back, PECVD	95

All muon measurements were performed at the low-energy muon facility (LEM) at the Swiss Muon Source (SμS), Switzerland [14, 15]. The samples were glued to a Ni-coated holder with silver paste and mounted onto a cryostat placed perpendicular to the muon beam. For the muon implantations, kinetic energies between 3 keV and 20 keV were used to probe depths down to 160 nm below the sample surface. For all measurements, the FWHM spot size of the muon beam was 12 mm. All measurements were performed at 10 K and with a magnetic field of 10 mT which was applied parallel to the beam axis. Scintillator detectors placed above, below, left and right of the beam axis were used for the detection of the decay positrons. For each energy point, around 10^6 muon decays were measured.

Fig. 2. Calculated muon stopping profiles for a low-density deposited SiO₂ layer (a) and a thermally grown SiO₂ layer (b) on top of 4H-SiC. For the simulations, Trim.SP was used [16,17].

Results and Discussion

Figure 2 shows calculated muon stopping profiles for both oxide types and for the range of muon energies used in this study. As indicated in the graph, implantation energies below 10 keV are mainly probing the oxide, while muons with larger energies also reach into the SiC bulk.

For all samples, the diamagnetic decay asymmetry A_D is determined. A_D is proportional to the fractions of muons in a diamagnetic state and decreases with an increasing fraction of Mu^0 formation. For the specific setup and measurement parameters, a maximum of $A_D \approx 0.2$ indicates that no Mu^0 is formed and all μ^+ decay as free muons. In the case of the investigated SiO_2/SiC interface, a decrease of the Mu^0 fraction may have different reasons. One possible scenario are oxidation-induced electron traps, causing an electron shortage around the implanted muons and hence a reduced Mu^0 formation probability. Another explanation for higher A_D values is oxidation-induced lattice strain, which may significantly change the availability of Mu^0 lattice sites and with that also the Mu^0 fraction in a disordered crystal.

In the case of the deposited SiO_2 (Si-PECVD), high asymmetry values are observed at energies below ~ 12 keV. The Mu^0 formation is strongly suppressed in the oxide region, pointing to a SiO_2 of very low quality. This is not very surprising as no post-deposition annealing for an oxide densification was performed after the PECVD process. With increasing implantation energies, more and more muons reach the SiC bulk and with an increased fraction of Mu^0 formed in the crystal, the diamagnetic signal decreases accordingly. The asymmetry data for an energy scan, taken at 10 K and at a magnetic field of 10 mT, is shown in Fig. 3(a).

Fig. 3. (a) Diamagnetic asymmetry A_D measured at 10 K and 10 mT for the sample with a PECVD-deposited SiO_2 . Parametrization of the experimental data is shown as lines. Two different densities of the SiO_2 layer, $\rho_{\text{SiO}_2} = 2.1 \text{ g/cm}^3$ and $\rho_{\text{SiO}_2} = 2.2 \text{ g/cm}^3$ are compared. (b) Muon stopping profiles which were considered for the parametrization. Depending on the implantation energy, different fractions of the muons stop either in the SiO_2 or in the SiC.

For a better understanding of the depth distribution of oxidation-induced defects, the measured asymmetries are simulated as described in Ref. [16]. For this sample with a deposited SiO_2 film (Fig. 3(b)), only two layers, a low-density SiO_2 layer and the SiC bulk are needed to describe measured A_D values. Comparison with the observed asymmetries from the other three samples reveal very different compositions of the interface for different oxidation processes, as summarized in Table 2. In the case of $\text{SiO}_2/4\text{H-SiC}$ interfaces with a thermally grown SiO_2 (C-DRY, Si-DRY+PECVD), a region with increased asymmetry is detected close to the interface, reaching both into the oxide (~ 15 -20 nm) and into the SiC bulk (~ 25 -30 nm). This increase of A_D is the result of a

suppressed Mu^0 formation, which may be caused by a lack of electrons near the implanted μ^+ . Furthermore, the thermally grown oxide bulk is found to be of higher structural order than the deposited oxide, exhibiting a much higher Mu^0 fraction.

Finally, the sample with a PECVD- SiO_2 layer, deposited after an etch-back of a thermally grown SiO_2 layer, supports the idea of a crystal modification during thermal oxidation: no contribution of a highly defective near-interface oxide region is required to describe the measured asymmetry, while an extended layer with low Mu formation probability is still present in the SiC bulk.

The reason for the experimentally observed electron depletion or SiC lattice distortion due to thermal oxidation is currently still not fully understood. An emission of C into the SiC bulk has been proposed [18, 19]. Spectroscopic ellipsometry and capacitance-voltage studies revealed a defective or strained SiC layer of a few nm [19, 20] although the extension of this defect region is an order of magnitude smaller than in our μSR analysis. More recently, oxidation-induced local lattice distortions up to 100 nm were observed with x-ray diffractometry [21]. Strain could explain the observed suppression of Mu^0 in the shallow SiC region; however, further work is needed to correlate the μSR signal to specific interface defects or other crystal deterioration mechanisms.

Table 2: Experimentally determined depth profiles of the SiO_2 /4H-SiC interface for all four samples. Indicated are the extensions of interface-near regions, both in the SiO_2 and in the SiC crystal, which exhibit an increased asymmetry compared to the bulk values.

Sample Name	Oxidation Process	SiO_2 , defective (nm)	SiC, defective (nm)
Si-PECVD	PECVD	95 nm	-
C-DRY	thermal	15 nm	20-25 nm
Si-DRY+PECVD	thermal + PECVD		25-30
C-DRY-PECVD	thermal, etch back, PECVD	-	25-30 nm

Summary

In conclusion, a first proof-of-concept of LE- μSR for the study of defects directly at or near the SiO_2 /4H-SiC interface was presented. While samples with deposited SiO_2 showed an abrupt transition from the oxide to the SiC bulk, two extended regions with lower Mu^0 fractions on the oxide and the SiC side of the interface were observed in the case of the thermally oxidized sample. A modification of the bulk region also remained when the thermally grown oxide was etched back and a PECVD- SiO_2 layer was deposited. The observed increase in diamagnetic asymmetry at dry SiO_2 /SiC interfaces is believed to be closely connected to either the formation of interface defects or the introduction of lattice strain.

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References

- [1] S. Dhar, S. T. Pantelides, J. R. Williams, et al., Silicon Dioxide–Silicon Carbide Interfaces: Current Status and Recent Advances, in: D. M. Fleetwood and R. D. Schrimpf (Eds.), Defects in Microelectronic Materials and Devices, CRC Press, Boca Raton, 2009.
- [2] M. Cabello, V. Soler, G. Rius et al., Mat. Sci. Semicon. Proc. 78, 22 (2018).
- [3] B. Hornetz, H.-J. Michel, J. Halbritter, J. Mater. Res. 9, 3088 (1995).
- [4] T. L. Biggerstaff, C. L. Reynolds, T. Zheleva, et al., Appl. Phys. Lett. 95, 032108 (2009).
- [5] A. F. Basile, J. Rozen, J. R. Williams, et al., J. Appl. Phys. 109, 064514 (2011).
- [6] X. Zhang, D. Okamoto, T. Hatakeyama, et al., Appl. Phys. Express 10, 064101 (2017).
- [7] A. Yaouanc, P. D. de Rétoir, Muon spin rotation, relaxation, and resonance: applications to condensed matter, Oxford University Press, Oxford, 2011.
- [8] E. Morenzoni, T. Prokscha, A. Suter, et al., J. Phys.: Condens. Matter 16, S4583 (2004).
- [9] H. V. Alberto, R. C. Vilão, R. B. L. Vieira, et al., Phys. Rev. Mat. 2, 025402 (2018).
- [10] B. D. Patterson, Rev. Mod. Phys. 60, 69 (1988).
- [11] E. Albert, S. Barth, A. Möslang et al., Appl. Phys. Lett. 46, 759 (1985).
- [12] E. Westhauser, E. Albert, M. Hamma et al., Hyperfine Interact. 32, 589 (1986).
- [13] J. Woerle, T. Prokscha, A. Hallén, et al., Phys. Rev. B 100, 115202 (2019).
- [14] T. Prokscha, E. Morenzoni, K. Deiters, et al., Nucl. Instrum. Meth. A 595, 317 (2008).
- [15] E. Morenzoni, H. Glückler, T. Prokscha et al., Phys. B: Condens. Matter 289-290, 653 (2000).
- [16] W. Eckstein, Computer Simulation of Ion-Solid Interactions, Springer Series in Material Science, Berlin, 1991.
- [17] E. Morenzoni, H. Glückler, T. Prokscha et al., Nucl. Instrum. Methods Phys. Res. B 192, 254-266 (2002).
- [18] S. T. Dunham and J. D. Plummer, J. Appl. Phys. 59, 2551-2561 (1986).
- [19] Y. Hijikata, H. Yaguchi and S. Yoshida, Mater. Sc. Forum, 679-680 (2011).
- [20] H. Hashimoto, Y. Hijikata, H. Yaguchi et al., Appl. Surf. Sc. 255, 8648-8653 (2009).
- [21] A. D. Hatmanto, K. Kita, Appl. Phys. Express 11, 011201 (2018).