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Depth-Resolved Study of the SiO₂-SiC Interface Using Low-Energy Muon Spin Rotation Spectroscopy

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Abstract. In this work, the interface between 4H-SiC and thermally grown SiO₂ is studied using low energy muon spin rotation (LE- μ SR) spectroscopy. Samples oxidized at 1300 °C were annealed in NO or Ar ambience and the effect of the ambience and the annealing temperature on the near interface region is studied in a depth resolved manner. NO-annealing is expected to passivate the defects, resulting in reduction of interface traps, which is confirmed by electrical characterization. Introduction of N during annealing to the SiC matrix results in a thin, carrier- rich region close to the interface leading to an increase in the diamagnetic asymmetry. Annealing in an inert environment (Ar) seems to have much less impact on the electrical signal, however, the μ SR signal shows a reduced paramagnetic asymmetry, indicating a narrow region of low mobility at the interface.

Introduction

The interface between SiC and thermally grown SiO₂ has large number of interface defects, resulting in low channel mobility and limited device performance. Although NO-annealing has shown to reduce the density of interface states (D_{it}), the resulting changes at the interface and the interplay between N and the SiC crystal are not completely understood [1,2]. Low energy muon spin rotation (LE- μ SR) spectroscopy is a powerful tool to study thin films and multi-layered structures. The implanted positive muons (μ^+) act as local probes in the material and enable the extraction of electrical information about the sample in a depth-resolved manner. Herein, we study the effect of thermal oxidation and post-oxidation annealing on the interface of the grown oxide and 4H-SiC using LE- μ SR.

Working Principle of μ SR

The μ SR measurements were performed at the low-energy muon (LEM) facility at the Paul Scherrer Institute (PSI), Switzerland [3]. During the experiment, the implanted μ^+ has the possibility to bind with an electron (e^-) in the semiconductor and form a neutral hydrogen-like paramagnetic state known as Muonium (Mu^0). In regions with large carrier concentration, Mu^0 can capture an electron to form Mu^- or capture a hole to form Mu^+ . Both Mu^+ and Mu^- are referred to as the diamagnetic states. The paramagnetic and diamagnetic states can be distinguished by their different muon spin precession frequencies in an externally applied magnetic field. The recorded decay asymmetries (diamagnetic: A_D , paramagnetic: A_{Mu}) and the calculated diamagnetic fraction (F_D) provide information about the local environment of the implanted μ^+ .

At LEM, μ^+ with tunable energy between 1-30 keV are implanted at mean depths between ~ 10 nm and ~ 200 nm. During slowing down, the μ^+ produces an ionization track of e^- . Below a few keV, the μ^+ does not generate e^- by ionization but loses energy by charge-exchange processes [4]. If the μ^+ leaves the charge-exchange regime as neutral Mu^0 , it is known as ‘prompt Muonium’. If the μ^+ does not form Mu^0 and the final charge state of the muon is positive, it can capture an electron from its ionization track and form what is known as ‘delayed Muonium’ [5]. Therefore, in order for the delayed Mu^0 to be formed, the electron at the end of the ionization track has to travel to the μ^+ under the influence of its electric field. The time taken by the electron to reach the μ^+ depends on the distance traveled and its mobility in that nanoscale of the semiconductor. Therefore, for a low-doped semiconductor and at low temperature where the only available electrons for delayed Mu^0 formation are from the muons’ ionization track, a measure of Mu^0 asymmetry (A_{Mu}) is directly proportional to the mobility of electrons around the implantation depth of μ^+ in the semiconductor [6]. Further details on the measurement procedure are described elsewhere [7].

Experimental Details

We study n-type, 4H-SiC samples having a 30 μm thick epitaxial layer, and a doping density (Nitrogen) of $2.8 \times 10^{15} \text{ cm}^{-3}$. Thermal oxidation was performed at 1300 °C in O_2 atmosphere and the samples underwent a post-oxidation anneal (POA) in NO and Ar atmosphere at 1300 °C for 70 minutes. A thin layer of SiO_2 was deposited on top of the thermal oxide using PECVD to reach a total thickness of 100 nm, such that the distance between the surface and the SiO_2 -SiC interface is equivalent for the three samples. An overview of the sample processing parameters is presented in Table 1.

Results and Discussion

Fig. 1(a) shows the diamagnetic asymmetry measured. At 10 K, the Mu^0 is expected to stay in the neutral charge state, due to lack of free carriers to form Mu^+/Mu^- . Therefore, the A_D recorded is similar across the three samples and independent of the POA. The paramagnetic signal as seen in Fig. 1(b) indicates the ease for the last ionization track electron to reach the implanted μ^+ to form delayed Mu^0 . The paramagnetic asymmetry recorded for the NO-annealed sample is very close to the non-annealed sample indicating that for the two samples, the mobility of electrons perpendicular to the SiO_2 -SiC interface is similar. However, the Ar-annealed sample has a smaller A_{Mu} indicating a hindrance to the carriers flow in the first 20 nm, beyond which the Mu^0 signal for the three samples is similar. This suggests that the annealing at 1300 °C in Ar ambience affects the first 20 nm of the semiconductor. The A_{Mu} recorded for the Ar and NO-annealed samples also show that Mu^0 formation is suppressed in the thermal SiO_2 layer. A_{Mu} rises sharply as the SiC region of the sample is reached, showing clearly that the μSR signal is very sensitive to the SiO_2 -SiC interface.

Table 1. Description of sample processing steps and resulting oxide layer thicknesses.

Name	Thermal SiO_2 thickness [nm]	Post-oxidation anneal (POA)	PECVD SiO_2 thickness [nm]
1300x	48	Not annealed	52
1300NO1300	56	NO at 1300 °C for 70 minutes.	44
1300Ar1300	48	Ar at 1300 °C for 70 minutes.	52

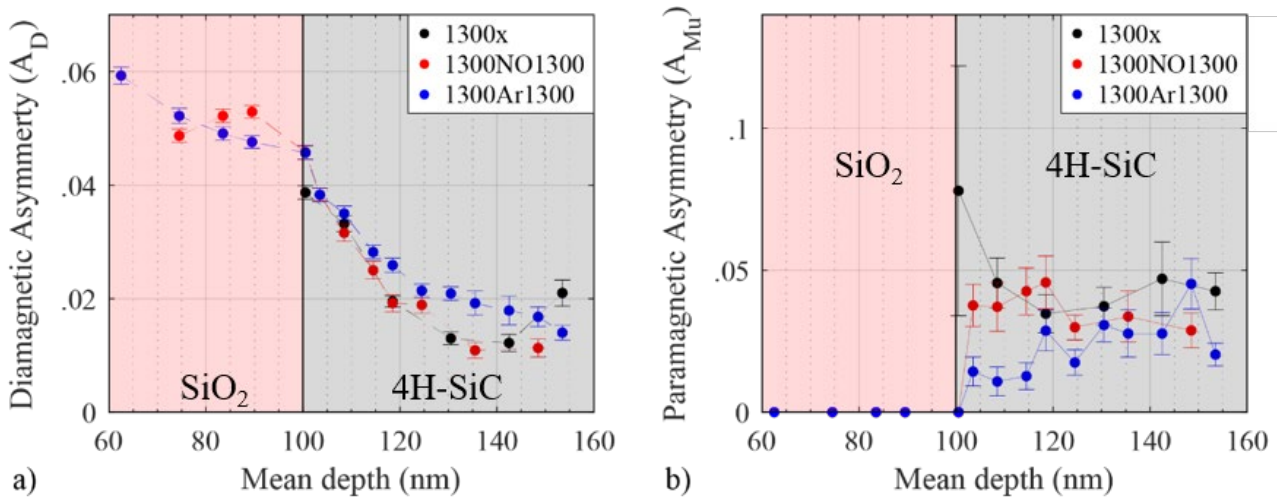


Figure 1: μ SR data recorded at $T = 10$ K and $B = 0.5$ mT. (a) At low temperatures, due to freeze out of carriers, the Mu^- formation is suppressed and A_D is similar for the three samples. (b) The A_{Mu} signal indicates a higher mobility in the NO-annealed sample when compared to the Ar-annealed sample in the first ~ 20 nm of SiC.

The experiments performed at 260 K and 0.5 mT (Fig. 2) show again a drop in the A_D signals for the non-annealed and the Ar-annealed sample, reaching quickly the bulk level [8]. However, the Ar-annealed sample exhibits a slightly smaller A_D signal, ~ 20 nm into SiC from the interface compared to the non-annealed sample. The drop in diamagnetic signal is likely due to the reduction in carrier concentration. This argument is also supported by an increase in the A_{Mu} signal for the Ar-annealed sample compared to the non-annealed sample beyond 20 nm into the SiC. In contrast, the NO-annealed sample has a much higher A_D signal compared to the non-annealed and Ar-annealed samples. The A_{Mu} signal for the NO-annealed sample is almost zero whereas it is significantly higher for the other two. Low A_{Mu} , together with the increased A_D suggests a region of large carrier concentration, which supports the formation of Mu^- .

The three samples were characterized electrically and the results of the capacitance-voltage (CV) measurements are shown in Fig. 3 a). Annealing in an Ar environment has very little effect on D_{it} , whereas with NO-annealing, the D_{it} is reduced significantly as shown in Fig 3 b). It has been suggested that the silicon vacancies (V_{Si}) are formed during thermal oxidation by the emission of silicon, contributing to the large D_{it} at the SiO₂-SiC interface [9]. Recently, μ SR was shown to be sensitive to the presence of V_{Si} in 4H-SiC, with V_{Si} enhancing the formation of Mu^0 , resulting in the reduction of the A_D [8]. In our experiment, nitrogen, released during NO-annealing, could passivate the D_{it} and more specifically V_{Si} , explaining the increase of A_D . The N on the substitutional Si site could act as a donor and result in a region of large carrier concentration. The A_D for the 1300NO1300 sample gradually reduces as we move farther away from the interface due to a decreasing N-concentration in the SiC. This trend suggests that the A_D for the NO-annealed sample would merge with that of the non-annealed sample beyond 70-80 nm into the SiC.

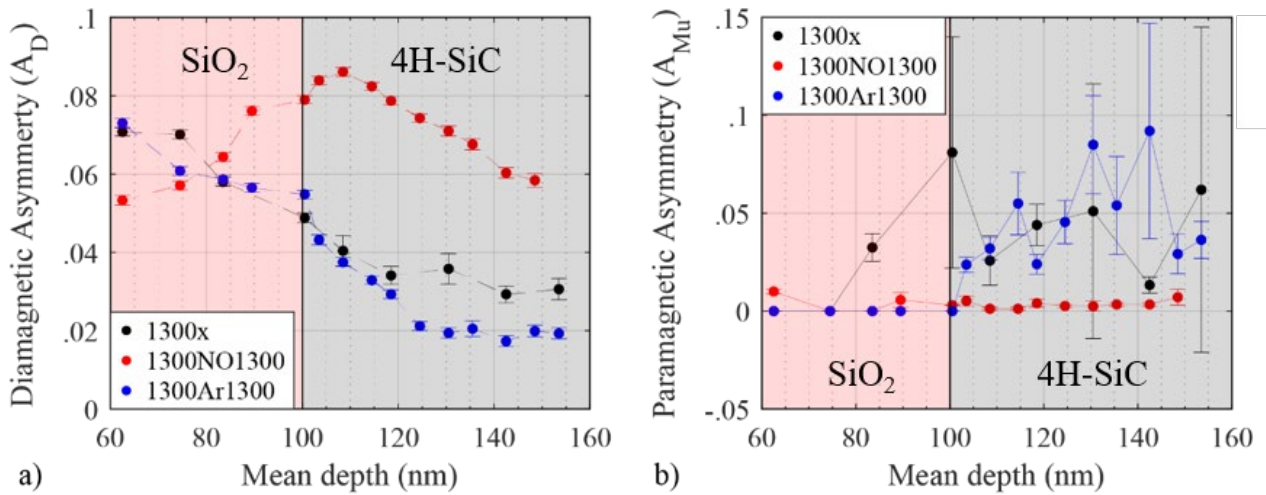


Figure 2: μ SR data recorded at 260 K and 0.5 mT. (a). The A_D for the NO-annealed sample is significantly higher than Ar and non-annealed samples. (b) A_{Mu} for the NO-annealed sample is suppressed, whereas it is significantly higher for the other two samples.

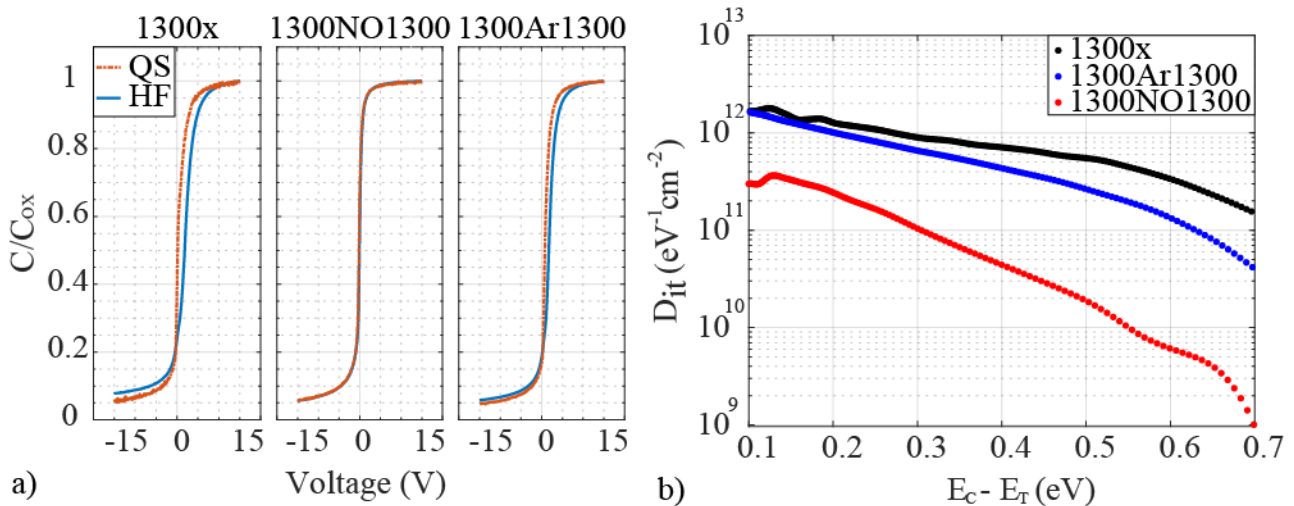


Figure 3: a) Quasi-static (QS) and high frequency (HF) measurement for the three samples. b) D_{it} calculated using quasi-static and high-frequency capacitance measurements. The D_{it} for the NO-annealed sample is significantly smaller than for the Ar and non-annealed samples.

Summary

The effect of oxidation and annealing on 4H-SiC has been studied with LE- μ SR. NO-annealing results in a region of high carrier concentration near the interface extending up to 70 nm into the SiC region but no appreciable effect on the carrier transport is observed, when compared to the non-annealed sample. Ar annealing has a very slight effect on the D_{it} (confirmed by CV measurements) and on the A_D signals. However, a reduction in A_{Mu} is observed which could suggest a narrow interface region (~ 20 nm) of reduced mobility.

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