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Use of graphite cap to reduce unwanted post-implantation annealing effects in SiC

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Abstract. 6H and 4H-SiC epilayers were Al-implanted at room temperature with multiple energies (ranging from 25 to 300 keV) in order to form p-type layers with an Al plateau concentration of $4.5 \times 10^{19} \text{ cm}^{-3}$ and $9 \times 10^{19} \text{ cm}^{-3}$. Post-implantation annealing were performed at 1700 or 1800 °C up to 30 min in Ar ambient. During this process, some samples were encapsulated with a graphite (C) cap obtained by thermal conversion of a spin-coated AZ5214E photoresist. From Atomic Force Microscope measurements, the roughness is found to increase drastically with annealing temperature for unprotected samples while the C capped samples show a preservation of their surface states even for the highest annealing temperature. After 1800°C/30 min annealing, the RMS roughness is 0.46 nm for the lower fluence implanted samples, slightly higher than for unimplanted samples (0.31 nm). Secondary Ion Mass Spectroscopy measurements confirm that the C cap was totally removed from the SiC surface. The total Al-implanted fluence is preserved during post-implantation annealing. A redistribution of the Al dopants is observed at the surface which might be attributed to Si vacancy-enhanced diffusion. An accumulation peak is also observed after annealing at 0.29 μm , depth corresponding to the amorphous/crystalline interface that was determined on the as-implanted samples by Rutherford Backscattering Spectroscopy in channeling mode. The redistribution of the dopants has an impact on their electrical activation. A lower sheet resistance ($R_{\text{sh}} = 8 \text{ k}\Omega$) is obtained for samples annealed without capping than for samples annealed with C capping ($R_{\text{sh}} = 15 \text{ k}\Omega$).

Introduction

Silicon Carbide (SiC) is a promising semiconductor material for new or improved power and high temperature applications [1]. During SiC device processing, ion implantation is required to locally dope, due to the low diffusion coefficients of the dopants. To activate the dopants by migration in substitutional sites and also to recover the matrix from the implantation-induced defects, high temperature annealing is necessary ($>1600^\circ\text{C}$), with the best activation rate for temperature in the 1800°C range [2]. At such high annealing temperature, a strong degradation of the surface occurs by sublimation of Si, resulting in a rough surface. Such surface roughness is critical for device performances since it can induce an increase of leakage currents in off-state and degradation of the breakdown voltages. Moreover, the etching of the surface might induce a significant loss of implanted dopants in the near surface region. In order to prevent Si sublimation, several solutions have been proposed as the use of AlN, graphite or SiO₂ capping [3-6], and by using silane partial overpressure during annealing [7]. However, the efficiency of the cap protection is shown to be temperature limited except for the graphite layer which resists up to 1800°C [4]. For the use of silane, an accurate control of the gas flux is necessary in order to prevent Si droplet deposition. Most of these studies were performed on samples implanted at high temperature or at room

temperature (RT) with a total implanted fluence below amorphisation threshold, and have mainly focused on the surface state after annealing. In the case of RT implantation, dynamic annealing of defects being less efficient, amorphisation of SiC can occur. Upon annealing, the recrystallisation by solid phase epitaxial growth (SPEG) can modify the surface morphology and have an effect on the doping profile.

In this paper, we present a study of the effects of a graphite (C) cap protection on high temperature annealed 6H-SiC amorphous layers created by Al ion implantation at RT. The study is focused on the modification of the dopant distribution profiles.

Experimental Procedure

Si polished N-type 6H and 4H-SiC wafers with epilayers (thickness 10 μm , doping $\sim 10^{16} \text{ cm}^{-3}$) commercialized by CREE ResearchTM have been Al implanted on whole surface at room temperature. The energies used ranged from 25 keV up to 300 keV with selected fluences (see Table 1) in order to obtain an Al plateau concentration of $4.5 \times 10^{19} \text{ cm}^{-3}$ or $9 \times 10^{19} \text{ cm}^{-3}$. The wafers were cut into 5 mm \times 5 mm samples. Before post-implantation annealing, some samples were encapsulated with a C cap. The C capping layer was formed by thermal conversion during annealing at 750°C for 30 min of a spin-coated AZ5214E photoresist layer deposited on the SiC implanted epilayers. This step was achieved in a conventional quartz tube furnace under nitrogen flux.

The post-implantation annealing to activate the Al dopants was performed in a JIPELEC rf-induction furnace under Ar atmosphere with a heating ramp of 40°C/s up to a plateau of 1700°C or 1800°C for 30 min. Subsequently, on the encapsulated samples, the capping layer was removed by an O₂ plasma in an Alcatel Nextral NE110 RIE reactor.

The surface state was analyzed after the different thermal treatments by Atomic Force Microscope (AFM), in contact mode, using Picoscan 5 Molecular Imaging.

To quantify the damage accumulation, RBS measurements in random and channeling conditions were performed on implanted samples before and after annealing using a 2 MeV He⁺ beam with a backscattering angle of 150°.

For electrical characterization, four-point probe measurements were carried out to investigate the sheet resistance R_{sh} .

Energy [keV]	Fluence [cm^{-2}]	Fluence [cm^{-2}]
300	8×10^{14}	1.6×10^{15}
190	3.9×10^{14}	7.8×10^{14}
115	2.8×10^{14}	5.6×10^{14}
60	1.9×10^{14}	3.8×10^{14}
25	9×10^{13}	1.8×10^{13}
Total implanted fluence [cm^{-2}] :		1.75×10^{15} 3.5×10^{15}

Table 1: Al ion implantation energies and fluences used to form the box dopant profile.

Results and discussion

The morphology of the surface has been studied as a function of annealing temperature and implanted fluence. Figure 1 shows the AFM images after annealing with or without the C cap compared to an unimplanted sample. The root-mean-square (RMS) roughnesses calculated from these measurements are resumed in Table 1. For the $1.75 \times 10^{15} \text{ cm}^{-2}$ total implanted fluence samples, the roughness is found to increase drastically with annealing temperature for unprotected samples, while the C capped samples show a very good preservation of their surface state even for the highest annealing temperature. In this case, after 1800°C/30 min annealing, the RMS roughness is 0.46 nm, slightly higher than for unimplanted sample (0.31 nm). The C cap has thus impeded Si sublimation

from the surface and the graphitization of the surface. Increasing the total implanted fluence to $3.5 \times 10^{15} \text{ cm}^{-2}$ results in a different surface morphology (Fig 1d). The surface is now showing a granulous aspect which tends to be oriented along a specific direction as observed in the case of furrows or macrosteps. The RMS roughness is found to slightly increase (1.49 nm) but remains in

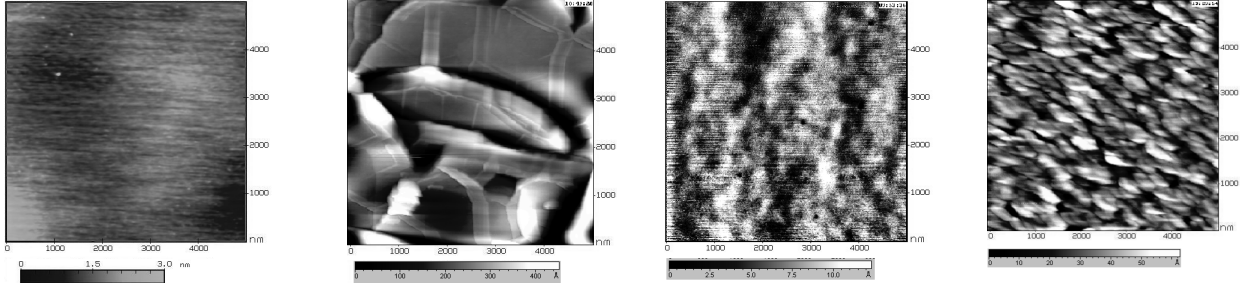


Fig. 1: $5 \times 5 \mu\text{m}^2$ AFM images for unimplanted sample (a), $1.75 \times 10^{15} \text{ Al.cm}^{-2}$ implanted sample after $1800^\circ\text{C}/30 \text{ min}$ annealed without (b), and with C cap (c), $3.5 \times 10^{15} \text{ Al.cm}^{-2}$ implanted sample after $1800^\circ\text{C}/30 \text{ min}$ annealed with C cap (d).

the same order of that of the lower fluence samples. Fig 2 presents RBS spectra of as-implanted samples showing the formation of an amorphous layer for the two different total fluences. Damage accumulation due to ion implantation seems to play a role on the modification of the surface. The surface morphology of implanted SiC after annealing does not depend only on annealing temperature, but also on the implantation fluence, even when a graphite cap protection is used.

Sample	Unimplanted	Annealed $1700^\circ\text{C}/30 \text{ min}$ ($1.75 \times 10^{15} \text{ Al.cm}^{-2}$)	Annealed $1800^\circ\text{C}/30 \text{ min}$ ($1.75 \times 10^{15} \text{ Al.cm}^{-2}$)	Annealed $1800^\circ\text{C}/30 \text{ min}$ with C cap ($1.75 \times 10^{15} \text{ Al.cm}^{-2}$)	Annealed $1800^\circ\text{C}/30 \text{ min}$ with C cap ($3.5 \times 10^{15} \text{ Al.cm}^{-2}$)
RMS roughness [nm]	0.31	14.43	24.67	0.46	1.49

Table 2: RMS roughness of the AFM analyzed samples

Secondary Ion Mass Spectrometry (SIMS) analyses performed after post-implantation annealing and O_2 plasma treatments confirm that the C cap was totally removed from the SiC surface. Flat Si and C profiles are obtained (Fig. 3a). This was confirmed by X-photoelectron spectrometry (XPS) measurements (not shown here). In Figure 3b, calibrated Al profile obtained by SIMS measurements

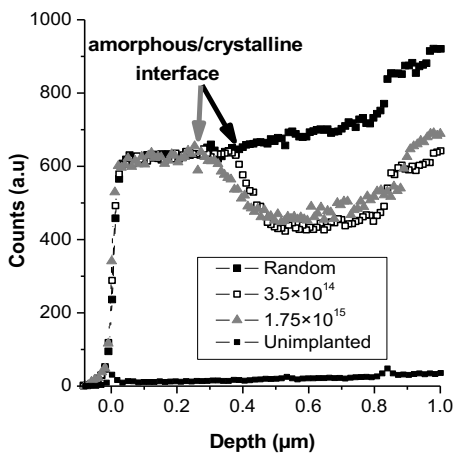


Fig.2: RBS spectra obtained on SiC implanted at room temperature at the indicated fluences

before and after annealing are shown. The total implanted fluence is preserved during post-implantation annealing with a C cap. The small variation from 1.88×10^{15} to $1.83 \times 10^{15} \text{ cm}^{-2}$ is not significant. Nevertheless, a redistribution of the Al dopants is observed at the surface, forming a uniform continuous plateau starting from the surface. This redistribution might be attributed to defect-enhanced diffusion close to the C/SiC interface formed by the presence of the C cap layer during post-implantation annealing. An accumulation peak is also observed after annealing with a C cap at a depth of $0.29 \mu\text{m}$. This depth corresponds to the amorphous/crystalline (a/c) interface which was determined on the as-implanted samples by RBS/C (Fig. 2). The region of the previous a/c interface is known to remain, after the SPEG process, a defective region (made of small dislocation loops and/or stacking faults) which can trap dopants and impurities. For samples annealed without the C cap, the redistribution is more pronounced with a strong diffusion at the deeper part of the profile. Moreover, a loss of dopants occurred at the surface. This loss can

be associated to the sublimation of Si from the surface (confirmed by the high RMS roughness obtained) resulting in the graphitization of the surface and in the out-diffusion of dopants. In contrast, for the capped sample where the C layer was deliberately deposited prior to annealing, it is rather observed an accumulation of dopants at the surface. The presence of the C cap has thus an influence on diffusion mechanisms during post-implantation annealing. The sublimation of Si from the surface might inject Si vacancies that will participate in the enhanced-diffusion of dopants while when a surface protection is used such a mechanism does not take place. Dopant diffusion mechanisms in SiC are poorly understood until now and further studies are needed to identify the mechanism responsible for such redistribution. The redistribution of the dopants has also an impact on their electrical activation. Indeed, the probability for the dopants to settle in a substitutional site

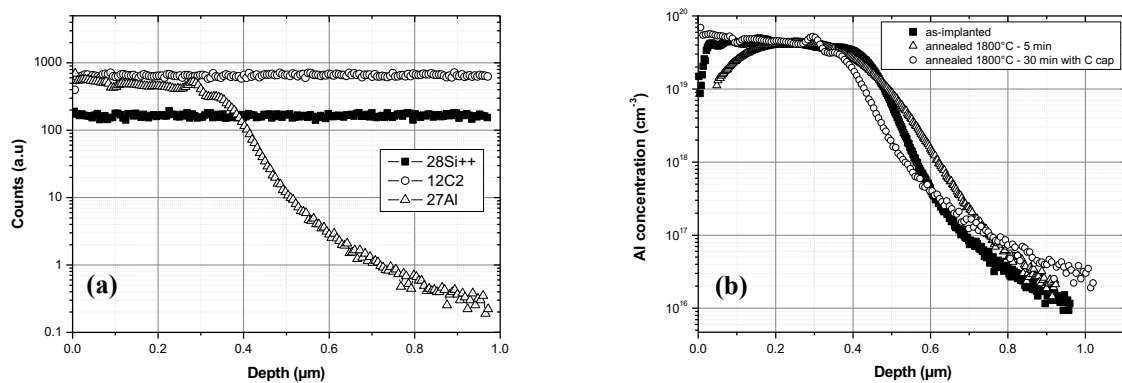


Fig.3: SIMS measurements: non calibrated C, Si and Al profiles in annealed samples after C cap removing (a), Al calibrated profiles on as implanted and annealed samples with/without C cap (b)

is higher when migration/diffusion of the dopants occurs. A lower sheet resistance ($R_{sh} = 8 \text{ k}\Omega$) is obtained for samples annealed without capping than for samples annealed with C capping ($R_{sh} = 15 \text{ k}\Omega$). The dopant diffusion thus leads to a better activation rate.

Summary

Graphite cap were used to reduce post-implantation annealing unwanted effects such as graphitization, roughening of the surface and out-diffusion of dopants. The surface morphology of implanted SiC after annealing was shown to depend on implantation parameters, even when using a graphite cap protection. The use of the graphite cap influences the diffusion of dopants and their activation rate.

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