Electrical activation of nitrogen heavily implanted 3C-SiC(100)

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Abstract

A degenerated wide bandgap semiconductor is a rare system. In general, implant levels lie deeper in the band-gap and carrier freeze-out usually takes place at room temperature. Nevertheless, we have observed that heavily doped n-type degenerated 3C-SiC films are achieved by nitrogen implantation level of ~$6x10^{20}cm^{-3}$ at 20K. According to temperature dependent Hall measurements, nitrogen activation rates decrease with the doping level from almost 100% ($1.5x10^{19}cm^{-3}$, donor level 15meV) to ~12% for $6x10^{20}cm^{-3}$. Free donors are found to saturate in 3C-SiC at ~$7x10^{19}cm^{-3}$. The implanted film electrical performances are characterized as a function of the dopant doses and post implantation annealing (PIA) conditions by fabricating Van der Pauw structures. A deposited SiO_2 layer was used as the surface capping layer during the PIA process to study its effect on the resultant film properties. From the device design point of view, the lowest sheet resistivity (~1.4mΩ.cm) has been observed for medium doped ($4x10^{19}cm^{-3}$) sample with PIA 1375°C 2h without a SiO_2 cap.

Keywords: 3C-SiC; post-implantation activation; Van der Pauw; Hall; degenerated film

1. Introduction

Silicon carbide (SiC) has been considered as the future power devices materials attributed to its superior electrical, mechanical and thermal properties. 3C-SiC has a smaller bandgap (2.3eV) than 4H-SiC (3.2eV), but still 2 times higher than Si (1.1eV) and can be grown directly on large area Si substrate via chemical vapour deposition (CVD) methods [1-3]. In addition, compared with 4H-SiC, 3C-SiC has a much better interface with SiO_2 [4], thus is promising in medium voltage (0.6-1.2kV) MOSFET applications, where channel resistance is crucial.

Highly doped regions are desired for good ohmic contact and low sheet resistance in power devices design. Due to the low diffusion coefficients of dopants in SiC, highly doped layers are mostly obtained by selective ion-implantation, and after that a high temperature (1400~1700°C) [5] PIA process is usually applied to repair the lattice damage induced by implantation, which can otherwise degrade the semiconductor layer electrical properties [6]. A roughened surface, enhanced at implanted regions, often emerges after PIA and can deteriorate the SiC interface performance such as the Schottky contact and FET channel [7-9]. A protection capping layer is often used to preserve the SiC surface during PIA, such cap materials studied include AlN [10, 11], BN/AlN [12] and graphite [8, 13]. AlN and BN/AlN process are found complex and expensive, thus not widely accepted. The graphite cap proved to be effective up to 1800°C [14] but can reduce the MOSFET channel mobility due to the excessive silicon vacancies induced by the reaction between diffused Si atoms and the graphite [14, 15]. A SiO_2 layer, which should not react with Si or C, can be easily deposited by CVD method and removed via HF etching. It was also studied and resulted a similar surface roughness level as a graphite cap

[16]. For 3C-SiC on Si wafers, the PIA temperature is limited by the melting point of Si (1412°C), well below that of SiO_2 (1610°C) [9] so a SiO_2 capping layer can be a good choice. Previously, n-type heavily implanted 3C-SiC was studied for different annealing conditions with [17] and without [18, 19] a graphite cap. A study of using deposited SiO_2 as the PIA capping layer for 3C-SiC on Si is demonstrated here. Results are reported for the implanted layer sheet resistivity, dopant activation (energy and rate) and free carrier mobility of nitrogen implanted 3C-SiC layer as following parameters: implantation doses, PIA conditions and SiO_2 capping effects.

2. Experimental Procedures

The materials studied in this work was a 10μm thick unintentionally doped ($<1x10^{16}cm^{-3}$) 3C-SiC film epitaxial grown on a 4 inch Si(100) substrate by NOVASiC. The wafer went through a chemical-mechanical polishing process [20] after the CVD growth with an initial surface RMS roughness ~0.2nm determined from atomic force microscopy (AFM) measurements. A Veeco multimode AFM with Nanonis controller was used to evaluate the surface roughness. The overall roughness value was determined by scanning three $10x10\mu m^2$ areas and averaging the results. The wafer was cut into 30 $8x8mm^2$ pieces and equally divided into 3 batches. An on-axis nitrogen implantation process was conducted on the plain surface of all samples at room temperature. A series of energies with increasing total doses were applied during implantation to form box profile at 3 doping levels, which will be called high dose, medium dose and low dose samples in following text. Nitrogen was selected as the dopant rather than phosphorous since it does less damage to the 3C-SiC film [17, 18], although it saturates more readily [8]. Prior to the PIA process, 1μm thick SiO_2 was deposited on the surface of half samples from each batch via plasma enhanced chemical vapour deposition (PECVD). To study the SiO_2 cap effect, one capped and one uncapped sample from each batch (6 all together), went through the PIA process with continuous Ar flow at 5slm. Five PIA conditions as shown in Table 1 (for each condition 2 samples from each batch) with different annealing temperature and time durations were applied to study PIA process effects on resultant 3C-SiC properties. The maximum annealing temperature applied was determined as 1375°C, just below the Si substrate melting point 1412°C [21]. The SiO_2 cap layer was removed by HF etching, after which, all samples were solvent cleaned followed by a standard RCA procedure. Van der Pauw structures were then patterned using lithography and ICP etching. A previously reported process [22] was used for the ohmic contacts formation in this work: Ti(30nm)/Ni(80nm) bilayer metal contacts were deposited at low pressure ($2x10^{-7}$mBar) in an E-beam evaporator and annealed for 1 min at 1000°C in a rapid thermal anneal (RTA) furnace with continuous Ar flow. The Van der Pauw structures were 1μm deep $1mm^2$ square mesas with $100x100\mu m^2$ contact pads on four corners. All contacts were isolated by 1μm deposited SiO_2 to reduce surface

recombination effects. Room temperature IV measurements were made on the Van der Pauw structures to study the implanted film sheet resistivity. Temperature dependent Hall measurements were also conducted with a magnetic field strength of 800mT and an input current of 10μA for free carrier concentration and mobility evaluation.

Table 1. Post-implantation activation annealing conditions

Temperature [°C]	1175	1275	1275	1375	1375
Duration [hour]	2	1	2	1	2

3. Results and Discussions

3.1. Surface roughness evolution

Fig 1 illustrates the surface morphology change of samples from each dose batch after a 1 hour PIA process at 1375°C. It can be noticed that regardless of the dose levels, all groups experienced a surface degradation, although the high dose sample surfaces were degraded more severely, indicating higher lattice damage [5, 18]. The SiO_2 capped samples were left with a higher roughness compared with the uncapped ones in all batches. A considerable amount of pits were observed on the SiO_2 capped high dose sample, resulting in a higher surface roughness value of 7.9nm compared with as implanted (0.4nm) and without a cap (4.3nm). The SiO_2 cap aimed at protecting the 3C-SiC surface led to a worse surface morphology, conflicting with the 4H-SiC case [16]. This is probably due to the interaction between SiO_2 and SiC, which was not supposed to occur at 1400°C but can be triggered by the high impurity concentration [23].

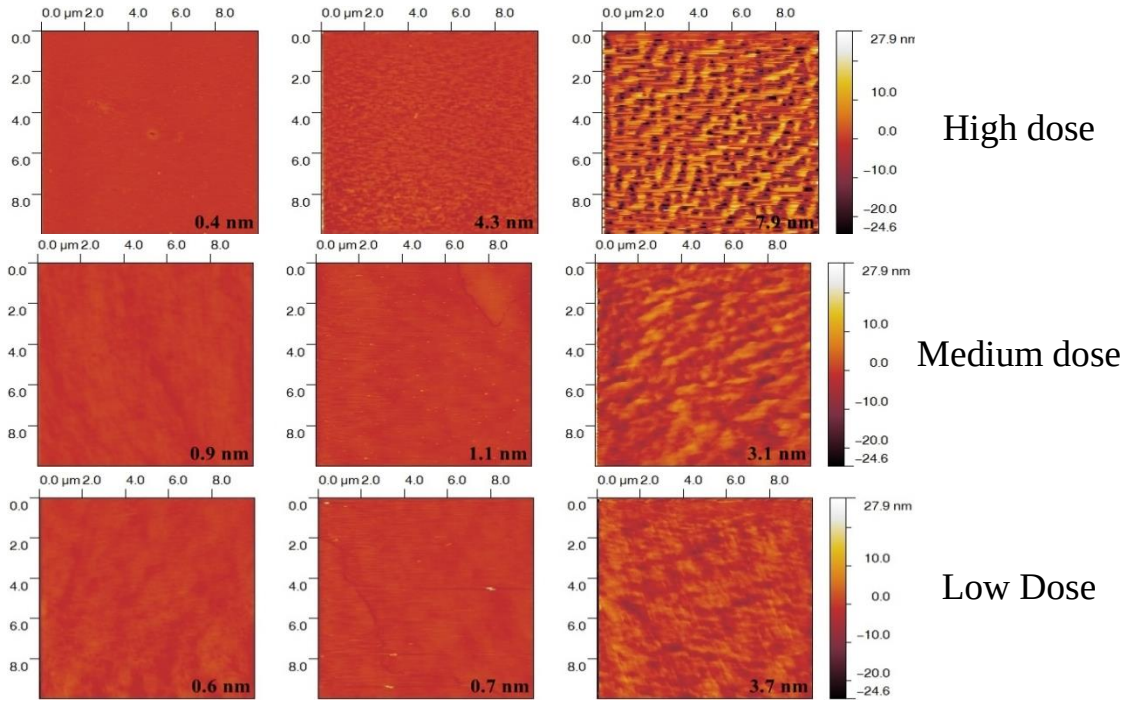

Fig 1. AFM images of 3C-SiC surface for 3 doses, inset values are RMS surface roughness values

3.2. *SIMS profiles*

After the PIA process, Second Ion Mass Spectrometry (SIMS) analysis was conducted on samples from each dose batch for the toughest PIA condition (1375°C, 2 hours) as shown in Fig 2. The profiles before PIA were not shown here but were expected to be much like the annealed ones due to the unlikely observable diffusion of nitrogen in 3C-SiC below 1800°C [17, 24]. High dose and medium dose samples both achieved an implantation depth of ~300nm with peak values around $6x10^{20}cm^{-3}$ and $4x10^{19}cm^{-3}$, respectively. For the low dose sample, a depth of ~500nm was observed and the peak doping value was determined as around $1.5x10^{19}cm^{-3}$.

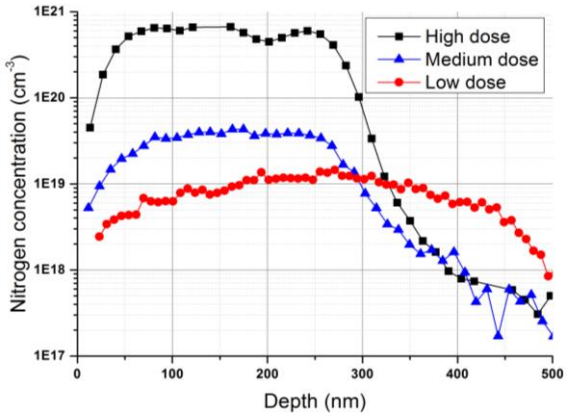

Fig 2. SIMS profiles for 3 dose samples after post implantation annealing at 1375°C for 2 hours

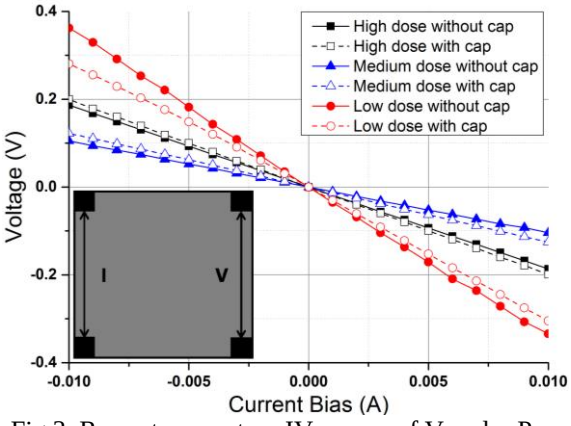

Fig 3. Room temperature IV curves of Van der Pauw structures annealed at 1375°C for 1 hour

3.3. *Room temperature IV measurements*

IV measurements were conducted on the fabricated Van der Pauw structures at room temperature. Linear IV curves were obtained for all samples (see Fig 3), indicating a typical ohmic behaviour for the Ti/Ni bilayer metal contacts. Sheet resistivity values can be calculated following equation (1) [25] below, where t is the film thickness and R is the resistance value obtained from IV measurements using the configurations shown in Fig 3.

$$\rho = \frac{\pi}{\ln(2)} tR \tag{1}$$

Due to the existence of a conduction n-type epilayer (see Fig 4), the resistance R can be considered as the result of paralleling the implantation and epilayer layer resistance and the implanted layer sheet resistivity $\rho_{sh,imp}$ can be calculated following equations (2)-(4), where T is the as grown epilayer thickness, t is the implanted film thickness, q is the electron charge, $N_{D,epi}$ is the epilayer doping $1x10^{16}cm^{-3}$ and μ_{epi} is the epilayer electron mobility $763cm^2/V.s$ [26].

$$\frac{1}{R} \approx \frac{1}{R_{imp}} + \frac{1}{R_{epi}} \tag{2}$$

$$R_{epi} = \left(\frac{\ln 2}{\pi}\right)\left(\frac{1}{T-t}\right)\rho_{sh,epi} = \left(\frac{\ln 2}{\pi}\right)\left(\frac{1}{T-t}\right)\frac{1}{qN_{D,epi}\mu_{epi}} \tag{3}$$

$$\rho_{sh,imp} = \frac{\pi}{\ln(2)} tR_{imp} = \frac{\pi}{\ln(2)} t\left(\frac{RR_{epi}}{R_{epi} - R}\right) \tag{4}$$

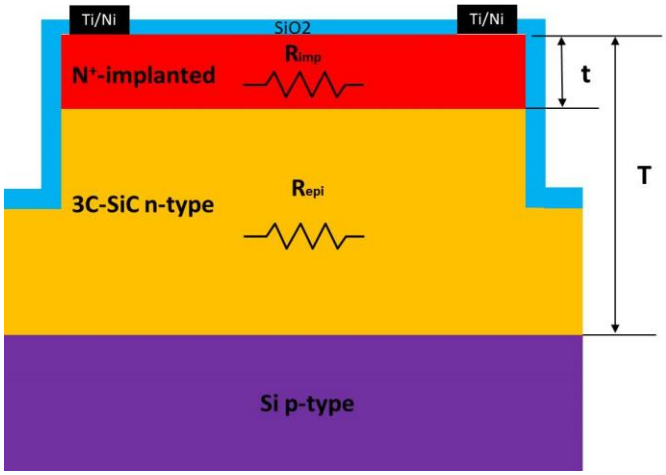

Fig 4. The implanted layer sheet resistivity calculation model

The resultant sheet resistivity values are plotted as a function of annealing conditions in Fig 5. It can be seen that for the medium dose sample, sheet resistivity values for capped and uncapped samples both gradually decrease with PIA process temperature and time durations. The lowest sheet resistivity value is ~1.4mΩ.cm obtained for the 2 hour 1375℃ annealing condition. There is more than 50% reduction comparing with the 2 hour 1175℃ process. This observed trend suggests an increasing nitrogen activation rate accomplished by raising annealing temperature as well as time durations. The SiO_2 capped samples for medium dose case all yielded a slightly higher sheet resistivity value. Relating this to the mild surface roughening observed on the SiO_2 capped sample compared with the uncapped one (2nm difference in Fig 1), it suggests that for a doping level of ~$4x10^{19}cm^{-3}$, surface roughness is an important indicator for evaluating implanted film electrical performance. For the high dose sample, the dependence of sheet resistivity on annealing conditions is much weaker that can be barely determined. This is most likely caused by the saturation of free donors in 3C-SiC [27], which means a higher concentration of active N donors is physically impossible even with higher annealing temperatures or longer time periods. There is almost no difference between capped and uncapped samples in high dose case, even with a bigger surface roughness difference (3.6nm difference) and much more obvious

surface morphology change (pits formation for SiO_2 capped sample). This indicates that in this high doping level ($6x10^{20}cm^{-3}$), surface roughness influence is negligible. The low dose samples, however, demonstrate quite random sheet resistivity behaviour. A possible explanation is that in this case, all nitrogen dopants have been activated and the fluctuations in the sheet resistivity curve actually come from the disturbance of contact resistance, which is known to be heavily dependent on the semiconductor doping level [5]. It was later found out the low dose sample contact resistance was one order of magnitude higher than the high dose and medium dose ones (not shown here). The Hall measurements results are also in favour of this assumption.

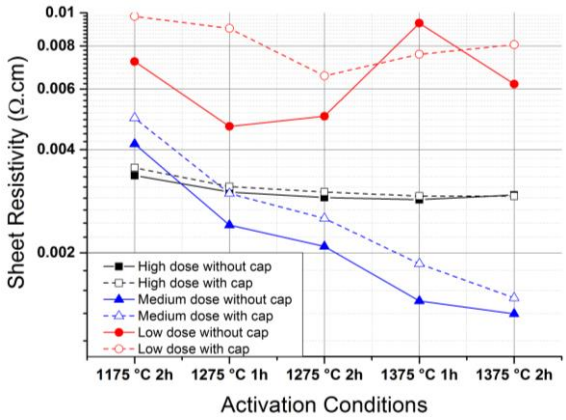

Fig 5. Effect of activation temperatures and time durations on implanted layer sheet resistivity

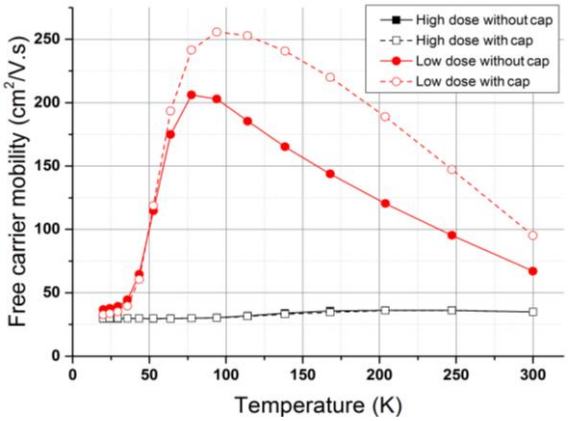

Fig 6. Temperature dependence of implanted 3C-SiC film electron mobility for high and low doses

3.4. Temperature dependent Hall measurements

Low temperature Hall measurements were made on the high dose and low dose sample annealed at 1375°C for 1 hour. The studied temperature range was between 20K and 300K and Fig 6 is the temperature dependent free carrier mobility curve. Due to a high impurity scattering effect, the high dose curve shows almost no temperature dependence. In low dose case, peak mobility value around $250cm^2/V.s$ is achieved at 100K for the uncapped sample and ~$95cm^2/V.s$ at room temperature. Compared with the literature mobility results summarised in Table 2, the values obtained in this work are in agreement with the general rule that higher impurity concentration results in reduced carrier mobility. Above 100K, free carrier mobility is dominated by phonon scattering so falls with increasing temperature roughly as $T^{-0.8}$, which is slower compared with the results obtained from more lowly doped epitaxial 3C-SiC layers, ~$T^{-1.8}$ in [28] and ~$T^{-1.2\sim1.4}$ in [29]. On the other hand, below 100K, free carrier mobility drops quickly with decreasing temperature. It is probably induced by the hopping phenomenon, which means a change of the carrier transport process from free electrons to quantum mechanical tunnelling of electrons between dopant atoms. It was previously found on heavily doped n-type and p-type 4H-SiC layers when temperature was so low that free carriers were frozen on the dopants [30, 31]. From

the almost perfectly overlapped curves for high dose sample (in Fig.6, 7, and 8), again it is confirmed that the SiO_2 cap has a negligible effect on high dose film electrical performance. While for the low dose sample, a considerable higher mobility is consistently observed in whole temperature range for the SiO_2 capped sample. This can be explained by a lower free carrier concentration, namely lower impurity scattering, found in the capped sample as seen in Fig 7. For N^+ implantation on n-type epilayer in this case, the following assumptions can be made: N_d-$N_a \approx N_d$ and $1+\alpha N_a << 4\alpha N_d$ with α defined in [32]. Then the temperature dependent semiconductor free carrier concentration $n(T)$ can be approximated as below:

$$n(T) = \frac{2\left(N_d - N_a\right)}{1+\alpha N_a + \sqrt{\left(1+\alpha N_a\right)^2 + 4\alpha\left(N_d - N_a\right)}} \approx \sqrt{(N_C/2)N_D}\, e^{-\frac{E_d}{2kT}} \tag{5}$$

Where N_D is the donor concentration, E_d is the dopant activation energy, k is the Boltzmann constant, T is the measuring temperature and N_C is the effective density of states in conduction band, which is further defined as:

$$N_c = 2(2\pi m_{d,e} kT / h^2)^{3/2} \tag{6}$$

Where $m_{d,e}$ is the 3C-SiC effective mass of density of states ($0.72m_0$) and h is the plank constant.

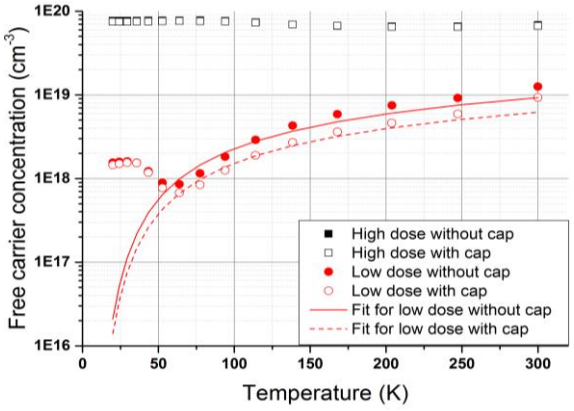

Fig 7. Temperature dependence of implanted 3C-SiC free carrier concentration for high and low doses

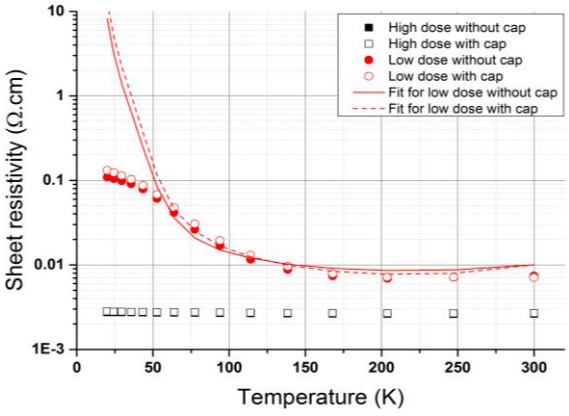

Fig 8. Temperature dependence of implanted 3C-SiC sheet resistivity for high and low doses

Using N_D and E_d as fitting parameters, best theoretical fits (red solid and dashed lines in Fig 7 and Fig 8) were achieved with $N_D \approx 1\text{x}10^{19}\text{cm}^{-3}$ and $\approx 1.5\text{x}10^{19}\text{cm}^{-3}$ for capped and uncapped low dose samples respectively, with activation energy of $E_d \approx 15$meV. Comparing with the SIMS profile in Fig 2, an N_D value of $1.5\text{x}10^{19}\text{cm}^{-3}$ indicates almost 100% activation for low dose uncapped case, while the SiO_2 capped sample yields a lower activation rate (≈67%). Previous results on the residual donor activation energy of 3C-SiC covered quite a range from 18 to 54meV using various characterizing tools as shown in Table 2. 15meV is lower than most of the literature values, but this is the first time that such a value ever reported for 3C-SiC epilayer with an

implantation level >1x10^{19}cm^{-3}. In [33] an expression was derived to show that the residual donor energy E_d in 3C-SiC decreased with the donor (Nitrogen) concentration, and when N_d was around 1x10^{19}cm^{-3}, E_d approached 0, which is similar to our case. For the high dose case, a theoretical fit is impossible indicating the degeneracy of the implanted layer. And the flat out free carrier concentration values (~7x10^{19}cm^{-3}, 12% activation rate) in the whole temperature range for the high dose sample confirms the donor saturation in 3C-SiC. The temperature dependent sheet resistivity curves (see Fig 8) were also obtained for high and low dose samples from Hall measurements. In Fig 8, even for the low dose sample, sheet resistivity starts to become temperature independent above 150K, which could mean all nitrogen donors have been thermally ionized by 150K. For both the free carrier concentration and sheet resistivity curves, the experimental data departs from the theoretical fit below 75K, which as mentioned before, is induced by hopping phenomenon [28]. A summary of the obtained results in this work are listed in Table 2 together with some literature results for comparison.

Table 2. Comparison between results obtained in this work and previous literatures on n-type 3C-SiC(100), RT is room temperature

Free carrier concentration (cm^{-3})	**Growing method**	**PIA process**	**Donor activation energy (meV)**	**RT electron mobility (cm^2/V.s)**	**Reference**
Not given (NID)	Solution	None	53 (luminescence)	Not given	[34]
1~3x10^{16} (NID)	CVD on Si	None	20 (Hall)	763	[26]
~2x10^{16} (NID)	CVD on Si	None	38 (ECR)	300	[35]
3~7x10^{16} (NID)	CVD on Si	None	18 (Hall)	400-550	[28]
1.2x10^{17} (NID)	CVD on Si	None	47 (Hall)	305	[33]
0.5~1x10^{18} (NID)	CVD on Si	None	40-50 (Hall)	120-200	[29]
~1x10^{19} (RT 1.5x10^{19}cm^{-3} nitrogen implanted)	CVD on Si and implanted	1375°C 1h with SiO_2 cap	15 (Hall)	95	This work
~1.5x10^{19} (RT 1.5x10^{19}cm^{-3} nitrogen implanted)	CVD on Si and implanted	1375°C 1h without cap	15 (Hall)	70	This work
~7x10^{19} (RT 6x10^{20}cm^{-3} nitrogen implanted)	CVD on Si and implanted	1375°C 1h with/without SiO_2 cap	Degenerated	35	This work

4. Conclusions

The dependence of the nitrogen implanted 3C-SiC layer electrical performance on the implantation doses, surface preparations and annealing conditions are investigated in this work. The surface morphology turns out to be an important indicator for low and medium doses layer properties, while not for the high dose layer. Nitrogen activation rate is found to be increasing with PIA process temperature and time durations in medium dose case. For the low and high dose samples, activation rates are not much PIA process dependent. This is due to 100% activation for low dose sample and only 12% activation rate but with donor saturation for high dose case, both confirmed by Hall measurements. By fitting the low dose sample experimental data points

with a theoretical curve, an activation energy value of ~15meV was extracted for nitrogen in 3C-SiC. The high dose film is found be completely degenerated. Also, it was found that nitrogen donors in 3C-SiC are probably fully thermally ionized at 150K. The results we obtained can be very useful for fabricating structures on heavily implanted 3C-SiC layers, which is very important during power devices manufacturing.

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