

Solid-state microwave annealing of ion-implanted 4H–SiC

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Available online 6 April 2007

Abstract

Solid-state microwave annealing was performed at temperatures up to 2120 °C for 30 s on ion-implanted 4H–SiC in N₂ ambient. The surface roughness in the samples annealed without a surface cap at 1950 °C is 2.65 nm for 10 μm × 10 μm atomic force microscopy scans. The sheet resistances measured on Al⁺- and P⁺-implanted 4H–SiC, annealed by microwaves, are lower than the best conventional furnace annealing results reported in literature. X-ray diffraction spectra indicate alleviation of the lattice damage induced by the ion-implantation and also incorporation of most of the implanted species into substitutional lattice sites.

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PACS: 01.30.Cc

Keywords: Ion-implantation; Microwave annealing; Silicon carbide; Sheet resistance; Surface roughness

1. Introduction

Silicon carbide (SiC) is the most suitable semiconductor material for fabricating devices that are used in high-power, high-frequency and chemically corrosive environments [1]. Ion-implantation is the only practical selective area doping technique available for making these devices [2,3], since diffusion coefficients of the technologically relevant dopants in SiC are very low even at temperatures as high as 1800 °C. Ion-implantation results in lattice damage and the implanted atoms predominantly reside in chemically inactive interstitial sites. Therefore, ion-implantation needs to be followed by annealing both for repairing the lattice damage and for moving the implanted dopants into chemically active substitutional lattice sites. Traditionally,

post-implantation annealing for SiC has been performed in furnaces at temperatures >1500 °C [4,5]. Often these furnaces have modest temperature ramping rates (few °C/s). This results in subjecting the SiC to high-temperatures for a prolonged period of time, hence, leading to a high degree of sublimation during annealing [6]. Due to the sublimation problem, the maximum annealing temperatures employed during conventional furnace annealing are limited to ≤1650 °C [1,2]. Consequently, sheet resistivities of p-type dopants in SiC have remained in the 10⁴ Ω/□ range [5–7]. Clearly, these values are too high for fabricating efficient SiC power devices [8]. Recently, we started investigating the use of a novel solid-state microwave technique for annealing ion-implanted SiC [9]. The microwave processing system is capable of providing a temperature rise rate of 600 °C/s and a fall rate of 400 °C/s, enabling short duration high-temperature (>1800 °C) annealing. In this paper, we present the surface morphology and electrical properties of Al⁺- and P⁺-implanted SiC material, annealed by the solid-state microwave technique.

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Report Documentation Page				Form Approved OMB No. 0704-0188	
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1. REPORT DATE 02 SEP 2007		2. REPORT TYPE		3. DATES COVERED 31-07-2006 to 01-08-2007	
4. TITLE AND SUBTITLE Solid-State Microwave Annealing of Ion-Implanted 4H-SiC				5a. CONTRACT NUMBER	
				5b. GRANT NUMBER	
				5c. PROGRAM ELEMENT NUMBER	
6. AUTHOR(S)				5d. PROJECT NUMBER	
				5e. TASK NUMBER	
				5f. WORK UNIT NUMBER	
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) George Mason University, Office of Sponsored Programs, 4400 University Dr, MSN 4C6, Fairfax, VA, 22030-4444				8. PERFORMING ORGANIZATION REPORT NUMBER ; 46232-MS-4	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Research Office, P.O. Box 12211, Research Triangle Park, NC, 27709-2211				10. SPONSOR/MONITOR'S ACRONYM(S)	
				11. SPONSOR/MONITOR'S REPORT NUMBER(S) 46232-MS-4	
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited					
13. SUPPLEMENTARY NOTES Federal Purpose Rights					
14. ABSTRACT					
15. SUBJECT TERMS					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT Same as Report (SAR)	18. NUMBER OF PAGES 4	19a. NAME OF RESPONSIBLE PERSON
a. REPORT unclassified	b. ABSTRACT unclassified	c. THIS PAGE unclassified			

2. Experimental

Multiple-energy Al^+ (10–400 keV) and P^+ (10–200 keV) implantations were performed into semi-insulating (SI) 4H-SiC. The P^+ - and Al^+ -implantations were performed at 500 °C. The P^+ - and Al^+ -implantations were designed to obtain a uniform implant concentration of $2 \times 10^{20} \text{ cm}^{-3}$ to a depth of $\sim 0.3 \mu\text{m}$ and $0.5 \mu\text{m}$, respectively. The total implant doses for the Al^+ - and P^+ -implantations were $1.37 \times 10^{16} \text{ cm}^{-2}$ and $1.3 \times 10^{16} \text{ cm}^{-2}$, respectively.

A detailed description of the solid-state microwave annealing system used in this study is provided elsewhere [9]. The annealing temperatures in this study ranged from 1750 °C to 2120 °C for the aluminum implanted samples and 1700–1950 °C for the phosphorus implanted samples. The typical anneal duration used in this work is 30 s. In this work, all the annealings were performed in a controlled atmosphere of 100% nitrogen to prevent oxidation of the SiC surface. Auger sputter profiling performed on the samples, microwave annealed in N_2 in the temperature range of 1800–2100 °C, revealed an unintentionally grown surface layer $< 80 \text{ \AA}$ thick. This surface layer consisted of silicon, carbon, oxygen and nitrogen. Apart from nitrogen, microwave annealing was also attempted in atmospheres of inert gases such as helium, argon and xenon. However, these latter gases were found to ionize (generating arcing) due to the intense microwave field in the vicinity of the SiC sample. The samples annealed in open air resulted in thick oxide layers [9].

X-ray diffraction (XRD) measurements were performed to determine the effectiveness of microwave annealing in alleviating the implantation-induced lattice damage in the SiC samples. Specular (00 ℓ) scans were obtained from an 18 kW Rigaku rotating anode diffractometer using $\text{Cu K}\alpha$ radiation. Very fine divergence and soller slits were used to increase the resolution of the XRD spectra. Surface roughness of the microwave annealed samples was evaluated using tapping mode atomic force microscopy (AFM) on $10 \mu\text{m} \times 10 \mu\text{m}$ areas. Hall measurements were performed, using the van der Pauw technique, for electrical characterization of the material, after depositing Ni (100 nm) and Ti/Al (20 nm/100 nm) contacts for the P^+ - and Al^+ -implanted samples, respectively. The contacts

were made ohmic by alloying at 1000 °C for 1 min in 1 atm UHP argon.

3. Results and discussion

3.1. Surface roughness of the microwave annealed samples

The RMS surface roughness extracted from $10 \mu\text{m} \times 10 \mu\text{m}$ tapping mode AFM scans of microwave annealed samples, as a function of annealing temperature, for 30 s microwave annealings is provided in Table 1. AFM images of an 1850 °C/35 s microwave annealed sample, and a 1500 °C/15 min furnace-annealed sample are provided in Fig. 1. The samples used for this study were Al^+ -implanted 4H-SiC. Furrows running along the face of the furnace-annealed sample can be clearly seen in Fig. 1. The formation of these furrows is believed to be due to the desorption and re-deposition of Si and C containing species from the SiC surface [10], possibly due to the long 15 min annealing duration. The surface of the microwave annealed sample shown in Fig. 1 is relatively smooth. It can be seen from the data presented in Table 1 that the RMS roughness values for the 1800–2000 °C microwave annealings are in the range of 2–4 nm. This is 1.5–3 times the measured surface roughness of the as-implanted sample (1.44 nm). Upon comparing these roughness values with those obtained after conventional uncapped and AlN/graphite capped annealings (not shown), the following observations can be made. Roughness increase in the microwave annealed samples is much lower than in the uncapped conventional furnace-annealed samples [11,12], which show an increase in roughness of ~ 15 times the value of the as-implanted sample for 1700 °C/15 min annealing [7]. Roughness

Table 1
RMS roughness extracted from $10 \mu\text{m} \times 10 \mu\text{m}$ tapping mode AFM scans of Al^+ -implanted SiC

Sample details	RMS roughness (nm)
As-implanted	1.44
1800 °C/30 s	2.18
1950 °C/30 s	2.65
2000 °C/30 s	4.29

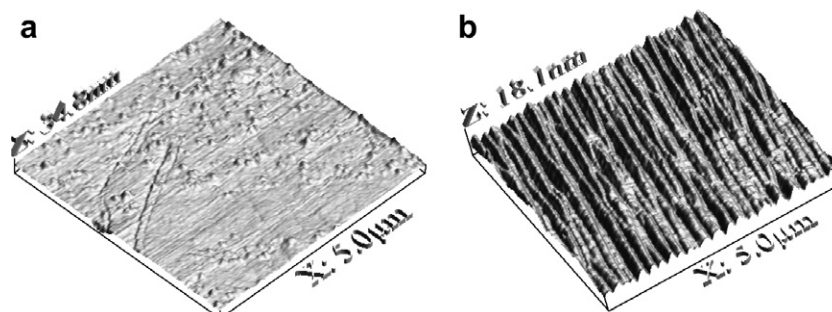


Fig. 1. Contact mode AFM images of (a) 1850 °C/35 s microwave annealed sample and (b) 1500 °C/15 min furnace annealed sample.

increase after uncapped microwave annealing of this work is comparable with the surface roughness obtained after furnace annealing using a graphite or AlN cap [13,14]. The results obtained indicate the attractiveness of high-temperature short duration microwave annealing for obtaining good surface morphology.

3.2. Electrical characteristics of aluminum and phosphorus implanted SiC

For the 4H-SiC, the sheet resistance measured at room temperature (RT), by the van der Pauw Hall technique, as a function of the annealing temperature for 30 s annealings is shown in Fig. 2 for the P⁺- and Al⁺-implantations. The variation of the electron/hole mobility, for the P⁺/Al⁺-implantation, respectively, with increasing annealing temperature is shown in Fig. 3. It can be observed from Figs. 2 and 3 that for both the aluminum and phosphorus implantations, increasing the microwave annealing temperature, in general, steadily lowers the sheet resistance, while increasing the carrier mobility. The microwave annealing yields sheet resistances, which are about one order of magnitude lower than those obtained after conventional fur-

nace annealing, which typically yields sheet resistances in the range of $10^4 \Omega/\square$ for the Al⁺-implantations [3–5]. Microwave annealing the aluminum implanted material at 2050 °C yields a sheet resistance of 2.81 k $\Omega/\square$ and a hole mobility of 6.2 cm²/V s. This is one of the best reported sheet resistance values for ion-implanted p-type SiC. Also, the increase of the hole mobility with increasing annealing temperature is an indication that an increasing number of implantation-induced defects is annealed.

For phosphorus implanted SiC, the microwave annealing performed at 1950 °C for 30 s yields a sheet resistance of 14 $\Omega/\square$ and an electron mobility of 100 cm²/V s. These results once again are the best reported values for implanted n-type (0001) oriented SiC. The conventional furnace annealing for n-type SiC typically yields sheet resistance values in excess of 100 $\Omega/\square$ [6,15,16].

In general, the carrier mobility at room temperature is limited by scattering of carriers by ionized impurities and also by defects in the crystal lattice. An increased implanted dopant substitutional activation with increasing annealing temperature results in an increased ionized impurity scattering and hence decreasing the carrier mobility. This initial trend (decrease of the carrier mobility with increasing sheet carrier concentration) can be seen in Fig. 3 for the phosphorus implanted samples for annealing temperatures <1800 °C. However, for annealing temperatures >1800 °C, the carrier mobility (for both Al⁺- and P⁺-implanted samples) kept increasing with increasing annealing temperature. This trend is believed to be due to a more effective annealing of implantation-induced lattice damage at these annealing temperatures. The X-ray diffraction and Rutherford backscattering spectrometry-channeling (RBS-C) measurements on the microwave annealed samples also indicated a lattice quality similar to the virgin sample.

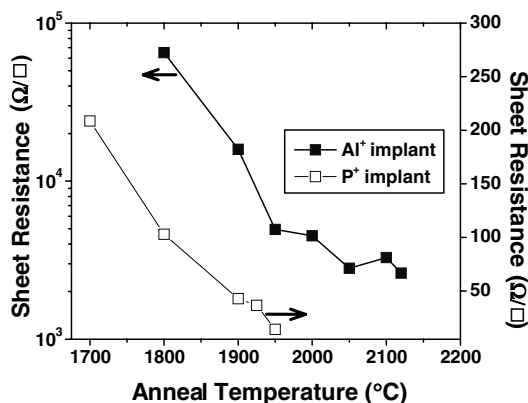


Fig. 2. Sheet resistance of aluminum and phosphorus implanted 4H-SiC, as a function of annealing temperature, for 30 s microwave annealing.

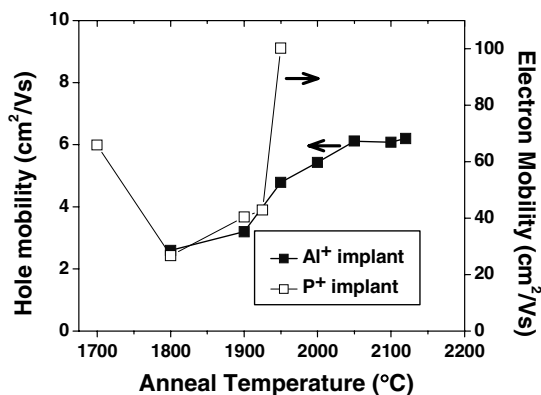


Fig. 3. Measured electron and hole mobility in the phosphorus and aluminum implanted 4H-SiC, respectively, as a function of annealing temperature, for 30 s microwave annealing.

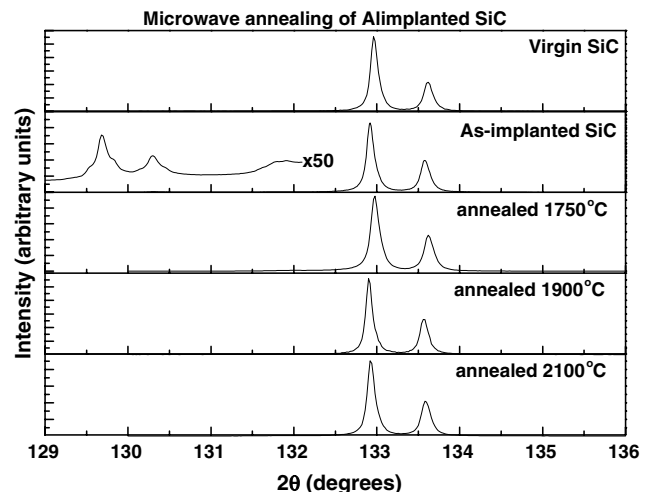


Fig. 4. X-ray diffraction scans of virgin, Al⁺ as-implanted and implanted/microwave annealed 4H-SiC samples.

3.3. Lattice quality of the aluminum implanted 4H–SiC

Fig. 4 shows the (0,0,12) out-of-plane X-ray diffraction scans for virgin, Al⁺ as-implanted, and implanted/microwave annealed 4H–SiC samples. Highly resolved X-ray diffraction peaks are obtained for the two wavelengths CuK α_1 and CuK α_2 . For the as-implanted sample, in addition to the main SiC (0,0,12) reflection, additional peaks can be observed on the low angle side of the main peak. The existence of the additional lower angle peaks with CuK α_1 and CuK α_2 components resolved indicates the presence of a sub-lattice with a larger d-spacing, as compared to the virgin sample, caused by the implanted Al occupying interstitial lattice positions [17]. The *c*-parameter of the low angle sub-lattice peak is found to be 10.2120 Å, which is 0.1314 Å larger than that of the 4H–SiC (0,0,12) peak. Furthermore, since the sub-lattice peaks are highly resolved for CuK α_1 and CuK α_2 wavelengths, it is an indication that a uniform Al implantation has been accomplished. The XRD profiles after microwave annealing show a disappearance of the sub-lattice peak indicating the movement of the implanted aluminum atoms from interstitial to substitutional lattice sites. Thus, it can be concluded that the microwave annealing was successful in activating the implanted dopants into electrically active substitutional sites and in relieving the implantation-induced lattice damage.

One of the problems with the solid-state microwave annealing at this time is SiC surface sublimation. The sublimation uniformly occurs all over the sample yielding smooth surfaces unlike furnace annealing, which yields rough surfaces. The sublimation problem can be solved by depositing a graphite cap on the implanted SiC surface prior to annealing. Our preliminary results on the use of the graphite cap indicate that the cap holds good for microwave annealing temperatures as high as 2100 °C.

4. Conclusion

Solid-state microwave annealing is an attractive method for performing high-temperature, short duration annealing of implanted SiC. Using this technique, temperatures as high as 2100 °C can be reached with a ramp-up rate of >600 °C/s and a fall rate of 400 °C/s. For 2050 °C/30 s microwave annealing in a nitrogen ambient, the RMS surface roughness is comparable to the surface roughness in capped samples subjected to conventional furnace annealing. Sublimation during microwave annealing can be prevented, at temperatures as high as 2100 °C, using a graphite cap on SiC. Electrical characterization of both

p-type (Al) and n-type (P) implanted material, subjected to microwave annealing, yielded record breaking low sheet resistance values, and high carrier mobilities. XRD measurements indicated an almost complete movement of the implanted species into substitutional lattice sites, and in relieving the implantation-induced compressive strain in the crystal lattice. This is an indication that the microwave annealing is effective in both activating the implanted dopants and healing the lattice damage.

Acknowledgements

We are grateful to Dr. A.V. Davydov of NIST for allowing the use of NIST facilities for performing the characterization. This material is based on the GMU work supported by Army Research Office (Dr. Prater) under Grant # W911NF-04-1-0428 and by a subcontract from LT Technologies under NSF SBIR Grant # 0539321.

References

- [1] C.E. Weitzel, J.W. Palmour, A.H. Carter, K. Moore, K.K. Nordquist, S. Allen, C. Thero, M. Bhatnagar, IEEE Trans. Electron Dev. 43 (1996) 1732.
- [2] J.B. Tucker, S. Mitra, N. Papanicolaou, A. Siripuram, M.V. Rao, O.W. Holland, Diamond Relat. Mater. 11 (2002) 392.
- [3] S.H. Ryu, S. Krishnaswami, M. O'Loughlin, J. Richmond, A. Agarwal, J. Palmour, A.R. Hefner, IEEE Electron Device Lett. 25 (2004) 556.
- [4] Y. Negoro, T. Kimoto, H. Matsunami, Mat. Sci. Forum 483–485 (2005) 599.
- [5] J.B. Tucker, M.V. Rao, M.C. Ridgway, O.W. Holland, N. Papanicolaou, J. Mittereder, J. Appl. Phys. 86 (1999) 752.
- [6] J. Senzaki, K. Fukuda, K. Arai, J. Appl. Phys. 94 (2003) 2942.
- [7] M. Rambach, F. Schmid, M. Krieger, L. Frey, A.J. Bauer, G. Pensl, H. Ryssel, Nucl. Instr. and Meth. B 237 (2005) 68.
- [8] H. Matsunami, Jpn. J. Appl. Phys. 43 (2004) 6835.
- [9] S.G. Sundaresan, M.V. Rao, Y.-L. Tian, J.A. Schreifels, M.C. Wood, K.A. Jones, A.V. Davydov, J. Electron. Mater., in press, doi:10.1007/s11664-006-0032-1.
- [10] M.A. Capano, S. Ryu, M.R. Melloch, J.A. Cooper Jr., M.R. Buss, J. Electron. Mater. 27 (1998) 370.
- [11] T. Kimoto, N. Inoue, Phys. Status Solidi A 162 (1997) 263.
- [12] M.A. Capano, S. Ryu, J.A. Cooper Jr., M.R. Melloch, K. Rottner, S. Karlsson, N. Nordell, A. Powell, D.E. Walker Jr., J. Electron. Mater. 28 (1999) 214.
- [13] K.A. Jones et al., J. Electron. Mater. 31 (2002) 568.
- [14] K.V. Vassilevski et al., Semicond. Sci. Technol. 20 (2005) 271.
- [15] E.M. Handy, M.V. Rao, O.W. Holland, K.A. Jones, M.A. Derenge, N. Papanicolaou, J. Appl. Phys. 88 (2000) 5630.
- [16] F. Schmid, M. Laube, G. Pensl, G. Wagner, M. Maier, J. Appl. Phys. 91 (2002) 9182.
- [17] J. Simonson, S.B. Qadri, M.V. Rao, R. Fischer, J. Grun, M.C. Ridgway, Appl. Phys. A. 81 (2005) 601.