Effect of RF power and gas flow ratio on the growth and morphology of the PECVD SiC thin films for MEMS applications

BHAVANA PERI, BIKASH BORAH and RAJ KISHORA DASH*

School of Engineering Sciences and Technology, University of Hyderabad, Prof. C R Rao Road, Gachibowli, Hyderabad 500046, India

MS received 11 April 2014; accepted 11 August 2014

Abstract. Low-temperature plasma enhanced chemical vapour deposition (PECVD) deposited silicon carbide (SiC) thin films are promising materials for the development of high-temperature working microelectromechanical system (MEMS) owing to their excellent mechanical properties, non-corrosive nature and ability to withstand high temperature. However, the surface roughness of such thin films is the main obstacle to achieve thicker thin films for MEMS applications as the surface becomes more rougher with the increase in the thickness of PECVD SiC thin films. Therefore, in this present study, thicker SiC thin films were deposited by PECVD process by using CH₄ and SiH₄ as the precursor gases in the presence of Ar as the carrier gas and two process parameters, i.e., radio frequency (RF) power with mixed frequency condition and flow ratio of silane to methane were varied by keeping the temperature and pressure constant to investigate the influence of these parameters on the growth rate, surface roughness and morphology of SiC thin films. It was observed that both the RF power (with the mixed frequency condition) and flow ratio of SiH₄/CH₄ can control the growth rate, surface roughness and morphology of the PECVD SiC thin films. Higher the carbon content in the thin films the surface became more smoother, whereas the surface became for rougher by increasing the RF power.

Keywords. PECVD; SiC; thin films; morphology; MEMS.

1. Introduction

Silicon carbide (SiC) thin films are potential candidates for several applications ranging from protective coatings to microelectromechanical system (MEMS) devices. The characteristic properties of withstanding high temperatures, corrosive environments along with excellent mechanical and dielectric properties have made SiC a suitable alternative material to silicon for such harsh environment applications.^{1,2} There are several methods such as low-pressure chemical vapour deposition (LPCVD),^{1,3} plasma-enhanced chemical vapour deposition (PECVD),⁴ sputtering,⁵ magnetron sputtering,⁶ and a few others^{2,7} and those were generally used for the growth of SiC thin films. Among all of them, LPCVD and PECVD are the two most commonly used methods for the deposition. However, LPCVD is a high-temperature process and mostly used for the growth of crystalline SiC thin films which has excellent mechanical properties such as hardness and young's modulus,⁴ whereas PECVD is a low-temperature process for growth of amorphous SiC thin films. Since, low-temperature process is more compatible with the current microfabrication technology and also has the capability to grow on different substrates or underlying thin film layers, the PECVD process is more suitable for the MEMS applications.

All the LPCVD SiC thin films exhibit polycrystalline texture with the most dominant phase is the 3C-SiC (111). The temperature range for such LPCVD processes is generally in the range of 900–1200°C.^{1–3} It was reported that the LPCVD process were generally used to deposit thick and uniform films on silicon substrates and it was seen that the flow ratio of SiH₂Cl and C₂H₂ plays a key role in controlling the deposition rate, surface morphology and microstructure.⁸ It was observed that the flow ratios can influence the chemical inertness properties of the deposited films. Fu *et al*⁹ had also shown that temperature and pressure have a significant role during the deposition of the LPCVD SiC thin films. The growth rate and residual stress of the thin films can be controlled by varying the pressure and temperature. Liu *et al*¹⁰ had also mentioned that the residual stress of the LPCVD grown SiC thin films on Si is high due to the variations in the lattice constants of the two materials.

In contrast to the LPCVD process, the PECVD process generally requires low temperature typically in the range of 200–400°C.^{4,11} Also, due to more adjustable growth parameters such as temperature, pressure, RF power and gas flow rates, the material properties (such as young's modulus, hardness, etc.) of the deposited SiC thin films can be controlled. It was reported by Liu *et al*⁴ that the residual stress of the SiC thin films were relatively lower and more controllable in PECVD method.⁴ Further, the deposited PECVD a-SiC thin films coefficient of thermal expansion (CTE) was seen very much matched with that of Si apart from the

*Author for correspondence (rkdse@uohyd.ernet.in, drrajdash@gmail.com)

ability to be deposited over various substrates. Several researchers had also reported that the composition of the SiC films can be controlled by varying the ratio of the methane and silane gas flow.^{12,13} Other properties such as the resistivity of the SiC film can also be improved by using ammonia gas as a dopant during the growth process^{14,15} in such deposition process.

In more recent work, Pelegrini *et al*¹¹ had shown that the amorphous hydrogenated SiC thin films deposited in a hydrogen-rich environment were more sensitive to an increase in the RF power. They had also mentioned that by increasing the RF power, the deposition rate can be doubled and the stoichiometry of the thin films can also be controlled. Liu *et al*⁴ had reported that the surface morphology was highly dependent on the growth temperature. Higher the growth temperature thin films became more rougher due to the crystalline in nature. However, the smooth surface of the SiC thin film was grown at 300°C and 300 W RF power. The LPCVD grown SiC thin films were also more tensile stress as compared to the PECVD samples. Prado *et al*¹⁶ studied the effects of the RF power, flow ratio and carrier gas (H_2) concentration on the properties of the SiC thin films by FTIR and reported that an increase in the RF power increased the carbon content of the SiC thin films and also use of hydrogen as a carrier gas the properties of the SiC thin films can be more controlled. Ricciardi *et al*¹³ had mentioned that by varying the gas flow ratio of CH_4 and SiH_4 , the deposition rate can be improved. They had also reported that PECVD SiC thin films surfaces were more smooth and the average root mean square (RMS) roughness of the deposited thin films were around 3 nm. It was also reported that by annealing the thin films the residual stress can be reduced.¹⁷ The stoichiometry of the thin films changed when the samples were annealed at 1300°C in an Ar atmosphere for 30 min. This process causes diffusion of Si from the substrate into the thin film there by causing the formation of Si nanocrystallites and SiC crystallites.¹⁷ It was also explained by Kim *et al*,¹⁸ that the annealing of the films causes the breaking of the Si-H and C-H_n bonds there by reducing the H_2 content in the thin films which improves the optical properties of the thin films. Awad *et al*¹⁹ mentioned that the roughness of the SiC thin films can be reduced to ~1 nm due to film densification when the samples were annealed. Therefore, more studies are required to understand the effects of RF power, flow ratio of the gases on the growth and morphology of the low-temperature PECVD process to control the mechanical and other properties for high-temperature MEMS applications.

In the present work, a low-temperature PECVD deposition process was carried out and the influence of deposition parameters such as RF power, mixed frequency and flow ratio of the gases on the growth and morphology of the SiC thin film are reported by keeping the deposition temperature and pressure constant. All the SiC thin films were deposited by using silane (SiH_4) and methane (CH_4) as the precursor gases by considering Argon (Ar) as the carrier gas. The deposition parameters (i.e., RF Power and flow ratio of the gases) were

optimized to get SiC thin films with higher growth rate and higher thickness. All the as-deposited SiC thin films were characterized by using X-ray diffraction (XRD) for structural analysis, cross-sectional field emission scanning electron microscopy (C-FESEM) for thickness, Raman spectroscopy for structural and Fourier transform infrared spectroscopy (FTIR) for bonding types in the samples. Finally, all the as-deposited samples were analysed by using atomic force microscopy (AFM) for growth and morphology variations due to different RF power, mixed frequency and flow ratio.

2. Experimental

All the SiC thin films were deposited by using a PECVD system (Oxford Instruments Plasmalab System 100) on a 2 in *n*-type Si(100) wafer (approximate substrate thickness of 250–300 μm) with the substrate resistivity in the range of 1–10 Ωcm . All the wafers prior to deposition were initially cleaned in Piranha ($H_2SO_4 : H_2O_2$ in the ratio 3 : 1 at a temperature of 80°C for 10 min) then rinsed thrice with the deionized (DI) water. Then all the samples were dipped in a hydrofluoric acid for 10 s in order to remove any native oxide present on the surface. Finally, they were rinsed with DI water and then blown with dry nitrogen gas. Prior to the deposition, the PECVD chamber was cleaned by using CF_4 plasma for 1.5 h and then the chamber was conditioned by depositing SiC using silane (SiH_4) and methane (CH_4) for 2 min at a pressure of 1000 mTorr, RF power of 300 W (Watt) and temperature of 350°C. The condition run is necessary in order to clear out all the fluorine molecules in the chamber. The substrate was then loaded into the chamber by using a load lock chamber and the deposition was carried out. Si substrate was heated to the desired temperature (380–400°C) before the gases were released into the chamber. All the gases (SiH_4 , CH_4 and Argon) get mixed prior to being injected into process chamber via a ‘showerhead’ gas inlet in the top electrode chamber. Depositions were carried out for a varying time range of 22–45 min to achieve SiC thin films with different thicknesses.

The PECVD deposition process parameters such as temperature, pressure, RF power and gas flow ratio which influence the growth rate along with the morphology of the SiC thin films. Therefore, the different process parameters were investigated in this study and are listed in table 1.

Table 1. Range of deposition parameters for deposition of PECVD SiC thin films.

Parameters	Range
Pressure	1200 mTorr
RF power	300–400 W (HF) and mixed (HF and LF)
Temperature	380–400°C
Gas flow ratio — $SiH_4 : CH_4$	1 : 4–1 : 7
Carrier gas (Ar) flow rate	700 sccm

Table 2. Growth rate, thickness and compositional C/Si ratio for all the as-deposited PECVD SiC samples (pressure = 1200 mTorr).

Sample no.	Temperature (°C)	RF power (W)	Flow ratio (SiH ₄ /CH ₄ /Ar) (sccm)	Deposition rate (nm min ⁻¹)	Thickness ($T = \mu\text{m}$) $T \pm 0.25 \mu\text{m}$	Cross-sectional EDS (C/Si) (C/Si $\pm 10\%$ variation)
1	380	400 W	20/100/700 (1 : 5)	~63	2.84	33/67
2	380	300 W	20/100/700 (1 : 5)	~67	3.03	35/65
3	380	300 W (HF) for 10 s; 200 W (LF) for 20 s	20/100/700 (1 : 5)	~30	1.35	39/61
4	400	350 W	25/100/700 (1 : 4)	~82	1.8	35/65
5	400	350 W	15/100/700 (1 : 7)	~34	0.74	37/63

Figure 1. FESEM cross-sectional micrographs and corresponding EDS spectrum of PECVD as-grown SiC thin films: (a) sample 2 (highest thickness of ~3 μm) and (b) sample 4 (highest growth rate (82 nm min⁻¹) with thickness of ~1.8 μm).

The pressure was kept constant at 1200 mTorr for all the depositions since it was reported in various literatures that it is the optimum value for better uniformity of the films.^{4,13,20} First set of SiC samples were deposited by varying the RF power for three different values, i.e., 400, 300 W and mixed frequency (300 W (high frequency (HF)) for 10 s; 200 W (low frequency (LF)) for 20 s), while keeping the other process parameters constant (temperature 350°C, flow ratio 1 : 5 ($\text{SiH}_4 : \text{CH}_4$ was 20 : 100 sccm), 700 sccm of Ar gas and pressure at 1200 mTorr). Another set of SiC samples were grown by varying the gas flow ratio for three different flow rates and by keeping the RF power constant at 350 W (HF), pressure at 1200 mTorr and temperature at 400°C. In this case, the CH_4 and Ar flow rates were fixed constant at 100 and 700 sccm, respectively, while the SiH_4 flow rate was varied from 25 to 15 sccm (the ratio of the gases $\text{SiH}_4 : \text{CH}_4$ varies from 1 : 4 to 1 : 7). There was no change in the chamber pressure even if flow ratios were changed from 25 to 15 sccm.

The thickness of all the as-deposited SiC films was measured by using the cross-sectional FESEM (C-FESEM) (Zeiss Gemini) and the attached EDX (Oxford Instruments) detector for analysing the composition of the cross-sectional thin films. The uniformity and the roughness of the deposited layers were analysed by using AFM (SPI 400, SEIKO Instruments). The structure and bonding were characterized by using X-ray diffraction (XRDD8, Bruker; $\text{CuK}\alpha$ radiation), Fourier transform infrared spectroscopy (FTIR, Nicolet 380, Thermo Scientific) and Micro-Raman spectroscopy (Witec-300, Focus Innovation 532 nm laser wavelength). The absorption spectra for FTIR was obtained after baseline and substrate absorption subtraction.

3. Results and discussion

The growth rate, thickness and composition of all the SiC samples are listed in table 2. From table 2, it is clear that sample 2 has the highest thickness, i.e., 3.03 μm with a deposition rate of $\sim 67 \text{ nm min}^{-1}$ whereas sample 4 has the highest deposition rate, i.e., $\sim 82 \text{ nm min}^{-1}$. The cross-sectional FESEM images for samples 2 and 4 are shown in figure 1. Higher thickness SiC thin film having thickness of $\sim 3 \mu\text{m}$ was achieved in a single run of 45 min with a growth rate of $\sim 67 \text{ nm min}^{-1}$ as shown in figure 1a. The maximum growth rate in the order of 82 nm min^{-1} was achieved with a thickness of $\sim 1.8 \mu\text{m}$ in a deposition time of 22 min as shown in figure 1b. The cross-sectional EDS analysis of the samples confirmed that all the samples are mostly Si rich.

Figure 2a shows the XRD plots for all the samples. It is seen from figure 2a that all the SiC thin films are amorphous in nature. Figure 2b shows the Raman spectra for all the SiC thin films samples. The peaks appearing in the low frequency range around 285 and 491 cm^{-1} are due to the amorphous Si-Si transverse acoustical (TA) and transverse optical (TO) mode of vibrations, respectively, sharp peak at around 520 cm^{-1} is due to the crystalline Si-substrate while

Figure 2. (a) XRD analysis, (b) Raman spectra and (c) FTIR spectra of all the as-deposited SiC thin films.

the peaks at 910 cm^{-1} are due to the second order transversal optical mode of Si–Si vibrations. The peaks at $760\text{--}790\text{ cm}^{-1}$ can be assigned to amorphous Si–C vibration.^{13,21} From the Raman analysis, it is clear that all the SiC thin films are showing broad bands peaks due to the inelastic scattering and hence all the thin films are amorphous in nature which also agrees with the XRD results.¹³

Figure 2c shows the FTIR spectra for all the samples which represent the different mode of vibrations which are

related to different bondings present in the SiC thin films. The significant peaks are those related to the stretching mode of vibration of Si–C bonds whose wavenumbers are in the range of $740\text{--}800\text{ cm}^{-1}$, the wagging mode of Si–H bonds are present around 600 cm^{-1} and the peaks around 1000 cm^{-1} are related to the wagging mode of Si–CH_n bonds. It is seen from figure 2c that corresponding to the change in the SiH₄/CH₄ flow ratio from 1:4 to 1:7 (comparing sample 4 and sample 5, mentioned in table 2)

Figure 3. AFM images of all the as-deposited SiC thin films: (a) sample 1, (b) sample 2, (c) sample 3, (d) sample 4 and (e) sample 5.

there is a peak shift from 740 to 765 cm^{-1} , respectively, which confirms the higher carbon content thin films^{11,22,23} with a decrease in the hydrogen content. The peaks around 2350 are due to the atmospheric CO_2 .²⁴ In the range of $2800\text{--}3000\text{ cm}^{-1}$, a very low signal is observed due to the vibrational stretching of the C-H_n bonds.

Figure 3 shows the AFM images of all the as-deposited SiC samples. The RMS value of the roughness is calculated over a scan area of $10 \times 10\text{ }\mu\text{m}^2$. From figure 3,

it is observed that the RMS surface roughness of the SiC thin films are less than $\sim 5\text{ nm}$. However, there is a small range of variation in the RMS values which indicates that the RF power, mixed frequency conditions and flow ratio ($\text{SiH}_4:\text{CH}_4$) can control the surface morphology and roughness. It is seen from figure 3 that as the RF power increases the surface roughness of the SiC thin films also increases and surface becomes more rougher. The differences in RMS are $\sim 0.8\text{ nm}$ (comparing sample 1 and sample 2).

Figure 4. AFM images of the growth morphology and microstructure of as-deposited SiC thin films: (a) sample 1, (b) sample 2, (c) sample 3, (d) sample 4 and (e) sample 5.

However, by considering the mixed frequency condition with lower RF power option, the surface roughness can be controlled further (comparing sample 2 and sample 3). In contrast to this, by increasing the flow ratio ($\text{SiH}_4 : \text{CH}_4$) the samples become more smoother. There is a decrease in the surface roughness by order of ~ 1.69 nm (comparing samples 4 and 5). This may be attributed to the fact that with higher RF power, both SiH_4 and CH_4 gases dissociate much faster which leads to a higher concentration of reactive ions in the plasma with a direct impact on the deposition rate and may increase the surface roughness by increasing the deposition rate. However, with the mixed frequency condition the dissociation of the CH_4 precursors is much faster as compared with SiH_4 , that may help to increase the carbon content in the thin films (table 2) and hence improve the surface roughness as compared with only low RF power condition.²⁰ It is also observed that by increasing the flow ratio (samples 4 and 5), the flow rate of methane is higher as compared with silane. Therefore, higher incorporation of CH_4 lowers the concentration of SiH_4 and SiH_n radicals which decreases the Si–C bond due to less availability of silicon.^{25,26}

Hence, surface roughness can be controlled by varying the deposition parameters such as RF power, mixed frequency and flow ratio. Such deposition parameters can also effect the growth morphology and microstructure of the SiC thin films. Figure 4 shows the AFM images of the microstructure and growth morphology of the SiC thin films for different SiC thin films (samples 1–5). It is observed from figure 4a–e that the microstructure and growth morphology of the SiC thin films are different. In the case of constant flow ratio ($\text{SiH}_4 : \text{CH}_4 = 1 : 5$) figure 4a–c the microstructure of the thin films are different as compared with the constant power (350 W) as shown in figure 4d and e. More study is still undergoing to understand the underlying mechanism of such dependency of process parameters on the growth morphology and microstructure. Once, the optimized process conditions are investigated, the PECVD SiC thin films materials properties can be tuned as per the requirement for several high temperature applications.

4. Conclusions

A low-temperature PECVD process for deposition of thicker SiC thin film was described in this paper with the influence of deposition parameters (RF power, mixed frequency and gas flow ratios) on the growth rate, morphology and microstructure. All the samples deposited using the PECVD process were amorphous hydrogenated SiC(a:SiC–H) which was confirmed from the XRD, Raman and FTIR analysis. All the as-deposited samples were Si rich from the EDX and Raman spectroscopy. Higher growth rate (~ 82 nm min^{-1}) was achieved with an average surface roughness of ~ 2.4 nm. The surface roughness increased with increase in the RF power whereas the mixed frequency condition can help to reduce the surface roughness further with increase in the carbon content in the sample. In contrast to this, increase

in the flow ratio the surface became more smoother. Hence, the RF power, mixed frequency and CH_4 flow rate can be properly tuned to obtain smoother surface, higher growth rate and also to control the growth and morphology of the PECVD SiC thin films for future high-temperature MEMS applications.

Acknowledgements

The financial support was provided by ADA: NP-MASS under Grant no. 1.27 to carry out this work. The PECVD depositions were carried out at CeNSE, IISc, Bangalore. We would like to thank the clean room manager Dr Raghavan and Aditi for their clean room assistance during the depositions of samples.

References

1. Azevedo R G, Jones D G, Jog A V, Myers D R, Chen L, Fu X, Mehregany M, Wijesundara M B J and Pisano A P 2007 *IEEE Sens. J.* **7** 568
2. Wu C, Zorman C A and Mehregany M 2006 *IEEE Sens. J.* **6** 316
3. Roy S, DeAnna R G, Zorman C A and Mehregany M 2002 *IEEE Trans. Electron. Devices* **49** 2323
4. Liu L, Tang W, Zheng B and Zhang H 2011 *Proceedings of the 2011 6th IEEE international conference on nano/micro-engineered and molecular systems*, February 20–23, Kaohsiung, Taiwan, p 146
5. El Khalfi A, El Maati Ech-chamikh, Ijdiyaou Y, Azizan M, Essaifi A, Nkhaili L and Outzourhit A 2014 *Spectrosc. Lett.: Int. J. Rapid Commun.* **47** 392
6. Chandra S, Kumar S and Bose G 2012 *J. Micromech. Microeng.* **22** 025010
7. Sarro P 2000 *Sens. Actuators* **82** 210
8. Zorman C A, Rajgopal S, Fu X A, Jezeski R, Melzak J and Mehregany M 2002 *Electrochem. Solid-State Lett.* **5** 99
9. Fu X, Dunning J L, Zorman C A and Mehregany M 2005 *Sens. Actuators A* **119** 169
10. Liu F, Carraro C, Pisano A P and Maboudian R 2010 *J. Micromech. Microeng.* **20** 035011
11. Pelegrini M V, Rehder G P and Pereyra I 2010 *Phys. Status Solidi C* **7** 786
12. Guo H, Wang Y, Chen S, Zhang G, Zhang H and Li Z 2006 *Proceedings of the 1st IEEE international conference on nano/microengineered and molecular systems*, January 18–21, Zhuhai, China
13. Ricciardi C, Primiceli A, Germani G, Rusconi A and Giorgis F 2006 *J. Non-Cryst. Solids* **352** 1380
14. Huran J, Hotovy I, Kobzev A P and Balalykin N I 2004 *Thin Solid Films* **459** 149
15. Milan P, Vladimír S, Miroslav M, Jozef H and Juraj O 2011 *34th international spring seminar on electronics technology (ISSE)*, 11–15 May, Trtatanska Lomnica
16. Prado R J, Fantini M C A, Tabacnills M H, Cardoso C A V, Pereyra I and Flank A M 2001 *J. Non-Cryst. Solids* **283** 1

17. Kunle M, Janz S, Nickel K G and Eibl O 2011 *Phys. Status Solidi A* **208** 1885
18. Kim Y, Cho S, Hong B, Suh S, Jang G and Yoon D 2002 *Mater. Trans.* **43** 2058
19. Awad Y, El Khakani M A, Brassard D, Smirani R, Camiré N, Lessard M, Aktik C, Scarlete M and Mouine J 2010 *Thin Solid Films* **518** 2738
20. Avram M, Avram A, Bragaru I A, Chen B, Poenar D and Iliescu C 2010 *Semiconductor conference (CAS), international* (Vol. 01), 11–13 October
21. Hua Z, Liao X, Diao H, Kong G, Zeng X and Xu Y 2004 *J. Cryst. Growth* **264** 7
22. Oliveira A R and Carreno M N P 2006 *J. Non-Cryst. Solids* **352** 1392
23. Kaneko T, Nemoto D, Horiguchi A and Miyakawa N 2005 *J. Cryst. Growth* **275** 1097
24. Mernagh V A, Kelly T C, Ahern M, Kennedy A D, Adriaansen A P M, Ramaekers P P J, McDonnell L and Koekoek R 1991 *Surf. Coat. Technol.* **49** 462
25. Mastelaro V, Flank A M, Fantini M C A, Bittencourt D R S, Carreno M N P and Pereyra I 1996 *J. Appl. Phys.* **79** 1324
26. Rava P, Crovini G, Demichelis F, Giorgis F and Pirri C F 1996 *J. Appl. Phys.* **80** 4116