International Journal of Thin Film Science and Technology

Volume 2
Issue 3 *Sep. 2013*

Article 2

2013

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Recommended Citation
K. E. Latha, H.; Udayakumar, A.; and Siddeswara Prasad, V. (2013) "Growth and effect of deposition pressure on microstructure and electrical properties of 3C-SiC thin films deposited using methyltrichlorosilane single precursor," *International Journal of Thin Film Science and Technology*: Vol. 2 : Iss. 3 , Article 2.
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International Journal of Thin Films Science and Technology

http://dx.doi.org/10.12785/ijtfst/020302

Growth and effect of deposition pressure on microstructure and electrical properties of 3C-SiC thin films deposited using methyltrichlorosilane single precursor

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Received: 29 Jul. 2013, Revised: 6 Aug. 2013, Accepted: 9 Aug. 2013
Published online: 1 Sep. 2013

Abstract: This paper presents effect of deposition pressure on the microstructure and electrical properties of 3C-SiC (111) thin films. These films are deposited at a temperature of 1040 $^\circ C$ and pressures of 1.5, 1.8, 2.5 and 2.7 mbar, on thermally oxidized Si (100) substrates from a mixture of methyltrichlorosilane and hydrogen gas using a vertical hot wall, low pressure chemical vapour deposition (LPCVD) reactor. The above properties are investigated for all films using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), Scanning electron microscopy (SEM) and four probe technique. An FTIR spectrum shows the formation of Si-C bonding of deposited films. The SEM and XRD investigations show improvement in the grain size and crystallanity, decrease in strain and dislocations of the films with increase in the deposition pressure from 1.5 to 2.5 mbar. AFM investigations show decrease in roughness of the films with increase in the deposition pressure from 1.55 to 2.5 mbar. The sheet resistance of 3C-SiC thin films is found to decrease with increase in temperature in the range of 35 to 550 $^\circ C$. Comparing all the films, 3C-SiC thin films deposited at a pressure of 2.5 mbar and temperature of 1040 $^\circ C$ exhibited improved structural and electrical properties.

Keywords: LPCVD, 3C-SiC, methyltrichlorosilane, XRD, Temperature coefficient of resistance.

1 Introduction

Silicon (Si) is a conventional semiconductor material having a narrow bandgap and widely used in sensor and device applications below 150 $^\circ C$. Mechanical properties of silicon are found to deteriorate for temperature above 500 $^\circ C$. Hence, there is a need for materials for high temperature applications and those materials should have wide bandgap, high hardness and high melting point [1,2].

Silicon Carbide (SiC) has intrinsic properties such as wide bandgap (2.2 eV for 3C-SiC), high melting point (2800 $^\circ C$), high break down field ($> 2\times 10^6 V cm^{-1}$), high electron saturation velocity ($2\times 10^7 cms^{-1}$) and high thermal conductivity (5-7 $W cm^{-1} K^{-1}$) [2,3,4,5]. These properties enable the use of SiC as sensing material for high temperature sensors applications. There are more than 200 different SiC polytypes and among these, only three crystalline structures namely cubic, hexagonal and rhombohedral are widely used for sensors and device applications. Among these three crystalline structures, 3C-SiC (cubic) material can be deposited on different substrates such as silicon, silicon dioxide, sapphire and silicon nitride [2,5,6]. The synthesis or deposition of 3C-SiC thin films can be done either by using double precursor or single precursor technique. The single molecular precursor technique is used for depositing the 3C-SiC material due to the accurate control of stoichiometry (1:1) between Si and C [7,8,9,10].

3C-SiC thin films can be deposited either by using atmospheric pressure chemical vapor deposition (APCVD) or low pressure chemical vapor deposition (LPCVD) technique. LPCVD is an ideal technique for depositing thinner 3C-SiC films because this provides higher film uniformity and lower contamination when compared to APCVD. The disadvantages of LPCVD are that, the deposition rate is more sensitive to temperature variations than APCVD and high temperatures

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are required to achieve significant deposition rate. Hence, for the growth of thin-films with high uniformity and low contamination level, LPCVD system is more suitable than an APCVD system [11,12].

This paper reports a technique for depositing 3C-SiC thin films by LPCVD from methyltrichlorosilane (MTS) single precursor molecule for sensor applications. Very little work has been reported regarding the electrical properties of 3C-SiC thin films using MTS. The present work explores the effect of deposition pressure on microstructure (growth rate and crystallinity) and electrical properties (temperature coefficient of resistance) of 3C-SiC thin films.

2 Experimentation

The 3C-SiC thin films are deposited on a thermally oxidized Si (100) substrate using a resistively heated vertical hot wall LPCVD reactor (M/s. Archer Technicoat limited, UK) and details of the reactor are reported elsewhere [13]. The precursors used are MTS (Purity: 99.8 %, M/s. Spectrochem, India) for SiC deposition and hydrogen (H_2) (Purity: 99.9995%, M/s. Inox air products, USA) as carrier gas. Gas flows are controlled by mass flow controllers (M/s. Bronkhorst Hightech, Netherland) through visual flow indicators. MTS is a colourless liquid at room temperature, is evaporated by heating at 70 $^\circ C$ in a flash evaporation system. The resulting vapour is mixed with H_2 in the mixing chamber and finally admitted in to the reaction chamber. Processing condition used for the deposition of 3C-SiC thin films are: temperature 1040 $^\circ C$ and MTS: H_2 ratio used is 1:16 to deposit stoichiometric SiC without co-deposition of free Si or carbon (C). These films are deposited using precise control of the various deposition pressures of between 1.5 and 2.7 mbar.

The Si (100) substrates are cleaned by RCA (Radio Corporation of America) cleaning method. To electrically isolate the deposited film for electrical characterization, a silica layer of 0.6 μm thicknesses is thermally grown on Si substrates. These thermally oxidized Si (100) substrates are placed on the graphite holder in the reaction chamber for deposition of 3C-SiC thin films. The reaction chamber is evacuated to a pressure of 0.1 mbar by using a combination of liquid ring pump (LRP) and booster pumps, and purged with argon (Purity:99.999%) continuously prior to film deposition for removing the residual air. The temperature of the reaction chamber is increased from room temperature to 960 $^\circ C$ at a rate of 4 $^\circ C/min$ with argon (Ar) purging. The MTS at a flow rate of 1.25 SLM (Standard Liters per Minute) and H_2 at a flow rate of 20 SLM are supplied into the chamber for a period of 20 min. During this period, the pores on the surface of the substrates are filled with 3C-SiC material due to the decomposition of MTS vapour in the presence of H_2. The temperature of the reaction chamber is increased from 960 $^\circ C$ to 1040 $^\circ C$ at a rate of 4 $^\circ C/min$ with Ar purging. The 3C - SiC thin films are deposited on thermally oxidized Si (100) substrates by supplying reactive gases (MTS+ H_2) for a period of 30 min. After the deposition process, reactive gas supply is stopped and the reactor is evacuated for 30 min to remove the byproducts. The reactor is backfilled with Ar and allowed to cool to room temperature.

Experiments are conducted for various deposition pressures to deposit 3C-SiC thin films by keeping other processing conditions constant.

PANalytical X-pert Pro X - ray diffractometer operated at 40 kV, 30 mA with a Cu Kα ray tube (λ = 1.542Å), configured in symmetrical $\theta - 2\theta$ mode is used to obtain XRD patterns of 3C-SiC thin films in order to analyze the crystal orientation, the scanning angle (2θ) is varied from 15° to 80° with scanning steps of 0.04°. Perkin Elmer spectrometer with a spectral resolution of $4cm^{-1}$ is used to carry out the Fourier transform infrared spectroscopy measurements to confirm the presence of Si-C bonding. Size, distribution of grains and elemental composition in the grown 3C-SiC thin films are analyzed using Jeol JSM-5500 Scanning electron microscopy (SEM). Film thicknesses of grown 3C-SiC thin film are determined by cross-sectional SEM images. The surface roughness of 3C-SiC thin films is investigated with atomic force microscope (AFM- XE-70 Park system) using diamond tip in contact mode. XEI software is used for data processing and surface roughness analysis. Sheet resistance of 3C-SiC thin films is measured by 4-probe method (in-situ) using a current source meter (Keithley 6221) and Nanovoltmeter (Keithley 2182A) in the range of 30 to 550 $^\circ C$ with a heating rate of 4-5 $^\circ C/min$ and an accuracy of $\pm 1^\circ C$. TCR of each 3C-SiC thin film are calculated for the above temperature range.

3 Results and discussion

3.1 Structural properties

Figure 1 shows the photograph of 3C-SiC thin film deposited on thermally oxidized Si substrate. It is observed that the film is free of any visible cracks, and peeling offs. It confirms the adhesion and uniformity of deposition.

The crystal orientation in the grown film is analyzed by means of X-ray diffraction (XRD) patterns. The XRD patterns obtained for Si (100) substrate and 3C-SiC thin films deposited on thermally oxidised Si (100) substrate at a temperature of 1040 $^\circ C$ and deposition pressures range of 1.5 to 2.7 mbar are shown in Figure 2. The peaks corresponding to 6H-SiC (101) polytype, 3C-SiC (311) and 3C-SiC (111) are observed in the film deposited at a pressure of 1.5 mbar. The

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Figure 1: Photograph of 3C-SiC thin film deposited on thermally oxidized Si

intensity of the diffraction peaks increased with increasing the deposition pressure. A strong peak observed in all the deposition pressures at $2\theta = 35.56^\circ$ corresponding to 3C-SiC (111) phase. The lattice constant of corresponding peak is $4.36\AA$, which agrees well with that of single-crystalline 3C-SiC [9,10,14]. The observed Full width at half maximum (FWHM,β) decreases from 0.32° to 0.24° with increase in deposition pressure from 1.5 to 2.5 mbar which indicates the enhancement in crystallite size from 26.5 nm to 35.3 nm (as obtained from Scherrer formula) and reduction in strain (1.3×10^{-3} to 0.98×10^{-3}) of films. But for the film deposited at pressure of 2.7 mbar exhibits an increase in FWHM of 0.34°, which indicates the reduction in crystallite size (24.82 nm) and enhance the strain of the film (1.9×10^{-3}). This shows the deposition pressure is an important parameter influencing the film crystallinity. The peaks observed at around 27.5° and 56.1° are attributed to the presence of SiO_2 layer between Si substrate and 3C-SiC thin film, and Si substrate respectively.

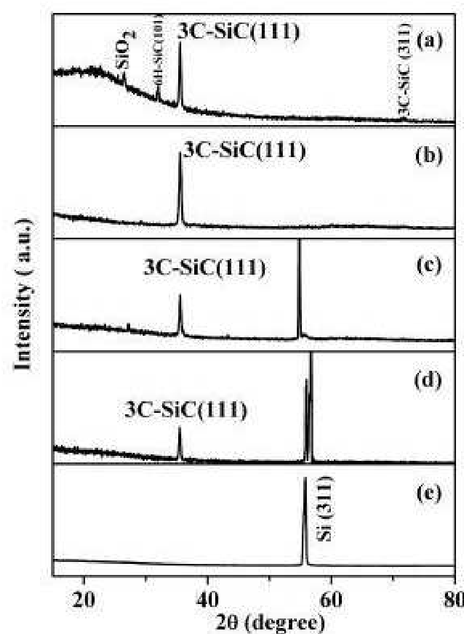

Figure 2: XRD patterns of 3C-SiC films deposited at various deposition pressures (a) 1.5 mbar (b) 1.8 mbar (c) 2.5 mbar (d) 2.7 mbar and (e) Si(100)-Substrate

The dislocation density (δ), defined as the length of dislocation lines per unit volume [15], are estimated using the equation

$$\delta = \frac{1}{D^2} \quad (1)$$

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Where D is the crystallite size of the films are estimated using the Scherrer formula. Strain of the thin films is estimated using the equation (2)

$$\varepsilon = \frac{\beta \cos\theta}{4} \tag{2}$$

These results reveled that changing the deposition pressure will change the microstructure of the deposited 3C-SiC thin films as reported in Table 1.

Table 1: Strain, dislocation and crystallite sizes for the peak(111)

Sl. No	Deposition pressure (mbar)	FWHM (10^{-3} rad)	Average Crystallite size, D(nm)	strain ε (10^{-3})	Dislocation density $\delta(10^{14}$ $lines/m^2)$
1	1.5	5.495	26.48	1.31	14.26
2	1.8	4.808	30.27	1.14	10.91
3	2.5	4.122	35.31	0.98	8.02
4	2.7	5.864	24.82	1.39	16.23

Dislocation density (δ) indicates a measure of crystallinity. Among these films, 3C-SiC deposited at pressure of 2.5 mbar gives the smallest δ indicating best crystalline structure compared to higher or lower deposition pressure.

The transmittance peaks of the transverse optical (TO) mode in the Fourier transform infrared spectra of 3C- SiC thin films deposited at pressures range of 1.5 to 2.7 mbar are shown in Figure 3. Since Si-C bond is highly IR active, a vibrational peak appears at around 800 cm^{-1} in all the samples and is attributed to Si-C bonding [10,16]. The 3C-SiC thin film deposited at pressure of 2.5 mbar show an increase in Si-C peak intensity as compared to the other films. The increased peak intensity is attributed to the decrease in mean free path of precursor molecules with increasing gas pressure. This result in increasing the number of molecules that collide with each other and precursor will decompose completely. This indicates the possibility of increased Si-C bonding with increase in deposition pressure [17]. The FWHM is found to decrease with increase in deposition pressure indicative of the enhancement in crystallinity and this result is in agreement with the XRD result. The peak observed at 606 cm^{-1} is from the Si substrate [10] and vibrational peak observed at around 1100 cm^{-1} (not shown in Figure 3.) is attributed to the presence of SiO_2 layer between Si substrate and 3C-SiC thin film [18,19].

The elemental compositions of the deposited 3C-SiC thin films are analyzed by Energy dispersive X ray spectroscopy (EDX) at different points on films. The elemental compositions of 3C-SiC deposited at pressures of 1.5, 1.8, 2.5 and 2.7 mbar is as shown in Figure 4 and results indicate the stoichiometric relationship (1:1) between Si and C.

The effect of pressure on the microstructure of deposited films can be determined by the comparison of films deposited under different pressures. The surface microstructure of 3C-SiC thin films as a function of deposition pressures are shown in Figure 5. Densely packed grains with increasing grain sizes (122 nm to 185 nm) are observed on the 3C-SiC thin films. This shows that the surface grain size increases with increase in deposition pressure (1.5 to 2.5 mbar). The grain boundaries are seen more clear on the film deposited at pressure of 2.5 mbar. But the film deposited with the pressure of 2.7 mbar, grains tend to decrease in size because of large strain in the film affects the growth of 3C-SiC, leading to poor crystallinity. These observations are consistent with the crystallite size predicted by the Scherrer equation.

With increasing deposition pressure, the growth rate rapidly increases (Figure 6) at pressures below 2.5 mbar, but decreases slightly at pressure above 2.5 mbar. This means that at pressures below 2.5 mbar, the SiC growth rate is significantly influenced by reactive species formed from the decomposed MTS. This is due to increase in deposition pressure and gas fluxes are constant, the mobile speed of reactive gases (MTS) to the growing surface decreases. Thus the resident time of molecules in the deposition zone increases results in increased growth rate. These observations are consistent with the increased peak intensity of (111) peak (XRD diffraction data from Figure 2). An increase in the deposition pressure (2.7 mbar) seems to reduce the mobile speed of reactive gases (MTS) to the growing surface, thereby decreasing the growth rate and deteriorates the crystal quality. The lowest FWHM for 3C-SiC (111) peak is observed at a pressure of 2.5 mbar, at which a change in growth rate is also observed. These results reveled that the microstructure properties is more sensitive to the deposition pressure variations and are good in case of film deposited with 2.5 mbar. Hence the growing temperature and pressure are main parameters influencing both film crystallinity and deposition rate [20].

The surface roughness of 3C-SiC thin films deposited at different deposition pressures are investigated using atomic force microscopy. The average surface roughness of the films decreases with increasing pressure (1.5 to 2.5 mbar). This shows that the surface roughness exhibits strong dependence on deposition pressure [14]. But there is increase in surface

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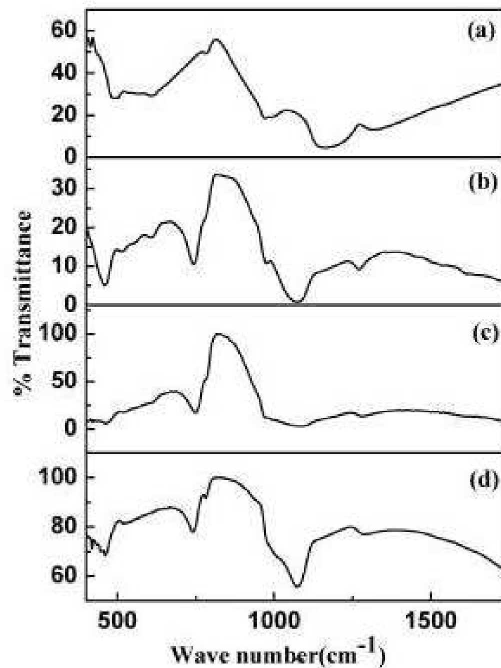

Figure 3: FTIR spectra obtained by 3C-SiC films deposited at various pressures (a) 1.5 (b) 1.8 (c) 2.5 and (d) 2.7 mbar

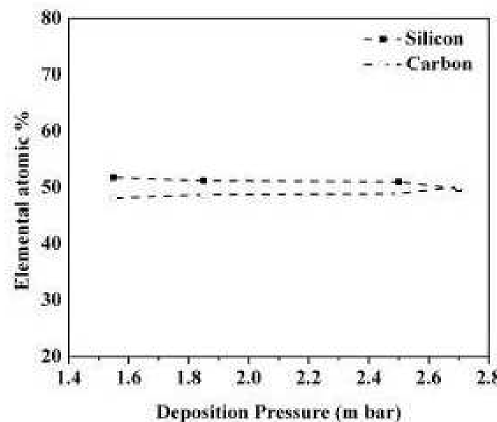

Figure 4: Elemental compositions of 3C-SiC thin films as a function of deposition pressure

roughness in the film deposited at pressure of 2.7 mbar due to reduction in grain size. Figure 7 shows the roughness variations with deposition pressure. Average surface roughness of the film deposited at 1.5, 1.8 and 2.7 mbar is too high for MEMS and sensor applications [10].

3.2 Electrical properties

For sensor applications, it is always desirable that the resistance variation shall be as low as possible over a wide range of temperature [4] and electrical properties are depends on grain size of the deposited film. There is improved microstructure properties are observed in film deposited at pressure of 2.5 mbar when compare to all the films. Hence for electrical properties characterization only film deposited at pressures of 1.5, 2.5 and 2.7 mbar are considered. The normalized sheet

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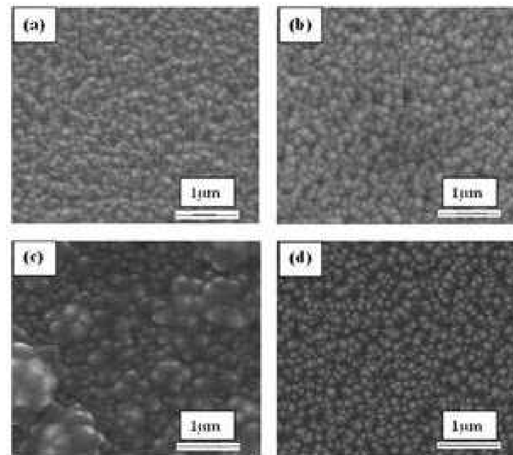

Figure 5: SEM micrographs of 3C-SiC films deposited at pressure of (a) 1.5, (b) 1.8, (c) 2.5 and (d) 2.7 mbar.

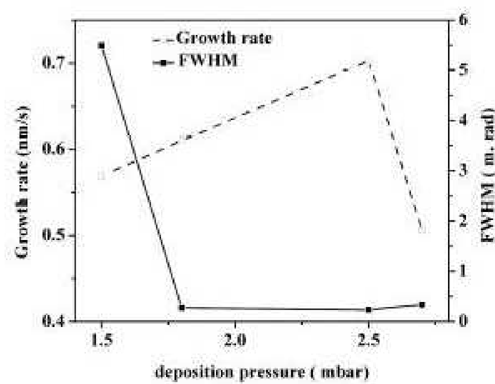

Figure 6: Growth rate of 3C-SiC thin films and FWHM of the 3C-SiC (111) peak as a function of deposition pressure

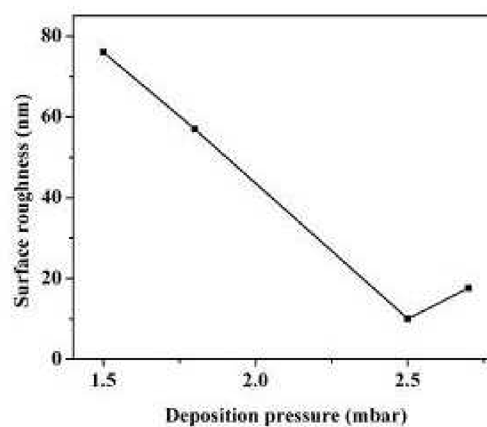

Figure 7: Roughness of 3C-SiC thin film deposited at different pressure

resistance variation of 3C-SiC thin films deposited at 1.5, 2.5 and 2.7 mbar with temperature in the range of 40 - 550°C is shown in Figure 8. The normalized sheet resistance of the 3C-SiC thin films deposited at pressures of 1.5, 2.5 and 2.7 mbar

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exhibits an exponential dependence on temperature. The thermionic emission of energetic carriers over a potential barrier is activated with increasing temperature, which results in decrease of sheet resistance. The film deposited at a pressure of 1.5 and 2.7 mbar has larger magnitude change in resistance ratio as compared to the film deposited at 2.5 mbar. This is attributed to the increased grain size (growth rate) of film deposited with 2.5 mbar. The resistance variation is found relatively low for temperature range of 330-550 $^{\circ}C$ for both the films.

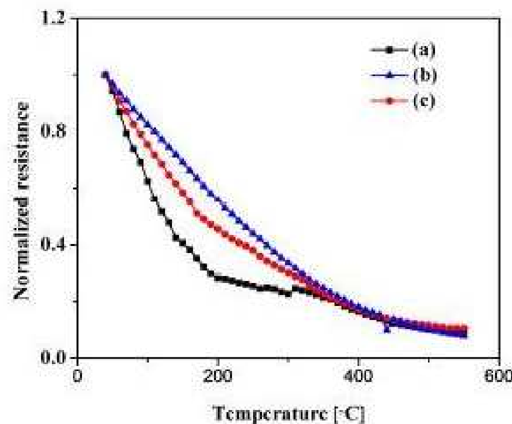

Figure 8: Normalized resistance versus temperature for 3C-SiC thin films deposited at a pressures of (a) 1.5, (b) 2.5 and (b) 2.7 mbar

The TCR variations of 3C-SiC thin films deposited at different pressure with temperature is shown in Figure 9. The TCR of 3C-SiC thin films deposited at a pressure of 2.5 mbar varies from $-3209 ppm/^{\circ}C$ to $-1803 ppm/^{\circ}C$ whereas for the film deposited at a pressure of 1.5 and 2.7 mbar varies from $-5650 ppm/^{\circ}C$ to $-1780 ppm/^{\circ}C$ and $-3970 ppm/^{\circ}C$ to $-1750 ppm/^{\circ}C$, respectively, for the temperature range of 40 to 550 $^{\circ}C$. The standard deviation of TCR for 3C-SiC thin film deposited at a pressure of 1.5, 2.5 and 2.7 mbar is $1385 ppm/^{\circ}C$, $188 ppm/^{\circ}C$ and $616 ppm/^{\circ}C$, respectively, in the temperature range of 40 to 320 $^{\circ}C$, whereas for temperature range of 330 to 550 $^{\circ}C$, standard deviation of TCR will be $231 ppm/^{\circ}C$, $211 ppm/^{\circ}C$ and $230 ppm/^{\circ}C$, respectively. The standard deviation of TCR for 3C-SiC thin films deposited at pressures of 1.5 and 2.7 mbar are found to be almost same, but standard deviation of film deposited at pressure of 2.5 m bar is less compared to other samples due to higher growth. Hence these films are suitable for high temperature (330 to 550 $^{\circ}C$) sensor applications.

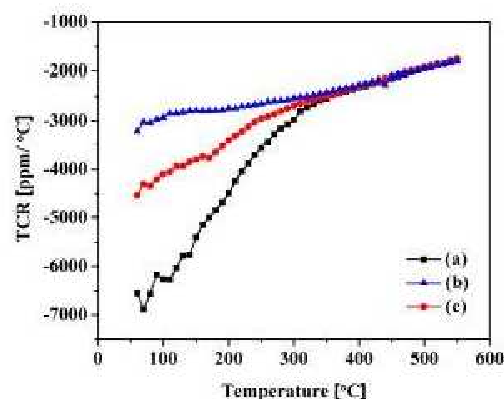

Figure 9: TCR versus temperature for 3C-SiC thin films deposited at pressures of (a) 1.5, (b) 2.5 and (b) 2.7 mbar

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4 Conclusions

3C-SiC thin films are successfully deposited using low pressure chemical vapour deposition for high temperature sensor applications. The effect of deposition pressures on microstructure, crystallinity and electrical properties of 3C-SiC thin films are analyzed. X-ray diffraction study is carried out on these thin films showed that the crystallinity and crystallite increased with increase in pressure from 1.5 to 2.5 mbar. The Si-C bonding peak in the film deposited at a pressure of 1.5 mbar is very weak and broad, where as the film deposited at 2.5 mbar shows stronger and sharper Si-C bonding, indicating the enhancement in crystallinity of 3C-SiC thin films and this result is in agreement with the XRD result. The grain size increases with increasing deposition pressure (1.5 to 2.5 mbar) as investigated from SEM analysis. Where as a decrease in grain size is observed in case of film deposited with 2.7 mbar and shows a significant variation in sheet resistance as compared to the films deposited at pressure of 2.5 mbar. The standard deviation of TCR for 3C-SiC thin films deposited at pressures of 2.5 mbar is 211 ppm $/^{\circ}C$ for temperature range of 330 to 550 $^{\circ}C$. These results reveled that effect of pressure on TCR of film for high temperature range is negligible. Because of reduction in the surface roughness and TCR in the film deposited at pressure of 2.5 mbar, this film is suitable for high temperature sensor applications.

Acknowledgement

The authors thank the management of National Aerospace Laboratories, Bangalore and Siddaganga Institute of Technology, Tumkur for their encouragement and support to carry out this work.

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