Print ISSN : 0022-2755

Journal of Mines, Metals and Fuels

Contents available at: www.informaticsjournals.com/index.php/jmmf

Investigation on Strain Sensitivity and Temperature Behaviour of Nitrogen Doped 3C-SiC Thin Films

H.K.E Latha[a*], Mala Sa and A. Udayakumar[b]

*aDepartment of Electronics and Instrumentation Engineering, Siddaganga Institute of Technology, Tumakuru-572103, India. *E-mail: lathahke@gmail.com*

bPrincipal scientist, Materials science division, Council of Scientific and Industrial Research- National Aerospace Laboratories, Bangalore-560017, India.

Abstract

Electrical resistance-strain behaviour of nitrogen doped 3C-SiC thin films was investigated to determine whether they could be used as strain gauges. Using a hot wall vertical low pressure chemical vapour deposition (LPCVD) reactor, these films were deposited on thermally oxidised Si (100) and alumina substrates at 2.5 mbar pressure and 1040°C temperature from methyltrichlorosilane (MTS) precursor. The nitrogen doping gas utilised was ammonia (NH_3). Using four-point bending method, the gauge factor (GF) was determined for all thin films (0, 9, 17, and 30 atomic % nitrogen doped). Systematic annealing of films in a vacuum atmosphere was used to determine the TCR of nitrogen doped 3C-SiC (111). Film sheet resistance was evaluated using a four-probe approach, and it was observed to decrease as the temperature rises from 40 to 550°C. The resistivity, average TCR, and strain sensitivity of film doped with 17 atomic % nitrogen concentration were 0.14 cm, -103 ppm/°C, and -9.6, respectively, indicating that it can be employed as a strain gauge material in high temperature applications. However, film doped with 30 atomic % of nitrogen concentration showed an increase in the resistivity, TCR and strain sensitivity.

Keywords: *LPCVD, MTS, 3C-SiC thin film, TCR, strain sensitivity.*

1.0 Introduction

Strain gauges made of thin film semiconductors have been widely studied and used in measurement of physical quantities like pressure, strain, flow, acceleration, and force due to their higher strain sensitivity (GF). Measurement of these parameters under harsh environment conditions (high temperature, high shock, high radiation, errosive flow/ impact and corrosive media) using sensors in aerospace, petrochemical and automotive sectors are very essential for the safe and efficient operation of industrial processes [1,2]. Currently, piezoresistive sensors based on silicon (Si) are utilised to measure the physical properties described above. Due to Si narrow bandgap, its electrical capabilities decrease at temperatures over 150°C, and its mechanical qualities worsen at temperatures beyond 500°C, limiting its use in high-temperature applications [3, 4]. As a result, semiconductor sensing materials with a wide bandgap, good mechanical and electrical properties over a broad temperature range are required. Wide bandgap semiconductor materials include GaAs, diamond and SiC. Thin film pressure sensors based on GaAs [5] and Diamond [6-8] have been employed up to temperatures of 300 and 450°C, respectively.

*Corresponding Author

SiC is a promising semiconducting material which finds applications in opto and microelectronics. SiC material has attracted the researchers due to its larger bandgap, higher hardness, melting point, break down field, electron saturation velocity and thermal conductivity, and is found to be suitable as sensing material in sensor applications for high temperature [1, 9-11]. There are about 200 distinct SiC polytypes, with cubic, hexagonal, and rhombohedral crystalline forms being the most used for sensors and devices. Among these polytypes, 3C-SiC (cubic) is the widely used sensing material for harsh environment applications and it can be easily deposited on Si, SiO_2 and Si_3N_4 substrates using CVD method [1,12-16]. 3C-SiC thin films can be deposited using either double or single precursor. However, the stoichiometry link between Si and C is poor in the double precursor approach. As a result, subsequent studies are concentrated on deposition of 3C-SiC thin films employing single precursors [1, 2, 17-21]. Using single precursors, the deposited films show good stoichiometry.

A strain gauge with good strain sensitivity, low TCR, and great thermal stability is useful for measuring physical parameters [15, 16, 22, 23]. Experimental analysis of the strain sensitivity and the TCR of 3C-SiC (nitrogen doped) for possible uses as a strain gauge is reported in this paper. These films were deposited using LPCVD technique with MTS as the precursor gas, and doping is done by introducing ammonia gas during the deposition process. The four-point bending method is used to test strain sensitivity.

2.0 Experimentation

2.1 Growth of 3C-SiC (nitrogen doped) thin films

The in-situ 3C-SiC (nitrogen doped) films were deposited on thermally oxidised Si (100) and alumina substrates using LPCVD reactor, details are reported elsewhere [24]. Technique used for deposition of these thin films are reported elsewhere [25]. By maintaining processing conditions constant, insitu 3C-SiC (nitrogen doped) thin film deposition tests were carried out for varied NH_3 flow rates. XRD patterns are obtained from PANalytical X-pert Pro X-ray diffractometer. Sheet resistance was measured by 4-probe method (in-situ) in the range of 40 to 550°C. Resistivity and TCR of thin films were calculated for temperature range 40 to 550°C. Electrical resistance – strain behaviour of films were studied at room temperature using four-point bending set up. The electrical resistance – strain graph was used to calculate the GF of thin films.

2.2 Experimental Set-Up to Study the Electrical Resistance – Strain Behaviour

Once the deposition process is completed, for strain gauge applications it is required to know the resistance-strain behaviour of the films. Therefore, it is essential to know the sensitivity (GF) of the 3C-SiC thin film to use it as strain gauge material.

In present work, four-point bending method (mechanical technique) was used to measure the GF of

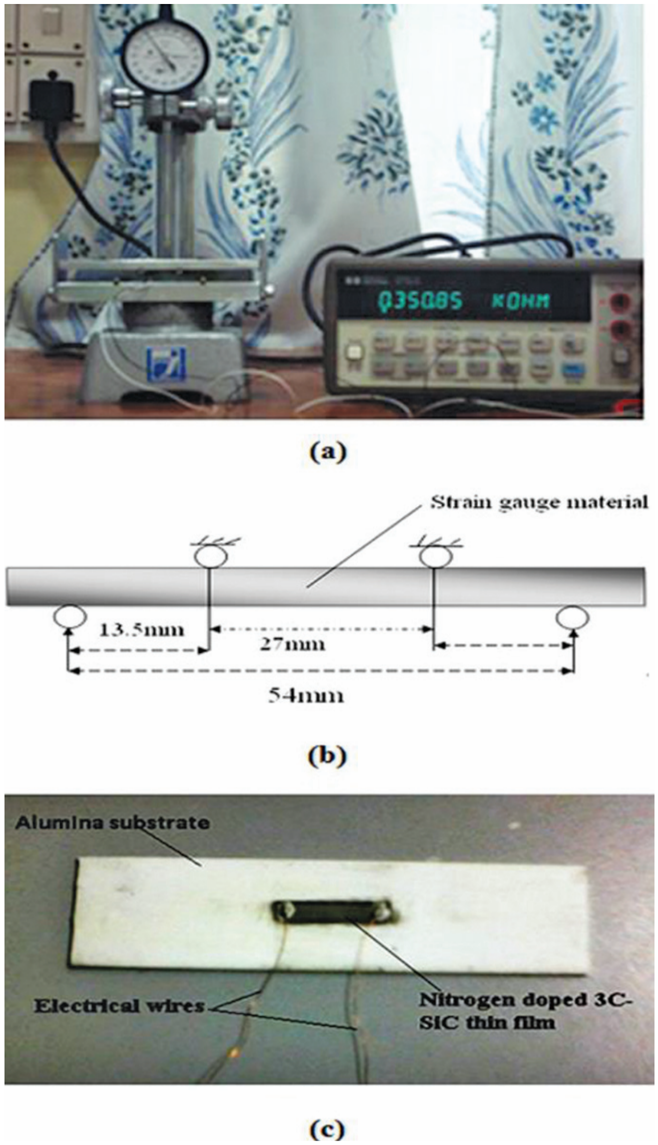

Fig.1. Photograph of (a) the four-point bending setup, (b) Dimension details of the four-point bending setup and (c) Nitrogen doped 3C-SiC thin film strip on alumina substrate.

thin films. The photography of four-point bending set up is shown in Fig.1 (a). It consists of two identical rectangular electroplated stainless steel (SS304) slabs of 7 mm thickness, 50 mm width and length of 125 mm and four tungsten carbide rolling pins of diameter 5 mm and length 50 mm.

Two V-grooves have been made in each of the slab along the width to position the tungsten carbide rolling pins at suitable locations on either slab so as to provide adequate bending moment under loading of the sample plate as shown in Fig.1(b). Two of the rolling pins were placed in the V-grooves on top of the bottom slab. In order to hold the rolling pins firmly, rollers pins were fixed by kapton tape. The sample plate (alumina) with the 3C-SiC thin film strip (strain gauge) and wire leads for resistance measurement is then placed over the rolling pins, which is as shown in Fig. 1(c). The upper slab, with the other two rolling pins in its V-grooves facing downwards, was placed over the sample plate.

The entire assembly was kept on a stand with a dial gauge (Mitutoyo, 2109S-10) of ±1μ accuracy. Thus the alumina plate on which the strain gauge thin films were deposited are placed between the rolling pins. At the centre of the top plate a hole of 9 mm diameter was made, through which the dial gauge spindle was placed so that the tip of the rod just touching the surface of thin film (sample plate) where maximum deflection occurs during loading. The four-point bending setup described above is similar to the set up reported in [26]. The calibration of the system was carried out using a foil type Nicrome strain gauge, which is a linear type strain gauge having a resistance of 350Ω and GF value 2.1 (IPA India, Bangalore) is bonded on to a micro slide (75 × 25 × 1.35 mm).

Load is applied in steps and corresponding deflection as well as change in resistance of the Nichrome foil strain gauge is measured using dial gauge and a 6 1/2 digit multimeter, respectively. GF is calculated and found to be 2.0975. Therefore, the accuracy of the four-point bending system used in the present investigation is found to be 0.12%.

When load (slotted weights) was applied, the sensor material (nitrogen doped 3C-SiC) would be in tension. The value of strain ε is given by

$$\varepsilon = \frac{4t|\delta_t|}{d^2} \quad \text{... (1)}$$

where t = thickness of alumina substrate(mm)

δt = deflection (μm) and

d = distance between outer rolling pins (mm)

Sample is loaded by placing slotted weights on top of the upper slab and for various loads the corresponding deflections indicated by the dial gauge are recorded. Simultaneously, the resistance change ΔR was measured using multimeter (HP 34401A).

Also, the initial resistance R of thin film strain gauge before loading was measured. GF of semiconductor material may be expressed as

$$GF = \frac{\Delta R/R}{\varepsilon} \quad \text{... (2)}$$

Where ΔR/R is relative change in resistance and strain ε is determined using equation (1). The GF is a scalar quantity [27] and depends on the fabrication method (diffusion, ion-implantation or epitaxial growth), crystallographic directions, dopant level and type (n- or p).

3.0 Results and Discussions

XRD pattern of 3C-SiC (undoped) film is depicted in Fig.2. The peaks at 27° and 56° correspond to SiO_2 layer and Si respectively.

The peaks at 2θ values of 35.6° corresponds to 3C-SiC (111) and it validates that, the processing parameters employed has resulted in 3C-SiC polytype with a lattice constant of 4.36 Å. The impact of doping on the crystallinity of films is depicted in Fig.3. It is observed that FWHM has raised from 0.23° (for undoped) to 0.63° (for film doped with 17 atomic % of nitrogen), exhibiting a reduction in the size of crystallite (from 36.2 to 13.2 nm). The detail discussions on the impact of nitrogen doping in 3C- SiC on the crystallinity are reported elsewhere [25].

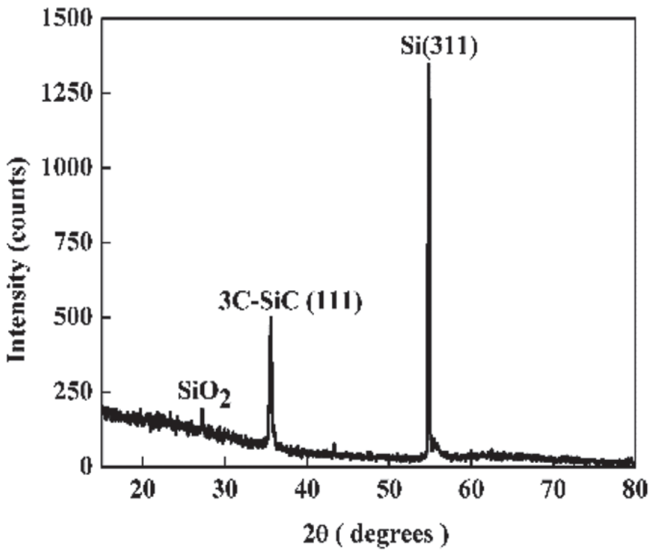

Fig.2. XRD pattern of undoped 3C-SiC thin film

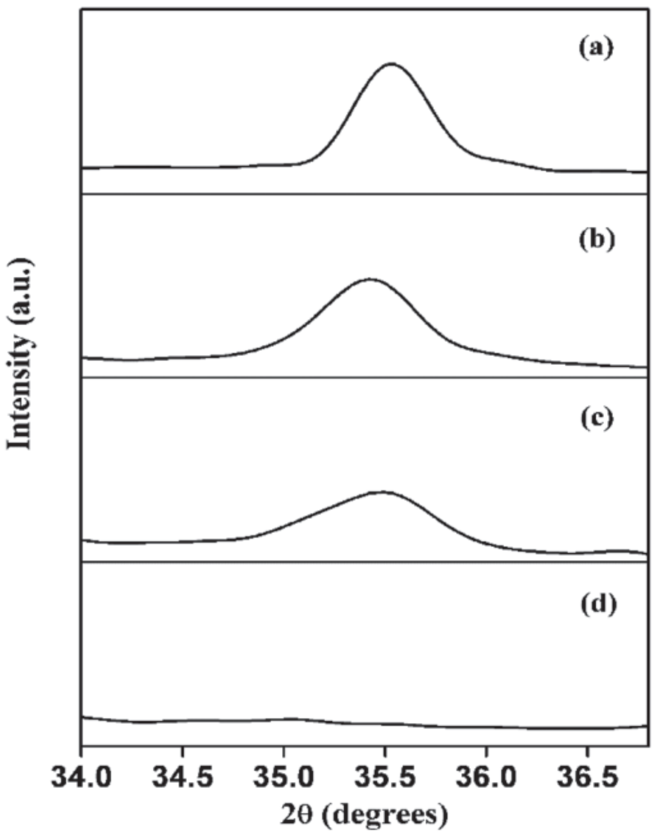

Fig. 3. XRD patterns of thin films with (a) 0 (b) 9 (c) 17 and (d) 30 atomic % of nitrogen doping

The temperature effect on resistivity of 3C-SiC (0, 9, 17 and 30 atomic % of nitrogen) films in the temperature region 40-550°C is depicted in Fig.4. Temperature impact on resistivity of undoped film exhibits a reduction with rise in temperature (up to 200°C).

Furthermore, as the temperature rises the variations in resistivity becomes lesser and tends to a value of 19 Ωcm at 550°C. In these films resistivity was found to decrease in the order of 14 as temperature increased from 40 to 550°C (Fig 4(a)). Doping the film reduces the resistivity significantly less than 1Ωcm at any given dopant concentration and variations in resistivity is slow down over a range of temperature [17, 28, 29]. The resistivity was found to decrease in the order of 1.4 for the film doped with 9 atomic % of nitrogen (Fig.4(b)), 1.07 for film doped with 17 atomic % nitrogen (Fig.4 (c)) and 1.5 for the film doped with 30 atomic % nitrogen (Fig.4 (d)) as temperature rise from 40 to 550°C. The film doped with 30 atomic % of nitrogen exhibited an increase in resistivity compared (Fig.4 (d)) to films doped with 9 and 17 atomic % of nitrogen (Fig 4 (b & c)). Film doped with 17 atomic % of nitrogen exhibits an insignificant change in resistivity over the entire temperature range of study. The detail discussions on temperature dependent resistivity of doped films were reported elsewhere [25].

The TCR plots of 3C-SiC (nitrogen doped) films with temperature are depicted in Fig.5 and were found to be negative. The obtained average TCR is - 2488 ppm/°C for undoped films, -661 ppm/°C for 9 atomic % of nitrogen, -103 ppm/°C for 17 atomic % of nitrogen and -658 ppm/°C for 30 atomic % of nitrogen. Film doped with 17 atomic % of nitrogen exhibits lower value of

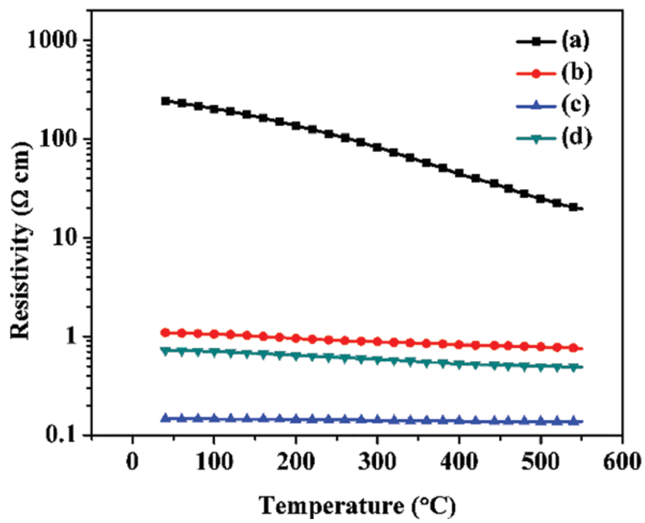

Fig.4. Impact of temperature variations on resistivity of films with (a) 0 (b) 9 (c) 17 and (d) 30 atomic % of nitrogen doping

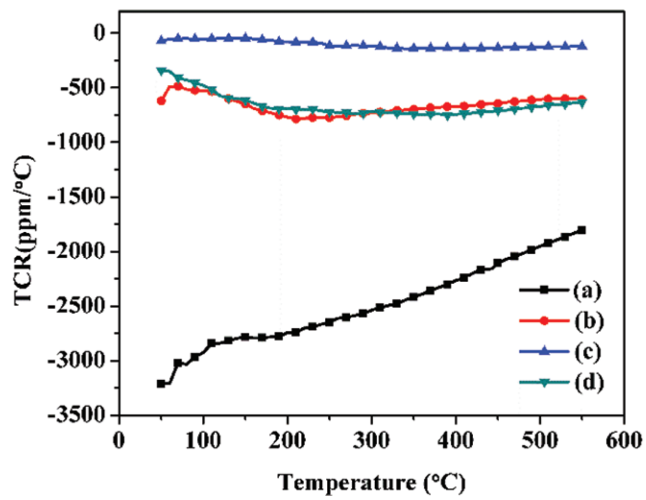

Fig. 5. Impact of temperature variations on TCR of thin films with (a) 0 (b) 9 (c) 17 and (d) 30 atomic % of nitrogen doping

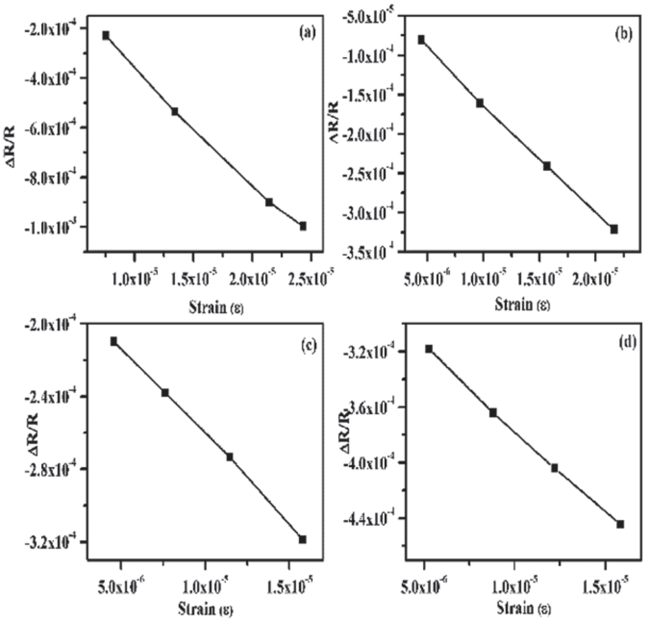

Fig. 6. Plot of ÄR/R and strain for thin films with (a) 0 (b) 9 (c) 17 and (d) 30 atomic % of nitrogen doping

average TCR. TCR of film (doped with 17 atomic % of nitrogen) varies from -71 to -118 ppm/°C (Fig 5 (c)) and that of undoped film varies from - 3209 to - 1803 ppm/°C (Fig 5 (a)) in the temperature range of 40 to 550°C. Results exhibit that 3C-SiC (undoped) film has greater TCR values than films doped with nitrogen. It is observed that for all doped film the TCR values are slightly increased in the temperature region 40 to 200°C (Fig.5 (b-d)) but start to reduce in its value for temperature above 200°C. The detail discussions on TCR variations of 3C-SiC (nitrogen doped) films with temperature are reported elsewhere [25].

The GF mainly depends on resistivity and bandgap of the semiconductor material used to construct the strain gauge. A graph of ΔR/R with strain measured at room temperature is depicted in Fig.6 for longitudinal strained 3C-SiC (nitrogen doped) film oriented in (111) direction and GF is computed from the slope of ÄR/R versus strain. A four-point bending technique is used to study the change in resistance - strain behaviour and calculate the GF of the nitrogen doped 3C-SiC thin film using equation (1) and (2).

GF was found to be reduced from - 46 to - 9.6 (Fig.6 (a-c)) with rise in nitrogen doping concentration from 0 to 17 atomic % and increased to -12 (Fig.6(d)) for film doped with 30 atomic % of nitrogen. Reduction of GF up to 17 atomic % of doping is due to the decrease in resistivity (Fig.4). However increase in GF of 30 atomic % of nitrogen doping can be contributed to the forming

Table 1: Electrical resistivity, Average TCR and GF of doped thin films

Sl.No.	Nitrogen doping concentration (at %)	Electrical resistivity in Ù cm	Average TCR (ppm/°C)	GF
1	0	246	-2315	-46
2	9	1.09	-686	-14
3	17	0.14	-123	-9.6
4	30	0.72	-709	-12

of amorphous structure in thin film (increase in resistivity). These results are in good agreement with XRD studies (Fig.3). It is also observed that the GF value is decreased by 79 % with a rise in doping concentration (0 to 17 atomic % of nitrogen). The results show that nitrogen concentration in the 3C-SiC film has a significant effect on GF. The summary of the results (electrical resistivity, average TCR and GF) obtained for various 3C-SiC (nitrogen doped) thin films are tabulated in Table 1.

4.0 Conclusions

3C-SiC thin films (with and without nitrogen doping) were obtained through LPCVD process on thermally oxidized Si and alumina substrates using MTS and NH3 precursors. The decrease in resistivity and GF of films was observed with rise in nitrogen doping concentration and their lowest values were observed at 17 atomic % of nitrogen at room temperature. Whereas the films doped with 30 atomic % of nitrogen showed an increase in the resistivity and GF. The doped film (17 atomic % of nitrogen) showed average lower values of TCR (-103 ppm/°C) from 40 – 550°C, whereas the film doped with 30 atomic % of nitrogen showed an increase in resistivity, TCR and strain sensitivity. Because of reduction in resistivity, TCR and strain sensitivity, thin film doped with 17 atomic % of nitrogen are suitable material for the possible applications as strain gauge.

Acknowledgements

The authors thank Mr. P. Kumara, Mr. S. Ganesh and Ms. M V Geetha for their help in carrying out the experiments, Dr. M M Nayak, Center for Nano- science and engineering, Indian Institute of Science, Bangalore for his kind suggestions and encouragement. The authors also thank the Directors, National Aerospace Laboratories, Bangalore and Siddaganga Institute of Technology, Tumakuru for their encouragement and support to carry out this work.

References

[1] Mehran Mehregany, Christian A Zorman, Narayanan Rajan, and Chien Hung Wu. (1998). Silicon Carbide MEMS for Harsh Environments. Proc. IEEE, vol. 86, no. 8, 1594-1610.

[2] Gerhard H. Kroetz, Martin H. Eickhoff, and Helmut Moeller. (1999). Silicon compatible materials for harsh environment Sensors. *Sensors and Actuators*, 74, 182-189.

[3] Joseph S Shor, David Goldstein and Anthony D. Kurtz. (1993). Characterization of n-type beta - SiC as a piezoresistor. *IEEE transactions on electron devices* 40, 1093 - 1099

[4] Okojie R.S., Ned A.A., Kurtz A.D., Carr W.N. (1998). Characterization of highly doped n- and p-type 6H-SiC piezoresistors. Electron Devices. IEEE T. Electron. Dev.;45: 785– 790

[5] Dehe, A., Fricke, K., Mutamba, K. and Hartnagel, H. L, J. (1995). A piezoresistive GaAs pressure sensor with GaAs/AlGaAs membrane technology. Micromech. *Microeng.* 5, 139

[6] Obermeier, E. (1995). High Temperature Microsensors Based on Polycrystalline Diamond Thin Films. Proceedings of 8th International Conference on Solid State Sensors and Actuators, Stockholm, Sweden, 178–181.

[7] Luo JK, Fu YQ, Le HR, Williams JA, Spearing SM, Milne WI (2007) "Diamond and diamond-like carbon MEMS". *J Micromech Microeng* 17:S147–S163

[8] M. Werner, M. Adamschik, P. Gluche, E. Kohn, and H.-J. Fecht. (1998). Review on diamond based piezoresistive sensors. Proceedings of the IEEE International Symposium on Industrial Electronics (ISIE '98), vol. 1, 147-152

[9] K. N. Bhat and M. M. Nayak (2012). MEMS Pressure Sensors - An Overview of Challenges in Technology and Packaging. Inst. Smart Struct. *Syst. J. Isss J. Isss*, vol. 1, no. 1, 1–10.

[10] Fraga MA, Furlan H, Pessoa RS, Rasia LA, Mateus CFR (2012) Studies on SiC, DLC and TiO2 thin films as piezoresistive sensor materials for high temperature application. *Microsyst Technol* 18:1027–1033

[11] Chang-Min Ohn., and Gwiy-Sang Chung. (2006). ohmic contacts of Polycystalline 3C-SiC Thin-Films Grown on Si (100) Wafers for Microsensors of Vehicle Engines. 2006 International Forum on Strategic Technology (IFOST) IEEE,), 301-304.

[12] J.S Shor, D Goldstein, A D Kurtz. (1991). Evaluation of ß-SiC for Sensors, Digest of Technical Papers, Transducers' 91, San Francisco, *IEEE*, 912-915.

[13] X. Song, S. Rajgolpal, J.M. Melzak, C.A. Zorman, and M.Mehregany. (2001). Development of a multilayer SiC surface micromachining process with capabilities and design rules comparable with conventional polysilicon surface micromachining. *Mat. Sci. Forum*, vol. 389-393, pp. 755-758,

[14] Hung-J Kuo, Christian A, Zorman and Mehran Mehregany.(2003). Fabrication and testing of single crystalline 3C-SiC devices using a novel SiC-on-insulator substrate. solid state sensors conference proceedings Boston, U.S.A., 742–745

[15] Dzung Viet Dao, Hoang-Phuong Phan, Afzaal Qamara and Toan Dinha .(2016). Piezoresistive effect of p-type single crystalline 3C-SiC on (111) plan. *RSC Adv*, 6, 21302-21307

[16] Hoang-Phuong Phan, Dzung Viet Dao, Koichi Nakamura, Sima Dimitrijev, and Nam-Trung Nguyen (2015). The Piezoresistive Effect of SiC for MEMS Sensors at High Temperatures: A Review. *Journal of Microelectromechanical Systems*, 24, 6, 1663-1677

[17] Sangsoo Noh, Xiaoan Fu, Li Chen, Mehran Mehregany. (2007). A study of electrical properties and microstructure of nitrogen-doped poly-SiC films deposited by LPCVD. Sensors and Actuators A 136, 613- 617

[18] Wijesundara, Muthu B.J., Gao, Di, Carraro, Carlo, Howe, Roger T., Maboudian, Roya (2003) Nitrogen doping of polycrystalline 3C-SiC films grown using 1,3-disilabutane in a conventional LPCVD reactor. *Journal of Crystal Growth*, 259 (1). 18-25

[19] Naoki Kubo, Takeshi Kawase, Shuichi Asahina, Nobuyuki Kanayama, Hiroshi Tsuda, Akihiro Moritani and Kuninori Kitahara. (2005). Epitaxial Growth of 3C-SiC on Si(111) Using Tetraethylsilane and Prevention of Void Formation at Heterointerfaces. *J. Surface Finishing Society of Japan*, 56, 227– 233

[20] Gwiy-Sang CHUNG and Kang-San Kim. (2007). Characteristics of Poly-Crystalline 3C-SiC Thin Films Grown for Micro/Nano-Electromechanical Systems by Using Single-Precursor Hexamethyldisilane. *Journal of the Korean Physical Society* 51, 1389- 1394

[21] Chien C.Chiu, Seshu B. Desu and Gang Chen, Ching Yi Tsai, William T Reynolds Jr. (1995). Deposition of epitaxial â-SiC films on porous Si(100) from MTS in a hot wall LPCVD reactor. *J. Mater.* Res. 10, 1099–1107

[22] M. A. Fraga, H. Furlan, R. S. Pessoa, M. Mass. (2014). Wide bandgap semiconductor thin films for piezoelectric and piezoresistive MEMS sensors applied at high temperatures: An overview. *Microsystem Technology*, 20, 9-21

[23] M. A. Fraga H. Furlan M. Massi I. C. Oliveira. (2010). Effect of nitrogen doping on piezoresistive properties of a-Si x C y thin film strain gauges. *Microsystem Technology,* 16, 925-930.

[24] A.Udayakumar, A Sri Ganesh, S. Raja, M. Balasubramanian (2011). Effect of intermediate heat treatment on mechanical properties of SiCf/SiC composites with BN interphase prepared by ICVI. *Journal of the European Ceramic Society*. 31, 1145-1153

[25] Latha H K E, A. Udayakumar and V Siddeswara Prasad. (2015). Microstructure and electrical properties of nitrogen doped 3C–SiC thin films deposited using methyltrichlorosilane. *Journal of Materials science in semiconductor processing*, 29, 117-123

[26] Stroud. P.T. (1972). "Some effects of strain and temperature on the resistance of oxygen ion implanted titanium thin films", *Thin solid films,* vol. 10, 205-215

[27] M. A. Fraga, H. Furlan, M. Massi, L. A. Rasia, and C. F. R. Mateus (2011). evaluation of the performance of piezoresistive strain sensors based on different semiconductor thin films. 21st Brazilian Congress of Mechanical Engineering, October 24-28,

[28] Jingchun Zhang., Roger T. Howe., and Roya Maboudian. (2006). Electrical Characterization of n-Type Polycrystalline 3C- Silicon Carbide Thin Films Deposited by 1,3-Disilabutane. *Journal of the Electrochemical Society*, 153, G548-G551

[29] Jingchun Zhang., Carlo carraro., Roger T Howe and Roya Maboudian (2007). Electrical, mechanical and metal contact properties of polycrystalline 3C-SiC films for MEMS in harsh environments. *Surface & Coatings Technology*, 201, 8893-8898