Materials Science Forum Vols. 645-648 (2010) pp 143-146
Online available since 2010/Apr/29 at www.scientific.net
© (2010) Trans Tech Publications, Switzerland
doi:10.4028/www.scientific.net/MSF.645-648.143

Growth rate effect on 3C-SiC film residual stress on (100) Si substrates

R. Anzalone[1,2,*], C. Locke[3], J. Carballo[3], N. Piluso[1], A. Severino[1], G. D'Arrigo[1], A. A. Volinsky[4], F. La Via[1], and S.E. Saddow[3,5]

[1]IMM-CNR, sezione di Catania, Stradale Primosole 50, 95121, Catania, Italy
[2]University of Catania, Phys. Dept., via Santa Sofia 64, 95125, Catania, Italy
[3]Dept. of Electrical Engineering, USF, Tampa, FL 33620, USA
[4]Dept. of Mechanical Engineering, USF Tampa, Florida 33620, USA
[5]Dept. of Molecular Pharmacology and Physiology, USF, Tampa, FL 33620, USA
[*]corresponding author: ruggero.anzalone@imm.cnr.it

Key words: 3C-SiC heteroepitaxy, growth rate, residual stress.

Abstract. SiC is a candidate material for micro- and nano-electromechanical systems (MEMS and NEMS). In order to understand the impact that the growth rate has on the residual stress of CVD-grown 3C-SiC hetero-epitaxial films on Si substrates, growth experiments were performed and the resulting stress was evaluated. Film growth was performed using a two-step growth process with propane and silane as the C and Si precursors in hydrogen carrier gas. The film thickness was held constant at ~2.5 μm independent of the growth rate so as to allow for direct films comparison as a function of the growth rate. Supported by profilometry, Raman and XRD analysis, this study shows that the growth rate is a fundamental parameter for low-defect and low-stress hetero-epitaxial growth process of 3C-SiC on Si substrates. XRD (rocking curve analysis) and Raman spectroscopy show that the crystal quality of the films increases with decreasing growth rate.

From curvature measurements, the average residual stress within the layer using the modified Stoney's equation was calculated. The results show that the films are under compressive stress and the calculated residual stress also increases with growth rate, from -0.78 GPa to -1.11 GPa for 3C-SiC films grown at 2.45 and 4 μm/h, respectively.

Introduction

Silicon Carbide (SiC) has long been recognized as an excellent material for high-power, high-frequency and high-temperature electronics due its outstanding electrical and thermal properties. SiC is now receiving added attention for its potential applications in micro- and nano-electromechanical systems (MEMS and NEMS) due its exceptional electrical, mechanical and chemical properties as compared with silicon (Si), which is currently the leading material in these technology areas.

Among the polytypes of SiC, cubic SiC (i.e., 3C-SiC) possesses unique properties, such as high electron drift velocity, which is more suitable for high-frequency power devices [1]. However the most important property of 3C–SiC is that it can be grown on large diameter Si substrates, which makes it very suitable for many industrial applications. The large area substrates offer the possibility for low-cost batch processing, which makes SiC more attractive for sensor and device applications. The heteroepitaxy of SiC on Si substrates results in the 3C-SiC/Si hetero-system, which is also a very interesting material system by itself [2].

Unfortunately, due to high residual stresses (which normally arrise during the growth process) and crystal defects stemming from the large lattice constant mismatch and the thermal expansion coefficient difference between SiC and Si, the use of SiC in Si-based MEMS fabrication techniques has been somewhat limited. The resulting stress relaxation has important implications with regard to processing, epitaxial quality, and the properties of films and coatings that undergo large temperature fluctuations [3]. Therefore, it is necessary to reduce and control the residual stress in 3C-SiC films for the design and performance of MEMS devices.

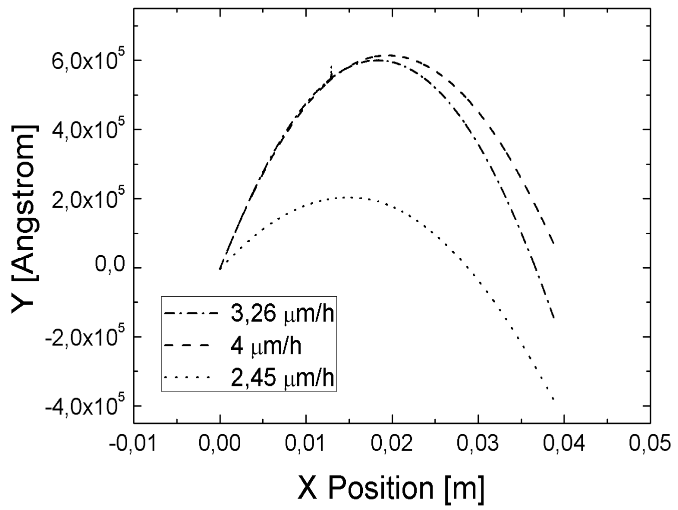

Fig. 1. Wafer profile measurements along the 50 mm diameter (orthogonal to the flow direction). These profiles are used to calculate the radius of curvature for estimating the residual stress.

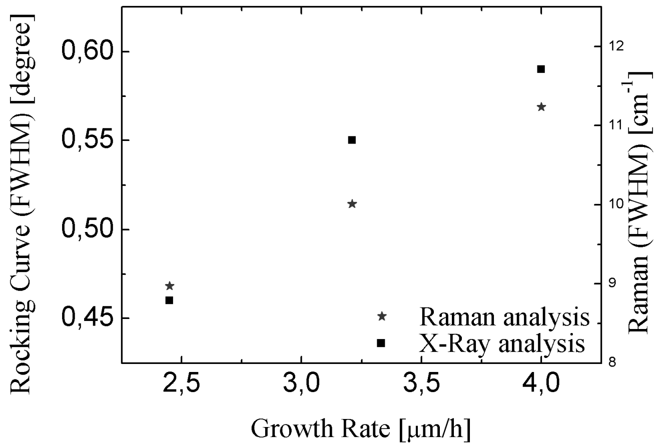

Fig. 2. XRD rocking curve data (square points) and Raman spectroscopy data of the TO vibration mode (star points). Both measurements were performed at the wafer centre.

Experimental

For this experiment, three equi-thick 3C-SiC films were grown at three different deposition rates on individual 50 mm diameter (100) Si wafers. A high-quality 3C-SiC epitaxial growth process was used to reduce the defect density in the growing layer and to improve its crystalline quality [4]. The entire deposition consisted of two different steps, namely carbonization followed by growth. During carbonization, propane (C_3H_8) and hydrogen (H_2) flow through the reactor while the temperature is ramped to 1135 °C at a process pressure of ~ 400 Torr. Once the temperature stabilizes at 1135 °C, the wafer is held under a steady-state condition of gases flow, temperature, and pressure for 4 minutes. This allows the conversion of the Si wafer surface to a 3C-SiC buffer layer. This process was adapted to reduce the formation of voids underneath the interface between 3C-SiC and Si, due to the selective out-diffusion of silicon from the substrate by a reaction of silicon with a suitable hydrocarbon gas [5]. After the 4 minute carbonization plateau, the growth phase began. Silane (SiH_4) was then introduced into the gas stream, the temperature was ramped to 1370 °C while the process pressure was maintained at 400 Torr. The SiH_4 flow was incrementally increased with the C_3H_8 flow in order to maintain a stoichiometric ratio conducive for growth of crystalline 3C-SiC. This process was used for the three 3C-SiC films grown on (100) Si substrates [6] at 2.45, 3.21, 4 µm/h of growth rate measured at the wafer centre, respectively sample #1, #2 and #3 .

Due to the lack of wafer rotation during film growth the sample thickness varied between 2.1 µm (center) and 2.5 µm (edge) across the 50 mm wafer diameter in a direction parallel to the main flat (transverse to the flow direction), while the thickness varied between 3.1 (upstream) and 2.2 µm (downstream) along a direction perpendicular to the main flat (flow direction).

Results and Discussion

The stress in the 3C-SiC films was determined using a profilometer system, which measures the changes in the surface profile caused by deposition of a stressed thin film. The stress (σ) was calculated using Stoney's equation: $\sigma = Eh^2/[(1-\nu)6\Delta Rt]$. The values of Young's modulus (E) along the <100> axis and Poisson's ratio (ν) for silicon were taken to be 130 GPa and 0.279, respectively [7]. Variables h and t are the individual thicknesses of the substrate and the film, respectively. ΔR is the change in radius of curvature determined from $(1/R)=(1/R_2)-(1/R_1)$, where R_1 is the average radius of the substrate prior to film deposition. The substrate deforms to a new radius R_2 after the film is deposited.

The film profiles are shown in Fig. 1 for the three growth rates. The profiles were measured along the direction parallel to the main flat along the 50 mm wafer diameter to avoid any errors caused by film thickness variations. Nevertheless the non-symmetry of the measured data is due to the slight bow of the wafer. From this analysis all three films are seen to be under compressive stress.

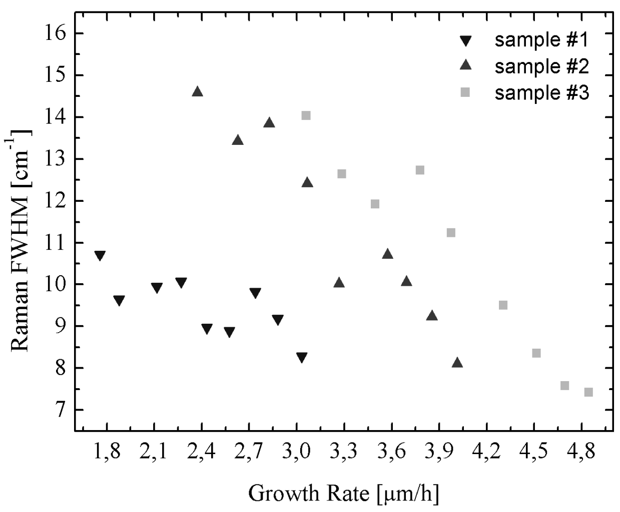

Fig. 3. TO Raman data as a function of the growth rate for three different equi-thick single crystal 3C-SiC films.

The crystalline quality of the samples was determined by high-resolution X-ray diffraction measurements of the (200) 3C-SiC diffraction plane via rocking curve analysis. The relative density of defects was determined from the full-width–at-half-maximum (FWHM) value of the rocking curve due to the proportionality between the defect density and the FWHM value [8]. Rocking curve measurements were performed with a Phillips X'pert Diffractometer and displayed FWHM values of 0.46°, 0.55°, and 0.59° for 3C-SiC films grown at 2.45, 3.21, 4 μm/h, respectively (Fig 2). The crystalline quality was also measured by Raman spectroscopy, where FWHM values of the TO vibration mode of 7.41 cm^{-1}, 8.08 cm^{-1} and 8.57 cm^{-1} were found for 3C-SiC films grown at the corresponding growth rates (Fig 3). As was observed from the X-Ray rocking curves, the Raman FWHM of the TO peak is closely related to the defects inside the film, therefore the crystalline quality increases with decreasing growth rate.

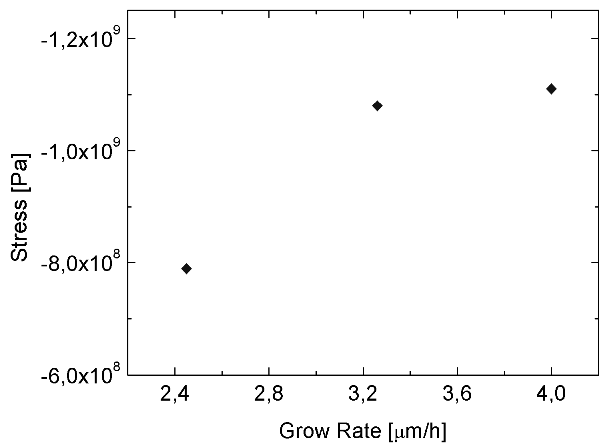

Fig. 4. Average residual stress in 3C-SiC films as a function of the growth rate. Data calculated by applying the modified Stoney's equation to the data of Fig. 1.

Due to the lack of wafer rotation during film growth a difference in thickness between the upstream and downstream sides of the wafer is observable. Multiple Raman analyses were performed along the flow direction on the three samples used for this experiment. Figure 3 shows the Raman FWHM analysis on the three different films measured along the flow direction. From the comparison between the different samples at a fixed thickness value (see Fig. 2 where a center of the three samples is compared), one could determine that the quality of the sample increases with decreasing growth rate. The same results reported in Fig 2, but not shown in this work, are also observable by a comparison in the lower part of the wafer (the thick part) and the upper part (the thin part).

From the measured sample curvature, one can determine the average residual stress within the layer using the modified Stoney's equation that accounts for the film thickness non-uniformity [9]. The results of the thin film stress calculations are reported in Fig. 4, where the calculated stress increases with the growth rate, from -0.78 GPa to of -1.11 GPa for 3C-SiC films grown at 2.45 and 4 μm/h, respectively. The minus sign means that film is in compression. The value used for the growth rate for the three different films is referred to at the centre of the wafer.

Conclusions

In this work a study of the influence of the growth rate on the residual stress and crystal quality of hetero-epitaxial 3C-SiC films grown on (100) Si has been presented.

With a common 3C-SiC epitaxial growth process, used to reduce the defect density in the grown layer and to improve its crystalline quality, three equi-thick 3C-SiC films were grown by Chemical Vapor Deposition at different deposition rates on 50 mm diameter (100) Si wafers.

Raman and X-Ray analyses were performed and showed the growth rate influence on the film quality, when the FWHM of (of XRD and Raman signal) decreased with increasing growth rate at fixed thickness value. Is well known that the FWHM is closely related to the amount of defects inside the film, so decreasing the growth rate appears to have improved the film quality.

Acknowledgments

The 3C-SiC growth in Prof. Saddow's laboratory was supported by the Army Research Laboratory under Grant No. DAAD19-R-0017 (B. Geil) and the Office of Naval Research under Grant No. W911NF-05-2-0028 (C. E. C. Wood).

This work was also supported from the PIA-INNOVATION Project No E01/0615/2 financed from the Ministry of University and Research and from the Ministry of Production Activity of Italy. Alex A. Volinsky would like to acknowledge support from NSF under CMMI-0600266 grant.

References

[1] W. E. Nelson, F. A. Halden, and A. Rosengreen, J. Appl. Phys., 37 (1966) p.33.

[2] P.M. Sarro, Sens. Actuators, A 82 (2000) p. 210.

[3] Deva Reddy, A.A. Volinsky, C. Frewin, C. Locke, S.E. Saddow, Mat. Res. Soc. Symp. Proc. 1049 (2008) p. AA3.6.

[4] C.L. Frewin, C. Locke, J. Wang, P. Spagnol and S.E. Saddow, Journal of Crystal Growth, Vol. 311, Issue 17 (2009) pp. 4179-4182.

[5] A. Severino, G. D'Arrigo, C. Bongiorno, S. Scalese, G. Foti, and F. La Via, J. Appl. Phys., 102 (2007) p. 023518.

[6] M. Reyes, Y. Shishkin, S. Harvey, S.E. Saddow, Mat. Res. Soc. Symp. Proc. 911 (2006) p. 79.

[7] J. J. Wortman and R. A. Evans, J. Appl. Phys. 36 (1965) p. 153.

[8] B.D. Cullity, Elements of X-ray Diffraction, second ed., Addison-Wesley, Reading, MA, USA, (1978) p. 284.

[9] A.A. Volinsky, G. Kravchenko, P. Waters, J. Deva Reddy, C. Locke, C. Frewin, S.E. Saddow, Mat. Res. Soc. Symp. Proc. Vol. 1069 (2008) p. D3.5.

Silicon Carbide and Related Materials 2009
doi:10.4028/www.scientific.net/MSF.645-648

Growth Rate Effect on 3C-SiC Film Residual Stress on (100) Si Substrates
doi:10.4028/www.scientific.net/MSF.645-648.143

References

[1] W. E. Nelson, F. A. Halden, and A. Rosengreen, J. Appl. Phys., 37 (1966) p.33.

[2] P.M. Sarro, Sens. Actuators, A 82 (2000) p. 210.
doi:10.1016/S0924-4247(99)00335-0

[3] Deva Reddy, A.A. Volinsky, C. Frewin, C. Locke, S.E. Saddow, Mat. Res. Soc. Symp. Proc. 1049 (2008) p. AA3.6.

[4] C.L. Frewin, C. Locke, J. Wang, P. Spagnol and S.E. Saddow, Journal of Crystal Growth, Vol. 311, Issue 17 (2009) pp. 4179-4182.
doi:10.1016/j.jcrysgro.2009.06.037

[5] A. Severino, G. D'Arrigo, C. Bongiorno, S. Scalese, G. Foti, and F. La Via, J. Appl. Phys., 102 (2007) p. 023518.
doi:10.1063/1.2756620

[6] M. Reyes, Y. Shishkin, S. Harvey, S.E. Saddow, Mat. Res. Soc. Symp. Proc. 911 (2006) p. 79.

[7] J. J. Wortman and R. A. Evans, J. Appl. Phys. 36 (1965) p. 153.
doi:10.1063/1.1713863

[8] B.D. Cullity, Elements of X-ray Diffraction, second ed., Addison-Wesley, Reading, MA, USA, (1978) p. 284.

[9] A.A. Volinsky, G. Kravchenko, P. Waters, J. Deva Reddy, C. Locke, C. Frewin, S.E. Saddow, Mat. Res. Soc. Symp. Proc. Vol. 1069 (2008) p. D3.5.