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Polycrystalline 3C-SiC thin films deposited by dual precursor LPCVD for MEMS applications

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Abstract

Polycrystalline silicon carbide (poly-SiC) thin films were deposited in a high-throughput, low pressure chemical vapor deposition (LPCVD) furnace using dichlorosilane (SiH_2Cl_2) and acetylene (C_2H_2) dual precursors. The deposition temperature ranged from 800 to 900 °C, and the pressure was varied between 0.46 and 5.00 Torr. Poly-SiC deposition with good uniformity is demonstrated on 150 and 100 mm diameter (1 0 0) silicon wafers. X-ray photoelectron spectroscopy (XPS) data indicated that stoichiometric SiC films were deposited over the entire range of temperatures and pressures. X-ray diffraction (XRD) data showed that all the stoichiometric films were highly textured (1 1 1) oriented, polycrystalline 3C-SiC (poly-SiC). The surface morphology and roughness as determined by atomic force microscopy (AFM) and scanning electron microscopy (SEM) indicated that the surface features consisted of spherulitic aggregates, and the surface roughness increased with increasing film thickness. The residual stress of the films varied from about 700 MPa (tensile) to −100 MPa (compressive) with the deposition pressure changing from 0.46 to 5.00 Torr at a deposition temperature of 900 °C. This observation indicates that the residual stress in poly-SiC can be controlled during deposition without affecting the process thermal budget.

Keywords: Silicon carbide; LPCVD; Chemical vapor deposition; Dual precursors; MEMS

1. Introduction

Recent advances in SiC surface and bulk micromachining techniques combined with outstanding mechanical, electrical and chemical properties make SiC a viable complement to silicon for microelectromechanical systems (MEMS) [1,2]. SiC is an excellent material for reliable and robust MEMS operating in harsh environments [3]. Polycrystalline 3C-SiC (poly-SiC), in particular, is a motivating consideration as a structural material since a variety of substrate materials (i.e., polysilicon, SiO_2) can readily be used as sacrificial and micromolding layers to create multilayered SiC devices with a complexity comparable to polysilicon surface micromachining [4,5]. Early SiC MEMS advances were realized using an atmospheric pressure chemical vapor deposition reactor constructed specifically for epitaxial growth of 3C-SiC, which, by its design and selection of precursors, required temperatures in excess of what would be necessary to produce poly-SiC. Encouraged by these initial advances, there have been several efforts in the development of poly-SiC deposition processes for MEMS applications in recent years. Low pressure chemical vapor deposition (LPCVD) has been demonstrated to be a viable approach to produce high quality poly-SiC films at substrate temperatures that are reasonably low [6–14].

Continued advancement of poly-SiC MEMS depends on the successful development of deposition techniques that are comparable in method and scale to conventional polysilicon LPCVD and capable of producing poly-SiC films that are suitable for MEMS products. Previously, we reported the successful development of a high-throughput, LPCVD furnace on large-area substrates using SiH_2Cl_2 and C_2H_2 dual precursors in a brief communication [14]. This work reports on an effort to develop deposition methods for this furnace

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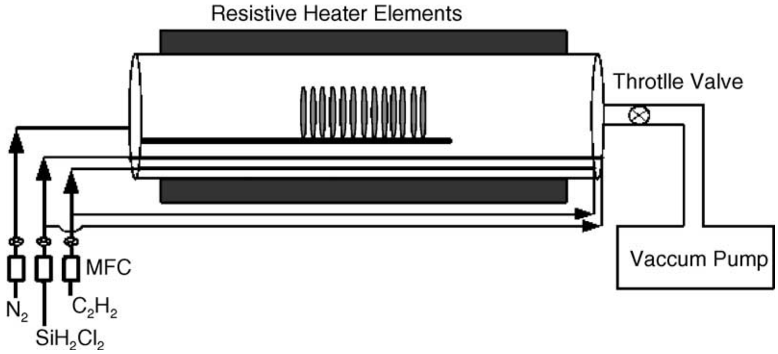

Fig. 1. Schematic diagram of the high-throughput, large volume, hot-wall LPCVD system.

that can be used to produce poly-SiC films with the physical properties required for SiC MEMS. The effects of deposition temperature and pressure on deposition rate, crystalline orientation, film surface roughness and residual stress were investigated.

Even though only (1 0 0) silicon substrates are used to study the residual stress and elastic modulus of the deposited films, the crystalline microstructure and physical properties of poly-SiC films deposited on sacrificial layers such as silicon dioxide or polysilicon are expected to be very similar. A previous study by us indicated that poly-SiC deposition rate and residual stress were nearly identical for depositions on silicon and SiO_2 under the same deposition conditions, and the films deposited on both substrates had (1 1 1) oriented texture [15]. The Young's modulus estimated from poly-SiC resonators fabricated from the film deposited on a SiO_2 sacrificial layer is very close to the Young's modulus estimated from poly-SiC diaphragms fabricated from the film deposited on silicon [16,17].

2. Experimental

SiC films were deposited on 100 mm-diameter (1 0 0) silicon wafers by LPCVD in a conventional, hot-wall horizontal furnace that was modified to perform SiC depositions [14]. Fig. 1 shows a schematic diagram of the LPCVD reactor setup. The reaction chamber consisted of a cylindrical quartz tube with disc-type, water cooled, metal flanges capping both ends. The reaction chamber was measuring 2007 mm in length and 225 mm in inner diameter. Wafers were held in a SiC boat that rested on a SiC paddle attached to the front flange. The boat and paddle were oriented such that the wafers were positioned at the center of the furnace tube. Precursor gases, in this case SiH_2Cl_2 and C_2H_2, entered the reaction chamber through two small-diameter injector tubes, one for each precursor, positioned parallel to the principle axis of the furnace and beneath the wafer boat. The vacuum system consisted of a roots blower and a mechanical pump that could reach a base pressure of <1 mTorr in a fully loaded system. It is worthwhile noting that although 100 mm-diameter wafers were used in this study, the reaction chamber is sized such that poly-SiC films can be deposited on 150 mm-diameter wafers, as evidenced by the SiC-coated Si wafer shown in the optical photograph of Fig. 2. The film thickness standard deviation for ∼700 nm-thick poly-SiC films deposited on 150 mm-diameter silicon wafers is ∼48 nm, while the film thickness standard deviation for ∼700 nm-thick poly-SiC films deposited on 100 mm-diameter wafers is ∼20 nm.

Although the furnace is capable of holding up to 100 wafers, each load consisted of 25, 100 mm-diameter wafers evenly distributed in a single, 50 slot silicon carbide boat. The first and last five wafers were always designated as baffling wafers to stabilize the gas flow within the boat. Wafers in position 6, 10, 13, 16 and 20 (13 is the center wafer in the boat) from the loading end were designated as samples for this study. Prior to loading the furnace, all wafers were cleaned using a standard RCA cleaning procedure. As shown in Table 1, the first set of depositions was performed at a fixed pressure of nominally 0.4 Torr at temperatures from 750 to 900 °C. A second set was performed at 900 °C using pressure

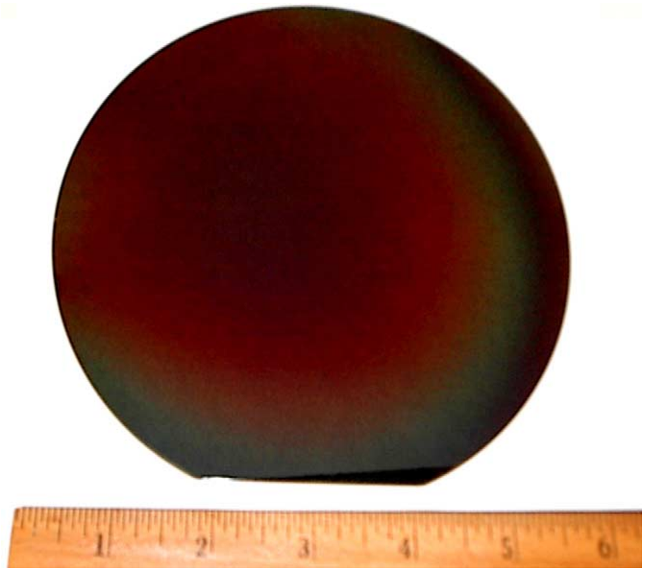

Fig. 2. Optical photograph of a 150 mm-diameter Si wafer coated with a 700 nm thick poly-SiC film deposited in the LPCVD system; the unit of the numbers on the scale is inch.

Table 1
Film thickness and residual stress of poly-SiC films deposited at the temperatures used in this study

Sample	*T* (°C)	Pressure (Torr)	Thickness (nm)	Stress (MPa)
1	800	0.42	156	−96
2	850	0.43	324	342
3	900	0.46	474	695

settings from 0.46 to 5 Torr as listed in Table 2. In general, the deposition time varied from 1 to 2 h, but in several cases, longer times were used to deposit thicker films. For all depositions, the flow rates of SiH_2Cl_2 and C_2H_2 (5% in H_2) were held constant at 54 sccm and 180 sccm, respectively. These flow rates correspond to a Si-to-C molar ratio in the gas flow of 3:1.

Following each deposition, the thickness of the films was measured optically using a Nanospec 4000 AFT spectrophotometer. Residual stresses were determined by measuring the curvature of each silicon wafer before and after film deposition using a laser-based curvature measurement system (Frontier Semiconductor Measurement, FSM 120). Since SiC films were deposited on both sides of the silicon wafers, reactive ion etching in a CHF_3/O_2 mixture was used to remove the film deposited on the backside. For each stress measurement, six scans were performed across the wafer surface, rotating the wafer by 30° between each scan. The repeatability of this technique was studied by successive measurements on the same wafer and was found to be within ±5 MPa of a measured value.

X-ray photoelectron spectroscopy (XPS) was used to analyze the chemical composition of the as-deposited films. The analysis was performed using a Perkin-Elmer 5500 XPS system that was configured with an Al Kα line X-ray source at 1486.6 eV. The base pressure in the analysis chamber was around $\sim 10^{-9}$ Torr. The chemical composition of the films was obtained by analyzing the Si 2p, C 1s and O 1s peaks [10,14]. Corrections to the binding energy shifts due to steady-state charging of the samples were made by taking the C 1s in SiC as reference at 282.7 eV [14]. Quantification of the XPS data was performed by normalizing the area of each peak using atomic sensitivity factors. Sputter depth profiling using a 4 keV argon beam was sometimes performed to expose the bulk region of the samples in order to establish the baseline bulk concentrations of the various elements as well as an upper bound on the thickness of any near-surface region. The crystal orientation was studied by conventional X-ray diffraction using a Scintag diffractometer with a Cu Kα X-ray tube (λ = 1.542 Å), and configured in a symmetrical θ–2θ mode. The surface morphology and roughness were determined by atomic force microscopy (AFM) (Digital Instrument Nanoscope Multimode III). The thickness and surface morphology were also characterized by scanning electron microscopy (SEM) using a Hitachi S-4500 field emission microscope.

Table 2
Thickness and residual stress of poly-SiC films deposited at 900 °C and pressures ranging from 0.46 to 5.00 Torr

Sample	*T* (°C)	Pressure (Torr)	Thickness (nm)	Stress (MPa)
1	900	0.46	474	695
2	900	1.00	425	541
3	900	2.50	531	172
4	900	2.85	269	−41
5	900	3.75	503	−87
6	900	5.00	615	−98

3. Results and discussion

3.1. *The effect of temperature on SiC deposition at fixed pressure*

XPS spectra were preformed for films deposited as a function of temperature at fixed pressure. Deconvolution of the C 1s (282.7 eV) and Si 2p (100.2 eV) peaks indicate that all films deposited at 800 °C and above were stoichiometric SiC. There was no detectable film deposition at 750 °C. An oxygen impurity level of 0.4–1 wt.% was typical for these films and was correlated to the procedure used during loading and pump-down as well as the condition of the vacuum seals. A chemical stability test was performed by immersing at least one wafer from each run into a 4 L, 3.57 M aqueous KOH etch solution at 55 °C for 24 h; conditions that are sufficient to completely dissolve unprotected 500 μm-thick Si wafers but should not etch SiC. For all samples grown at 800 °C and above, the wafers were completely unetched, indicating that the films were continuous and free of pinholes on both sides.

XRD spectra of the films deposited on Si(1 0 0) at 800, 850 and 900 °C are shown in Fig. 3. The spectra exhibit a strong peak positioned at $2\theta = 35.6°$, indicating a (1 1 1) 3C-SiC polycrystalline texture to the films. Peaks corresponding to the (2 0 0), (2 2 0), and (3 1 1) 3C-SiC orientations are not present. The average of the full width at half maximum intensity (FWHM) decreased from 0.41° for the film deposited at 800 °C to 0.35° for the film deposited at 850 °C to 0.31° for

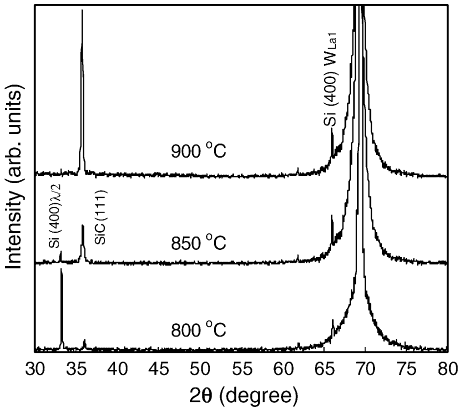

Fig. 3. XRD spectra of SiC films deposited at a pressure of ∼0.4 Torr and temperatures ranging from 800 to 900 °C.

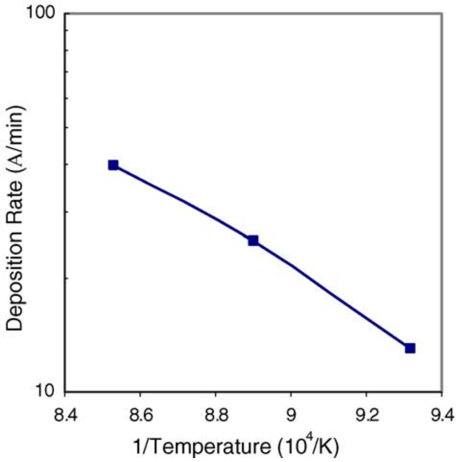

Fig. 4. Arrhenius plot of the SiC film deposition rate as a function of temperature.

the film deposited at 900 °C. Fig. 4 shows an Arrhenius plot of the deposition rate as a function of temperature from 800 to 900 °C. From this plot, an activation energy of 33.5 kcal/mol was calculated for the formation of SiC. As shown in Fig. 4, the deposition rate increases as the deposition temperature increases, suggesting that the deposition process in this temperature region is kinetically controlled, with the controlling step being the SiC-forming reaction. This finding is in contrast to the conclusions reported in Ref. [12], where a constant deposition rate was reported for LPCVD poly-SiC films deposited over the same temperature range, but when an alternating supply of SiH_2Cl_2 and C_2H_2 precursors and a pressure of 150 mTorr were used.

As stated above, the lowest temperature that yielded measurable SiC films was 800 °C, which is consistent with the findings reported in Ref. [12]. Although it has been reported that amorphous SiC films can be deposited at 750 °C from SiH_2Cl_2 and C_2H_2 precursors when using a deposition pressure of 9 Torr [13], we did not observe this for films deposited in the pressure range used in our study. In addition, it was reported that poly-SiC films deposited at 900 °C and 9 Torr were polycrystalline with a (2 2 0) 3C-SiC orientation [13], which is in stark contrast to the (1 1 1) 3C-SiC oriented poly-SiC films deposited at the same temperature in our reactor. By comparing our findings with those reported in Refs. [12] and [13], it can be inferred that higher deposition pressures lead to a decrease in the threshold temperature for the deposition of stoichiometric SiC films. More importantly, the microstructure appears to be dependent on deposition pressure as well, with higher pressures favoring the formation of polycrystalline (1 1 0) 3C-SiC and lower pressures favoring the (1 1 1) 3C-SiC texture. These findings are significant, since it has been shown that the texture of the poly-SiC affects the mechanical properties important for MEMS, such as Young's modulus and residual stress [18].

Fig. 5 shows AFM surface profiles for the films deposited at 800 and 900 °C. As shown in these micrographs, the surface morphology of the poly-SiC film consists of spherical, particle-like grains. The average surface grain size for the films deposited at 800 and 900 °C are 42 and 47 nm, respectively. Likewise, the average surface roughness for the films deposited at 800 and 900 °C are 6.9 and 7.7 nm, respectively.

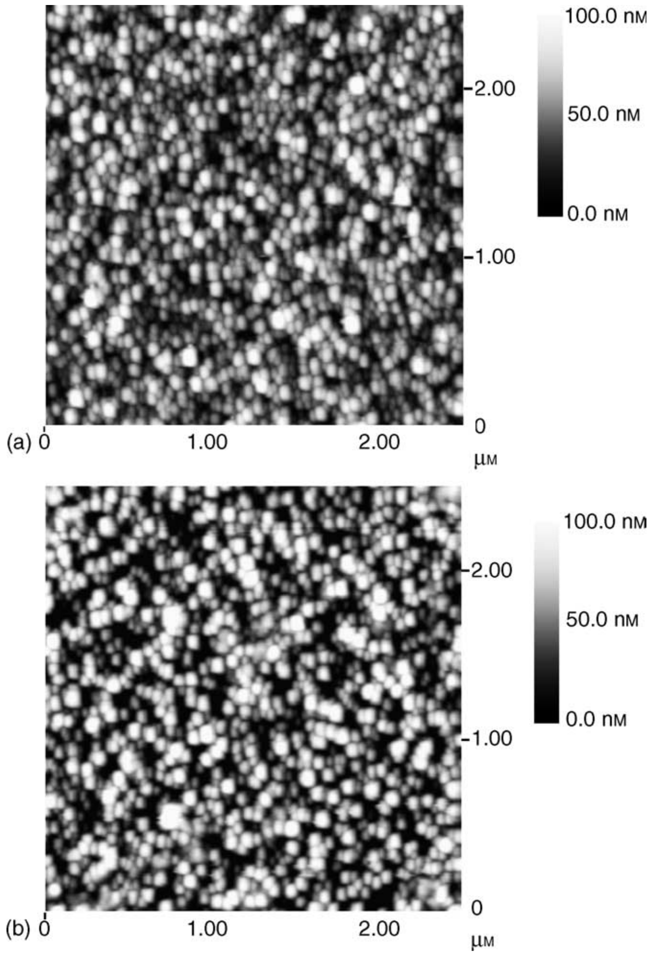

Fig. 5. AFM micrographs of poly-SiC films grown at a nominal pressure of 0.46 Torr and: (a) 800 °C (thickness = 156 nm, rms roughness = 6.9 nm), and (b) 900 °C (thickness = 474 nm, rms roughness = 7.7 nm).

Fig. 6 shows SEM micrographs of the surface of SiC films deposited at 900 °C, 0.46 Torr for deposition periods of 1 and 7.5 h. The thickness of SiC films in Fig. 6(a) and (b) are 0.24, 1.49 µm, respectively. The surface roughly consists of spherulites formed by small-sized crystallite aggregates regardless of thickness. The thicker film (Fig. 6(b)) has much larger spherulitic aggregates than the thinner film, indicating that the surface grain size increases with increasing thickness.

Table 1 lists the residual stresses in the poly-SiC films for wafers positioned in the center slot of the boat during film growth. For films deposited using otherwise identical process parameters, the residual stress in the films deposited at 800 °C was moderately compressive (−96 MPa), but for films deposited at 900 °C, the stress was highly tensile (695 MPa). In general, single crystalline 3C-SiC films deposited on Si wafers using CVD methods have tensile residual stresses as

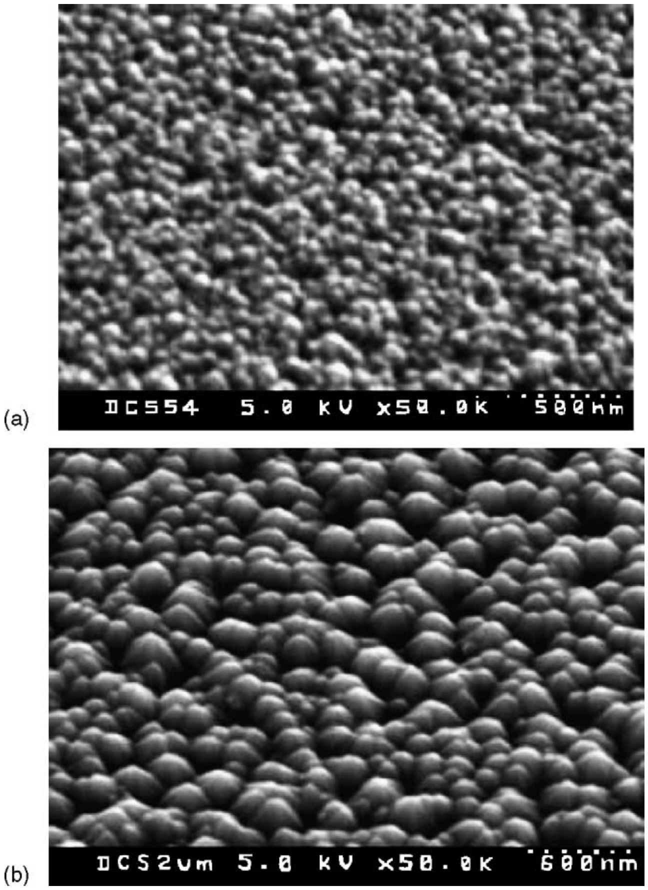

Fig. 6. SEM micrographs of a poly-SiC film grown at 900 °C and 0.46 Torr for (a) 1 h and (b) 7.5 h.

a result of the thermal mismatch between SiC and Si, and the high temperatures in the deposition process as well as the lattice mismatch between the two materials. Poly-SiC films deposited by APCVD in epitaxial reactors also exhibit tensile residual stresses for much the same reasons [19]. Although not commonly observed, a temperature dependent transition between compressive stresses and tensile stresses has previously been reported for poly-SiC films grown on Si by LPCVD at 0.4 Torr over a temperature range of 930–1150 °C using tetramethylsilane as a single Si- and C-containing precursor, with compressive stresses observed for the lower deposition temperatures [11]. To the best of our knowledge, we are the first to report of such a transition using dual precursors as Si- and C-containing sources and deposition temperatures below 900 °C. These observations are significant in that they suggest that mechanisms other than thermal mismatch have a significant effect on residual stress as the deposition temperature is reduced below 1000 °C.

3.2. The effect of pressure on SiC deposition at fixed temperature

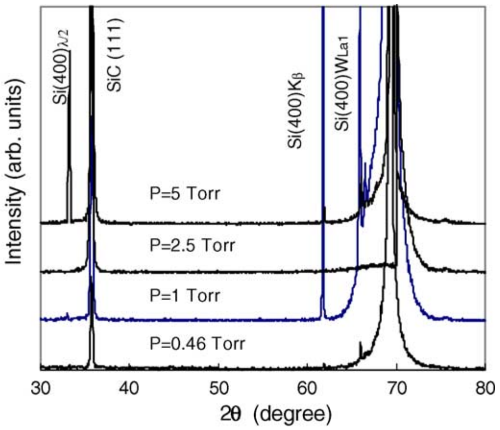

Fig. 7. XRD spectra of SiC films deposited at 900 °C and pressures ranging from 0.46 to 5.00 Torr.

Fig. 7 shows typical XRD spectra from poly-SiC films deposited on Si(1 0 0) at 900 °C and pressures ranging from 0.46 to 5.00 Torr. In all cases, the films exhibit a strong peak positioned at $2\theta = 35.6^\circ$, indicating a (1 1 1) 3C-SiC polycrystalline texture regardless of deposition pressure. The average FWHM generally increased as the deposition pressure increased from 0.46 to 5.00 Torr. Fig. 8 shows the effect of deposition pressure on SiC deposition rate. SiC deposition rate has a linear relationship with logarithmic deposition pressure for pressures above 2.50 Torr and exhibits a deposition rate transition region for deposition pressures between 1.00 and 2.50 Torr. The deposition rate is controlled by the precursor diffusion rate of the reaction species to the surface of the wafers and chemical reaction kinetics of SiC formation. This study by itself is not sufficient to reach a conclusion as to the cause of this transition.

Fig. 9 shows typical AFM surface profiles for films deposited at 1.00 and 3.75 Torr, respectively. As seen in these images, the surfaces consist of spherical, particle-like grains with average surface grain sizes ranging from 72 nm for the films deposited at 1.00 Torr to 95 nm for the films deposited at 3.75 Torr. Recalling from Fig. 5(b) that the surface grain size of a film deposited at 900 °C and 460 mTorr was 47 nm, the data shown herein indicate that the surface grain size increases with increasing deposition pressure at similar film

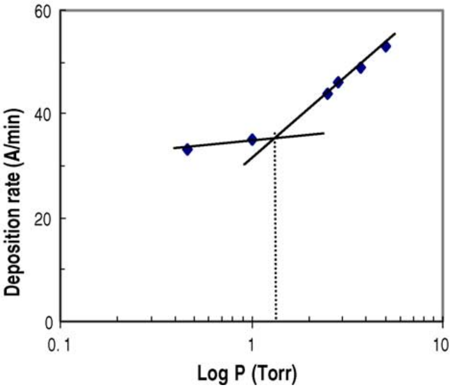

Fig. 8. Pressure-dependent deposition rate for poly-SiC films deposited at 900 °C.

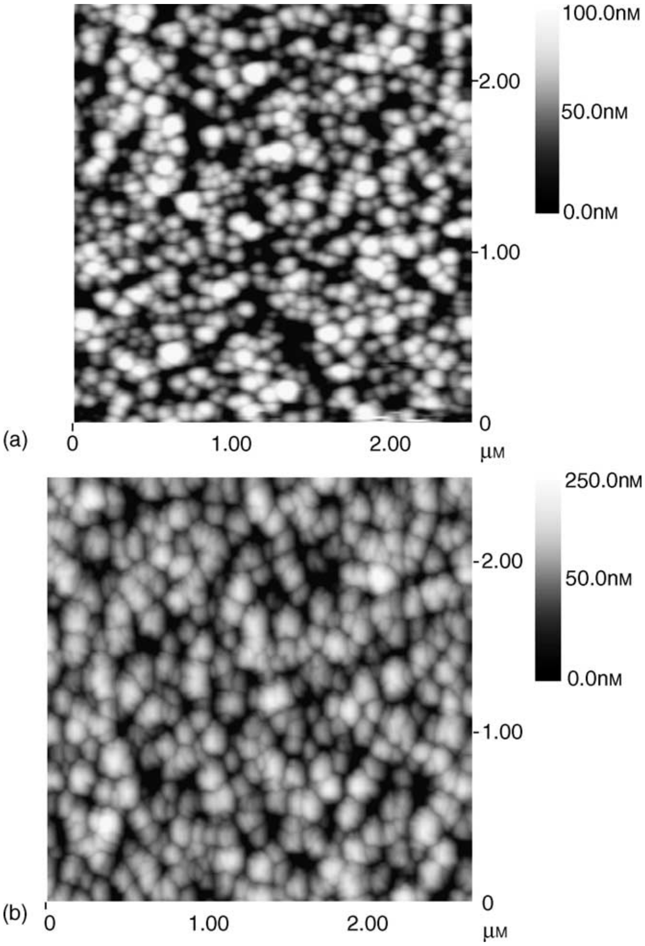

Fig. 9. AFM micrographs of poly-SiC films grown at: (a) 900 °C and 1.00 Torr (thickness = 425 nm, rms roughness = 8.9 nm), and (b) 900 °C and 3.75 Torr (thickness = 503 nm, rms = 15.4 nm).

thicknesses for films deposited at 1.00 and 3.75 Torr as listed in Table 2. Like the deposition rate and grain size, the surface roughness exhibits a dependence on deposition pressure, with the average surface roughness for the film deposited at 1.00 Torr being 8.9 nm, while the average surface roughness for the film deposited at 3.75 Torr being 15.4 nm.

Table 2 summarizes the residual stress data for wafers from the center slot for the pressure related experiments. In general, the residual stress depends on temperature, pressure and film thickness. At a deposition temperature of 900 °C, the residual stress ranged from 695 MPa (tensile) for a deposition pressure of 0.46 Torr to −98 MPa (compressive) for a deposition pressure of 5.00 Torr. To the best of our knowledge, these data are the first to indicate a connection between residual stress and deposition pressure for poly-SiC films [20]. The pressure dependence in the residual stress may be induced by changes in grain size and grain boundaries [20–22]. Regardless of the actual physical mechanism, the fact that the stress changes state from tensile to compressive over a practical range of pressures enables the use of deposition pressure as a controlling parameter in the production of poly-SiC films with the desired value of residual stress without increasing thermal budget.

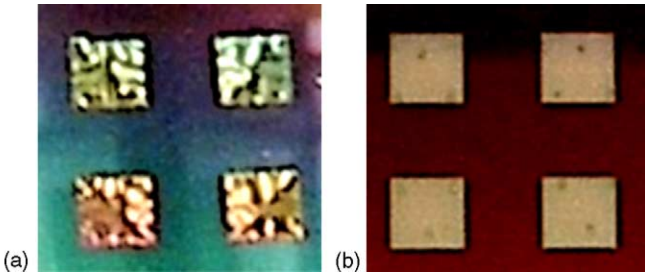

Fig. 10. A 4 mm × 4 mm SiC membranes made from poly-SiC films deposited at: (a) 900 °C and 5.00 Torr, membrane thickness 0.6 μm and (b) 900 °C and 2.50 Torr, membrane thickness 1.0 μm.

Fig. 10 shows optical photographs of 4 mm × 4 mm size membranes made from poly-SiC films deposited at 900 °C and 5.00 Torr, as well as 900 °C and 2.50 Torr. The membranes were fabricated using conventional KOH-based Si bulk micromachining techniques [23,24]. The thickness of the membranes in Fig. 10(a) is 0.6 μm, while that of the membranes in Fig. 10(b) is 1.0 μm. The buckled membranes in Fig. 10(a) indicate that the film deposited at 5.00 Torr had a compressive residual stress, while the taut diaphragms in Fig. 10(b) indicate that the film deposited at 2.50 Torr had a tensile residual stress. These findings are consistent with the wafer curvature measurements reported above. The fact that free standing membranes could be made from both the compressive and tensile films suggests that both films are stoichiometric SiC that are seemingly absent of Si crystallites or amorphous inclusions, thus ruling out differences in chemical composition as a reason for the observed differences in residual stress.

4. Conclusions

Poly-SiC thin films have been deposited on (1 0 0) Si substrates in a high-throughput, large volume, horizontal hot-wall LPCVD furnace using SiH_2Cl_2, C_2H_2 as Si- and C-containing precursors at temperatures from 800 to 900 °C and pressures from 0.42 to 5.00 Torr. The growth rate of the SiC films increases with increasing deposition temperature and pressure. The films exhibited a (1 1 1) 3C-SiC texture over the entire temperature and pressure ranges. The faceted grains were spherulitic aggregates. The surface roughness of the films increases with increasing film thickness due to an increase in grain size. Most significantly, the residual stress exhibits a discernable relationship to both deposition temperature and pressure in a way that enables in situ control of residual stresses during the formation of poly-SiC films. The control of residual stress by deposition pressure at constant temperature is also important in terms of the process thermal budget.

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Biographies

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Christian A. Zorman, Ph.D. received his B.S. cum laude in physics and B.A. cum laude in economics from the Ohio State University in 1988, and M.S. and Ph.D. in physics from Case Western Reserve University in 1991 and 1994, respectively. His doctoral research involved an investigation of the secondary electron emission properties of CVD diamond films for vacuum electronics. Dr. Zorman joined the MEMS program at CWRU in 1994 as a Research Associate and immediately began working in the SiC MEMS area. He was promoted to Senior Research Associate in 1997 and Researcher in 2000. In addition to his research positions within the University, Dr. Zorman has held appointments as Adjunct Assistant Professor in the Department of Electrical Engineering and Computer Science and Interim Administrative Director of the Microfabrication Laboratory. He currently is an Associate Professor in EECS at CWRU. He has been instrumental in the construction of AP- and LPCVD reactors for SiC thin films, and has led the development of recipes for the growth of single and polycrystalline 3C-SiC films for micromachined sensors and actuators. In addition to the development of novel bulk and surface micromachining techniques for SiC, Dr. Zorman was a key contributor in the development of novel polishing, wafer bonding, and low defect density growth processes for SiC. His current research interests include the development of SiC for NEMS. He has published over 120 technical papers, five book chapters, and has taught several short courses on SiC for MEMS. Prof. Zorman is a past chairman of the MEMS Technical Group in the American Vacuum Society and is currenty serving as co-chairman.

Mehran Mehregany received his B.S. in Electrical Engineering from the University of Missouri in 1984, and his M.S. and Ph.D. in Electrical Engineering from Massachusetts Institute of Technology in 1986 and 1990, respectively. From 1986 to 1990, he was a consultant to the Robotic Systems Research Department at AT&T Bell Laboratories, where he was a key contributor to ground-breaking research in microelectromechanical systems (MEMS). In 1990, he joined the Department of Electrical Engineering and Applied Physics at Case Western Reserve University as an Assistant Professor. He was awarded the Nord Assistant Professorship in 1991, was promoted to Associate Professor with tenure in July 1994, and was promoted to Full Professor in July 1997. He held the George S. Dively Professor of Engineering endowed chair from January 1998 until July 2000, when he was appointed the Goodrich Professor of Engineering Innovation. He served as the Director of the MEMS Research Center at Case from July 1995 until December 2002. Since January 2003, Professor Mehregany has been serving as Chairman of the Electrical Engineering and Computer Science Department. Professor Mehregany is well known for his research in the area of MEMS, and his work has been widely covered by domestic and foreign media. He has over 250 publications describing his work, holds 15 U.S. patents, and is the recepient of a number of awards/honors. He served as the Editor-in-Chief of the Journal of Micromechanics and Microengineering from January 1996 to December 1997, and is Assistant-to-the-President of the Transducers Research Foundation. His research interests are in micro- and nano-electromechanical systems (MEMS and NEMS), including sensors, actuators, micromachining and microfabrication technologies. His additional specialized interest centers on developing silicon carbide as an enabling material for MEMS and NEMS, in particular for applications in harsh environments.Professor Mehregany is the founder/cofounder of several technology companies, including Advanced Micromachines Incorporated (now part of The Goodrich Corporation), FLX Micro, Inc. (www.flxmicro.com) and NineSigma, Inc. (www.ninesigma.com).