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Deposition of Polycrystalline 3C-SiC Films on 100 mm Diameter Si(100) Wafers in a Large-Volume LPCVD Furnace

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The chemical and microstructural characteristics of silicon carbide films deposited on 100 mm diam, silicon (100) wafers in a large-volume, low-pressure chemical vapor deposition (LPCVD) furnace using dichlorosilane (SiH_2Cl_2) and acetylene (C_2H_2) were investigated. The deposition temperature was held constant at 900°C and the pressure ranged between 460 and 510 mTorr. X-ray photoelectron spectroscopy data indicated that stoichiometric SiC was deposited using SiH_2Cl_2-to-C_2H_2 molar ratios of 4:1, 6:1, and 8:1. X-ray diffraction showed that the stoichiometric films were highly textured, 3C-SiC(111) at all locations across each wafer. These findings indicate that the SiH_2Cl_2/C_2H_2 precursor system has great potential for use in large-scale LPCVD furnaces and produces SiC films with a microstructure that has advantageous properties for use in high-frequency resonator micromechanical devices.

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SiC is well known for its excellent properties, making it an outstanding addition to the microelectromechanical systems (MEMS) technology toolbox. Increasing interest in SiC for coating and structural device applications, combined with recent demonstrations of SiC surface micromachining processes,[1,2] have provided substantial impetus for developing deposition and process technologies similar to those for polysilicon; films that are typically deposited on large-area silicon wafers in large horizontal low-pressure chemical vapor deposition (LPCVD) furnaces. This paper reports the development of recipes for the deposition of polycrystalline SiC (poly-SiC) films in a high-throughput, LPCVD furnace on large-area substrates at 900°C. This technology is being developed to support a multilayer SiC surface micromachining process named MUSiC™ (for Multi-User SiC).[3] The MUSiC process is a four-layer poly-SiC process that generally embodies the design rules and capabilities of the now well established Cronos Integrated Microsystems polysilicon MUMPs™ process.

While a few LPCVD processes have been reported for deposition of poly-SiC in recent years,[4-8] it appears that these processes were performed in research-sized chambers too small in volume to meet the batch processing needs required of commercially viable technologies. Nevertheless, the successes reported in these early studies provide sufficient motivation to develop similar large-scale LPCVD systems for the deposition of SiC.

Silicon carbide films were deposited on 100 mm diam silicon (100) wafers in a conventional, hot-wall horizontal furnace using SiH_2Cl_2 and C_2H_2 as silicon and carbon-containing precursors. The furnace tube was large by SiC deposition standards, measuring 2007 mm in length and 225 mm in diameter. Although the furnace is capable of holding up to 100 wafers, each load consisted of 10 wafers held in a single, 50 slot quartz boat, with the first three and last three slots in the boat filled with wafers, and the other four wafers evenly distributed throughout the remaining slots. Prior to loading the furnaces, all wafers were cleaned using a standard RCA cleaning procedure. For characterization purposes, the depositions were performed for 1 h at a fixed temperature of 900°C, although in several cases, longer times were used to deposit thick films. The flow rates of SiH_2Cl_2 and C_2H_2 were held constant during each deposition run. To study the effect of precursor concentration on growth rate, microstructure, and surface morphology, runs were made using SiH_2Cl_2 flow rates ranging from 18 to 72 standard cubic centimeters per minute (sccm), with the flow rate of C_2H_2 (5% in H_2) remaining fixed at 180 sccm for all depositions. This resulted in a series of deposition runs with the SiH_2Cl_2-to-C_2H_2 molar ratios ranging from 2:1 to 8:1. The furnace pressure was held constant during each deposition, but ranged between 460 and 510 mTorr over the series of runs, with higher pressures associated with higher SiH_2Cl_2 flow rates. Following each deposition, optical reflectometry and scanning electron microscopy (SEM) were used to study the thickness and surface morphology, X-ray photoelectron spectroscopy (XPS) and wet chemical etching were used to characterize the chemical composition, and X-ray diffraction (XRD) was used to determine the microstructure of the as-deposited films.

Figure 1 shows the relationship between deposition rate and molar ratio of the precursor gases. Except for ratios above 6:1, an increase in the molar ratio resulted in an increase in deposition rate, ranging from a minimum of 90 nm/h for a molar ratio of 2:1 to a maximum of 240 nm/h for molar ratios equal to or above 6:1. This result is in contrast to the findings reported in Ref. 6 and 8, which reported that the deposition rate of SiC using SiH_2Cl_2 and C_2H_2 at reduced pressures exhibits a first order dependence on the SiH_2Cl_2 concentration for molar ratios over the same range. Our data suggests that the exact mechanism governing the deposition rate is more complex than a simple first order dependence on the relative precursor concentration, especially given the likelihood of multiple, complex gas phase and surface reactions possible with this dual precursor system.[9] It is conceivable that reactor geometry may also play an important role. Regardless, the maximum deposition rate is adequate to produce films of sufficient thickness for microelectromechanical systems (MEMS) applications, as evidenced by the SEM cross section in Fig. 2a, which is from a 1.5 μm thick film deposited using a molar ratio of 6:1 for 7.5 h.

For all molar ratios, the as-deposited films exhibited a high degree of thickness uniformity, both across each wafer and across the quartz boat. Visually, each film had a uniform monochromatic appearance, although plan-view SEM micrographs indicate a somewhat grainy surface texture (Fig. 2b) which increases with increasing film thickness. Optical reflectometry measurements taken from at least five points across each wafer indicate an areal thickness variation of less than 0.5% for films deposited using a molar ratio of 2:1, increasing to only 1.7% for a molar ratio of 8:1. As for the thickness variation across the quartz boat, the values ranged from a low of 0.2% for a molar ratio of 2:1 to a high of only 1.5% for a molar ratio of 8:1. These parameters, in concert with the deposition rate data indicate that this precursor gas system is well suited for scale-up to production sized reactors.

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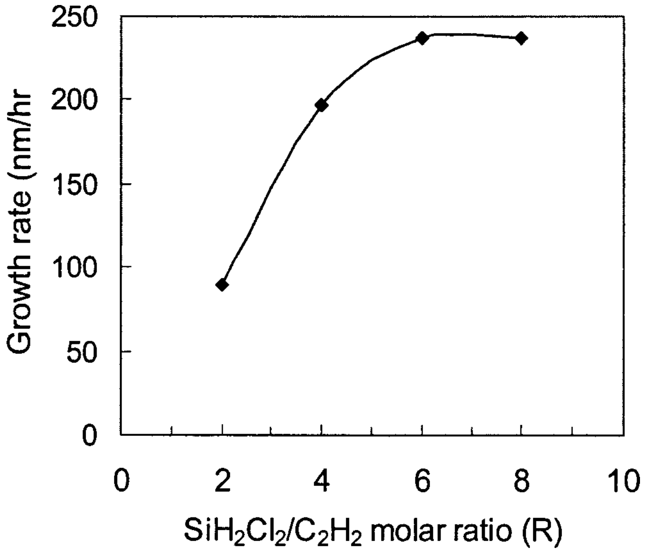
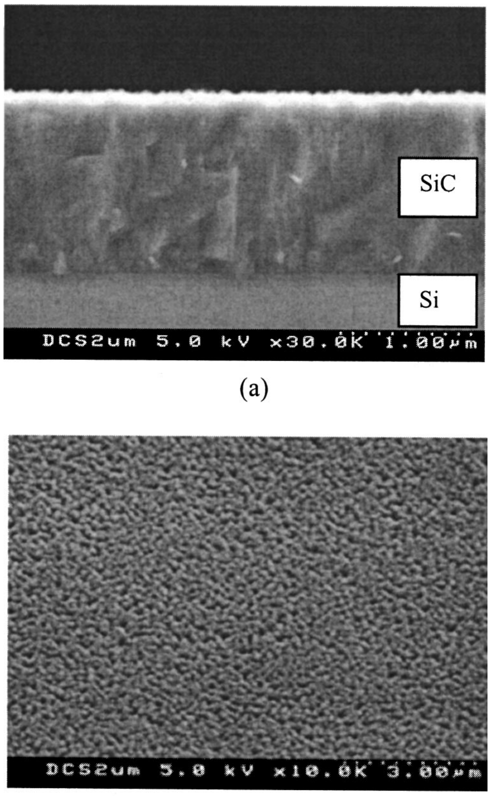


Figure 1. Deposition rate *vs.* SiH_2Cl_2/C_2H_2 molar ratio for SiC films deposited at 900°C.

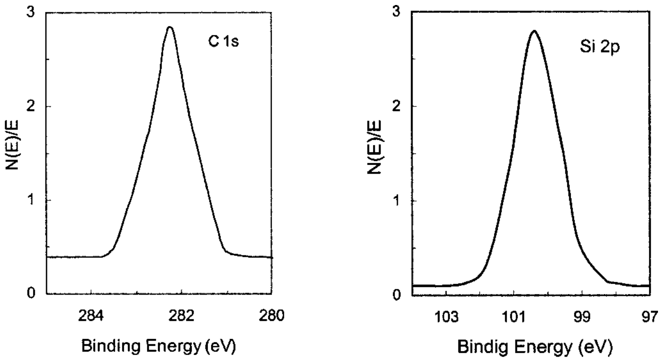
Initial chemical testing was performed by immersing at least one wafer from each run in a 4 L, 3.57 M KOH/H_2O etch solution at 55°C for 24 h; conditions that are sufficient to completely dissolve unprotected 500 μm thick Si wafers but should not etch SiC. For all samples except the film deposited using a molar ratio of 2:1, the wafers and films were unetched, indicating that the films were continuous, pinhole-free, and likely to be SiC. In contrast, the substrate supporting the film grown using a molar ratio of 2:1 was completely dissolved. This result indicated that the film was either not stoichiometric or plagued with pinholes. XPS sputter depth profiling indicated that for a molar ratio of 2:1, the as-deposited films were carbon-rich, with a carbon concentration of approximately 56 atom %. In contrast, stoichiometric SiC films were obtained for molar ratios of 4:1, 6:1, and 8:1. These findings are further substantiated by examining the position and shape of the C 1s and the Si 2p peaks of the unetched films, which are detected at 282.7 and 100.2 eV, respectively, and exhibit the shapes expected for stoichiometric SiC (Fig. 3). Under none of the conditions were Si-rich films deposited. The ability to deposit SiC over such a wide range of molar ratios differs greatly from the findings reported in Ref. 6, where stoichiometric SiC could only be deposited at a SiH_2Cl_2/C_2H_2 molar ratio of 8:1 for a deposition pressure of 9 Torr. Our findings suggest that lower total pressures improve precursor mixing and gas phase diffusion such that the reactions necessary to form stoichiometric SiC are relatively insensitive to the concentration of Si except when SiH_2Cl_2 concentrations are low.



(a)

(b)

Figure 2. (a) Cross-sectional and (b) plan-view SEMs of a stoichiometric SiC film deposited using a SiH_2Cl_2/C_2H_2 molar ratio of 6:1. The film is ~1.5 μm thick and was deposited at 900°C for 7.5 h.



(a)

(b)

Figure 3. XPS C 1s (a) and Si 2p (b) peaks for a stoichiometric SiC film deposited using a SiH_2Cl_2/C_2H_2 molar ratio of 6:1.

The crystal structure of the films was characterized by means of XRD. The samples were randomly selected from various areas of each 4 in. wafer to observe any spatial variation in crystallinity. Figure 4 shows a typical XRD spectrum from a SiC film deposited

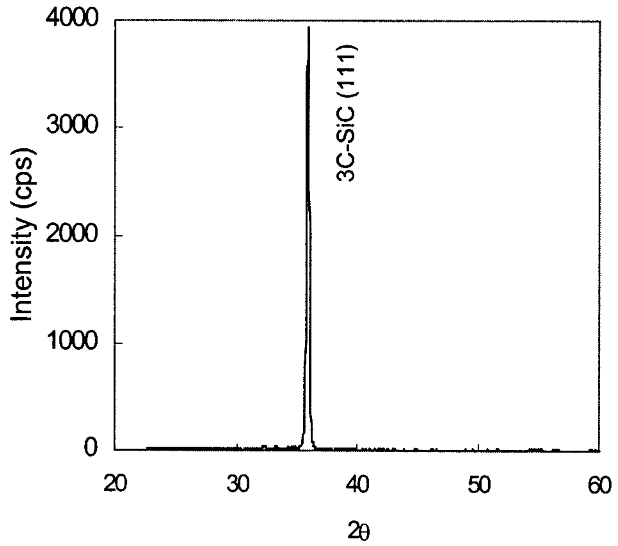


Figure 4. XRD spectrum from a 1.5 μm thick SiC film deposited using a SiH_2Cl_2/C_2H_2 molar ratio of 6:1.

at a molar ratio of 6:1. The peak position at $2\theta = 35.8°$ indicates that the film is 3C-SiC with a strong (111) texture. Peaks corresponding to the (200) or (220) 3C-SiC planes were not detected at any of the locations in any of the stoichiometric samples. For the carbon-rich film, no diffraction peaks were found. By comparison, it has been reported that using the same precursors and furnace temperature, SiC films deposited at a total pressure of 9 Torr exhibited a (110) texture,[6] suggesting that pressure alone can be used as a means of controlling the microstructure of the as-deposited films.

The results of this initial study clearly indicate that the SiH_2Cl_2/C_2H_2 precursor gas system is well suited for use in production-style furnaces. But perhaps equally important, the ability to use this furnace to deposit highly-oriented 3C-SiC(111) films may result in the improved performance of micromechanical SiC devices that capitalize on the high Young's modulus (E) of SiC, such as resonant beam structures. The resonant frequency of these devices depends directly on the acoustic velocity of the structural material, which is defined as $(E/\rho)^{1/2}$, making SiC attractive for high frequency applications. For SiC in general and 3C-SiC in specific, the Young's modulus exhibits an anisotropic behavior with respect to crystal orientation, whereas the density is constant. Theoretical calculations indicate that for 3C-SiC, the (111) orientation has the highest Young's modulus,[10] suggesting that the films deposited using the conditions detailed in this report have the preferred microstructure for high frequency resonator applications.

Acknowledgments

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