Stress control of polycrystalline 3C-SiC films in a large-scale LPCVD reactor using 1,3-disilabutane and dichlorosilane as precursors

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Abstract

Control of residual stress and strain gradient of polycrystalline SiC films deposited via low-pressure chemical vapor deposition on 100 mm Si wafers is achieved by varying dichlorosilane (DCS) and 1,3-disilabutane (DSB) fractions in the inlet gas mixture. For films deposited at 800°C and 45 sccm DSB, stress decreases from 1.2 GPa tensile with no added DCS to 240 MPa tensile with 40 sccm DCS added to the inlet gas stream. The lowest magnitude strain gradient achieved is $3.1 \times 10^{-5}$ µm$^{-1}$ with 20 sccm DCS added. Electron probe microanalysis indicates that the films change from being slightly carbon-rich in the absence of DCS to successively more silicon-rich with the addition of DCS.

1. Introduction

Silicon carbide in the 3C cubic phase is a promising material for use as a structural layer in micro- and nanoelectromechanical systems (MEMS and NEMS). It is well suited for operation in harsh environments due to its high melting point, wide bandgap, wear resistance, and chemical resistance [1–4]. In addition, SiC is used to fabricate high frequency MEMS and NEMS resonators since such devices demand a structural material with a high modulus to density ratio [5, 6]. Recent efforts towards SiC-based MEMS and NEMS have included scaling up low-pressure chemical vapor deposition (LPCVD) methods for deposition of polycrystalline SiC on 100 and 150 mm Si wafer substrates to allow processing compatibility with standard MEMS manufacturing [7, 8].

MEMS applications require the deposition of structural films with residual stresses that are low and preferably tensile. High tensile residual stress leads to film cracking [9] and compressive residual stress leads to beam buckling [10]. Residual stress in SiC films is known to be affected by deposition temperature [11] and, in certain cases, by deposition pressure [12]. However, recent large-scale deposition of LPCVD SiC films from 1,3-disilabutane (DSB) has yielded films with high residual stresses in excess of 1.3 GPa tensile across a range of deposition temperatures [8]. Alteration of deposition pressure often requires expensive pumping system modifications; hence, alternative stress reduction techniques are desirable. Strain gradient is another film property that must be controlled for successful MEMS device fabrication, since large strain gradients lead to microstructure curling. Tailored doping with ammonia is one method which can be used to control the strain gradient of SiC films [13].

It has long been known that residual stress in silicon nitride films can be tuned by changing inlet gas flow rates and thus adjusting the silicon-to-nitrogen ratio in the resulting film [14]. In this paper, an analogous method to reduce and control the residual stress and strain gradient in polycrystalline SiC films is presented in which dichlorosilane is introduced as a second feed gas in the reactor. Stress and strain gradient values are reported as functions of the fraction of DCS to total gas flow rate in the inlet gas mixture. The results show that, by using this approach, one can effectively control stress and strain gradient of the films.
2. Methods

Polycrystalline 3C-SiC films are deposited in a 100 and 150 mm wafer compatible hot wall LPCVD reactor from the precursors 1,3-disilabutane (DSB) and dichlorosilane (DCS). Deposition is performed on 100 mm Si(1 0 0) wafers in a single 15-wafer closed boat. The reactor supports up to three boats, allowing deposition on up to 45 wafers simultaneously. Details of the reactor and closed boat are described elsewhere [8]. All films reported here are deposited at 800 °C for 1 h. The DSB flow rate is held constant at 45 sccm for each film and the DCS flow rate is varied between depositions. One run is performed for each set of deposition conditions.

Film stress is measured on four equally spaced wafers in each run using a Tencor FLX-2320 (Flexus) system to measure wafer curvature, a micrometer to measure wafer thickness, and optical reflectometry to measure SiC film thickness [8]. The average strain is measured on one wafer of each run using microfabricated strain gauges [11]. The gradient in strain, which is measured on one wafer of each run, is measured in two ways. If the magnitude of the strain gradient is of the order of $10^{-4} \mu m^{-1}$ or less, tip deflections of microfabricated cantilever beams are measured using a Wyko NT3300 white light interferometer. Strain gradient can be calculated using

$$\Gamma = 2\Delta z/L^2. \quad (1)$$

If the magnitude of the strain gradient is of the order of $10^{-3} \mu m^{-1}$, cantilever beams are deflected too far to allow the use of interferometry. Instead, beams are examined using a Leo 1550 scanning electron microscope. The radius of curvature $R$ is measured and strain gradient is calculated using (2):

$$\Gamma = 1/R. \quad (2)$$

Film stoichiometry is measured on one wafer of each run with electron probe microanalysis (EPMA) using a Cameca SX-51 microprobe with a 200 nm sampling depth (3.5 kV acceleration voltage and 200 nA incident beam current). A single crystal 6H-SiC (Cree, Inc.) standard is used as calibration.

3. Results and discussion

The SiC growth rates, averaged based on measurements made on wafers occupying the same four equally spaced positions in each run, vary between 0.23 and 0.32 μm per hour depending on the amount of dichlorosilane added to the inlet flow (figure 1). The error bars reflect the standard deviation of the growth rate across all four wafers measured for each condition. The within-wafer non-uniformity of the growth rate ranges from 2% to 5%.

Figures 2 and 3 depict film stress and Si:C ratio, respectively, as a function of the fraction of DCS flow rate in the inlet gas mixture. The error bars in figure 2 reflect the errors in measuring film thickness, radius of curvature and substrate thickness, all of which are used to calculate residual stress. Increasing DCS in the inlet gas mixture results in increased Si:C ratio and decreased film stress. The change in stoichiometry is in agreement with the expectations since a DSB molecule contains both Si and C atoms, while DCS contains one Si atom. The reduction in residual tensile stress may partly be due to silicon’s larger atomic radius than carbon, which has been used to explain low-stress nitride [16]. Excess Si in a film increases the average bond length, thus reducing tensile stress; however, the largest change in the Si to C ratio corresponds to the smallest change in residual stress (figures 2 and 3) which highlights that this may not be the sole cause for stress reduction. The addition of a chlorinated precursor introduces new chemistry in the reactor which could also be responsible for a reduction in stress.

Figure 4 depicts a microfabricated strain gauge fabricated from a film deposited with 20 sccm DCS. For films with less than 20 sccm DCS, these structures were broken due to the high strain gradient of the films. The films deposited with 20 and 40 sccm DCS both had residual strain of $5 \times 10^{-4}$.

Figure 5 contains scanning electron micrographs of cantilever beam arrays under different deposition conditions. Figure 6 depicts strain gradient as a function of the fraction...
To test the chemical robustness of these films, the coated substrates are immersed in hot 33% KOH (80 °C), an aggressive silicon etchant. No microcracks are observed for any condition indicating the films are of high quality. In addition, the etch rate in KOH is found to be less than 1 nm h⁻¹ using optical reflectometry to measure the film thickness before and after etching. Therefore, these SiC films are superior to silicon dioxide and silicon nitride as masking layers for wet anisotropic KOH bulk micromachining [18].

4. Conclusion

By altering the DCS flow rate in a DSB-based LPCVD SiC reactor, Si:C ratio, residual stress and strain gradient were varied. With increasing DCS flow rate, residual stress decreases while Si:C ratio increases. The lowest residual stress achieved is 240 MPa tensile while the lowest strain gradient realized is $3.1 \times 10^{-5}$ µm⁻¹. The reduction in stress means this polycrystalline SiC deposition technology is no longer limited to only thin MEMS coating layers, but can now also be used as a MEMS structural layer.

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