Novel Low-Temperature CVD Process for Silicon Carbide MEMS

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SUMMARY

A low-temperature, single precursor CVD process for the realization of SiC-based MEMS and SiC-coated MEMS is described using 1,3-disilabutane. With this deposition method, the fabrication of an all-SiC cantilever beam array is demonstrated using standard microfabrication processes. Also, SiC coating of released Si micromechanical structures is realized using this process. The SiC-coated microstructures are shown to have superior chemical stability when compared to their Si analogs, as well as exhibit highly favorable mechanical properties.

Keywords: silicon carbide; CVD; resonators; MEMS

INTRODUCTION

In recent years, silicon carbide (SiC) has emerged as an important material for microelectromechanical systems (MEMS) due to its favorable physicochemical, mechanical, tribological, and electrical properties [1,2]. Conventionally, atmospheric pressure chemical vapor deposition (CVD) using SiH_4 and propane is employed, requiring temperatures in excess of 1000°C. In addition, elaborate methods are used to pattern the SiC films. As a consequence, SiC technology remains technically demanding and non-standard in Si-based integrated circuit fabrication laboratories. The development of a simple method for integrating SiC structural layers and conformal, thin SiC coatings into Si-based micromechanical systems is of immediate interest.

Here, we report on a novel method for obtaining high quality SiC films by CVD from a single precursor molecule. The precursor molecule used in these studies is 1,3-disilabutane (DSB; molecular structure CH_3-SiH_2-CH_2-SiH_3) [3]. This paper presents the first application of this precursor molecule for the realization of SiC-based MEMS and SiC-coated Si MEMS [4]. The process has several key advantages over the conventional CVD SiC deposition method: 1) High quality polycrystalline films at temperatures as low as 650 °C are demonstrated. This temperature is low enough to enable SiC deposition on released Si microstructures. 2) The precursor is a liquid at room temperature with vapor pressure of 27 Torr, and is rather benign. These characteristics make the handling aspects much simplified when compared to conventional CVD utilizing such gases as SiH_4. 3) The use of the single precursor eliminates the need for an elaborate gas handling system. 4) No pre-carbonization step is needed for deposition on Si and SiO_2. 5) The SiC films can be patterned using SiO_2 masking and a simple lift-off using HF.

EXPERIMENTAL

The CVD process utilized here employs DSB pressures between 10^{-4} and 10^{-5} Torr and a growth temperature of approximately 800ºC. The SiC films are grown on patterned Si(100) substrates, single crystalline Si atomic force microscope (AFM) cantilevers, and released polycrystalline Si (polySi) microstructures. Complementary experiments are performed on HF-etched Si(100) and polySi wafers in order to produce larger samples on which the composition, structure, and thickness of the films are determined.

A number of analysis and characterization techniques are employed to investigate the deposited SiC films. These include Auger electron spectroscopy (AES) to confirm chemical composition, X-ray diffraction (XRD) to determine structural crystallinity, and AFM to investigate SiC film topology. Scanning electron microscopy (SEM) is used to investigate the SiC film morphology, the quality of the SiC/Si interface, and to estimate film thickness by cross-sectional imaging.

The Si microstructures used in this study [5] are fabricated at the Berkeley microfabrication laboratory, using standard surface micromachining techniques. These microstructures are comprised of a 2 μm thick polySi structural layer that is heavily doped with phosphorous. After fabrication, wafers are diced and annealed at 950°C to relieve the residual stress and reduce the stress gradient in the structural film. The microstructures are then released by a 10 minute etch in 1:1 (v/v) mixture of 49 wt% HF and 37.6 wt% HCl. Next, the micromachines are treated with a 1-octadecene anti-stiction monolayer, described in detail elsewhere [6]. The

octadecene treatment allows high yield release of microstructures that are free of surface oxide. Moreover, the alkene film protects the polySi microstructure from oxidation while being handled in ambient, yet desorbs from the microstructures upon heating in vacuum to 350°C [6]. All micromachine actuation was performed in laboratory air at 20°C and 33% relative humidity.

RESULTS AND DISCUSSION

SiC film characterization

In Fig. 1, a SEM image is displayed showing the typical cross-section of a deposited SiC film. Note that the SiC film shows excellent conformality with no void formation at the SiC/Si interface. An AFM topography scan of a 210 nm thick SiC coating (not shown) yields regular cubic features having an average diameter of 100 nm and a rms roughness of less than 10 nm. AES shows the deposited films to be carbidic and stoichiometric, while XRD on thick (>1μm) films shows bulk polycrystalline cubic structure.

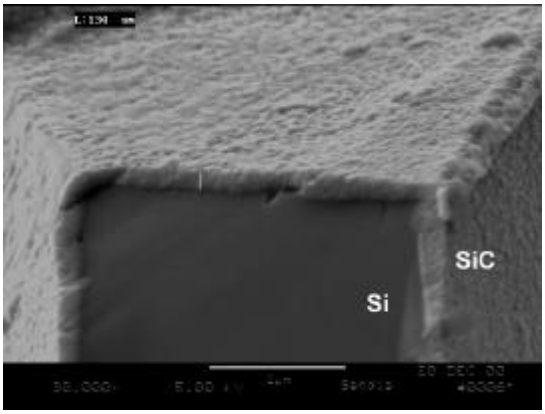

Fig. 1: *SEM image of the cross-section of a 130 nm thick SiC film deposited on a Si AFM cantilever.*

The elasticity of the deposited SiC coatings is characterized by the following method, the details of which are reported elsewhere [7]. SiC films of varying thickness are deposited on Si AFM cantilevers. The resonance frequency of the cantilever is observed to increase in proportion to film thickness, indicative of a uniformly deposited SiC film. The conformality of the SiC coatings is confirmed by cross-sectional SEM. The SiC-coated cantilever is then modeled as a composite beam of rectangular cross-section assuming a uniform coating on all sides. An elastic modulus of about 380 GPa is estimated, in good agreement with previous estimates for the elastic modulus of cubic SiC thin films [8] and cantilever beams [9].

SiC-based microstructures

The realization of all-SiC micromechanical structures is of particular interest for applications in harsh environments. Here, we employ our low-temperature CVD method to fabricate all-SiC cantilever beam arrays on Si(100) substrates. The key steps are illustrated in Fig. 2.

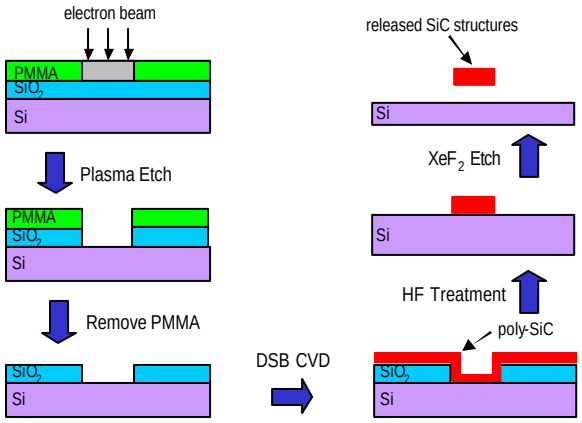

Fig. 2: *Schematic of the key fabrication steps for an all-SiC cantilever beam array.*

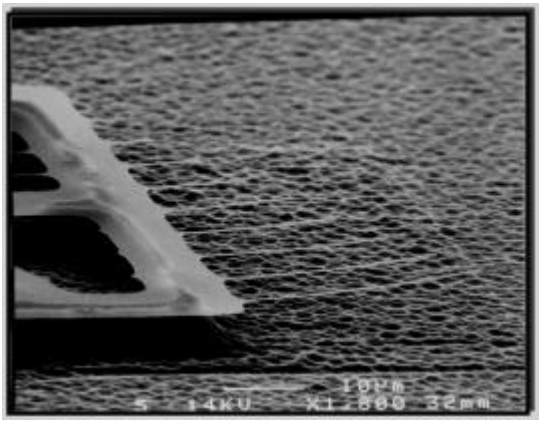

Fig. 3: *SEM image of an all-SiC cantilever beam array.*

A 100 nm SiO_2 masking layer is deposited on a Si(100) substrate and patterned using standard lithographic techniques. The SiC film is then deposited on top of the SiO_2 mask, covering the entire substrate. Next, the SiC-coated sample is immersed in conc. HF, which lifts the SiO_2 mask. SiO_2 is previously shown to be an effective mask for epitaxial SiC deposition [10]. A XeF_2 etch then releases the patterned SiC microstructure.

A SEM image showing the fully released SiC cantilever beam array is shown in Fig. 3. The SiC beams are approximately 200 nm thick and up to 30 μm in length. This approach is technically simple and is designed to leverage off established microfabrication processes.

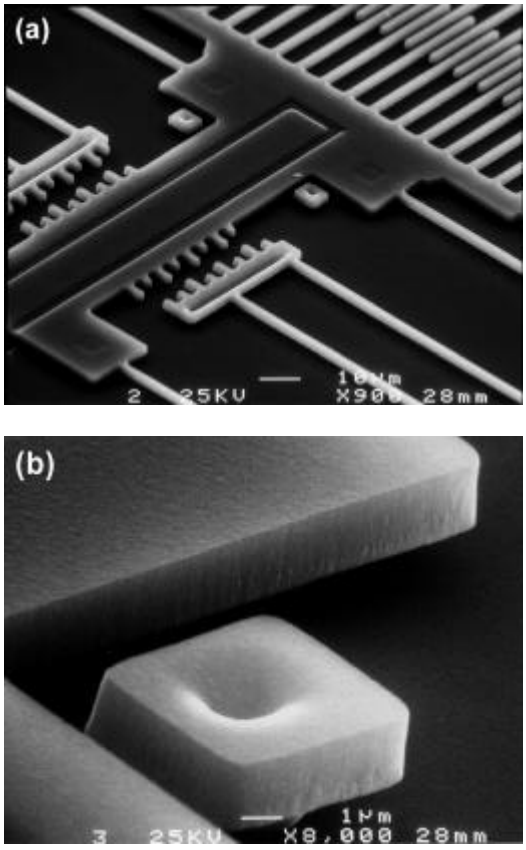

Fig. 4: *SEM images of a released polySi lateral resonator coated with a thin SiC film. (a) Top view of resonator. (b) Close-up of resonator sidewall.*

SiC-coated microstructures

The DSB CVD process is also used successfully to coat *released* polySi microstructures. In Fig. 4, SEM images of a lateral resonator are shown following the deposition of a 35 nm thick SiC coating. The film thickness is estimated by carrying out the same deposition on a patterned Si(100) substrate and measuring the film thickness by profilometry. For thin SiC coatings, no deformation of the released structures is detected following deposition, as evidenced in Fig. 4(a). Furthermore, Fig. 4(b) shows the SiC coating to be smooth, covering the microstructure's sidewalls.

Resonance frequency data for the lateral resonators are collected over approximately six devices with varying folded beam suspension length. Table 1 summarizes the resonance frequency data. Data analysis is done using the Rayleigh method [11], as shown in Eqn. 1.

$$IE = \frac{L^3}{24}\left(4\pi^2 f_r^2 m_{eff}\right) \qquad (1)$$

This cursory analysis enables an estimate of the elastic modulus, E, of the resonator's structural layer as a function of its mass, m_{eff}, its folded beam suspension length, L, its resonance frequency, f_r, and its moment of inertia, I.

folded beam length (μm)	uncoated sample (kHz)	SiC-coated sample (kHz)
150	24.9	27.9
200	15.6	18.1
250	10.8	12.7
300	8.2	9.6
350	6.4	7.6
400	5.2	6.2

Table 1: *The resonance frequency measurement reproducibility is ±0.2 kHz. The uncoated sample experienced only the thermal cycle of a deposition treatment.*

Using the data for the uncoated sample, the Rayleigh method analysis gives a modulus of 120 GPa for the polySi structural layer. In order to deconvolute the modulus information from SiC coated structures, the simple model must be extended to account for a composite beam structure. Assuming a uniform thin film thickness of δ, and following similar analysis as in Ref. 7, the modified relation between the elastic moduli of the films and the resonance frequency is given in Eqn. 2.

$$\frac{E_1 wt^3}{12} + E_2\left(\frac{wt^2}{2} + \frac{t^3}{6}\right)\delta = \frac{L^3}{24}\left(4\pi^2 f_r^2 m_{eff}\right) \qquad (2)$$

Here, E_1 and E_2 are the elastic modulus of the polySi and deposited SiC film, respectively. Using the numerical value for E_1 calculated for the uncoated sample, and an assumed film thickness of 35 nm for the SiC overlayer, the average value for the modulus of the deposited film (E_2) is 370 GPa. The biggest uncertainty in this analysis is the deposited film thickness, which is measured indirectly. A direct measurement of film thickness is destructive and will be performed

subsequent to sample characterization and analysis, in turn yielding a more accurate estimate for E_2.

Chemical and mechanical stability

Using a tungsten probe tip, the thin SiC coatings prove to be scratch resistant, while an uncoated polySi surface is readily scratched. The uniformity and chemical stability of the SiC coatings are tested by immersion in 33wt% KOH solution at 65ºC for 60 sec. Under these conditions, the Si etch rate is approximately 1.2 μm/min. while the SiC etch rate is negligible. The results of this test are shown in Fig. 5. As seen in Fig. 5(a), a SiC-coated micromachine remains intact following immersion in hot KOH, indicating that the SiC overlayer is conformal, dense, and pin-hole free. Inspection of the underside of the SiC-coated structural layer and ground plane verify the conformality and chemical stability of the SiC films, as no degradation of these surfaces is observed subsequent to immersion in hot KOH. Shown in Fig. 5(b), an uncoated micromachine is completely etched from the substrate under these conditions.

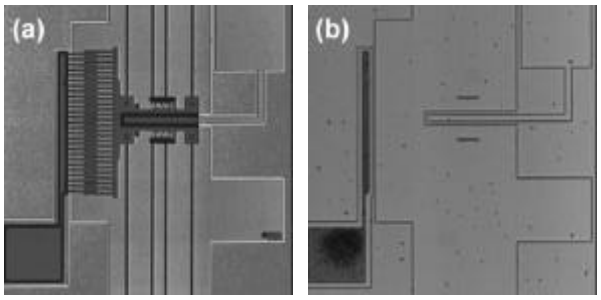

Fig. 5: *Optical images of (a) SiC-coated and (b) uncoated polySi micromachines following immersion in hot KOH.*

CONCLUSIONS

In conclusion, the low-temperature, single precursor CVD process described here is shown to be readily adaptable to current MEMS technologies. Both SiC-based and SiC-coated micromechanical structures are realized, and an initial evaluation of their mechanical and chemical properties is reported. Furthermore, this CVD method proves promising for applications in harsh environments where current Si technologies cannot be employed.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge fruitful discussions with Prof. Roger Howe. This work is supported by DARPA, Sandia National Laboratories, and the Arnold and Mabel Beckman Foundation. M.C.F. acknowledges the support of the Erwin Schrödinger Fellowship.

REFERENCES

[1] M. Mehregany and C. A. Zorman, "SiC MEMS: opportunities and challenges for applications in harsh environments", Thin Solid Films, 355-356, pp. 518-524, 1999.
[2] P. M. Sarro, "Silicon carbide as a new MEMS technology", Sens. Actuators, 82, pp. 210-218, 2000.
[3] K-W. Lee, K.-S. Yu, and Y. Kim, "Heteroepitaxial growth of 3C-SiC on Si(001) without carbonization", J. Crys. Growth, 179, pp. 153-160, 1997.
[4] Patent disclosure filed, University of California at Berkeley case no. B00-099.
[5] U. Srinivasan, M. R. Houston, R. T. Howe, and R. Maboudian, "Alkyltrichlorosilane-Based Self-Assembled Monolayer Films for Stiction Reduction in Silicon Micromachines", J. MEMS, 7, pp. 252-260, 1998.
[6] W. R. Ashurst et al., "Alkene based monolayer films as anti-stiction coatings for polysilicon MEMS", Sens. Actuators A, 2001, in press.
[7] C. R. Stoldt, M. C. Fritz, C. Carraro, and R. Maboudian, "Micromechanical Properties of Silicon Carbide Thin Films Deposited using Single Source CVD", submitted to Appl. Phys. Lett., February 2001.
[8] M. Mehregany, L. Tong, L. G. Matus, and D. J. Larkin, "Internal Stress and Elastic Modulus Measurements on Micromachined 3C-SiC Thin Films", IEEE Trans. Electr. Dev., 44, pp. 74-79, 1997.
[9] C. Serre, A. Pérez-Rodríguez, J. R. Morante, P. Gorostiza, and J. Esteve, "Determination of micromechanical properties of thin films by beam bending measurements with an atomic force microscope", Sens. Actuators, 74, pp. 134-138, 1999.
[10] J. H. Edgar et al., "Selective epitaxial growth of silicon carbide on SiO_2 masked Si(100): The effects of temperature", J. Appl. Phys., 84, pp. 201-204, 1998.
[11] M. Lim, "Polysilicon Microstructures to Characterize Static Friction", Masters Thesis, Dept. of Mechanical Engineering, University of California at Berkeley, 1990.