

Micromechanical properties of silicon-carbide thin films deposited using single-source chemical-vapor deposition

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(Received 27 February 2001; accepted for publication 11 May 2001)

1,3-Disilabutane is used as a single-source precursor to deposit conformal silicon-carbide films on silicon atomic-force-microscopy cantilevers. By measuring the resonance frequency of the cantilever as a function of silicon-carbide film thickness and developing an appropriate model, the value of the film's elastic modulus is determined. This value is in good agreement with those reported for silicon-carbide films deposited using conventional dual-source chemical-vapor deposition. Additionally, we comment on the feasibility of integrating this process into the fabrication technology for microelectromechanical systems. © 2001 American Institute of Physics. [DOI: 10.1063/1.1383277]

Silicon carbide (SiC) is an attractive material for demanding mechanical and high-temperature applications, as well as for use in abrasive, erosive, and corrosive media. It is tough, possesses low-friction characteristics, and is second only to diamond in wear resistance. SiC is also a semiconductor of great interest in high-power, high-temperature, and high-radiation applications.^{1,2} Furthermore, SiC is one of the foremost biocompatible materials,³ making it a potential candidate for biological applications.

The superior mechanical, chemical, and electrical capabilities make SiC an exceptionally attractive material in microelectromechanical systems (MEMS).⁴⁻⁶ As it stands, SiC technology remains technically demanding and a nonstandard process in the Si-based integrated circuit (IC) fabrication laboratories. As a consequence, the development of a simple method for integrating thin, conformal SiC coatings as well as structural layers into Si-based micromechanical systems is of immediate interest. Conventional SiC chemical-vapor-deposition (CVD) processes generally utilize multiple precursors such as silane and acetylene, and require elevated substrate temperatures in excess of 1000 °C. High temperatures cause significant deformation of released polycrystalline-Si microstructures,⁷ making postprocess SiC coating challenging if not impossible. This indicates that low-temperature alternatives to conventional SiC CVD methods must be considered. Ideally, a simple CVD method should utilize a single precursor that forms high-quality SiC films at temperatures low enough to enable SiC deposition postprocessing.

Lee and co-workers⁸ have demonstrated that epitaxial cubic-SiC films can be formed on single-crystalline Si at temperatures of 900–1000 °C utilizing 1,3-disilabutane (DSB) as a single precursor. Polycrystalline-SiC films at temperatures as low as 650 °C have been achieved, although temperatures near 800 °C yield a higher growth rate and improved crystallinity.⁹ These temperatures make postprocessing of Si-based MEMS tangible. At room temperature, DSB is a liquid with a vapor pressure of 27 Torr, making the

handling aspects much more simplified when compared to conventional dual-source CVD. Also, the use of a single precursor insures stoichiometry, hence, it eliminates the need for an elaborate gas handling system. These features make this method of deposition ideal for integrating SiC into MEMS technology. However, the micromechanical properties of SiC films produced using DSB have not been investigated. In this letter, we report a method for determining the elastic modulus of polycrystalline-SiC films using Si cantilevers commonly employed in atomic-force microscopy (AFM). Through this study, the feasibility of using a single CVD precursor for coating Si-based micromechanical structures is also demonstrated.

The SiC deposition proceeds as follows. Before deposition, the DSB (Gelest Inc., >95% purity) is further purified by freeze–pump–thaw cycles using liquid nitrogen, after which it is introduced into the vacuum chamber via a variable leak valve and nozzle. The base pressure of the CVD chamber is below 2×10^{-9} Torr. Prior to introduction into the CVD chamber, each Si AFM cantilever is dipped in concentrated HF to remove the native oxide, then rinsed with acetone and dried under N₂. This procedure is confirmed not to change the previously determined cantilever resonance frequency. In the CVD chamber, the cantilever is positioned 3 cm below the exit of the nozzle. The cantilever is radiatively heated from below by a spiral tungsten filament, and its temperature is calibrated and monitored by a thermocouple attached to the heating stage. Deposition begins by pressurizing the CVD chamber with DSB to 5×10^{-5} Torr with the cantilever temperature held at 100 °C. The cantilever temperature is then increased at a rate of 50 °C/min to the growth temperature of 780 °C at constant DSB pressure, where it is then held for the duration of the deposition (from 0 to 180 min). The growth rate under these conditions is 3.5 nm/min. Control experiments performed on uncoated cantilevers show that prolonged annealing at 780 °C in the absence of DSB does not change the resonance frequency.

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A number of analysis and characterization techniques are employed to investigate the SiC-coated cantilevers. Complementary experiments are performed on HF-etched Si(100) wafers in order to produce larger samples on which the composition and structure of the SiC films is determined. Auger electron spectroscopy shows the deposited films to be carbidic and stoichiometric, while x-ray diffraction measurements on thick ($>1 \mu\text{m}$) SiC films reveal a bulk polycrystalline cubic structure. Scanning electron microscopy (SEM) is used to investigate the SiC film morphology and the quality of the Si/SiC interface, and to estimate the SiC film thickness on the cantilevers by cross-sectional imaging. Finally, topography and resonance frequency measurements are performed with a commercially available scanning probe microscope (Nanoscope III Multimode, Digital Instruments, Inc.). Oxygen-plasma-treated Si_3N_4 cantilevers with a nominal force constant of 0.58 N/m are used in contact mode to image the SiC films deposited on Si(100) substrates. Tapping-mode Si cantilevers (Digital Instruments, Inc.) with a nominal force constant of 20–100 N/m are used for resonance frequency measurements.

SEM images of a SiC-coated cantilever beam are displayed in Fig. 1. In Fig. 1(a), the cantilever is shown following the deposition of a 210-nm-thick film. No deformation of the cantilever is detected, indicating that either the residual stress is minimal or the deposited film has excellent conformality. In Figs. 1(b) and 1(c), cross sections of the cantilever tip and sidewall confirm that the coating is homogeneous, dense, and uniform. An AFM topography scan of the SiC coating (not shown) yields regular cubic features having a rms roughness of less than 10 nm.

The elasticity of the deposited SiC coatings is characterized by examining the change in resonance frequency of the coated cantilever as a function of deposition time (effectively, film thickness). AFM has been previously shown to be a very sensitive tool for recording changes in cantilever resonance frequency.^{10,11} In Fig. 2, the ratio in resonance frequency of the coated AFM cantilever is plotted versus SiC film thickness. It is readily observed that the resonance frequency increases in proportion to film thickness, indicative of a uniformly deposited SiC film whose stiffness increases with thickness. Conversely, a decrease in frequency with increasing film thickness is indicative of a mass-loaded cantilever that lacks uniform coverage.¹¹

The SiC-coated cantilever is modeled as a composite beam of rectangular cross section assuming a uniform coating on all sides. The Si AFM cantilevers used in this study have nearly a rectangular cross section with the following nominal dimensions as determined by SEM analysis. The length of the beam, l , is 125 μm , thickness t varies between 3.5 and 5 μm , and width w between 30 and 40 μm (see Fig. 3). The variation in t has the largest effect on the elastic behavior. For an uncoated cantilever of rectangular cross section, the lowest resonance frequency (ν_u) is given by¹²

$$\nu_u = \frac{3.52}{2\pi l^2} \sqrt{\frac{EI}{\rho S}}, \quad (1)$$

where $I = wt^3/12$ is the principal moment of inertia about an axis perpendicular to the beam and to its displacement. $S = wt$ is the cross-sectional area, E is the elastic modulus, and

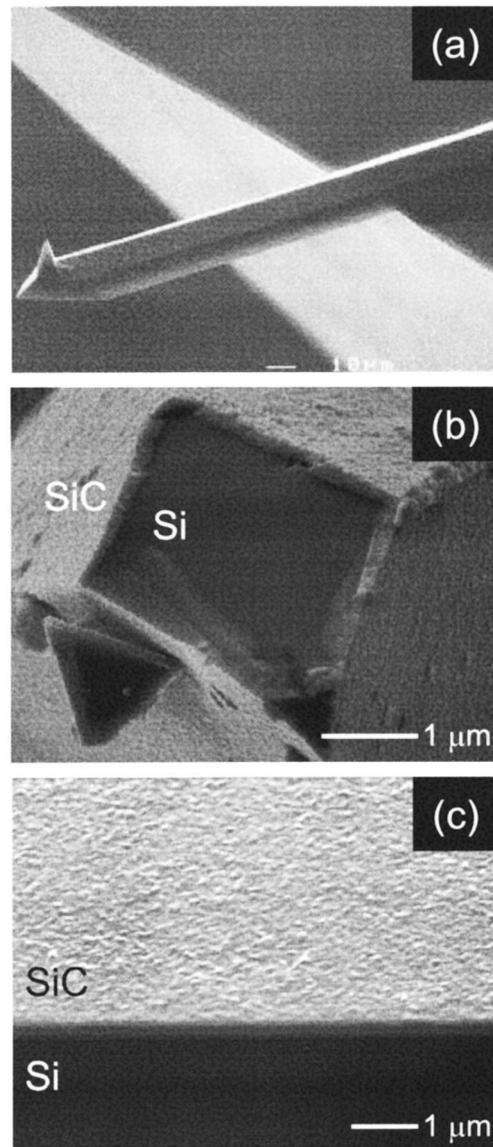


FIG. 1. SEM images of (a) a Si AFM cantilever coated with a 210-nm-thick SiC film; (b) the cross section of a Si AFM tip coated with SiC illustrating the film's conformality; and (c) the sidewall cross section of a coated AFM tip showing the SiC–Si interface.

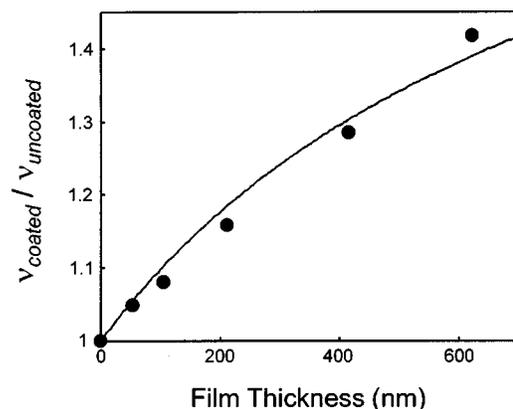


FIG. 2. Measured resonance frequency ratio of the SiC-coated Si AFM cantilevers plotted vs the deposited SiC film thickness (solid circles). The solid line is the theoretical resonance frequency ratio ν_c/ν_u fit to the measured resonance frequency ratio, yielding the elastic modulus of the deposited SiC films.

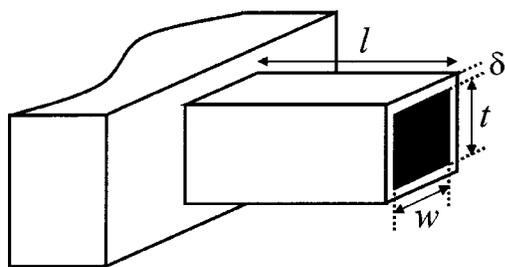


FIG. 3. Schematic showing the key linear dimensions for a composite cantilever beam of rectangular cross section (not to scale). Uncoated cantilever length l , thickness t , and width w ; and coating thickness δ .

ρ is the material density. A thin, uniform coating of thickness δ causes a change in both the moment of inertia (numerator) and the mass (denominator). Accounting for the changes in E and ρ through first order in film thickness δ , Eq. (1) is transformed to

$$\nu_c = \frac{3.52t}{2\pi l^2} \sqrt{\frac{E_1 \frac{wt}{12} + E_2 \delta \left(\frac{w}{2} + \frac{t}{6} \right)}{\rho_1 tw + 2\rho_2 \delta(w+t)}}, \quad (2)$$

where l , t , and w are still the length, thickness, and width of the uncoated beam, respectively. E_1 and ρ_1 are the elastic modulus and density of the silicon beam, and E_2 and ρ_2 are the elastic modulus and density of the SiC coating. The elastic modulus and density of silicon are 190 GPa and 2.3 g/cm³, respectively, and the density of cubic silicon carbide, ρ_2 , is 3.2 g/cm³.¹³ By fitting the theoretical frequency ratio ν_c/ν_u to the measured frequency ratio (shown as the solid line in Fig. 2), elastic modulus E_2 of the deposited coating is determined to be ~ 360 GPa. Numerical analysis accounting for the actual shape of the cantilever (whose cross section is not perfectly rectangular) shows that Eq. (2) causes the elastic modulus of the thin SiC film to be underestimated by approximately 6%. The value of the elastic modulus thus obtained (380 GPa) is in good agreement with previous estimates for the elastic modulus of cubic SiC thin films¹⁴ and cantilever beams.¹⁵

The uniformity and chemical stability of the SiC coating are tested by immersing a cantilever with a 100-nm-thick SiC coating in 33 wt % KOH solution at 65 °C for up to 6

min. The resonance frequency of the cantilever decreases by less than 2%, whereas an uncoated silicon cantilever shows a 60% decrease in resonance frequency after only 1 min of immersion in KOH. SEM analysis reveals that the uncoated silicon cantilever beam is etched from an original thickness of 3.5 to 2.1 μm .

In conclusion, we report that the resonance frequency of AFM cantilevers can be used to determine elastic properties of thin deposited films. We have demonstrated that a single precursor for depositing polycrystalline-SiC thin films can be used to coat Si-based micromechanical structures. Conformal and uniform SiC films are grown on Si AFM cantilevers at relatively low temperatures, giving highly favorable elastic properties. This method is technically simple and can easily be adopted in current IC fabrication laboratories. Furthermore, this CVD method proves promising for applications in harsh environments where current technologies cannot be employed.

The authors gratefully acknowledge support for this work from DARPA, Sandia National Laboratories, and the Arnold and Mabel Beckman Foundation. One of the authors (M.C.F.) acknowledges additional support from the Austrian Science Foundation (Schrödinger Fellowship).

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