SILICON CARBIDE FOR ENHANCED MEMS RELIABILITY

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ABSTRACT

The tribological characteristics of SiC films, both as a structural material and as a thin passivating coating, are characterized using micromachined cantilever beam arrays, doubly clamped beams, and sidewall adhesion and friction testers. Our results indicate that using SiC as the substrate or structural layer or thin film coating significantly reduces stiction of free-standing structures to the substrate, and virtually eliminates stiction and wear of sidewall MEMS structures.

INTRODUCTION

A major reliability problem with silicon-based MEMS is unwanted adhesion, friction and wear (collectively called tribology). Adhesion, which is also referred to as "stiction" in MEMS research community, results from the dominance of surface forces, such as capillary, chemical, electrostatic, or van der Waals, over body forces at the micrometer scale. Stiction can be categorized into two classes: release stiction and in-use stiction. Release stiction occurs during the release step of MEMS devices, where the sacrificial material is removed in liquid etchants and the microstructures are subsequently dried. During the drying process, the liquid capillary forces produced at the liquid-vapor interface are sufficient to draw compliant microstructure surfaces into contact and cause stiction. The stiction remains if the restoring force generated by the micromechanical structure is not large enough to overcome the adhesion. Release stiction problems are most commonly solved by designing new processes to dry the MEMS devices without creating the liquid-vapor interface (such as critical point drying technique), and modifying the surface texture to reduce the contact area (such as roughening the surfaces and using dimples) or changing the water meniscus shape (such as self-assembled monolayer coating technique). Although release stiction can be avoided using these techniques, microstructure surfaces may still come into contact and impact or shear against each other when the device is in operation, in storage or improperly handled after the release stage, which may lead to the second class of stiction, in-use stiction. The in-use stiction poses a great threat to MEMS reliability, and in most cases requires surface modification of the structural material to prevent it from occurring.

To date, coating the MEMS devices with either solution-based or vapor-based anti-stiction monolayers appears to be an effective method to overcome the in-use stiction problem. However, some microstructures, such as the hubs on microgears, experience tremendous wear in operation, which is another issue that plagues the MEMS reliability. Although some of the anti-stiction monolayers are shown to significantly reduce the static friction between the sliding surfaces, they are susceptible to large number of wear cycles. Therefore, the integration of hard materials, as either structural or coating layers, into MEMS technology is of great interest to the current research on the wear

issue in MEMS. One approach is to find a material that can be processed by microfabrication methodologies, and is able to reduce the in-use stiction and to provide wear resistance simultaneously. SiC is one such promising material. Recent advances in polycrystalline SiC technology for MEMS [1,2] enable the use of SiC both as a structural layer and as a thin passivating coating for MEMS devices. In this paper, we report on the tribological characteristics of SiC films for enhanced MEMS reliability. These include the characterization of SiC-to-Si and SiC-to-SiC stiction, as well as the effectiveness of SiC as a wear resistant coating material. In addition, SiC-to-SiC sidewall stiction is examined. Our results indicate that using SiC as the substrate or structural layer or thin film coating significantly reduces stiction of free-standing structures to the substrate, and virtually eliminates stiction and wear of sidewall MEMS structures.

EXPERIMENTAL DETAILS

The SiC films used in our experiment are deposited by a low-pressure chemical vapor deposition (LPCVD) process at 800 °C using a single precursor 1,3-disilabutane (DSB) in a commercial 3-inch horizontally oriented hot-wall tubular reactor (TekVac CVD-300-M). Details of the LPCVD process are described elsewhere [3]. When the SiC films are used as substrate or structural materials, hydrogen-diluted ammonia (5% NH₃ in H₂) is introduced to the reactor along with the precursor DSB (with flow rate ratio of 1:5 for diluted NH₃ to DSB) to dope the SiC films. The resistivity of the SiC films is about 45 mΩ·cm in experiments reported here. The SiC films are etched using HBr/Cl₂ transformer coupled plasmas (TCP) in a commercial LAM TCP 9400 etcher [4]. The etch process parameters are chosen so that relatively straight SiC sidewalls are obtained.

Cantilever beam arrays (CBA) and doubly clamped beams are employed to characterize the SiC-to-Si and SiC-to-SiC stiction when SiC is used either as the structural or substrate material. Specifically, structures with the following combinations of structural and substrate materials are fabricated and tested: SiC on polycrystalline Si (poly-Si), SiC on SiC, and poly-Si on SiC. As a comparison, stiction of poly-Si structures to poly-Si substrate is also examined. To characterize the stiction of SiC-to-SiC sidewalls, sidewall stiction testing structures are employed. Such a structure consists of a doubly clamped beam and an anchored pad with one side of their sidewalls facing each other. Schematic views of these testing structures are shown in Fig. 1. The fabrication of these structures involves the following steps: (i) deposition of the substrate material (~1 μm thick) on Si wafers, (ii) deposition of sacrificial silicon oxide (~2 μm thick), (iii) deposition of the structural material (~2 μm thick), (iv) patterning the structures using one mask photolithography, and (v) etching of the structural

Travel support has been generously provided by the Transducers Research Foundation and by the DARPA MEMS and DARPA BioFlips programs.

material. All the test structures are released by time-etching the sacrificial silicon oxide in 49% HF aqueous solution, followed by rinsing with deionized water and isopropanol. They are dried in air of normal laboratory ambient (20 °C and 40% relative humidity) to examine the release stiction. To characterize the in-use stiction, the surfaces to be examined are manually pushed into contact by a tungsten probe while sustaining the contact pressure for several seconds. Some testing parts adhered together due to the release stiction are manually separated using tungsten probes before investigating the in-use stiction. The residual strain information for the structural layer is obtained from the strain gauges fabricated around the stiction testing structures to provide parameters for the calculation of the apparent work of adhesion [5].

Figure 1. A schematic view of the testing structures employed to characterize the stiction of the structural material to the substrate (cantilever beam and doubly clamped beam), and the sidewall stiction of the structural material (sidewall stiction tester).

To evaluate the effectiveness of SiC as a coating material, sidewall friction testers fabricated from poly-Si are coated conformally with a thin SiC coating. The design and the testing scheme of the sidewall friction testers are detailed elsewhere. [6]. Briefly, the device consists of a fixed post and a flexible beam. The beam is pulled to contact and rub against the post during the wear test. Before the structures are encapsulated with SiC, poly-Si sidewall friction testers are released and coated with an octadecene-based monolayer to avoid the stiction and to protect the poly-Si surface from being oxidized by air [7]. The samples are next transferred into the LPCVD reactor and coated with ~40 nm SiC films. The SiC films are not doped when used as coating materials in these experiments. With these devices, the static friction coefficient of the SiC films is obtained as a function of the wear cycles and compared with that of the native oxide on poly-Si surfaces.

The probing system used for all micromachine actuation is a Lucas-Signatone S-1160 with a Mitutoyo FineScope 60 microscope, equipped with a Sony CCD-IRIS camera. A Nomarski style differential interference contrast prism (manufactured by Olympus) is used with a Mitutoyo M Plan Apo 10× objective on the microscope to measure the detachment length of CBA. The testing structures are also examined by scanning electron microscopy (SEM) afterwards.

RESULTS AND DISCUSSION

CBA with poly-Si beams (0.1 to 1.5 mm long, 10 μm wide, 2 μm thick and 2 μm away from the substrate) are fabricated on both poly-Si and SiC substrates and tested. The in-use stiction

results for poly-Si to poly-Si and poly-Si to SiC contacts are listed in Table 1. The apparent work of adhesion is calculated from the detachment length using Eqn. (1) [8].

$$\gamma_s = \frac{3Et^3h^2}{8l^4}, \quad (1)$$

where γ_s is the apparent work of adhesion, E is the Young's modulus of the structural material, h is the thickness of the sacrificial layer, t is the thickness of the beam, and l is the detachment length.

Table 1. In-use stiction results for contacts of poly-Si to Si and poly-Si to SiC surfaces. The apparent work of adhesion is calculated from the detachment length of cantilever beams.

Structural Material	Substrate Material	Treatment	Cantilever Beams	
			Detachment Length (μm)	Apparent Work of Adhesion (mJ/m ²)
poly-Si	poly-Si	After release	200	20
poly-Si	SiC	After release	>1500	<0.006
		After exposed to room air for 1 week	>1500	<0.006
		After exposed to room air for 2 weeks	1000	0.034

It is observed that the apparent work of adhesion of newly released poly-Si to SiC contacts is more than 4 orders of magnitude less than that of poly-Si to poly-Si contacts, which makes the detachment length of cantilever beams more than 1.5 mm long. The detachment length only begins to decrease (and hence, the work of adhesion to increase) after the structure is exposed to room air (40% relative humidity) for two weeks. Even then, the replacement of poly-Si with SiC still significantly reduces the stiction of MEMS structures. We expect that under hermetic encapsulation, SiC will retain its extremely low stiction characteristics indefinitely.

The AFM image of the SiC film as a substrate material is shown in Fig. 2. We believe that the relatively rough surface and the film topography with sharp spikes contribute significantly to the reduced in-use stiction.

Due to the relatively high residual strain gradient ($8 \times 10^{-5} \mu\text{m}^{-1}$) of the SiC films, SiC cantilever beams curve down, making the stiction characterization by CBA impractical. Doubly clamped SiC beams (0.5 to 4 mm long, 10 μm wide, 2 μm thick and 2 μm away from the substrate) are also present on the mask set. All the doubly clamped SiC beams up to 4 mm-long (the longest on the mask set) on both Si and SiC substrates are found to be free-standing after being released and dried in air without employing the critical point drying technique, and remain stiction-free after manually pushing into contact with the substrate (Fig. 3). Since no beam was observed to adhere to the substrate, the detachment length could not be determined. If one assumed it to be equal to the length of the longest beam in the mask set, one would determine an upper bound to the apparent work of adhesion from Eqn. (2) after taking the residual strain ϵ_R into account [8]:

$$\gamma_s = \frac{128Et^3h^2}{5l^4} \left[1 + \frac{4l^2}{21t^2} \epsilon_R + \frac{256}{2205} \left(\frac{h}{t} \right)^2 \right]. \quad (2)$$

Because of the large residual strain of the SiC films (1.14×10^{-3}), the upper bound to the apparent work of adhesion is found to be

$\sim 7 \text{ mJ/m}^2$ versus about 20 mJ/m^2 for Si-to-Si contacts. Although the actual apparent work of adhesion for SiC-to-Si and SiC-to-SiC contact is not acquired from this set of experiments, the result is still promising for SiC MEMS devices with clamped-clamped structures, which are very likely free of both release and in-use stiction.

Figure 2. AFM image of the SiC film as a substrate material. The SiC film is about $1 \mu\text{m}$ thick, and has a RMS roughness of 12.4 nm . The scan size is $5 \times 5 \mu\text{m}$, and vertical scale bar is 100 nm .

Figure 3. SiC doubly clamped beams up to 4 mm long on Si substrates. The beams are free-standing after being released and dried in air (without employing the critical point drying technique), and remain stiction-free after manually pushing them into contact with the substrate.

Figure 4 shows the SiC sidewall stiction testers after they are released and manually actuated. The doubly clamped beams are 0.5 to 4 mm long, $2 \mu\text{m}$ wide, $2 \mu\text{m}$ thick and $2 \mu\text{m}$ away from the fixed sidewall. Similar to the in-plane stiction testing, stiction on the SiC-to-SiC sidewall is not observed either upon release and drying, or upon manual actuation.

Sidewall friction testers (Fig. 5) fabricated from poly-Si are tested with and without SiC coating. The SEM image of a rubbing beam of a SiC-coated side-wall friction tester after 1 million wear cycles is shown in Fig. 6(a). Another rubbing beam without any coating which has failed due to high friction and wear after about 11,000 cycles is shown in Fig. 6(b). By comparing these two images, one can clearly see the wear debris and the worn surface of poly-Si without SiC coating, which are not obviously observed with SiC coating. This observation is consistent with earlier reports of enhanced wear resistance observed by AFM studies on 3C-SiC coated Si (deposited by dual precursor APCVD method) compared to uncoated Si [9]. It is postulated that the enhanced wear resistance of the SiC film is due to the higher hardness and fracture toughness of SiC.

Static friction coefficient as a function of rubbing cycle is reported in Fig. 7 and compared to the base line of Si-to-Si contact (i.e., without SiC coating). As is observed, without any coating, the friction coefficient increases as the wear cycles increase and the device fails after 11,000 cycles. But with a thin

SiC coating, the device still works after a million rubbing cycles, and the friction coefficient first decreases and then stabilizes as the number of wear cycles increases, indicative of the self-lubricating characteristics of SiC. A similar behavior has been reported previously when SiC-SiC contacts are exposed to oxygen with partial pressures greater than 1 Pa [10]. The authors attribute the tribochemically generated lubrication to the sp^2 -hybridized carbon. We suggest that a similar phenomenon might be responsible for the observed reduction in the friction coefficient in our experiments.

Figure 4. (a) The SiC-to-SiC sidewall stiction testers. The SiC doubly clamped beams are 0.5 to 4 mm long, $2 \mu\text{m}$ thick, and $2 \mu\text{m}$ wide and separated from the anchored sidewalls by $2 \mu\text{m}$. Only the 0.5 mm long testers are shown in this image. (b) A close-up of the sidewalls and gap of the tester in (a). Stiction is never observed either upon release and drying in air, or upon manual actuation of these testers.

Figure 5. An optical image of the contacting parts of the sidewall friction tester.

Figure 6. (a) SEM image of the rubbing beam of a SiC-coated side-wall friction tester after 1 million wear cycles. Note the absence of wear scarring or debris formation on the beam. (b) SEM of the tester without any coating which has failed due to high friction and wear after about 11,000 cycles. Note the wear debris in this case.

Figure 7. Wear information for sidewall friction testers with various surface treatments. In the case of oxide samples, the last data point represents failure of the device. The SiC device was not observed to fail by wear.

CONCLUSIONS

We have investigated the stiction, friction, and wear characteristics of SiC films, when used as structural materials and as thin coatings for MEMS devices. Our results indicate that using SiC as the substrate or structural layer significantly reduces the in-use stiction of MEMS structures, and the application of a thin coating of SiC can dramatically improve the wear resistance of MEMS devices. In addition, self-lubricating

phenomenon is observed for SiC-SiC sidewall contacts during wear and friction testing.

ACKNOWLEDGMENT

The authors wish to thank Dr. Muthu Wijesundara for his assistance with the SiC film deposition. The financial support of DARPA MTO NMASP and MPG programs and the Sandia National Laboratories is gratefully acknowledged. One of the authors (D. Gao) acknowledges additional support from the Applied Materials, Inc. in the form of a graduate fellowship.

REFERENCES

1. C. R. Stoldt, C. Carraro, W. R. Ashurst, D. Gao, R. T. Howe and R. Maboudian, "A Low-Temperature CVD Process for Silicon Carbide MEMS," *Sensor Actuat. A*, vol. 97, 410 (2002).
2. D. Gao, M.B.J. Wijesundara, C. Carraro, C. W. Low, R. T. Howe, and R. Maboudian, "High Modulus Polycrystalline 3C-SiC Technology For RF MEMS," in *Proc. Transducers'03, The 12th Int. Conf. Solid-State Sensors & Actuators* (2003), pp.1160-1163.
3. M.B.J. Wijesundara, G. Valente, W. R. Ashurst, R. T. Howe, A.P. Pisano, C. Carraro, and R. Maboudian, "Single-source chemical vapor deposition of 3C-SiC films in a LPCVD reactor I. Growth, structure, and chemical characterization," *J. Electrochem. Soc.*, 151 (3), C210 (2004).
4. D. Gao, R. T. Howe, and R. Maboudian, "High-selectivity Etching of Polycrystalline 3C-SiC Films using HBr-based Transformer Coupled Plasma," *Appl. Phys. Lett.*, 82(11), 1742 (2003).
5. D. Gao, M.B.J. Wijesundara, R. T. Howe, and R. Maboudian, "Characterization of Residual Strain in SiC Films Deposited using 1,3-Disilabutane for MEMS Applications," *J. Microlithography, Microfabrication, & Microsystems*, 2 (4), 259 (2003).
6. W. R. Ashurst, C. Yau, C. Carraro, R. Maboudian, and M. T. Dugger, "Dichlorodimethylsilane as an anti-stiction monolayer for MEMS: A comparison to the octadecyltrichlorosilane self-assembled monolayers," *J. Microelectromech. Syst.* 10 (1), 41 (2001).
7. W. R. Ashurst, C. Yau, C. Carraro, C. Lee, G. J. Kluth, R. T. Howe, and R. Maboudian, "Alkene based monolayer films as anti-stiction coatings for polysilicon MEMS," *Sensors and Actuators A* 91, 239 (2001).
8. C. H. Mastrangelo, "Adhesion-related failure mechanisms in micromechanical devices," *Tribol. Lett.*, 3, 223 (1997).
9. S. Sundararajan, and B. Bhushan, "Micro/nanotribological studies of polysilicon and SiC films for MEMS applications," *Wear*, 217, 251 (1998).
10. J. M. Martin, and T. le Mogne, "Interpretation of friction and wear of ceramics in terms of surface analysis," *Surface and Coatings Technology*, 49, 427, (1991).