

Vapor Phase Anti-Stiction Monolayer Coatings for MEMS

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(Invited Paper)

Abstract—Despite the progress in micromachining technology, adhesion and friction remain key issues that limit the realization and reliability of many microdevices. Conventional approaches to abate the deleterious effects adhesion and friction rely on the deposition of organically-based anti-stiction monolayers produced from liquid-phase processes. It has become widely accepted that liquid-phase monolayer processes are less desirable than vapor-phase processes, especially for manufacturing purposes. Thus, current research is aimed at the development of vapor-phase anti-stiction processes that yield comparable or better films than their corresponding liquid-phase processes. To date, a variety of monolayer systems that have been well established via liquid-phase deposition processes have been adapted to vapor processes. In this review paper, current trends in anti-stiction technology and a discussion of available vapor-phase anti-stiction methods are presented.

Index Terms—MEMS, Monolayer Coatings, Stiction, Vapor Coatings

I. STICTION IN MEMS

THE integration of miniaturized mechanical components with microelectronic components has spawned a new technology, known as microelectromechanical systems (MEMS). This technology promises to extend the benefits of microelectronic fabrication to sensing and actuating functions [1]–[5]. Some notable commercial examples of MEMS include Analog Devices ADXL series accelerometers and the Texas Instruments DMD™.

One of the core technological processes underlying MEMS is termed surface micromachining, and it involves the fabrication of micromechanical structures from deposited thin films [6]–[8]. Typical microstructures made by surface micromachining have lateral dimensions of 10–500 μm , with thicknesses of 0.1–2.5 μm , and are offset 0.1–2 μm from the substrate. Here, the large surface-area-to-volume ratios of surface and bulk micromachined micromechanisms bring the role of surface and interfacial forces into the foreground [9]–[15]. Stiction is a term (originally from the disk drive industry) that has been applied to the unintentional adhesion of compliant microstructure surfaces when restoring forces are unable to overcome interfacial forces such as capillary, chemical, van der Waals and electrostatic attractions.

Release stiction, the adhesion of surface-micromachined structures to the underlying substrate following the final sacrificial layer etch, is caused primarily by liquid capillary forces. Engineering solutions to this problem include surface roughening, critical point drying, freeze-sublimation, polymer

ashing and gas phase etching of sacrificial layers and have been extensively reviewed elsewhere [16]–[23].

These techniques, however, do not prevent adhesion from occurring during micromachine operation. Microstructure surfaces may come into contact unintentionally through acceleration or electrostatic forces, or intentionally in applications where surfaces impact or shear against each other. When surface adhesive attractions exceed the intrinsic microstructure restoring forces, surfaces permanently adhere to each other causing device failure—a phenomenon known as in-use stiction. To reduce in-use stiction, the approaches fall under two categories, physical and chemical modifications of surfaces. In the physical approach, the surfaces are roughened to reduce the effective contact areas [9], [24]. Typically selective etching is employed to change the surface topography. With this approach, the largest reported reduction in in-use stiction is a factor of 20 [25]. In the chemical approach, the surface chemical composition is altered. With this approach, stiction can be reduced by about four orders of magnitude. Hence, more research has focused on chemical modification of surfaces for stiction reduction.

For commercial viability and industrial growth of MEMS to continue, micromechanical systems must be built with high yields and reproducible device properties and must exhibit reliability over the expected device lifetime. However, the traditional high yields experienced in the IC industry extend to MEMS production only up to the point of microstructure release, and virtually every type of micromechanical device is susceptible to stiction. Also, microactuators have surfaces in normal or sliding contact, friction and wear are also important issues. Indeed, stiction and friction are commonly cited as major failure modes for micromachines [26].

II. ANTI-STICTION MONOLAYERS

Various types of chemical modifications have been explored as possible anti-stiction treatments for MEMS, and have been reviewed elsewhere [13], [27]. In general, these chemical modification techniques have employed solution based (liquid-phase) synthesis schemes which result in monolayer formation. It is well known that the application of hydrocarbon or fluorocarbon chlorosilane-based self assembled monolayers (SAMs) can greatly improve the adhesion properties of micromechanical surfaces [11]–[14], [27], [28].

Although these monolayer systems provide many favorable characteristics, they also possess qualities that limit their applicability. Such qualities can be grouped into two types, those that arise due to the chemistry involved, and those that are

due to the solution based SAM formation processes. Although chemistry related limitations can be significant, the issues related to the SAM deposition *process* are what generally limit the widespread application of the SAMs as anti-stiction methods in industry [27]. One limitation is that the coating process is cumbersome because the SAM solution must be freshly made and appropriately conditioned immediately before each coating. This is due to the sensitivity of the SAM solution to ambient humidity, and the ability of the SAM precursors to polymerize.

Another difficulty related to the coating process is one of "portability." The large number of process variables, varying expertise and care of operators, as well as the lack of standardized wafer level coating equipment make the success rate of SAM coating processes vary considerably from run to run and laboratory to laboratory. Perhaps the greatest limitation is that of scaling up the process from single dies to whole wafers and eventually multi-wafer cassettes. Much of the work up to this stage has consisted of releasing dies $1 \times 1 \text{ cm}^2$. Scaling up the release and coating process to full-wafer level and eventually to multi-wafer cassettes poses substantial engineering challenges.

Recent developments in anti-stiction monolayer technology address some of these issues by performing the coating process in the vapor phase. Vapor phase processing eliminates the use of organic solvents and greatly simplifies handling of the samples. Moreover, the stoichiometry of the precursor molecules can be more precisely controlled. With vapor processing, scaleability is achieved, and the process control schemes can be implemented with relative ease.

Although there are a wide variety of anti-stiction precursors available, the methods used to deposit them onto microstructures and other substrates follow similar steps. In general, a vapor phase anti-stiction coating process consists of three distinguishable steps. The first step is the release process. The release process should be designed and implemented such that release stiction is avoided, and high device yield is achieved. Following the release step, a surface preparation step is used. Typically, this involves a combination of surface cleaning and surface reactivity modification. In the specific case of chlorosilane based chemistry, a downstream oxygen or water plasma is useful for this purpose since it both strips adsorbed organic material and oxidizes the surface. Finally, the released and prepared microstructures are exposed to a vapor containing the chosen precursor for a given amount of time. After this surface reaction step is completed, the excess precursor is purged, and the sample is ready for testing or further processing (such as packaging).

Chlorosilanes represent one class of precursors used for anti-stiction coatings. Other classes include amines, alcohols, carboxylic acids, siloxanes and dimethylaminosilanes. The structural formulas for some of these precursors can be found in Fig. 1. In the sections that follow, each class of precursors is discussed.

III. CHLOROSILANE BASED MONOLAYERS

Chlorosilane-based monolayer coatings which function as anti-stiction layers for micromachines have received much

attention in recent years. The commercial availability of the chlorosilane precursors, and the apparent simplicity of the coating process make them attractive to a wide variety of researchers. Moreover, the availability of a wide variety of functionalized pendant groups makes chlorosilanes a useful intermediate to many applications, such as the control of protein binding on silicon based devices (see e.g. [29], [30]). Chlorosilanes have the general formula $R_{4-n}SiCl_n$, where $1 \leq n \leq 3$, and the tail group(s), R, may be hydroxylated, fluorinated or otherwise functionalized carbon chains or aromatic substituents. In the case of $n < 3$, each R is not necessarily the same. The reactions that are involved in attaching chlorosilane precursors to silicon surfaces are all the same. The first reaction is the hydrolysis on the Si-Cl bonds to form Si-OH bonds and HCl. The number of water molecules required and HCl molecules produced in the case of complete hydrolysis is n . After the silanol form of the precursor is generated, some of the -OH groups participate in a condensation reaction with a -OH group on the oxidized silicon surface. This process liberates one water molecule per condensation reaction. The exact fate of every Si-OH bond in tri- and di-chlorosilanes is uncertain, since cross polymerization (condensation reactions with other precursor molecules) is also possible.

In the sections that follow, information about some chlorosilane-based monolayer is presented. These are grouped according to the chlorine number (n). Property data for these chlorosilane precursors can be found in Table I. Also in the table are typical values for untreated (oxidized) silicon and the conventional benchmark anti-stiction film octadecyltrichlorosilane (OTS) deposited from solution. Work of adhesion data are obtained by the cantilever beam array technique [31], and coefficient of static friction data are obtained with a sidewall friction device [32].

A. Trichlorosilanes

1) *FOTS*: It has been demonstrated that monolayer films that are produced from the precursor tridecafluoro-1,1,2,2-tetrahydrooctyltrichlorosilane $(CF_3(CF_2)_5(CH_2)_2SiCl_3$, FOTS) in a low-pressure CVD style reactor exhibit low adhesion energies [33], [34]. Figure 1 shows a structural diagram of FOTS. This precursor can be deposited in the general way described previously. In one experimental setup, *in-situ* ellipsometric measurement of the film growth was performed. A quartz crystal microbalance was also used to independently measure the film thickness. From these analytical techniques, it was concluded that the VFOTS monolayer was about 70–90% of a complete monolayer. Atomic force microscopy (AFM) scans on treated Si(100) revealed smooth, low-particle surfaces. Adhesion data found from micromachine samples (cantilever beams) indicates that the VFOTS film is quite effective at stiction reduction. Overall, it was found that the deposition process provide excellent process uniformity, reproducibility and monitoring capability [33].

2) *FOTS*: Another related precursor hepta-decafluoro-1,1,2,2-tetrahydrodecyltrichlorosilane $(CF_3(CF_2)_7(CH_2)_2SiCl_3$, FOTS) has also been successfully

deposited on micromachines from the vapor phase (VFDTs). The main difference between FDTS and FOTS process is that the FDTS liquid source must be heated to about 100°C (by a boiling water bath) to generate enough vapor pressure to carry out the process.

In order to confirm that FDTS was applied to the sample X-ray photoelectron spectroscopy was used. Figure 2 shows XPS survey spectra of VFDTs and OTS coated Si(100). Note the large F (1s) peak present in the VFDTs spectrum which is not present in the OTS spectrum. Also note the C (1s) doublet in the VFDTs spectrum versus the singlet in the OTS spectrum. Figure 3 shows a close-up spectrum of the C (1s) peak. Here, it is important to observe the splitting in the carbon peak that is indicative of -CF₂- (and -CF₃), which could only be present due to the FDTS precursor. The presence of F, -CF₂- and -CF₃ in these spectra confirm that the FDTS precursor has been applied to the Si(100) sample.

AFM has also been used to quantify the roughness of coated Si(100) samples. It was found that the film roughness was about 0.1 nm, which was close to that of bare oxidized Si(100). This VFDTs process has also been successfully applied to micromachine samples. Property data for this coating can be found in Table I. Specifically, cantilever beams and sidewall friction testers have been coated and used to determine the film characteristics. Cantilever beams show an apparent work of adhesion of about 3 μJ/m². Moreover, the beams exhibit a sharp transition from free-standing to arc shaped. Sidewall friction testers are used to measure a μ_s of about 0.12 for the VFDTs coating.

B. Dichlorosilanes

The dimethyldichlorosilane ((CH₃)₂SiCl₂, DDMS) monolayer has also been shown to be an effective anti-stiction surface coating for MEMS. Figure 1 shows a structural diagram of DDMS. As discussed elsewhere, the DDMS monolayer is quite effective at reducing friction and adhesion, although not quite as effective as OTS [32], [35]. This precursor has several properties that make it an attractive candidate for vapor phase processing. One is that the DDMS precursor has a lower tendency to polymerize than other trichlorosilanes. Another is that it has a vapor pressure at room temperature that is much greater than any other chlorosilane precursor used in anti-stiction processes. Moreover, for every DDMS molecule that gets hydrolyzed, only two molecules of HCl will result, as opposed to three from OTS or FDTS. Perhaps the most compelling distinction that DDMS has over OTS is that it has a much greater thermal stability in an oxidizing ambient. In fact, the DDMS films can withstand upwards of 400°C whereas OTS films begin to degrade at about 225°C [36]. The vapor DDMS (VDDMS) films have been shown to have less particles than the corresponding solution deposited films, see Fig. 4.

IV. ALCOHOLS AND AMINES

Some other monolayer processes have been developed by Zhu *et al.* which exploit Si-O or Si-N linkages [37]. In particular, the surface reactions of amines and alcohols of

the form R-NH₂ and R-OH (R is an alkane or perfluorinated alkane) with Cl-terminated Si, in both vacuum and solution have been studied. The Cl terminated surface can be generated by exposing a H-terminated Si sample to Cl₂ in vacuum while either heating the substrate to about 80°C or illuminating it with a tungsten filament. The surface reaction can be accomplished by exposure of the Cl-terminated Si to the R-NH₂ or R-OH precursor in vacuum or by dipping the sample in a solution containing the precursor molecules. In the case of R-NH₂, the reaction involves bridge bonding of the N to two adjacent Si atoms by the elimination of 2 HCl. In the case of the R-OH type precursors, the reaction involves the elimination of HCl and the formation of a single O-Si bond. The resultant film quality has been verified by X-ray photoelectron spectroscopy and interfacial force microscopy, and shown to be similar to other close packed alkyl films formed by other methods [38].

V. SILOXANES

Another vapor coating approach with enhanced thermal stability is reported by the Analog Devices, Inc. [39], [40]. It involves the use of a silicone or siloxane that has high resistance to temperature and oxidation. The procedure for applying this coating is quite different from the others reported here, as it is applied during the packaging step. In production, a small amount of the chosen precursor liquid is dispensed into each device package before it is sealed. As the temperature is raised in the sealing furnace, the liquid evaporates. Most of the vapor escapes but not before contacting the surfaces in the package cavity, including the MEMS sensor. The hot MEMS surfaces initially possess high surface energies and hence the aforementioned molecules contacting the surfaces chemically react and bond to them. As the surface becomes enriched in organic groups, its surface energy lowers, and hence, its reactivity towards the vapor diminishes. The result is a monolayer coating with a high organic content. Some precursors that have been used in this manner include trimethylpentaphenyl trisiloxane oil, dimethyl siloxane, cyclic dimethyl siloxane, tetramethyltetraphenyl trisiloxane, and cyclic silicones such as octaphenylcyclotetrasiloxane.

VI. tris-DIMETHYLAMINOSILANES

Recently, a different binding chemistry has been developed, and successfully applied to micromachines. Here, the precursors are (tridecafluoro-1,1,2,2,-tetrahydrooctyl)tris-dimethylamino silane (PF8TAS) and (heptadecafluoro-1,1,2,2,-tetrahydrodecyl)tris-dimethylamino silane (PF10TAS) [41]. Figure 1 shows a structural diagram of PF10TAS. These precursors are not commercially available, but can be synthesized from their corresponding trichlorosilanes (FOTS and FDTS, respectively) and dimethylamine. It should be noted that the aminosilane precursors are extremely sensitive to water, and must be kept rigorously anhydrous.

The process for applying the aminosilane to micromachines is essentially the same as that used for the chlorosilanes. However, an important distinction is that there is no water vapor added to the chamber during the deposition. Although

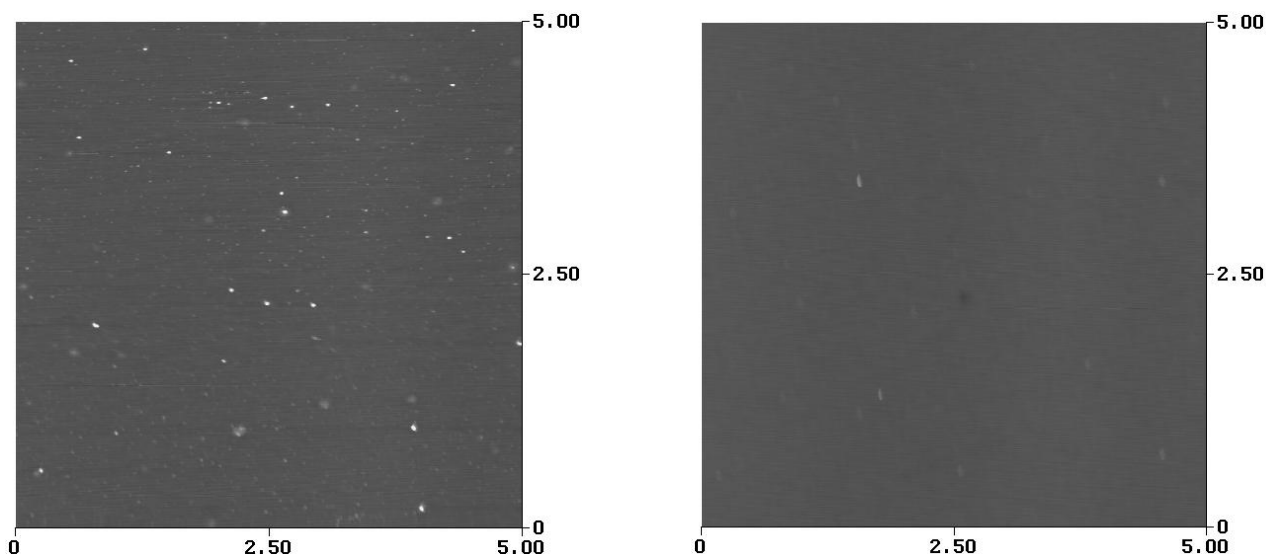


Fig. 4. AFM images of LDDMS (left) and VDDMS (right) coated Si(100). It is evident that the vapor deposited DDMS sample has significantly less particles than the solution deposited sample.

TABLE I

A COMPARISON OF PROPERTY DATA FOR A VARIETY OF MONOLAYER COATINGS. FILM TYPES THAT BEGIN WITH "L" ARE SOLUTION DEPOSITED AND THOSE THAT BEGIN WITH "V" ARE VAPOR DEPOSITED.

Film Type	Contact Angle		Work of Adhesion $\mu\text{J}/\text{m}^2$	Coefficient of Static Friction	Particles	Ref.
	Water	Hexadecane				
LDDMS	103°	38°	45	0.28	intermediate	[32]
VDDMS	102°	38°	62	0.35	low/none	[36]
LFDTs	111°	72°	<10	~0.10	very high	[43], [44]
VFDTs	111°	72°	3	0.12	low/none	[45]
VFOTs	≥110°	—	≤20	~0.3	low/none	[33], [46]
LOTS	110°	38°	12	0.07	very high	[32]
Oxide	~0–30°	~0–20°	~20,000	1.1	n/a	[32], [47]

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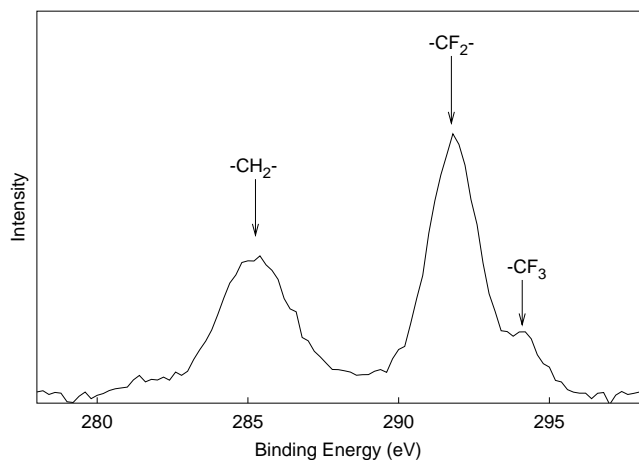


Fig. 3. Close-up XPS scan of the C (1s) region for VFDTS coated Si(100).



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