A NOVEL PROCESS FOR FABRICATING SLENDER AND COMPLIANT SUSPENDED POLY-SI MICRO-MECHANICAL STRUCTURES WITH SUB-MICRON GAP SPACING

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ABSTRACT

This paper reports on the development of a novel wet-release process for the fabrication of slender and compliant micro-mechanical structures with sub-micron gap spacing developed in an in-house CMOS compatible 8" pilot line. A Self Assembled Monolayer (SAM) of n-decyltrichlorosilane (DTS) is used as an anti-stiction material to realise suspended structures. Process characterisation includes studies on the DTS SAM, release of the polycrystalline Silicon and polycrystalline Silicon Germanium test structures. We have obtained work of adhesion values of ***11μJ/m²*** and ***0.06μJ/m²***, slenderness ratios of ***218*** and ***1000***, compliance of ***12 m/N*** and ***350 m/N***, and contact angle of ***126°*** and ***129°*** for poly -Si and poly-SiGe cantilevers respectively. Our results compare favourably well with results from the literature.

Key words: MEMS, surface micromachining, stiction, slendernes ratio, compliance, SAM, DTS, contact angle, work of adhesion

1. INTRODUCTION

Polysilicon surface micromachining technology [1] is widely used among the MEMS research community throughout the world. Many commercial foundry services [2] are offered to the research community to fabricate a variety of application specific devices. Typically, device structures with large gap spacing (> 2μm) and relatively thick structural layer (~ 2μm) are realisable in such services. One drawback of such a scheme is that rather large actuation voltages are needed in the case of electrostatic MEMS structures. There is a clear need however, for the structures with narrow gap spacing, and thin structural layers. Such structures are for instance necessary for the realisation of low-voltage electrostatic switches (<10 V) and for the easy integration into CMOS. Although, there are many ways to release the structural layer, wet release technology is the preferred technique because of the associated simplicity, the low cost and the high throughput. Despite many advancements, wet sacrificial layer etching technology is not sufficiently robust when it comes to realising compliant, slender and stiction-prone (small gap distance and large overlap area to the substrate) micro-mechanical structures. In the wet sacrificial layer etching technology, dominant forces are the surface tension force and the restoring mechanical spring force. An ideal release process guarantees successful release of micro-mechanical structures independent of the strength of these forces. Stiction is the key bottleneck for the successful realisation. However, researchers have successfully released structures that are 1-2 μm thick and as long as 2-3 mm but with a rather large gap distance (>1 μm) [3, 6, 10], or, have released short and stiff structures with a sub-micron gap distance [4]. The intermediate region encompasses structures that are long (<1 mm), thin (0.5 - 1 μm) and with a gap distance extending into sub-micron regime (0.5 - 1 μm). This work primarily focuses on the release process development covering the intermediate region. In this work, the wet release of slender, compliant and low tensile stress poly-Si and poly-SiGe micro-mechanical structures is carried out by using a novel process sequence, implementing n-decyltricholorosilane (DTS) as an anti-stiction material. We have deposited DTS in the form of self assembled monolayers (SAMs) [5] as an alternative to the currently used OTS (octadecyltrichlorosilane) and FDTS [6] (perfluorodecyltrichlorosilane). The release sequence method has been successfully applied to realise electrostatic actuators such as the pull-in structures and the micro-resonators as well as the slender poly SiGe structures with thicknesses of 0.46μm, 1μm and 2μm, and gap spacings of 0.45μm and 1μm. Further, surface, thermal, and the compositional analysis of the DTS SAM coated poly-Si surface and a comparison of the contact angle between OTS and DTS coated oxide surface is performed. A comparative study is made with our process characterisation results and results from the literature. Finally some application examples are described.

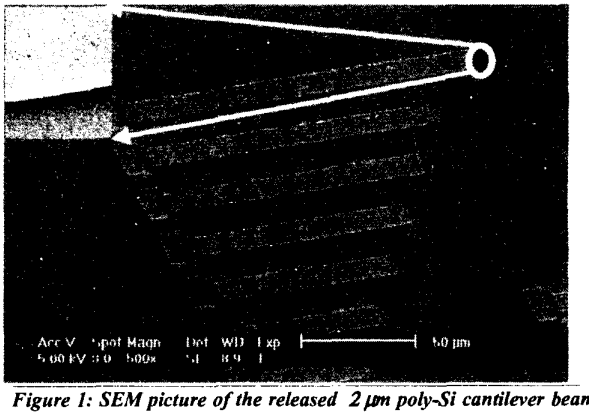

Figure 1: SEM picture of the released 2 μm poly-Si cantilever beams with 0.45 μm gap spacing

2. RELEASE PROCESS SEQUENCE

Fabricated unreleased structures are diced into individual dies of the order of few square centimetres. The dies are subjected to ultrasonic rinse in IPA (Iso Propyl Alchohol), followed by a DI water rinse and dried with a N_2 gun. The release process is illustrated schematically in Fig. 2. The novel features of the release process sequence are highlighted in Fig. 2. Our approach for chemical

passivation (stiction reduction) of the stop layer (poly-Si) surface is based on the use of Self-Assembled Monolayers (SAMs)[5]. These films were formed with n-decyltrichlorosilane (DTS) precursor molecules CH_3-$(CH_2)_9$-$SiCl_3$. Dissolution of these precursor molecules in a suitable solvent (*e.g.* dry toluene) and insertion of an oxidised surface, allows the hydrolysation of the trichlorosilane head group and subsequent chemisorption on the surface. A short temperature treatment at 150^{0}C, after initial film formation, ensures further SAM stabilisation by crosslinking between the chains. The silane SAM deposition is often referred to as 'silanisation'. Toluene was used as the final rinse liquid to keep the water content in the liquid to a minimum level thus ensuring proper conditions for the DTS SAM layer formation. A special Teflon sample holder is fabricated for the easy transferring of the sample from one beaker to the next beaker. The sample holder is built in such a way that the sample stays in the liquid medium all the time and one liquid enters by displacing the previous liquid. A SEM picture of the released poly-Si cantilever test structures is presented in Fig. 1.

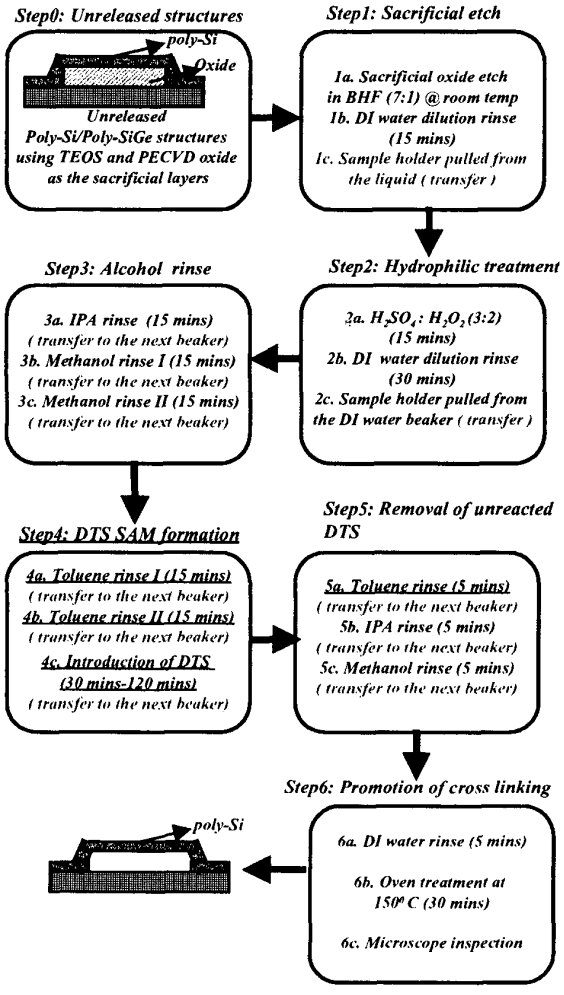

Figure2: Release process sequence implementing DTS SAM layer

3. PROCESS CHARACTERISATION

The process has been characterised in several ways, including the determination of the maximum slenderness ratio, of the contact angle after SAM deposition, XPS analysis of the SAM layer, the work of adhesion and the SAM layer thermal stability. In the following sub sections, these are explained in detail.

Slenderness Structures

Test structures are fabricated using PECVD poly-SiGe as the structural layer material and the PECVD oxide as the sacrificial layer. The PECVD oxide was deposited at 460°C, at 2 Torr pressure, with 20 sccm of SiH_4, 400 sccm of N_2, 400 sccm of H_2, 1200 sccm of N_2O, and a plasma power of 100 W. The PECVD poly-SiGe was deposited at 710°C, 2 Torr pressure, 30 sccm of SiH_4, 100 sccm of GeH_4, 40 sccm of PH_3, and plasma power of 30 W. This process results in 43% Ge incorporation into the deposited film. Using the simple linear rule of mixtures, a first order estimate of the elastic modulus is made, which is equal to 152.2 GPa. This value is used to estimate the compliance as well as the work of adhesion for the released poly-SiGe structures using the release process sequence described in Fig 2. Cantilever beams as long as 1 mm with slenderness ratio of 1000 and compliance of 443 m/N are released successfully following the release procedure described in Fig. 2. Fig. 3 shows SEM picture of the released poly-SiGe structure.

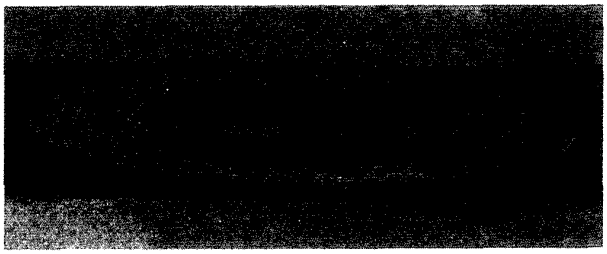

Figure3: slender wet released poly-SiGe test structures of 1 μm thickness and 1 μm gap spacing

DTS SAM Surface

To study the hydrophobic nature of the deposited DTS SAM layer, static water contact angles for various surfaces before and after the SAM formation are measured. After SAM deposition, the coated surface exhibits a strong hydrophobic nature as shown in the Fig. 4. We have deposited DTS SAM layers on the commonly used MEMS stop layer surfaces. The results of the static contact angle measurements are summarised in the table 1.

XPS Analysis

To examine the chemical nature of the surface of DTS-coated PolySi, X-ray Photo-electron Spectroscopy (XPS) analysis was performed (Table 2). After SAM-deposition, a decrease in the Silicon (*Si*) and Oxygen (*O*) concentration and an increase in the carbon (C) content were observed, indicative of successful DTS deposition on the poly-Si

surface. Moreover, the binding energy for the *Si* photo-electron peak shows a pronounced shift after the SAM formation. Before the SAM formation, *Si* has a largely metallic character (±73 % of the total *Si* has

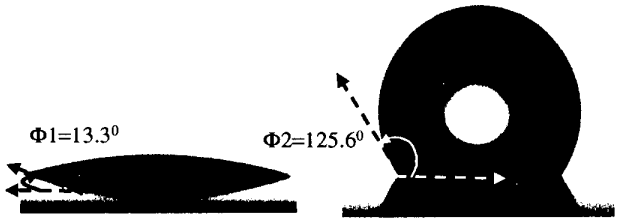

Figure 4: Wettability of a water droplet on (left) a clean surface and (right) the DTS coated surface.

Table 1: Values for static contact angles (CA) on various surfaces (values for averages and SD's are based on at least five drops).

Surfaces studied	*Contact angle without DTS*	*Contact angle with DTS*
Blanket thermal SiO_2	51.4 ±2.3°	109.3 ±1.1°
Blanket LPCVD Si_3N_4	30.2 ±1.4°	110.3 ±0.6°
Blanket Al/Si	67.9 ±0.8°	101.8±2.6°
Blanket SiC/ SiO_2/Si	57.3 ±4.4°	104 ±1.4°
Blanket PolySi	13.3 ±1.3°	125.6±0.8°
Device wafer (Si)	40°	129°

a binding energy at ±99.5 eV). After DTS SAM formation, most of the *Si* is oxidised (±70 % of the total *Si* has a binding energy at ±102.8 eV). This shift in binding energy is consistent with the occurrence of crosslinking between the DTS chains and with binding of DTS on the poly-Si surface (*i.e.* the formation Si-O-Si bonds).

Table 2: Elemental concentration on the PolySi surface, before and after DTS SAM formation (assessed with XPS).

	Si (in %)	*O (in %)*	*C (in %)*	*Contact angle with water*
PolySi blanc	45.0	43.9	11.2	13.3 ±1.3°
PolySi & DTS	35.7	28.1	36.2	125.6 ±0.8°

Thermal Stability

Encapsulation and subsequent housing of the MEMS device structures is an important criterion for the long lasting lifetime of the device. Most often encapsulation and the packaging of the fabricated MEMS structures is a step carried out at the post processing level. Thus any processed layer prior to the post processing must satisfy thermal budget requirements of the post-processing step. The thermal stability of the DTS coated poly-Si surface is determined by the temperature annealing of the sample at various temperatures in N_2 ambient. Fig. 5 shows that the DTS SAM layer can withstand temperatures as high as 400° C.

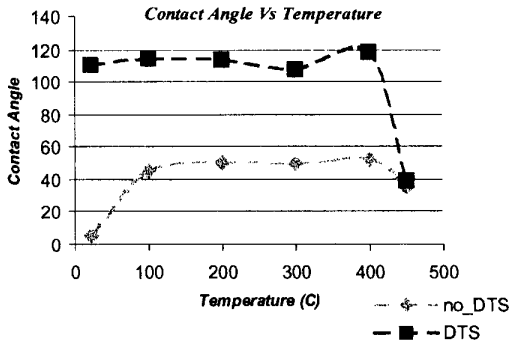

Figure 5: Contact Angle Vs Temperature on the DTS coated poly-Si surface

Comparative study

Comparison was made between SAMs of DTS and OTS, on the basis of contact angle measurements. Both the silanes were deposited from the toluene on SiO_2. It is observed that the OTS renders slightly more hydrophobic layers.

Table 3: Contact angles with water on SiO_2 samples silanised with the DTS and the OTS (in static, advancing and receding mode).

	Static	*Advancing*	*Receding*	*hysteresis*
DTS	110.9 ±1.3°	121.0 ±1.4°	96.5 ±1.3°	24.5 ±1.7°
OTS	117.1 ±1.9°	127.8 ±2.3°	106.7 ±0.6°	21.2 ±2.8°

On the contrary, when deposited from the dry CCl_4, no observable difference between DTS and OTS SAMS on SiO_2 (data not shown) noticed. Moreover, to our knowledge, the SAM deposition from OTS is more stringent than for DTS, concerning water content in the deposition solvent, deposition time and deposition ambient.

Detachment length, work of adhesion, and contact angle are the parameters often used to gauge the release process. The collected data from the literature and from our process on slenderness ratio (Sr), Compliance (C), work of adhesion (W), and contact angle are summarised in Table 4. The significant difference between our results (last two rows) is mainly due to the fact that poly-Si and poly-SiGe are build with different stack of underlying layers.

Table 4: comparison of slenderness ratio (Sr), compliance (C), work of adhesion (W), and contact angle data of the released cantilever beams

Sr	*C [m/N]*	*W [μJ/m²]*	*SAM layer (contact angle)*	*Reference*
56	3.4E-2	42E3	NA (0°)	[7]
216	4E0	45E0	DDMS (103°)	[10]
309	23E0	10E0	NA (84°)	[9]
442	68E0	3E0	OTS (114°)	[8]
218	12E0	11E0	DTS (126°)	This work (Poly-Si)
1000	3.5E2	6E-2	DTS (129°)	This work (Poly-SiGe)

4. APPLICATION

A five-level mask process has been developed to realise the electrostatic actuators for use in switches (pull-in structures) and micro-resonators. The devices are fabricated in an in-house 8" CMOS pilot line. Poly-Si deposited at 600°C with a thickness of 0.46 μm and subsequent annealing carried out at 1050°C is used as the structural layer and TEOS oxide deposited at 670°C with a thickness of 0.45 μm is used as the sacrificial oxide. These structures are released with stop layer as poly-Si, using the release sequence described in Fig. 2. Fabricated structures are tested electrically for pull-in voltage and electro-mechanically for the resonance.

The pull-in voltage has been obtained from the Capacitance-Voltage (C-V) characteristics of structures with varying length from 100 μm till 40 μm, thickness of 0.46 μm, width of 8 μm and gap spacing of 0.45μm. From the measured pull-in voltage data, the effective elastic modulus and the residual stress of the Poly-Si layer re determined using a closed-form expression of the pull-in voltage given in the reference[11]. Fig. 6 shows the least square fit for extracting the material properties. The extracted value of the elastic modulus is used to obtain an estimate of the work of adhesion. The following expression has been used to estimate the work of adhesion from the reference [12].

$W = 3\hat{E}h^3d^2/8l_d^4$, where $\hat{E}$ is the effective elastic modulus, h is the thickness of the cantilever beam, d is the gap spacing, and l_d is the detachment length obtained from the optical observation.

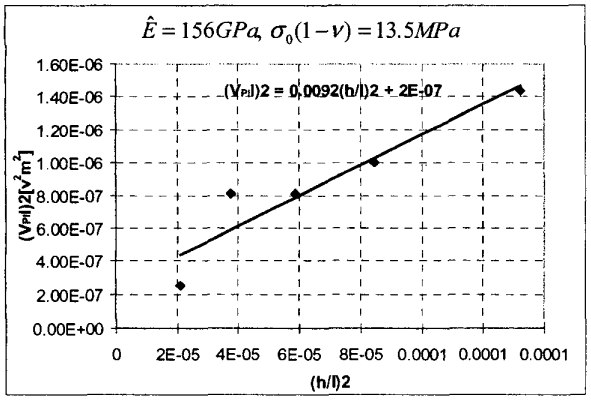

Figure 6: Method of extraction of the material properties based on the pull-in voltage data [11]

The poly-Si microresonators of length 100 μm, width 8 μm, thickness 0.46 μm, and the gap spacing of 0.45μm are successfully excited for the mechanical resonance. Because of the high compliant nature of these beams and very narrow gap spacing (high electromechanical coupling coefficient), they could be easily excited in air.

5. CONCLUSIONS

DTS SAM layer as an anti-stiction coating material is successfully integrated into the sacrificial wet release technique resulting in a novel release process. Formation of the DTS SAM layer is verified by contact angle measurement, the XPS and the thermal characterisation. The release process sequence is employed to successfully release slender and compliant structures of poly-Si as well as poly-SiGe. We have found that the values of the slenderness ratio, compliance, and the work of adhesion to be depending strongly on the stop layer at the end of the sacrificial layer etching and also on the underlying layer stack below the sacrificial oxide. Sub-micro joule adhesion energies per unit area have been achieved for our release process. DTS coated fabricated electrostatic actuators are successfully tested electrostatically for the pull-in and electrodynamically for the resonance. Noteworthy is that stiction has not been observed after repeated pull-in tests indicating the usefulness of DTS for anti-stiction coatings also during the in-use operation.

6. ACKNOWLEDGEMENTS

The authors would like to thank Cristina Rusu for timely help in depositing the PECVD Oxide and the PECVD SiGe and for the SEM analysis. The authors would also like to thank Bert Du Bois, T.Conard, R. De Palma and A. Campitelli for their technical help.

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