A Fluoro-ethoxysilane-Based Stiction-Free Release Process for Submicron Gap MEMS

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ABSTRACT

Nowadays, stiction remains one of the biggest reliability problems in the fabrication of micro- electromechanical systems (MEMS), especially when a small gap is used. To avoid the adhesion, a Self-Assembled Monolayer (SAM) could be coated. Main research in that field focuses on aliphatic chloro-silanes. We developed a novel wet-release CMOS compatible process for the fabrication of surface-micromachined beams using a perfluorated SAM. Uniform 9 Å thick monolayers were observed. A static contact angle of 117° up to 300°C was measured. Furthermore, a complete 1 μm thick (gap 0.5 μm) polysilicon RF MEMS capacitor with aluminium interconnects realized with this process demonstrated the CMOS compatibility.

Keywords: MEMS, SAM, stiction, release process, hydrophobization

1 INTRODUCTION

Surface micromachining using deposited polysilicon films is a technology that is now widely used for the fabrication of MEMS such as sensors or radio-frequency (RF) resonators and switches. In this last application, low actuation voltages ($<$ 10 V) are desired, which requires small gaps ($<$ 1 μm) between electrodes. On the other hand, the process steps have to be compatible with CMOS technology.

One of the biggest reliability problems in the fabrication of suspended structures is known as the stiction, i.e., their permanent adhesion to the substrate when their surfaces come into contact. Indeed, the surface forces become more important as the dimensions shrink, as the surface over volume ratio increases. The collapse can occur while processing or during its use in humid environment.

The phenomenon is most of the time divided into two stages: first, a mechanical collapse mainly caused by the attractive capillary forces, and second, a permanent adhesion to the substrate caused by the van der Waals forces, the electrostatic forces, the hydrogen bridging, or the solid bridging. If the surface functionality of the polysilicon beams is transformed from hydrophilic to hydrophobic, the capillary forces would become repulsive and the collapse would not occur [1]. In that way both process-linked and in-use stiction are avoided.

In order to get hydrophobic surfaces, it has already been proposed to coat silane monolayers [2]–[4]. This technique has the advantage to reduce wear and friction in microengines [5]. Main research in that field focuses on aliphatic chloro-silanes, such as octadecyltrichlorosilane (OTS) and 1H,1H,2H,2H - perfluorodecyltrichlorosilane (FDTS). As with any chloro- silane based SAM precursor molecule, the first step in the reaction sequence for binding the molecule to the substrate is the hydrolysis of the Si-Cl bonds, one equivalent HCl is created for each Si-Cl bond that is hydrolysed. The presence of HCl in the SAM coating solution might be problematic if metal compounds, as aluminum interconnects, are present [6]. Furthermore, those kind of SAM are extremely sensitive to humidity and the quality of the film significantly depends on temperature and coating time, leading to reliability problems. Dialkyldichloromethylsilane (DDMS) was reported to have better chemical stability than monoalkyltrichlorosilane and simplified coating process [7]. To avoid some of the limitations imposed by the chlorosilane chemistry, alkene based monolayers were successfully coated as anti-stiction layers [6].

Also, we developed here a novel wet-release CMOS compatible process for the fabrication of surface micromachined beam using a perfluorated SAM. The wet release technology was preferred because of its simplicity and low cost. The release sequence was successfully applied to fabricate electrostatic variable capacitors used in the RF field.

2 EXPERIMENTAL PROCEDURE

As aluminum interconnects should not be etched, the etch of silicon dioxide sacrificial layer is performed by concentrated (73 %) hydrofluoric acid. Indeed in HF solutions the aluminum is attacked via

$$2Al + 6H_3O^+ \rightarrow 3Al^{3+} + H_2 \uparrow + 3H_2O \qquad (1)$$
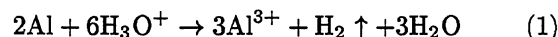
The low H_3O^+ concentration avoids this reaction, increasing the selectivity SiO_2/Al. In order to get a better control on the etch rate, HF can be diluted in isopropanol [8]. Water should be avoided for the subsequent rinses, preventing from reaction (1). The rinces

were so performed with isopropyl alchohol, which has the further advantage to be a low-tension fluid, decreasing the probability of stiction at that step.

Monolayer of (heptadeca fluoro-1,1,2,2,-tetrahydrodecyl) triethoxysilanes was used as anti-stiction coating. To allow the SAM to grow, the samples were first oxidized through immersion in H_2O_2. Hydrogen peroxide was then displaced to isopropanol. Next, wafers were put in a 1 : 600 mixture of the silane and dried isopropanol (through vacuum distillation on CaO) under argon environment for 20 min, 40 min, 60 min, 2 hours and 3 hours, respectively. Humidity should be avoided to prevent the silane from polymerization. The reaction is stopped by immersion in acetone and polymers which have not reacted are eliminated by a dichloromethane soxhlet extraction.

This complete release process was applied to 1 μm and 2 μm thick polysilicon beams. The polysilicon was deposited in a LPCVD oven at 625°C with a 90 sccm SiH_4 flow. Two thicknesses of the sacrificial PECVD silicon dioxide layer were used: 0.5 μm and 1 μm. In parallel to that beams, samples of bulk silicon and LPCVD polysilicon, PECVD SiO_2 and PEVCD silicon nitride deposited on bulk silicon wafer were prepared in order to measure their contact angle. Those samples followed the same process as the polysilicon beams but only from the oxidation step in H_2O_2. The bulk silicon references wafers were oxidized through immersion in the pirahna mixture (H_2SO_4:H_2O_2, 1 : 1) before the silanization.

In order to check reproducibility, the same experiment was done three times under the same conditions at several weeks of interval.

3 PROCESS CHARACTERIZATION

In two of the three experiments, monolayers where obtained after 20 min in the silane/isopropanol mixture, as confirmed by both contact angle and X-ray reflectivity measurements. In the last experiment, the measured contact angle was about 87° for the samples let 20 and 40 min in the silane solution, and about 111.8° for the others. X-ray reflectivity measurements on reference wafers showed that in each case a uniform monolayer of 9 Å is obtained (Fig. 1), which is in agreement with the chain length of precursor molecule.

3.1 Contact Angle Measurements

After the coating, a high degree of hydrophobicity was obtained. We measured the static contact angle by deposition of a 0.5 μl ultra pure water droplet on 10 different locations on each sample. Mean values are given in the following. No real dependance on silanization time was observed. For instance, the contact angle for polysilicon is kept between 117.1° and 119.4° for silanization time between 20 min and 3 hours.
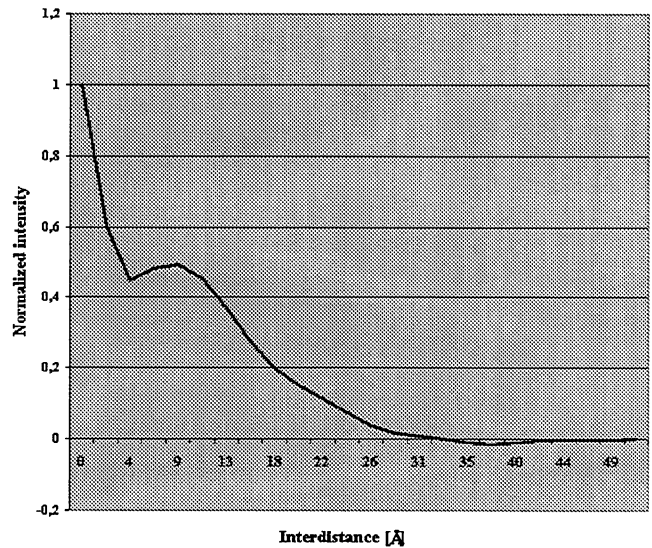

Figure 1: Patterson function from the X-ray measurements of the SAM.

Table 1: Contact angle of various materials.

Silicon	Polysilicon	PECVD SiO_2	PECVD Si_3N_4
117.3°	118.2°	109.7°	108.2°

Values for the bulk silicon wafers between 111.8° and 117.0° were found in mean for each of the three experiments. A maximum contact angle of 121.6° was found after three hours in the silane solution for one of the three experiments. The lack of reproducibility may be linked to the difference of ambient (temperature, humidity) between the experiments. The contact angle of the considered materials after 20 min silanization are found in Table 1. Note that without the SAM, all those materials exhibit contact angles close to 10° (value for the oxide layer).

3.2 Temperature Dependance

The last processing step of any microsystems is the packaging. Any layer deposited before this step must satisfy the thermal budget requirements of the packaging. The thermal stability of the SAM was determined by exposing it at high temperatures during at least one hour in nitrogen ambient. Experimental results showed that hydrophobicity is maintained up to 300°c (Fig. 2). X-ray reflectivity confirmed the presence of the monolayer after the annealing.

3.3 Micro-beams fabrication

As already mentioned, this release process was used to fabricate micro-beams of various lengths. One and two microns thick, up to 540 μm long cantilevers and
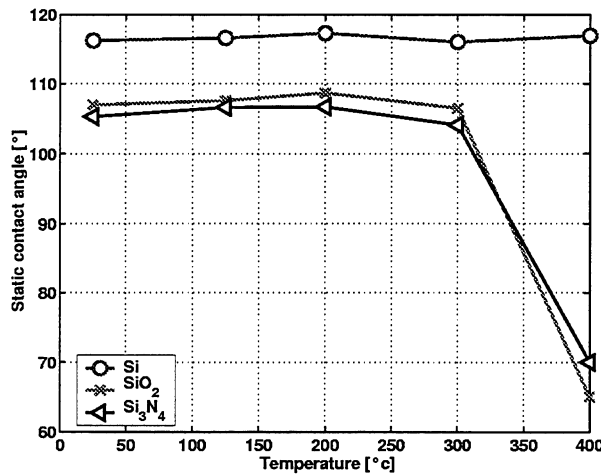
Figure 2: Temperature dependance of the contact angle for various silanized materials.

clamped-clamped beams on a 0.5 μm SiO$_2$ sacrificial layer were successfully released (Fig. 3). Without SAM, even for beams as short as 45 μm, stiction problems occurs. We extracted a work of adhesion W equal to $W = 1.75$ μJm^{-2} for the coated beams using the following equation [9] :

$$W \quad = \quad \frac{3\tilde{E}h^2t^3}{8l_d^4} \qquad (2)$$

where $\tilde{E}$ is the effective elastic modulus, h the gap spacing, t the beam thickness, and l_d the detachment length.

Furthermore, a complete 1 μm thick (gap 0.5 μm) polysilicon RF MEMS capacitor with aluminium interconnects realized with this process demonstrated the CMOS compatibility (Fig. 4).

4 CONCLUSIONS

A CMOS fully compatible release process for MEMS was proposed. Attack of aluminum interconnects is avoided by the use of concentrated HF and a fluoro-ethoxysilane as anti-stiction layer. This monolayer was characterized by X-ray reflectivity. The contact angle measurements showed a high degree of hydrophobicity and thermal stability up to 400°c even if the SAM is partially destroyed. Reproducibility of the experiment is nevertheless still questionable as the several experiments did not give exactly the same results. However, in each case, high hydrophobicity and stable monolayer were observed; this process can so be used for the aimed application.

One micron thick polysilicon beams on a 0.5 μm gap and a complete MEMS variable capacitor were properly released. We noted in this case a detachment length of 540 μm.
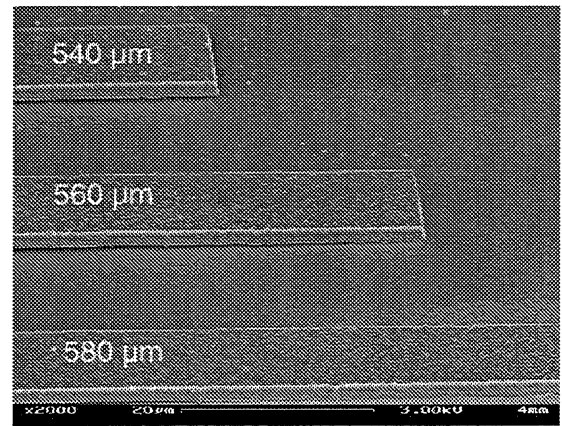
(a)
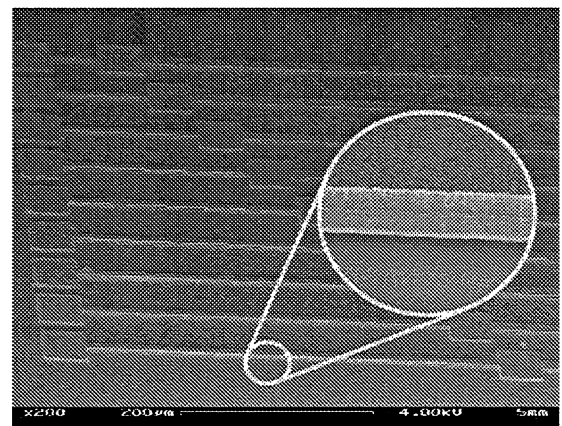
(b)

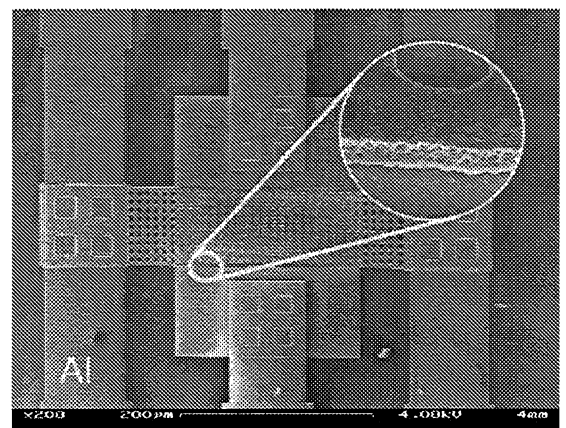Figure 3: Cantilever (a) and clamped-clamped (b) beams released with the proposed process.

Figure 4: Top view of a polysilicon MEMS capacitor on a 0.5 μm gap and its aluminum interconnects.

5 ACKNOWLEDGEMENTS

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