

Investigation of attractive forces between PECVD silicon nitride microstructures and an oxidized silicon substrate

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Abstract

A troublesome phenomenon encountered during the realization of free-standing microstructures, for example, beams, diaphragms and micromotors, is that initially released structures afterwards stick to the substrate. This effect may occur during wafer drying after the etching process has been completed, as well as during normal operation as soon as released structures come into contact with the substrate. In this paper the most important types of attractive forces are discussed with respect to their possible influence on the performance of micromachined structures. It is concluded that the main reason for sticking of PECVD silicon nitride micromachined structures is adsorption of water molecules. The water molecules, adsorbed on both surfaces, attract each other as soon as the surfaces come into contact. It is shown that a chemical surface modification, in order to achieve hydrophobic surfaces, is an effective method for avoiding adsorption of water, and therefore reduces sticking. Sticking of micromachined structures during drying is reduced by rinsing with a non-polar liquid before wafer drying.

1. Introduction

Silicon micromachining has become an important tool for the fabrication of many types of mechanical sensors and actuators. Examples are a recently developed capacitive pressure sensor and electret microphone [1]. At the University of Twente a condenser microphone with a silicon nitride diaphragm is being developed. The movable diaphragm will be fabricated by means of sacrificial layer etching. This process, which has also been used for the fabrication of beams [2] and micromotors [3], consists of the following successive steps. First, the so-called sacrificial layer is deposited (evaporated aluminium) and patterned, followed by the diaphragm material deposition (plasma-enhanced chemical vapour deposition (PECVD) of silicon nitride). Small access-holes are etched in the diaphragm material. The movable diaphragm is formed by sacrificial layer etching via the access-holes. The final step is drying the wafer with the diaphragms.

Figure 1 shows a schematic cross section of a diaphragm after the etching of the sacrificial layer

has been completed. After rinsing with water or other liquids in order to remove the etchant, the wafer is still covered with liquid. When the liquid above the diaphragm has evaporated, only the cavity under the diaphragm is filled with liquid. This liquid evaporates through the access-holes in the diaphragm. Adhesive forces between the shrinking liquid and the thin diaphragm pull the diaphragm towards the substrate. It has been observed that, after all the liquid has disappeared, diaphragms may stick permanently to the underly-

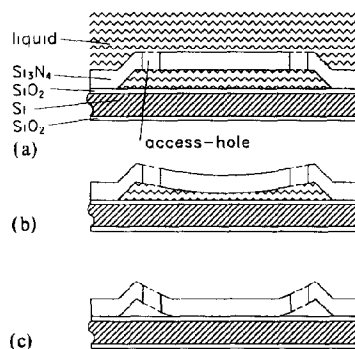


Fig. 1 The drying process of diaphragms fabricated by means of sacrificial layer etching: (a) after completion of sacrificial layer etching, (b) only the air gap filled with liquid, (c) the diaphragm stuck to the substrate.

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ing substrate material. Another effect that has been observed is that diaphragms, which have been dried without sticking, may stick to the substrate as soon as the surfaces come into contact with each other. This may occur during normal operation due to (sound) pressure overload. Both effects have also been described in the literature for other structures [2–5].

It is clear that an attractive force is present between the diaphragms and the substrate, with a magnitude inversely proportional to their separation. During drying of the wafer, the evaporating liquid causes the diaphragms to contact the substrate. The attractive force causes the diaphragms to remain stuck to the substrate.

Linder and de Rooij [4] investigated sticking of doubly clamped polysilicon microbeams. They used APCVD silicon dioxide as a sacrificial layer. By applying a voltage between the beams and the silicon wafer, stuck beams could be released from the substrate. It was concluded that sticking was caused by an electrostatic force.

Mehregany *et al.* [3] fabricated polysilicon micromotors using silicon dioxide as sacrificial layer. They found that sticking occurred as soon as native oxide was formed on the silicon substrate and the polysilicon rotors. It was assumed that because of the insulating native oxide layer, an electrostatic force would cause the sticking. Guckel *et al.* [2] suggested that sticking was caused by the presence of an etch residue. Lober and Howe [5] observed that water condensation between microstructures and the substrate may cause sticking.

A technique to dry micromachined structures successfully has been developed by Guckel *et al.* [2]. After completion of sacrificial layer etching, the wafer is rinsed with a water/methanol mixture. By applying a very rapid evaporation in a vacuum chamber, the liquid freezes. Under carefully chosen conditions, the ice does not melt, but sublimates. In this way the presence of liquid, and thus sticking during drying, is avoided. Furthermore, the authors report that the attractive force causing sticking is eliminated by covering the substrate and the microstructures with LPCVD silicon nitride.

It can be concluded that no literature exists about sticking of PECVD silicon nitride microstructures. The objective of this paper is to investigate systematically which type of attractive

force causes sticking of released PECVD silicon nitride structures. Silicon nitride cantilever beams and square diaphragms will be used as test structures and will be described in Section 2. In Section 3, several attractive forces that may cause sticking will be considered. In Section 4, surface treatments to improve fabrication and operation will be presented.

2. Test structures and technology

Besides diaphragms, silicon nitride cantilever beams have been used as test structures in order to study mechanical effects. The cantilever beams and diaphragms have been realized on the same 2 inch p-type 5–10 Ω cm <100> silicon wafers. First, the wafers were provided with thermally grown SiO_2 , with a thickness of 2100 Å. As a sacrificial layer, 1 μm aluminium was evaporated. After patterning the sacrificial layer, 1 μm PECVD silicon nitride was grown for use as the beam and diaphragm material. The conditions of the PECVD process are shown in Table 1. Diaphragm and beam structures were realized by reactive ion etching of the silicon nitride layer, followed by wet etching with a $\text{H}_3\text{PO}_4/\text{CH}_3\text{COOH}/\text{HNO}_3/\text{H}_2\text{O}$ 80/5/5/10 mixture at a temperature of 50 °C. The wafers were dried in normal environmental air.

The lateral dimension of the diaphragms was 200 μm . Cantilever beams have been made with a length of 100, 200, 300 and 400 μm and a width of 20 and 50 μm , resulting in eight different types of beams. The mask layout of a test structure containing 16 cantilever beams is shown in Fig. 2.

The magnitude of the attractive force per unit area required for sticking of cantilever beams can be estimated by calculating the force per unit area, P , which causes a tip deflection y of the cantilever beam. This force per unit area is equal to

$$P = \frac{2Eh^3}{3L^4} y \quad (1)$$

TABLE 1 The deposition conditions of the PECVD silicon nitride

| | |
|--------------------------------|-------------------------------------|
| Reactor | parallel plate (Electrotech PF 310) |
| Temperature | 300 °C |
| Pressure | 650 mTorr |
| Power | 20 W |
| Frequency | 13.56 MHz |
| SiH_4/N_2 (2%) | 2000 sccm |
| NH_3 | 10 sccm |

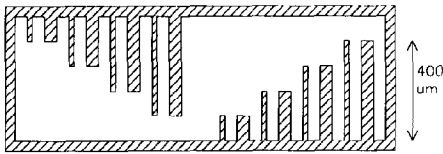


Fig 2 Mask layout of a testing structure, containing a total number of 16 cantilever beams with lengths of 100–400 μm and a width of 20 or 50 μm

where E is Young's modulus of the beam material, h is the beam thickness and L is the length of the beam. Assuming the tip deflection y to be equal to the distance between the beam and substrate, P can be calculated for different values of L , using $E = 10^{11} \text{ N/m}^2$ for silicon nitride, y as 1 μm and h as 1 μm . The theoretical results are shown in Table 2. As can be seen from the SEM photograph in Fig 3, free-standing beams show an upward curvature. Thus the air gap is larger than 1 μm and therefore the force per unit area required to cause sticking will be higher than the values of Table 2.

TABLE 2 The minimal force per unit area required to cause sticking of cantilever beams, calculated for different cantilever beam lengths. Young's modulus is 10^{11} N/m^2 , beam thickness is 1 μm and distance between beam and substrate is 1 μm

| Cantilever beam length (μm) | Force per unit area (N/m^2) |
|--|--|
| 100 | 670 |
| 200 | 42 |
| 300 | 8 |
| 400 | 3 |

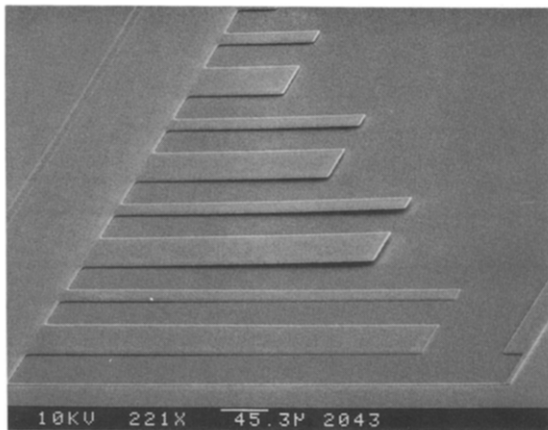


Fig 3 SEM photograph of stuck and released cantilever beams. The released beams all show an upward curvature due to an internal bending moment in the beam material.

3. Investigation of attractive forces

Sticking of cantilever beams may be caused by an internal bending moment in the beam material, which is a mechanical effect, or by different types of attractive forces. The mechanical effect will be explained and investigated in Section 3.1. The most important types of attractive forces that may cause sticking of cantilever beams will be discussed in Sections 3.2–3.4.

3.1 Internal bending moment

Free-standing cantilever beams have an average stress of zero, because they are clamped at only one side. Note that the average stress is also zero if the beams are made of silicon nitride with tensile stress. A non-uniform distribution of the stress across the thickness of the nitride layer always causes an internal bending moment, even in the case of zero average stress. This moment causes cantilever beams to bend, in contrast with beams that are clamped at two sides. An internal bending moment may cause the cantilever beams to bend downward, thus keeping the beams stuck to the substrate.

To investigate if sticking of the cantilever beams is caused by an internal bending moment, stuck beams have been released from the substrate by means of a small probe. As can be seen from the SEM photograph in Fig 3, released beams all showed an upward curvature, indicating the presence of an internal bending moment, but of opposite polarity to that required for the cantilever beams to stick. Therefore, it can be concluded that the presence of an internal bending moment is not the reason why the beams stick to the substrate.

3.2 Electrostatic forces

If the silicon nitride microstructure or the substrate contains a certain amount of fixed electric charge, an electric field will exist in the air gap between the beams or diaphragms and the substrate. For two parallel plates, the attractive force per unit area, P , is

$$P = \frac{1}{2} \epsilon_0 E_a^2 \quad (2)$$

where ϵ_0 is the permittivity of the air gap and E_a is the constant electric field strength between both plates. The force per unit area, P , can be made equal to zero if $E_a = 0$, which can be achieved by an external field that is opposite to the field caused

by the built-in charges. This method has been applied successfully by Linder and de Rooij [4].

The external voltage that is required to achieve a zero internal field can be estimated by considering a simplified representation of the structure connected to an external voltage source V_u , as shown in Fig. 4. The substrate is assumed to be conducting, and is covered with an SiO_2 layer that contains a surface charge per unit area σ_{ox} . Furthermore it is assumed that the silicon nitride diaphragm contains a surface charge per unit area σ_n . The capacitances per unit area of the air gap, the SiO_2 and the silicon nitride layer are C_a , C_{ox} and C_n , respectively. The charge present on the plates of C_a determines the electric field in the air gap, E_a . Using Gauss's law, the electric field strengths in the silicon nitride, E_n , the air gap, E_a , and the SiO_2 , E_{ox} , can be calculated as a function of the charges σ_n and σ_{ox} . By eliminating E_n and E_{ox} and applying $\oint E_s ds = 0$, it can be shown that

$$E_a = \frac{C_a C_i}{\epsilon_0 (C_i + C_a)} \left[V_u + \frac{\sigma_n}{C_n} - \frac{\sigma_{\text{ox}}}{C_{\text{ox}}} \right] \quad (3)$$

where ϵ_0 is the permittivity of vacuum and

$$C_i = \frac{C_{\text{ox}} C_n}{C_{\text{ox}} + C_n} \quad (4)$$

It can be seen from eqn (3) that E_a is equal to zero for

$$V_u = \sigma_{\text{ox}}/C_{\text{ox}} - \sigma_n/C_n$$

$|V_u(E_a = 0)|$ will be maximum if σ_{ox} and σ_n have opposite signs, and is given by

$$|V_u(E_a = 0)| = \left| \frac{\sigma_{\text{ox}}}{C_{\text{ox}}} \right| + \left| \frac{\sigma_n}{C_n} \right| \quad (5)$$

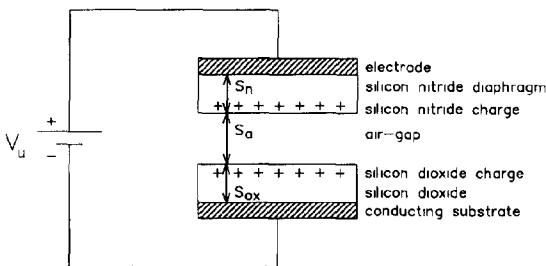


Fig. 4 Schematic representation of a free-standing silicon nitride diaphragm provided with an electrode, and a conducting substrate covered with a silicon dioxide layer. The upper electrode and the substrate are connected by an external voltage source V_u .

A charged insulator can be characterized by its built-in voltage V , which is related to the surface charge per unit area, σ . For the SiO_2 and Si_3N_4 layers the voltages are given by

$$V_{\text{ox}} = \frac{\sigma_{\text{ox}} s_{\text{ox}}}{\epsilon_0 \epsilon_{\text{ox}}} = \frac{\sigma_{\text{ox}}}{C_{\text{ox}}} \quad (6a)$$

$$V_n = \frac{\sigma_n s_n}{\epsilon_0 \epsilon_n} = \frac{\sigma_n}{C_n} \quad (6b)$$

where s_{ox} , s_n and ϵ_{ox} , ϵ_n are the thicknesses and the relative dielectric constants of SiO_2 and Si_3N_4 , respectively, and $\epsilon_0 \epsilon_{\text{ox}}/s_{\text{ox}}$, $\epsilon_0 \epsilon_n/s_n$ are equal to the SiO_2 and Si_3N_4 capacitances per unit area, C_{ox} and C_n . The voltages V_{ox} and V_n can easily be measured with a vibrating reed electrostatic voltmeter [6]. However, measurements with a Monroe Iso-probe 244 electrostatic voltmeter have shown that the built-in voltages of SiO_2 and Si_3N_4 layers, fabricated as described in Section 2, were both below the detection threshold of this instrument, which is 1 V. Thus $|V_{\text{ox}}| < 1$ V and $|V_n| < 1$ V. Assuming V_{ox} and V_n to be equal to 1 V, an upper limit for the value of $|V_u(E_a = 0)|$ can be calculated using eqns (6a), (6b) and (5). The upper limit for $|V_u(E_i = 0)|$ is 2 V, so $|V_u(E_i = 0)| \leq 2$ V.

The internal field compensation test has been carried out with $200 \mu\text{m} \times 200 \mu\text{m}$ silicon nitride diaphragms, provided with aluminium electrodes. Using a Hewlett Packard 4145 B parameter analyser, the external voltage was swept between -30 and $+30$ V using steps of 100 mV. It was not possible to release stuck diaphragms this way. Using higher voltages it was observed that the contacting area between the diaphragms and the substrate increased. This indicates that the attractive force, caused by the externally applied electrostatic field, increases. Therefore it is not to be expected that voltages of more than 30 V will release stuck diaphragms.

According to eqn (3), an electrostatic attraction can always be eliminated by compensating the internal electric field. However, by sweeping the external voltage in a theoretically predicted range it has not been possible to release stuck diaphragms. Considering these results, it can be concluded that, in our case, sticking is not caused by electrostatic attraction. Note that some built-in charge may have been present in the diaphragm material or the silicon dioxide layer, but this is not the main reason for sticking.

3.3 Van der Waals forces

The attraction between neutral atoms or non-polar molecules, with a mutual separation too large to cause their electron clouds to overlap, is called the van der Waals force. Its origin is that atoms or molecules without a static dipole moment exhibit a fluctuating dipole moment, due to the fact that electrons are not able to screen the charge of the atomic nucleus in all directions at one and the same moment because of their motion.

As a consequence of the van der Waals attraction between atoms, this type of attraction is also present between macroscopic bodies. The force between macroscopic bodies can be calculated by summation of the forces between individual atoms or molecules of the bodies. By means of this microscopic theory, it has been shown that the attractive force per unit area, P , between two flat, infinite plates is [7]

$$P = A/6\pi D^3 \quad (7)$$

where D is the distance between the plates and A is a so-called Hamaker constant. Equation (7) is only valid for distances less than about 10 nm. These short-range van der Waals forces are called normal van der Waals forces.

As the distance between the plates becomes larger, the attractive force per unit area can be written as [7]

$$P = B/D^4 \quad (8)$$

where B is a Hamaker constant for long-range van der Waals attraction. These long-range van der Waals forces are called retarded van der Waals forces.

Using eqns (7) and (8), the van der Waals force per unit area can be calculated as a function of the mutual distance between two flat plates. Figure 5 shows the calculated force per unit area for SiO_2 plates, using $A = 5.4 \times 10^{-20} \text{ J}$ and $B = 10^{-28} \text{ J m}$ [8]. Values for the Hamaker constants of PECVD silicon nitride are not available.

Comparing the results of Table 2 and Fig. 5, theoretically the van der Waals force between the free-standing beams and the substrate can become strong enough to cause sticking if their mutual separation is less than about 10–100 nm, of course depending on the length of the beams. It will be clear that the effective distance between the microstructures and the substrate is determined by

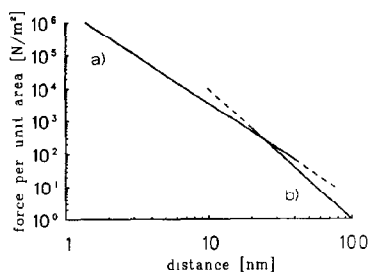


Fig. 5 The van der Waals force per unit area between two flat, infinite plates as a function of distance. The calculation is based on literature values for SiO_2 ($A = 5.4 \times 10^{-20} \text{ J}$ and $B = 10^{-28} \text{ J m}$) [6] (a) normal forces (eqn (7)) (b) retarded forces (eqn (8))

their surface roughness, which will result in such a large uncertainty in the measured van der Waals force that these measurements will not be reliable and are therefore not performed. Therefore it can only be concluded that the van der Waals force is theoretically able to cause sticking.

3.4 Water adsorption

If an SiO_2 or Si_3N_4 surface is surrounded by humid air, water molecules are absorbed to the surface. Adsorption can be divided into chemisorption and physisorption. Chemisorbed water molecules are chemically bonded to the surface as hydroxyl ($-\text{OH}$) groups. Physisorbed water molecules are hydrogen bonded to the hydroxyl groups. This is shown schematically in Fig. 6(a). If two surfaces are contacted, an attraction may occur between the adsorbed water molecules. This is shown schematically in Fig. 6(b). Stengl *et al* [9] have shown that two oxidized silicon wafers bond as soon as they touch because

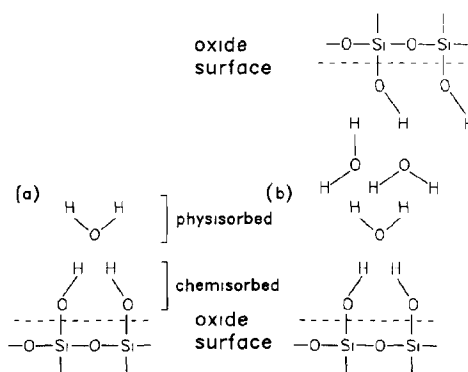


Fig. 6 (a) SiO_2 surface after adsorption of water (b) Two SiO_2 surfaces kept together by hydrogen bonding between physisorbed water molecules as proposed by Stengl *et al* [7] — = chemical bond — = hydrogen bond

of this mechanism. They calculated a surface energy of 104 mJ/m^2 for the bonded wafers. The bond between bonded wafers is very strong. Values of $2\text{--}5 \times 10^5 \text{ N/m}^2$ have been measured by Shimbo *et al.* [10]. Therefore this mechanism is expected to be a possible cause for sticking.

If sticking of free-standing silicon nitride microstructures to the oxidized silicon substrate is caused by water adsorption, the sticking would be correlated with the presence of adsorbed water molecules. The adsorption of water on SiO_2 surfaces can be described by the BET adsorption theory, which predicts that only a certain fraction R_c of the total surface is covered with one or more monolayers of physically adsorbed water molecules. This fraction can be written as a function of the relative humidity of the surrounding air [11]

$$R_c = \frac{Ch}{1 + (C - 1)h} \quad (9)$$

where h is the relative humidity and C is a dimensionless constant, which is approximately 0.04 for SiO_2 [11]. It can be concluded from eqn (9) that adsorbed water molecules can be removed from the surface ($R_c = 0$) by making the relative humidity equal to zero.

A zero relative humidity has been realized by placing the samples in a continuous flow of dry nitrogen. Adsorption of water molecules occurred when a test sample was exposed to room air with a relative humidity of 40–50%. Three test samples, each containing five test structures as shown in Fig. 2, with initially stuck beams have been used for this experiment. The cantilever beams have first been released from the substrate with a small probe. After this release the test samples were placed in a dry (nitrogen) or a humid (air) environment for a period of 3–4 h to enable the adsorption or desorption process to reach an equilibrium situation. The attractive forces in a dry nitrogen ambient and in a humid air environment have been compared by contacting the beams with the substrate. This has been done by pushing on top of the beams with the probe and pressing the beams and substrate together. After each test, the number of beams that remained stuck to the substrate was counted.

The experiment was repeated alternately in humid air ($h = 40\text{--}50\%$) and dry nitrogen. The resulting sticking ratios are shown in Table 3. The

TABLE 3 The average sticking ratio of silicon nitride cantilever beams in humid air (relative humidity 40–50%) and dry nitrogen as a function of the beam length. Beam thickness is $1 \mu\text{m}$. All samples contained 20 beams of each length

| Beam length (μm) | Sticking ratio | |
|----------------------------------|----------------|--------------|
| | Humid air | Dry nitrogen |
| 400 | 0.63 | 0.19 |
| 300 | 0.41 | 0.05 |
| 200 | 0.11 | 0.01 |
| 100 | 0 | 0 |

sticking ratio is defined as

$$\text{sticking ratio} = \frac{\text{number of stuck beams}}{\text{total number of beams}}$$

The values from Table 3 are the average sticking ratios of the three test samples. It can be observed from Table 3 that the sticking ratio decreases in dry nitrogen, and increases with increasing beam length. This is expected, because less force per unit area is required to deflect longer cantilever beams, as calculated in Table 2. Furthermore it was observed that the sticking ratio was a reversible function of the relative humidity.

Thus a correlation between the attractive force and the presence of adsorbed water molecules is obviously present. It can be concluded that adsorption of water molecules on the beams and the substrate is a main factor for sticking under normal environmental circumstances. Another force also seems to contribute to the sticking, because in a dry nitrogen atmosphere some beams are always able to stick to the substrate. However, this second force is of minor importance.

4. Surface treatments

In Section 3 it was shown that sticking of free-standing microstructures is mainly caused by the presence of water molecules adsorbed on the surfaces. It is very likely that sticking during drying of microstructures is due to the same effect. To produce free-standing microstructures, which can operate in a normal (humid) environment, both sticking during drying and during normal operation should be avoided. Therefore, a treatment to avoid sticking during drying of the microstructures

and a surface treatment to avoid adsorption of water afterwards will be discussed here

4.1 Rinse procedures

During drying of micromachined structures, the withdrawing liquid film between the structures and the substrate pulls the surfaces together as soon as only the air gap under the microstructures is filled with liquid. Once the surfaces touch, they are kept together by the attractive forces between adsorbed water molecules. Sticking during drying of the microstructures may be reduced or even eliminated if the adhesive force between the withdrawing liquid and the microstructures or the substrate is minimized. The force, caused by the liquid, is a function of the surface tension of the liquid and the contact angle between the liquid and the surface. In general, a lower surface tension will result in a smaller force. Furthermore, rinsing with another liquid will remove water from the air gap between the beams and the substrate. Therefore it is expected that rinsing with a liquid with a lower surface tension after completion of sacrificial layer etching will reduce the sticking ratio.

The following rinse cycles have been tested after completion of the sacrificial layer etching. First, rinsing with deionized (DI) water only. Next, rinsing with DI water and ethanol and, finally, rinsing with DI water, ethanol and n-hexane. Rinsing with ethanol before rinsing with n-hexane is necessary, because (non-polar) n-hexane cannot be mixed with (polar) water. After the samples have been dried in normal environmental air, the number of stuck beams is counted. Table 4 shows the results.

As can be seen from Table 4, rinsing with a non-polar liquid reduces the number of stuck beams.

4.2 Hydrophobic surface modification

Free-standing micromachined structures may stick as they come into contact with the substrate

TABLE 4 The average sticking ratio as a function of the length of the cantilever beams after different rinse cycles. All samples contained 20 beams of each length.

| Beam length (μm) | DI water | DI water, ethanol | DI water, ethanol n-hexane |
|----------------------------------|----------|----------------------|----------------------------------|
| 400 | 1.0 | 0.98 | 0.82 |
| 300 | 1.0 | 0.95 | 0.68 |
| 200 | 1.0 | 0.93 | 0.28 |
| 100 | 1.0 | 0.95 | 0 |

during normal operation due to the presence of adsorbed water molecules from environmental humid air. To avoid adsorption of water molecules on the surface, it should be hydrophobic. One method to replace polar hydroxyl groups by non-polar groups is a chemical surface modification with hexamethyldisilazane (HMDS) [12]. The $-\text{OH}$ groups on the silicon dioxide or silicon nitride surface are then replaced by non-polar $-\text{O}-\text{Si}(\text{CH}_3)_3$ groups.

Samples with cantilever beams, which have been released with a probe, have been used to investigate the effect of a vapour phase HMDS treatment. All samples contained 96 cantilever beams. Two samples were left untreated and two samples were treated with HMDS vapour at 120°C for 30 min. The attractive force between the beams and substrate has been tested in humid air by deliberately contacting the surfaces with a probe, as explained in Section 3.4. The average sticking ratios are shown in Table 5.

Too many of the $100\text{ }\mu\text{m}$ long beams were damaged during release from the substrate. Therefore no conclusions have been drawn from the results of these beams. It can be observed from Table 5 that the sticking ratio of the $200\text{ }\mu\text{m}$ long beams is significantly reduced by the HMDS treatment. The sticking ratio of the 300 and $400\text{ }\mu\text{m}$ long beams shows no significant decrease. Furthermore it was observed that the contact area between stuck beams and the substrate of the HMDS-treated beams was typically less than 10% of the total beam area. The contact area of the untreated beams was about 50% of the total beam area.

The sticking ratios from Table 5 for untreated beams in humid air are higher than the values shown in Table 3. This is because the beams used for testing the HMDS treatment were fabricated in another batch. The free-standing cantilever beams

TABLE 5 The average sticking ratio as a function of the beam length of untreated and HMDS-treated samples with released cantilever beams after deliberately contacting the beams with the substrate in normal environmental air ($h = 40-50\%$). All samples contained 24 beams of each length.

| Beam length (μm) | Sticking ratio | |
|----------------------------------|----------------|--------------|
| | Untreated | HMDS treated |
| 400 | 1.0 | 1.0 |
| 300 | 1.0 | 0.98 |
| 200 | 0.96 | 0.02 |

of this batch showed less upward curvature than the beams shown in Fig 3. Therefore these beams do stick more easily to the substrate, as explained in Section 2, and higher sticking ratios are expected.

As shown in Section 3.4, adsorption of water molecules is the main reason for sticking of PECVD silicon nitride cantilever beams. Modification of the hydrophilic surface into a hydrophobic one reduces water adsorption. The number of stuck beams with a length of 200 μm decreases significantly after HMDS treatment. The sticking ratio of the 300 and 400 μm long beams is not reduced significantly. In Table 2 it was shown that a smaller force per unit area is required to cause sticking of longer cantilever beams. Therefore it may be concluded that the attractive force between the beams and the substrate has been reduced by the hydrophobic surface modification, but is still able to cause sticking of the longer cantilever beams. The HMDS treatment is a useful way to reduce sticking of microstructures during normal operation.

5. Discussion and conclusions

A correlation between the sticking of initially released PECVD silicon nitride cantilever beams and the relative humidity of the surrounding air has been found. It can be concluded that adsorbed water molecules on the surfaces attract each other as soon as the surfaces are brought into contact. Stengl *et al.* [7] have found that water adsorption causes direct bonding of polished silicon wafers at room temperature. Apparently, the same mechanism is responsible for both phenomena.

It was not possible to reproduce the experiment of Linder and de Rooij [4] using PECVD silicon nitride diaphragms. Stuck diaphragms could not be released by applying a voltage between the diaphragm and the substrate. In our experiments the silicon under the diaphragm was undoped, and therefore some voltage drop will occur across the depletion region when the air-gap capacitor is reversely biased. However, this was not the reason why it was impossible to release the diaphragms, because the voltages have been investigated up to values higher than +30 or -30 V, where the attraction only increased, as explained in Section 3.2.

It was observed in Sections 3.4 and 4.2 that some of the untreated beams in a dry nitrogen

atmosphere, as well as the relatively long HMDS-treated beams, are able to stick to the substrate. Apparently, another force also contributes to the sticking. This may be van der Waals forces, as explained in Section 3.3, or electrostatic forces. However, the main reason for the sticking of PECVD silicon nitride cantilever beams is adsorption of water molecules, and the other forces are of minor importance.

The fabrication of PECVD silicon nitride cantilever beams has been improved by rinsing with non-polar n-hexane after completion of sacrificial layer etching. In order to avoid sticking of microstructures during normal operation, water adsorption should not occur. A hydrophobic surface modification with HMDS has been shown to reduce sticking.

Summarizing, it can be stated that the fabrication and operation of micromachined devices can be improved by applying the rinse procedures and surface treatments discussed in this paper.

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