Micromachined SiO$_2$ microcantilever for high sensitive moisture sensor

Qi Chen · Ji Fang · Hai-Feng Ji · Kody Varahramyan

Received: 29 April 2007 / Accepted: 25 November 2007 © Springer-Verlag 2007

Abstract Ultra-sensitive and selective moisture sensors are needed in various industries for processing control or environmental monitoring. As an outstanding sensor platform, surface-stress sensing microcantilevers have potential application in moisture detection. To enlarge the deflection of the microcantilever under surface stress induced by specific reactions, a new SiO$_2$ microcantilever is developed which features a much lower Young’s modulus than conventional Si or SiN$_x$ microcantilevers. For comparing SiO$_2$ cantilever with Si cantilevers, a model of the cantilever sensor is given by using both analysis and simulation, resulting in good agreement with the experimental data. The results demonstrate the SiO$_2$ cantilever can achieve a much higher sensitivity than the Si cantilever. In order to fabricate this device, a new fabrication process using isotropic combined with anisotropic dry etching to release the SiO$_2$ microcantilever beam by ICP (Inductively Coupled Plasma) was developed and investigated. This new process not only obtains a high etch rate at 9.1 µm/min, but also provides good profile controllability, and a flexibility of device design. Attributed to the high sensitivity, a significant deflection amplitude of the surface modified SiO$_2$ microcantilever was observed upon exposure to 1% relative humidity. The SiO$_2$ cantilevers are promising for inexpensive and highly sensitive moisture detection.

1 Introduction

In recent years, moisture sensors have been widely used for measurement and control in industrial or household environment. The sensing and control of moisture is very important in various areas and processes of industries such as chemical, petrochemical, food processing, agriculture, textile industries, etc., as the presence of moisture is highly undesirable and deteriorates the quality of the product. Different types of moisture sensors have been reported, such as capacitive humidity sensors (Nahar and Khanna 1998; Boisen et al. 2000; Kang and Wise 2000) resistive humidity sensors (Barkauskas 1997; Chou et al. 1999; Arshak and Twomey 2002), optical humidity sensors (Somani et al. 2001), hygrometric humidity sensors and gravimetric humidity sensors (Rittersma 2002). However, the moisture sensor, which has the capability to detect low relative humidity (RH) like 1% or lower than 1%, is rarely reported. Environmental detection of RH often needs to be performed at very low concentrations. Therefore, more efforts should be paid to develop the sensitivity, linearity, and stability of the moisture sensors.

Microcantilevers have proven to be an outstanding sensor platform for extremely sensitive chemical, biological and environmental sensors (Chabot and Moreland 2003; Thundat et al. 1997; Xu et al. 2002; Ji et al. 2001; Hansen et al. 2001; Wu et al. 2001; Ji and Thundat 2002; Belaubre et al. 2003; Fritz et al. 2000). In general, microcantilever technology has several advantages besides high sensitivity. The entire sensor could fit in an area less than a few millimeters. Because the cantilever bending signal is driven by molecular recognition, the only power required is for the detection and display, allowing use of light-weight battery power or photovoltaic cells (Oden et al. 1996). Electronics for operation and control are relatively simple and...
inexpensive. Microcantilever arrays could be used to record numerous properties, gain specificity and provide replication (Quist and Badia 2003). Therefore, developing a high sensitivity moisture sensor based on microcantilevers will have promising applications in the chemical petroleum and transportation industries.

Microcantilevers can undergo bending due to molecular adsorption or absorption, by confining the adsorption and absorption to one side of the cantilever where the sensing material is coated. Adsorption or interaction of the analyte will change the surface characteristics of the microcantilever or the film volume on the cantilever, and result in bending of the microcantilever. This concept has already been used to demonstrate the feasibility of chemical detection of a number of vapor phase analytes as well as highly sensitive detection of chemical and biological species in solutions. Most of these microcantilever sensors were made of silicon. Due to the relatively large Young’s modulus of silicon material, the bending response of the silicon microcantilever is too weak to be measured when surface-stress change is rather small. The silicon dioxide (SiO$_2$) microcantilever can provide a much higher mechanical sensitivity compared to its silicon or silicon–nitride counterparts, as thermally grown SiO$_2$ film features a much lower Young’s modulus than single-crystalline silicon or silicon nitride (Tang et al. 2004; Bao 2000). It can be clearly seen that (http://www.memsnet.org/material) lists Young’s modulus values of thermal SiO$_2$, single crystalline Si and LPCVD (low pressure chemical vapor deposition) Si$_3$N$_4$ as 57–79, 170 and 290–380 GPa, respectively. The high mechanical sensitivity leads to a large surface-stress-induced bending of the cantilever.

Many methods have been developed in the field of microcantilever fabrication in order to meet a wide range of specifications. These specifications include beam size, thickness, connectivity, beam protection, materials, alignment, and cost. Optimizing fabrication process usually needs careful evaluation of advantages and trade-offs. A key step of the fabrication process for cantilever sensors is release of the suspended microcantilever beam at the final stage of the fabrication process. Wet etching always gets involved in the fabrication of microcantilever structures for the releasing the cantilever beam from the substrate. However, surface tension-induced stiction during the release and drying step is a big challenge for the wet etching process. Although some preventive measures are taken to avoid this problem (Chatzandroulis et al. 2002), especially in the fabrication of SiO$_2$ microcantilevers due to the low etching selectivity of SiO$_2$: Si using either KOH or EDP etchants, the processing conditions are hard to control (Tang et al. 2004). Recently, we used a plasma anisotropic dry etching process to release the SiO$_2$ microcantilever beams, avoiding stiction of the released structure to the adjacent silicon substrate (Chatzandroulis et al. 2002) and the low SiO$_2$: Si etching selectivity in wet etching (Tang et al. 2004). To completely release the cantilever beam, the anisotropic dry etching must go through the 500-μm-thick sacrificial silicon from the backside of wafer. Common photoresists cannot be used as an etching mask. The etching mask material must be carefully selected. In our present work, we developed a dry isotropic plasma etching with ICP for front-sided releasing cantilever beam. Therefore, the entire fabrication process of SiO$_2$ cantilever was to divide the release process into three dry etching steps, the 3 μm thick photoresist 1813 can be an appropriate mask layer for releasing the cantilever beam.

In this article, we present a novel guard-armed SiO$_2$ cantilever sensor for high sensitive moisture detection. To compare the Si and SiO$_2$ cantilevers, the deflections of both microcantilever beams induced from uniform surface-stress are analyzed. The simulation results proved that the SiO$_2$ cantilever beam has a higher sensitivity compared to the Si beam. A comparison experiment between Si and SiO$_2$ microcantilevers was done by exposing both to the same concentration of aminoethanethiol solution. The experimental results also fit perfectly with the simulation results. The moisture sensor based on the surface modified SiO$_2$ cantilever beam showed a good response to 1% RH.

2 Device design and simulation

A schematic diagram of the SiO$_2$ microcantilever structure designed is shown in Fig. 1. The device consists of two adjacent SiO$_2$ cantilever beams, connecting wings on both sides, and three guard arms. The dimensions of the designed SiO$_2$ microcantilever beams are 250 μm in length, 100 μm in width, and 1 μm in thickness. The connecting wings were connected with the adjoining cantilever bodies after the beams were released, so that all the cantilevers were held on the substrate for further processing. The guard arm was designed to protect the microcantilever beam from damage by collision during separation and handling procedures.

In many cases, adsorption-induced bending of cantilever beams can be predicted by a model shown in Fig. 2a, in which the beam tip was under a uniformly distributed loading, $\sigma$. The unit of $\sigma$ is N/m, and $\sigma > 0$ is tensile. A concentrated moment $M$ applied at the cantilever beam’s free end can represent this surface-stress effect. Therefore, the radius of microcantilever curvature $R$ can be derived as

$$\frac{1}{R} = \frac{6\Delta\sigma(1-\nu)}{E\tau^2}$$

(1)
Here, $E$ and $v$ represent the cantilever beam’s Young’s modulus and the Poisson’s ratio, respectively. $t$ stands for the thickness of the cantilever beam. $\Delta \sigma$ represent differential surface stress, and $\Delta \sigma = \sigma^+ - \sigma^-$, $\sigma^+$ and $\sigma^-$ are surface stresses on the top and bottom surfaces of the cantilever beam, respectively.

In fact, the Eq. (1) is the famous Stoney’s formula. In terms of the radius of curvature $R$, the deflection at the end of the cantilever beam can be expressed as

$$y_{\text{max}} = \frac{L^2}{2R} = \frac{3L^2(1-v)}{E t^2} \Delta \sigma \quad (2)$$

Although Stoney’s formula serves as a cornerstone for curvature based analysis and a technique for the measurement of surface stress, it does not agree well with the experiment data for structures under large deflection or “thick” film/coating on the substrate (Klein 2000; Freund et al. 1999). In addition, the above model regards the surface stress as a moment applied at the beam’s free edges does not take into account the influence of the surface stress on structure stiffness. Thus, Zhang et al. (2004) provided a new model which is demonstrated in Fig. 2b. This model assumes an area stress $s$ uniformly distribute on the top surface of the beam. They modeled this as a uniformly distributed axial stress $sw$ along the beam’s neutral axis and a uniformly distributed bending moment $m$ along the beam. In terms of this, they derived the governing equation as

$$E I \frac{d^4 y}{dx^4} - sw(L-x) \frac{d^2 y}{dx^2} \frac{d^2 y}{dx^2} + sw \frac{dy}{dx} = 0 \quad (3)$$

and boundary conditions as

$$\begin{align*}
y(x)|_{x=0} &= 0 \\
dy \bigg|_{x=0} &= 0 \\
d^2 y \bigg|_{x=L} &= 0 \\
E I \frac{d^4 y}{dx^4} + m &= 0 \quad (4)
\end{align*}$$

This second model can solve the problems, which the first model cannot explain. A beam module ($L = 250 \mu m$, $w = 100 \mu m$, $t = 1 \mu m$) was built in CoventorWare (Coventer Inc., Cary, NC, USA) to compute the mechanic deformation induced by uniform surface stress through the finite element method (FEM). When applying boundary conditions to the meshed model, we divided the load force applied on the beam surface into a uniform concentrated force in each element, which is equivalent to the condition described in the aforementioned second model. If the cantilever is made of silicon dioxide, the Young’s modulus $E = 73.0$ GPa, the Poisson’s ratio $v = 0.17$, then the maximum deflection of the silicon dioxide beam appeared at the end of the beam with the value 164.9 nm. On the other hand, if the cantilever beam is made of silicon, the Young’s modulus $E = 169.0$ GPa, the Poisson’s ratio $v = 0.3$, then the maximum deflection of the cantilever beam is 6.96 nm. The simulation results of beam deflection are shown in Fig. 3. The SiO$_2$ beam can give a 2.37 times larger bending amplitude under the same conditions compared with the silicon beam. A higher sensitivity could be achieved through using the silicon dioxide instead of silicon as the cantilever beam.

### 3 Device fabrication

#### 3.1 Investigation of ICP isotropic plasma etching for beam release

In addition to conventional optical lithography, SiO$_2$ wet etching and ICP plasma dry etching, a new cantilever beam release processing was developed for the device fabrication. This process includes two steps. The cantilever beam was first patterned by anisotropic dry etching, followed by the isotropic plasma dry etching to release the cantilever beam completely. In the first step, anisotropic etching was applied to open a window and to ensure the fluorine...
The starting material was a commercially available (Silicon Inc.) double side polished 4-in. (100-mm), <100> silicon wafer with 500 μm thick and a 1 μm thick SiO₂ layer on both surfaces. To achieve the structure of microcantilever described previously, four masks were designed and applied. Among these, mask 2 was specifically designed for cantilever beam release by isotropic plasma dry etching combined with an anisotropic etching process in Fig. 4. Here, Fig. 4b depicts an enlarged part of the mask pattern in Fig. 1a and the related dimension of masks 1 and 2. The photoresist pattern of mask 2 served as an etching mask in the ICP etching process to release the microcantilever beam from bulk silicon. In order to protect the edges of the cantilever beam, the photoresist pattern covering on the cantilever beam was about 5 μm longer along the three edges than the SiO₂ beam. This also compensated for any slight alignment errors. To ensure that the cantilever beam released completely and the foot of the beam remained at the expected position after release, the photoresist pattern at the foot of beam was designed at 60 μm longer than in mask 1. Figure 5 illustrates the main steps of the fabrication process. The fabrication process is described as follows:

Fig. 3 The deflection contour of cantilever beam. a SiO₂, b Si radicals could react efficiently with the silicon underneath the SiO₂ beam during the isotropic plasma etching. The silicon isotropic etching procedure is modified from the standard Bosch process. In this modified process, the passivation step was dismissed, and SF₆ was the only process gas. Because no passivation layer was formed along the sidewall of the trench, the ions reacted freely with the silicon atoms under the mask horizontally. The isotropic etching was realized largely by ion-enhanced chemical etching.

Dry etching with ICP has been extensively studied for the fabrication of high aspect ratio micromechanical structures. Among the available process schemes and recipes, the Bosch process (Laerm and Schilp 1996; Laerm et al. 1999) has made high aspect ratio silicon structures with vertical sidewalls possible (Quev et al. 2002). The standard Bosch process is an anisotropic etching procedure and works as follows. It starts with a shallow etching step under a low pressure SF₆ plasma generated with an RF source, fluorine radicals generated in SF₆ plasma then react with silicon to form volatile SiF₄, and continues with a passivation step enhanced via an ICP source, where CₓFₓ gas molecules are dissociated to form a polymeric layer over the exposed silicon surface. During the subsequent etch step, this layer is preferentially withdrawn on the horizontal surface rather than on the sidewalls because of ionic physical etching. Both steps repeat until complete etching of the silicon layer is accomplished. Experiments and optimization settings for anisotropic plasma etching with ICP system have been reported (Aachboun and Ranson 1999; Aachboun et al. 2000).

To take advantage of high etching rate with ICP, we investigated isotropic plasma etching for the release of suspended structure from a silicon substrate. A high-density ICP system (Alcatel 601E) was used to conduct deep dry plasma etching investigation. We have applied different isotropic etching process recipes to 16 processed wafers for 10 min, respectively, Table 1 shows the processing parameters and the sample profiles. Pure SF₆ is the only process gas in the silicon isotropic etching. The etch rate, undercut rate, and isotropic ratio have been taken into consideration to obtain an optimal set of plasma etching parameters. In fact, for the isotropic etch, we hope to achieve higher etch rates as well as better isotropic ratios at the same time. Therefore, recipe 13 in Table 1 (46 mTorr pressure, 1,800 W source power, substrate power 20 W, 300 sccm SF₆ flow rate, and 20°C chuck temperature) was adopted as the best one. For this recipe, the etching rate was about 9.1 μm/min, uniformity was 92% and isotropic ratio was 66%. Using this process, the cantilever beam was successfully released from the substrate.
3.2.1 Pattern the SiO\textsubscript{2} cantilever beam

Shipley 1813 positive tone photoresist was spun on one surface of a silicon wafer. A microcantilever beam pattern was transferred with mask 1 to the photoresist layer on the front side of the wafer by a standard photolithography process and then the SiO\textsubscript{2} cantilever shapes were defined by wet etching with Buffered Oxidation Etchant (BOE HF-N\textsubscript{2}H\textsubscript{4}F = 1:6 in volume). In the meantime, the entire SiO\textsubscript{2} layer on the backside was etched off. The photoresist was then cleaned by acetone and DI water (Fig. 5a).

3.2.2 Wafer backside etching 1: pattern cantilever base with guard arm

A 3 \textmu m thick 1813 photoresist layer was spun on the backside of the wafer and then patterned with a...
photolithography process to form the base of the cantilever sensor. The photoresist pattern served as a mask for ICP plasma etching to create the guard arm in the next fabrication step. The wafer was etched by about 70 μm by the ICP anisotropic etching process (Fig. 5b). The etching depth of about 70 μm determined the thickness of the connecting arm. This etched area will be completely opened through the wafer after releasing the cantilever beam.

3.2.3 Wafer backside etching 2: pattern cantilever connecting wing and guard arm

A 3 μm layer of photoresist 1813 was spun on the backside of the wafer again and then plasma anisotropic etching applied to etch about 260 μm. This etching step is also applied to etch the connecting wings, guard arms, and open window from the backside of the substrate (Fig. 5c).

3.2.4 Cantilever beam release

A new process, which involves two steps, was developed to release the SiO$_2$ cantilever beam. The cantilever beam was first patterned by anisotropic dry etching and followed by the isotropic plasma dry etching to completely release the cantilever beam. In the first step, anisotropic etching was applied to open a window and to ensure that the fluorine radicals can react efficiently with the silicon underneath the SiO$_2$ beam during the isotropic plasma etching. Then, the isotropic plasma etching was applied to release the SiO$_2$ beam from the bulk silicon substrate.

A 3 μm thick film of photoresist 1813 was spun on the front side of the wafer and then the front side of the wafer was patterned with mask 2 (Fig. 4). The photoresist pattern served as an etching mask for ICP etching process to release the microcantilever beam from the bulk silicon. The cantilever beam was released by two plasma dry etching steps: 90-μm thick anisotropic etching and then isotropic etching until all microcantilever beams were released (Fig. 5d). The processing time for releasing the cantilever was 20 min. The optimal recipe for isotropic etching in this step was chosen by the following investigation and discussions. Figure 6 shows the scanning electron microscopy (SEM) pictures of one cantilever. The configuration of the several devices is shown in Fig. 7. The connecting wing between two adjacent microcantilever bodies should be about 50–70 μm thick, so that the thickness is not only sufficient to connect them together, but also easily broken just by applying a little force. The cantilevers can easily be coated with a sensing polymer or films for different chemical or biological sensing applications.

4 Measurement

The above simulation result indicates that a SiO$_2$ cantilever has the potential to give a larger deflection amplitude than another advantage of this process is that it is easy for us to handle the problem associated with the photoresist mask, which occurred in our previous experiment. During an anisotropic dry etching go through an entire wafer with ICP, the photoresist mask was damaged or etched off because the ions bombard the surface of the photoresist. The fabrication process we developed calls for dividing the release process into three dry etching steps. Meanwhile, the release time is greatly reduced at the higher isotropic etching rate (9.1 μm/min). Therefore, the 3 μm thick photoresist 1813 can be an appropriate mask layer for releasing the cantilever beam.

Fig. 7 SEM image of SiO$_2$ microcantilevers
shows that the new moisture sensor responds to the decrease in the humidity. The excellent real time monitoring property gives the device a promising application in environments requiring high sensitivity.

5 Conclusion

A high sensitivity guard-armed SiO$_2$ cantilever-based sensor platform was successfully designed, modeling-analyzed, micro-fabricated, and tested experimentally. A front-sided microfabrication process has been developed, which was valuable for fabricating SiO$_2$ cantilever beams and releasing other suspending parts from the silicon substrate. The new cantilever’s design and fabrication process solve some major issues, which occurred in the conventional design and fabrication, such as cantilever release, etching mask endurance, and beam protection. Based on the developed ICP isotropic release process, a special mask was designed. The cantilever beam was completely released with a 3 μm thickness photore sist layer and 20 min etching time by ICP plasma dry etching (46 mTorr pressure, 1,800 W source power, substrate power 20 W, 300 sccm SF$_6$ flow rate, and 20°C chuck temperature). Both simulation and experimental results proved that the SiO$_2$ microcantilever can achieve higher sensitivity than the equivalent Silicon microcantilever due to its lower Young’s modulus. With specific modification on the surface of the sensing cantilever, the sensors have been shown experimentally to detect 1% RH. It can be seen that using the SiO$_2$ microcantilever as a platform with appropriate...
Acknowledgments This work was supported by NSF Sensor and Sensor Network ECS-0428263 and Board of Regents Industrial Ties and Research subprogram under contract number LEQSF (2005-04)-RD-B-19.

References