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Vapor Hydrofluoric Acid Sacrificial Release Technique for Micro Electro Mechanical Systems Using Labware

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We have developed a novel technique of sacrificial layer etching for micro electro mechanical systems (MEMS). Our technique uses vapor of hydrofluoric acid (HF) to etch sacrificial silicon oxide and to make freestanding silicon microstructures. The advantages of this technique are: (1) no subsequent water rinse is needed, (2) freestanding silicon microstructures can be successfully released without sticking to the substrate, (3) equipment for our vapor phase HF etching simply consists of Teflon beakers only. Conditions for the technique have been optimized by estimating etching rate with test patterns made of silicon-on-insulator (SOI) wafers and by observing water droplets condensation on the sample surface with thermally oxidized silicon chips. By this technique we have successfully obtained freestanding microstructures of SOI wafers. Microcantilevers of as long as 5000 μm (a 5-μm-wide, 10-μm-thick, and 5000-μm-long cantilever over a 0.6-μm-gap) have been successfully released without adhering to the base substrate or contacting the neighboring cantilevers. We have also fabricated and actuated electrostatic comb-drive actuators of 60 and 200 comb pairs to demonstrate high processing yield of our nonstick releasing technique. [DOI: 10.1143/JJAP.42.3690]

KEYWORDS: hydrofluoric acid, vapor HF, MEMS, SOI wafer, silicon oxide, releasing process, sacrificial etching, adhesion, sticking problem

1. Introduction

Silicon dioxide (SiO_2) sacrificial-release technique has long been used in micro electro mechanical systems (MEMS) of polysilicon surface micromachining, in which functional polysilicon microstructures are formed on a silicon oxide layer and released at the final step by selectively removing the underlying sacrificial oxide. Sacrificial-release technique still holds its significance in today's bulk-micromachining processes using deep reactive ion etching (DRIE).

Aqueous solution of hydrofluoric acid (HF) is used for selective sacrificial etching of silicon oxide. However, it requires special caution in handling because freestanding polysilicon microstructures are very fragile after release, and they easily stick onto the base substrate due to the surface tension force of the rinsing liquid while it evaporates. Various techniques have been developed to avoid the surface sticking problem.

To replace the water after the rinsing process, a liquid of low surface tension such as isopropanol is commonly used. Phase change at the liquid-gas interface or a supercritical state is also useful. For instance, freeze-dry technique of sublimation[1–4] was used in the early version of the nonstick releasing process. Supercritical-point dryer of CO_2 has also been used for both organic and inorganic materials.[5]

As a mechanical design solution to the sticking problem, microdimples[6,7] or rough surfaces[8] are intentionally used to reduce the pull-down force or effective contact area. Photoresisit-asist releasing combined with subsequent O_2 plasma ashing is useful for most silicon and metal microstructures.[9] Another example of a mechanical solution is to use temporary supports such as tethers[10] and columns[11] that are to be removed after the releasing step. Microstructures made of nickel electroplated on a silicon substrate have been released by isotropic dry etching of the silicon layer.[12] Some MEMS processes can be replaced with sacrificial-etch-free methods. Instead of making a gap by sacrificial etching, one could prepare a recess on the back surface of a movable pattern before wafer bonding.[13,14]

On the other hand, the concept of removing the sacrificial layer with vapor phase etching is very attractive because it eliminates the whole sequence of wet etching, rinsing steps, and elaborate drying procedure and enables us to obtain freestanding microstructures easily. Several researchers have developed the vapor HF technique[15,16] but with complicated apparatus. In this paper, we report a very simple method of vapor phase etching using lab-ware only.

2. Vapor HF Releasing Technique

Figure 1 schematically illustrates our experimental apparatus. An aqueous solution of HF (HF 50%, H_2O 50%) is kept in a Teflon dish and vaporized to induce a chemical reaction with silicon oxide. One more Teflon dish is used to cap the HF vessel to keep the HF vapor inside. A light bulb [an incandescent lamp, 110 V, 40 W WHITE LAMP (TOKI)] is used in order to warm up the sample and to control the temperature during this process. For better uniformity of etching, we set a sample upside down to the HF vapor. For a small sample, we use a Teflon film with some holes to hold the chips in the vessel.

We have made microstructures with a silicon-on-insulator

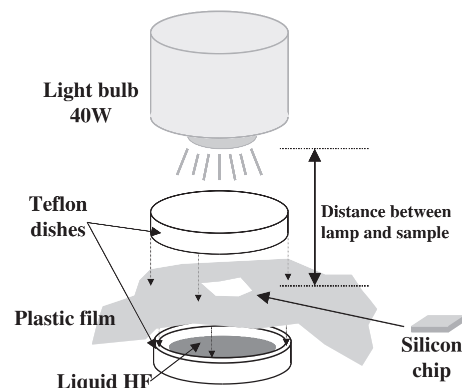


Fig. 1. Schematic illustration of vapor HF apparatus. Sample is placed on a plastic film over HF acid in the teflon beaker.

Table I. The relationship between temperatures and etching results of the thermal oxide layers on silicon wafers. In order to change the temperature in the HF vessel, the height of the light bulb was changed. Etching time was 10 min.

Light Bulb Position	Temperature	Water condensation	Etch rate (compared with a 10 cm distance setup)	Overall Evaluation
No lamp	24°C (Cleanroom temperature)	Uniformly wet	Fast	Not good
15 cm distance	Approx. 30°C	Many droplets formed	Fast	Not good
10 cm distance	Approx. 40°C	No condensation	Medium	Good
5 cm distance	Approx. 60°C	No condensation	Slow	Acceptable

(SOI) wafer of a 10-μm-thick active layer on a 0.6-μm-thick buried oxide (BOX). Trenches of 1 to 20 μm wide and 4 mm long were used to estimate the undercut of silicon oxide. In addition to this, by using silicon chips with thermal oxide, the effects of temperature and water condensation have been investigated.

Moreover, with the SOI wafer, cantilevers of 10 to 5000 μm long with various widths (5, 10, 20 and 40 μm) were prepared for the releasing test. Comb-drive micro-actuators were fabricated and tested on the same wafer.

3. Results

3.1 *Etching results of vapor HF*

Figure 2 shows the side etch length of silicon dioxide as a function of etching time. We measured the undercuts of side etching which are generated under the trench structures made of SOI wafers (Fig. 3). The undercut values plotted in Fig. 2 are the average length measured on different trench openings (5, 10, 15, and 20 μm). From this result, we estimated the etching time for fully releasing a 40-μm-wide cantilever to be 120 min.

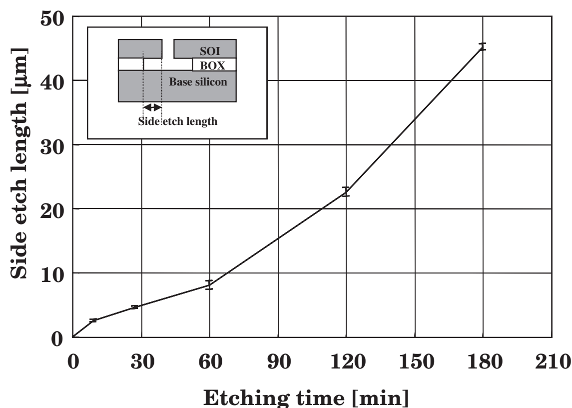


Fig. 2. Undercut by side etch as a function of etching time. SOI wafers were used to estimate the undercut of SiO_2. Side etch variation of different trench opening widths is shown by the error bar.

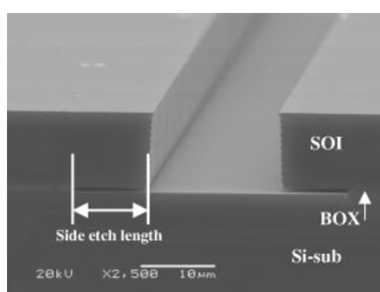


Fig. 3. Scanning electron microscope (SEM) image of side etching of SiO_2.

Maximum deviation of undercut has been measured to be 0.4 μm, 0.3 μm, 1.5 μm, 1.6 μm, and 1.1 μm after 9 min, 27 min, 60 min, 120 min, and 180 min, respectively. Moreover, the undercuts of each etching time have no dependence on trench width. Thus we have concluded that the difference in etch rates caused by trench width are negligible in the etching process. We have also found that the uniformity of this etching technique is good because we have visually confirmed that more than 90% of 560 micromachines in a 3 cm square chip have been successfully released and driven by electrostatic force.

3.2 *Effect of temperature and water during vapor phase HF etching*

Table I shows the relationship between temperatures and etching results of the thermal oxide layers (initial thickness 7565 Å) on silicon wafers. The height of the light bulb was changed from 5 cm to 15 cm to change the temperature in the HF vessel. Observed parameters are wafer condensation on the surface, etch rate of the first 10 minutes, and temperature in the vessel. The etch rate is compared with the value when the light bulb is at a distance of 10 cm.

When no light bulb is used, the silicon oxide layer was completely removed at room temperature (24°C) but water condensation was observed. When the electric lamp was set at a distance of 15 cm from the sample, the sample warmed up to approximately 30°C and the etch rate of the silicon oxide was high, nevertheless water condensation occurred.

When the distance was 10 cm, the sample temperature became approximately 40°C and no droplets were found. Since we confirmed no silicon oxide left on the surface of the sample, the etch speed was sufficiently fast to release microstructures. When the distance was 5 cm, the sample became approximately 60°C and no water condensation was observed. However, silicon oxide was not etched completely.

Figure 4 shows the surfaces of the samples after the first one minute in the vapor HF. On the sample without light illumination [Fig. 4(a)] and that of a 15 cm setup [Fig. 4(b)], we found stains of water droplets. The pattern of the stains in Fig. 4(b) indicates that etching has taken place more homogeneously than the etching indicated by the pattern in Fig. 4(a). On the other hand, surfaces were uniformly etched and no stains of droplets were observed on the surface of the 10 cm setup [Fig. 4(c)] and 5 cm setup [Fig. 4(d)].

Figure 5 shows etching rate as a function of light bulb height measured with the samples used in Fig. 4. Variation of etching depth in one sample represents the nonuniformity

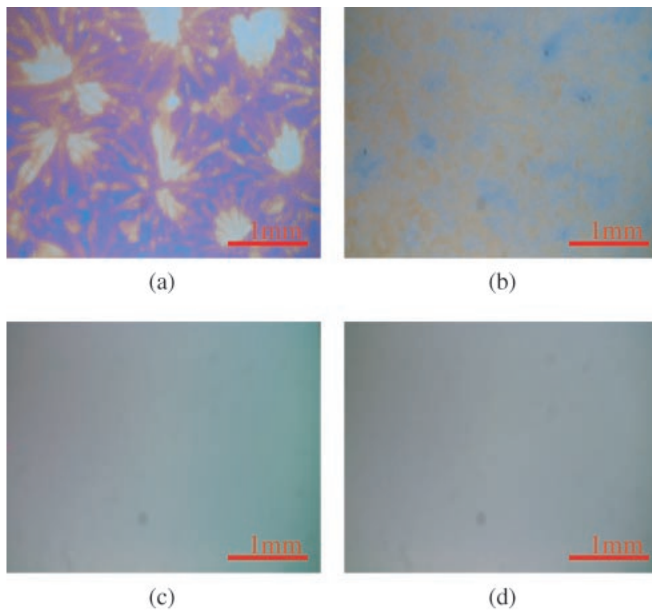


Fig. 4. Optical microscope photographs of SiO_2/Si wafers after 1 minute in vapor HF. Each etching depth is shown in Fig. 5. (a) Processed without using the light bulb. A lot of water droplet stains of irregular patterns are formed due to water condensation. (b) Processed with the light bulb at 15 cm. Surface is stained but the etching reaction is more homogeneous than (a). (c) Processed with the light bulb at 10 cm. (d) Processed with the light bulb at 5 cm. On samples (c) and (d), the oxide is uniformly etched and no marks of water droplets are observed.

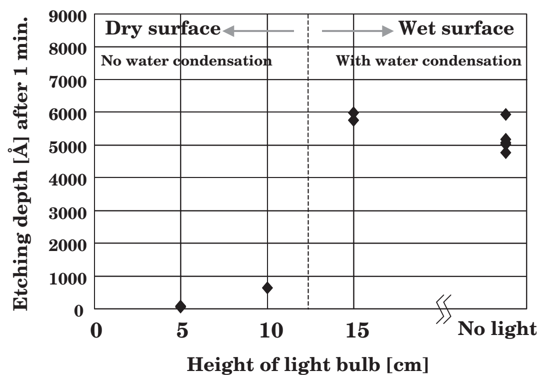


Fig. 5. Etching depth obtained after 1 minute in vapor HF, evaluated with thermal silicon oxide.

of the surface caused by water condensation. When the light bulb was placed 10 cm or nearer, etch speed was not so fast, nevertheless, etching conditions were dry (no water was observed) and results were sufficiently uniform to realize freestanding microstructures. To the contrary, when the light bulb was placed over 10 cm, etch speed was fast but water was condensed on the sample surface to create dots resulting in nonuniform etching.

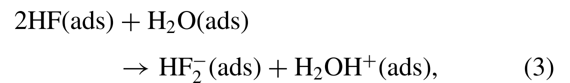
Etching results significantly change in quantity and quality by the effect of the light bulb position around 10 cm to 15 cm as shown in Fig. 5. In the left-hand side region in the plot, where the light bulb is placed near the sample, the etch rate is relatively slow. However, the releasing result is successful because the wafer surface remains dry without causing water condensation. On the other hand, in the right-hand side region in Fig. 5, when the light bulb is farther than 15 cm or when no light is used, silicon oxide is etched fast. However, the released microstructures are not free from the sticking problem because the wafer surface becomes wet due to water condensation.

4. Discussion

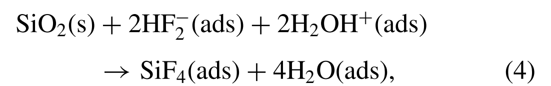
Chemical reaction of SiO_2 etching by HF is described by the following equations[16–18)]

$$HF(g) \rightleftharpoons HF(ads), \quad (1)$$

$$H_2O(g) \rightleftharpoons H_2O(ads), \quad (2)$$



$$2HF(ads) + H_2O(ads) \rightarrow HF_2^-(ads) + H_2OH^+(ads), \quad (3)$$



$$SiO_2(s) + 2HF_2^-(ads) + 2H_2OH^+(ads) \rightarrow SiF_4(ads) + 4H_2O(ads), \quad (4)$$

$$SiF_4(ads) \rightleftharpoons SiF_4(g), \quad (5)$$

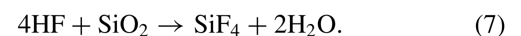
$$H_2O(ads) \rightleftharpoons H_2O(g). \quad (6)$$

The reaction occurs on the SiO_2 surface, where water adsorbed on the surface is believed to catalyze the reaction by increasing the concentration of the active bifluoride species as described by eq. (3).

Temperature control is, therefore, important to maintain an adequate amount of water for fast etching as well as to avoid water condensation. The etching process of HF vapor of silicon oxides at various temperatures has been intensively investigated.[20)] It has been reported that etch rates of silicon oxides produced by various methods (thermally grown, chemical vapor deposition, etc.) depend on temperature. In case of thermal oxide, the etch rate drops more than two orders of magnitude when the temperature was raised from room temperature to 40°C.

Time dependence of etch rate can be found in Fig. 2. The etch speed during the first 10 min is much faster than that of the following 10 min. After the first 10 min, etch rate seems to be constant for a while, and it becomes fast again after 60 min.

Our hypothesis to explain the change in etch rate is as follows. The reaction speed is governed by the presence of absorbed water which behaves as a catalyst on the silicon oxide surface. At the beginning of the process, when the temperature of the sample is about to increase and is still low, the water condenses on the sample surface to enhance the etching reaction. During this period, the water is supposed to be the result of the reaction or from liquid HF, which contains H_2O. After the first 10 minutes, since the sample is warmed up to approximately 40°C, and the temperature becomes constant, the amount of water existing on the surface is thought to be constant thanks to the balance of water evaporation and water generation [eq. (4)]. When etching time is long, etch rate becomes fast to generate more H_2O by the following reaction:



$$4HF + SiO_2 \rightarrow SiF_4 + 2H_2O. \quad (7)$$

This is the sum of eqs. (3) and (4) showing that more water is produced by the SiO_2 etching process. When the undercut length is large after long etching time, generated water is trapped inside, and evaporation rate decreases. As a result,

the etch rate of oxide increases again due to the catalytic effect, as seen in our experiment.

Using an electric light bulb is a simple and useful method to warm up the sample and to control water condensation on the sample surface. From Fig. 2, Table I and Fig. 5, we conclude that the dry region in Fig. 5 is suitable for the releasing step. Furthermore, samples should be kept at approximately 40°C during the step in order to obtain high-speed etching.

From our experience, we have optimized vapor HF etching procedure as follows: First, samples were warmed up to 40°C by the light bulb for 30 min in order to prevent water condensation. The samples were then exposed to the vapor HF for 120 min to produce more than a 20 μm undercut. During the process, the samples were kept warm by the light bulb. After etching, the samples were warmed again for 3 min to eliminate remaining HF vapor.

5. Demonstration of Releasing Microstructures

By using the vapor HF technique, compliant freestanding microstructures can be easily released without the sticking problem. Figure 6 shows cantilevers made by this method. Figure 6(a) shows the entire view of the cantilever array. Figures 6(b) and 6(c) show close-up views. The longest cantilever is 5 μm wide, 10 μm high, and 5000 μm long. The gap between the structures and base substrate is only 0.6 μm.

Figure 7 shows an electrostatic comb-drive actuator that has been successfully released by the same manner. The comb part consists of 60 pairs of electrodes. The suspensions are 500 μm long, and the movable central part is 500 μm × 100 μm in area. We have successfully driven the actuator by electrostatic force. We also released and demonstrated a larger version of the comb-drive actuator, which has 200 pairs of comb electrodes and a 1500 μm × 100 μm movable part.

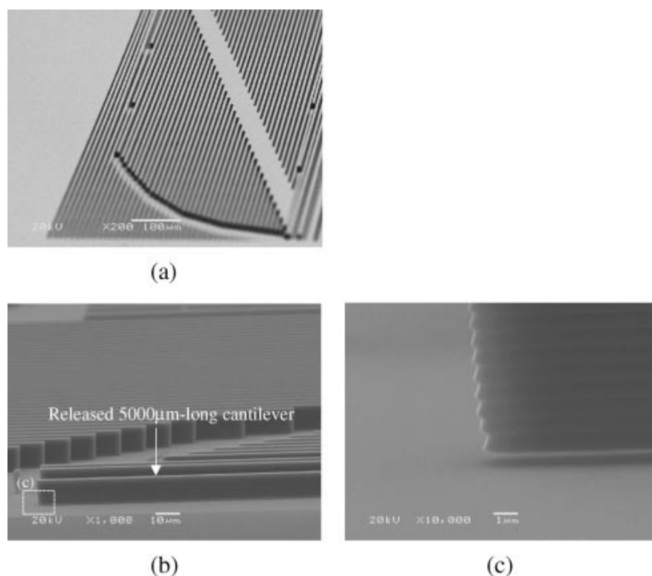


Fig. 6. (a) SEM images of cantilevers made of SOI wafer released by vapor HF technique. The longest cantilever is 5000 μm long, 5 μm wide and 10 μm high. The gap between the cantilever and base substrate is 0.6 μm. (b) SEM image of the 5000 μm-long cantilever. The cantilever is free of the sticking problem. (c) Close-up view of the tip of the released 5000 μm-long cantilever.

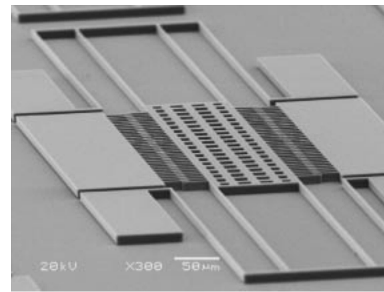


Fig. 7. SEM image of a comb-drive actuator fabricated by vapor HF technique. The central frame (100 μm × 500 μm) is supported by a set of 500 μm-long cantilevers.

6. Conclusion

A very simple and reliable sacrificial releasing technique for MEMS devices has been developed using vapor HF in Teflon beakers and an electric light bulb as a heater. The etch rate of silicon oxide and its time dependence have been investigated with test structures made of SOI wafer. By observing water droplets condensing on the surface of the silicon oxide, we have optimized the light bulb height to be 10 cm above the chips to maintain them at 40°C. Under this condition, we have no sticking problem for the microstructures. Nevertheless, the water on the silicon surface works as a catalyst for silicon oxide etching, it should be constantly removed in order to avoid the sticking problem and achieve freestanding microstructures.

We have obtained a freestanding silicon cantilever 5000 μm long, 5 μm wide, and 10 μm high suspended over a 0.6 μm air gap. By the same technique, we have successfully released a comb-drive actuator with a 500 μm × 100 μm large movable frame suspended with a set of 500 μm suspensions without any part sticking to the substrate. A comb-drive actuator with a 1500 μm × 100 μm large movable frame has been successfully released and electrostatically actuated.

This vapor HF technique eliminates wet etching sacrificial release and significantly improves production yield of MEMS devices.

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