Research Report

A New Full-Dry Processing Method for MEMS

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MEMS用気相エッチング技術

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Abstract

A new full-dry processing method has been developed that includes a new sacrificial layer dry etching technique, which enables the microscopic structures used for microsensors and micro-electro-mechanical systems (MEMS) to be released from silicon substrates with high repeatability, and a new water-repellent dry coating technique, which prevents the released structures from sticking to the substrates during operation. The usefulness of this full-dry processing method from etching to coating, with respect to the length of releasable cantilevers, was evaluated by comparison with the conventional wet method. It was confirmed that the full-dry processing method permits sustainable cantilevers to be about three times longer than those released using the conventional wet method.

Keywords Surface micromachining, Microsensor, Micro-electro-mechanical systems, MEMS, Sacrificial layer dry etching, Water-repellent dry coating

要　旨

マイクロセンサや微小電気機械システム(MEMS) に用いる微小な構造体が再現性良くシリコン基板よりリリースできる犠牲層ドライエッチング技術とリリースに成功した構造体が動作中において基板に付着しないようにするための撥水性ドライコーティング技術を開発した。従来のウエット法と本ドライ法によるリリース可能なカンチレバー長さを比較し，オールドライ処理の有用性について評価した。エッチングからコーティングまでのオールドライ処理は，従来のウエット法に比べ，カンチレバーの形成，保持長さを3倍以上にできることが確認された。

キーワード 表面マイクロマシニング，マイクロセンサ，微小電気機械システム，MEMS，犠牲層ドライエッチング，撥水性ドライコーティング

1. Introduction

Surface micromachining techniques for forming microscopic movable structures on silicon substrates are widely applied to the development of microsensors and MEMS. Each of such microstructures is fabricated by undercut etching of an underlying layer called a sacrificial layer. Conventionally, a solution for etching the sacrificial layer is selected from hydrogen fluoride or alkaline solutions, depending on the materials of the sacrificial layer and the microstructures. However, in the drying process after rinsing with deionized water that follows wet etching, the structure sticks to the silicon substrate due to the surface tension of the water, if its rigidity is low. In addition, even if the structure is formed successfully, there is a possibility that it may stick to the substrate during operation. This sticking occurs because the sticking force, such as the intermolecular force, is greater than the restitutive force of the structure. These difficulties limit the formation of low-rigidity structures, which are needed to improve the sensitivity of microsensors, when using the conventional wet method.

To overcome these difficulties, dry-phase sacrificial layer etching techniques, which do not require the rinsing and drying processes after etching, have recently been proposed.[1-3)]

A full-dry micromachining method that includes sacrificial layer dry etching and water-repellent dry coating to prevent sticking after release has been developed. Use of a water-repellent coating is one method that prevents sticking by reducing the surface energy of the structure. We have previously reported on a water-repellent silanization dry coating technique using organosilicon compounds.[4)] When the substrate is coated with an organosilicon compound, a film composed of regular arrays of long-chain alkyl groups standing vertical to the substrate is formed. This is referred to as a self-assembled monolayer. Because it is a monomolecular layer, the mechanical properties of the structure are scarcely changed by the water repellent processing, which could make this coating technique effective in forming low-rigidity structures.

This paper begins with a discussion of the micromachining system for full-dry processing, and then proceeds to the basic features of sacrificial layer etching of silicon dioxide using a mixed gas of anhydrous hydrogen fluoride (HF) and methyl alcohol (CH_3OH). This is followed by an examination of the sacrificial layer etching of polysilicon (poly-Si) using xenon difluoride (XeF_2) gas, and water-repellent coating using (tridecafluoro - 1, 1, 2, 2 - tetrahydrooctyl) trichlorosilane ($C_8F1_3H_4SiCl_3$) gas and water vapor (H_2O). The paper concludes with a discussion of the usefulness of the full-dry processing method from etching to coating.

2. Micromachining system

Figure 1 shows the overall appearance of the micromachining system. The system consists of a main body that contains an etching and a coating chamber, a cylinder cabinet that houses an HF cylinder, a waste gas abatement system, and a controller. The dimensions of the system are 1.9m W, 1.3m D, and 1.7m H.

Figure 2 is a line diagram of the micromachining system, in which an etching and a coating chamber are installed. A mixed gas of HF and CH_3OH and XeF_2 gas can be separately fed into the etching chamber. In order to etch poly-Si with XeF_2 gas, it must be stripped of its natural oxide. For this pretreatment, a diluted HF solution is conventionally used. The new system enables dry-phase pretreatment using a mixed gas of HF and CH_3OH. In addition, both the mixed gas and XeF_2 gas do not etch aluminum. This permits sacrificial layer

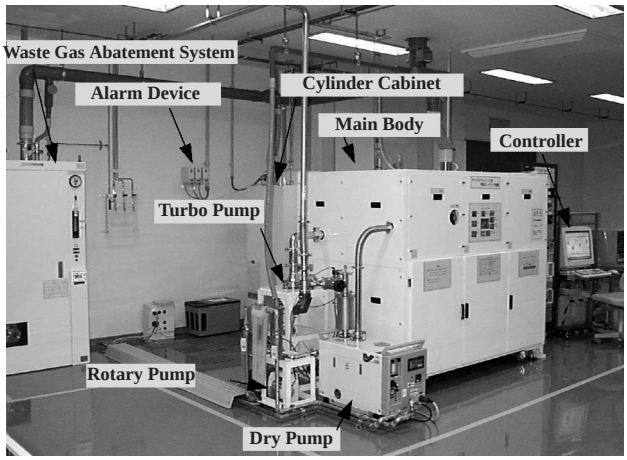


Fig. 1 A photograph of the micromachining system.

etching at the end of the sensor process after the formation of aluminum wiring.

The exhaust system consists of a vacuum purge line with a rotary pump and a turbo-molecular pump and a treating line with a dry pump. Acceptable wafer sizes are 4, 5, and 6 inches. A customized Visual Basic program was used to control the treating process.

3. Characteristics of sacrificial layer etching of silicon dioxide with HF/CH_3OH mixed gas

3. 1 Etching procedure and samples for evaluation

As shown in Fig. 2, HF and CH_3OH gases are separately fed into the etching chamber while the flow rate is controlled by a mass flow controller. The pressure in the chamber can be adjusted with a throttle valve. The mechanism of silicon dioxide (SiO_2) etching with the HF/CH_3OH mixed gas can be expressed by the following equations:

$$2HF + M \rightarrow HF_2^- + MH^+ \quad \cdots\cdots (1)$$

$$SiO_2 + 2HF_2^- + 2MH^+ \rightarrow SiF_4 + 2H_2O + 2M \quad \cdots\cdots (2)$$

where, M represents CH_3OH.

Figure 3 shows the process of fabricating the samples used to evaluate the performance and progress of SiO_2 sacrificial layer etching. The silicon substrates used were p-type(100)-oriented wafers with a resistivity of 10 to 20Ω·cm. The process is summarized as follows.

(a) An SiO_2 sacrificial layer is deposited on the silicon substrate.

(b) A poly-Si layer, which is resistant to the HF/CH_3OH mixed gas, is deposited on the SiO_2 coated substrate by low-pressure CVD.

(c) The poly-Si layer is partially etched by reactive ion etching (RIE) to make a round hole of 10μm in diameter.

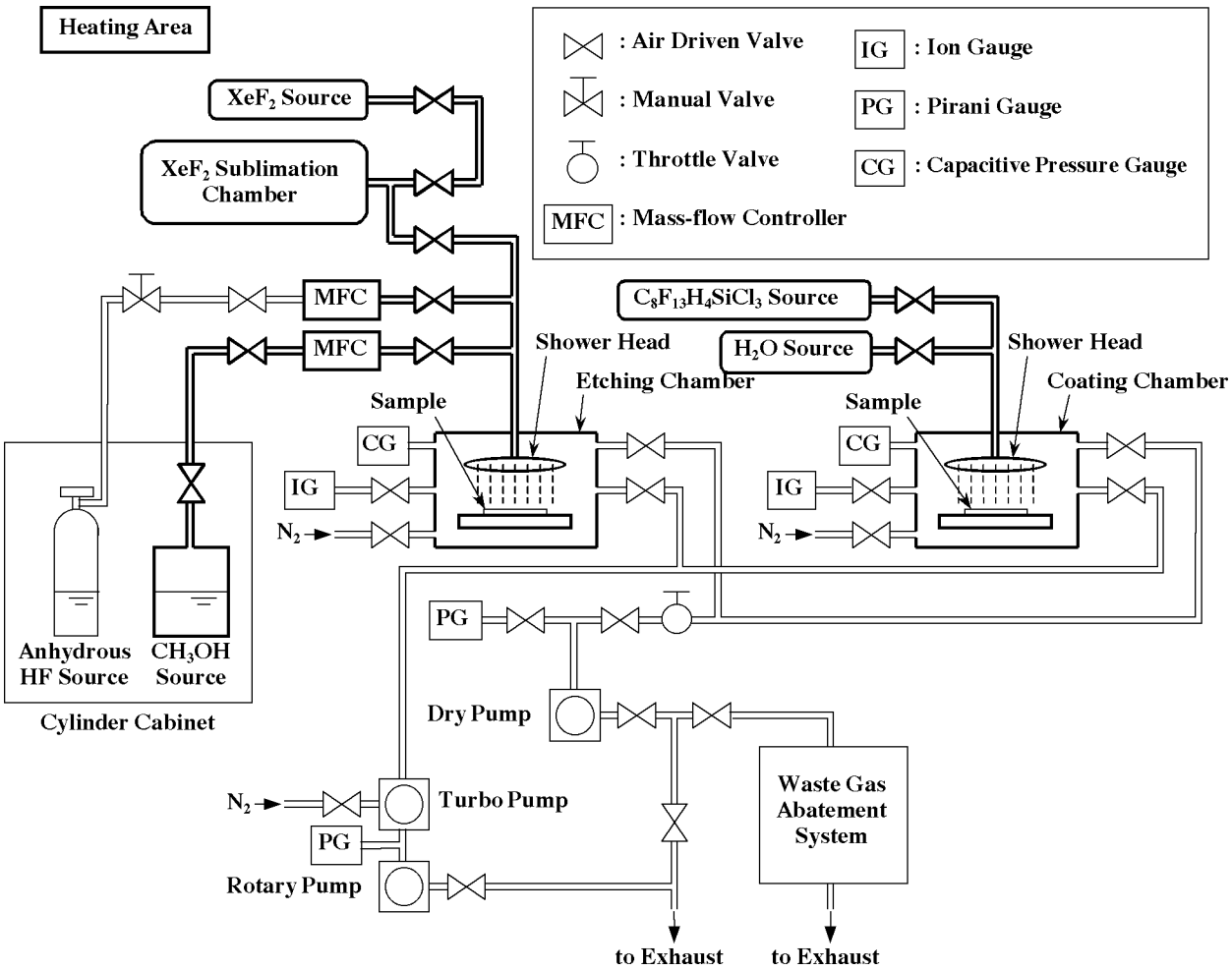


Fig. 2 A schematic of the micromachining system.

(d) The SiO_2 is undercut etched with the HF/CH_3OH mixed gas.

The progress of undercut etching was evaluated by observing the dark field image under a metallurgical microscope as shown in **Fig. 4**. The undercut etching distance was measured with a micrometer attached to the microscope. Etching characteristics were investigated in terms of the etching rate obtained, by dividing the SiO_2 undercut etching distance by the etching time.

3. 2 Test results

Figure 5 shows the relation between the CH_3OH/HF flow rate ratio and the undercut etching rate. The HF flow rate was fixed at 400sccm, while the CH_3OH flow rate was varied from 0 to 400sccm. The other conditions were a gas pressure of 30Torr, a sample temperature of 30°C, and an etching time of 60min. It was found that the undercut etching rate shows a maximum at a CH_3OH/HF flow rate ratio of 0.6.

Following this test, the dependence of the undercut etching rate on gas pressure, gas flow rate, and sample temperature was investigated to determine suitable treating conditions for SiO_2 sacrificial layer etching. The suitable conditions obtained are listed in **Table 1**. Under these conditions, the SiO_2 sacrificial layer etching rate was 115nm/min.

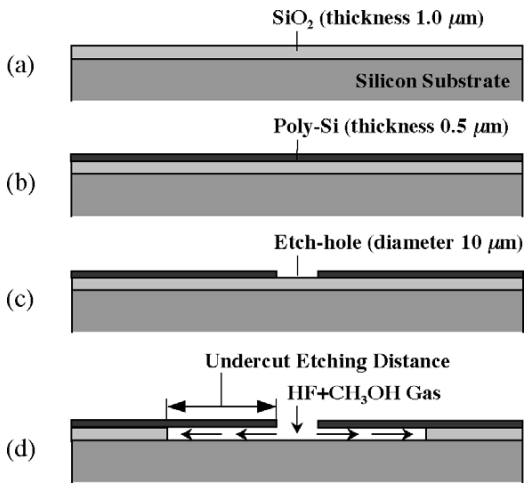


Fig. 3 Process sequence for fabrication of an SiO_2 sacrificial layer etching sample.

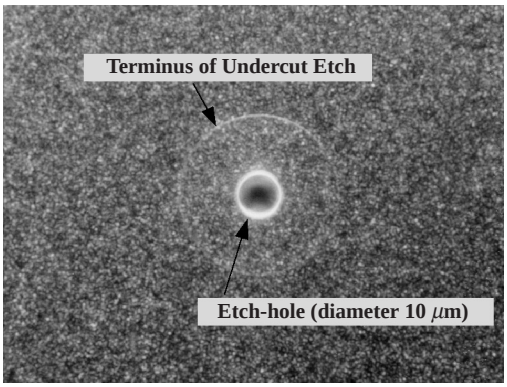


Fig. 4 A top view photomicrograph of an etched SiO_2 sacrificial layer.

4. Characteristics of sacrificial layer etching of polysilicon with XeF_2 gas

4. 1 Etching procedure and samples for evaluation

The poly-Si sacrificial layer etching was performed by "pulse etching," in which the sample is alternately exposed to XeF_2 gas and a vacuum. An outline of the steps involved is as follows. XeF_2 gas is fed into the etching chamber until the required

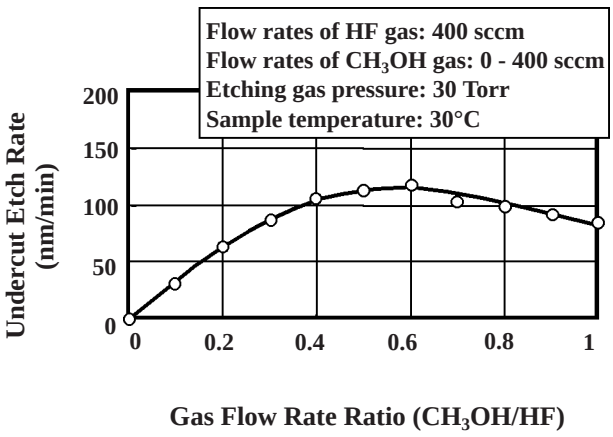


Fig. 5 Dependence of the undercut etch rate on gas flow rate ratio (CH_3OH/HF).

Table 1 Suitable treating conditions for SiO_2 sacrificial layer etching.

Flow Rates of HF Gas	200 sccm
Flow Rates of CH_3OH Gas	120 sccm
Etching Gas Pressure	30 Torr
Sample Temperature	30 °C

gas pressure is reached. It is then held for several minutes. Finally the gas is exhausted with a dry pump. This is performed automatically by opening and closing an air driven valve. The silicon (Si) etching mechanism with XeF_2 gas is expressed by the following equation.[5)]

$$2XeF_2 + Si \rightarrow 2Xe + SiF_4 \cdots\cdots\cdots\cdots\cdots (3)$$

Figure 6 shows the process of fabricating the samples used to evaluate the characteristics and progress of poly-Si sacrificial layer etching. The silicon substrates used were p-type(100)-oriented wafers with a resistivity of 10 to 20Ω·cm. The process is summarized as follows.

(a) An SiO_2 layer, which is resistant to XeF_2 gas, is deposited on the silicon substrate.

(b) A poly-Si layer is deposited on the SiO_2-coated substrate as a sacrificial layer by a low-pressure CVD process.

(c) A silicon nitride (SiN) layer, which is resistant to etching, is deposited on the poly-Si-coated substrate by a plasma CVD process.

(d) The SiN layer is partially etched by RIE to make a round hole of 10μm in diameter.

(e) The poly-Si is undercut etched using XeF_2 gas.

4. 2 Test results

To investigate the effect of XeF_2 gas pressure on the progress of poly-Si sacrificial layer undercut etching, the undercut etching distance per pulse of etching was measured under conditions of a XeF_2 gas duration time of 1 min and a sample temperature of 25°C. **Figure 7** shows the undercut etching distance with respect to the number of etching pulses with XeF_2 gas pressure as a parameter. From the figure, it is apparent that: (1) the higher the XeF_2 gas pressure, the faster the progress of the undercut etching; and that (2) the undercut etching distance increases as the number of etching pulses increases, although the rate of increase decreases with the progress of etching and the progress becomes saturated. It was presumed that (2) was caused by the feed rate of the XeF_2 gas. In addition, the effect of XeF_2 gas holding time on the progress of undercut etching was evaluated to determine suitable treating conditions for poly-Si sacrificial layer etching. The suitable conditions obtained are listed in **Table 2**.

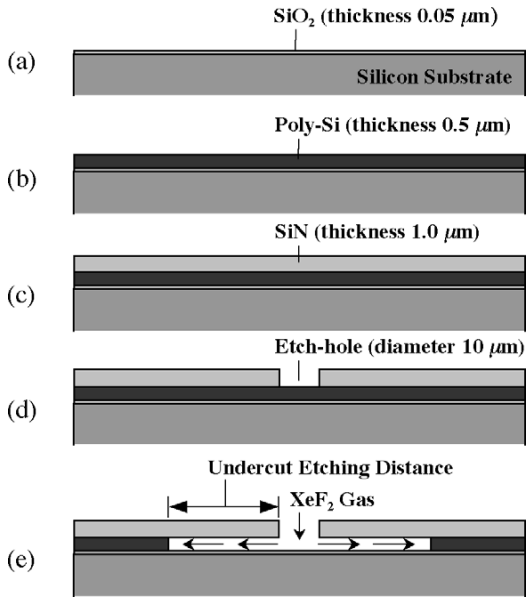


Fig. 6 Process sequence for fabrication of Poly-Si sacrificial layer etching samples.

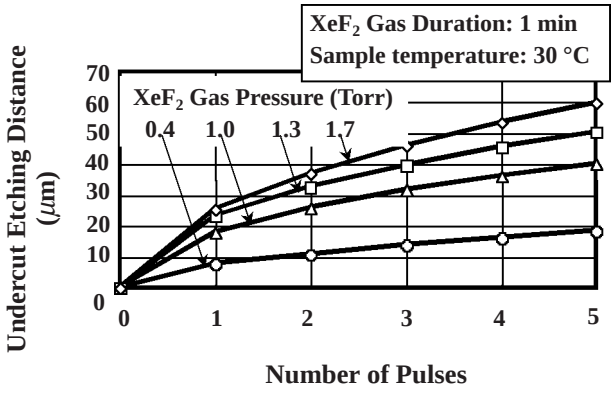


Fig. 7 Undercut etching distance vs. number of pulses with different XeF_2 gas pressure.

Table 2 Suitable treating conditions for Poly-Si sacrificial layer etching.

XeF_2 Gas Pressure	1.0 Torr
XeF_2 Duration Time	3 min
Waste Gas Exhaust Time	5 min
Sample Temperature	25 °C

5. Characteristics of water-repellent coating using $C_8F_{13}H_4SiCl_3$ and H_2O gases

5. 1 Coating procedure and evaluation

The procedure for the coating is as follows. The coating chamber is fed with H_2O gas until the required gas pressure is reached. This pressure is then held for a specified time, after which the gas is exhausted from the chamber for a specified time with a dry pump. Subsequently, $C_8F1_3H_4SiC_{l3}$ gas is fed into the chamber in the same manner as the H_2O gas. This cycle is automatically repeated by opening and closing an air driven valve. A schematic representation of the silanization coating reaction using $C_8F_{13}H_4SiCl_3$ and H_2O gases is shown in **Fig. 8**.

The water-repellency characteristics were evaluated in terms of water contact angle. Water was dripped onto the surface of a water-repellent coated silicon substrate. **Figure 9** shows the photomicrograph of a water droplet on the silicon surface after a silanization coating of 10 iteration, indicating that the water contact angle is 102 degrees. When the water contact angle is greater than 90 degrees, sticking on wet samples can be prevented.

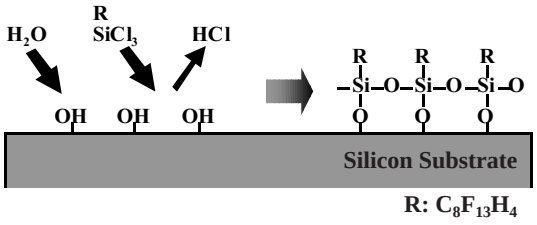


Fig. 8 A schematic of the silanization reaction.

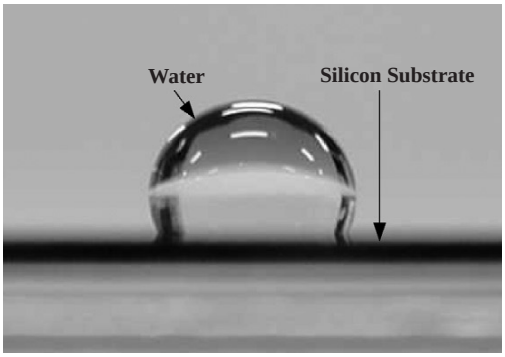


Fig. 9 A water contact angle with a silicon substrate after silanization coating of 10 iteration.

5. 2 Test results

To investigate the dependence of water repellency on the frequency of the coating, the water contact angle at each treating cycle was measured. **Table 3** shows the conditions for the water-repellent treatment. For these experiments, p-type(100)-oriented silicon wafers were used as substrates. As a pretreatment, the substrates were stripped of natural oxide using a diluted HF solution (HF/H_2O: 1/50, dipping time: 30s).

Figure 10 shows the dependence of the water contact angle on the frequency of the coating. The initial water contact angle is the value just after dipping in diluted HF. It was found that the water contact angle increases as the frequency of the coating increases, exceeding 100 degrees at four cycles.

6. Evaluation of usefulness of the full-dry processing

The usefulness of the full-dry processing method was evaluated by comparing the new dry method with the conventional wet method, with respect to the length of releasable cantilevers.

Figure 11 shows the schematic structure of the cantilevers used for the evaluation. A cantilever pattern made in the silicon layer was released from the silicon substrate by etching the SiO_2 sacrificial layer. The cantilevers were 5μm wide, 15μm thick and 25 to 1000μm long. The gap between the cantilevers was 5mm. Since the rigidity of the cantilevers is lower in the horizontal direction than in the vertical direction with regard to the substrate surface, they stick more readily to each other than to

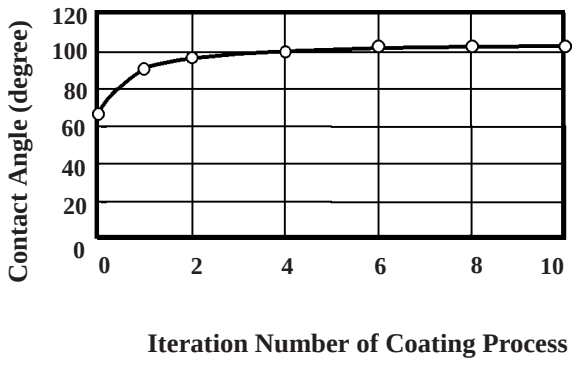
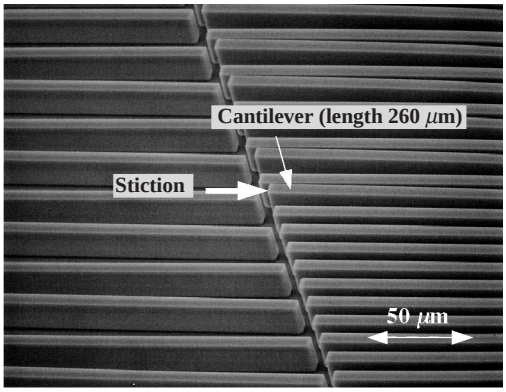


Fig. 10 Water contact angles of silicon substrates treated by the silanizatuion coating.

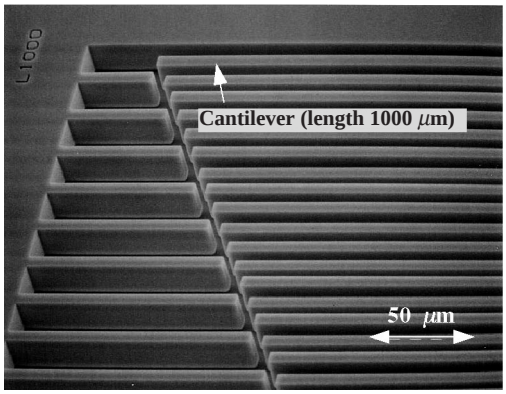
the substrate.

The lengths of the released cantilevers formed by the dry method and the wet method were compared. In the wet method, the SiO_2 sacrificial layer was etched for 30 min with surfactant buffered hydrofluoric acid (LAL-1000 made by Hashimoto Kasei Kogyo), rinsed in deionized water for 10 min and was then left to stand to dry. In the dry method, the SiO_2 sacrificial layer was etched for 30 min under conditions of an HF flow rate of 200sccm, CH_3OH flow rate of 120sccm, a gas pressure of 30Torr, and a sample temperature of 30°C. **Figure 12** shows the released cantilevers fabricated by both the wet and the dry methods. The cantilevers fabricated using the wet method that are longer than 260μm stick to each other. On the other hand, cantilevers fabricated using the dry method that are up to 1000μm in length, are released without any trouble. This proves that the dry method enables the fabrication of releasable cantilevers that are about three times longer than those fabricated using the wet method.

To evaluate the usefulness of the water-repellent coating, cantilevers of up to 1,000μm in length were released by the dry method. Half of these cantilevers were coated five times with the water-repellent coating, under the conditions shown in **Table 3**. Then, these two groups of samples were immersed in water for 1 min and left to stand until dry. Next, the retainable cantilever lengths of the two groups were compared. The cantilevers that were longer than 290μm without the water-repellent coating stuck to each other. On the other hand, the cantilevers whose length was up to 1,000μm with the water-repellent coating did not stick to each other. These results indicate that the full-dry processing method from etching to coating permits sustainable cantilevers to be about three times longer than those released using the conventional wet method.



(a)



(b)

Fig. 12 SEM photographs of the fabricated silicon cantilevers: (a) wet etching (b) dry etching.

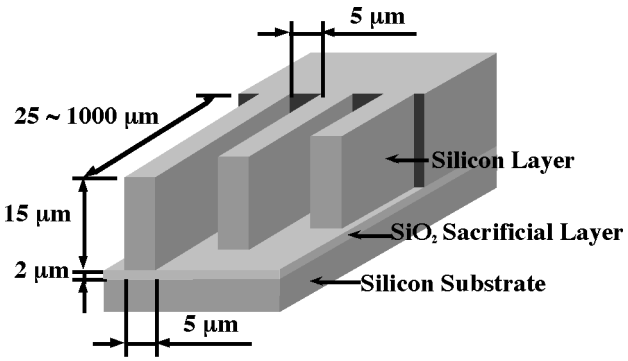


Fig. 11 A schematic perspective view of an experimental sample.

Table 3 Silanization coating conditions.

H_2O Gas Pressure	1.0 Torr
H_2O Duration Time	1 min
H_2O Exhaust Time	3 min
$C_8F_{13}H_4SiCl_3$ Gas Pressure	0.1 Torr
$C_8F_{13}H_4SiCl_3$ Duration Time	10 min
$C_8F_{13}H_4SiCl_3$ Exhaust Time	3 min

7. Conclusion

A new full-dry processing method has been developed that includes a new sacrificial layer dry etching technique, which permits the microscopic structures used for microsensors and MEMS to be released from silicon substrates with high repeatability, and a new water-repellent dry coating technique, which prevents released structures from sticking to substrates during operation. This full-dry processing method from etching to coating, permits sustainable cantilevers to be about three times longer than cantilevers released using the conventional wet method. The full-dry processing method is expected to be useful for improving the sensitivity of microsensors and MEMS.

References

1) Offenberg, M., Elsner, B. and Larmer, F. : “Vapor HF etching for sacrificial oxide removal in surface micromachining”, Electrochem. Soc. Fall Meet., **94**-2(1994), 1056-1057
2) Chang, F. I., Yeh, R., Lin, G., Chu, P. B., Hoffman, E., Kruglick, E. J. J., Pister, K. S. J. and Hecht, M. H. : “Gas-phase silicon micromachining with Xenon Difluoride”, Proc. SPIE Microelectronic Structures and Microelectromechanical Devices for Optical Processing and Multimedia Applications, Austin, TX, (1995), 117-128
3) Lee, Y. -I., Park, K. -H., Lee, J., Lee C. -S. and Yoo, H. J. : “Dry Release for Surface Micromachining with HF Vapor-Phase Etching”, J. Microelectromech. Syst., **2**-3(1997), 226-233
4) Sakata, J., Tsuchiya, T., Inoue, A., Tokumitsu, S. and Funabashi, H. : ”Anti-Stiction Silanization Coating to Silicon Micro-Structures by a Vapor Phase Deposition Process”, Proc. of the 10th Int. Conf. on Solid-State Sensor and Actuators, Sendai, Japan, (1999), 26-29
5) Williams, K. R. : “Etch Rates for Micromachining Processing”, J. Microelectromech. Syst., **5**-4(1996), 256-269

(Report received on June 28, 2002)

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