Wet Etched High Aspect Ratio Microstructures on Quartz for MEMS Applications

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Keywords : quartz, MEMS, anisotropic etching, high aspect ratio, microcapillary, tilt sensor

1. Introduction

α -quartz draws great attention for its piezoelectricity, excellent mechanical characteristics and electrical insulation in comparison with the conventional MEMS material, silicon. The major obstacles limiting the wide use of α -quartz are its complex anisotropic etching profile and the lack of fabrication technologies. This research focused on wet etching of z-cut quartz wafers for high aspect ratio trench and through-hole, which are desired in MEMS fabrication.

2. Experiment

Experimental mask patterns were set up with initial widths of 5, 10, 20, 50, 100 and 200 μm , the large width 200 μm pattern was for observing the whole etching profile and minimum 5 [μm] pattern was for discussing the possible high aspect ratio microstructure [aspect ratio=etch depth/(undercut + initial width)]. The intermediate initial widths patterns were designed to observe the effect of initial width an etching time on the aspect ratio. A 100 μm Z cut wafer was used in this research, and Au/Cr thin film was deposited as the etch mask. Saturated ammonium bifluoride solution at 87 degrees C was used as the quartz etchant.

3. Results

After 40[min] etching, samples were cut vertical to the etching line and cross sections were observed using the SEM (scanning electron microscope). Etching profile was plotted at a polar interval of 5° . The etch rate at the Z plane was 110 $\mu\text{m}/\text{h}$, which was the same at all polar direction. High aspect ratio structure was obtained at the polar angles from 30° to 60° . Fig.1 (a) and (c) show the basic structure of high aspect trench, and the etching aspect ratio dependence on the polar angle after 20, 40, 70 min etching respectively. The aspect ratio is defined as etch depth to (etching undercut+initial width). An example of experimental micrograph (etched at polar angle β of 45° for 70 min) with a etch depth 50.7 μm and top width 16.4 μm is presented in Fig.1 (b).

4. Conclusion and Applications

High aspect ratio microstructure (> 3) which was considered impossible by wet etching technology was achieved. This result suggests that anisotropic etching of Z cut α -quartz can be applied to microcapillary electrophoresis, where high aspect ratio microchannel is desired for improving detecting sensitivity. Trench at polar angle of 45° is demonstrated to be the best choice. We successfully fabricated nearly vertical trench feature at $\beta=45^\circ$ with 50.7 μm depth and 16.4 μm width, the aspect ratio of 3.1.

Double-sided etching showed great interest for the twofold symmetry around the X axis (Fig. 2). According to this property, two same sensors at ($\beta=15^\circ$ and 105°) and ($\beta=-45^\circ$ and 45°) can be arranged at 90° on one quartz chip, which propose a novel two axis tilt sensor.

Fig. 1. Definition of the basic microstructure and aspect ratio: (a) the basic microstructure, u_1, u_2 , and D_b represent side etch and bottom etch depth respectively; (b) SEM photograph of 70 min etched trench at 45° ; (c) Plotted trench aspect ratio [$AR=D_b/(\text{initial width}+u_1+u_2)$] dependence on the polar direction based on the 20, 40, 70 min etching results

Fig. 2. The twofold symmetry around X axis

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Z cut α -quartz wafers were etched in saturated ammonium bifluoride solution at 87 degrees C. The side wall profiles were observed using the scanning electron microscopy (SEM) and plotted dependent on the polar direction. This research focused on investigating high aspect ratio trench and through-hole, which were dependent on the polar direction to the crystal axis. Aspect ratio in dependence on polar direction was also plotted and microchannels with aspect ratio > 3 could be achieved at the polar angle between 30° to 60° . The possibility of application for microcapillary was discussed, and the trench at 45° was considered best. Double-sided etching technique was used for manufacturing through-hole structures. Through-hole at 0° was demonstrated effective for fabrication of capacitive MEMS tilt sensor. Through-holes at 15° and 105° were proposed for fabrication of 90° -arranged two axis capacitive tilt sensor, taking advantage of the twofold symmetry property around X axis and threefold symmetry property around Z axis.

Keywords : quartz, MEMS, anisotropic etching, high aspect ratio, microcapillary, tilt sensor

1. Introduction

Quartz draws great attention for its piezoelectricity, excellent mechanical characteristics and electrical insulation in comparison with the conventional MEMS material, silicon. Further, it is also well known to be the most appropriate material for bioMEMS, especially for microcapillaries because it is UV transparent and chemically inert. The major obstacles limiting the wide use of quartz wafers are its complex anisotropic etching profile and the lack of fabrication technologies.

The precise 3D microstructures with a high aspect ratio are needed in many advance applications⁽¹⁾. For example, high aspect ratio microchannel would provide high sensitivity in the microcapillary electrophoresis (μ -CE) when using UV absorption detection, because absorbance is proportional to the optical path length⁽²⁾. Current CE microdevices are mostly fabricated by isotropic etching fused silica or glass, which suffer from the shallow channel depths, because the resulting channels are twice as wide as they are deep. For solving this problem, several groups have made great efforts in recent decades. Ujiie et al.⁽³⁾ successfully fabricated vertical trench features with $50\ \mu\text{m}$ depth and $20\ \mu\text{m}$ width, the aspect ratio of 2.5, using dry etching technology. But dry etching method is an expensive process and special equipment is needed. Harrison et al.⁽⁴⁾ reported a planar device design with an increased sensitivity, in which the measuring beam was launched through a U-cell, increasing the path length by allowing the beam to pass in a longitudinal direction along the flow channel. Unfortunately, the insertion of optical fibers into etched channels within the device made it difficult to manufacture. Further,

capacitance based sensor is commonly used for acceleration, tilt detection. In this case, high aspect ratio through-hole derives big capacitance and high sensitivity.

Ueda et al.⁽⁵⁾ systematically studied the etching anisotropy of quartz by using 21 kinds of cut angles twenty years ago, and after that Cheng et al.⁽⁶⁾ reported characterization results of anisotropic etching properties using a spherical specimen. But the undoubted fact is that the actual application of quartz for MEMS was very few in comparison with Si. Z cut quartz wafers show the most potentials to fabricate microsensors and microactuators due to the high etch rate at the Z plane. Hedlund et al.⁽⁷⁾ anisotropically etched z cut quartz using the mixture solution of HF and NH_4F at different concentrations, temperatures and gave the etching profiles. But the etching rates of side wall planes were not provided, which should be important in designing microdevices. This research experimentally studied wet-etching profile and etching rates of appeared etching planes using traditional quartz wafer etchant, saturated ammonium bifluoride solution at 87 degrees C. Furthermore, we focused on discussing the possible high aspect ratio microstructures and introducing the proposed and prospective MEMS applications, such as MEMS tilt sensor, microcapillary.

2. Experimental

2.1 Mask Pattern Design Experimental mask patterns were set up with initial widths of 5, 10, 20, 50, 100 and $200\ \mu\text{m}$ (Fig. 1). Large width $200\ \mu\text{m}$ pattern was for observing the whole etching profile and minimum $5\ \mu\text{m}$ was for discussing the possible high aspect ratio microstructure [Aspect ratio = etch depth/(undercut + initial width)]. The intermediate spaces were design to observe the procedure of disappearing of etching plane. Because α -quartz is threefold symmetry, we just studied the polar angle from 0° to 120° with an interval of 5° at the beginning of +x

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Fig. 1. Designed etch mask with initial spaces of 5, 10, 20, 50, 100 and 200 μm (from left to right) and definition of the pattern polar direction β starting from +X axis

Fig. 2. Definition of the sidewall profile with plane P_1 , P_2 , P_3 and Z , and the angle α_1 , α_2 , α_3 and etching rate V_1 , V_2 , V_3 and u

Fig. 3. Etching planes and the track of their intersection

axis.

2.2 Quartz Etching Process A 100 μm Z cut quartz was used in this research. The etching process was as follow. (1) Washing quartz wafers in piraha solution ($\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2=3:1$) at 110 degrees C; (2) Sputtering Cr (50nm) and Au (180nm) mask on the quartz wafer; (3) Patterning the Cr and Au thin films with the designed photoresist mask; (4) Etching quartz wafer in saturated ammomium bifluoride solution at 87 degrees C. In this research, a stirrer was not used and no chemicals or water were supplemented.

2.3 Analysis Method

2.3.1 Definition of Etching Profile After substrate etching, samples were cut vertically to the etching line and cross sections were observed using scanning electron microscopy (SEM). Etching profile was defined in Fig. 2.

2.3.2 Track of the Intersection of Two Etching Planes

The appeared etching planes would advance parallelly and the

Fig. 4. Examples of SEM picture for measuring sidewall profiles, etching rates (a); etching undercut (b)

intersection of the etching planes is decided by the following formula (1)⁽⁸⁾. (Fig. 3)

$$R_\theta \sin \zeta = R_0 \sin(\zeta + \theta) \quad (1)$$

2.3.3 Observing and Calculating the Etching Profiles

Fig. 4(a) shows an example of the SEM picture for measuring the etching profile. Fig. 4(b) gives an example picture for measuring undercut. The undercut was clearly observed at the SEM photograph (Fig. 4(b)), the dark and bright part represent films with and without quartz, respectively.

Based on the Fig. 3, the etching rates of side wall P_1 , P_2 , P_3 and the Z plane can be calculated by the following formula (2), (3), (4) and (5), respectively.

$$V_1 = \frac{(W_1 + U) \cos \alpha_1}{t} \quad (2)$$

$$V_2 = \frac{(W_2 + U) \cos \alpha_2}{t} \quad (3)$$

$$V_3 = \frac{U \cos \alpha_3}{t} \quad (4)$$

$$V_z = \frac{D}{t} \quad (5)$$

V_1, V_2, V_3 : etching rate of side wall P_1, P_2 and P_3 respectively

V_z : etching rate of Z plane

U : etching undercut

t : etching time

$W_1 (W_2)$: distance between the intersection of $P_3 (P_2)$, Au/Cr mask

Fig. 5. Plotted etching profiles dependence on polar angle. Side wall inclination angle (a); Etching rate (b)

Fig. 6. SEM pictures of polar angle at 40° for observing the trench profile dependence on initial width and etching time

and the intersection of P_1 (P_2), Au/Cr mask

3. Results

3.1 Characterization of the Anisotropy The angle α_1 , α_2 , α_3 (in Fig. 2) were measured and plotted in dependence on the polar angle as Fig. 5(a). No minus inclination angle α was observed. We paid more attention on the angle α_3 , which tended to increase from $\beta=0^\circ$ to 15° and from $\beta=90^\circ$ to 120° , but also suddenly decrease from $\beta=15^\circ$ to 30° . In a wide range from $\beta=30^\circ$ to 90° , it appeared to be nearly 0° . In fact, it was difficult to measure and discriminate the angle in this range, because no bigger than 2° was observed. The side wall etching rates of V_1 , V_2 , V_3 were calculated and plotted as Fig. 5 (b). The etching rate V_Z was $110 \mu\text{m/h}$, which was the same at all polar directions. So we did not plot V_Z .

3.2 Trench Profile Dependence on Initial Width and Etching Time Fig. 6 shows the typical SEM pictures including all initial widths (5, 10, 20, 50, 100 and $200 \mu\text{m}$). These SEM pictures were used to study high aspect ratio microstructures.

3.3 High Aspect Ratio Microstructure For MEMS applications, high aspect ratio microstructures are required in

microdevices design. Aspect ratio was defined as depth/width. Structures at polar angel β should have a opposite shape with at polar angel $\beta+60^\circ$ because of the threefold symmetry property. So we just discussed the trench structures from $\beta=0^\circ$ to 60° . 40 min etched results are presented in Fig. 7.

According to the 40 min etching results (Fig. 7), we defined the basic microstructure as it is presented in Fig. 8 (a). The 20 min, 40 min and 70 min etched results with initial width $5 \mu\text{m}$ were plotted in Fig. 8 (c). The aspect ratio did not arise as the increase of etching time due to the disappearing of Z plane, P_1 or P_2 as shown in Fig. 6. Based on the 40 min results shown in Fig. 7, the trench profiles can be classified as (1) $\beta=0^\circ$ to 15° , in this range Z plane and P_1 disappeared rapidly and high aspect ratio structure can not be achieved because of the big angle α_3 (Fig. 5 (a)); (2) $\beta=15^\circ$ to 30° , P_2 plane appeared as bottom surface, but still no high aspect ratio structure can be achieved because of the low etching rate of V_2 (Fig. 5 (b)); (3) $\beta=30^\circ$ to 45° , α_2 increased gradually, but V_2 increased quickly. So high aspect ratio microstructure can be achieved in this area; and (4) $\beta=45^\circ$ to 60° , V_2 became bigger, but attention should be paid to the side B, where α_2 appeared gradually, and α_3 became bigger. At $\beta=60^\circ$, feature was achieved and

Fig. 7. Cross section SEM pictures from $\beta=0^\circ$ to 55° for 40 min etching with a initial width of $5\ \mu\text{m}$

returned to the $\beta=0^\circ$ feature. Fig. 8 (b) was an example SEM picture of 70 min etched trench with initial width $5\ \mu\text{m}$ at $\beta=45^\circ$.

4. Discussion and Applications

Double-sided etching experiment was also performed using the same mask pattern. The result showed that the bottom side has the same etching profile at the opposite polar direction (β) due to the twofold symmetry in the positive X axis (Fig. 9).

4.1 Application for Microcapillary

In the case of

microchannel for microcapillary, rectangular channel should be ideal, because slope side wall decreases the UV light pass length, resulting in low sensitivity. Considering the side etching (u_1, u_2), side wall slope α , and bottom surface etching rate V_2 , $\beta=45^\circ$ should be the best choice. Fig. 8 (b) shows the 70 min etched trench with an aspect ratio of 3.1 at $\beta=45^\circ$.

4.2 Application for Capacitive Tilt Sensor

For capacitance based sensor, quartz wafer should be etched through for achieving movable part and static part, so called

Fig. 8. Definition of the basic microstructure and aspect ratio: (a) the basic microstructure, u_1 , u_2 , and D_b represent side etch and etch depth respectively; (b) SEM photograph of 70 min etched trench at 45° ; (c) Plotted trench aspect ratio $[AR=D_b/(\text{initial width}+u_1+u_2)]$ dependence on the polar direction based on the 20, 40, 70 min etching results

Fig. 9. The twofold symmetry around X axis

comb-electrode. The key feature of this kind of sensor is to fabricate high aspect ratio through-hole, which is used for constructing variable capacitor. Double-sided etching technique shows great interest for the twofold symmetry. We have developed a tilt sensor⁽⁹⁾ using +X direction ($\beta=0^\circ$) for the high etching rate of V_1 (both sides of up and down) and simple side wall profile (up-down symmetry). The sensor is composed of comb-electrode, mass proof and long elastical cantilever. Fig. 9 shows the pictures of the fabricated tilt sensor.

In general consideration, for designing two axis sensor on one chip, sensor 2 will be arranged 120° to the sensor 1 (+x direction) for the well-known threefold symmetry property. However, 90°

Fig. 10. Fabricated capacitive tilt sensor using +X direction

Fig. 11. Cross section of double-sided etched high aspect ratio ($AR=5$) through-hole at $\beta=15^\circ$, 90 min

arranged two axis sensor should also be achieved, if we let β satisfy the following formula (6) taking advantage of the twofold symmetry around X axis (Fig. 9).

$$\beta - (-\beta) = 90^\circ \quad (6)$$

Namely, at $\beta=45^\circ$ and -45° same two sensors can be achieved, which just have up-down counter etching profiles. Further, considering the threefold symmetry property, two same sensors can also be achieved if satisfying the following formula (7), which gives $\beta=15^\circ$, arranging two sensors at $\beta=15^\circ$ and 105° ($-15^\circ+120^\circ$).

$$120 - \beta - \beta = 90^\circ \quad (7)$$

Actually, through-holes at $\beta=45^\circ$ and 105° have the same etching profiles, which are only left-right counter. In conclusion, there is only one kind of through-hole etching profile which can be used for fabricating 90° arrangement two axis tilt sensor. Fig. 11 shows the cross section of double-sided etched high aspect ratio through-hole at $\beta=15^\circ$, which was etched with an initial width $5 \mu\text{m}$ for 90 min.

5. Conclusion

High aspect ratio microstructure (> 3) which was considered impossible by wet etching technology⁽³⁾ was achieved. This result suggests that anisotropic etching of Z cut α -quartz can be applied to microcapillary electrophoresis, where high aspect ratio microchannel is desired for improving detecting sensitivity. The dependence of aspect ratio on polar angle would give important guidance to the design layout. Trench at polar angle of $\beta=45^\circ$ is demonstrated to be the best choice. We successfully fabricated nearly vertical trench feature at $\beta=45^\circ$ with $50.7\ \mu\text{m}$ depth and $16.4\ \mu\text{m}$ width, the aspect ratio of 3.1. Double-sided etching showed great interest for the twofold symmetry around the X axis. According to this property, two same sensors at ($\beta=15^\circ$ and 105°) and ($\beta=-45^\circ$ and 45°) can be arranged at 90° on one quartz chip, which propose a novel two axis tilt sensor.

Acknowledgement

This research has been supported by the Grant-In-Aid of Fukuoka Industry, Science and Technology Foundation (IST).

(Manuscript received Jul. 24, 2006, revised Jan. 4, 2007)

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