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Etching characteristics of Si{110} in 20 wt% KOH with addition of hydroxylamine for the fabrication of bulk micromachined MEMS

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Abstract

Anisotropic wet etching is a most widely employed for the fabrication of MEMS/NEMS structures using silicon bulk micromachining. The use of Si{110} in MEMS is inevitable when a microstructure with vertical sidewall is to be fabricated using wet anisotropic etching. In most commonly employed etchants (i.e. TMAH and KOH), potassium hydroxide (KOH) exhibits higher etch rate and provides improved anisotropy between Si{111} and Si{110} planes. In the manufacturing company, high etch rate is demanded to increase the productivity that eventually reduces the cost of end product. In order to modify the etching characteristics of KOH for the micromachining of Si{110}, we have investigated the effect of hydroxylamine (NH_2OH) in 20 wt% KOH solution. The concentration of NH_2OH is varied from 0 to 20% and the etching is carried out at 75 °C. The etching characteristics which are studied in this work includes the etch rates of Si{110} and silicon dioxide, etched surface morphology, and undercutting at convex corners. The etch rate of Si{110} in 20 wt% KOH + 15% NH_2OH solution is measured to be four times more than that of pure 20 wt% KOH. Moreover, the addition of NH_2OH increases the undercutting at convex corners and enhances the etch selectivity between Si and SiO_2.

Keywords: Wet anisotropic etching, Bulk micromachining, Silicon, Corner undercutting, KOH, MEMS, Hydroxylamine (NH_2OH), TMAH, Si{110}, Etching characteristics

Background

Wet anisotropic etching is a main process of silicon bulk micromachining for the fabrication of different types of microstructures (e.g. cantilever, diaphragm, cavity, etc.) [1–5]. It is a low cost technique and suitable for batch process. Potassium hydroxide (KOH) and tetramethylammonium hydroxide (TMAH) are the two main etchants used for wet anisotropic etching-based silicon bulk micromachining [6–12]. These etchants are thoroughly investigated under various etching conditions. KOH is preferred over TMAH to achieve high etch selectivity between {111} and {110}/{100} planes. In the fabrication of microstructure using etching process, etch rate is a key parameter in manufacturing as it influences production rate which eventually affects the cost of final product. Mostly high etch rate is desirable to reduce production cost. An etchant with high etch rate may also provide high etch selectivity between silicon and mask layer, which is very useful to use same thickness mask layer for prolonged etching. The important parameters which affect the etch rate and surface morphology are etchant concentration, etching temperature, ultrasonication/microwave irradiation during etching, and the addition of different kinds of additives to etchant. Each method has its own benefits and drawbacks. Maximum etch rate of Si{100} and Si{110} is obtained in KOH with a concentration range from 15 to 25 wt% [8–11]. Different kinds of additives (e.g. redoxsystem or complexants, oxidizing agent, surfactants, and metal impurities) [13–17] are

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added into KOH to get high speed etching and the alcohols/surfactants are incorporated to improve the surface morphology [18–24]. Moreover, etching at the boiling point of the etchant [11, 25], microwave irradiation of the etchant [26], ultrasonic agitation of the etchant [27] have been employed to increase the etch rate. The ultrasonic method may rupture the fragile structures and microwave irradiation technique causes irradiation damage to the structures. Anisotropic etching at very high temperature (e.g. boiling point of etchant) is not preferable as it affects the anisotropy and the etch selectivity between SiO_2 and silicon. Therefore, it is required to improve the etching characteristics by adding some additives.

In all kinds of wet anisotropic etchants, Si{111} planes exhibit minimum etch rate. If the mask edges are aligned along the directions comprises {111} planes, wet anisotropic etching provides microstructures with smooth sidewalls due to the emergence of {111} planes at these directions. The angle between sidewall and wafer surface depends on the wafer orientation. Moreover, the number of directions along which {111} planes appear depend on the orientation of wafer surface. In the case of {100} wafer, four {111} planes making an angle of 54.7° with wafer surface expose at ⟨110⟩ directions. Hence {100} wafer is suitable to fabricate rectangular shaped cavities or suspended structures over rectangular shape cavity using wet anisotropic etching [3, 6, 12, 21]. In the case of the wafer with {110} surface, two slanted planes making an angle of 35.5° with wafer surface and four vertical planes with respect to wafer surface appear along ⟨110⟩ and ⟨112⟩ directions, respectively. Therefore, in order to fabricate microstructures with vertical sidewalls {110} wafer is a most appropriate choice [10, 28–34]. It can be used to fabricate deep channels/cavities with vertical sidewall.

In this paper, we have studied the etching characteristics of {110}-oriented silicon wafer in 20 wt% KOH solution without and with addition of NH_2OH. It is mainly focused to investigate the effect of NH_2OH in 20 wt% KOH to achieve improved etching characteristics for applications in silicon bulk micromachining for the formation of MEMS structures.

Experimental details

In this work, 4-in. diameter {110}-oriented p-type doped Czochralski-grown silicon wafers with 5–10 Ω-cm resistivity are used. One micron thick oxide layer grown by thermal oxidation process has been used as mask to protect unwanted places from etching. Photolithography method is used to pattern the thermal oxide layer. After transferring the mask pattern on photoresist layer, oxide etching is employed in buffered hydrofluoric acid (BHF). Thereafter, wafer is rinsed in DI water followed by removal of photoresist using acetone. After patterning of oxide layer, wafer is diced into small chips. 20 wt% KOH (99.99%, Alfa Aesar) is used as main etchant, while hydroxylamine solution (NH_2OH) is used as an additive solution. In order to investigate the effect of NH_2OH on the etching characteristics of KOH, various concentrations of NH_2OH (5, 10, 15 and 20%) added into KOH solution. Experiments are performed in one litre etchant at 75 ± 1 °C. In order to maintain the constant temperature of the etchant during etching process, a constant-temperature water bath is used. Teflon made cylindrical container equipped with reflux condenser is used to avoid change in etchant concentration due to evaporation. PFA made chip holder with multiple slots is used to hold samples in order to etch many samples at a time under same etching conditions. Before starting experiment, samples are cleaned in piranha bath (1:1; H_2O_2:H_2SO_4) to remove organic impurities. This step is followed by thorough rinse in DI water. The presence of very thin oxide layer (i.e. native oxide) delay silicon etching. Therefore, prior to dipping the samples into etchant, the samples are immersed in 1% HF for 30 s followed by DI water rinse. After silicon etching process, etch depth, surface roughness and undercutting are measured using 3D measuring laser microscope (Olympus, OLS4000). Moreover scanning electron microscope (SEM) is employed to inspect etched surface morphology. The thickness of oxide layer after different times of etching is measured using ellipsometry. In this work, mask patterns with simple shapes such as cantilever, parallelogram/rhombus shapes are used to determine different etching characteristics, e.g., etch rate, surface morphology, undercutting at convex corners. Rhombus shape geometries (mesa and opening) are used to measure the etch rate, surface morphology, and undercutting at convex corners, while cantilever patterns are used to demonstrate the application of proposed etchant for the fabrication of suspended MEMS structures. The dimensions of the parallelograms and cantilever shape patterns vary from 300 µm × 300 µm to 1000 µm × 1000 µm and 50 µm × 100 µm to 200 µm × 400 µm, respectively.

Results and discussion

Etching characteristics of Si{110} including etch rate, surface roughness/morphology, and undercutting at convex corners are studied on the samples etched in pure and NH_2OH-added 20 wt% KOH. To study etching characteristics, the concentration of NH_2OH is varied from 5 to 20% in steps of 5%. Detailed descriptions of these characteristics are presented in following subsections.

Etch rate

Etch rates of Si{110} in pure and various concentrations of hydroxylamine (NH_2OH) added 20 wt% KOH solution are presented in Fig. 1. The etch rate increases with increase of NH_2OH concentration up to 15% and starts decreasing if the NH_2OH concentration is further increased. The etch rate in 15% NH_2OH-added KOH is four times more than that in pure 20 wt% KOH. The standard deviation indicated by error bars is calculated by taking six measurements on the same sample at different locations. The mechanism behind the increase of etch rate in NH_2OH-added KOH may be interesting finding, but it is not the main focus of the present work. The major objective of this work is to investigate the effect of NH_2OH to achieve high etch rate and undercutting to promote the application of wet anisotropic etching in MEMS fabrication. In several studies, it has been claimed that the etch rate of an etchant increases if its wettability improves such as the wettability of KOH increases when anionic additives are added [35, 36]. In the case of NH_2OH-added KOH solution, NH_2OH and OH^- ions participate in the reaction to forms NH_2O^- ions and water until it reaches in chemical equilibrium [37]. NH_2O^-, OH^- and water are active species in NH_2OH-added alkaline solution. The etching mechanism in KOH solution is well known. First step of etching process is the chemical oxidation in which hydrogen-terminated silicon (Si–H) becomes a hydroxyl-terminated silicon atom (Si–OH) [38]. It is a slow process. We speculate that in NH_2OH-added KOH solution H of Si–H is replaced by NH_2O^-. After becoming OH^- or NH_2O^- terminated, the Si–Si backbonds exhibit significant polarity due to the large electronegativity of O that results in weakening of the backbonds which are easily attacked by the polar water molecules, leading to the removal of silicon atom as a $Si(OH)_4$ product. Due to the more electronegativity of oxygen in NH_2O^- than in OH^-, the polarization and weakening of the backbonds in Si-ONH_2 is more prominent. Thus silicon backbonds are speedily hydrolysed (or attacked by water) in NH_2OH-added KOH. In addition to H_2O, NH_2O^- and OH^- ions, other intermediate compounds of self-decomposed NH_2OH in the solution may also participate in the reaction to dissolve silicon [37, 39]. Thus etching in NH_2OH-added KOH proceeds at a faster rate in comparison to that in pure KOH. Increasing concentration of NH_2OH above 15% decreases etch rate. It may be because of the decrease in NH_2O^- and OH^- ions concentration and/or decrease in freely available water due to the formation of clusters (K^+, OH^-, NH_2O^-, etc.) [40, 41].

Etch selectivity with thermal oxide

Silicon dioxide is extensively used in silicon micromachining as etch mask layer to create various kinds of grooves and cavities. Moreover, it is employed as structural layer for the fabrication of MEMS structures such as cantilever. It can easily be grown by various techniques such as thermal oxidation, chemical vapor deposition (CVD), and anodic oxidation [42, 43]. However thermal oxidation is most widely used as it provides excellent quality oxide layer and interface. Oxide layer is usually patterned by photolithography followed by oxide etching in HF/BHF. The etch rate is calculated by measuring the oxide thickness at different locations on the same sample after different times of etching. Figure 2 shows the etch rate of SiO_2 in 20 wt% KOH with varying concentration of NH_2OH. It can be noticed that the oxide etch rate increases with increase of NH_2OH concentration. In the fabrication of MEMS structures using silicon wet anisotropic etching based bulk micromachining, the etch selectivity between silicon and mask/structural layer (e.g. SiO_2) is an important concern. It is defined as the

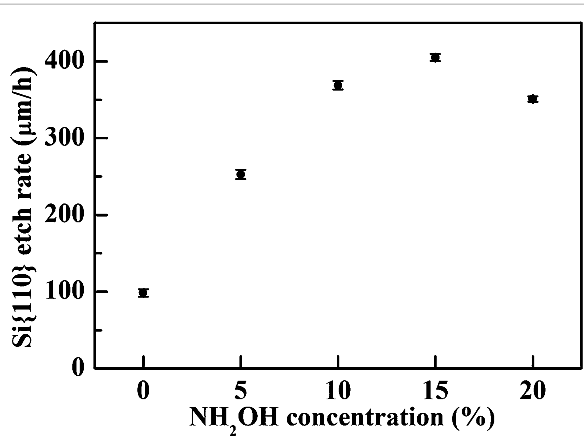

Fig. 1 Etch rate of Si{110} in pure and various concentrations of NH_2OH-added 20 wt% KOH solution at 75 °C

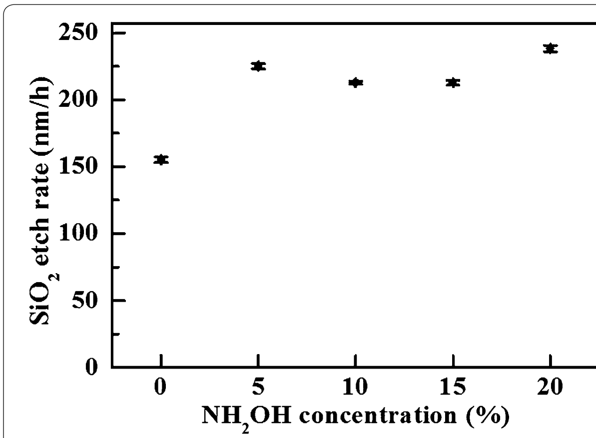

Fig. 2 Etch rate of thermal silicon dioxide (SiO_2) in pure and different concentrations of NH_2OH-added 20 wt% KOH at 75 °C

ratio of the etch rate of silicon to that of SiO_2. If an etchant provides high etch selectivity, mask/structural layer can be exposed in the etchant for a longer time, which is needed to fabricate deep cavities/grooves or freestanding structures (e.g. cantilever) on silicon wafer. The etch selectively between Si{110} and SiO_2 calculated using the results of Figs. 1 and 2 is presented in Fig. 3. It can easily be noticed in Fig. 3 that the etch selectivity increases significantly with increase of NH_2OH concentration. In other words, we can say that the addition of NH_2OH to KOH solution considerably improves the etch selectivity between Si{110} and SiO_2. Hence it can be concluded that the same thickness oxide layer in NH_2OH-added KOH can be used to form larger depth cavities and grooves in comparison to pure KOH. In addition to that oxide layer can be used as structural layer to fabricate freestanding structures using NH_2OH-added KOH as anisotropic etchant.

Etched surface morphology

Surface morphology is one of major concerns in optical MEMS applications and designing high-efficiency solar cell, etc. Etched surface morphology primarily depends on the etchant type, etchant concentration, etching temperature, additives, and agitation of etchant during etching process. The average etched surface roughness decreases with the increase of etching temperature and KOH concentration [11, 25, 44, 45]. Surface roughness of the samples etched in 20 wt% KOH solution without and with addition of various concentrations of NH_2OH is measured using 3D measuring laser microscope (Olympus, OLS4000) and presented in Fig. 4. The standard deviation indicated by error bars is calculated by taking six measurements on the same sample at different locations. Figure 5 presents SEM images of the etched

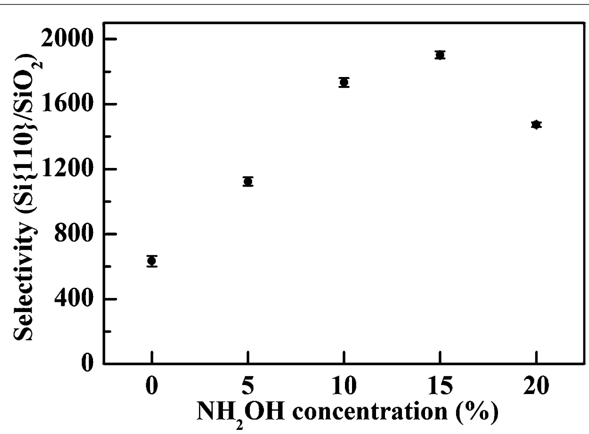

Fig. 3 Etch rate selectivity between Si{110} and silicon dioxide (Si{110}/SiO_2) in pure and NH_2OH-added 20 wt% KOH at 75 °C

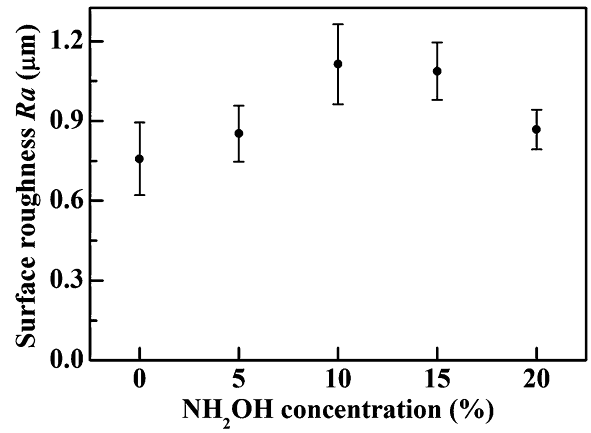

Fig. 4 Average surface roughness (*Ra*) in 20 wt% KOH solution without and with addition of NH_2OH solutions at 75 °C

samples corresponding to surface roughness shown in Fig. 4. Average surface roughness (*Ra*) of the samples etched in NH_2OH-added 20 wt% KOH solution is nearly same as those are etched in pure 20 wt% KOH. It means that the etched surface of Si{110} is not affected significantly when NH_2OH is added into KOH solution. Main cause of surface roughness in the wet etching process is micromasking by the hydrogen bubbles and/or impurities on the surface during the etching process [24, 44–46].

Undercutting at convex corner

In silicon wet anisotropic etching, the corners formed by the intersection of {111} planes are termed as concave and convex corners. These corners are produced by the intersection of ⟨110⟩ and ⟨112⟩ directions on Si{110} surface as these directions contain {111} planes which expose during wet anisotropic etching. Although both types of corners (concave and convex) are shaped by the intersection of {111} planes, they have opposite etching characteristics. Concave corners do not encounter any kind of undercutting, while convex corners face severe undercutting, depending on the type of etchant, in all kinds of alkaline solutions [46–52]. Si{110} wafer is a primary choice when the microstructures with vertical sidewalls formed by {111} planes are fabricated using wet anisotropic etching [28–34, 52]. These vertical sidewalls appear at ⟨112⟩ directions which form a rhombus shape structure containing two types of convex corners (acute and obtuse corners) as presented in Fig. 6. Undercutting rate (undercutting length along ⟨112⟩ direction/etch time) and undercutting ratio (undercutting length along ⟨112⟩ direction/etch depth) at both types of convex corners as a function of NH_2OH concentration are presented in Fig. 6. Undercutting rate increases as the concentration of NH_2OH increases up to 15% NH_2OH and is

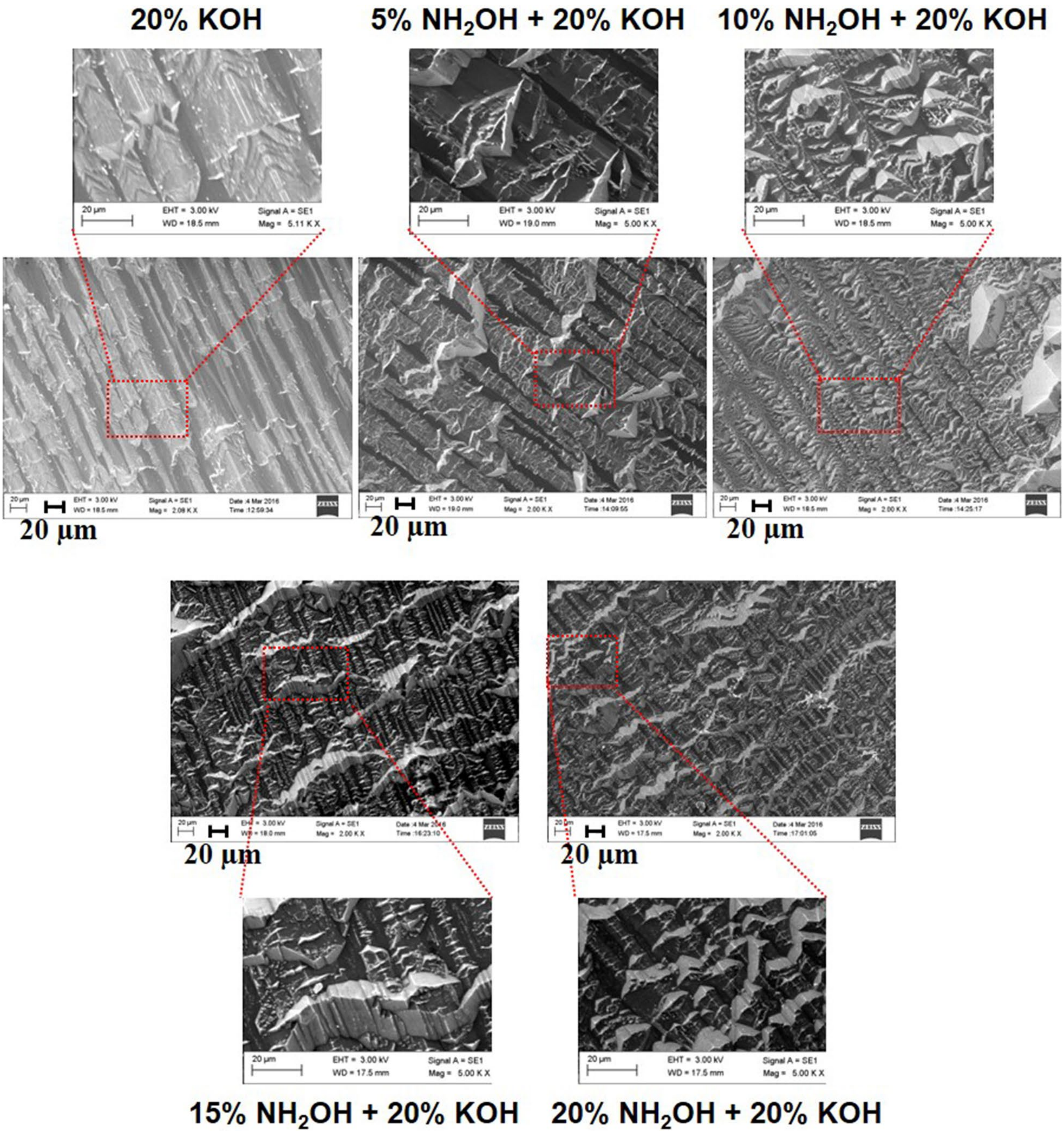

Fig. 5 SEM photographs showing the surface morphology of the samples etched in 20 wt% KOH solution without and with addition of NH_2OH at 75 °C

around four times more than that in pure 20 wt% KOH. The undercutting at convex corners takes place mainly due to the emergence of high index planes [29, 46–52]. The main reason behind the increase in undercutting is the increase of the etch rate of high index planes appearing at convex corners during etching process.

In the fabrication of suspended MEMS components (e.g. cantilever beams), underneath material is removed by undercutting process. Hence high undercutting is desirable for the fast removal of underneath material to release the microstructures. In the present research NH_2OH-added KOH provides high undercutting at

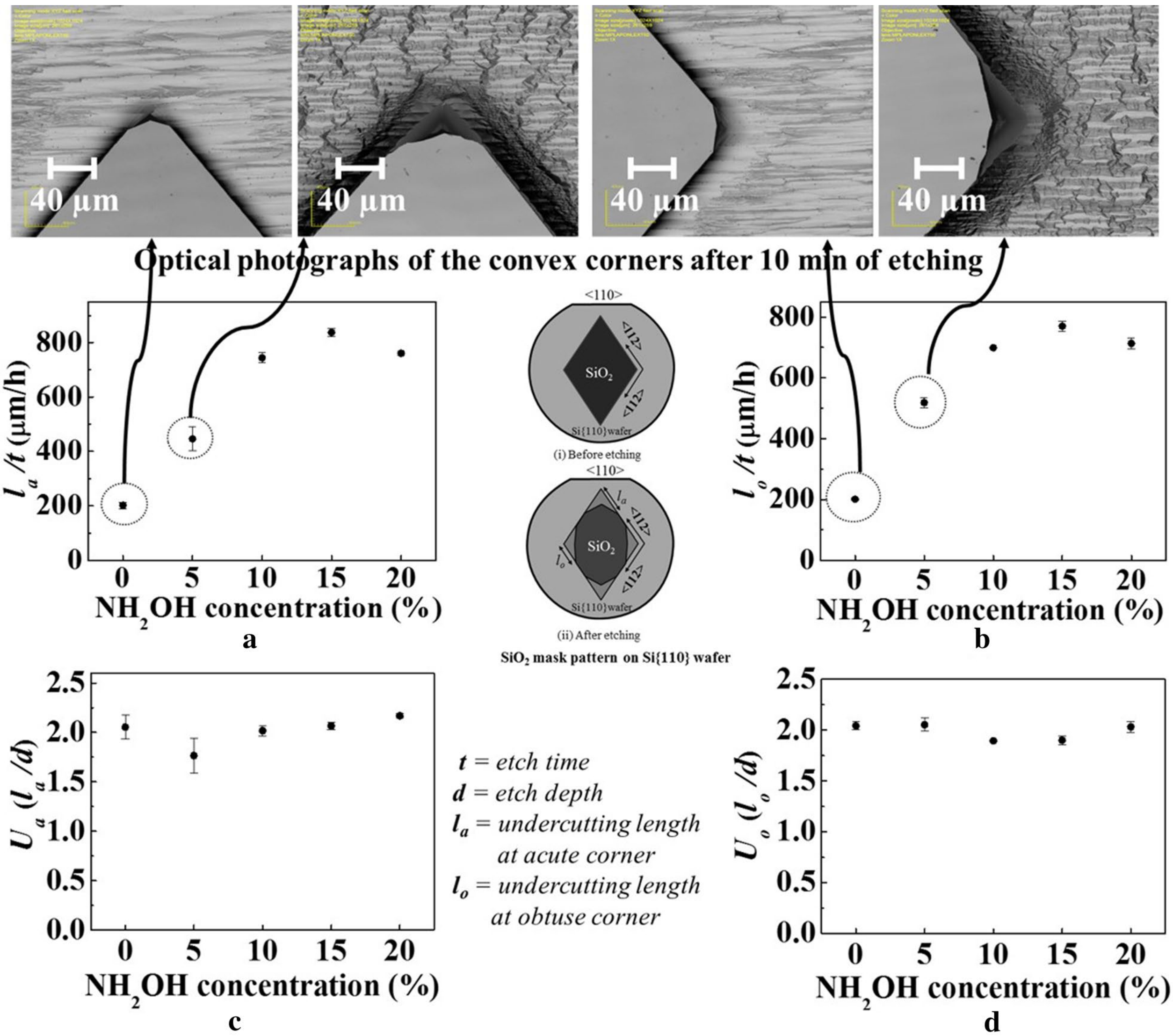

Fig. 6 Undercutting at acute and obtuse corners of a rhombus shape structure formed by ⟨112⟩ directions on Si{110} wafer in pure and various concentrations of NH_2OH-added 20 wt% KOH solution: undercutting rate (*l*/*t*) at **a** acute and **b** obtuse corners; undercutting ratio (*l*/*d*) at **c** acute and **d** obtuse corners

convex corner and therefore it is very useful for the fabrication of freestanding structures. In addition to that it exhibits high etch selectivity between Si{110} and SiO_2 as presented in Fig. 3 and explained in "Etch selectivity with thermal oxide", which is required for the fabrication of SiO_2 microstructures. To demonstrate the application of high undercutting for the realization of suspended structures for MEMS, silicon dioxide cantilever beams are fabricated in 15% NH_2OH-added 20 wt% KOH solution. Figure 7a, b present the schematic views of patterned oxide layer on Si{110} and released cantilever beam after anisotropic etching, respectively. Optical and SEM images of freestanding cantilever beam are presented in Fig. 7d, e, respectively. The fabrication of oxide cantilever beam indicates that the NH_2OH-added KOH exhibits high etch selectivity between silicon and silicon dioxide. Pure KOH provides very low etch selectivity between Si{110} and SiO_2 in comparison to NH_2OH-added KOH as presented in Fig. 3. Due to this reason, oxide layer cannot survive for a longer time in pure KOH solution and therefore it cannot be used as structural layer for the fabrication of freestanding structure. Hence we can say that NH_2OH-added KOH is a suitable wet anisotropic etchant for the fabrication of SiO_2 microstructures.

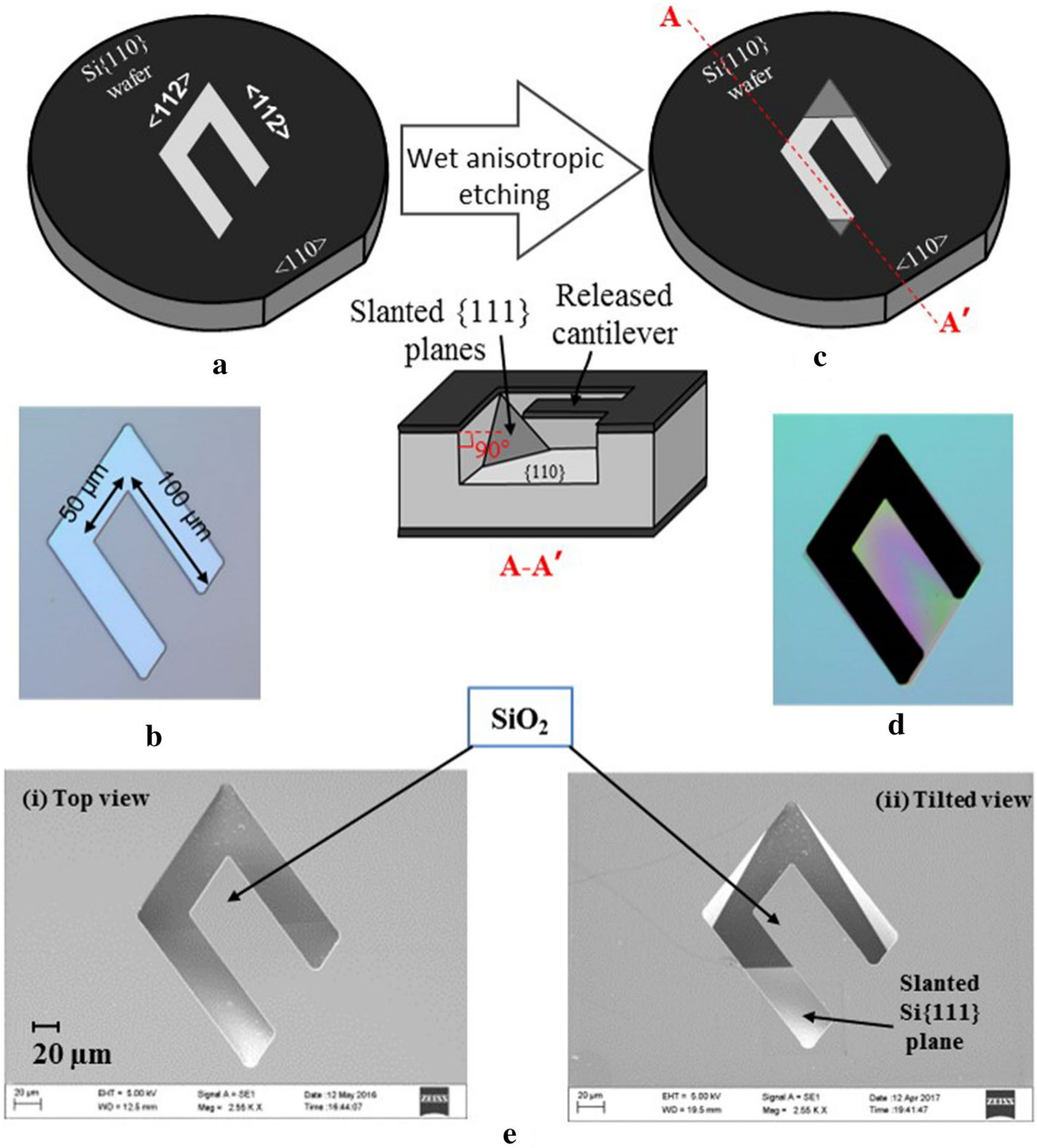

Fig. 7 Silicon dioxide cantilever beam fabricated in NH_2OH-added KOH solution: **a** patterning of oxide layer using photolithography and oxide etching, **b** optical image of cantilever beam patterned in oxide layer, **c** wet anisotropic etching to release the beam, **d** optical and **e** SEM images of released cantilevers

Conclusions

Etching characteristics of Si{110} surface in pure and different concentration of NH_2OH-added 20 wt% KOH are studied for applications in silicon wet bulk micromachining. The etching characteristics of KOH solution are changed drastically when NH_2OH is added. The etch rate and undercutting are improved significantly. High etch rate is very useful to achieve larger etch depth in less time in comparison to common etchant. Increase in undercutting at convex corner is beneficial for the fast release of the microstructures. Both these characteristics are indispensable for reducing etch time and therefore useful for industries to increase the productivity. Moreover, NH_2OH-added KOH provides high etch selectivity between silicon and oxide (i.e. Si/SiO_2) in comparison to pure KOH. High etch selectivity can be exploited for the fabrication of MEMS structures using silicon dioxide as mask/structural layer. It can be concluded that the results presented in this paper are highly useful for research and industrial applications.

Authors' contributions
AVNR and VS did experiments. AVNR and PP wrote the manuscript. All authors read and approved the final manuscript.

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Competing interests
The authors declare that they have no competing interests.

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