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Micromirror angle dependence with etchant choice on <100> silicon via wet etching

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***Abstract*—In creating mirrored silicon structures for micro-optics, the smoothness of the surface and etch rate are crucial parameters. We demonstrate a method of creating both 45° and 90° etch-planes from monocrystalline silicon for use as retro-reflective sidewalls in a microfluidic device. The technique uses the same photolithographic pattern orientation, but with two different etchants. Etching on <100> direction in Si(100) with potassium hydroxide (KOH) gives vertical surfaces (where e.g. the high surface tension influences etching of crystallographic silicon planes), whilst tetramethylammonium hydroxide (TMAH) gives 45° sidewalls . We illustrate the use of these fabricated structures by creating arrays of micromirrors that enable an optical beam to be reflected parallel back and forth from 45° and -45° tilted vertical structures. This device has potential uses in optofluidic spectroscopic applications, where there is a need to increase the effective pathlength of a beam through a sample whilst keeping the device as small as possible.

I. INTRODUCTION

There is an increasing interest in creating platforms where optics can be integrated with fluid movements, particularly in the field of optofluidics for analytical measurement. In such devices optical components need to be fabricated at precise locations close to or aligned with microfluidic channels or cavities. Silicon micromachining and MEMS processing technologies both lend themselves well to such applications enabling complex microstructures to be created that can shape or direct optical beams.

Previously silicon bulk micromachining technology has been developed and is commonly carried out using a wide variety of different etches, the most common of which is the wet anisotropic etch of crystalline wafers for the fabrication of a wide variety of the microstructures (including e.g. microcavities, cantilevers, diaphragms, etc.) [1], [2], [3]. In addition these same etches are also integral to the manufacture of various integrated devices such as accelerometers [4], beam splitters [5], transistors [6] and comb structures [7]. The development of optics apparatus has recently been stimulated by the ability to use MEMS-micromachining to produce arrays of micromirrors which afford an optical 90° reflection of light [8]. The technique is both low-cost and scalable and is therefore suitable for large scale batch processing. The resulting surface morphology and etch rate depend on etchant concentration, additives, solution temperature and mass transfer/convection (stirring) speed [9]. In order to enhance the surface morphology, surfactants and alcohols are often combined and added to the etchant to produce a suitable process [10], [11], [12], [13], [14], [15]. Potassium hydroxide (KOH) and tetramethylammonium hydroxide (TMAH) are the two main etchants, commonly used for such bulk silicon micromachining [12], where KOH is often used to attain a higher etch selectivity between {111} and {110}/{100} planes than is the case for TMAH [16].

Anisotropic silicon wet etching using a Si(100) wafer has previously been shown to achieve vertical sidewalls using KOH [17], although the surfaces were not smooth enough to produce mirrors. Similarly 45° slanted smooth sidewalls in Si(100) have been created [18] using TMAH with a variety of different additives, concentrations, and stirring speeds. The angle between etched sidewall and the wafer surface depends on the wafer crystal structures and photopattern orientation. In all cases, the formation of a preferred sidewall plane relies on the surface tension which varies both with concentration and temperature of the etchant mixture [19]. For example, adding *tert*-butanol decreases the surface tension [20], making smoother sidewall as it decreases the etch rate of both {100} and {110} planes [21]. In order to achieve vertical sidewalls using Si(100), a surface tension (>74mN/m) should be used in the etching process [19], allowing etching of {110} plane until reaching the vertical plane {100}, shown in Fig. 1.

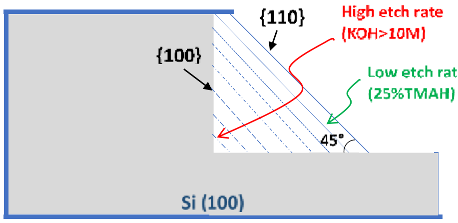

Fig. 1. Schematic representation of dependence of solution's etch rate on preferential formatted planes of Si(100) in aqueous TMAH and KOH etchants.

We show that by adding a low percentage of tert-butanol (0.1M) to achieve smooth (or mirrored) sidewall whilst maintaining a high surface tension it is possible to form 90° sidewalls at high KOH concentrations (10M). We subsequently demonstrate that we can attain both 90° and 45° smooth sidewalls from the same silicon cut and pattern orientation (mask aligned 45° to <110> direction) for the formation of MEMS structures in bulk silicon micromachining.

II. MATERIALS AND METHODS

Etching experiments with KOH and TMAH solutions were conducted using 3'', 4'', and 6'' (100) silicon wafers of 380, 500, and 659 µm thickness respectively, supplied by Chalmers Nanofabrication Laboratory, University Wafer, and James Watt Nanofabrication Centre (JWNC)/UK respectively. The 3'' wafer was double sided polished and has nitride layer over thermal oxide layer on both sides of 60/40 nm respectively in order to obtain masking window. 4'' and 6'' wafers had100 nm LPCVD-nitride layer masking material. The wafers were patterned in photolithography on one side, which was followed in the process-flow by dry etching of the nitride and/or thermal oxide layer to give a desired pattern. Subsequently, after removal of the photoresist from the hard mask, selective etching in BHF (10:1 buffered hydrofluoric acid) solution for one minute was used to remove the native oxide before etching process, it rinsed thoroughly with deionised water and dried with N2.

The KOH solution was prepared from potassium hydroxide pellets, supplied by Sigma-Aldrich, and deionised water containing 0.1M *tert*-butanol alcohol was supplied by Sigma-Aldrich. For TMAH etching, 25% TMAH supplied by KMG Ultra Pure Chemicals used with Triton X-100 surfactant (Sigma-Aldrich). During etching, the samples were maintained vertically to promote the detachment of hydrogen bubbles during etching by a Teflon holder immersed in the etching solution of the volume 1500 ml, contained in a glass vessel.

The etching processes were carried out under atmospheric pressure and at stabilized 80°C temperature. Stirring of the etching solution was carried out mechanically at a fixed rate of rotation using IKA Laboratechnik stirrer with a Teflon paddle. The Teflon paddle was fully immersed in the solution during etching, although it was located above the sample holder. The process was performed in a condenser to control the evaporation over a time. The etching time for fabrication of micromirror structures was selected for each structure so that the etching rate was varied. The surface morphology, roughness, sidewall angle, and etch depth of the etched spatial microstructure were investigated using SEM (scanning electron microscope).

In order to achieve 45° and 90° slanted sidewalls, the mask patterns were aligned in the <100> direction in Si(100) wafer which is essentially at 45° to the <110> wafer flat [12], as shown in Fig. 2.

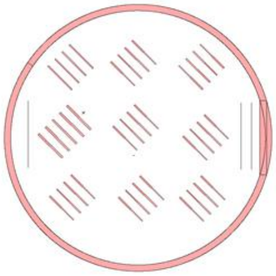

Fig. 2. Photomask design of 45° tilted trenches to the flat edge <110> wafer flat, i.e. <100> direction in Si(100).

The crystallographic property of silicon comprises {110} wall at the <100> direction and this {110} wall was inclined at 45° to the wafer surface ({100} surface). In an anisotropic wet etching process, the essential condition was to ensure that etch rate at the {110} plane remained the lowest for the planes between {100} and {110} planes. When this silicon orientation is wet etched, it gives {111} sidewalls owing to its having the slowest etch rate (the etch rate of {100} and {110} is high). However, this property can be tuned to ensure that the {110} plane is the slowest etching plane and not {100}. For this, a surfactant is used (e.g. addition of Triton to TMAH based etchant). This reduces the etch rate of {110} planes and since there are no {111} planes at the <100> direction, the etching process will give 45° walls.

The order of the rate of etching by aqueous KOH solution for different crystal planes is {110}>{100}>{111} [16]. When KOH:*tert*-butanol mixture was used , the relative etch rates were modified to {100}>{110}>{111} for the different crystal planes [21]. In this case, the etching process will give 90° sidewalls.

III. RESULTS AND DICUSSION

In our experiment, we used 25% TMAH with 0.1% Triton X-100 with a stirring speed of 130 rpm and 80°C. Smooth 45° sidewall were attained at an etch rate of 0.43 µm/min, Fig. 3.

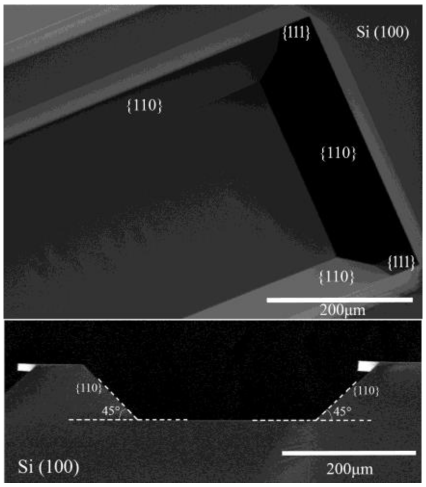

Fig. 3. SEM micrographs of etched grooves ending with 45° sidewalls of 45° tilted trenches mask fabricated with the 25%

TMAH and 0.1% Triton (groove depth ~92 µm) of Si(100) substrate which has SiO2 and SiNx mask layers.

When *tert*-butanol was added to high concentrated KOH (10M) in the solution, {110} plane was etched and reached {100} plane (90° sidewall), due to the effect of surface tension as indicated in Sec. I, with a stirring speed of 130 rpm and 80°C at an etch rate of 0.96 µm/min, Fig. 4.

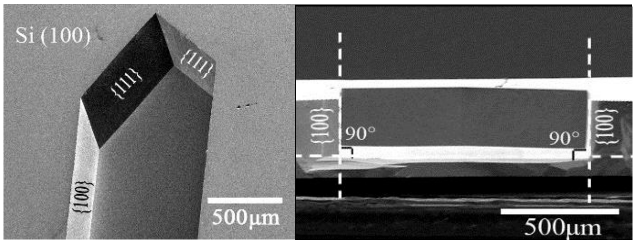

Fig. 4. SEM micrographs of etched grooves ending by vertical sidewalls of 45° tilted trenches mask with respect to the flat edge. Groove depth is ~250 µm, fabricated in 10M KOH and 0.1M *tert*-butanol on Si(100) substrate which has SiO2/SiNx mask layers.

For lower KOH concentrations (low surface tension), i.e. when using 8M, with same concentration of *tert*-butanol there is a remaining artefact of {110} planes along with {111} planes, as depicted in Fig. (5).

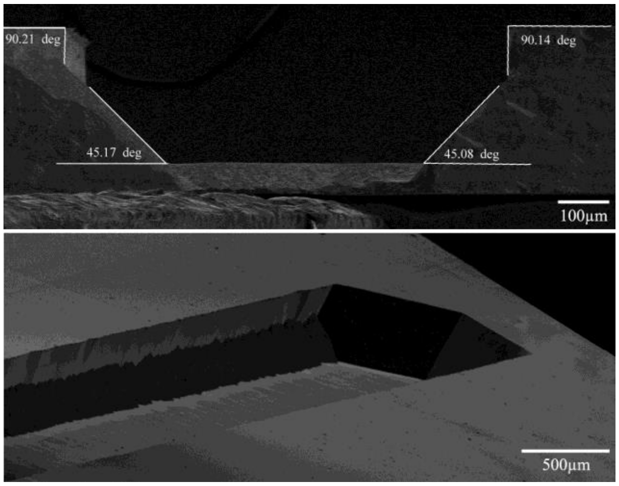

Fig. (5): SEM micrographs of etched grooves ending by ~45° and 90° sidewalls fabricated by 45° tilted trenches mask with respect to the wafer flat edge with the 8 M KOH and 0.1 M *tert*-butanol of Si(100) substrate which has SiO2/SiN mask layers.

IV. APPLICATION OF DEEP VERTICAL MICROMIRRORS TO ATTAIN 45° CAVITIES

Vertical sidewalls with patterns inclined 45° and -45° were fabricated for applications in multipass micromirrored cavities. Smooth 45° micromirrors can be obtained with high etching rate by taking advantage of the resulting smooth vertical sidewalls due to the high surface tension micromachining processes of <100> direction in Si(100), see Fig. 4. The advantage of this process is evident in the ability to achieve deep, smooth 45° micromirrors with high etch rates when compared to those fabricated using TMAH.

Structures where the sidewalls form multiple mirrors for bringing parallel optical beams through a cavity were designed. The structure in Si(100) substrate was etched in 10M KOH mixed with 0.1M *tert*-butanol. The fabricated micromirror array cavity are presented in Fig. 6 (a). The edges between two {110} planes forming the mirrors were not sharp after the etching process due to the formation of the {111} during the etch process. The structure can serve as a cavity or waveguide via retroreflection at an angle 90° parallel to the substrate, Fig. 6 (b).

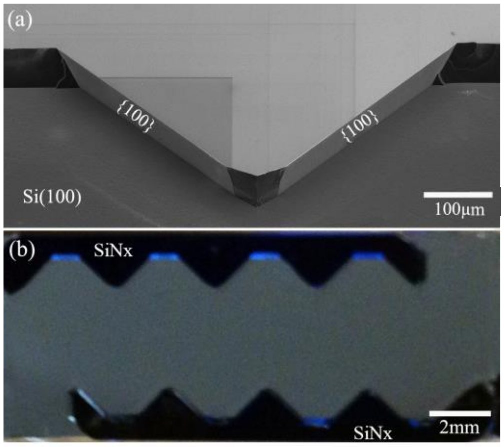

Fig. 6. (a) SEM micrograph of etched walls ending by 90° sidewalls with 45° and -45° tilted pattern with respect to the wafer flat edge of Si(100) substrate which has SiO2/SiNx mask layers using 10 M KOH and 0.1 M *tert*-butanol etchant mixture (depth ~200 µm). (b) Photograph of multiple retro-reflection cavity which makes parallel beam reflections via 45° tilted vertical mirrors.

The structure shown in Fig. 6 (b) provides the possibility of providing a large number of optical passes through a given sample, which may, for example be within a microfluidic device, where the sample being probed could either be part of a medical (e.g. serum or urine) or environmental (e.g. waste-water) study. By providing a greatly increased path length using this method, we can produce an integrated, self-aligned retroreflective topology, which can potentially increase the performance of instruments. The sensitivity of the measurement will be proportional to effective optical path length, and we can therefore increase this without a concomitant increase in form factor.

V. CONCLUSIONS

We show results demonstating the development of high quality wet etch fabricated sidewalls in monocrystalline silicon. High quality 45° and 90° sidewalls have been achieved by varying the etch chemistry while using the same mask. Surface tension has an adverse effect on the 45° slanted wall while it is preferable to attain the vertical etched wall from the same window direction. Surfactant and alcohol are crucial in the aim of achieving smooth sidewalls. KOH has high etch rate which is suitable for high throughput micromirror devices application.

Using this fabrication technology we have created a platform which in future can find a range of opto-microfluidic applications, enabling multiple reflections to increase the optical path length. It is anticipated that this type of device could enable low cost, scaleable optofluidic technologies in spectroscopic measurements within the clinical, biomedical and environmental sciences.

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