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Abstract This paper examines wet and dry fabrication of vertical micro-mirrors in (110) silicon for use in an innovative BioMEMS integrating gripping and micro force sensing functionalities. Wet anisotropic chemical etching in potassium hydroxide (KOH) and tetramethyl ammonium hydroxide (TMAH) with and without isopropanol alcohol (IPA) additive was examined. Deep Reactive Ion Etched samples were produced using inductive coupled process. 3D surface roughness of samples was examined using scanning electron microscope, interferometric profilometer and atomic force microscopy. An optic fiber displacement sensor was exploited to measure the reflectivity of uncoated or coated samples with evaporated metallic thin film. The research aimed to find optimal fabrication technique for fabricating vertical micro-mirrors in polymer based BioMEMS. TMAH etched silicon samples with surface roughness R_a = 15.1 nm showed highest reflectivity

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TECHNICAL PAPER

3D surface topography and reflectivity of anisotropic etched silicon micromirrors for BioMEMS

R. E. Mackay · N. Lionis · H. R. Le

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Abstract This paper examines wet and dry fabrication of vertical micro-mirrors in (110) silicon for use in an innovative BioMEMS integrating gripping and micro force sensing functionalities. Wet anisotropic chemical etching in potassium hydroxide (KOH) and tetramethyl ammonium hydroxide (TMAH) with and without isopropanol alcohol (IPA) additive was examined. Deep Reactive Ion Etched samples were produced using inductive coupled process. 3D surface roughness of samples was examined using scanning electron microscope, interferometric profilometer and atomic force microscopy. An optic fiber displacement sensor was exploited to measure the reflectivity of uncoated or coated samples with evaporated metallic thin film. The research aimed to find optimal fabrication technique for fabricating vertical micro-mirrors in polymer based BioMEMS. TMAH etched silicon samples with surface roughness $R_a = 15.1$ nm showed highest reflectivity of all structures fabricated, reflectivity was more than doubled by adding a 10 nm layer of evaporated aluminum coating.

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1 Introduction

MEMS (Micro-electro-mechanical systems) technology was introduced a few decades ago and MOEMS (micro-opto-electro-mechanical systems) during early 1990s, but it was not until late 1990s when their reliability and effectiveness made them commercially viable. Since then, the market of MEMS and especially MOEMS has experienced an exponential growth in demand as more sectors in industry require their capabilities. The automotive industry is a characteristic example of where MEMS are used extensively nowadays. Mostly in the form of sensors, MEMS are implemented in many parts of a modern automobile. An example is the accelerometers used to detect a collision and inflate an airbag (Matsunaga and Esashi 2002). As the market grows MEMS is being introduced to new fields, one rapidly expanding field is BioMEMS (Biological micro-electro-mechanical systems) (Grayson et al. 2004; Bashir 2004). Currently researchers are working on a project developing polymer micro-grippers with an optical micro force sensor (Fig. 1) (Mackay and Le 2008; Mackay et al. 2011). This is an exciting example to incorporate BioMEMS with MOEMS to fulfill dual requirements of micro object handling and micro force sensing. This project aims to characterise the mechanical properties of the epithelium tissue. The mechanical characterization of tissues will help scientists to understand fundamental cell physiology. With respect to cancer mechanical properties of normal cells could be compared to those of abnormal cells. This could lead to new early diagnostic tools and therapies in the treatment of colon cancer (Suresh 2007). However, the silicon mirror requires through wafer etching to leave a free standing silicon mirror.

Deep etching through silicon wafers has been a problem for many years; the use of these deep etched structures as

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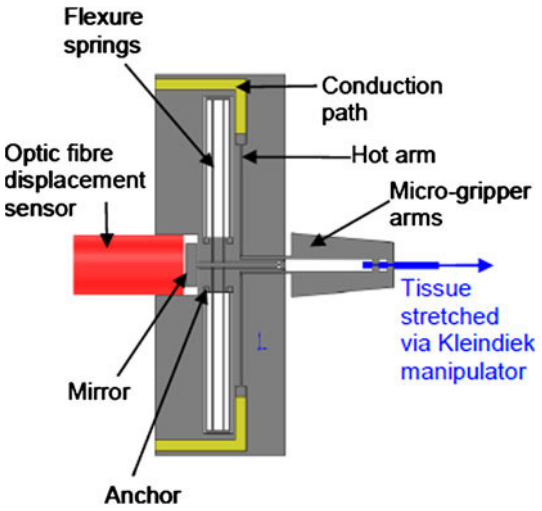

Fig. 1 Design concept for the gripping and force measurement system

mirrors for MOEMS remains a considerable challenge. Through wafer etching must be incorporated in the fabrication of micro-grippers to allow a mirror to be created for displacement sensing. Wet anisotropic etching is the lowest cost and most commonly adopted solution; however geometries are significantly limited by crystal orientation (Agarwal 2007). LIGA can be used to create vertical mirrors; however this requires deep X-ray lithography which is not available in the majority of fabrication units. Deep reactive ion etching allows for complex geometries to be fabricated through wafers, however this generally results in high sidewall roughness although some groups have managed to fabricate mirrors using this etching technique alone (Marxer et al. 1997). A number of groups have amalgamated dry and wet etch techniques to produce complex geometries with smooth sidewalls (Agarwal 2007; Yun et al. 2006). The wafers are orientated and patterned as if for wet etching, however the first step is DRIE followed by a polishing step in a wet etchant to produce {111} planes (Price 1973). However, this increases the manufacturing costs.

DRIE can produce non-vertical sidewalls which can have spherical deviation which along with high roughness cause optical losses in MOEMS. DRIE was developed from RIE, a dry isotropic etching process using SF_6 radicals to etch silicon. One DRIE technique utilizes two gases to create an anisotropic etch; SF_6 is used as the etchant due to fluorine atoms reacting with the silicon substrate, C_4F_8 is used to passivate the sidewalls between each SF_6 cycle to allow deep holes to be etched in silicon. The cycles of etching and passivation cause a curtaining effect to occur on the sidewalls of the wafer creating high surface roughness (Craciun et al. 2002). Deep etches cause large defects to occur at the top of the sidewall which has been subjected to a large number of etching and passivation cycles.

Wet anisotropic etching is a lower cost process and requires simple experimental setup which results in high etch rates with smooth walls being fabricated with a high level of anisotropy. Wet chemical etched structures are limited to specific geometries dictated by crystal orientation of the specific silicon wafer type, i.e. (110). Three main types of chemicals are used for anisotropic etching of silicon; alkaline metal hydroxides (i.e. potassium hydroxide (KOH) or sodium hydroxide (NaOH)) are relatively cheap, non-toxic and result in low roughness sidewalls; diamine based etchants (i.e. ethylenediamine pyrocatecol (EDP)) require complex etch apparatus, have short shelf life and produce highly toxic gases during the reaction with silicon; quaternary ammonium hydroxides (i.e. tetramethyl ammonium hydroxide (TMAH)) has excellent selectivity, is non-toxic, however it is more expensive than alkaline or diamine etchants but can be doped with silicic acid and ammonium persulfate to increase sidewall smoothness (Biswas and Kal 2006). Etching of (110) silicon gives high anisotropy due to the etch rates of the different planes {110} etches faster than {100} which is faster than {111} by a ratio of 400:200:1 at 85°C (Kendall 2003).

Isopropanol alcohol (IPA) has been added to alkaline and TMAH etchants to help improve sidewall surface finish. IPA has no active role in the reaction between etchant and silicon (Williams and Muller 1996), however it does reduce the reaction rate, therefore lowering sidewall roughness. Palik et al. (1983) used Raman spectroscopy to understand the reactions occurring during alkaline etching of silicon (Eq. 1)

$$Si + 2H_2O + 2OH^- = Si(OH)_2O_2^- + H_2O + 2H_2 \quad (1)$$

The etchant must be mixed mechanically to ensure striation does not occur giving different etch rates throughout the solution (Palik et al. 1983); also if IPA is present this does not readily dissolve in solution, mixing ensures even concentration of IPA throughout the entire solution. Hydrogen bubbles readily form on the wafer surface, as seen from Eq. 1 (Seidel et al. 1990), agitation of the etchant helps remove hydrogen bubbles which can act as a 'pseudo' mask stopping small areas from being etched, increasing surface roughness, due to the formation of hillocks (Yang et al. 2005). Mechanical agitation ensures hydrogen bubbles are removed quickly from the surface of the silicon being etched.

Both DRIE and wet chemical etching rely on a large number of variables to ensure smooth, defect free, vertical sidewalls are obtained. Wet chemical etching depends on type of etchant, concentration of etchant, temperature, mixing rate, additives and alignment to {111} plane. DRIE variables include pressure and flow rate of etchant and

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passivation chemicals, RF power, distribution of reactive fluorine species and concentration and distribution of waste products. Finally wet etching can cause stiction of the envisaged free hanging MEMS structure, which must be taken into account when designing for BioMEMS and MOEMS, whilst DRIE avoids this due to the dry etchants being used.

In total, 42 wet etching experiments were carried out using KOH, KOH + IPA, TMAH and TMAH + IPA. Deep reactive ion etched samples were also examined which were etched using inductive coupled plasma process. Sample reflectivity was studied using Philtec D6 fibre optic displacement sensor. Uncoated bare Si samples were tested along with samples coated with a 20 nm layer of evaporated Au–Pd and 10 nm Al. Samples were examined using light microscopy and SEM. Surface profiling was done using a Dektak (Veeco) surface profiler and Zygo 3D interferometric profilometer.

2 Experimental procedure

Silicon wafers (110) p-type (resistivity 1–5 ohm cm) single side polished with a 76 mm diameter and thicknesses of 381 μm were used for anisotropic etching experiments. Wafers were thermally oxidized in air to create a 1 μm SiO_2 insulating layer. The wafer was spin coated with Shipley 1813 photoresist of about 1.2 μm; this was baked on a hotplate at 115°C for 3 min. Two photomask patterns were used throughout the experiments (Fig. 2). The wafer was aligned to the photomask so the {111} plane was parallel to the mirror pattern. The wafer was exposed to the mask pattern using an OAI J500 photo aligner. The wafer was exposed for 4 s and the photoresist developed by immersion in MICROPOSIT MF 321 diluted with deionized H_2O at a ratio of 1:3 respectively at room temperature for 20 s. The sample was then placed in buffered hydrofluoric acid (BHF) for 7 min 30 s until the oxide was removed leaving bare silicon. Remaining photoresist was removed by rinsing in acetone. The thickness of the oxide layer was verified using a Dektak surface profiler.

Wet etching experiments were carried out in a Teflon beaker (Fig. 3); a magnetic stirrer was placed in the bottom of the beaker to agitate the chemicals as a reaction rate controlled process requires a constant diffusion rate and also avoids stratification of the etchant to maintain even etching across the wafer. A small Teflon guard was placed over the stirrer to avoid collision between this and the silicon wafers being etched. The speed of the stirrer was set to a constant speed of 250 rpm when aqueous solutions of KOH or TMAH were used. However, for solutions with IPA added, the speed was set to 500 rpm to ensure proper mix and distribution of the alcohol into the solution. The beaker

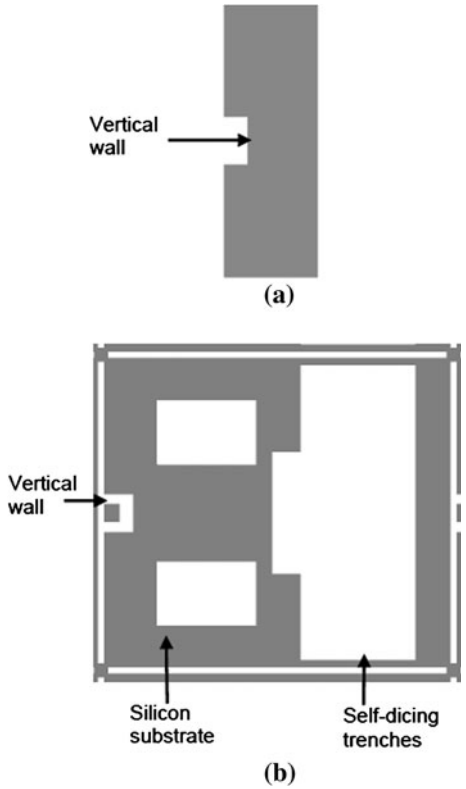

Fig. 2 Mask patterns used for back etching on (110) silicon wafers **a** early mask used for initial experiments; **b** mask designed for polymer micro-gripper system

was placed in a water bath which was placed directly onto a hotplate. The hot plate temperature controller was used in order to set the etching temperature. A probe was placed in the etchant to sense the temperature within the solution and provide feedback to the controller in order for the temperature to remain constant. The hotplate was set at a temperature two times larger than the etching temperature, due to the etchant being in a Teflon beaker immersed in a water bath, which gave a temperature tolerance to the etchant of ±5°C. Outside of this range the etching temperature either would not be reached or it would be exceeded. Evaporation of the solution was an issue so the plastic beaker was sealed with a cap which featured a small hole for the temperature probe which was placed directly into the solution in order to provide the necessary feedback.

KOH solutions were made by dissolving KOH pellets in DI H_2O; this will give KOH concentration ±5% due to the absorption of moisture into the KOH pellets (Powell 2001). TMAH solutions were made by mixing 20 wt% TMAH solution with DI H_2O. 4% IPA was added to solutions which contained the alcohol. Wet chemical experiments were carried out using 20 wt% KOH, 25 wt% KOH, 30 wt% KOH, 25 wt% KOH + IPA, 30 wt% KOH + IPA, 20 wt% TMAH, 10 wt% TMAH, 13 wt% TMAH and 13 wt% TMAH + IPA.

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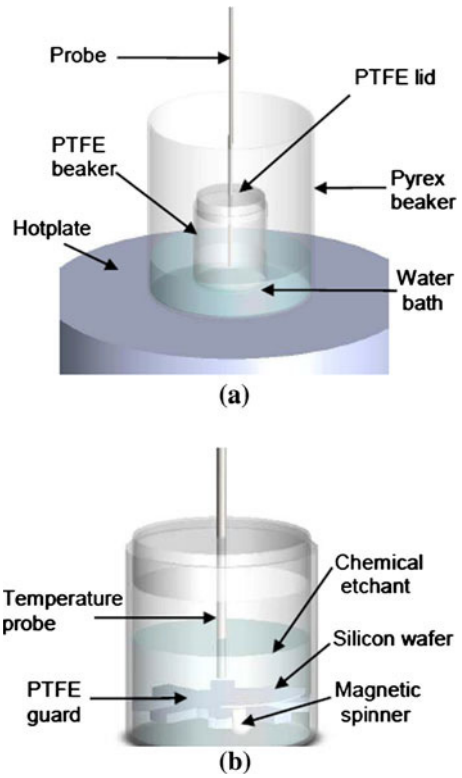

Fig. 3 Experimental setup for silicon etching experiments **a** Overview of experimental setup **b** teflon beaker for silicon etching

DRIE samples were obtained from the Scottish Microelectronics Centre Edinburgh. These samples were produced on 76 mm Si wafers with 200 μm thickness. SF_6 is used for etching the silicon; this is an anisotropic process however it causes an initial undercut in the silicon. The C_4F_8 is used to passivate the sidewall to stop the area being isotropically etched during the next etching cycle. This helps restrain isotropy but this cannot be completely eliminated as a small amount of undercutting occurs in every etch cycle.

2.1 Reflectivity tests

Reflectivity tests were carried out in order to relate surface roughness with optimal reflectivity of samples. Samples tested were polished and unpolished silicon, 30 wt% KOH + IPA, 25 wt% KOH + IPA, 13 wt% TMAH, 13 wt% TMAH + IPA, 13 wt% TMAH coated with thermally evaporated Al, DRIE and DRIE then coated with sputtered Pd/Au.

A 3D micromanipulator by Kleindiek Nanotechnik was used to hold and manipulate samples, whilst a specially designed fibre optic holder was used to ensure the displacement sensor was parallel to the chip (Fig. 4). This allowed for movement of samples in 3 axes with step sizes ranging from 0.25 to 500 nm. Precise alignment between the {110} sidewall and fibre optic displacement sensor

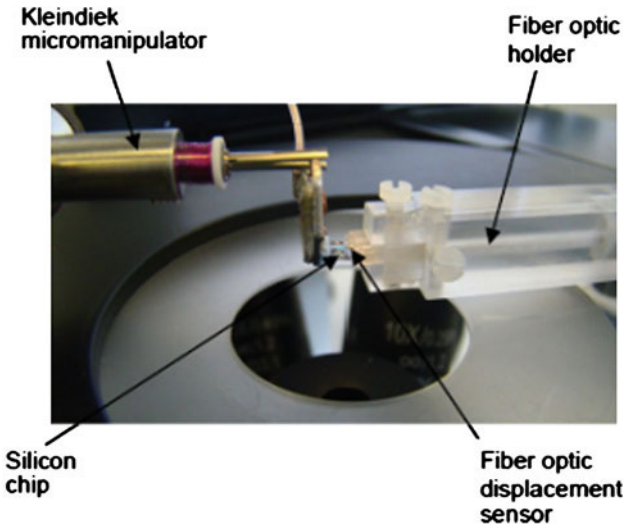

Fig. 4 Reflectivity testing setup

(Philtec D6) is paramount to retrieve accurate results, sidewall thickness is 381 μm. The fibre optic displacement sensor had wavelength 670 nm.

Static experiments were used to test optimal reflectivity of samples whilst dynamic testing examined the range of voltages that could be acquired when the chip was displaced cyclically over ±288 μm. Nanocontrol software supplied with the micromanipulator was used to create a macro program to run a series of displacement loops. A Labview program was used to retrieve optimal voltages and displacement voltages.

3 Results and discussions

The surface finish of samples was analyzed using a Dektak surface profiler and Zygo interferometric profilometer and Veeco CPii atomic force microscope (AFM). Samples were also examined using both light microscopy to help with sample selection and then scanning electron microscope to examine the microstructure of the etched {111} sidewalls.

3.1 Deep reactive ion etched samples

Deep reactive ion etched samples showed even etching throughout the majority of the sample. Samples show striated lines due to the process of DRIE (Fig. 5a), cycling etching and passivation. AFM results showed the depth of trenches formed due to the DRIE process (Fig. 5b), the figure shows a 50 × 50 μm area and the resulting curtaining pattern is clearly visible. Etch trenches were measured in the centre of a Si wafer sidewall showed depths of ~7.5 μm which increases surface roughness significantly. DRIE samples varied greatly depending on etch parameters. Average roughness of DRIE samples was found to be R_a = 1,707 nm

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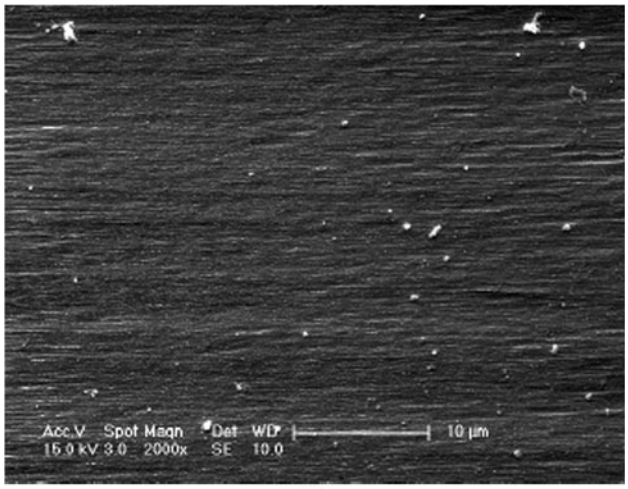
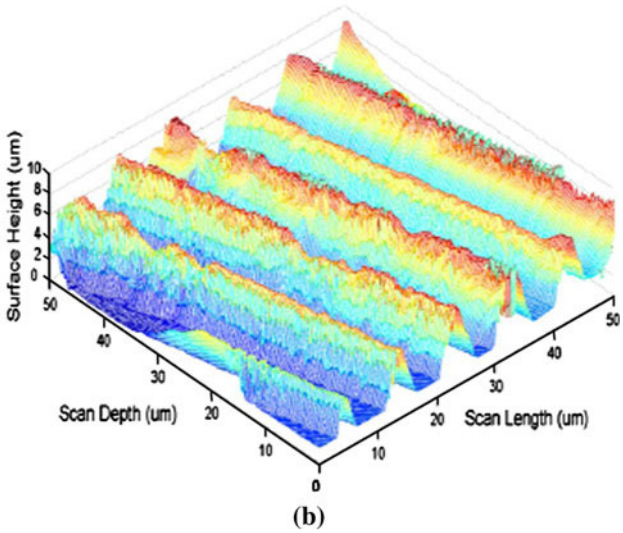


(a)



(b)

Fig. 5 Sidewall surface of deep reactive ion etched samples **a** SEM image and **b** AFM scan profile

and rms = 2,027 nm. However the majority of samples showed poor reflectivity. One sample with much lower surface roughness $R_a = 533$ nm and rms = 684 nm was selected for reflectivity tests. SEM examination of DRIE samples showed large defects occurring at the top of the sidewall where wafers were exposed to thousands of etching and passivation cylces (Fig. 6). These deep trenches could seriously affect reflectivity if they penetrate >50 µm deep into the sidewall. An area of 150 × 150 µm is needed for reflection to the optical displacement sensor. However, the majority of these defects do not penetrate beyond 10 µm.

3.2 KOH etched samples

One of the greatest difficulties the authors faced when etching with KOH was using SiO_2 as a mask for KOH

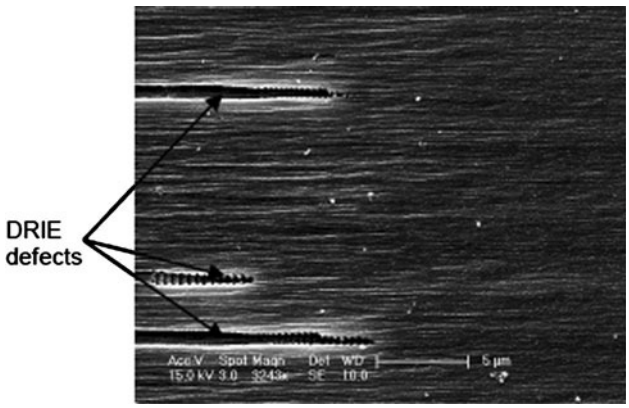


Fig. 6 DRIE defects seen at the top of the wafer where the material is subjected to hundreds of etching and passivation cycles

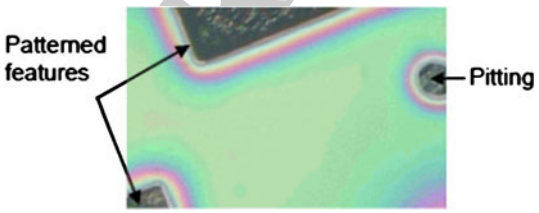


Fig. 7 KOH etched wafers with 1 µm SiO_2 mask layer

etched samples. Some pitting was seen on the surface of the wafers due to uneven etching of SiO_2 across the surface of the wafer, this resulted in masked areas of silicon etching significantly (Fig. 7). KOH etched samples featured a number of circular trenches forming on the surface of the {111} plane when etched at low concentrations ≤20 wt%. In order to etch vertical mirrors in (110) silicon KOH concentrations in the range of 25–35 wt% were found to be more desirable and fewer circular trenches appeared parallel to the {111} plane. Additions of IPA at these concentrations greatly reduced surface roughness; however some pitting was still seen on the surface. The addition of IPA reduces the etch rate due to changes in surface energy of Si. Figure 8a shows a number of hillocks, which have formed due to hydrogen bubbles acting a 'pseudo' masks throughout the etching process. The AFM results show an uneven surface with significant areas of pitting and the edge of a hillock formed (Fig. 8b).

KOH samples showed high surface roughness, samples etched in 25 wt% KOH at 75°C showed $R_a = 3{,}127$ nm. 25 wt% KOH + 4% IPA showed lower average roughness of $R_a = 1{,}113$ nm and rms = 1,316 nm when etched at 75°C. Increasing KOH concentration to 30 wt% + 4% IPA decreased surface roughness to $R_a = 616$ nm and rms = 763 nm. Increasing concentrations of KOH etching solution and addition of IPA significantly decreases the etch rate, slower etch rates show better surface finish due to less

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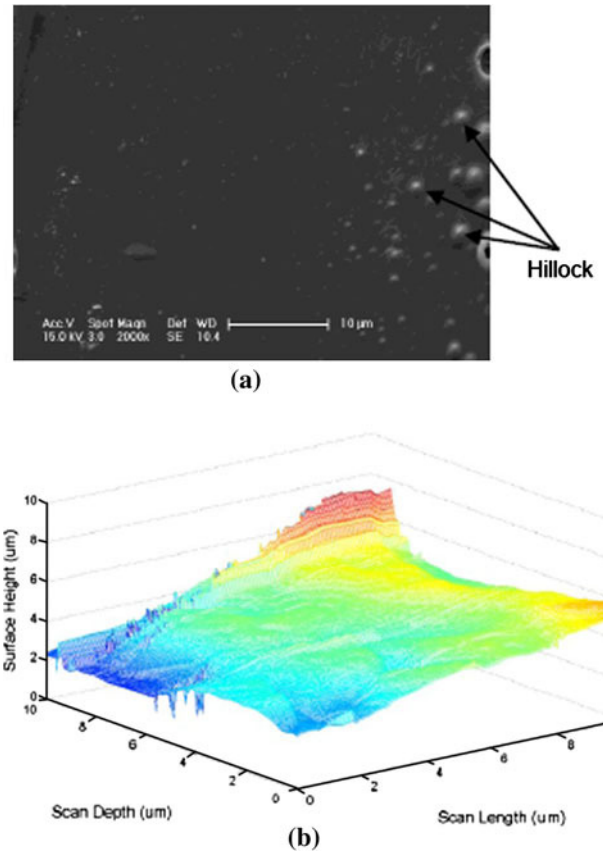

Fig. 8 SEM image of silicon sidewall etched in 25 wt% KOH +4% IPA, 70°C, 4 h 30 min **a** SEM image of vertical mirror **b** AFM surface profile

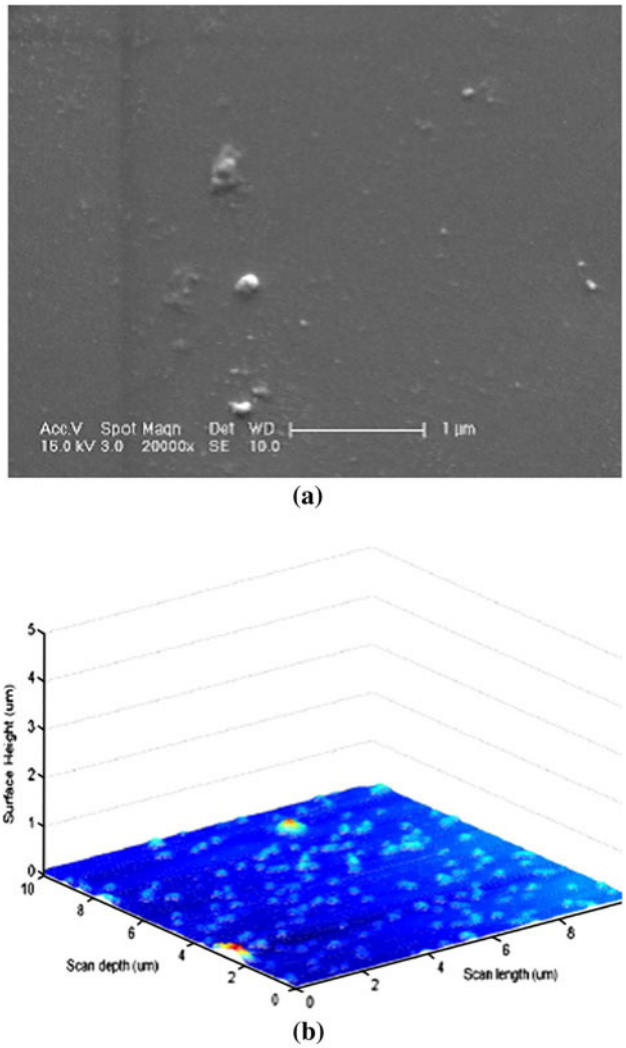

Fig. 9 AFM surface profile of silicon sidewall etched in 13 wt% TMAH at 85°C **a** SEM image of vertical mirror **b** AFM surface profile

defects being formed on the Si surface. High surface roughness of KOH samples could be due not only to slight misalignment with {111} plane but also depth of etch >200 μm (Sato et al. 1999b).

3.3 TMAH etched samples

TMAH showed much better selectivity to the SiO_2 mask than KOH. Vertical sidewalls aligned to the {111} plane were produced with fewer defects due to this high selectivity. Uniformity of the etched surface was observed to be greater in TMAH than KOH. TMAH samples etched with concentrations of 13 wt% showed lower surface roughness than those etched at 10 and 20 wt%. The optimum etching temperature was found to be 85°C.

TMAH samples had a lowest roughness of all samples produced with $R_a = 14$ nm and rms = 20 nm. Figure 9b shows an area 10 × 10 μm and the average surface height over this area. Two large hillocks can be seen in this area but their height is ~100 nm.

Samples etched with TMAH only were observed to be less rough than those with the addition of 4% IPA. Addition of IPA caused striations to occur across the wafer increasing surface roughness, however, the addition of IPA did eliminate the formation of hillocks on the {111} surface (Fig. 10). Hillocks formed during TMAH etches were significantly smaller and fewer than seen in KOH etchants.

3.4 Reflectivity tests

Optimal alignment was achieved by manipulating the chip when set at a distance of 200 μm from the tip of the fiber optic displacement sensor. This distance gives the optimal voltage output from the displacement sensor. The maximum voltages were retrieved during static testing (Table 1). Silicon wafer surface samples of polished silicon and unpolished silicon were used as reference values.

Polished silicon showed the highest static output voltage, 1.66 V, whilst unpolished the lowest; maximum voltage output for the displacement sensor is 5 V. Polished

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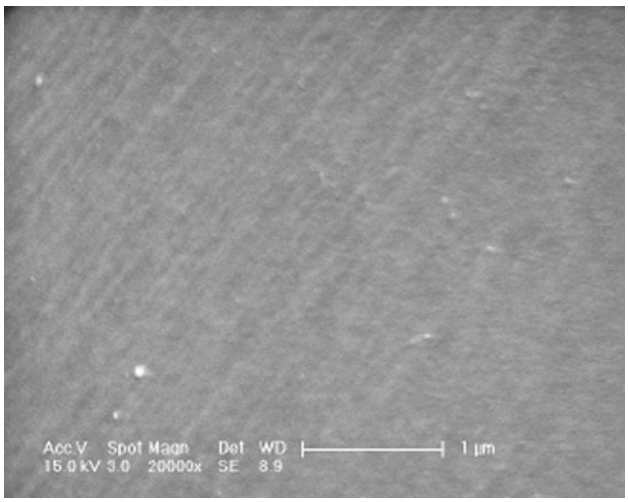

Fig. 10 Samples etched in 13 wt% TMAH + IPA showed striation and rougher surfaces than those etched with TMAH only

and unpolished silicon samples were much easier to align than sidewall samples as they had a much greater depth >2 mm when compared to etched samples which had a depth 200–381 μm. TMAH showed lowest surface roughness and highest maximum voltage, 0.83 V during static tests, this increased significantly to 2.48 V once coated with 10 nm Al. DRIE samples tested were those with the lowest roughness (R_a = 533 nm). DRIE samples showed output voltage of 0.72 V which increased to 1.12 V when the Pd-Au coating was sputtered on the surface. TMAH + IPA samples showed much lower static output voltage of 0.35 V due to the striations scattering light away from the fiber optic sensor. Samples etched in 30 wt% KOH + IPA gave an output voltage of 0.24 V whilst 25 wt% KOH + IPA gave an output voltage of 0.04 V showing slower etch rates increase reflectivity. However due to defects in KOH etched silicon arising because of poor masking, reflectivity is lower than DRIE samples. Samples etched with KOH only could not be tested as

Table 1 Summary of optimal voltage outputs and voltage variation for ±288 μm of displacement

Etching technique/solution	Optimal voltage (V)	Voltage variation (V)
Polished silicon	1.66	0.26
DRIE metalized	1.12	0.1
13 wt% TMAH metalized	2.48	0.07
13 wt% TMAH	0.83	0.11
DRIE	0.72	0.17
13 wt% TMAH + IPA	0.35	0.05
30 wt% KOH + IPA	0.24	0.05
Unpolished silicon	0.15	–
25 wt% KOH + IPA	0.04	–

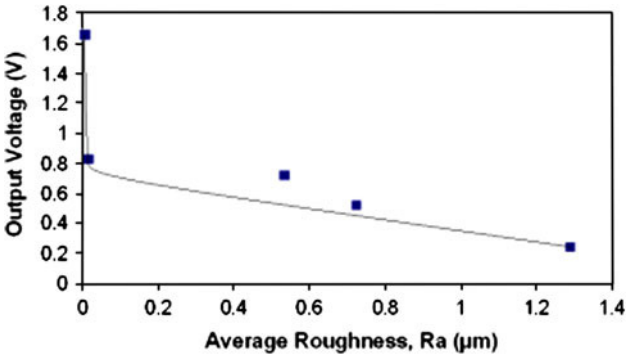

Fig. 11 Static reflectivity tests showing optical sensor output voltage vs average roughness of silicon sidewall mirror

samples did not reflect enough light to produce an output voltage. Static output voltages were related to average roughness (R_a) measurements (Fig. 11). Polished silicon (R_a = 5 nm) shows significantly higher output voltage when compared to TMAH samples (R_a = 14 nm), it is believed this is due to the small area being tested and difficulty in aligning the 200 μm thick sample to the tip of the optic fiber. Sato et al. found orientation dependence of etching differs between the two wet etchants, within the current experiments TMAH was found to be easier to align to {111} orientation (Sato et al. 1999a). It can be seen that DRIE samples (R_a = 533 nm) with lower roughness than KOH + IPA samples (567 nm) showed higher output voltage than the slightly rougher KOH + IPA samples. Addition of highly reflective metals (i.e. Au–Pd), sputtered onto the silicon sidewalls greatly increased static voltage output. Au–Pd has excellent reflectivity of near 100% for the wavelength of light (690 nm) being reflected. Al has slightly lower reflectance around 95% for the specific displacement sensor however it is much lower cost. Si showed lowest reflectivity and is highly dependent on surface roughness (Hashim and Salih 2005).

Displacement experiments were carried out using the Kleindiek micromanipulator, the range of output voltage difference for displacement of 566 μm is shown in Table 1. Results were poorer than expected but it is believed this is due to parasitic motion in the micromanipulator, resulting in rotation of samples, this resulted in misalignment of the wafer to the fiber optic sensor.

4 Conclusions

Etching of silicon in multifunctional BioMEMS represents a complex procedure due to the number of variables and the associated outcomes of each. Fabrication of vertical mirror surfaces via anisotropic deep etching through wafers for BioMEMS is achievable by controlling the etch rate and extraction of gaseous products from the etchant. One

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obvious limit of anisotropic etching is the limit of the pattern to the crystal orientation of (110) wafers, it is impossible to produce rectangular structures and trapezoids are formed in the structure; however it is proved possible to orientate the wafer so the mirror is present and perfectly aligned to the {111} plane. A new method was developed to examine the reflectivity of micro-engineered surface using optic fiber displacement sensor and 3D piezoelectric manipulator.

The results show that concentration of both KOH and TMAH is an important factor to produce optimal surfaces. Generally higher percentages of KOH result in smoother walls, here the optimal is 30 wt%, this slows down the etch rate resulting in smoother walls when examined using SEM. KOH must be used with the addition of IPA, to reduce surface energy of Si and cause fewer hillocks to be formed, samples etched in KOH only could not be used as vertical silicon mirrors. The addition of IPA to TMAH solutions resulted in rougher, striated surfaces being formed. For TMAH, the optimal etchant concentration was found to be 13 wt%. Temperature of etchant also results in variability of smooth sidewalls, for KOH + IPA lower temperatures, ≤70°C resulted in optimal smoothness, the lowering of temperature helps slow the etch rate. For TMAH etchants, the optimal was found to be 85°C.

The argument by previous authors that TMAH etches can result in smoother sidewalls than KOH etched samples, was proved. TMAH showed good results with the lowest R_a recorded and highest output of uncoated micro-samples. It was also found that IPA did not improve the surface quality when TMAH was used. KOH + IPA showed higher roughness due to the formation of sizable hillocks and significant pitting in the SiO_2 mask. If KOH is to be used to form vertical sidewalls SiN must be employed as the mask.

DRIE samples showed significantly higher surface roughness than TMAH but performed well during reflectivity results. Uncoated samples gave a reasonable static output voltage from the fiber optic displacement sensor. Coated samples of DRIE and TMAH gave the best overall reflectivity results, as expected.

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