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

Mackay, RE

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MICROSYSTEM TECHNOLOGIES-MICRO-AND NANOSYSTEMS-INFORMATION STORAGE AND PROCESSING SYSTEMS

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	Organization	University of Plymouth
	Address	Plymouth, UK
	Email	huirong.le@plymouth.ac.uk
Author	Family Name	<b>Mackay</b>
	Particle	
	Given Name	<b>R. E.</b>
	Suffix	
	Division	Centre for Electronic Systems Research, School of Engineering and Design
	Organization	Brunel University
	Address	UB8 3PH, Uxbridge, Middlesex, UK
	Email	ruth.mackay@brunel.ac.uk
Author	Family Name	<b>Lionis</b>
	Particle	
	Given Name	<b>N.</b>
	Suffix	
	Division	Cass Business School
	Organization	City University
	Address	London, UK
	Email	sinoiljr@hotmail.com
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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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2 **3D surface topography and reflectivity of anisotropic etched**  
3 **silicon micromirrors for BioMEMS**

4 **R. E. Mackay · N. Lionis · H. R. Le**

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7 **Abstract** This paper examines wet and dry fabrication of  
8 vertical micro-mirrors in (110) silicon for use in an inno-  
9 vative BioMEMS integrating gripping and micro force  
10 sensing functionalities. Wet anisotropic chemical etching  
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12 hydroxide (TMAH) with and without isopropanol alcohol  
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15 3D surface roughness of samples was examined using  
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19 or coated samples with evaporated metallic thin film.  
20 The research aimed to find optimal fabrication technique  
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25 adding a 10 nm layer of evaporated aluminum coating.  
26

A1 R. E. Mackay  
A2 Centre for Electronic Systems Research,  
A3 School of Engineering and Design, Brunel University,  
A4 Uxbridge, Middlesex UB8 3PH, UK  
A5 e-mail: ruth.mackay@brunel.ac.uk

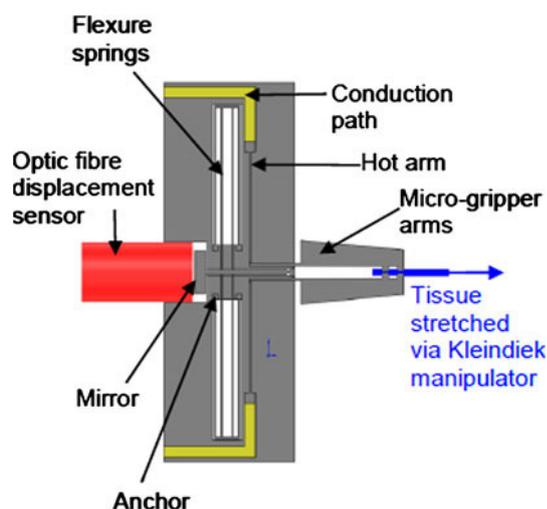
A6 N. Lionis  
A7 Cass Business School, City University, London, UK  
A8 e-mail: sinoiljr@hotmail.com

A9 H. R. Le (✉)  
A10 School of Marine Science and Engineering,  
A11 University of Plymouth, Plymouth, UK  
A12 e-mail: huirong.le@plymouth.ac.uk

**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 effec-  
tiveness 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 auto-  
mobile. 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 pro-  
ject 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 epi-  
thelium tissue. The mechanical characterization of tissues  
will help scientists to understand fundamental cell physi-  
ology. 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



**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  $\text{SF}_6$  radicals to etch silicon. One DRIE technique utilizes two gases to create an anisotropic etch;  $\text{SF}_6$  is used as the etchant due to fluorine atoms reacting with the silicon substrate,  $\text{C}_4\text{F}_8$  is used to passivate the sidewalls between each  $\text{SF}_6$  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)



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

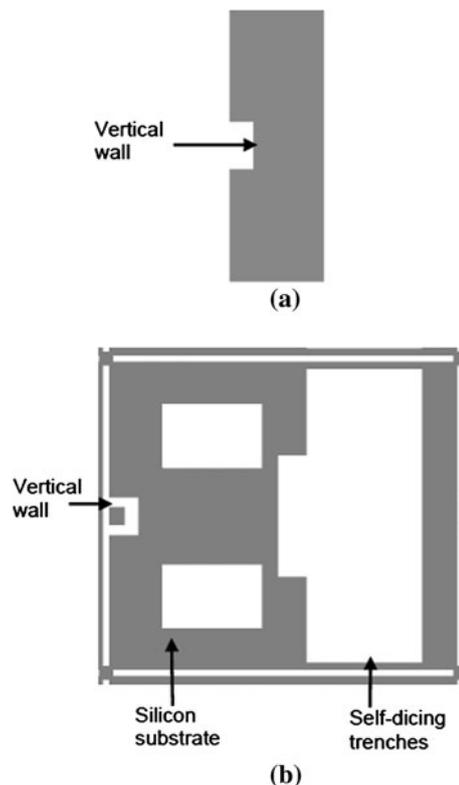
147 passivation chemicals, RF power, distribution of reactive  
 148 fluorine species and concentration and distribution of waste  
 149 products. Finally wet etching can cause stiction of the  
 150 envisaged free hanging MEMS structure, which must be  
 151 taken into account when designing for BioMEMS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

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

## 165 2 Experimental procedure

166 Silicon wafers (110) p-type (resistivity 1–5 ohm cm) single  
 167 side polished with a 76 mm diameter and thicknesses of  
 168 381  $\mu\text{m}$  were used for anisotropic etching experiments.  
 169 Wafers were thermally oxidized in air to create a 1  $\mu\text{m}$   
 170  $\text{SiO}_2$  insulating layer. The wafer was spin coated with  
 171 Shipley 1813 photoresist of about 1.2  $\mu\text{m}$ ; this was baked  
 172 on a hotplate at 115°C for 3 min. Two photomask patterns  
 173 were used throughout the experiments (Fig. 2). The wafer  
 174 was aligned to the photomask so the {111} plane was  
 175 parallel to the mirror pattern. The wafer was exposed to the  
 176 mask pattern using an OAI J500 photo aligner. The wafer  
 177 was exposed for 4 s and the photoresist developed by  
 178 immersion in MICROPOSIT MF 321 diluted with deionized  
 179  $\text{H}_2\text{O}$  at a ratio of 1:3 respectively at room temperature  
 180 for 20 s. The sample was then placed in buffered hydro-  
 181 fluoronic acid (BHF) for 7 min 30 s until the oxide was  
 182 removed leaving bare silicon. Remaining photoresist was  
 183 removed by rinsing in acetone. The thickness of the oxide  
 184 layer was verified using a Dektak surface profiler.

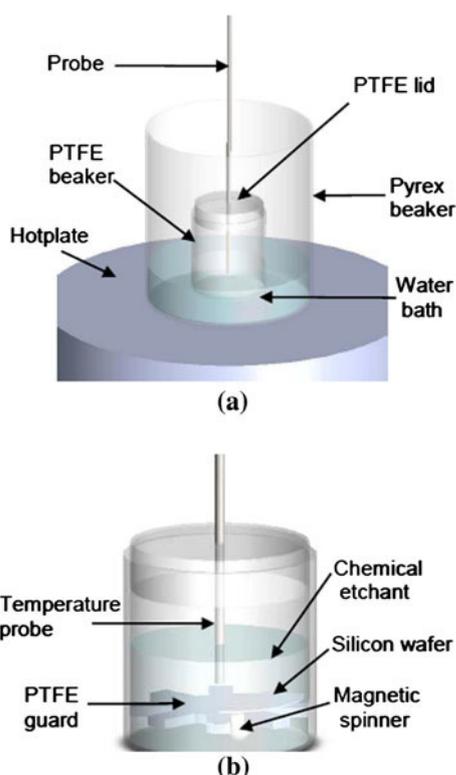
185 Wet etching experiments were carried out in a Teflon  
 186 beaker (Fig. 3); a magnetic stirrer was placed in the bottom  
 187 of the beaker to agitate the chemicals as a reaction rate  
 188 controlled process requires a constant diffusion rate and  
 189 also avoids stratification of the etchant to maintain even  
 190 etching across the wafer. A small Teflon guard was placed  
 191 over the stirrer to avoid collision between this and the sil-  
 192 icon wafers being etched. The speed of the stirrer was set to  
 193 a constant speed of 250 rpm when aqueous solutions of  
 194 KOH or TMAH were used. However, for solutions with IPA  
 195 added, the speed was set to 500 rpm to ensure proper mix  
 196 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

197 was placed in a water bath which was placed directly onto a  
 198 hotplate. The hot plate temperature controller was used in  
 199 order to set the etching temperature. A probe was placed in  
 200 the etchant to sense the temperature within the solution and  
 201 provide feedback to the controller in order for the temper-  
 202 ature to remain constant. The hotplate was set at a tem-  
 203 perature two times larger than the etching temperature, due  
 204 to the etchant being in a Teflon beaker immersed in a water  
 205 bath, which gave a temperature tolerance to the etchant of  
 206  $\pm 5^\circ\text{C}$ . Outside of this range the etching temperature either  
 207 would not be reached or it would be exceeded. Evaporation  
 208 of the solution was an issue so the plastic beaker was sealed  
 209 with a cap which featured a small hole for the temperature  
 210 probe which was placed directly into the solution in order to  
 211 provide the necessary feedback.

212 KOH solutions were made by dissolving KOH pellets in  
 213 DI  $\text{H}_2\text{O}$ ; this will give KOH concentration  $\pm 5\%$  due to the  
 214 absorption of moisture into the KOH pellets (Powell 2001).  
 215 TMAH solutions were made by mixing 20 wt% TMAH  
 216 solution with DI  $\text{H}_2\text{O}$ . 4% IPA was added to solutions  
 217 which contained the alcohol. Wet chemical experiments  
 218 were carried out using 20 wt% KOH, 25 wt% KOH,  
 219 30 wt% KOH, 25 wt% KOH + IPA, 30 wt% KOH +  
 220 IPA, 20 wt% TMAH, 10 wt% TMAH, 13 wt% TMAH and  
 221 13 wt% TMAH + IPA.



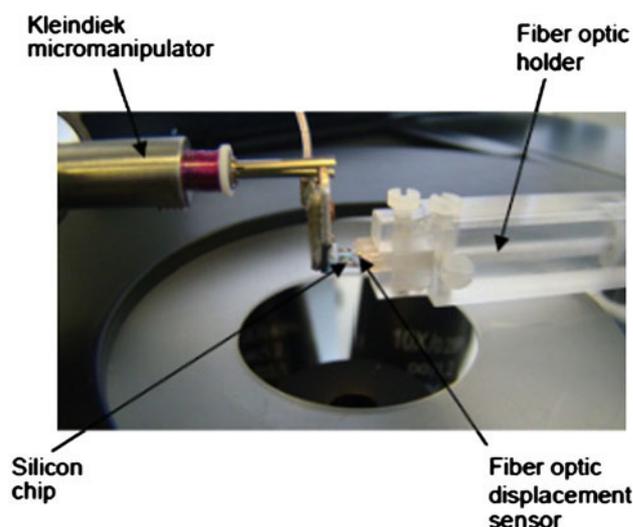
**Fig. 3** Experimental setup for silicon etching experiments **a** Overview of experimental setup **b** teflon beaker for silicon etching

222 DRIE samples were obtained from the Scottish Micro-  
 223 electronics Centre Edinburgh. These samples were produced  
 224 on 76 mm Si wafers with 200  $\mu\text{m}$  thickness.  $\text{SF}_6$  is used for  
 225 etching the silicon; this is an anisotropic process however it  
 226 causes an initial undercut in the silicon. The  $\text{C}_4\text{F}_8$  is used to  
 227 passivate the sidewall to stop the area being isotropically  
 228 etched during the next etching cycle. This helps restrain  
 229 isotropy but this cannot be completely eliminated as a small  
 230 amount of undercutting occurs in every etch cycle.

### 231 2.1 Reflectivity tests

232 Reflectivity tests were carried out in order to relate surface  
 233 roughness with optimal reflectivity of samples. Samples  
 234 tested were polished and unpolished silicon, 30 wt%  
 235 KOH + IPA, 25 wt% KOH + IPA, 13 wt% TMAH,  
 236 13 wt% TMAH + IPA, 13 wt% TMAH coated with ther-  
 237 mally evaporated Al, DRIE and DRIE then coated with  
 238 sputtered Pd/Au.

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



**Fig. 4** Reflectivity testing setup

(Philtec D6) is paramount to retrieve accurate results, 246  
 sidewall thickness is 381  $\mu\text{m}$ . The fibre optic displacement 247  
 sensor had wavelength 670 nm. 248

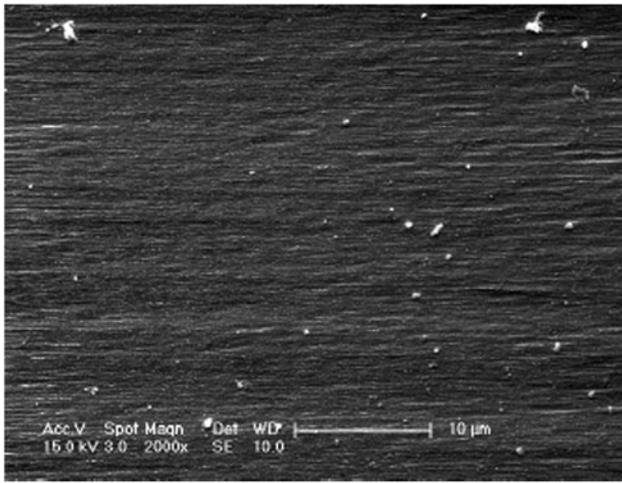
Static experiments were used to test optimal reflectivity 249  
 of samples whilst dynamic testing examined the range of 250  
 voltages that could be acquired when the chip was dis- 251  
 placed cyclically over  $\pm 288 \mu\text{m}$ . Nanocontrol software 252  
 supplied with the micromanipulator was used to create a 253  
 macro program to run a series of displacement loops. 254  
 A Labview program was used to retrieve optimal voltages 255  
 and displacement voltages. 256

## 257 3 Results and discussions

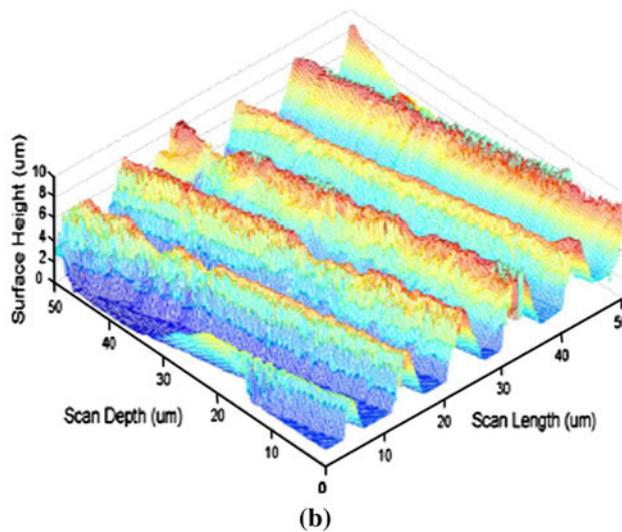
The surface finish of samples was analyzed using a Dektak 258  
 surface profiler and Zygo interferometric profilometer and 259  
 Veeco CPIi atomic force microscope (AFM). Samples were 260  
 also examined using both light microscopy to help with 261  
 sample selection and then scanning electron microscope to 262  
 examine the microstructure of the etched  $\{111\}$  sidewalls. 263

### 264 3.1 Deep reactive ion etched samples

265 Deep reactive ion etched samples showed even etching 265  
 throughout the majority of the sample. Samples show striated 266  
 lines due to the process of DRIE (Fig. 5a), cycling etching 267  
 and passivation. AFM results showed the depth of trenches 268  
 formed due to the DRIE process (Fig. 5b), the figure shows a 269  
 $50 \times 50 \mu\text{m}$  area and the resulting curtaining pattern is 270  
 clearly visible. Etch trenches were measured in the centre of 271  
 a Si wafer sidewall showed depths of  $\sim 7.5 \mu\text{m}$  which 272  
 increases surface roughness significantly. DRIE samples 273  
 varied greatly depending on etch parameters. Average 274  
 roughness of DRIE samples was found to be  $R_a = 1,707 \text{ nm}$  275



(a)



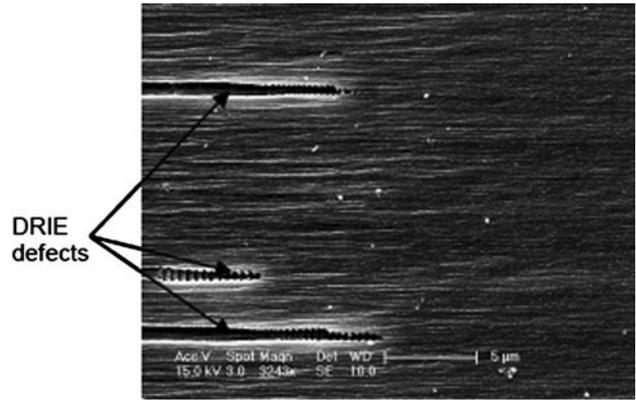
(b)

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

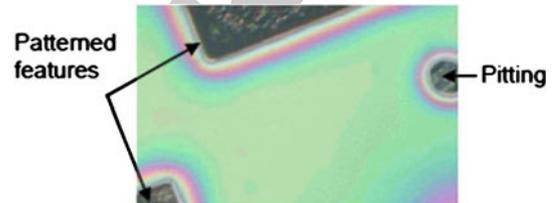
276 and  $rms = 2,027$  nm. However the majority of samples  
 277 showed poor reflectivity. One sample with much lower surface  
 278 roughness  $R_a = 533$  nm and  $rms = 684$  nm was  
 279 selected for reflectivity tests. SEM examination of DRIE  
 280 samples showed large defects occurring at the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

287 **3.2 KOH etched samples**

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



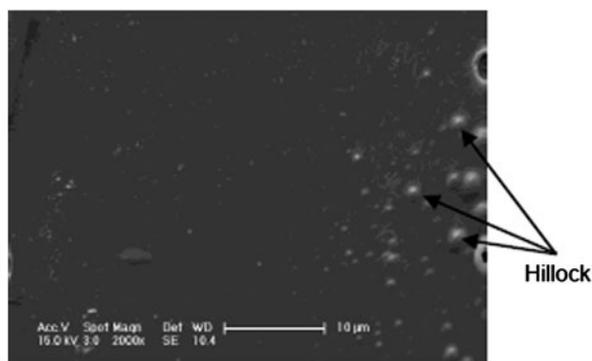
**Fig. 6** DRIE defects seen at the top of the wafer where the material is subjected to hundreds of etching and passivation cycles



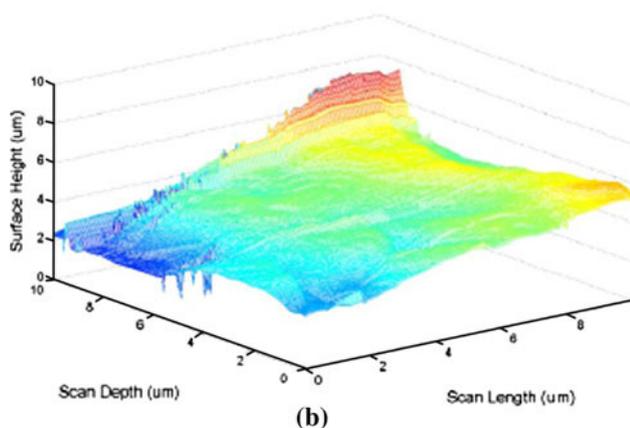
**Fig. 7** KOH etched wafers with  $1$   $\mu m$   $SiO_2$  mask layer

290 etched samples. Some pitting was seen on the surface of  
 291 the wafers due to uneven etching of  $SiO_2$  across the surface  
 292 of the wafer, this resulted in masked areas of silicon  
 293 etching significantly (Fig. 7). KOH etched samples fea-  
 294 tured a number of circular trenches forming on the surface  
 295 of the  $\{111\}$  plane when etched at low concentrations  
 296  $\leq 20$  wt%. In order to etch vertical mirrors in  $(110)$  silicon  
 297 KOH concentrations in the range of  $25$ – $35$  wt% were  
 298 found to be more desirable and fewer circular trenches  
 299 appeared parallel to the  $\{111\}$  plane. Additions of IPA at  
 300 these concentrations greatly reduced surface roughness;  
 301 however some pitting was still seen on the surface. The  
 302 addition of IPA reduces the etch rate due to changes in  
 303 surface energy of Si. Figure 8a shows a number of hillocks,  
 304 which have formed due to hydrogen bubbles acting as  
 305 ‘pseudo’ masks throughout the etching process. The AFM  
 306 results show an uneven surface with significant areas of  
 307 pitting and the edge of a hillock formed (Fig. 8b).

308 KOH samples showed high surface roughness, samples  
 309 etched in  $25$  wt% KOH at  $75^\circ C$  showed  $R_a = 3,127$  nm.  
 310  $25$  wt% KOH +  $4\%$  IPA showed lower average roughness  
 311 of  $R_a = 1,113$  nm and  $rms = 1,316$  nm when etched at  
 312  $75^\circ C$ . Increasing KOH concentration to  $30$  wt% +  $4\%$  IPA  
 313 decreased surface roughness to  $R_a = 616$  nm and  $rms =$   
 314  $763$  nm. Increasing concentrations of KOH etching solu-  
 315 tion and addition of IPA significantly decreases the etch  
 316 rate, slower etch rates show better surface finish due to less



(a)



(b)

**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

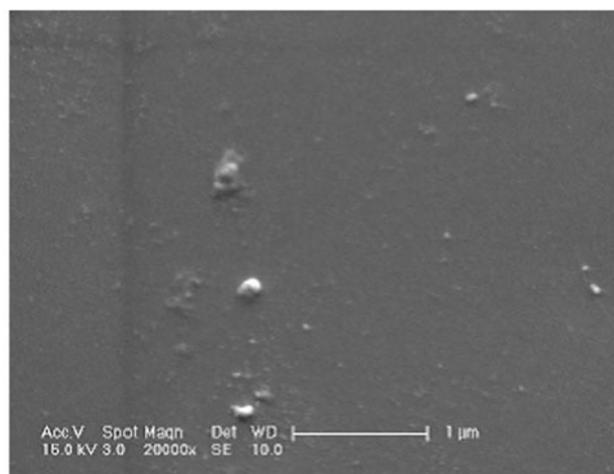
317 defects being formed on the Si surface. High surface  
318 roughness of KOH samples could be due not only to slight  
319 misalignment with {111} plane but also depth of etch  
320 >200 µm (Sato et al. 1999b).

### 321 3.3 TMAH etched samples

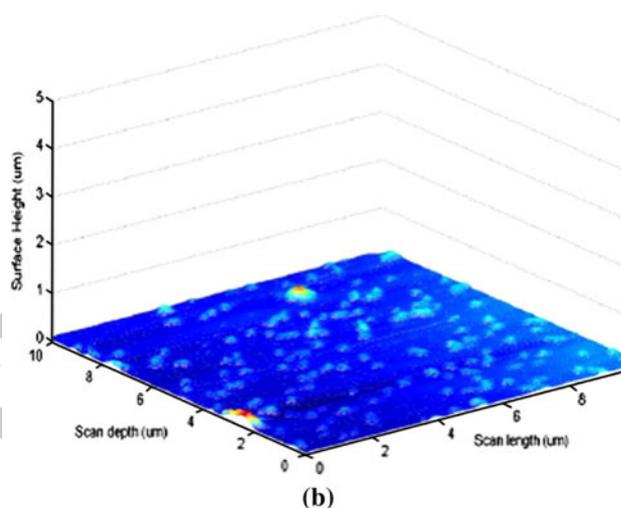
322 TMAH showed much better selectivity to the SiO<sub>2</sub> mask  
323 than KOH. Vertical sidewalls aligned to the {111} plane  
324 were produced with fewer defects due to this high selectivity.  
325 Uniformity of the etched surface was observed to be  
326 greater in TMAH than KOH. TMAH samples etched with  
327 concentrations of 13 wt% showed lower surface roughness  
328 than those etched at 10 and 20 wt%. The optimum etching  
329 temperature was found to be 85°C.

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

335 Samples etched with TMAH only were observed to be  
336 less rough than those with the addition of 4% IPA. Addition  
337 of IPA caused striations to occur across the wafer



(a)



(b)

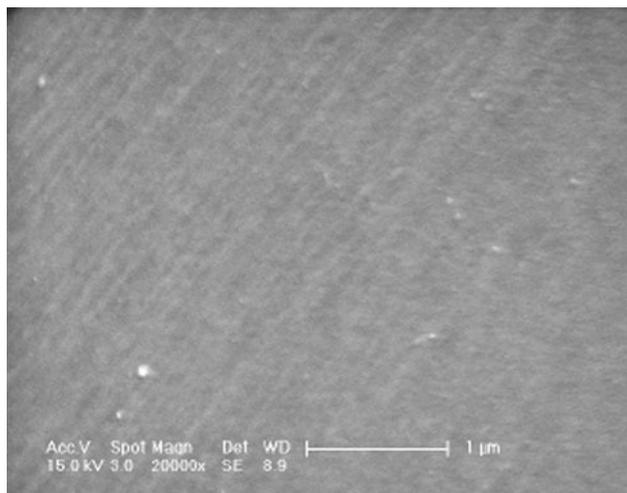
**Fig. 9** AFM surface profile of silicon sidewall etched in 13 wt% TMAH at 85°C **a** SEM image of vertical mirror **b** AFM surface profile

increasing surface roughness, however, the addition of IPA 338  
did eliminate the formation of hillocks on the {111} sur- 339  
face (Fig. 10). Hillocks formed during TMAH etches were 340  
significantly smaller and fewer than seen in KOH etchants. 341

### 342 3.4 Reflectivity tests

Optimal alignment was achieved by manipulating the chip 343  
when set at a distance of 200 µm from the tip of the fiber 344  
optic displacement sensor. This distance gives the optimal 345  
voltage output from the displacement sensor. The maxi- 346  
mum voltages were retrieved during static testing 347  
(Table 1). Silicon wafer surface samples of polished silicon 348  
and unpolished silicon were used as reference values. 349

Polished silicon showed the highest static output volt- 350  
age, 1.66 V, whilst unpolished the lowest; maximum 351  
voltage output for the displacement sensor is 5 V. Polished 352

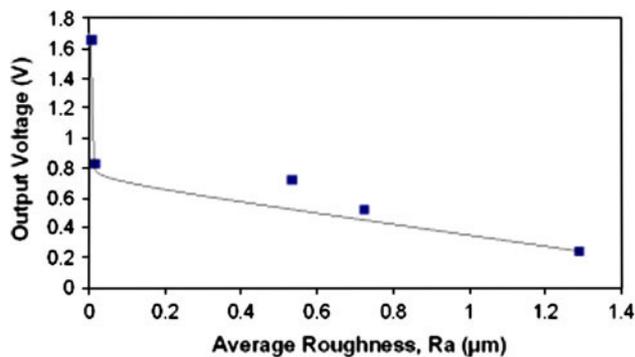


**Fig. 10** Samples etched in 13 wt% TMAH + IPA showed striation and rougher surfaces than those etched with TMAH only

353 and unpolished silicon samples were much easier to align  
 354 than sidewall samples as they had a much greater depth  
 355 >2 mm when compared to etched samples which had a  
 356 depth 200–381 μm. TMAH showed lowest surface roughness  
 357 and highest maximum voltage, 0.83 V during static  
 358 tests, this increased significantly to 2.48 V once coated  
 359 with 10 nm Al. DRIE samples tested were those with the  
 360 lowest roughness ( $R_a = 533$  nm). DRIE samples showed  
 361 output voltage of 0.72 V which increased to 1.12 V when  
 362 the Pd-Au coating was sputtered on the surface. TMAH + IPA  
 363 samples showed much lower static output voltage  
 364 of 0.35 V due to the striations scattering light away from  
 365 the fiber optic sensor. Samples etched in 30 wt%  
 366 KOH + IPA gave an output voltage of 0.24 V whilst  
 367 25 wt% KOH + IPA gave an output voltage of 0.04 V  
 368 showing slower etch rates increase reflectivity. However  
 369 due to defects in KOH etched silicon arising because of  
 370 poor masking, reflectivity is lower than DRIE samples.  
 371 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

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

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

#### 4 Conclusions

401  
 402 Etching of silicon in multifunctional BioMEMS represents  
 403 a complex procedure due to the number of variables and  
 404 the associated outcomes of each. Fabrication of vertical  
 405 mirror surfaces via anisotropic deep etching through wafers  
 406 for BioMEMS is achievable by controlling the etch rate  
 407 and extraction of gaseous products from the etchant. One

Author Proof

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,  $\leq 70^\circ\text{C}$  resulted in optimal smoothness, the lowering of temperature helps slow the etch rate. For TMAH etchants, the optimal was found to be  $85^\circ\text{C}$ .

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  $\text{SiO}_2$  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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## References

- Agarwal R (2007) Fabrication of vertical mirrors using plasma etch and KOH: IPA polishing. *J Micromech Microeng* 17(1):26–35
- Bashir R (2004) BioMEMS: state-of-the-art in detection, opportunities and prospects. *Adv Drug Deliv Rev* 56(11):1565–1586
- Biswas K, Kal S (2006) Etch characteristics of KOH, TMAH and dual doped TMAH for bulk micromachining of silicon. *Microelectron J* 37(6):519–525
- Craciun G, Blauw MA, Van Der Drift E, Sarro PM, French PJ (2002) Temperature influence on etching deep holes with  $\text{SF}_6/\text{O}_2$  cryogenic plasma. *J Micromech Microeng* 12(4):390–394
- Grayson ACR, Shawgo RS, Johnson AM, Flynn NT, Li Y, Cima MJ (2004) A BioMEMS review: MEMS technology for physiologically integrated devices. *Proc IEEE* 92(1):6–21
- Hashim MR, Salih KQ (2005) Optical properties of treated and untreated monocrystalline  $\langle 111 \rangle$ ,  $\langle 100 \rangle$ ,  $\langle 110 \rangle$  wafers in the visible region at room temperature. *Microelectron Eng* 81(2–4):243–250
- Kendall DL (2003) Vertical Etching of silicon at very high aspect ratios. *Annu Rev Mat Sci* 9(1):373–403
- Mackay RE, Le HR (2008) Development of micro-tweezers for tissue micro-manipulation. In: *Proceedings of the 2nd International Conference on Bioinformatics and Biomedical Engineering*, pp 1551–1554, Shanghai, China, 2008
- Mackay RE, Le HR, Keatch RP (2011) Design optimization and fabrication of SU-8 based electro-thermal micro-grippers. *J Micro/Nano Mechatron* 6(1–2):13–22
- Marxer C, Thio C, Gretillat MA, de Rooij NF, Battig R, Anthamatten O (1997) Vertical mirrors fabricated by deep reactive ion etching for fiber-optic switching applications. *J Microelectromech Syst* 6(3):277–285
- Matsunaga T, Esashi M (2002) Acceleration switch with extended holding time using squeeze film effect for side airbag systems. *Sens Actuators A Phys* 100(1):10–17
- Palik ED, Gray HF, Klein PB (1983) A Raman study of etching silicon in aqueous KOH. *J Electrochem Soc* 130(4):956–959
- Powell O (2001) Anisotropic etching of 100 and 110 planes in (100) silicon. *J Micromech Microeng* 11(3):217–220
- Price JB (1973) Anisotropic etching of silicon with KOH– $\text{H}_2\text{O}$ –isopropyl alcohol. In: Huff HR, Burgess RR (eds) *Semiconductor silicon*. Princeton, NJ, pp 339–353
- Sato K, Shikida M, Yamashiro T, Tsunekawa M, Itob S (1999a) Roughening of single-crystal silicon surface etched by KOH water solution. *Sens Actuators A* 73(1–2):122–130
- Sato K, Shikida M, Yamashiro T, Asaumi K, Iriye Y, Yamamoto M (1999b) Anisotropic etching rates of single-crystal silicon for TMAH water solution as a function of crystallographic orientation. *Sens Actuators A* 73(1–2):131–137
- Seidel H, Csepregi L, Heuberger A, Baumgartel H (1990) Anisotropic etching of crystalline silicon in alkaline solutions. *J Electrochem Soc* 137(11):3612–3626
- Suresh S (2007) Biomechanics and biophysics of cancer cells. *Acta Biomater* 3(4):413–438
- Williams KR, Muller RS (1996) Etch rates for micromachining processing. *J Microelectromech Syst* 5(4):256–269
- Yang CR, Chen PY, Chiou YC, Lee RT (2005) Effects of mechanical agitation and surfactant additive on silicon anisotropic etching in alkaline KOH solution. *Sens Actuators A* 119(1):263–270
- Yun SS, You SK, Lee JH (2006) Fabrication of vertical optical plane using DRIE and KOH crystalline etching of (110) silicon wafer. *Sens Actuators A* 128(2):387–394