

# Anisotropic etching of silicon in KOH + Triton X-100 for 45° micromirror applications

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**Abstract** The etching process of monocrystalline silicon in potassium hydroxide solution with addition of Triton X-100 surfactant at different temperatures is studied. It is shown that decreasing the temperature lowers etch rates and improves surface morphology of Si (*hkl*) planes. Based on Arrhenius plots of etch rates, activation energies for Si (100) and (110) planes are determined. The obtained activation energies are lower than the ones for pure KOH solutions, reported in the literature. The possible explanation of this phenomenon, based on the temperature dependence of surfactant adsorption, is presented. Besides, it is established that the convex corners of microstructures etched at different temperatures are slightly curved, probably formed by planes vicinal to {221} plane. Furthermore, the {110} sidewalls inclined at 45° to Si (100) substrate are characterized by low surface roughness in a whole range of considered temperatures (60–90 °C). Thus, the {110} sidewalls could be used as MEMS micromirrors for reflecting light beam at an angle of 90°. It is indicated that the KOH solution containing Triton X-100 is of particular interest for fabrication of elongated mesa structures containing 45° micromirrors and waveguides concurrently.

## 1 Introduction

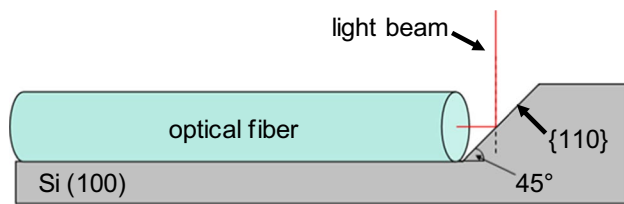
Bulk micromachining of silicon is a popular method of fabricating spatial structures of microsystems, such as micro-electro-mechanical systems (MEMS) and micro-opto-electro-mechanical systems (MOEMS). The technology is often implemented through wet anisotropic etching (Hoffmann and Voges 2002; Sankar and Das 2013; Niesel and Dietzel 2014), which is not only a cheap technique (as compared to dry etching), but also allows one to obtain sidewalls defined by crystal planes, i.e. inclined at specific angles towards a surface of the substrate. This unique feature of the anisotropic etching process gives, for example, a possibility of fabricating {110} sidewalls inclined at 45° to the surface of the (100) substrate. Such sidewalls can serve as micromirrors for 90° out-of-plane light reflection (Strandman et al. 1995; Resnik et al. 2005; Yagyu et al. 2010; Xu et al. 2011; Rola et al. 2014), as shown schematically in Fig. 1, which is attractive in the field of MOEMS, provided that those sidewalls have low surface roughness.

The {110} planes develop as sidewalls of spatial structures in (100) substrates on condition that edges of the masking windows are aligned in <100> directions and the etch rate ratio  $R_{(100)}/R_{(110)}$  is distinctly higher than one. The second condition can be fulfilled in alkaline solutions containing amphiphilic compounds, such as surfactants (Resnik et al. 2005; Yagyu et al. 2010; Xu et al. 2011; Rola et al. 2014; Pal and Sato 2009). It has been shown recently that nonionic surfactant Triton X-100 can be successfully applied as an additive to potassium hydroxide solution (Rola and Zubel 2013a, b; Rola et al. 2014; Pal et al. 2015). The KOH + Triton X-100 solution may be an alternative to popular etching compositions, such as KOH + isopropanol (due to a smoother {110} surface) and TMAH

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**Fig. 1** Schematic illustration of light reflection (coming out of optical fiber) from a {110} sidewall inclined at 45° to Si (100) substrate

(tetramethylammonium hydroxide) + Triton X-100 (due to a lower cost of KOH than TMAH).

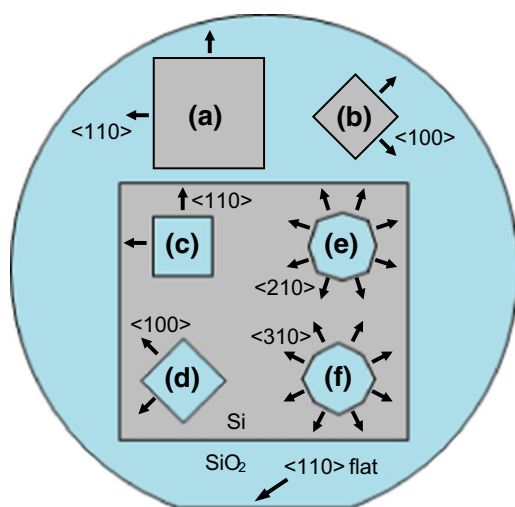
It has been already established that the best surface morphologies and the most regular shapes of spatial microstructures etched in KOH + Triton X-100 are achievable at low hydroxide concentrations (2–3 M) (Rola and Zubel 2013a) and, additionally, the etching process does not significantly depend on Triton X-100 concentration within a wide range (10–200 ppm of the surfactant) (Rola and Zubel 2013b). In this paper, the effect of temperature and solution stirring on silicon etching process is investigated. The research is aimed at finding optimal etching conditions in low concentrated potassium hydroxide solution with the Triton surfactant. The possible application of the above-mentioned etchant for fabrication of spatial microstructures employing 45° micromirrors is also discussed.

## 2 Experimental details

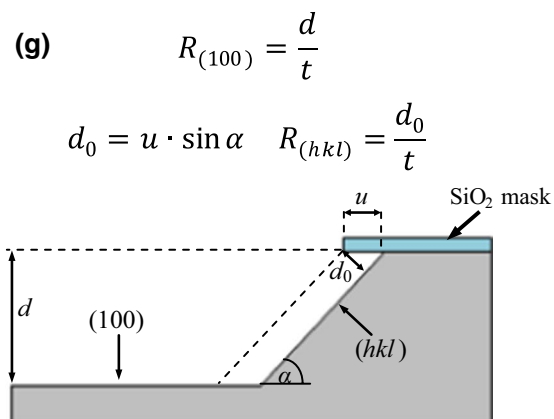
The etching experiments were conducted using 3" (100) and (110) silicon wafers (single side polished) of 2–10

Ω cm resistivity and 380 μm thickness, supplied by Institute of electronic materials technology (ITME), Poland. In order to obtain masking windows, the wafers were thermally oxidized up to a thickness of about 1 μm and then patterned in photolithography (on the polished side), which was followed by selective etching in BHF (buffered hydrofluoric acid) solution. Subsequently, the wafers were diced into 17 mm × 17 mm samples. The etching masks containing different patterns were used. The shapes of masking patterns utilized for investigation of temperature dependence of etching process are shown in Fig. 2a–f.

The Si samples were etched in 2 M (10 wt%) potassium hydroxide (KOH) aqueous solution containing 50 ppm (by volume) of Triton X-100 surfactant, supplied by Sigma-Aldrich. The KOH solution was prepared from potassium hydroxide pellets (supplied by Chempur, Poland) and deionized water (1 MΩ cm). During etching, the samples were situated vertically in a Teflon holder immersed in the etching solution of the volume 1.5 dm<sup>3</sup>, contained in a glass vessel. The etching processes were carried out under atmospheric pressure and at stabilized, elevated temperatures (60–90 °C). Stirring of the etching solution was carried out mechanically with a frequency of rotation 210 rpm using the Heidolph RZR 2020 stirrer equipped with a Teflon paddle. The Teflon paddle was fully immersed in the solution during etching, though it was located markedly above the samples holder. Due to an expected increase of etch rates with temperature (according to Arrhenius equation), the etching time was dependent on temperature: 240 min for 60 °C, 150 min for 70 °C, 105 min for 80 °C, 82 min for 90 °C. This was supposed to yield comparable etching depths at different etching temperatures. The etching time for fabrication of micromirror structures was



**Fig. 2** a–f Shape of masking patterns on Si (100) wafer (not to scale) having edges aligned in directions: a, c <110>, b, d <100>, e <210>, f <310>, g illustration of determining etch rate on Si (100) substrate: by etching depth method ( $R_{(100)}$ ) and by mask undercut method ( $R_{(hkl)}$ )



**Table 1** Etch rates of Si (100) and (110) planes in non-stirred (N) and stirred (S) KOH solution with Triton X-100 at different temperatures, determined by etching depth measurements

$T$ (°C)	$R_{(hkl)}$ ( $\mu\text{m}/\text{min}$ )				$R_{(100)}/R_{(110)}$ N	$R_{(100)}/R_{(110)}$ S
	(100) N	(110) N	(100) S	(110) S		
60	0.032	0.025	0.052	0.040	1.278	1.320
70	0.062	0.040	0.080	0.061	1.556	1.311
80	0.101	0.055	0.110	0.084	1.817	1.305
90	0.148	0.099	0.180	0.092	1.496	1.952

selected individually for each structure so that the etching depth was adequate.

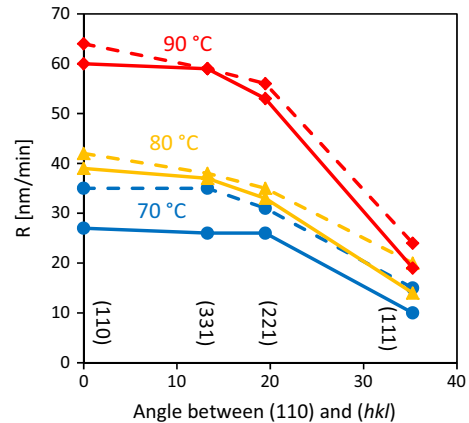
The etch rates  $R$  of the Si (100) and (110) substrates were evaluated by the etching depth measurement with a micrometric tool (within the accuracy of 1  $\mu\text{m}$ ). The etch rates of several ( $hkl$ ) planes were determined based on mask undercut of mesa structures on Si(100) substrate in specific  $\langle uvw \rangle$  directions (see Fig. 2c–g). The surface morphology and the shapes of the etched spatial microstructures were investigated using SEM (scanning electron microscope). The surface morphology of (100) and (110) substrates was examined within a representative area near the center of the large etched region (about 3 mm  $\times$  3 mm) for each of both the substrates. The surface roughness of {110} planes forming sidewalls of spatial microstructures was visually evaluated based on SEM images of the microstructures. Prior to the microscopic observations (except for imaging of convex corners), the SiO<sub>2</sub> masks were removed from the Si samples by etching in the 20 % HF (hydrofluoric acid) solution.

### 3 Results and discussion

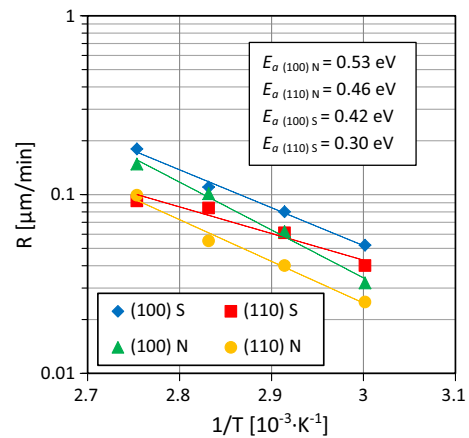
#### 3.1 Effect of temperature and stirring on etch rates

The etch rates of (100) plane are higher than those of (110) plane (which results in  $R_{(100)}/R_{(110)}$  anisotropy ratios higher than one) in a whole range of considered temperatures (60–90 °C), with stirring as well as without stirring of the etching solution containing surfactant, as shown in Table 1. The etch rates increase as the temperature rises, though the influence of temperature on etch rate ratio  $R_{(100)}/R_{(110)}$  is not evident. Generally, stirring of the solution increases the etch rates of both planes. The etch rate increase under influence of stirring may be a result of the impact of diffusion on etch rate at low hydroxide concentration, as suggested by Glembocki et al. (1991). On the basis of Table 1, one cannot determine the impact of stirring on the etch rate anisotropy of (100) and (110) planes.

The temperature rise and application of stirring also increase the etch rates of ( $hkl$ ) planes that were determined on the basis of mask undercut measurements (see Fig. 2c–g), as shown in Fig. 3. The impact of stirring on the etch



**Fig. 3** Etch rates of Si ( $hkl$ ) planes in non-stirred (solid line) and stirred (dashed line) KOH solution with Triton X-100 at different temperatures (filled circle 70 °C, filled triangle 80 °C, filled diamond 90 °C), determined based on mask undercut measurements



**Fig. 4** Arrhenius plots and calculated activation energies for etching of Si (100) and (110) planes in non-stirred (N) and stirred (S) KOH solution with Triton X-100 (based on etching depth measurements)

rates is more pronounced at low temperature (70 °C). The etch rates of the considered planes are in a following relation:  $R_{(110)} > R_{(331)} > R_{(221)} > R_{(111)}$ , which can be also presented as:  $R_{SM} > R_{SM/TM} > R_{TM}$ , where SD (step dihydride), SM (step monohydride) and TM (terrace monohydride) are types of surface bonds (Kasparian et al. 1997; Gosálvez

et al. 2007). Nonetheless, it should be stated that the differences between etch rates of (110), (331) and (221) planes are relatively small.

The dependence of the etch rates on temperature can be also presented in the form of Arrhenius plot, which allows one to determine values of activation energy (Fig. 4), based on the equation:

$$R = R_0 \exp\left(\frac{-E_a}{kT}\right) \quad (1)$$

where  $R$  etch rate,  $R_0$  pre-exponential factor,  $E_a$  activation energy,  $k$  Boltzmann constant,  $T$  temperature.

One can see that stirring of the solution decreases the activation energy of etching reaction, though the reason for this phenomenon is not known. One can also notice that the values of activation energy obtained from the etch rates in KOH solution with Triton X-100 ( $E_{a(100)} = 0.53$  eV,  $E_{a(110)} = 0.46$  eV) are lower than those in the case of etching in pure potassium hydroxide solutions, obtained by Seidel et al. (1990) ( $E'_{a(100)} = 0.57$  eV,  $E'_{a(110)} = 0.59$  eV) and Shikida et al. (2000) ( $E'_{a(100)} = 0.62$  eV,  $E'_{a(110)} = 0.60$  eV). As seen above, the differences are definitely larger for (110) plane. Although it is worth mentioning that the KOH concentration used in the present work (10 wt%) is lower than the KOH concentrations used by Seidel et al. (1990) and Shikida et al. (2000) (20 and 34 wt%, respectively), the activation energy does not depend much on the potassium hydroxide concentration, as shown by Seidel et al. (1990). Furthermore, reduction of activation energy under the influence of nonionic surfactant has been also observed for TMAH solutions by Tang et al. (2010).

The observed decrease of activation energy is puzzling since such a phenomenon is normally associated with the increase of the reaction rate whereas the results indicate that the silicon etch rates are reduced in the presence of the surfactant. In this context, it should be noted that surfactant molecules are believed to be adsorbed on Si surface in aqueous solutions of hydroxides, such as KOH or TMAH, thus impeding access of reactants (water molecules and hydroxide ions) to the Si surface and, as a consequence, reducing the etch rate (Allongue et al. 1995; Pal et al. 2009). Therefore, possibly, the Triton X-100 surfactant is not involved in the etching reaction and its role in the process is limited only to hindering the access of reactants to the silicon surface. In such a case, the activation energy of the reaction itself should remain unchanged after addition of the surfactant. Thus, if the Arrhenius plot-derived activation energy ( $E_a$ ) decreases, it can be presumed that the real activation energy ( $E'_a$ ) of the reaction is masked by the dependence of surfactant adsorption on temperature. As shown by Tang et al. (2009), the thickness of Triton X-100 surfactant layer on Si (100) and (110) surfaces increases as

temperature rises up to 80 °C. Consequently, the increase in surfactant adsorption with temperature may counteract the increase in the etch rate with temperature, which is observed on the basis of Arrhenius plots as the activation energy decrease.

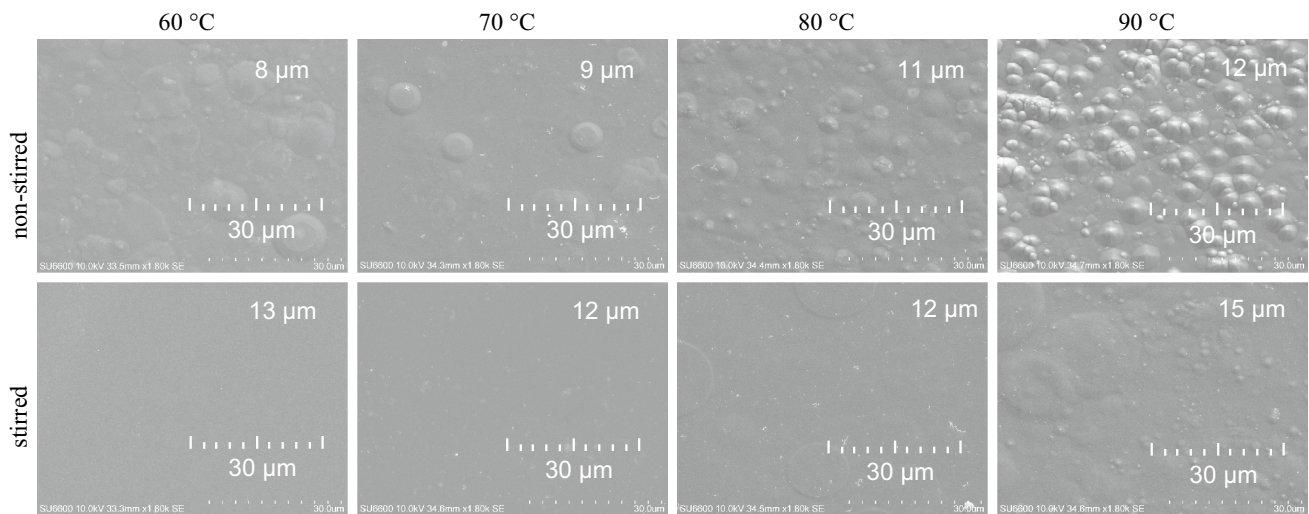
The above hypothesis can be also discussed on the basis of Arrhenius Eq. (1). The pre-exponential factor  $R_0$  is, inter alia, a function of frequency with which reactant molecules collide. In the case of silicon etching in alkaline solution (e.g. KOH) this corresponds, in a nutshell, to the frequency of collisions between liquid reactants ( $H_2O$  and  $OH^-$ ) and silicon surface. This frequency is sure to be limited if a layer of surfactant molecules is adsorbed on the Si surface. Therefore, as the surfactant adsorption density increases with temperature, the  $R_0$  factor decreases. Because the temperature dependence of Triton X-100 adsorption on Si surface in aqueous solution follows an Arrhenius relationship (Tang et al. 2009), it is presumed that the equation for the etch rate in the presence of nonionic surfactant should be written as:

$$\begin{aligned} R &= R_0 \exp\left(\frac{-E'_a}{kT}\right) = \frac{R'_0}{\exp\left(\frac{-E_s}{kT}\right)} \exp\left(\frac{-E'_a}{kT}\right) \\ &= R'_0 \exp\left(\frac{-(E'_a - E_s)}{kT}\right) = R'_0 \exp\left(\frac{-E_a}{kT}\right) \end{aligned} \quad (2)$$

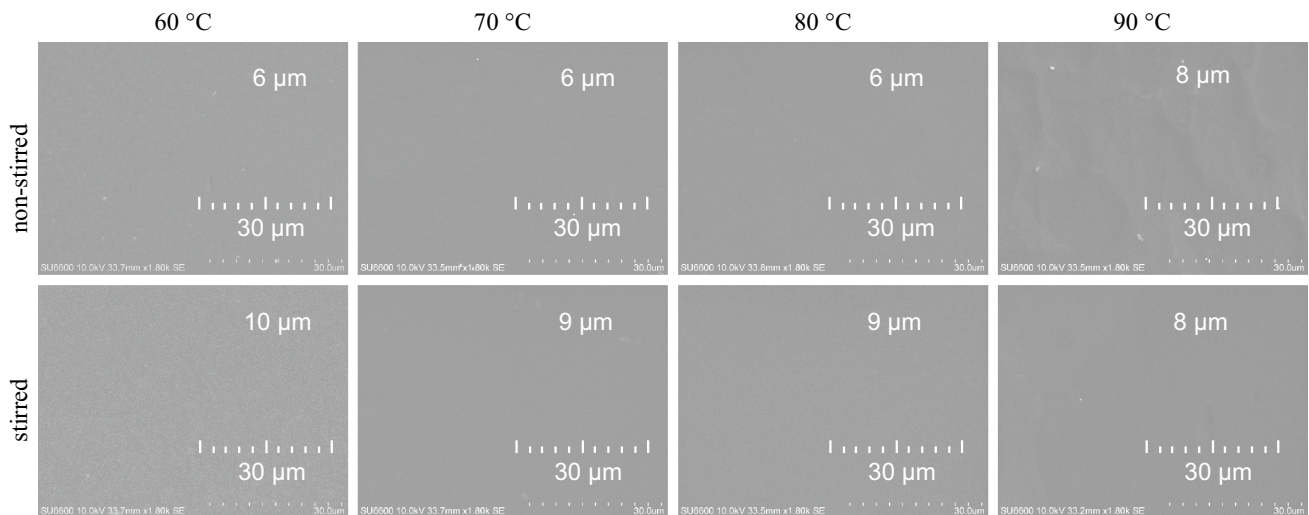
where  $E_a$  energy derived from Arrhenius plot (see Fig. 4),  $E'_a$  activation energy of etching reaction (the same in pure and surfactant-added solutions),  $E_s$  activation energy of surfactant adsorption.

As mentioned in one of the previous paragraphs of this section, the difference between  $E_a$  and  $E'_a$  is especially pronounced for (110) plane ( $(E'_a - E_a) = 0.13$  eV (Seidel et al. 1990),  $(E'_a - E_a) = 0.14$  eV (Shikida et al. 2000)). It can be concluded from (2) that this difference should be equal to activation energy of the surfactant adsorption ( $E_s$ ) on silicon surface. Tang et al. (2009) have shown that the activation energy of Triton X-100 adsorption on Si (110) surface in aqueous solution is about 0.15 eV, which is quite close to the above values. The bigger inconsistency appears for (100) plane:  $E_s \approx 0.007$  eV (Tang et al. 2009) whereas  $(E'_a - E_a) = 0.04$  eV (Seidel et al. 1990),  $(E'_a - E_a) = 0.09$  eV (Shikida et al. 2000).

Nevertheless, it has to be said that the solution to the problem may not be so straightforward. One should be aware that the explanation presented above is a simplified model. It does not take into consideration certain factors. First of all, the etching reaction of silicon in alkaline solutions is not a one-step reaction, i.e. it probably undergoes several elementary steps. Moreover, when the step flow model of anisotropic etching is considered, it should be



**Fig. 5** SEM images (*slanted view*) of Si (100) surfaces etched in stirred and non-stirred KOH solution with Triton X-100 at different temperatures (etching depths are *marked*)

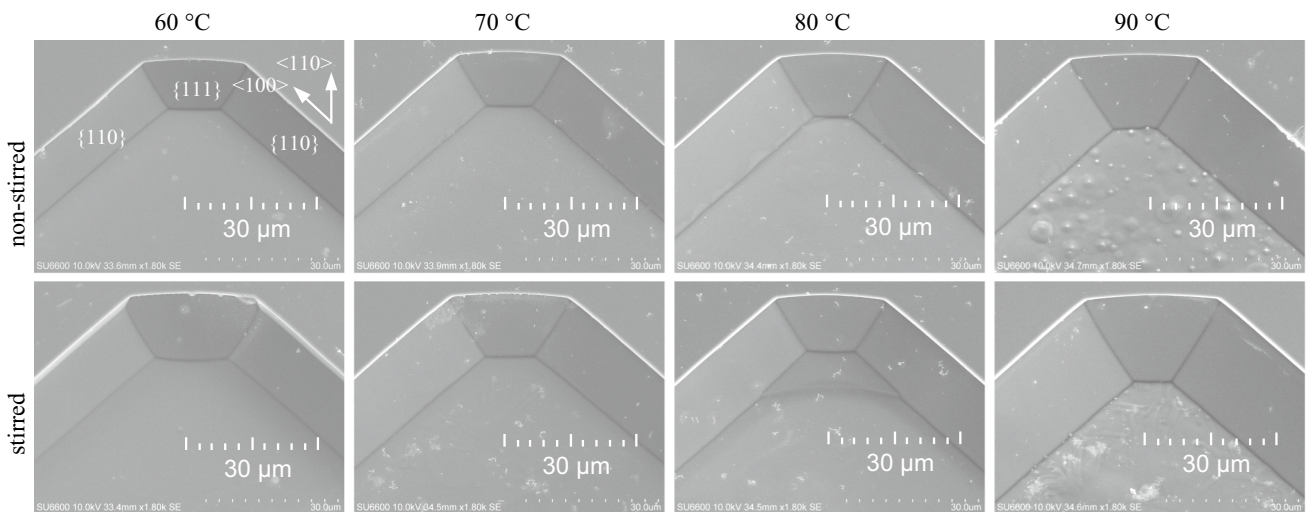


**Fig. 6** SEM images (*top view*) of Si (110) surfaces etched in stirred and non-stirred KOH solution with Triton X-100 at different temperatures (etching depths are *marked*)

also taken into account that, during etching, each silicon crystal surface is composed of steps and terraces, of which each type has its specific activation energy of etching (Wind et al. 2002; Gosálvez et al. 2006). This implies that the activation energy for an etched (*hkl*) surface depends on fractions of step and terrace sites forming a particular surface. One of conceivable methods of verifying the hypothesis discussed in the previous paragraphs is to carry out computer simulations of silicon anisotropic etching that include temperature dependence of surfactant adsorption on Si surface. It is also desirable that the experimental values of activation energy would be confirmed by other researchers.

### 3.2 Effect of temperature and stirring on surface morphology and shape of spatial structures

The change of temperature and application of stirring affects surface morphology of Si (100) and (110) substrates (Figs. 5, 6), though the impact on the topography of (110) surface is little. The (110) surface is generally smooth after etching in the KOH solution with the surfactant, and only at high temperature (90 °C) and when the solution is not stirred during etching process the (110) substrate exhibits surface roughness that is visible in SEM images. The roughness of the (100) surface increases as the temperature rises. Furthermore, stirring of the solution makes the



**Fig. 7** SEM images (slanted view) of concave structures with  $\{110\}$  sidewalls in Si (100) substrates etched in stirred and non-stirred KOH solution with Triton X-100 at different temperatures

(100) surface smoother. This is consistent with the results for (110) surface and suggests that, generally, decreasing temperature and application of stirring is beneficial for surface smoothness of Si ( $hkl$ ) planes etched in KOH solutions with Triton X-100 surfactant.

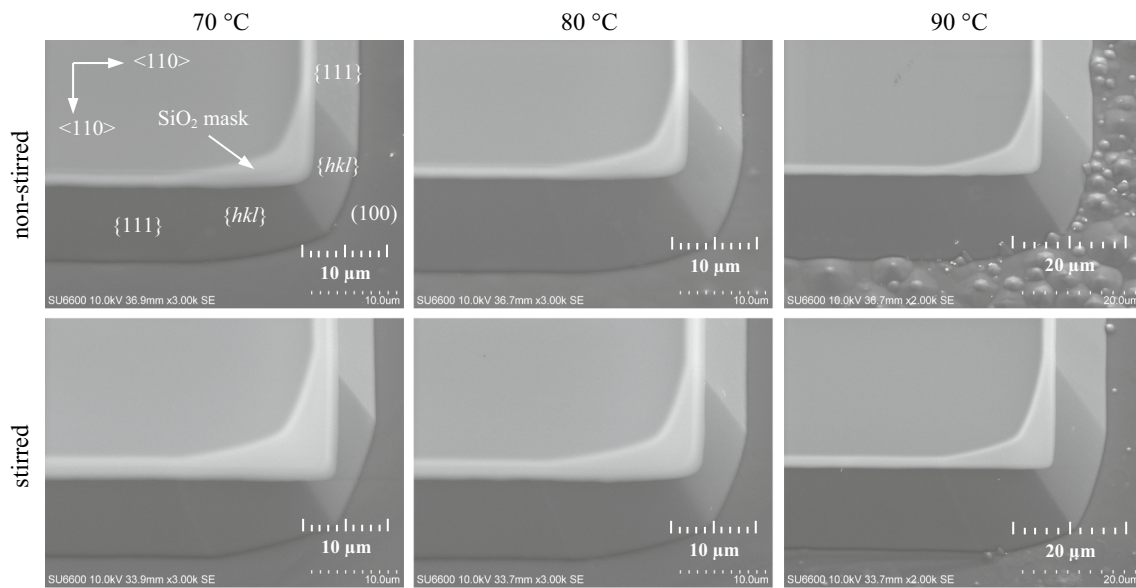
When comparing the above results with the literature data by Palik et al. (1991), one finds that the Si surface roughness is also reduced under the influence of stirring of a pure KOH solution (without surfactant). In the case of the temperature impact on the Si surface roughness in pure alkaline solutions, the literature data from different sources (Palik et al. 1991; Yang et al. 2005; Dutta et al. 2011) are not consistent with each other. Moreover, Yang et al. (2005) have shown that the roughness of Si (100) surfaces etched in KOH or TMAH solutions containing nonionic surfactant decreases as the temperature increases, which is contrary to the results obtained in the present work. The aforementioned difference may result from different molecular structures of surfactants used by Yang et al. (2005).

Because the anisotropy ratio  $R_{(100)}/R_{(110)}$  is larger than one throughout the temperature range from 60 to 90 °C (see Table 1), etching processes conducted at these temperatures result in appearing of  $\{110\}$  sidewall planes inclined at 45° to the (100) substrate when the etching mask is aligned in  $\langle 100 \rangle$  directions (see Fig. 2b), as shown in Fig. 7. The fact that sidewalls etched in 2 M KOH solution containing Triton X-100 using  $\langle 100 \rangle$ -oriented mask are truly  $\{110\}$  planes of 45° inclination has been already proven in the previous paper (Rola and Zubel 2013a). It can be seen from SEM images in Fig. 7 that the  $\{110\}$  sidewalls are smooth at all considered temperatures, in the case of both stirring and non-stirring conditions. One can also notice bright lines appearing above the  $\{110\}$  sidewalls,

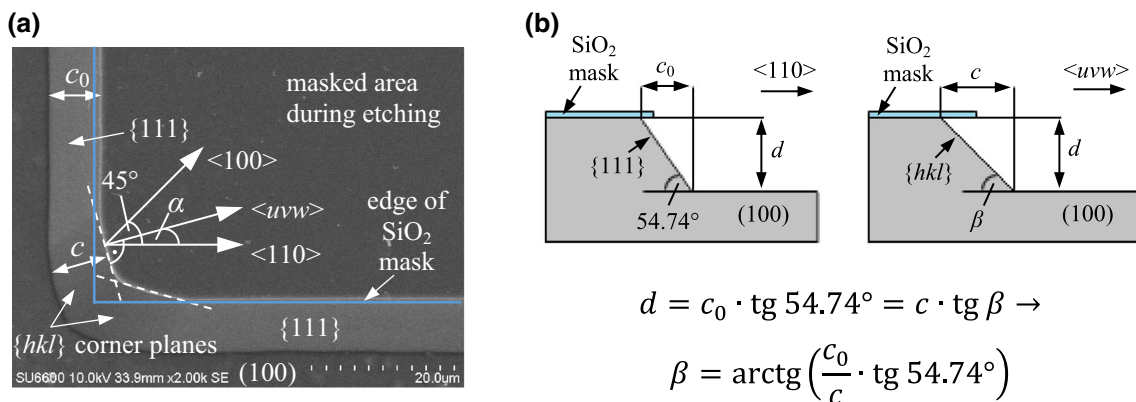
which are probably the  $\{100\}$  planes since the etch rate ratios  $R_{(100)}/R_{(110)}$  are only a few hundredths greater than one (see Table 1). Additionally, the concave structures have  $\{111\}$  planes (inclined at 54.74° to the substrate) in the corners. The  $\{111\}$  plane exhibits the lowest etch rate of all ( $hkl$ ) planes in silicon crystal and, as a result, always is developed (to a greater or lesser extent) as a sidewall of a concave structure, despite the shape of the etching mask. Furthermore, one can observe in Fig. 7 that the lower is the temperature, the larger is the area of the  $\{111\}$  plane in the concave corner with respect to the  $\{110\}$  sidewalls.

As it has been demonstrated in the previous paper (Rola and Zubel 2013a), addition of Triton X-100 surfactant to the KOH solution causes undercut reduction of convex corners of structures bounded by  $\{111\}$  sidewalls. The Fig. 8 shows that the shape of the convex corners does not change significantly as a function of temperature and under influence of solution stirring. Therefore, one can imagine that those corners are formed by the same or very similar (i.e. vicinal)  $\{hkl\}$  planes. Each corner consists of two symmetrical slanted planes, so it can be presumed that both the corner planes belong to the same  $\{hkl\}$  family of planes. This assumption is supported by the fact that each of these convex corners is intersected by a  $\{100\}$  plane that is perpendicular to the (100) substrate and which is a plane of symmetry for this substrate.

In order to evaluate which  $\{hkl\}$  planes appear in the convex corners, a direction  $\langle uvw \rangle$  in which those planes develop and an inclination angle of those planes to the (100) substrate need to be known. The wanted  $\langle uvw \rangle$  direction can be determined on the basis of an angle which it forms with the  $\langle 110 \rangle$  direction. The method of establishing values of both above-mentioned angles is shown in Fig. 9. The obtained



**Fig. 8** SEM images (*slanted view*) of convex structures with {111} sidewalls in Si (100) substrates etched in stirred and non-stirred KOH solution with Triton X-100 at different temperatures



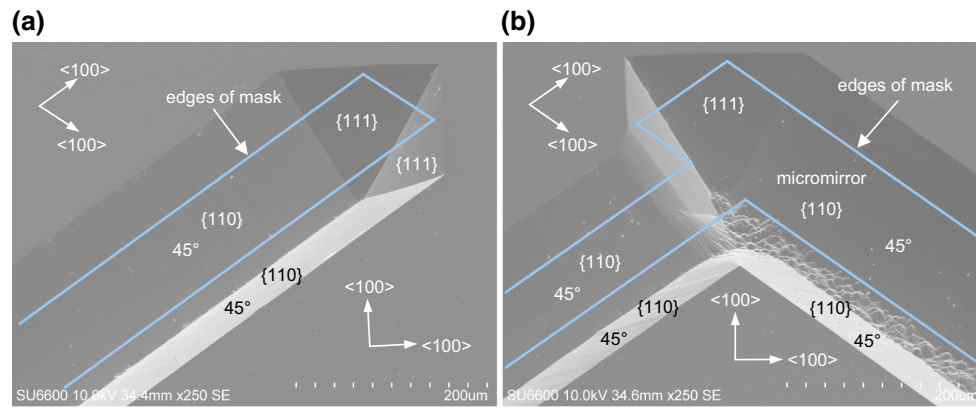
**Fig. 9** Method of determining α and β angles of {hkl} planes of etched convex corner: **a** SEM image (*top view*), **b** schematic illustration (*cross-sectional view*)

results are shown in Table 2. The α angle varies from about 15.5° to 19.5°, and β angle varies from about 48° to about 51.5°. Values of α and β within the above ranges occur for a {221} plane, for which these quantities are 18.43° and 48.19°, respectively. Because of a quite large spread of α and β values, the corner plane might not be exactly the {221} plane, but a high-index plane that is vicinal to the {221} plane. Moreover, the corner sidewalls seem to be a little curved in SEM images (Fig. 8), which suggests that a single sidewall of the convex corner may be composed of more than one {hkl} plane vicinal to the {221} plane. When comparing those results with microstructures etched in 25 % TMAH + Triton X-100 solution

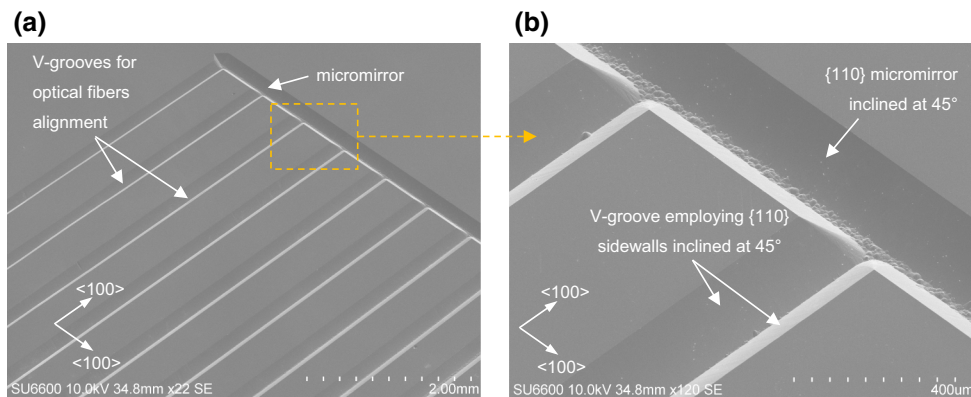
**Table 2** Values of α and β angles of {hkl} convex corner sidewalls etched in stirred and non-stirred KOH + Triton X-100 solution at different temperatures

T (°C)	Non-stirred		Stirred	
	α (°)	β (°)	α (°)	β (°)
70	15.6 ± 3.0	51.1 ± 0.8	16.4 ± 1.6	51.4 ± 1.0
80	16.3 ± 2.1	50.6 ± 0.6	16.1 ± 2.2	51.0 ± 0.7
90	19.5 ± 2.4	50.5 ± 1.2	17.8 ± 2.4	48.1 ± 1.4

Each value is an average of measurements of 12 different corner sidewalls etched on a single substrate (for each value, standard deviation is shown)



**Fig. 10** SEM images (*slanted view*) of concave structures etched through windows of different shapes on Si (100) substrate in stirred KOH solution with Triton X-100 at 90 °C



**Fig. 11** SEM images (*slanted view*) of a structure consisting of V-grooves and a single micromirror etched in Si (100) substrate in stirred KOH solution with Triton X-100 at 90 °C

(Pal et al. 2011), one can notice that the convex corners etched in the TMAH-based etchant are sharper, though they are probably also composed of more than one crystal plane, presumably planes vicinal to {110} plane.

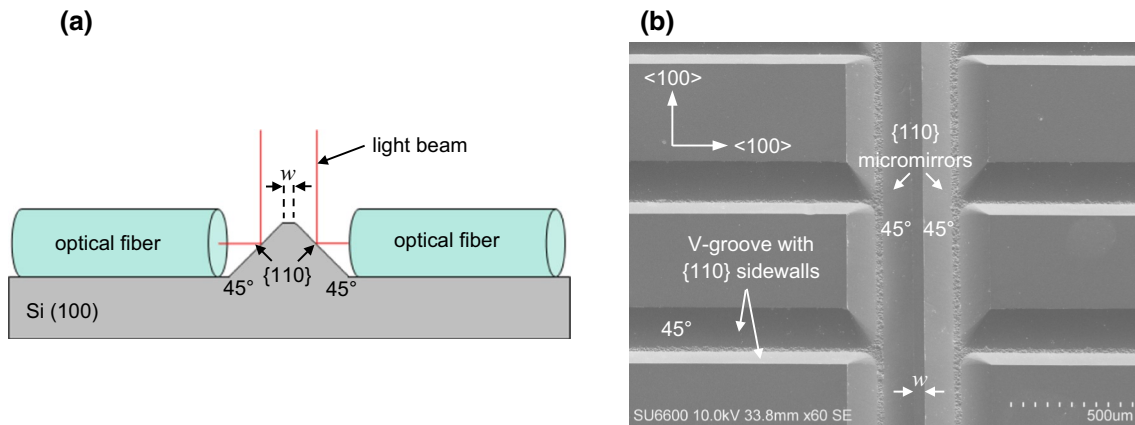
### 3.3 Anisotropic etching of 45° micromirrors

As it was mentioned in the introduction section of the present paper, the smooth {110} planes inclined at 45° to the Si (100) substrate can be used as micromirrors for 90° out-of-plane reflection of light in MOEMS. The results described in a previous section of the present paper indicate that the KOH solution containing Triton X-100 surfactant allows one to obtain such microstructures in a wide range of etching temperatures (60–90 °C). Increasing the temperature up to 90 °C, and consequently increasing etch rate up to almost 0.2 μm/min, might be necessary if one desires to fabricate (in a timely manner) a deep structure of 45° micromirrors in the (100) substrate, such as that employing optical fibers having diameter 125 μm.

The potential drawback of the KOH + Triton X-100 solution in MOEMS technology is a distinctive development of {111} planes in the concave corners of the structures (see Fig. 7), since it hampers fabrication of 45° micromirrors and V-grooves for optical fibers alignment at the same time. When the etching process is carried out long enough to fabricate a structure for optical fibers of 125 μm diameter, the groove formed of {110} sidewalls becomes completely bounded at its end by {111} planes (Fig. 10a). Nevertheless, the problem can be solved by widening the mask window in a place of micromirror (Fig. 10b). As a consequence of the window widening, the smooth {110} sidewall inclined at 45° to the (100) substrate is developed at the end of the groove.

Although the above-mentioned widening increases an area occupied by a structure on silicon substrate, this ceases to be a problem in the case of etching of a large structure which consists of many grooves combined with a long single micromirror. Figure 11 shows an example of such a





**Fig. 12** A silicon structure for making close two beams leaving optical fibers: **a** schematic illustration (*cross-sectional view*) of a structure and **b** SEM images (*slanted view*) of a structure etched in Si (100) substrate in stirred KOH solution with Triton X-100 at 90 °C

structure which was etched in KOH solution with Triton X-100. Owing to application of a common micromirror for several grooves, the problem of the window widening turns out to be minor. In other words, the structure is widened only at two ends of the micromirror, which is negligible in view of a size of the whole structure. Such a structure can be applied for optical interconnecting of two separate optoelectronic modules, as it has been demonstrated for a similar structure etched in KOH solution containing isopropanol by Hsiao et al. (2009).

Another possible application of the {110} micromirrors inclined at 45° to the (100) substrate is a structure for making close two parallel light beams coming out from opposite optical fibers (Fig. 12a). The similar structure, but using a micro prism for light reflection, has been already proposed by Mori et al. (2005). The structure suggested in the present paper is simple, i.e. it can be fabricated in a single silicon substrate in a one technological step. A distance between two reflected beams can be controlled by adjusting a space between two micromirrors ( $w$  in Fig. 12) and monitoring an etching depth which determines a height at which the reflection occurs. The  $w$  distance depends on a degree of the etching mask undercut in the  $\langle 100 \rangle$  direction (i.e. the undercut over {110} sidewalls), which can be predicted based on the etch rate of the {110} plane and the etching time. The example of a structure discussed above, etched in the KOH solution containing Triton X-100, is shown in Fig. 12b. The structure consists of two smooth long micromirrors, each combined with several grooves for optical fibers alignment.

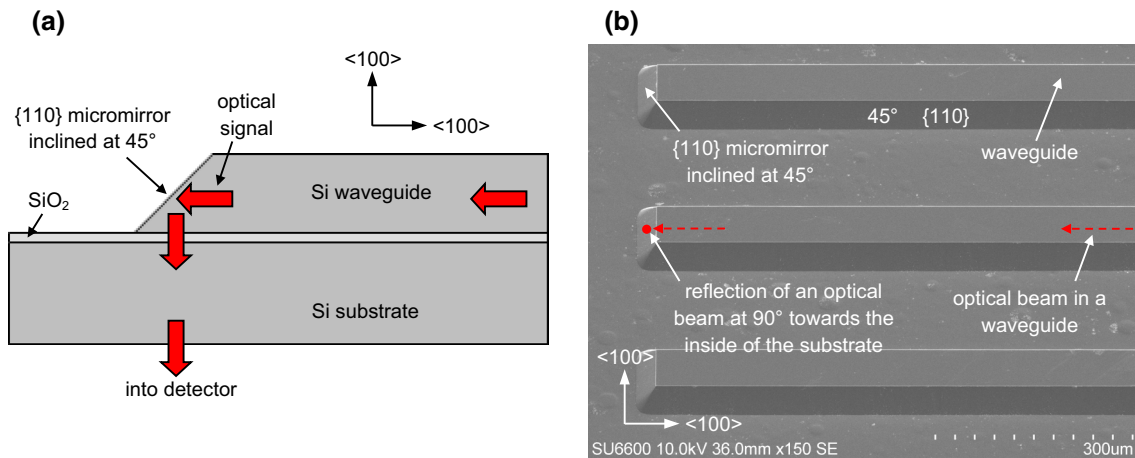
The potassium hydroxide solution with Triton X-100 surfactant can be also used for etching of elongated mesa structures which serve as trapezoidal silicon waveguides employing {110} micromirrors inclined at 45° to the (100) substrate. Such microstructures are potentially attractive for optical interconnecting, as it has been suggested by

Shen et al. (2012). The schematic illustration of a simple waveguide-micromirror structure is shown in Fig. 13a and a view of such mesa structures etched in the KOH solution with the surfactant is shown in Fig. 13b. The path of a light beam in a waveguide and its reflection from a micromirror are also schematically demonstrated (the light beam is reflected in the direction perpendicular to the substrate). The structures shown in Fig. 13b were etched directly on the Si (100) substrate. The real waveguide structures should be separated from the silicon substrate by a thin SiO<sub>2</sub> layer (see Fig. 13a), which, due to a low refractive index as compared to silicon, allows one for trapping a light in a waveguide during propagation within it.

The convex corners of the microstructures shown in Fig. 13b hold the shapes defined by the mask, so a separate micromirror can be used for each waveguide, without the need for the mask widening. The structures employing silicon waveguides do not have to be etched as deeply as in the case of the structures with V-grooves for optical fibers, thus the etching process can be conducted for a shorter time and at a lower temperature (i.e. lower etch rate). Taking also into consideration a low roughness of {110} sidewalls, it can be stated that the KOH + Triton X-100 solution is especially attractive for etching of silicon waveguides with 45° micromirrors.

## 4 Conclusions

The results of anisotropic etching of Si (100) and (110) substrates in KOH solution containing Triton X-100 surfactant were shown and discussed in this paper. It was demonstrated that rising temperature and application of stirring both increase the etch rates of (100) and (110) substrates as well as the etch rates of {110}, {331}, {221} and {111} planes that were determined based on the mask undercut



**Fig. 13** A silicon structure employing a waveguide with micromirror: **a** schematic illustration (*cross-sectional view*) of a structure and **b** SEM images (*slanted view*) of structures etched on Si (100) substrate in stirred KOH solution with Triton X-100 at 75 °C

measurements. In addition, the activation energies of etching were calculated on the basis of Arrhenius plots. It was shown that activation energy for (100) and (110) planes decreases when the etching solution is stirred during process. Comparing the results of the present paper with the results from the literature, one could notice that addition of the Triton X-100 surfactant to the KOH solution decreases the activation energy of etching process. It was shown that this phenomenon can be explained, provided that temperature dependence of surfactant adsorption on silicon surface is taken into consideration.

The surface morphology and the shape of spatial microstructures etched in the KOH solution with Triton X-100 were investigated. It was disclosed that the decrease of temperature and the use of stirring of the etching solution both are beneficial for surface quality of Si (100) and (110) substrates, though the (110) surface is very smooth in almost all considered etching conditions. The change of above-mentioned etching conditions does not influence notably the shapes of convex structures bounded by {111} planes and concave structures bounded by {110} planes, both etched in Si (100) substrate. It was shown that the convex corners are slightly curved, probably composed of planes vicinal to the {221} plane and the {110} sidewall planes of the concave structures are smooth.

The smooth {110} sidewalls can be used as micromirrors for 90° out-of-plane light reflection because of the angle of 45° which they form with the (100) substrate. Therefore, the possibility of application of the KOH + Triton X-100 solution for fabrication of such micromirrors was closely studied. It was suggested that high etching temperatures (90 °C) and special design of the etching mask should be utilized when one desires to obtain deep microstructures employing 45° micromirrors and V-grooves for optical fibers alignment. The microstructures which can be

fabricated in this manner include modules for optical interconnecting and structures for making close parallel optical beams. Besides, the KOH solution containing Triton X-100 surfactant seems to be particularly useful in terms of etching mesa structures which can be used as waveguides and 45° micromirrors at the same time.

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