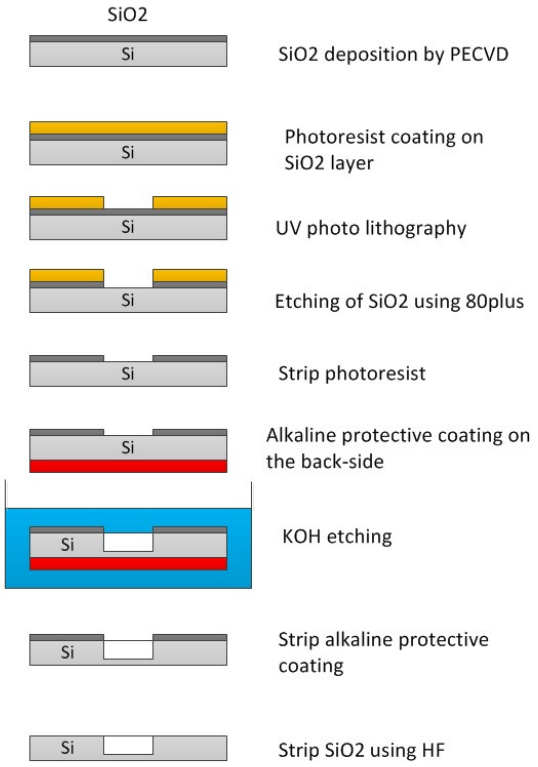


Figure 1. Process flow of KOH etching of (100) Si wafer

Table 1. PECVD silicon oxide deposition parameters of default and Test recipes.

	Default	Test
Set Pressure (Torr)	1.0	1.8
Temperature (°C)	350	350
LF Power		
Forward (W)	0	0
Pulse Time (sec)	0	0
Pulsed	OFF	OFF
LF First	OFF	OFF
RF Power		
Forward (W)	20	140
Pulse Time (sec)	0	0
Pulsed	OFF	OFF
HF First	ON	OFF
RF Automatch		
Capacitor 1	0	77
Capacitor 2	0	26
Auto/Manual/Hold	Auto	Auto
Gas flow (sccm)		
Silane (90% He)	50	265
NH3	710	1000
N2	90	500

1. Introduction

The goal of this project is to perform on-site inspection of potassium hydroxide (KOH) wet etching process, using the tools available at Quattrone Nanofabrication Facility. It has previously been reported[1] that the apparent etch rate along the (100) direction of Si wafer through a hard mask of silicon nitride is 0.9-1.5 µm/min. This report describes KOH etching of (100) Si wafer through a hard mask of silicon oxide, and reveals that the scattered etch rate is ascribed to the etch rates of the different crystal planes exposed during the etching. Figure 1 shows a process flow of KOH etching of (100) Si wafer.

2. Experimental Section

A. Deposition of 300 and 420 nm thick silicon oxide film

A (100) Si wafer was sonicated in acetone and isopropyl alcohol (IPA) for 5 min each, and dried using nitrogen gun. 316 and 424 nm thick silicon oxide films were deposited on the Si wafers for hard masks upon KOH etching, using the default recipe, given by Oxford Instruments, and Test recipe,[2] respectively, in Oxford Plasma Lab 100 (Plasma Enhanced Chemical Vapor Deposition (PECVD)). Table 1 indicates silicon oxide deposition parameters of the default and Test recipes. The deposition rates of the default and Test recipes were 53 and 283 nm/min, respectively.

B. UV lithography using SUSS MicroTec MA6 Gen3 Mask Aligner

Hexamethyldisilazane (HMDS) was vapor primed on the silicon oxide film as an adhesion promoter, using YES oven (Yield Engineering Systems), followed by spin-coating positive photoresist S1818 (Microchem) at 5500 rpm for 30 sec. The photoresist film was baked at 115°C for 5 min on a hot plate, and was exposed to 405 nm UV light with the power of 150mJ/cm^2, using SUSS MicroTec MA6 Gen3 Mask Aligner. The exposed photoresist film was developed in MF319 (Microchem) for 1 min, and was rinsed with deionized (DI) water. The sample was then dried using nitrogen gun.

[1] I. Bajwa, http://repository.upenn.edu/scn_protocols/18/
[2] M. Metzler, http://repository.upenn.edu/scn_tooldata/34/

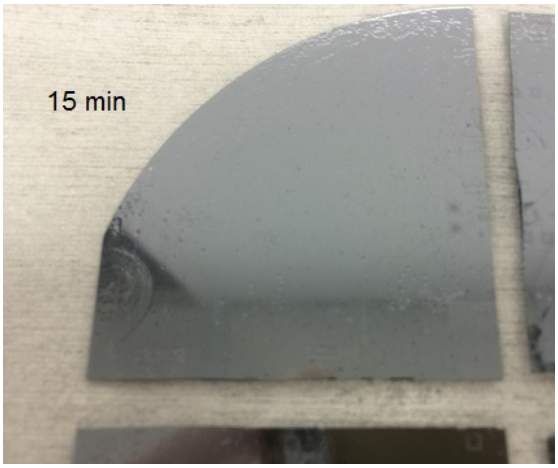


Figure 2. A photo image of a piece of the Si wafer with 316 nm thick silicon oxide film, prepared by the default recipe, on its surface. The piece was immersed into 30 wt% KOH solution at 80 °C for 15 min.

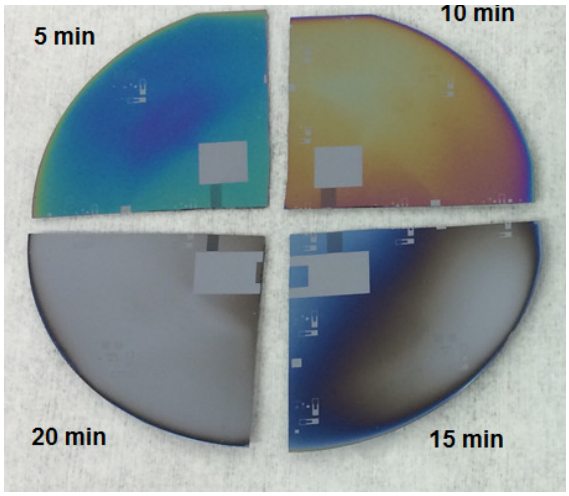


Figure 3. A photo image of four pieces of the Si wafer with 424 nm thick silicon oxide film, prepared by the Test recipe, on its surface. The four pieces were immersed into 30 wt% KOH solution at 80 °C for 5, 10, 15, and 20 min, respectively.

C. Dry etching of silicon oxide layer using Oxford 80 plus RIE

The silicon oxide film was dry-etched in three steps through the developed photoresist film, using Oxford 80 plus RIE. For the first 5 min, the sample was etched with the following condition: O_2 = 4 sccm; CHF_3 = 100 sccm; pressure = 50 mTorr; power = 150 W; T = 17.5 °C.[3] The etch rate was 37 nm/min. For the second 5 min, the sample was rotated at 180 °, and etched with the above condition to ensure the uniform etch. Then, for the last 2 min and 30 sec, the sample was etched with the following condition to avoid fluorocarbon polymer residue buildup on the exposed Si surface[4]: CF_4 = 20 sccm; pressure = 65 mTorr; power = 150 W; T = 17.5 °C.[5] After the etching, the photoresist film was removed by O_2 plasma treatment using Anatech SCE-108 Barrel Asher.

D. Alkaline protective coating on the back side of the Si wafer

ProTEK B3 Primer (Brewer Science) was spin coated on the back side of the Si wafer at 1500 rpm for 60sec, as adhesion promoter. Then, ProTEK B3 Protective Coating (Brewer Science) was spin coated on the primer at 1000 rpm at 60 sec, and was baked at 120 °C for 120 sec on a hot plate, followed by the 2nd baking at 205 °C for 60 sec.

E. KOH etching

Figure 2 shows a photo image of a Si piece with 316 nm thick silicon oxide film, prepared by the default recipe, on its surface. The piece was immersed into 30 wt% KOH aqueous solution at 80 °C for 15 min. As can be seen in figure 2, the silicon oxide film prepared by the default recipe was dissolved in the KOH solution, and the surface had uneven cavities all over.

Figure 3 shows a photo image of four Si pieces with 424 nm thick silicon oxide film, prepared by the Test recipe, on its surface. The four pieces were immersed into 30 wt% KOH aqueous solution at 80 °C for 5, 10, 15, and 20 min, respectively. The silicon oxide film was almost dissolved

[3] M. Metzler, http://repository.upenn.edu/scn_tooldata/38/
[4] It is not efficient to use this recipe throughout due to its lower selectivity.
[5] M. Metzler, http://repository.upenn.edu/scn_tooldata/36/

during the 20 min KOH etching, but the surface was still smooth. The etch rate of the silicon oxide prepared by the Test recipe is estimated to be around 21 nm/min, as shown above. On the other hand, the etch rates of PECVD silicon oxide un-annealed and annealed at 1000 °C for 60 min have been reported to be 15 and 7.8 nm/min in 30 wt% KOH solution at 80 °C, respectively,[6] showing that the annealing decreases the etch rate due to decreasing in the deficiency of amorphous silicon oxide, such as dangling bond of Si and the defects. The etch rate of ~21 nm/min suggests that PECVD silicon oxide prepared in QNF should have the large amount of the deficiency in the film.

F. Strip silicon oxide layer and alkaline protective coating

It was very difficult to remove the alkaline protective coating film using ProTEK Remover 100 (Brewer Science). However, the alkaline protective coating film could easily be peeled off during the process of stripping the silicon nitride film in 49wt% HF aqueous solution, when the silicon oxide film was removed.

3. Results

Figure 4 shows SEM images of 82.5 µm x 82.5 µm squares etched for (a) 5, (b) 10, (c) 15, and (d) 20 min, respectively. It is known that a (100)-orientated wafer forms square-based pyramids with (111) crystal planes upon KOH etching because the bonding energy between Si atoms depends on the crystal planes, resulting in the highly anisotropic etching. As can be seen in figure 4, the (111) crystal plane is revealed on the edge of the squares, and the area of (111) crystal plane increases with increasing in KOH etching time. Figure 5 shows SEM images of cross-sections of 20 µm width opened and 20 µm width masked lines etched for 10 min, indicating that the length of cross-section of (111) crystal plane etched is 16.6 µm, and the etching depth to the (100) surface is 13.3 µm. The angle between the (100) and (111) crystal planes is estimated to be 53.2 ° from the lengths. The etching depths of 82.5 µm x 82.5 µm squares etched for 5, 10, 15, and 20 min along the (100) direction can be estimated to be 6.5, 13.8, 21.6, and 31.2 µm, respectively, from figures 4 and 5. Figure 6 shows a plot of etching depth along (100) direction versus etching time. The etch rate along (100) direction is estimated to be 1.4 µm/min, which is consistent with the literature value =1.3-1.4 µm/min (for the (100) direction in 30wt% KOH solution at 80 °C).[7]

Figures 7 to 10 show SEM images of 20 µm width opened and 20 µm width masked lines and 2.4 µm width opened and 3.6 µm width masked lines, etched for 5 to 20 min. The apparent etch rates along the (100) direction for 5, 10, 15, and 20 min etching are 0.4, 1.3, 0.2, and 0.8 µm/min, respectively. As can be seen in figure 8, when the (100) crystal plane still remains on the surface, the etch rate of 1.3 µm/min is almost the same as the literature value. However, when the (100) crystal plane is etched away completely, the etch rate is much smaller than the literature vale because the etch rate along the (111) direction is extremely small (0.005 µm/min in 30 wt% KOH solution at 70 °C),[8] as shown in figures 7, 9, and 10. In other words, the etching of the (100) crystal plane dominates the etching along (100) direction until the (100) crystal plane is etched away, leaving the extremely slow etching (111) crystal plane, which forms the pyramidal structure with (111) crystal plane.

[6] K. R. Williams, J. Microelectromech. Syst. 12, 761 (2003).
[7] http://www.cleanroom.byu.edu/KOH.phtml.
[8] http://www.virginiasemi.com/pdf/siliconetchingandcleaning.pdf.

Figure 11 shows SEM images of 5 µm width right angle lines etched for (a) 5, (b) 10, (c) 15, and (d) 20 min, respectively, indicating that the etching behavior of the right angle line is different from that of the straight line. As can be seen in figure 11, although the etching of the line is very slow due to the (111) crystal plane, the etching of the exterior angle line is much faster than the etching of the (111) and (100) crystal plane, so that the (100) surface is revealed on the surface again. Furthermore, the area of (100) surface increases with increasing in the etching time, expressing that the different fast etching crystal plane, such as (110) surface (the etch rate = 2.0 µm/min[8]), is revealed on the exterior angle line.

4. Summary

KOH wet etching of (100) Si wafer was performed through a hard mask of silicon oxide, using the tools available at Quattrone Nanofabrication Facility. The 316 nm thick PECVD silicon oxide hard mask prepared by the default recipe was dissolved during 15 min KOH etching, and the resultant surface had uneven cavities all over. On the other hand, the 424 nm thick silicon oxide film prepared by the "Test" recipe worked as the hard mask against 20 min KOH etching.

The etch rate along the (100) direction was determined to be 1.4 µm/min by etching 82.5 µm x 82.5 µm squares. The straight line etching indicated that the etching of the (100) crystal plane dominates the etching along (100) direction until the (100) crystal plane is etched away, leaving the extremely slow etching (111) crystal plane, which forms the pyramidal structure with (111) crystal plane. On the other hand, the right angle line etching showed that the exterior angle line is etched faster than the (100) crystal plane, indicating that the different fast etching crystal plane, such as (110) surface, is revealed on the exterior angle line.

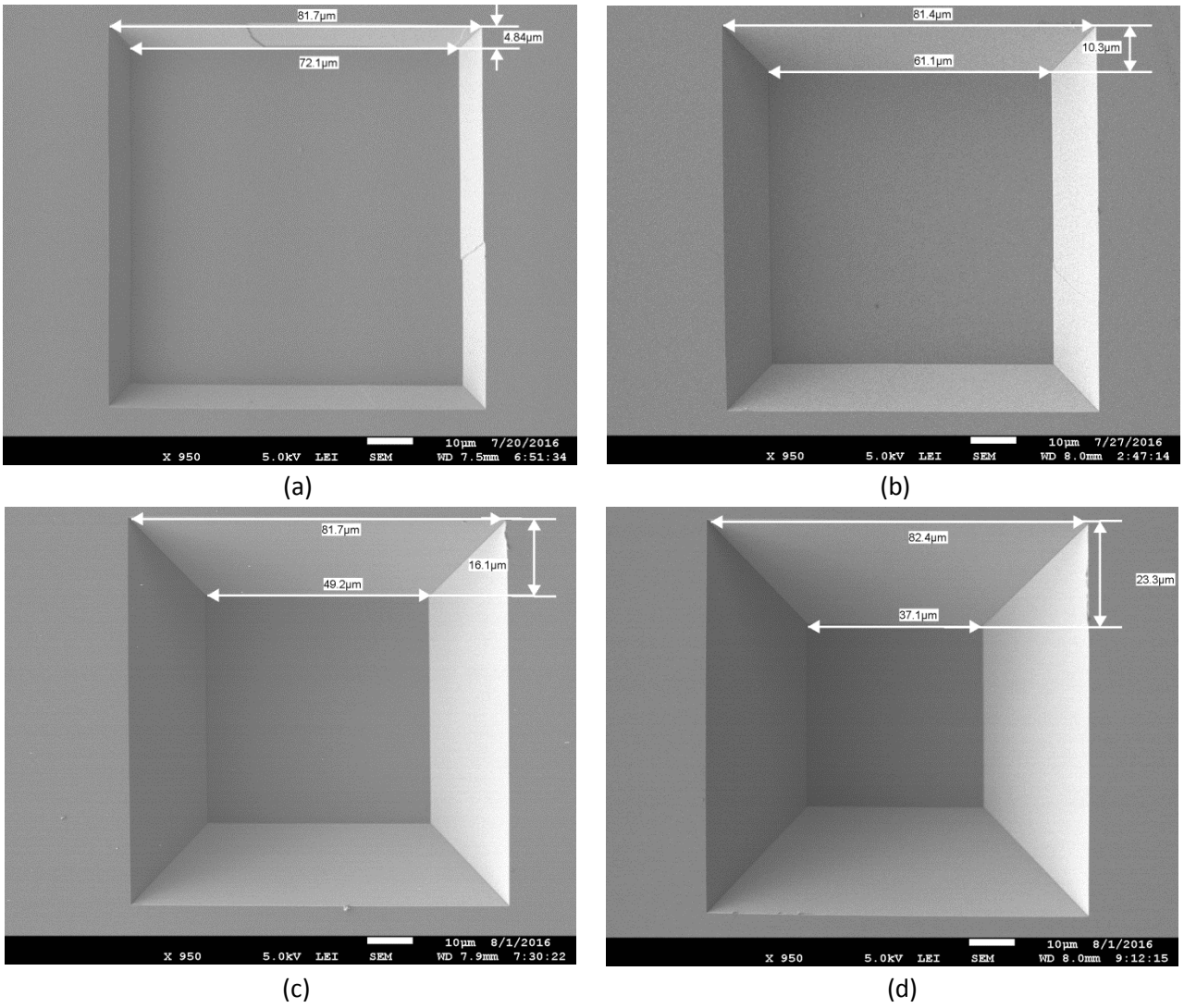
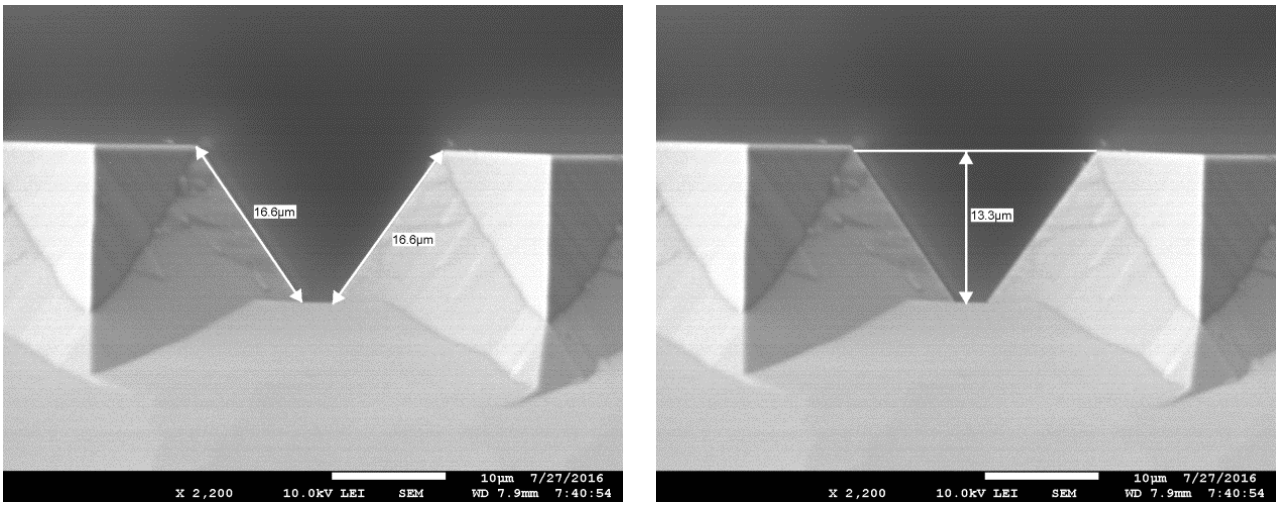


Figure 4. SEM images of 82.5 µm x 82.5 µm squares etched in 30 wt% KOH solution at 80 °C for (a) 5, (b) 10, (c) 15, and (d) 20 min, respectively.



(a) (b)

Figure 5. SEM images of cross-sections of 20 µm width opened and 20 µm width masked lines etched in 30 wt% KOH solution at 80 °C for 10 min. (a) The length of cross-section of (111) crystal plane etched is 16.6 µm; (b) the etching depth to (100) crystal plane is 13.3 µm. The angle between the (100) and (111) crystal planes is calculated to be 53.2 ° from the lengths.

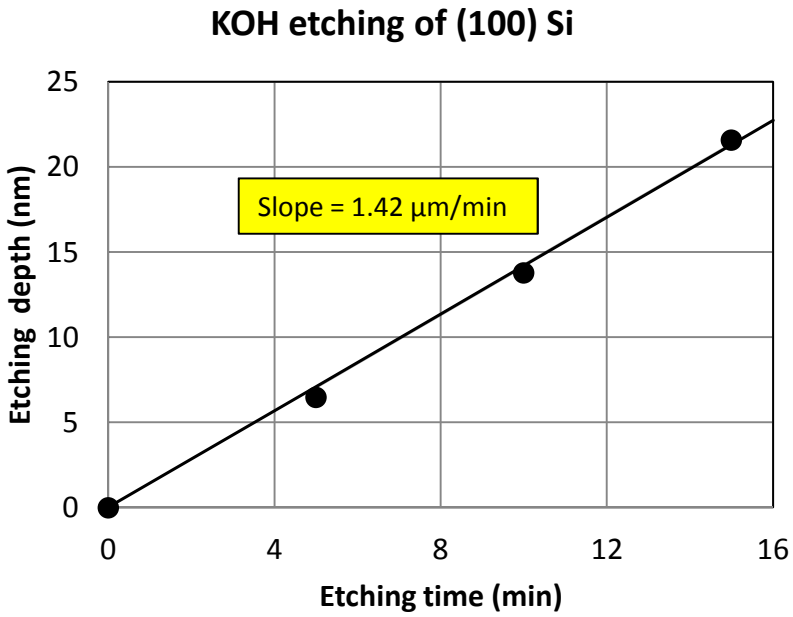
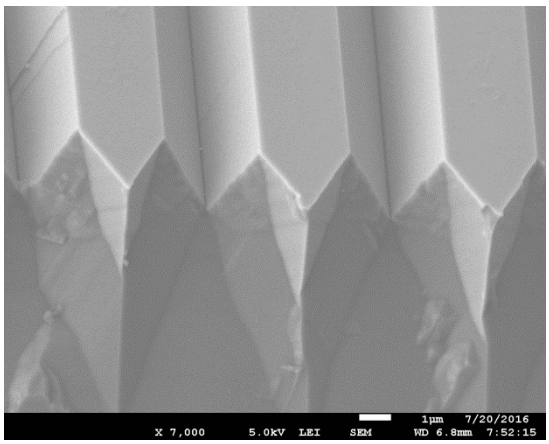
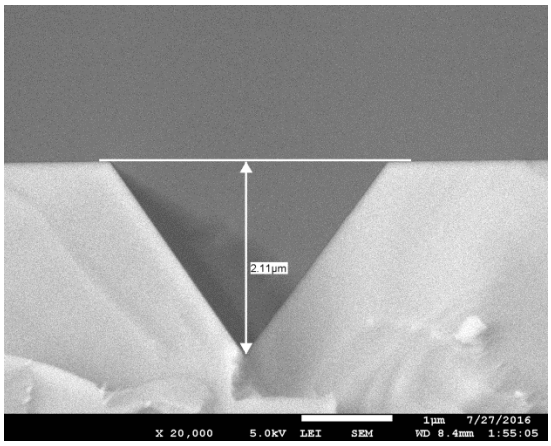


Figure 6. A plot of etching depth along (100) direction versus etching time. The 31.2 µm etching depth for 20 min KOH etching is removed because the silicon oxide hard mask should almost be dissolved in KOH solution.

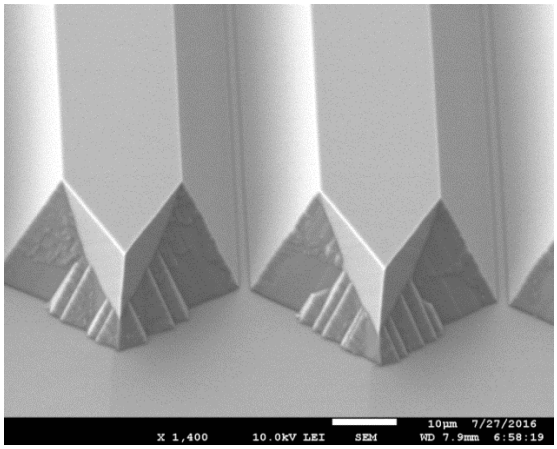


(a)

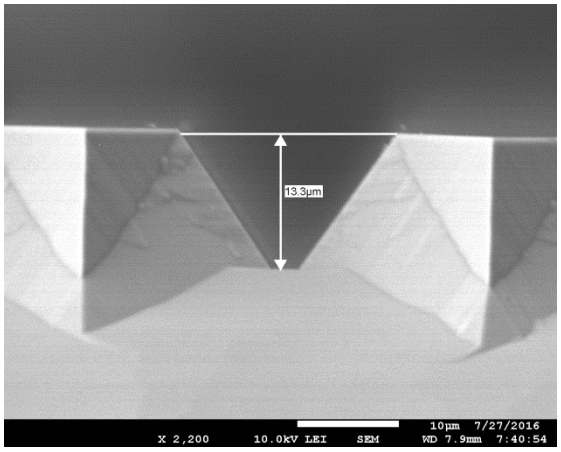


(b)

Figure 7. SEM images of 2.4 µm width opened and 3.6 µm width masked lines etched in 30 wt% KOH solution at 80 °C for 5 min. (a) tilted at 45 °; (b) cross-section.

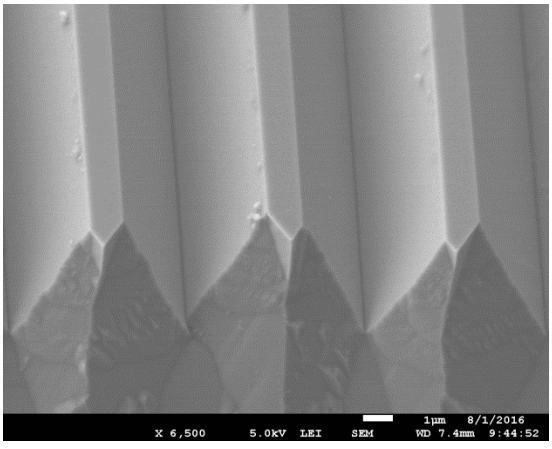


(a)

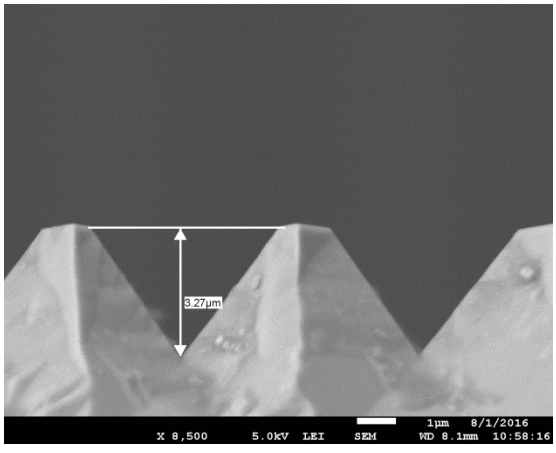


(b)

Figure 8. SEM images of 20 µm width opened and 20 µm width masked lines etched in 30 wt% KOH solution at 80 °C for 10 min. (a) tilted at 45 °; (b) cross-section.

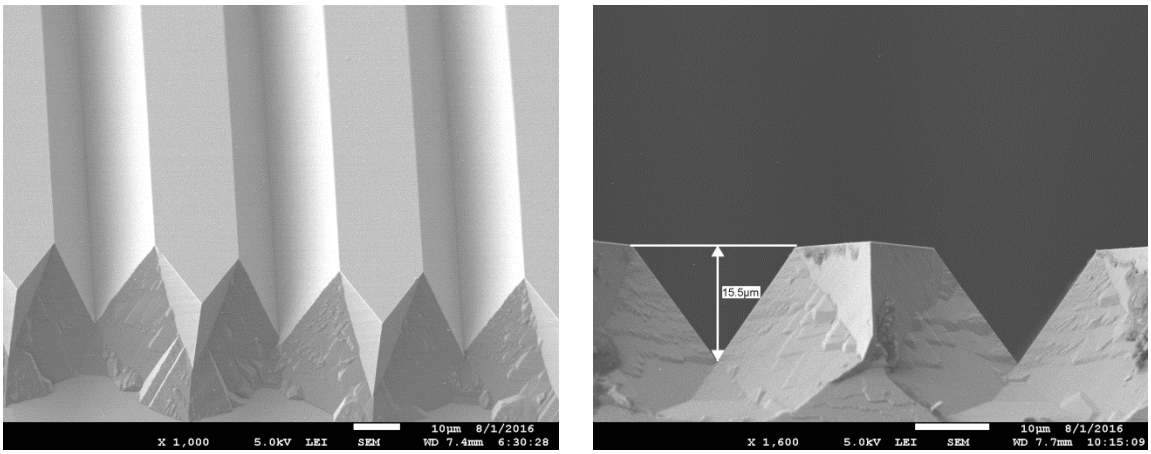


(a)



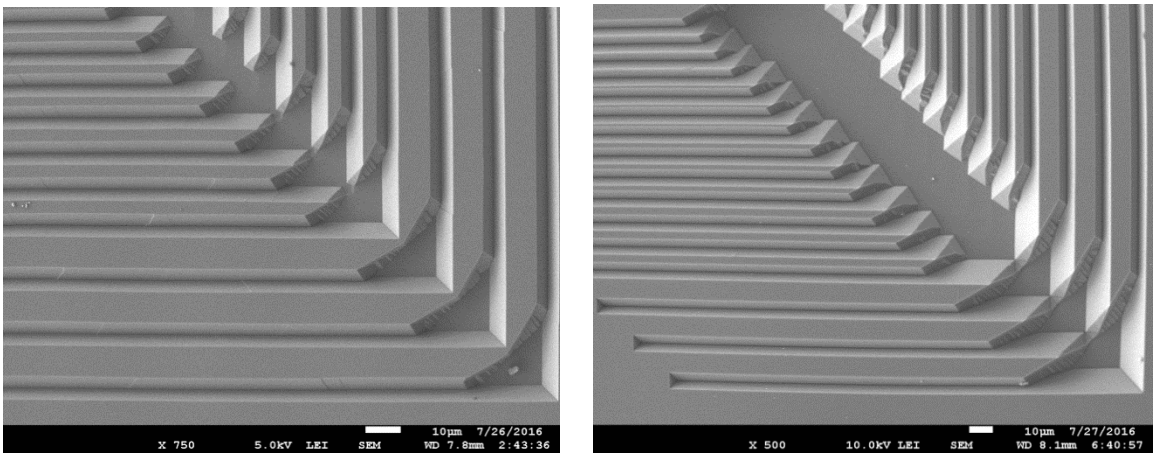
(b)

Figure 9. SEM images of 2.4 µm width opened and 3.6 µm width masked lines etched in 30 wt% KOH solution at 80 °C for 15 min. (a) tilted at 45 °; (b) cross-section.

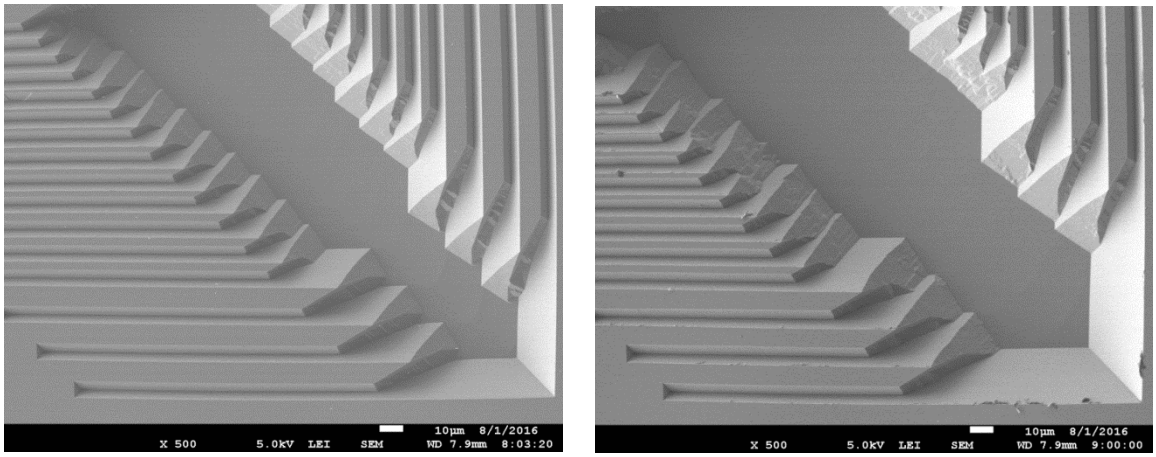


(a) (b)

Figure 10. SEM images of 20 µm width opened and 20 µm width masked lines etched in 30 wt% KOH solution at 80 °C for 20 min. (a) tilted at 45 °; (b) cross-section.



(a) (b)



(c) (d)

Figure 11. SEM images of 5 µm width right angle lines etched in 30 wt% KOH solution at 80 °C for (a) 5, (b) 10, (c) 15, and (d) 20 min, respectively.