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Formation of 45° Silicon (110) Surface Using Triton X-*n* Surfactants in Potassium Hydroxide for Infrared Applications

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Silicon (Si) micromirrors are an integral feature for many micro-optomechanical systems (MOEMS). Such mirrors are generally wet etched in alkaline solution at elevated temperature. For 90° beam steering applications, 45° slanted Si (110) plane is the prime choice fabricated with the incorporation of tensioactive surfactants. Here, Triton-Si and Triton-hydroxide (OH⁻)/H₂O interaction using varying hydrophilic chain length Triton (X-45, X-100 and X-405) were investigated. The surfactant concentration was varied from 0 to 1000 ppm in potassium hydroxide (KOH). Triton molecules were shown to adsorb preferentially on (110) than on (100) surface. Longer chain length Triton hampered OH⁻ access to Si surface resulting in slower etch rate. In contrast, contact angle measurement suggested that shorter Triton interfaced better with Si surface. Later, Si wafers etched in Triton 10 ppm – KOH were examined. The measured output for (110)_{X-45}, (110)_{X-100}, (110)_{X-405} and polished Si wafer reference ($R_q < 5 \text{ Å}$) mirrors were 0.58, 0.76, 0.72 and 1.25 mW, respectively. Subsequently, Si-SiO₂ thin film in [HLHL]²-substrate configuration was fabricated. Broadband micromirror for use in 3.0–5.5 μm spectrum range was experimentally realized with reflected efficiency of 73%. © 2018 The Electrochemical Society. [DOI: 10.1149/2.0141812jss]

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Silicon micromirrors play an important role in MEMS and MOEMS devices.¹ Generally, they are employed to guide and direct free space propagated light on chip.² To date, micromirrors are often fabricated using either dry or wet etching. The former is fabricated using deep reactive ion etching (DRIE).³ A near vertical mirror can be obtained with proper optimization. However, Bosch process usually creates large DRIE scallops due etching and passivation steps cycling. Those scallops can cause propagation loss due to surface scattering.⁴ Anisotropic wet etching, on the other hand, enables formation of smooth facets which depends on the direction of crystal facets. For example, vertical Si (100) sidewall^{5,6} as well as Si (111) which angles 54.7° with respect to Si (100) plane could be realized by etching in tetramethylammonium hydroxide (TMAH), potassium hydroxide (KOH) and ethylene diamine (EDA). Apart from wet etching, other fabrication techniques that were reported include laser ablation,⁷ molding⁸ and dicing.⁹

45° angled mirror is highly desirable for 90° optical beam steering. It allows vertically propagated light to be directed parallel to chip surface and vice versa. So far, notable techniques have been demonstrated to create such mirrors on Si. Namely, movable MEMS micromirror¹⁰ as well as fixed mirror based on Si (110) plane.¹¹ For the latter, tensioactive alcohol additives such as isopropanol (IPA) were added to KOH or TMAH with etching performed at elevated temperature between 60–90°C.¹² These additives were found to be capable of decreasing the etch rate for (110) plane. However, rapid IPA evaporation at elevated etching temperature necessitated constant alcohol replenishment to maintain saturation level. Zuber et al. proposed the use of higher boiling point diols in place of monohydric alcohols which eliminates the need to refill constantly.¹³ High concentration of alcohol (3 – 5 M) is required to obtain sufficient tensioactive effect.

Apart from alcohol additives, non-ionic amphiphathic polymers such as polyoxyethylene alkyl phenyl ether (NCW) and Isooctylphenoxy polyetoxyethanol (Triton X-100)¹⁴ have been investigated. The chief advantage of these surfactants is that they are effective even in ppm (20–100 ppm) range.¹⁵ These high boiling point (> 200°C) surfactants permit etching to be performed at higher temperature which is necessary for the fabrication of smooth (110) surface. Numerous studies had examined the effects of etching temperature, stirring speed, base and surfactant concentration on (100) and (110) planes surface quality as well as that for convex corners.^{16–20} Among published works, KOH/Triton X-100, KOH/IPA and TMAH/Triton X-100 are the most widely reported systems.²¹ So far, TMAH/Triton etchant combination is known to create the smoothest (110) plane. One main disadvantage of TMAH is that it produced a magnitude rougher (100)

surface than KOH.²² The rougher (100) surface limits further fabrication processes such as deposition of antireflective coating, etc to be performed. Moreover, the cost of TMAH is approximately ten times that of KOH.

Previously, authors had described Triton X-*n* hydrophobic head interaction with hydrophobic surfaces like Si.^{23–25} In particular, Jeon et al. investigated the adsorption/desorption of Triton X-*n* from Si (100) using Fourier-transform Infrared Spectroscopy (FTIR) and contact angle measurement.²⁶ To the best of our knowledge, Si wet etching in KOH-Triton of different hydrophilic moiety lengths for the formation of (110) plane has not been investigated. This work aims to explain how Triton X-*n* molecules interact with Si (100) and (110) during etching. Molecular interaction at the Si – Triton and Triton – OH⁻/H₂O interfaces will be discussed. In all, Triton of three different tail lengths, namely, X-45, X-100 and X-405, in KOH were examined. A mechanistic study will be presented in later part of this paper. Finally, to explore the use of fabricated Si (110) plane as on-chip fixed mirror, optical measurement conducted at 1.55 μm and 3.80 μm will be discussed.

Experimental

Si wafer preparation.—The technique to obtain 45° mirror on Si is as follows. P-doped Si (100) wafers were thermally oxidized to form 1 μm of thermal oxide. Prior to UV exposure, the mask patterns were aligned 45° with respect to the primary flat. No special alignment process was adopted. The prepared wafers were subjected to reactive ion etching (RIE) to form hard mask for subsequent wet etching.

Wet etch bath.—Triton X-*n* (*n* = 45, 100 and 405) were used without further treatment. The surfactant solutions were freshly prepared prior to etching. An amount of Triton was extracted into deionized water (DI) under vigorous stirring to facilitate complete dissolution of Triton. Subsequently, a 30% potassium hydroxide (KOH) bath was prepared using KOH pellets (Kanto) in Triton X-*n* solution in a glass vessel as shown in Fig. 1A. The experiment temperature was set to 80°C and was closely monitored using a chemical resistant temperature probe, modulating the bath temperature. 80°C was selected as Si wet etching was commonly reported to be performed between 60–100°C.²⁷ The suspended Si wafers were stirred in the etching bath at 250 rpm for 2 h. Thereafter, the wafers were thoroughly rinsed in deionized water and dried under dry N₂ stream. The experiment was repeated a few times with freshly prepared etching solution.

Si and SiO₂ thin film deposition.—Etched samples were subjected to buffered oxide etch (BOE) to remove native oxide. This was

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Figure 1. A) Experimental setup for fabrication of Si (110) micromirror on-chip and B) Optical measurement setup for quantifying light reflection efficiency at 1.55 and 3.80 μm using Si (110) surfaces fabricated in Triton X-*n*.

followed by the deposition of 770 nm of SiO_2 thin film by plasma enhanced chemical vapor deposition (PECVD, ASM P5000). Subsequently, 320 nm of Si was deposited via sputtering (Denton).

Characterization.—Atomic force microscopy (AFM) measurement was performed on etched Si wafers to elucidate the root mean square surface roughness (R_q) of both Si (100) and (110) surfaces. Static sessile contact angle measurement using water and Triton X-*n* on cleaned Si (100) and (110) wafers. As shown in Fig. 1B, reflectivity measurement of (110) surface was conducted using 1.55 and 3.80 μm laser as light sources. The sample was then mounted on a rotation stage and the single mode fiber (SMF) was placed as close to the (110) surface as possible. Objective lenses, 20X DIN (Edmund Optics) and LFO-5-6 with working distance of 5.3 mm (Innpho) were used for near-infrared (NIR) and mid-infrared (MIR) reflectivity measurement, respectively. Throughout the experiment, all optical components except the sample were fixed in space. Free space light propagation was terminated at the single integrating sphere InGaAs photodetector for NIR measurement and single thermopile for MIR demonstration.

Results and Discussion

Various shapes had been etched in KOH-Triton previously.²⁰ In this case, millimetre length rectangular mesa were etched to reveal (110) plane. Other shapes were not considered in this paper as the main focus was to fabricate reasonably long mirrors for on-chip applications.

Etch rates in three different Triton chain length solutions – KOH solutions are shown in Fig. 2. Pillars etched in the absence of Triton served as reference. In the absence of Triton, (110) plane was rapidly etched away as shown in Fig. 3A with vertical (100) sidewall. Similar to previous report, an etching rate of 75 $\mu\text{m}/\text{h}$ was obtained.²⁸ Further etching of such vertical mesa resulted in tiny stumps on the Si surface. This evidenced that (110) was rapidly etched away in the absence of Triton. When Triton X-*n* was introduced, even at low concentration, (110) plane was preserved.²⁹

Etch depth is influenced by Triton concentration and chain length. Pronounced difference in etch rate could be observed in all cases for Triton X-*n* between 1 and 100 ppm. According to Fig. 2, the etch rate for Triton X-100 – KOH was found to be approximately 55 $\mu\text{m}/\text{h}$. (100) etch depth is determined by Triton chain length such that longer Triton effects slower etch rate per unit time. In this case, the etch depth trend could be arranged in the following order: X-45 > X-100 > X-405. When Triton X-*n* concentration exceeds 10 ppm in all three scenarios, there was a significant decrease in etch depth. At higher concentration, when Triton X-*n* concentration exceeded 1000 ppm, etch rate declined sharply to approximately 10 $\mu\text{m}/\text{h}$.

It could be established that the presence of Triton X-*n* has a significant impact on retaining both (100) and (110) Si planes. Therefore, for effective use of such bulk micromachined mirrors, it is necessary to

balance the concentration of X-*n* in relation to the intended outcomes which are decided by the (100) and (110) surface roughness and (110) vertical mirror height.

Representative SEM images of the fabricated mesa are as shown in Fig. 3. In Fig. 3A, it could be established that in the absence of Triton, the (110) plane was quickly etched away. In Figs. 3B, 3C and 3D which correspond to Triton X-45, X-100 and X-405 1 ppm in KOH, respectively, at low Triton X-*n* concentration, pronounced “caterpillar legs” could be observed. It was partially alleviated when longer Triton at similar surfactant concentration was used. At higher Triton X-*n* concentration, as seen in Figs. 3E, 3F and 3G, the “caterpillar legs” effect was no longer visible for X-100 and X-405; however, the phenomenon persisted for X-45. Caterpillar legs formation could be a result of low concentration short chain length Triton X presents in etching bath that partially revealed higher order planes. It could be attributed to the presence of adsorbed Triton X-*n* that interacted with those planes while etching was in progress. The authors suggest that a separate study to be performed in subsequent work to confirm the postulation.

SEM images confirmed that (100) surface irregularities which peaked at Triton X-*n* 10 ppm. As shown in Figs. 3H–3N, (100) surface was visualized to be smoother at higher Triton X-*n* concentration. The results obtained were similar to those reported by Tanaka et al.³⁰ and Rola et al. which investigated the effects of Triton X-100 from 0.01 to

Figure 2. Si (100) etch rate with respect to Triton X-*n* concentration. Fig. 2 (insert) shows the molecular structure of Triton X.

Figure 3. SEM images of Si etched in various Triton X-*n* concentration.

10 ppm.¹⁸ This could be explained by the fact that at low Triton X-*n* concentration, etching behavior would be similar to that of KOH-only etchant which formed smooth (100) plane.

It is imperative to fabricate Si (110) mirrors with sufficient vertical height (at least 70 μm) for optical measurement using SMF. Since 10 ppm Triton X-*n* in KOH has been proven to be advantageous in terms of height etched per time, pillars etched in the 10 ppm Triton X-*n* - KOH solution would be evaluated in subsequent sections.

Surface roughness.—AFM was performed on samples prepared in 10 ppm Triton X-*n* - KOH etching bath with results shown in Fig. 4. It was observed that R_q values for (100) worsen at longer Triton chain length. On the contrary, Si (110) R_q improved with longer Triton chain length from 1.31 μm X-45 - KOH to 0.186 μm in X-405 - KOH. The surface texture on (110) is critical for the fabrication of micromirrors. Rough surface attenuates the amount of light reflected by scattering loss and would affect output efficiency. It is quintessential to minimize surface irregularities by establishing sound fabrication tech-

nique in addition to the use of longer Triton such as X-100 and X-405 which were observed to produce smoother (110) plane as illustrated in Fig. 3.

It was observed that surface roughness differs between that of (100) and (110) planes with the latter being highly striated while the former granulated in appearance.²¹ The striated texture could likely to be due to the exposure of (551) plane as reported by Sato et al.³¹ Surface roughness measured was within the same order of magnitude with that reported by Sato et al. (110) was more striated when X-45 was used which could be observed in Fig. 3. Striation was not observed when longer Triton (X-100 and X-405) or larger concentration of X-45 were used.

Si - triton interface.—Contact angle experiment has to be conducted as soon as the BOE process is completed in order to preserve the cleaned Si surface to be oxide free as much as possible. However, in reality, it is nearly impossible to obtain oxide-free silicon surface due to the rapid formation of native oxide in ambient environment.³² According to Morita et al., a rapid increase in native oxide thickness

	R_q surface roughness (μm)		
Si facets	X-45	X-100	X-405
100	0.275	0.444	0.88
110	1.31	0.221	0.186

Figure 4. AFM measurement on (100) and (110) surfaces etch in 10 ppm Triton X-*n*.

begins at 100 min post BOE cleaning. To reduce data discrepancies due to native oxide formation, measurements were completed within 60 min post Si surface preparation. DI water was dispensed directly onto Si surface as a controlled study. The measured angle for water on Si surface was approximately 75° . The result obtained agree with published data.³³

It was postulated that Triton X-*n*, when added to KOH bath, would interact with Si during the etching process. Previously, Pal et al. reported the effects of Triton X-100 adsorption on Si in TMAH etching solution by spectroscopic analysis.³⁴ It was described that more Triton X-100 preferentially adsorbed on (110) than (100). To complement

previous works, which necessitated complicated experimental setup, this paper presents contact angle measurement by static sessile drop method conducted on both (100) and (110) wafers which is sensitive to change in surface properties. The test commenced with BOE cleaning step to remove native oxide layer on Si. A $3\mu\text{l}$ drop of freshly prepared Triton X-*n* ranging from 1 to 1000 ppm was dispensed on the Si surface and solid-liquid angle was measured. The results of the contact angle measurement are shown in Fig. 5.

Generally, an increase in Triton X-*n* concentration entails greater Si wetting with the decrease in contact angle.²¹ The trend is common for both (100) and (110). (110) surface experienced greater wetting

Figure 5. Sessile drop contact angle measurement using Triton X-*n* solution on A) (100) and B) (110) wafers.

than (100) due to a higher packing density of Si for all Triton lengths. Moreover, Triton preferentially adsorbed on (110) given the difference in contact angle for (100) and (110) at higher Triton X-*n* concentration for all Triton X-*n* analyzed. Comparing the contact angles between different Triton X-*n*, longer Triton X-*n* exhibited larger measured contact angle. One plausible explanation is that the interaction with shorter Triton is preferred over longer Triton due to stronger repulsion between molecules along the tail with the latter which comprises repeating ether group. The ether groups confer hydrophilicity effect which will be explained in the subsequent section. Therefore, shorter Triton molecule effects better Triton-Si interaction in which the Triton molecules experience less repulsion.

Triton - liquid interface.—OH[−]/H₂O – Triton interface was studied to understand the effects of varying chain lengths on etching characteristics. Primarily, OH[−] ions are responsible for the chemical reaction with Si as Triton X-*n* was previously ascertained to be non-participating.³⁵ To understand this process, the above mentioned sessile drop method was modified slightly and authors assumed that Triton molecules present would assemble instantaneously on Si sur-

face before etching starts. To mimic the scenario, Triton X-*n* was spin dropped onto BOE cleaned (100) and (110) Si wafers. The wafers were then dried at 80°C for 5 min. Thereafter, 3 μl of DI was dispensed directly onto the Si surface stained with Triton. Although KOH in water system was employed during the actual etching process, its use during contact angle measurement was avoided due to safety reason as well as the fact that KOH etching would induce surface roughness on both Si (100) and (110) surfaces. The change in Si surface condition over time would render static contact angle measurement impossible especially for high Triton concentration.^{36,37} Moreover, previous work suggested that the presence of KOH would not change the contact angle significantly.³⁸ As shown in Fig. 6, to simplify the study, a water-only system was represented instead.

One can observe that longer Triton has higher wettability than the shorter counterpart. In this instance, wettability (highest to lowest) is as listed in the following sequence: X-405 > X-100 > X-45. In part, this is due to the presence of repeating ether group found along the hydrophilic tail of the Triton molecule as explained in the earlier section. Ether groups form hydrogen bonds with the OH[−] ions and water molecules present in the solution.³⁹ By this argument, the tail

Figure 6. Sessile drop contact angle measurement using H₂O on Triton X-*n* treated A) (100) and B) (110) surfaces.

Figure 7. Schematic diagram showing the adsorption of Triton molecules on Si surfaces and its interaction with reactants as well as products evolution. The impression depicts the consequence of Triton polar tail trapping hydroxyl ion and water which affects the forward reaction.

likely presents a shielding effect which prevents rapid etching of Si surface by OH^- ions and H_2O molecules when those reactants are “trapped” along the Triton tail. This aids in explaining slower etching rate with longer Triton.

Molecular mechanistic insight.—The schematic diagram in Fig. 7 shows the overall wet etching reaction involving Triton X-*n*. Ether bonds ($-\text{C}-\text{O}-\text{C}-$) located along the chain possibly contribute to intermolecular hydrogen bonding with water and hydroxyl ions by being hydrogen bond acceptors.⁴⁰ This explains higher solubility of X-405 than X-100 and X-45 in aqueous solution as shown in Fig. 8. While X-100 and X-405 were fully solubilized in water, X-45 formed a turbid suspension. Ether bonds possibly “trapped” etching ions, preventing access of hydroxyl ions to effect Si etching as well as removal of by-products such as $\text{SiO}_2(\text{OH})_2^{2-}$, etc. By this argument, the presence of Triton inhibits forward chemical reaction as described:⁴¹

On the other hand, it is apparent that Si (100) and (110) facets, due to different atomic packing density, surface energy, etc, present distinct

interactive environment with Triton surfactant. Si (100) and (110) have unique preferential sites which attract the Triton hydrophobic moiety with its hydrophilic moiety pointed toward the aqueous environment. Previously, Tang et al. reported a larger volume of Triton X-100 molecules were selectively adsorbed on (110) than (100) by measuring the thickness of Triton layer using ellipsometry.³⁵ Since Si (110) plane is more closely packed than (100), more Triton molecules could possibly accumulate on (110) than (100) surface. Concerning Triton of different lengths, shorter molecules experience less steric repulsion than longer molecules which allows more X-45 than X-100 and X-405 molecules to accumulate on both (100) and (110). The phenomenon was observed by better surface wetting depicted in Fig. 6B. Although X-45 confers better Si surface wetting,²⁶ it is not as effective as longer Triton in preventing Si removal resulting in faster etch and the formation of rougher (110) plane as shown in Figs. 3 and 4. In the same light, longer Triton tail imposed a more diffusion controlled access of reactants and by-products.³⁴ Thus, it promoted a smoother etched (110) surface.

Since (100) surface is less densely packed, it is highly possible that there are fewer adsorption sites than (110). (100) surface with and without adsorbed Triton molecules experienced different etch characteristics as compared to (110). Unlike (110), (100) results in a granulated surface topography. Plausibly, Triton adsorbed on active sites where it resulted in uneven etch rates on the (100) surface. Unprotected Si were etched away faster than Si with adsorbed Triton molecules. This caused the appearance of granules. The authors suggest more studies to be conducted in the future to affirm the postulation. Surface adsorbed X-45 molecules somewhat present less effective reactants “trapping” as explained earlier which results in smoother (100) etched surface as compared to X-100 and X-405. At higher surfactant concentration, when more molecules populate Si surface, such problems were resolved, affording smoother (100) and (110) surfaces. Although longer Triton affords better (110) surface quality albeit slower etch rates, longer Triton polymer enables easier polymer removal post etching due to its better interaction with the aqueous environment.

Optical measurement.—Samples prepared in 10 ppm of Triton X-*n* – KOH bath were subjected to optical testing. Initial verification

Figure 8. Prepared Triton X-*n* in H_2O with increase solubility at higher X-*n*.

Figure 9. Power output for 1.55 μm beam reflected from (110) mirrors created by 10 ppm Triton X-*n* in KOH. The figure also shows power loss due to other test structures.

was performed using 1.55 μm laser source coupled into SMF-28 fiber. The complete measurement setup is shown in Fig. 1B. The sample was then mounted on a rotation stage and the SMF was placed as close to the (110) surface as possible. Throughout the experiment, all optical components except the sample were fixed in space. The sample holding stage enables one degree of freedom through rotation. Finally, free space light propagation was terminated at the single pixel uncooled InGaAs photodetector with an attached integrated sphere.

Results obtained from the reflectivity measurement are shown in Fig. 9. The laser was modulated to provide a power output of 5.43 mW. It was realized that 1.75 mW of power was attenuated by the objective lens. A polished Si wafer ($< 5 \text{ \AA}$) was mounted in between the fiber tip and lens to align the entire optical setup. The lens was adjusted to produce a sharp and bright circular profile visualized using a CCD detector system. The polished Si substrate reflected a measured power output of 1.25 mW. Si reflectivity is approximately 40% at 1.55 μm . The results proved that longer Triton assured smoother (110) surface. Results garnered from this reflectivity study coincided with surface topography shown in Fig. 3 and Fig. 4. On the contrary, rougher Si (110) from X-45 – KOH produced higher surface scattering loss which reduced the amount of light reflected at (110) plane.

MIR free space optical interconnect.—Given the favorable reflection measured with samples fabricated using X-100 10ppm surfactant, prepared samples proceeded with deposition of dielectric Bragg reflector (DBR). Alternating layers of high refractive index Si (H) and low refractive index SiO_2 (L) were deposited via RF sputtering and PECVD respectively to create a [HLHL]-substrate configuration. Lumerical FDTD simulated and FTIR measured DBR transmission profiles were shown in Fig. 10.

Simulated [HLHL] reflectivity data revealed a mirror working range from 3–7 μm . However, fabricated thin film filter had a working range of 3.0–5.5 μm . The slight discrepancy between simulated and actual data could be attributed to the presence of amorphous Si instead of crystalline Si which refractive index differed from that of crystalline Si, as well as slight deviation from desired Si and SiO_2 thicknesses. Thereafter, the samples were subjected to MIR reflectivity testing using a 3.8 μm laser. The results obtained are shown in Fig. 11. The power output value measured by a thermopile detector registered approximately 73% of the power from the sample after accounting for propagation loss from fiber and lens. The as-is fabricated mirror had surface roughness well within $\lambda/10$ optical flatness limit.

Figure 10. Transmission profiles of FDTD simulated and experimentally measured $[\text{Si-SiO}_2]^2\text{-Si}$ thin film.

Conclusions

Triton X-*n* with varying molecular length were investigated as surfactants for Si etching in KOH at elevated temperature. Si (100) surface roughness (smoothest to roughest) in the following order was obtained: X-45 < X-100 < X-405. On the contrary, (110) surface roughness was measured to be roughest for X-45 and smoothest for X-405. Longer Triton was hypothesized to shield Si from rapid etching more effectively than shorter Triton. It was shown that X-45 wetted (100) and (110) more than X-100 and X-405 given its higher overall hydrophobicity. On the contrary, less wetting was observed for Si-X-45 surface than Si- X-100/X-405 using H_2O due to the presence of more ether repeating groups along the hydrophilic moiety. Regardless of Triton X-*n* surfactant, (110) wetted more than (100) especially at high Triton X-*n* concentration. Contact angle measurement revealed the subtle effect of surface atomic density to Triton X-*n*'s hydrophobic head adsorption and reactants “trapping” sites along the hydrophilic tail. With 10 ppm Triton in KOH, shorter Triton resulted in highly striated and rough (110) but smooth (100) planes. (110) surface roughness improved with longer Triton but (100) surface worsened. Reflectivity measurement using 1.55 μm laser demonstrated high reflectiv-

Figure 11. Power output for 3.8 μm beam reflected from (110) mirror created by 10 ppm Triton X-100 in KOH with $[\text{Si-SiO}_2]^2$. The figure also shows power loss due to other test structures.

ity for Si (110) formed using longer Triton X-*n*. Finally, this work realized highly reflective and broadband MIR mirror by depositing Si-SiO₂ [HLHL-substrate] layers as DBR and up to 73% of power was recorded. Although TMAH/Triton system is proven to be unrivaled for the production of smooth (110) mirror today, exploration of surfactants of varying chain lengths in cost effective KOH could potentially bridge that gap in the future.

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