Pyramid textured Si{100} surface with low reflectivity in CMOS compatible solution

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Wet anisotropic etching of Si{100} results in micro-pyramids/hillocks bounded by four {111} planes. These kinds of geometrical structures are needed to reduce the front surface reflectance from silicon by taking multiple internal reflections of the incident ray and thus increase the efficiency of the solar cells. In this work, surface texturing of Si{100} is performed at different temperatures in a very low concentration CMOS compatible tetramethylammonium hydroxide (TMAH) without using any additive and agitation. The present research is aimed to significantly reduce the etching time in an extremely low concentration TMAH to texture the silicon surface for obtaining the lowest possible reflectance. Etching temperature is varied from 80 to 95°C with a step of 5°C. For each temperature, the etching time is also varied from 5 to 40 min with an interval of 5 min, whereas etching concentration is made a fix to 0.5 wt%. Surface morphology with dense and uniform pyramidal structures is achieved on the silicon samples etched at 85°C for 25 min. These samples provide the average solar weighted reflectance (R_{sw}) of around 10% and the lowest reflectance of 6.7% at 790 nm.

1. Introduction: Conventional sources of energy, e.g. coal, petroleum, natural gas etc. are limited in quantity and are being used for a long time. Moreover, these sources of energy are responsible for degrading the environment and human health in many ways, such as global warming and climate change. Renewable energy sources, particularly solar and wind energy, are secure, sustainable, and environment friendly. Among all renewable energy resources, solar energy is the most abundant, clean, and inexhaustible [1, 2]. A photovoltaic or solar cell can be used to convert solar energy into electricity. It offers several benefits compared to conventional energy sources. It has a noise-free operation with minimal maintenance requirements and a modular electricity solution [1]. Silicon-based solar cells have dominated the photovoltaic market because of several benefits such as higher efficiency, excellent stability, non-toxicity, and outstanding reliability in outdoor conditions [1–3]. There are several ways to improve the efficiency of solar cells. One way is to abate the reflection of light [1–5]. Antireflective coatings and surface texturing are the two most common ways used to reduce the reflection of light on the front surface of the cell [2]. In surface texturing, the Si surface is intentionally made rough to increase the optical path length by multiple internal reflections, which results in more photon generation and thus increase the efficiency [2]. Surface texturing is usually done by wet etching, dry etching, and lithography [3–23]. For generating anti-reflective structures on Si surface, many techniques based on lithographies such as photolithography, electron-beam lithography, and nanoimprint lithography have been used. However, due to the requirement of expensive equipment, time-consuming, and complicated procedures, these techniques are restricted in terms of practical applications [9, 10]. Among all, wet etching is a low cost, high throughput, and easy handling procedure, which brings a good balance between the efficiency and cost of the solar cells [2, 11, 24]. Potassium hydroxide and tetramethylammonium hydroxide (TMAH) are the two most extensively used alkaline solutions for surface texturing of silicon [5, 8, 12–14, 24]. Out of these two etchants, TMAH is popular because it provides better etch selectivity between silicon and silicon dioxide and offers compatibility with the CMOS process [3, 8, 12, 14–16, 24]. In wet etching, final etched surface morphology is defined by many parameters, i.e. etchant type, etching time, etchant concentration, and etching temperature [15, 16, 24]. Surface texturing using low concentration TMAH (<5%) results in high surface roughness, where high temperature provides a high etch rate and reduces etching time [18, 19]. These are a few remarkable observations that can be applied in solar cell industries to reduce the cost of surface texturing to improve light absorption or to minimise the reflection. TMAH over a wide range of concentrations (1–25 wt%) without and with different kinds of additives has been studied [4, 5, 20]. Moreover, other effects such as mechanical agitation on etched surface morphology have also been investigated [8]. In previous studies, 2% TMAH is reported as an optimal concentration for lower reflectance [3, 4, 8, 15, 21].

In this work, the effect of etching temperature on the surface texturing of Si{100} in a very low concentration CMOS compatible TMAH (0.5 wt%) solution is investigated in detail. The present study is aimed to identify the etching process with low chemical usage at the lowest possible etching duration to texture Si{100} surface to achieve low reflectance. The use of low concentration TMAH offers two major advantages. First, it minimises chemical waste. The second benefit is the ability to reduce the production cost. The achievement of low reflectance in very less etching time increases the industrial throughput. Therefore, the present work offers the large-scale industrial viability of the process.

2. Experimental: One side polished Czochralski grown {100} oriented single-crystalline p-type silicon wafers with 1–10 Ω.cm resistivity are used. Experiments are performed in a constant temperature oil bath system. The wet etching system contains a temperature controller, Teflon bowl, reflux condenser, and a thermometer. Fig. 1 presents the optical photograph of the wet etching system.

The process starts with a dicing of 3-inch diameter silicon wafer into four equal parts and each part (i.e. a one-quarter of 3-inch diameter wafer) is referred to as a sample. Fig. 2 schematically represents the steps followed during surface texturing and characterisation. The first Si sample is dipped in a piranha bath (H_2SO_4:H_2O_2::1:1) for 10 min to clean its surface. This step is followed by thorough rinsing in running de-ionised (DI) water. Chemically grown oxide layer in piranha bath is removed by dipping in 1% hydrogen fluoride solution for 1 min followed by a rinsing in DI water. For surface texturing, 0.5 wt% TMAH is used as an

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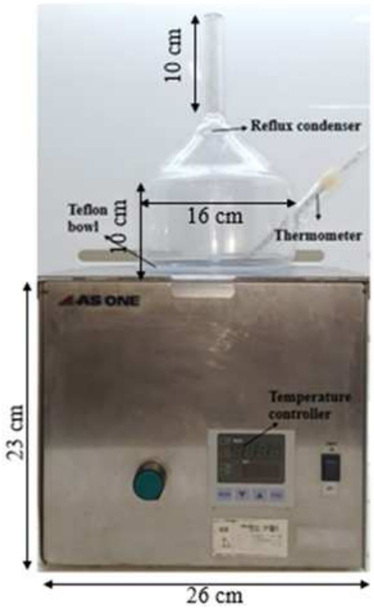

Fig. 1 *Optical photograph of a constant temperature oil bath system*

etchant and etching temperature is varied from 80 to 95°C in the step of 5°C. A 0.5 wt% TMAH is prepared by diluting 25 wt% TMAH (Alfa Aesar; purity 99.9999%) in DI water. Wet etching is performed in 1 L of TMAH solution in a cylindrical Teflon bowl (etching bath), which is placed inside a constant temperature oil bath (±1°C). A cone-shaped reflux condenser made of thick glass with a narrow opening at the top is used to avoid changes in the etchant concentration during etching as shown in Fig. 1. The temperature of the etchant is monitored in the proximity of the samples during the experiment by using a thermometer (±0.1°C). A magnetic stirrer is used to equalise the temperature in the oil bath. The etching experiment is performed for 40 min, where samples are taken out after every 5 min to investigate the effect of etching time. Etched samples are rinsed with DI water and dried using an air blower. Now the samples are ready for characterisation. Surface morphology is analysed using a SEM (Zeiss), while the root mean square (RMS) surface roughness (Sq) and surface area (SA) of etched Si samples are measured using a 3D laser scanning microscope (3D-LSM). Total reflectance (%R) is measured using an ultraviolet–visible (UV–Vis) spectrophotometer (UV 3092; Make: LabIndia Analytical Pvt. Ltd) in integrating sphere mode.

3. Results and discussion: Firstly, the etched Si samples are characterised using 3D-LSM to measure the surface roughness. Fig. 3 shows the surface roughness variation of the Si samples etched at different etching temperatures (80, 85, 90, and 95°C) for 5, 10, 15, 20, 25, 30, 35, and 40 min. RMS roughness (Sq) values are measured at ten different points on each sample to calculate the average and standard deviation (SD) values. The resolution of 3D-LSM in the *XY*-plane and *z*-direction (i.e. height) is 0.12 and 0.01 µm, respectively, while measurement accuracies in the *XY*-plane and *z*-direction are within ± 2% of the measurement value and $0.2 + L/100$ (or less), respectively, where L is the measured length in µm. Experiments are consecutively repeated at least three times for each etching temperature and time. Subsequently, the combined SD and mean of the measurements are calculated. As we can observe in Fig. 3 that the Sq values are influenced by etching time and etching temperature. The highest Sq values of around 1 µm are obtained for the sample etched for 40 min at 95°C. The error bars shown by red lines signify the SD of the mean value of Sq.

As one can observe here that the SD in Sq values (shown by red error bars) are higher for 90 and 95°C in comparison with that for 80 and 85°C etching temperatures. The higher SD in mean values indicates the variation in the sizes of pyramidal structures on the same sample at different places and epitomises that the etched surface morphology is non-uniform.

The absorption (or reflectance) of the incident light is one of the most important parameters governing the efficiency of the solar cell. As stated previously, surface texturing is a simpler and cost-effective technique to minimise the reflectance loss from the silicon surface. In surface texturing, reflectance depends on many parameters such as surface roughness, uniformity, density, and the size of pyramidal structures. In this work, the surface texturing is intended to reduce the reflectance or to enhance the absorption of light. After analysing surface roughness, optical properties are measured using a UV–Vis spectrophotometer. A rough surface reflects light in random directions, known as diffuse reflection. Hence minimising the total reflectance (specular and diffuse) is a requirement for solar cells. Therefore, total reflectance is measured to characterise the reflection from the surface using integrating sphere mode. In integrated sphere mode, light reflected in all directions is collected and measured by a detector. Fig. 4 presents the reflectance (%R) spectra of the samples etched at various temperatures (80–95°C) for different etching times (5–40 min) and Fig. 5 shows the corresponding R_{sw} values. As one can observe in Fig. 4 (at 80°C), the reflectance value is decreasing with an increase in etching time from 5 to 40 min, and the lowest R_{sw} of 14.7% (Fig. 5) is found at 40 min of etching time. Moreover, it can be noticed from Fig. 5 that the reflectance decreases with the increase of etching temperature from 80 to 85°C and the lowest reflectance of 10% is obtained on the sample etched for 25 min at 85°C. However, a further increase in etching temperature from 85 to 90°C and then to 95°C results in an increase in reflectance values where the lowest reflectance of 18.5 and 21% (Fig. 5) is found at 20 and 15 min of etching times, respectively. Hence, the lowest reflectance of around 10% is achieved on the sample etched

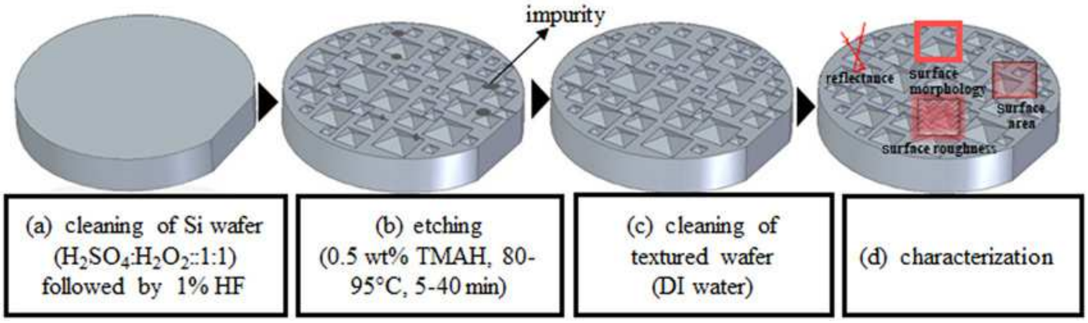

Fig. 2 *Steps involved in experimental and characterisation*

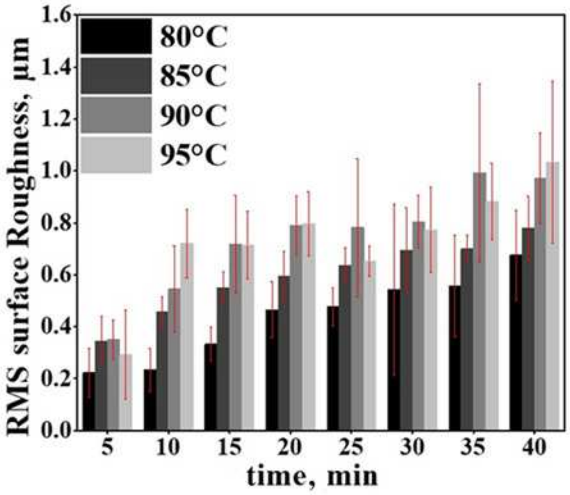

Fig. 3 *RMS surface roughness variation with respect to etching time (5–40 min) for samples etched in 0.5 wt% TMAH at different etching temperatures (80–95°C)*

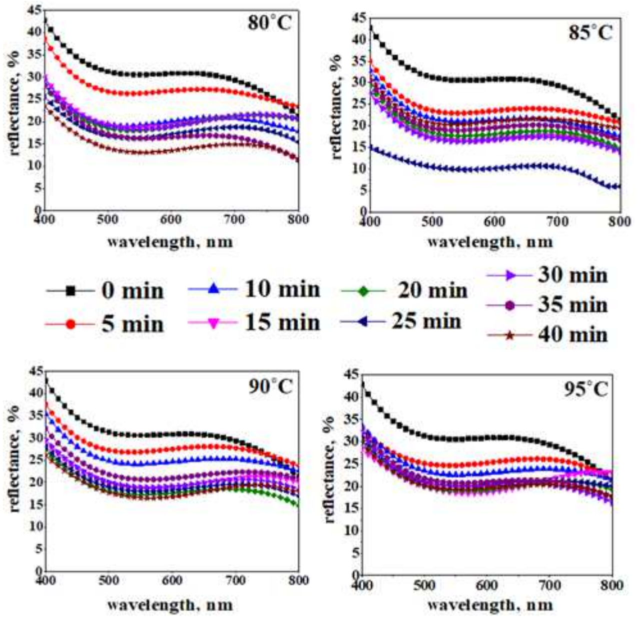

Fig. 4 *Reflectance (%R) spectra for samples etched in 0.5 wt% TMAH at different etching temperatures (80, 85, 90, and 95°C) for 5–40 min of etching time*

at 85°C for 25 min. This may be because of favourable surface morphology in terms of hillocks' uniformity and density. Etched samples are further characterised by SEM to investigate the uniformity, and density of the micro-pyramidal structures qualitatively to correlate with the optical values. The samples etched at 85°C show the lowest reflectance value. Therefore, the surface morphology study is primarily performed on these samples and the results are compared with the samples etched at other temperatures. Fig. 6 presents SEM micrographs of the samples etched at 85°C for different etching times 5, 10, 15, 20, 25, 30, 35, and 40 min.

The wet etching process using alkaline etchant generally involves two steps, i.e. oxidation and reduction. In the oxidation, adsorption of hydroxide ions occurs on the Si surface where Si–H bonds are broken and new Si–OH bonds are formed. Owing to more electronegativity of oxygen in hydroxide, Si–Si back bonds are weakened.

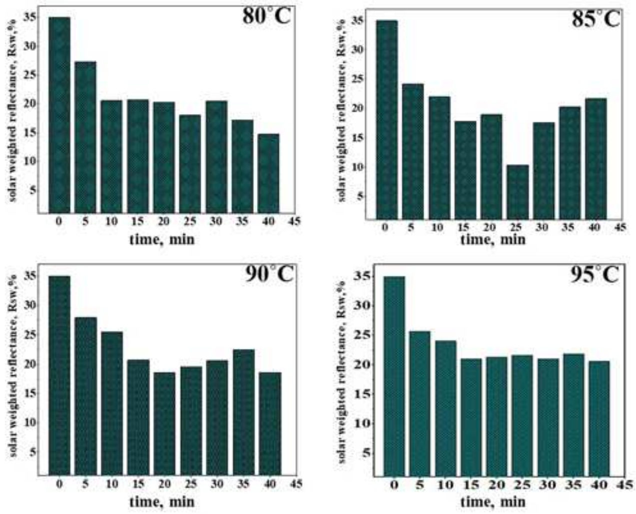

Fig. 5 *Solar WR values for samples etched in 0.5 wt% TMAH at different etching temperatures (80, 85, 90, and 95°C) for 5–40 min of etching time*

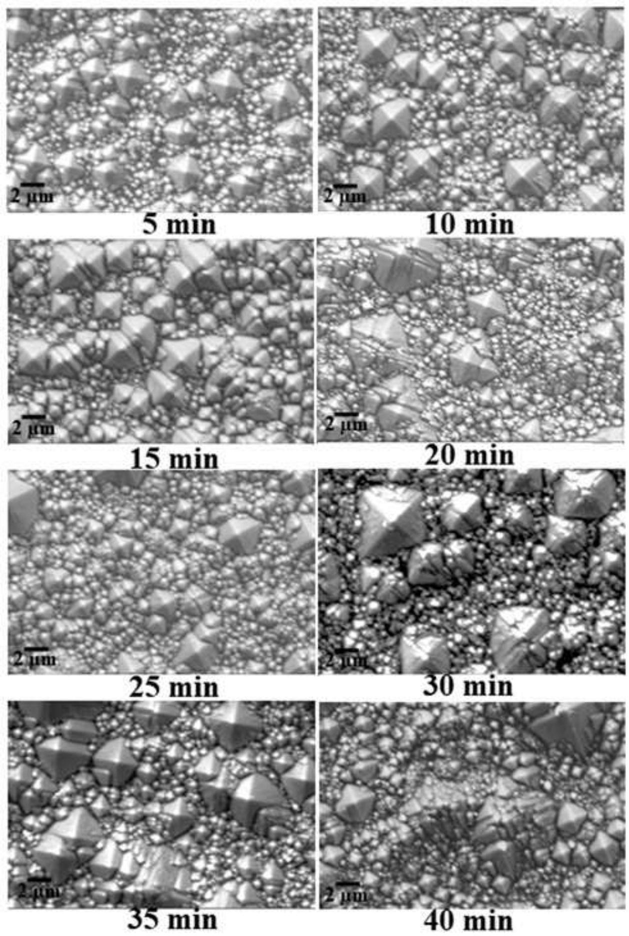

Fig. 6 *SEM images of samples etched in 0.5 wt% TMAH for different etching times from 5 to 40 min at 85°C*

In reduction, these weakened Si–Si back bonds are attacked by water molecules. This results in the breaking of back bonds and subsequently $Si(OH)_4$ and H_2 are formed as products by removing the surface Si atom. These hydrogen bubbles are stuck on the Si surface and act as a pseudo-mask during the etching process that results in the formation of micropyramids [21, 24].

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It can be noticed in Fig. 6 that the etching of Si{100} produces random sized pyramidal hillocks of sizes varying from 1 to 6 µm, but the surface is not fully covered with pyramids at 5 min of etching. It can also be observed in Fig. 6 that the size of pyramidal structures increases with an increase in etching time from 5 to 20 min, but the pyramidal structures are non-uniform. The pyramidal structures on the surface etched for 25 min are more uniform and cover the Si surface nicely in comparison with other samples. Further increase in etching time from 25 to 40 min increases non-uniformity in the pyramidal structures. To compare the etched surface morphology of the samples etched at different etching temperatures (80, 85, 90, and 95°C), the SEM images are taken on the samples etched for the same etching time. Fig. 7 shows the SEM images of the samples textured in 0.5 wt% TMAH at 80, 85, 90, and 95°C etching temperatures for a fixed etching time of 25 min.

It can easily be noticed that the pyramidal structures on the surfaces etched at a lower temperature (80 and 85°C) are more uniform and dense in comparison with those etched at higher temperatures (90 and 95°C). The same trend can also be seen in Fig. 3 where the SD in Sq value is more on the samples etched at 90 and 95°C in comparison with those etched at 80 and 85°C. This is the main reason that the lower reflectance is achieved on the samples etched at 85°C. If the samples etched at 80 and 85°C are closely inspected, pyramidal structures on the sample etched at 85°C are more uniform and dense in contrast with those etched at 80°C. It can be concluded here that the higher values of surface roughness are not directly related to lower reflectance values, but uniformity and density of pyramidal structures play an important role to minimise the reflectivity through multiple reflections [5, 8, 11]. Incident light falling to the non-uniform and less dense surface may not find any nearer structure to take another bounce and therefore went back to the atmosphere without having multiple internal reflections. As shown in Fig. 5, there is a particular etching time for each etching temperature, where the reflectance value is a minimum. The lowest R_{sw} values on the samples etched at 80, 85, 90, and 95°C for 40, 25, 20, and 15 min are 14.7, 10, 18.5, and 21%, respectively. Fig. 8 presents the SEM micrographs of samples etched in different temperature solutions for different etching times at which minimum reflectance is obtained. It can obviously be seen that the sample etched at 85°C for 25 min etching time exhibits denser and uniform surface morphology compared to others, which result in the lowest R_{sw} of around 10%.

To know more about the uniformity of pyramidal structures quantitatively, the SA of the etched samples is measured using 3D-LSM. Here, the SA at 15 different places is measured by scanning a 40×40 µm^2 area of the samples etched at 80, 85, 90, and 95°C. Thereafter, the corresponding mean and SD values are calculated. Fig. 9*a* shows the SA on the samples etched at different temperatures

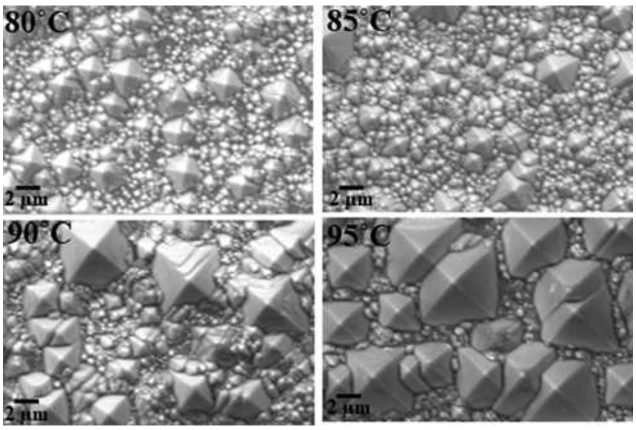

Fig. 7 *SEM images of samples etched in 0.5 wt% TMAH for 25 min at different temperatures*

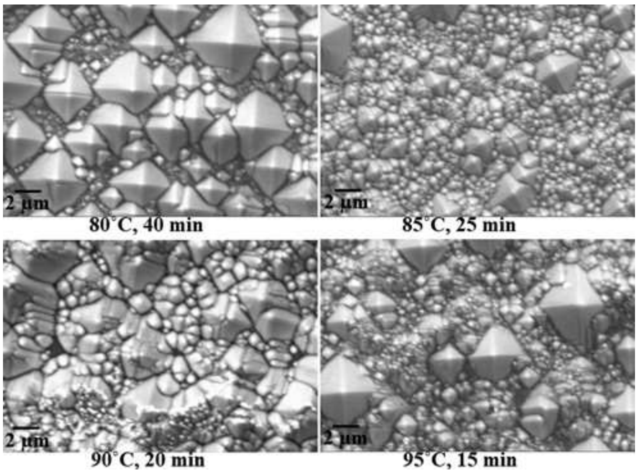

Fig. 8 *SEM images of pyramid-textured surfaces on which minimum reflectance is obtained*

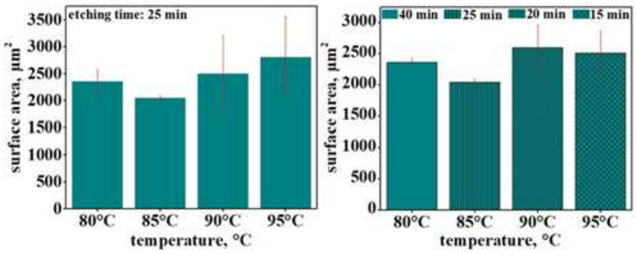

Fig. 9 *Variation in SA measured by scanning 40×40 µm^2 area on the samples etched*
a For 25 min at different temperatures (80, 85, 90 and 95°C),
b For different times (40, 25, 20, and 15 min) at various temperatures (80, 85, 90, and 95°C), respectively

80–95°C for a 25 min etching time, whereas Fig. 9*b* presents the SA for the samples etched for 40, 25, 20, and 15 min at 80, 85, 90, and 95°C, respectively. The error bars shown by red lines indicate the SD of the mean value of the SA. As one can observe in Fig. 9*a*, the variation in SA values in the case of 85°C is very less compared to 80, 90 and 95°C. The SA values in the case of 85°C are varying in the range of 1958–2091 µm^2, however, in the case of 95°C it is fluctuating in the range of 1992–4277 µm^2. This further confirms uniform structures at 85°C compared to other temperatures. Fig. 9*b* exhibits the SA on the samples etched for different times (40, 25, 20, and 15 min) at different temperatures (80–95°C) where the lowest reflectance is observed. As we can observe, SD in SA values is less at 85°C. Hence, it can be concluded that 0.5 wt% TMAH at 85°C for 25 min etching time provides optimal surface morphology in terms of uniformity and density of pyramidal structures to achieve lower reflectance in the visible range.

To understand the importance of the results presented in this paper, Table 1 shows a comparison of the reflectance values obtained on the surfaces textured under various etching conditions in different concentrations of TMAH [4, 8, 14, 15, 21]. In this study, we have achieved the lowest R_{sw} of 10% in the visible wavelength and minimum reflectance of 6.7% at 790 nm on the silicon surface etched at 85°C for 25 min, which is less than the values reported by other researchers on the silicon surface textured in TMAH solution under different etching conditions as can be noticed in Table 1. In a nutshell, the pyramid-textured Si{100} surface with low reflectivity is achieved in a very low concentration of TMAH with a reduced etching time. The results presented in this paper are very useful to scale-up for large-area surface texturing for Si solar cells.

Table 1 Comparison of reflectance values obtained on the wet anisotropically textured surface under various etching conditions

S. no	Etchant	TMAH concentration, %	Sample size	Temperature, °C	Additive	Agitation	Etching time, min	Reflectance	References
1	TMAH	2	2″ wafer	80	10% isopropyl alcohol (IPA)	—	30	10.7 at 799 nm	Wang *et al.* [4]
2	TMAH	2	4″ wafer	80	—	magnetic stirring	40	9.8 at 600 nm	Rosa *et al.* [15]
3	TMAH	2	—	80	8% IPA	ultrasonic agitation	30	weighted reflectance (WR) = 13	Papet *et al.* [8]
4	TMAH	1.5	—	80	9% IPA	—	60	average reflectance <10	Chen *et al.* [21]
5	TMAH	1	2×1.5 cm^2	95	—	—	15	average reflectance ≃12	Ou *et al.* [14]
6	TMAH	0.5	one-quarter of 3″ wafer	85	—	—	25	6.7 at 790 nm & $R_{sw} = 10$	present study

4. Conclusions: In summary, the effects of etching temperature and etching time on the surface texturing of Si{100} in a very low concentration CMOS compatible TMAH are investigated in detail. Surface roughness, surface morphology, and SA are characterised using SEM and 3D-LSM. Etching time (5–40 min) and etching temperature (80–95°C) are optimised to texturise the Si surface to obtain minimum reflectance. The Sq values are increased from 0.2 to 1 µm when the etching temperature is increased from 80 to 95°C for etching times of 5–40 min. The highest Sq of 1 µm is achieved for 40 min etching time at 95°C. In this study, 85°C with 25 min etching time is observed as an optimum etching condition to obtain a Si{100} surface with uniform and dense pyramidal structures to minimise reflection. The uniformity in the pyramidal structures is assured by measuring the SA variation at different locations of the same sample. SD in SA values is found to be less for 85°C etching temperature compared to other temperatures, which confirms uniform structures. For these etching parameters, R_{sw} of 10% is achieved for visible light while a minimum reflectance of 6.7% is attained at 790 nm. Reflectance values achieved in this work under mild etching conditions are very encouraging for large-area surface texturing for solar cell applications. Moreover, the use of very low concentration TMAH with less etching duration can minimise chemical waste and increase industrial throughput.

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