

Texturing of large area multi-crystalline silicon wafers through different chemical approaches for solar cell fabrication

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ABSTRACT

Surface texturing of silicon can reduce the reflectance of incident light and hence increase the conversion efficiency of solar cells. Comparatively lesser concentrated (10%) standard alkaline (NaOH/KOH) solution does not give good textured multi-crystalline silicon (mc-Si) surface, which could give satisfactory open-circuit voltage. This is due to grain-boundary delineation with steps formed between successive grains of different orientations. In this work an attempt has been made to obtain a well-textured mc-Si surface through three different approaches. The first two are with two different types of acid solutions and the third with concentrated alkaline NaOH. Solutions of HF–HNO₃–CH₃COOH/H₂O system with different concentrations of HF and HNO₃ were used for texturing. The results on the effect of texturing of these three solutions on the surface morphology of very large area (125 mm × 125 mm) mc-Si wafer as well as on the performance parameters of solar cell are presented in this paper. Attempts have been made to study extensively the surface morphologies of mc-Si wafers in two effective regions of the isoetch curves of the HF:HNO₃:diluent's system. Also we studied the reflectance, uniformity, spectral response, short-circuit current, open-circuit voltage, fill factor and dark current–voltage of the cells fabricated using wafers textured with the three different methods. Short-circuit current of the solar cells fabricated using acid-textured wafers were measured to be in the range of 4.93 A. This value is 0.37 and 0.14 A higher than the short-circuit current values measured in the cells fabricated with isotextured and alkaline-textured wafers, respectively.

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1. Introduction

Multi-crystalline silicon (mc-Si) surface texturing is a key issue in fabricating low cost and high-efficiency solar cells in mass production level. The well-established texturing method for mono-crystalline silicon is the anisotropic etching with aqueous solutions of potassium or sodium hydroxide (KOH/NaOH), which results in the surface covered with pyramids. This is due to the difference in etch rates of the $\langle 100 \rangle$ and $\langle 111 \rangle$ orientated planes of silicon [1]. The conventional alkali texturing is not so effective for mc-Si where the grains are randomly oriented. Moreover, mc-Si texturing with standard NaOH/KOH solution at comparatively lower concentration (10%) does not give textured surface, which could give satisfactory open-circuit voltage. This is mainly due to the grain-boundary delineation with steps formed between successive grains of different orientations. Several texturing techniques are under investigation, but none have reached the status of mass production for standard screen-printed solar cells [2–6]. Texturing with concentrated NaOH solution

(40%) at high temperature introduces less effect on mc-Si grain boundaries. Although reactive ion etching (RIE) is one of the well established, one out of many techniques under investigation for texturing mc-Si surface for solar cell fabrication [7,8], it is a complicated processes and requires expensive instrumentation. Another disadvantage of RIE technique is the essential requirement of an additional etching step in order to achieve better cell performance, which ultimately hamper industrial production throughput. On this background, we made an attempt to texture the mc-Si wafers without exposing the etched steps at the grain boundaries and to obtain a near uniform surface. We also examined the viability of this approach for industrial application.

The wet texturing of silicon surface with solutions containing HF/HNO₃ leads to isotropical etching resulting in features with rounded structures on surfaces. We have investigated the effect of texturing the mc-Si wafer for solar cell application using solutions chosen from two different regions of the isoetch [9] curves of HF–HNO₃–CH₃COOH/H₂O system. In some regions of isoetch curves of HF–HNO₃–CH₃COOH/H₂O system, huge amount of heat is evolved during rapid etching, which results in polished surface. Solutions belonging to some other regions of the curve generate porous structures on the silicon surface, which ultimately convert

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into a uniform surface with significant etch pits to capture appreciable percentage of incident light.

An comparative study of the surface morphology of mc-Si wafers treated with solutions from the two different regions of the isoetch curves for silicon (HF:HNO₃:diluent's system) as well as with NaOH has been carried out in this work, along with the study of the performance parameters of the fabricated solar cells.

2. Experiment

2.1. Texturization

p-Type, mc-Si wafers with resistivity of about 0.5–2 Ω cm were used in this experiment. HF–HNO₃–CH₃COOH solution with varying volume ratios of HF, HNO₃, and CH₃COOH was employed in acid texturing process of the silicon surface. The etching time in each process was also varied. Seven sets of etching solutions with different ratios of HF, HNO₃, and CH₃COOH, which fall in the isoetch region, were used to carry out the etching process (Table 1). Another experiment was carried out with HNO₃–HF–H₂O solution where H₂O was used as the diluent instead of CH₃COOH. In a third set of experiment, mc-Si wafers were etched with 30% and 40% NaOH solutions at different period of etching. During the texturing process with NaOH solution, a temperature-controlled hot plate and a flat polypropylene squire's tub filled with water were used to cover the etching vessel in order to prevent loss of chemical. Seven liters of solution of each type was taken to carry out the texturing. The surfaces of the mc-Si wafers were observed with an optical microscopic (Olympus 671998) after each etching process. A spectrophotometer (Scinco S-3100) was used to measure their surface reflectance. The surface morphology of selected multi-crystalline wafers was also studied by a scanning electron microscopy (SEM).

2.2. Solar cell fabrication

Conventional process steps such as POCl₃ diffusion; plasma-enhanced chemical vapor deposition (PECVD) for silicon nitride (SiN_x) deposition as anti-reflection coating (ARC), and screen-printed metallization were followed for the fabrication of mc-Si solar cells. All textured p-type silicon wafers (125 mm × 125 mm) were diffused by pentavalent impurity (phosphorus) in a open-tube furnace using a conventional POCl₃ diffusion source at 850 °C for 7 min as pre-deposition, followed by a 16 min of drive-in. After PSG removal by short time dipping in diluted HF solution, DI-water rinsing and drying were carried out. The sheet resistance of n⁺ emitter layer was 50–55 Ω/cm. All the textured multi-crystalline wafers were then oxidized at 850 °C for 13 min. After edge isolation and oxide layer removal, about 70-nm-thick layer of SiN_x was deposited on the front side by means of PECVD at 450 °C as ARC. The refractive index of SiN_x film was maintained at 2.0.

Table 1

Different experimental conditions for acid etching in different regions of isoetch curves as given in Fig. 1

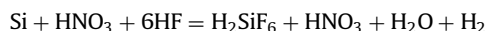
Etching point	Chemical composition	Volume ratio	Time of etching (min)
A	HF–HNO ₃ –CH ₃ COOH	2:15:5	1, 2, 3, 4, 5, 6
B	HF–HNO ₃ –CH ₃ COOH	1:8:1	1, 2, 3, 4, 5
C	HF–HNO ₃ –CH ₃ COOH	1:7:2	1, 2, 3, 4
D	HF–HNO ₃ –CH ₃ COOH	1:6:3	1, 2, 3, 4
E	HF–HNO ₃ –CH ₃ COOH	1:5:4	1, 2, 3, 4, 5, 6
F	HF–HNO ₃ –CH ₃ COOH	1:4:5	1, 2, 3, 4, 5, 6
G	HF–HNO ₃ –CH ₃ COOH	1:3:6	1, 2, 3, 4, 5, 6
H	HF–HNO ₃ –CH ₃ COOH	1:2:7	1, 2, 3, 4, 5, 6

The front and back metallization was carried out by screen-printing technique using standard Ag (product no. 3349, Ferro Electronic Materials) and Al pastes (product no. Fx53-038, Ferro Electronic Materials). This was followed by baking and co-firing at a temperature of 750 °C in a conveyer belt furnace (Seirratherm).

The illuminated current–voltage (LIV) and dark current–voltage (DIV) characteristics and the spectral response (SR) of the mc-Si solar cells fabricated using the three different surface etching methods were measured.

3. Results and discussion

Most of the experiments related to acid texturing were carried out with a HF–HNO₃–CH₃COOH mixture in volume ratios and etching time as mentioned in Table 1. The composition of the acids in the mixture was such that the etching rate fell in the lower region of the isoetch curves as shown in the Fig. 1. In this region, the etching contour runs parallel to the lines of constant HF. There is an excess of HNO₃ and the etch rate is governed by the ability of the HF to dissolve the SiO₂ as it is formed and by the removal of soluble complexes by diffusion into the volume of the etchant. The overall etching process is preceded by an induction period during which autocatalysis of HNO₃ is initiated. This is followed by cathodic reduction of HNO₃, resulting in a supply of holes, which enter into the oxidation reaction to produce SiO₂ and reacts with HF to form soluble complex H₂SiF₆. The overall reaction equation is given as



These etches are “self-passivating” in the sense that a freshly etched surface is already covered with a relatively thicker layer of SiO₂ (30–50 Å). Etching the silicon surface with this chemical in the lower region of the isoetch curves can yield orientation-independent texturing. It may be helpful for mc-Si polishing as well as texturing because mc-Si consists of different grains having different orientations. It is observed from the optical microscopic studies that there was no removal of saw damage marks from the surface of the mc-Si wafers after 1 min etching with different

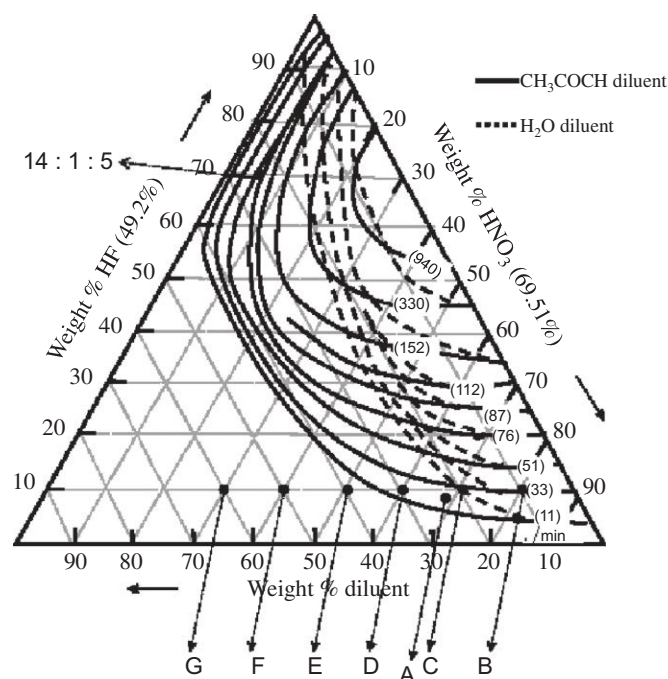


Fig. 1. Isoetch curves of HF–HNO₃–CH₃COOH/H₂O system.

recipes as mentioned in Table 1. The surfaces of mc-Si samples are observed as polished as well as textured, as shown in Fig. 2(a), after 3 min etching with the solution belonging to the A, B, C, D

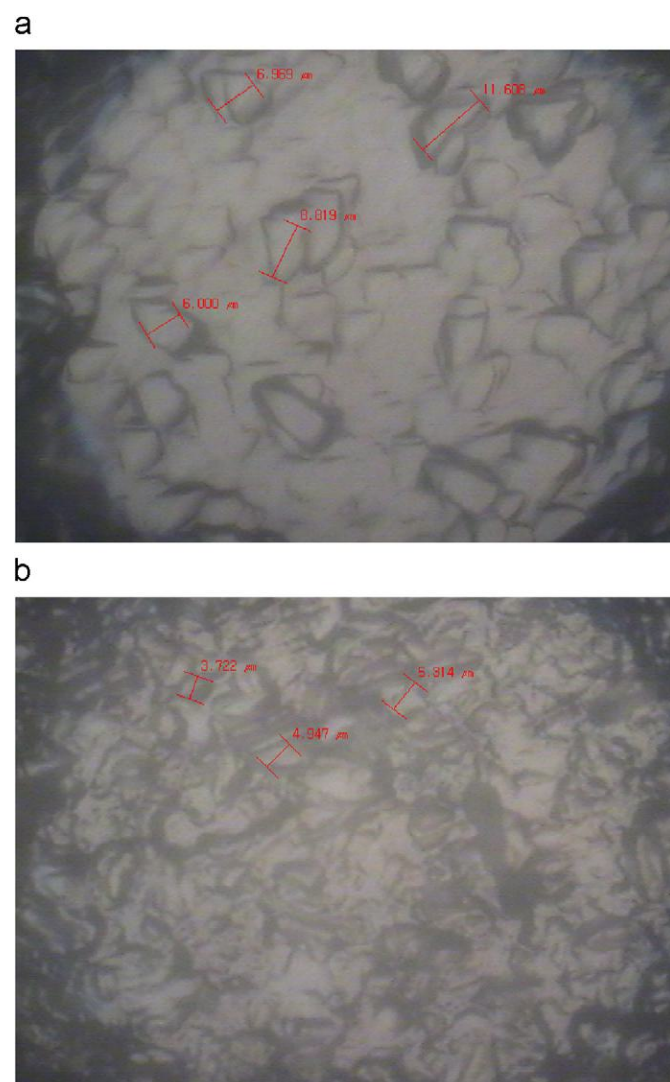


Fig. 2. Optical micrographs of mc-Si wafers textured with HF–HNO₃–CH₃COOH solution of A,B,C and D regions (a) and solutions of E, F and G regions (b).

regions of the isotech curve (Table 1 and Fig. 1). For the same etching time (3 min), the mc-Si surfaces are not quite satisfactorily etched with the solutions corresponding to the E, F, G regions (Fig. 2(b)). This may be due to the lesser etch rate of these solutions than the other solutions used for carrying out experiment in the regions A, B, C, D. It is also clearly evident in the optical microscopic images of Fig. 3. More etching time is required in order to achieve the same surface as produced by regions A, B, C, and D. In industrial application point of view, less time is usually preferred to achieve high throughput. The time and the volume ratio of the acids were kept fixed at 3 min and HF:HNO₃:CH₃COOH = 2:5:15, respectively, for etching during the solar cell fabrication. From Fig. 3 it is also seen that wafers etched for 4, 5 and 6 min also have good textured surface similar to the 3 min etching. Since short duration etching is preferable for industrial applications, the 3 min etching process is followed in the experiment.

It is seen from Fig. 4 that the grains of the mc-Si were textured but the texture geometry is different from grain to grain. This is due to the difference in oxidation and etching mechanisms of the grains oriented at different directions. Moreover, as the etching is isotropic in nature, no grain-boundary delineation has been observed in the optical microscopic studies of all the etched surfaces. SEM micrograph of a typical acid-etched sample using HF:HNO₃:CH₃COOH = 2:15:5 solution is shown in Fig. 5.

Investigation was done on the etching of mc-Si surface near the upper vertex of the isoetch curve shown in Fig. 1. Acidic solution with a volume ratio of HF:HNO₃:H₂O = 14:1:5 was taken for this experiment. The etching contour runs parallel to the lines of constant HNO₃ in this region and the etching rate is controlled by the concentration of HNO₃. This is due to the fact that there is an excess of HF to dissolve the SiO₂ formed during the reaction and the etching is limited by the rate of the oxidation–reduction reaction. It tends to be less orientation-dependent. The reaction in this region shows a totally different surface structure (Fig. 6) when compared to the surface structure after etching in the lower region of the isoetch curve (Fig. 5). This is due to the formation of porous silicon (PS) structure on the surface of mc-Si after 2 min etching with the solutions of HF:HNO₃:H₂O mixed in volume ratio 14:1:5. The whole surface of this large area mc-Si wafer looks uniformly blackish after 2 min etching with the aforesaid ratio. It is also noted that no grain boundary is exposed after etching.

There is a good difference between the etching behavior of the solution having higher percentage of HNO₃ and very lower percentage of HF (lower region of isoetch curve), and the solution

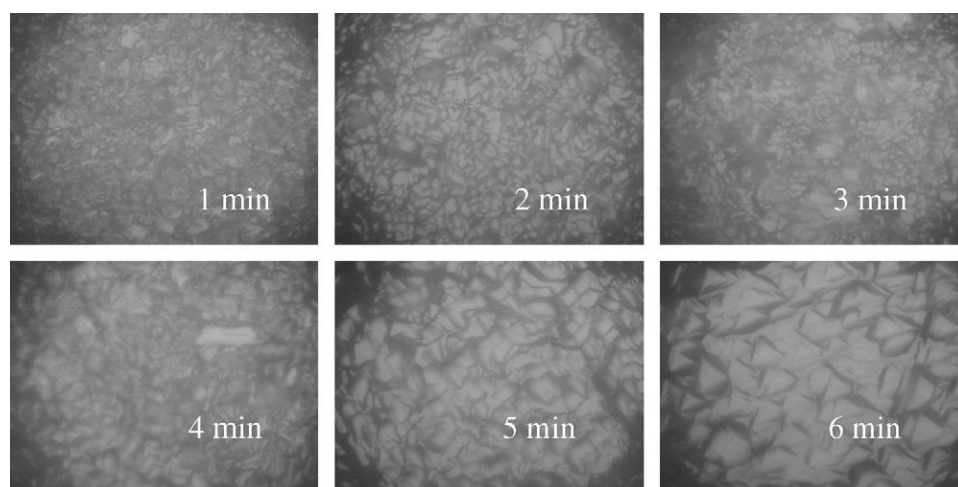


Fig. 3. Optical micrographs of mc-Si wafers textured with HF:HNO₃:CH₃COOH = 2:15:5 solution for different etching times.

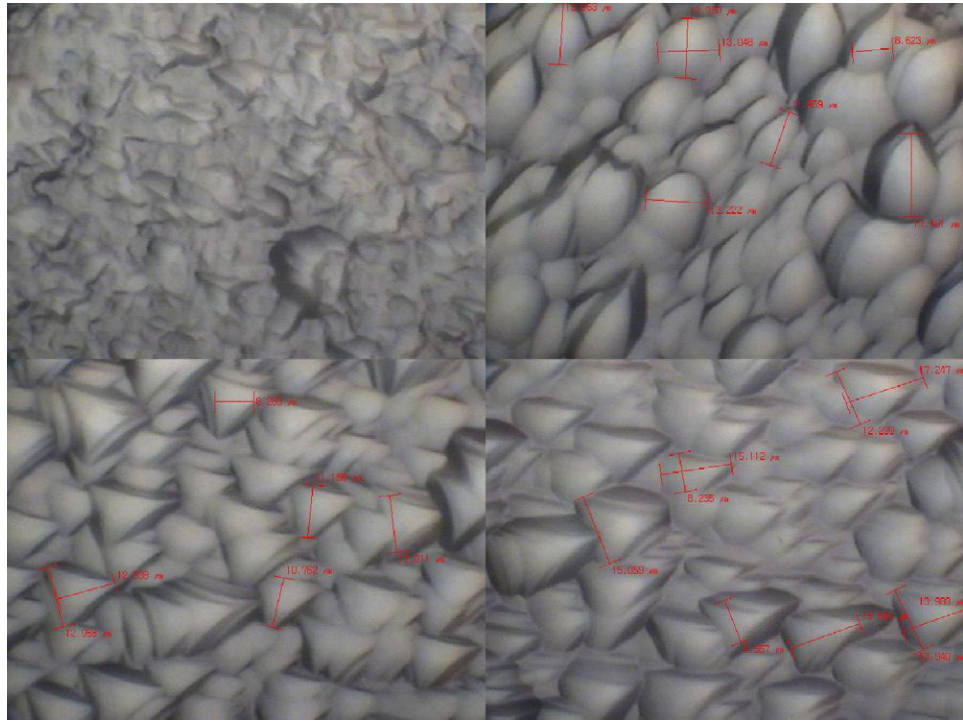


Fig. 4. SEM micrograph of the different portions of a wafer textured with $\text{HNO}_3:\text{CH}_3\text{COOH} = 2:15:5$ solution showing different texture geometry from grain to grain.

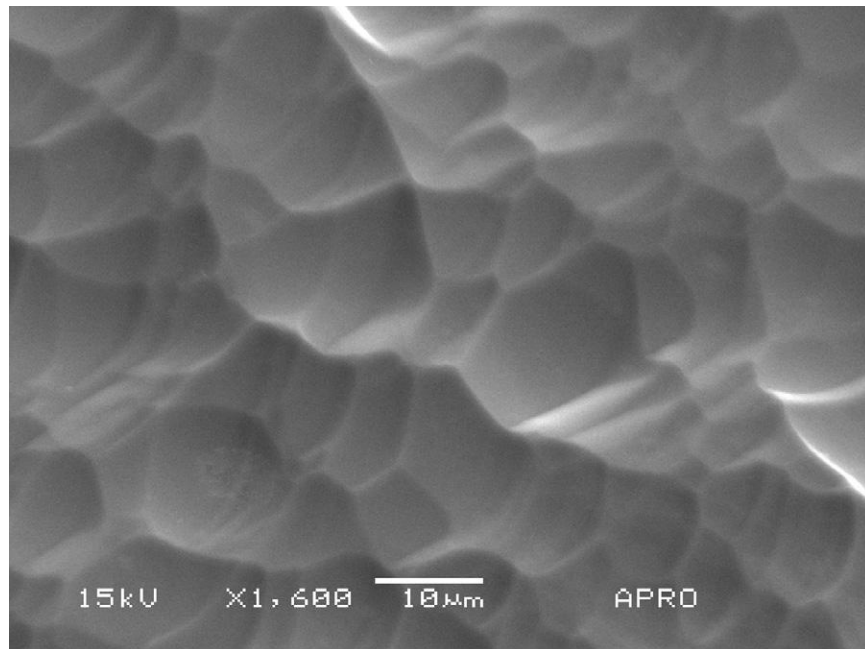


Fig. 5. SEM micrograph of an optimized acid-etched sample using $\text{HF}:\text{HNO}_3:\text{CH}_3\text{COOH} = 2:15:5$ solution. Etching duration is 3 min.

having higher percentage of HF and very lower percentage of HNO_3 (upper region near vertex of the isoetch curve). The etchant reaction of the former one shows little texturing effect on the surface of mc-Si whereas the later one gives well-textured surface with uniform pore structure of dimension around 10 nm after etching.

In order to compare the etching effect of higher percentage of NaOH with the acid etching approach, another set of experiments with 30% and 40% concentrated NaOH solutions were carried out.

It is observed from optical microscopic studies that the different grains attain different structural features after 3 and 5 min of texturing with 30% NaOH solution (Figs. 7 and 8), whereas, almost all the grains get almost similar feature after texturing with 40% NaOH (Fig. 9). In the case of texturing with the solution containing 30% NaOH, the chance of grain-boundary delineation is little more than that with 40% NaOH solution. Therefore, mc-Si texturing was performed with a solution with 40% NaOH during solar cell fabrication process.

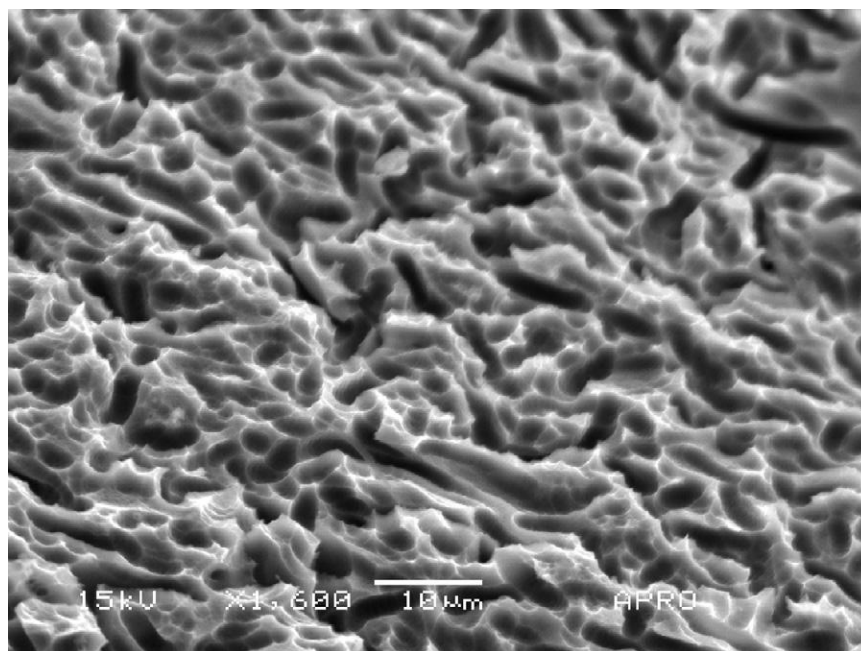


Fig. 6. Surface morphology of a wafer textured with $\text{HF}:\text{HNO}_3:\text{H}_2\text{O} = 14:1:5$ solution for 2 min.

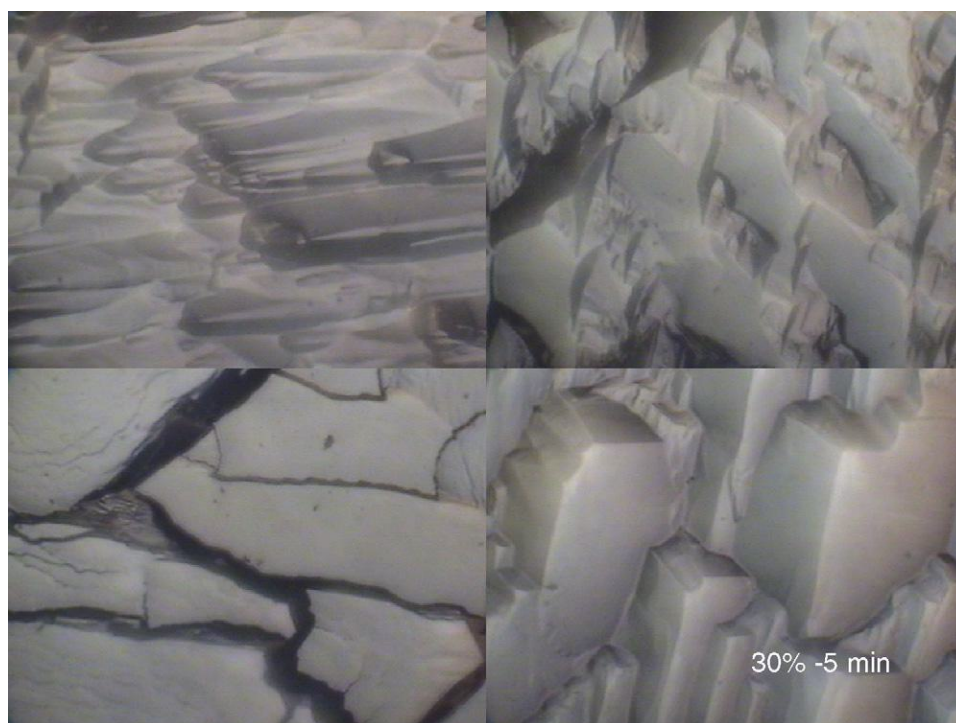


Fig. 7. Optical micrograph showing the structural features of different portions of a mc-Si wafer textured for 3 min with 30% NaOH solution.

It is observed from the SEM micrograph (Fig. 10(a)) that the surface is not fully textured with an extremely short time (30 s) etching using 40% NaOH solution. The height of the steps gradually increases with time (2 min) as shown in Fig. 10(b). Time and temperature need to be controlled precisely for appreciable texturing. The possibility of the high throughput rate due to very short time texturing is the main advantage of this process. The thinning of mc-Si wafers during texturing also leads

to the improvement in the cell performance due to good back surface field effect.

To compare the light-trapping effect on the surfaces of mc-Si wafers etched with the three different approaches, we measured the surface reflectance using a spectrophotometer after each etching. It is observed from Fig. 11, that the reflectance of the isotextured ($\text{HF}:\text{HNO}_3:\text{H}_2\text{O} = 14:1:5$) mc-Si sample without silicon nitride (SiN_x) AR coating shows lower values compared with

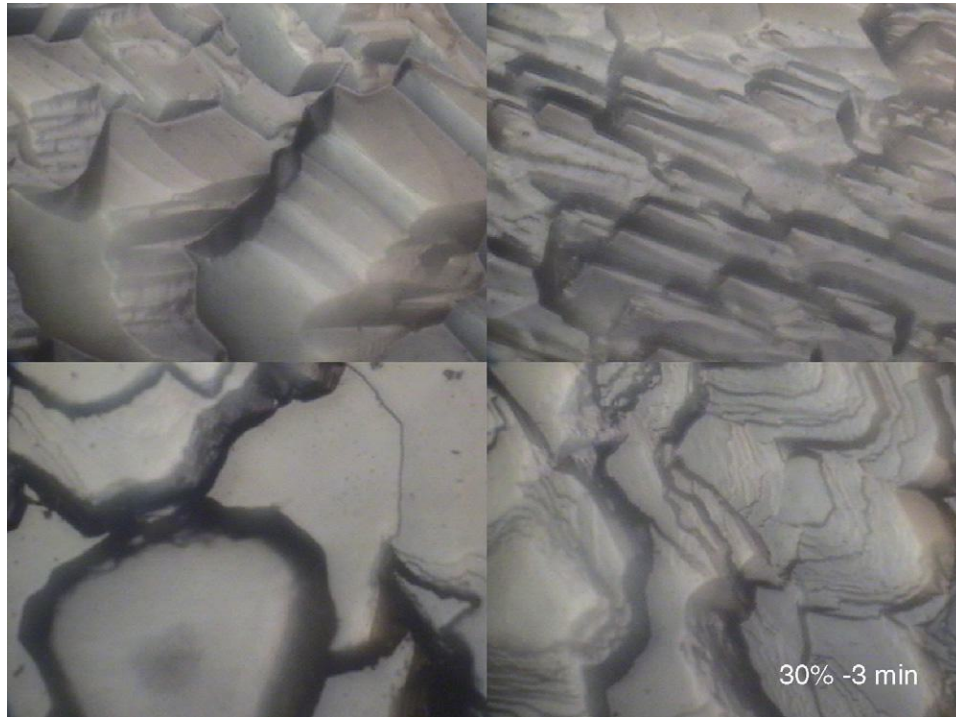


Fig. 8. Optical micrograph of the different portions of a mc-Si wafer textured for 5 min with 30% NaOH solution.

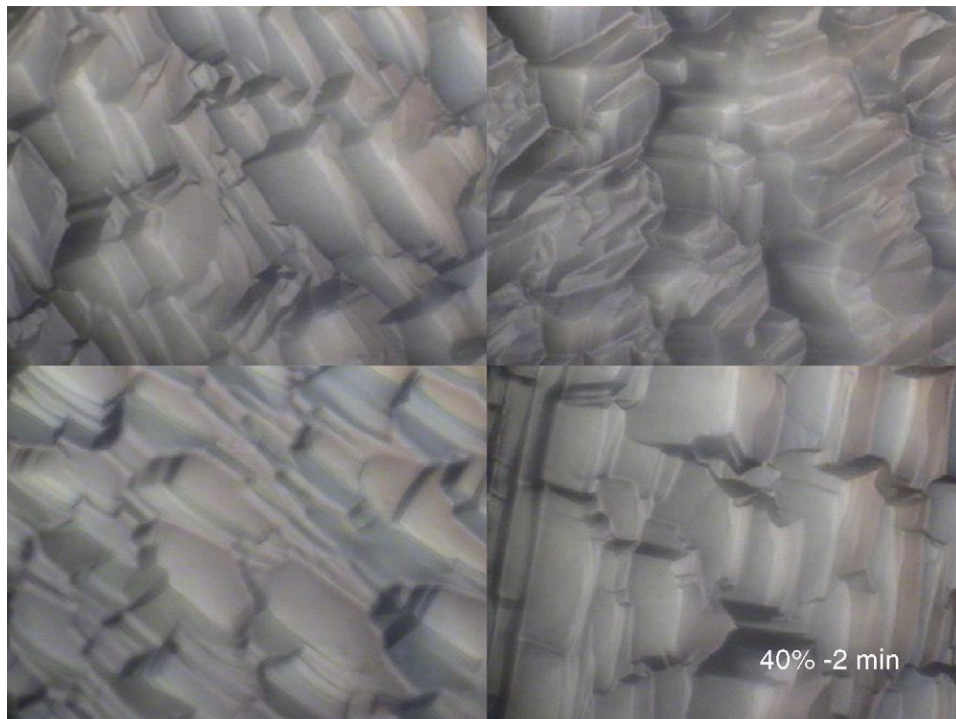


Fig. 9. Surface features of the different portions of a mc-Si wafer textured for 2 min with 40% NaOH solution.

NaOH (40%) textured and acid-textured ($\text{HF}:\text{HNO}_3:\text{CH}_3\text{COOH} = 2:15:5$) samples. The significant reduction of the reflectance of the isotexture mc-Si sample might be associated with two main factors. There is a possibility of reduction in refractive index due to the formation of the porous structure in the silicon substrate. The refractive index of PS is lower than that of the bulk silicon but higher than that of air [10]. The effective refractive index of the PS

layer will also depend on depth, since PS formation leads to the generation of Si structures with different porosity corresponding to the thickness of the PS layer. This porosity gradient leads to a refractive-index gradient, which notably reduces the values of the reflectance. On the other hand, the reduction observed in the reflectivity could also be partially explained by the higher roughness of the surface after PS formation. Higher the roughness,

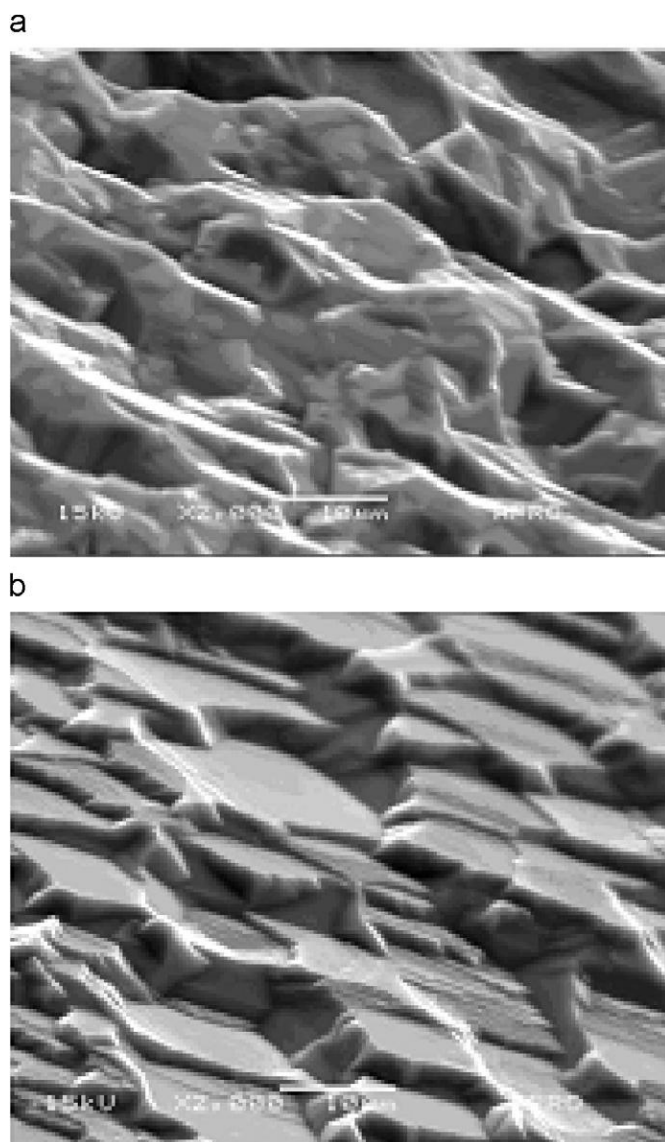


Fig. 10. Magnified ($\times 2000$) SEM image of 40% NaOH solution-etched wafer surface. (a) Etching duration 30 s and (b) etching duration 2 min.

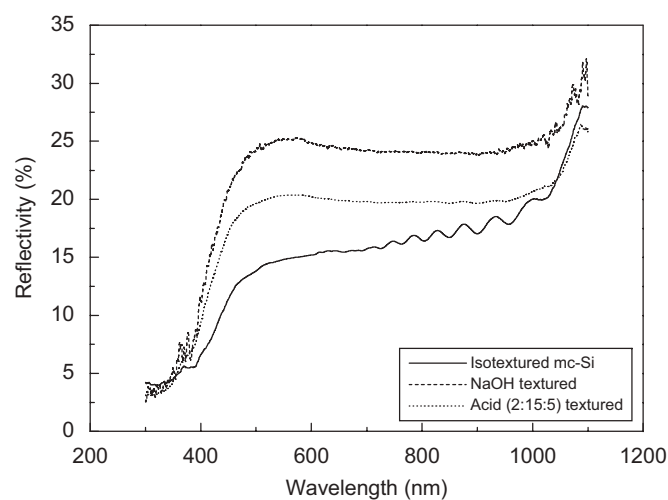


Fig. 11. Reflectance spectrum (without AR coating) of the mc-Si sample textured with the three different solutions.

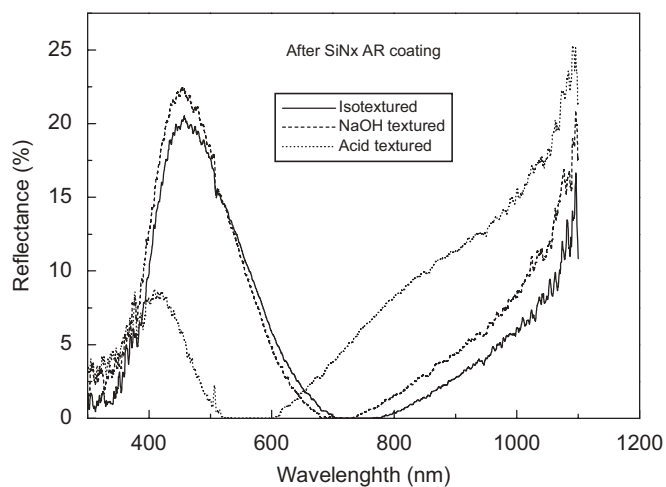


Fig. 12. Reflectance spectrum of the mc-Si sample textured with the three different solutions. The surface of the textured sample was coated with SiN_x.

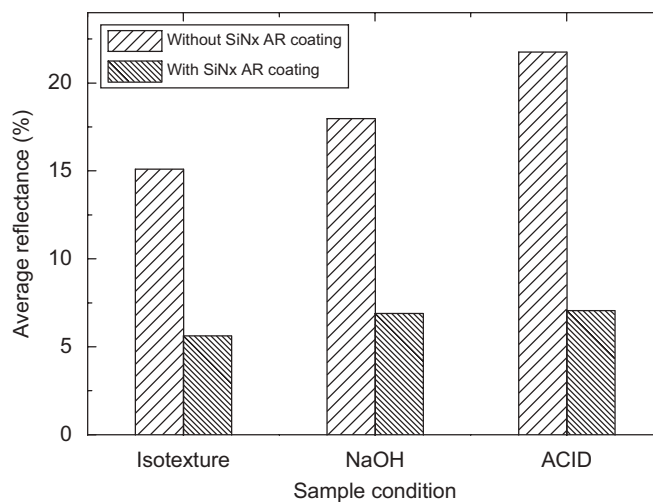


Fig. 13. Average reflectivity of mc-Si surface with and without AR coating.

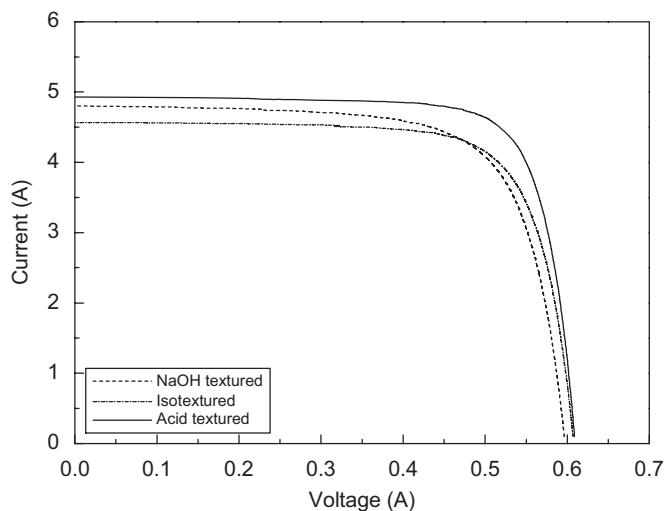


Fig. 14. Illuminated current-voltage (LIV) characteristics of the fabricated mc-Si solar cells with the NaOH texturing, acid texturing (2:15:5) and isotexturing (14:1:5) wafers.

Table 2

Performance parameters of large area (125 mm × 125 mm) mc-Si solar cells fabricated under different surface etching conditions

Surface condition	V_{oc} (V)	I_{sc} (A)	V_m (V)	I_m (A)	P_m (W)	FF	Eff (%)	R_s (m Ω)	R_{sh} (Ω)
NaOH texturing ^a	0.608	4.57	0.496	4.19	2.08	0.75	13.33	9	35.78
Isotexturing ^b	0.597	4.80	0.490	4.18	2.05	0.71	13.14	10	30.73
Acid texturing ^c	0.610	4.94	0.507	4.60	2.33	0.77	14.94	8	34.90

V_{oc} —open-circuit voltage, I_{sc} —short-circuit current, V_m —voltage at maximum power point, I_m —current at maximum power point, P_m —maximum power, FF—fill factor, Eff—efficiency, R_s —series resistance, R_{sh} —shunt resistance.

^a NaOH concentration = 40%.

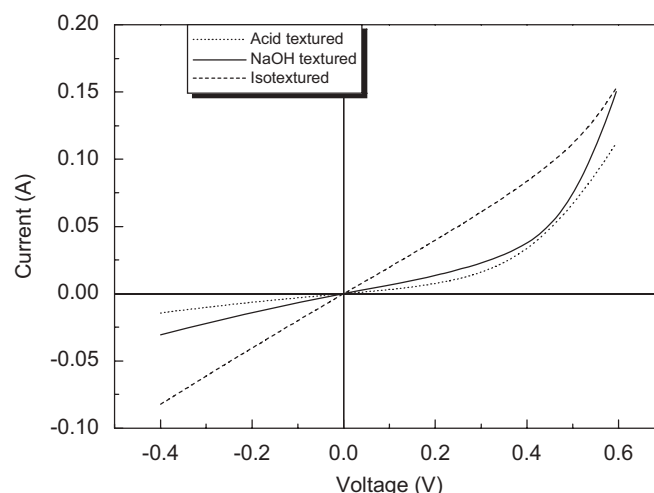
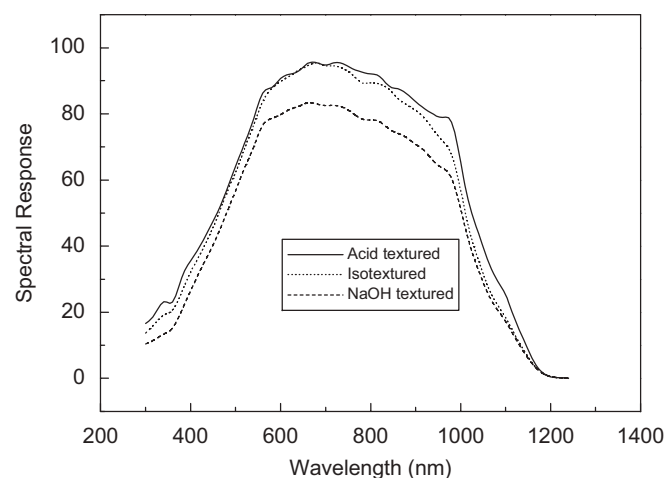
^b HF:HNO₃:H₂O = 14:1:5.

^c HF:HNO₃:CH₃COOH = 2:15:5.

higher the scattering and lower the reflectance. It can also be observed in Fig. 11 that the reduction in the reflectance is higher at lower wavelengths. This behavior can be explained from the significant reduction of the grain size compared with that of mc-Si, since the typical value of the grain size after PS formation is about 10 nm. For lower wavelengths, the scattering is higher and thus, the reflectance decreases. The higher value of reflectance in acid-textured (2:15:5) mc-Si sample compared with the isotexture and NaOH texture samples is due to lower light scattering from its polished surface. NaOH-textured samples show lower reflectance than acid-textured mc-Si because of higher light trapping effect due to its textured nature, which further helps for the greater light scattering compared with the polished mc-Si surface.

SiN_x film as AR coating as well as a surface passivation layer plays an importance role in commercial mc-Si solar cells [11,12]. It is observed from Figs. 12 and 13 that the average reflectivity of mc-Si surface drops from 15.10% to 5.62% for isotextured, 17.96% to 6.84% for NaOH textured and 21.77% to 7.05% for acid-textured surfaces, respectively, after SiN_x film coating. It is clear from Fig. 13 that the SiN_x film as AR coating significantly reduces the reflectance. The reduction in reflectance after SiN_x coating is higher in NaOH and acid-textured mc-Si surfaces than isotextured mc-Si surface.

LIV characteristics of the fabricated mc-Si cells with the NaOH texturing, acid texturing (2:15:5) and isotexturing (14:1:5) approaches were observed under global spectrum of AM 1.5 and are shown in Fig. 14. It is noted from the figure that the overall efficiency achieved in the case of acid-textured cell is much higher than NaOH (40%) texturing and isotexturing approaches. Almost all electrical parameters of the solar cell with acid-textured mc-Si wafer is much more favorable for achieving higher efficiency compared with the NaOH textured and isotextured mc-Si wafers (Table 2). This is due to different surface structures prevailing throughout the large area (125 mm × 125 mm) mc-Si resulting from the different texturing approaches. It is also observed from the Table 2 that all electrical parameters, except the short-circuit current (I_{sc}), of isotextured mc-Si solar cell are lower than acid-textured and NaOH-textured solar cell. The lower open-circuit voltage (V_{oc}) is due to inhomogeneous phosphorous concentration on porous structure surface, which leads to the increase in the carrier recombination [13] and thereby increasing the leakage current. This factor is clearly seen in the dark current–voltage (DIV) characteristic of mc-Si solar cell (Fig. 15). The lower fill factor is due to poor metal coverage by the finger and busbar structure and moreover it is very difficult to pass metal particle through the pores of lower diameter. The higher value of I_{sc} for isotextured mc-Si solar cell is due to its very low average reflectance value (5.62%) after SiN_x AR coating. Most of the

**Fig. 15.** Dark current–voltage (DIV) characteristic of mc-Si solar cell.**Fig. 16.** Spectral response of acid-textured mc-Si, NaOH-textured and isotextured mc-Si solar cells.

electrical parameters of the acid-textured mc-Si solar cell are higher than that of isotextured and NaOH-textured mc-Si solar cells. Surface flatness helps to improve fill factor and open-circuit voltage due to better metal coverage of the front metal grid patterning over the polished surface. Also, the leakage current in the acid-textured cell is even lesser than NaOH-textured cells. The higher value of the leakage current of the NaOH-textured cell is due to the steps across the grain boundaries. The conversion efficiency of the acid-textured cell is higher than the rest of the two cells fabricated with the other two texturing approaches.

It is observed from Fig. 16 that the spectral response of acid-textured mc-Si solar cell is better, in comparison to that of NaOH textured and isotextured mc-Si solar cells. This is due to the reasonable short-circuit current in the acid-polished cell. It can be concluded that the better electrical characteristics observed in the acid-textured mc-Si solar cells are due to the proper etching that gives uniform and smooth texture surface independent of the crystalline orientations. The only problem observed in acid texturing is the generation of fumes during vigorous exothermic reaction of the acid solution. Special etching bath with cooling system and with proper exhaust could solve this problem. Thus,

the acid texturing appears to be a better candidate to accomplish the task of mc-Si texturing with higher open-circuit voltage, short-circuit current and fill factor than the NaOH and isotexturing approaches.

4. Conclusion

We have investigated the texturing of mc-Si solar cells with solutions from the two different regions of isoetch curves of $\text{HF-HNO}_3\text{-CH}_3\text{COOH/H}_2\text{O}$ system and with NaOH solution of high concentration. Most of the electrical parameters of the acid-textured ($\text{HF:HNO}_3\text{:CH}_3\text{COOH} = 2:15:5$) mc-Si solar cell are higher than that of isotextured ($\text{HF:HNO}_3\text{:H}_2\text{O} = 14:1:5$) and NaOH-textured mc-Si solar cells. Surface flatness helps to improve fill factor and open-circuit voltage due to better metal coverage of the front metal grid patterning over the polished surface obtained due to acid texturing. Also, the leakage current in the acid-textured cell is even lesser than NaOH-textured cells. Reproducibility of the process with the acid texturing approach is also a very significant advantage. We have successfully fabricated large area ($125\text{ mm} \times 125\text{ mm}$) mc-Si solar cells using wafers textured with $\text{HF:HNO}_3\text{:CH}_3\text{COOH} = 2:15:5$ acid solution in a conventional industrial solar cell production line and achieved an efficiency of $\sim 15\%$.

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