Large-scale preparation of 22.06% efficiency single-crystalline silicon solar cells with inverted pyramid microstructure through nanostructure rebuilding treatment

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Abstract
The inverted pyramid microstructures (IPMS) were fabricated jointly by metal-assisted chemical etching (MACE) technology with extremely low concentration of silver ions to formed nanopores and nanostructure rebuilding (NSR) treatment of NH4HF2 solution. The results show nanopores were achieved with the aid of Ag-MACE technology, which diameter was mainly affected by the action of Ag ions, and depth was yet mainly influenced by HF concentration. Textured IPMS single-crystalline silicon (sc-Si) solar cells with the diameter of 1 μm and reflectivity of 8.62% were large-scale prepared. Benefiting from better light-trapping ability, the photoelectric conversion efficiency of IPMS sc-Si solar cells in standard size of 156.75 × 156.75 mm² achieved 22.06%. Simultaneously, the short-circuit current was increased by 71 mA. This promising technology could become a viable solution for industrial production of highly efficient sc-Si solar cells in the foreseeable future.

1. Introduction
Crystalline silicon solar cell has a dominant position in the solar cell market due to its low cost and high photoelectric conversion efficiency, especially single-crystalline silicon solar cell (sc-Si) [1–4]. However, it is troublesome to continue to improve the conversion efficiency of sc-Si solar cells with ultimate optimization of subsequent matching processes such as passivation and diffusion technique [5–7]. As reported in practical production, sc-Si solar wafers with UPMS fabricated in plant production have an average reflectivity of 10%–14%, which almost has reached the limit of one-step alkaline chemical texturing technique [8, 9]. It was reported that IPMS have ultra-low reflectivity that secondary reflection of light proceeds inside its structure [10]. Thus, the conversion efficiency of sc-Si solar cells could improve contributed to excellent light-trapping ability of IPMS.

In the past, many correlative attempts have done to improve conversion efficiency of sc-Si solar cells. Only by changing the surface structure to enhance the light trapping ability and ameliorating the passivation technology to reduce the surface recombination can improve the photoelectric conversion efficiency of sc-Si solar cell [11–13]. Armin Richter et al showed that the maximum theoretical photoelectric conversion efficiency of solar cells made of undoped silicon is 29.43% through new calculations [14], which provided us with reference for future. Moreno M et al used SF6/O2 plasmas in a reactive ion etching (RIE) system making it was possible to switch from random texture to pyramid-like texture and finally to inverted pyramid-like texture, which resulted in average reflectance values as low as 6% [15]. Due to poor controllability of practical production and high cost, this technology is not advisable for large-scale production in spite of improvement of conversion efficiency. Yang W et al used low concentration of nitric acid (HNO3) instead of hydrogen peroxide (H2O2) as oxidant to obtain black silicon wafers with surface reflection of 6.46% [16]. However, nitrogen emissions polluted environment making this technology undesirable. Faced with current trend of thinning of solar cells and improvement of conversion efficiency, it is a key step to apply atomic layer deposition technology into surface
passivation to reduce loss of silicon wafer surface recombination. Stapf A et al made full use of new acidic solutions containing hydrofluoric acid (HF), hydrochloric acid (HCl) and hydrogen peroxide (H₂O₂) fabricating random inverted pyramid structure that shows best performance, however, this conclusion has not been confirmed [17]. Wang S-D et al introduced that it is no trouble to obtain quasi-inverted pyramid structures with NaF/H₂O₂ solutions after MACE process [18], however, the reflectivity of silicon wafers with unevenness of nanostructures would be raised. This MACE technology (metal = Au, Ag, Cu, Co, Ni) [19–25] was well known for its application to crystalline silicon, however, on one hand, Au metals are so expensive and rare, on the other hand, Cu metals ions will accelerate corrosion of apparatus of reaction apparatus and reaction solutions containing Cu ions are difficult to handle resulting in environmental pollution. With these in consideration, this technology is difficult to get practical applications. Our laboratory previously reported that IPMS was prepared with the aid of Ag-MACE technology combined with NaOH solution [26], however, this method was difficult to control the morphology of IPMS and the photoelectric conversion efficiency was low.

In this work, we fabricated sc-Si solar cells with IPMS through Ag-assisted chemical etching technology and nanostructure rebuilding (NSR) treatment of NH₄HF₂ solution. The parameter conditions of obtained nanopores structure and IPMS were studied. The IPMS sc-Si solar cells express excellent photoelectric performance, in which ultra-low reflectance enhances light trapping ability to improve short-circuit current density. Thereby the conversion efficiency of IPMS sc-Si solar cells exceeded 22%.

2. Experimental

2.1. Methods

Diamond wire sawed (100)-oriented P-type sc-Si wafers (180 ± 10 μm thickness, 0.5–1.5 Ωcm) with size of 156.75 × 156.75 mm² were selected in this experiment. Sc-Si wafers were pre-cleaned with NaOH (0.12 M) and H₂O₂ (0.30 M) at 65 °C to remove surface oil and wafer residual cutting liquid in order to ensure the smooth progress of this experiment. With the help of Ag-MACE technology, mixed solutions containing H₂O₂ (2.49 M), AgNO₃, HF, and NH₄HF₂ (0.35 M) are used to polish to remove mechanical damage, plate silver and dig holes on the surface of sc-Si wafers, which nanopores silicon (NP-Si) were obtained. Then immerse in deionized water for 1 min. The deposited simple silver was removed in mixed solutions of H₂O₂ (0.50 M) and aqueous ammonia (0.19 M) for 5 min at room temperature. Then sc-Si wafers were successively rinsed in pure water for 1 min and HF (0.06 M) solution for 2 min to remove surface aqueous ammonia. Immediately after, NP-Si structures were anisotropically etched into IPMS in NSR treatment solutions of H₂O₂ (2.99 M), NH₄HF₂ (0.53 M) and 0.5% commercial additive B (Nanjing Naxin New Material Co., Ltd China) at 55 °C. Finally, optimized sc-Si textured wafers with inverted pyramid structure were prepared by hydrofluoric acid and pure water washing and drying.

The main processes of fabricating sc-Si solar cells wafer with IPMS are as follows: The conversion of light energy to electrical energy is realized by diffusion of phosphorus atoms to form P-N junction emitter. Acidic solution chemical etching method is used to remove phospholipases glass formed on the surface of silicon wafer after diffusion and consolidation. Plasma etching was used to etch doped silicon around solar cells to remove P-N junction at the edge of cells. Plasma-enhanced chemical vapor deposition (PECVD) was prepared for SiNx film in order to reduce surface reflection. The most promising Passivated Emitter Rear Cell (perc) technology is passivated solar cells back surface utilizing Al₂O₃ film, which can effectively reduce back surface recombination to enhance open circuit voltage and increase back surface reflection to raise short-circuit current. Screen printing is the most widespread production process for making solar cell electrodes currently. Then the conductive lines on silicon wafers are sintered to form electrode by rapid sintering at high certain temperature.

2.2. Material characterization

The weight loss of silicon wafers was measured by electronic balance (Hochoice, China). The optical reflectance exponent of 350 to 1050 nm was measured by a UV–vis and NIR spectrophotometer (UV-3101PC, Japan, with an integrating sphere). The microstructure of sc-Si was observed under atungsten filament scanning electron microscope (SEM; Regulus 8220, Japan). The film thickness measurement system (Filmetrics, F20-UV, USA) was used to measure the SiNx film. The internal/external quantum efficiency and photoelectric conversion efficiency of sc-Si solar cells could be obtained depending on the Enlitech QE-R and PVIV-411V systems separately. The electrical performances of (sc-Si) solar cells were characterized by current voltage (I–V) measurement system (Berger PSL-SCD, Germany).

3. Results and discussion

The formation of NP-Si is a complex process in which multiple reactions are performed simultaneously in mixed solutions composed of diverse chemical reagents. Figure 1(a) shows scheme of NP-Si obtaining
procedure we called ‘Plating, Polishing and Digging (PPD)’, which nanopores were prepared under the action of silver ions, HF and H2O2 followed by mechanical damage layer was removed with NH4HF2 solution. Among them, H2O2 was reduced as oxidizing agent and provided holes. HF was mainly reflected in the formation of nanopores, but reaction process of HF dissolving silicon and its compounds was slow, and the addition of NH4HF2 solution could speed up the reaction and remove mechanical damage layer as soon as possible. The mechanism of formation of nanopores structure was based on electrochemical corrosion reaction, chemical reaction equations are as follows:

Anode reaction:

\[
\text{Si} + 6\text{HF} \rightarrow 4\text{e}^- \rightarrow \text{SiF}_6^{2-} + 6\text{H}^+ \tag{1}
\]

Cathodic reaction:

\[
\text{Ag}^+ + \text{e}^- \rightarrow \text{Ag} \tag{2}
\]

\[
2\text{H}^+ + 2\text{e}^- \rightarrow \text{H}_2 \uparrow \tag{3}
\]

\[
\text{H}_2\text{O}_2 + 2\text{H}^+ \rightarrow 2\text{H}_2\text{O} + 2\text{h}^+ \tag{4}
\]

Overall reaction:

\[
\text{Si} + 6\text{HF} + \text{H}_2\text{O}_2 \rightarrow \text{H}_2\text{SiF}_6 + 2\text{H}_2\text{O} + 6\text{H}^+ \tag{5}
\]

As shown in figure 1(b), the mechanical damage layer was irregularly distributed on the surface of sc-Si wafers, such as damaged pits, cracks, and raised parallel marks [27], which were removed with NH4HF2 solution. After the PPD process, the nanopores structure formed in 3 M HF, 2.49 M H2O2, 5 M NH4HF2 and 5 ppm silver ions solutions at 50 °C for different time was seen in figures 1(c)–(e). As shown in figure 1(c), the diameter of NP-Si was about 80 nm for etching 3 min. With expending etched time, the diameter of NP-Si enlarged gradually. Figures 1(d), (e) show the diameter of NP-Si increased from about 140 nm to 180 nm for etching time from 5 min to 7 min, yet nanopores structure began to merge owing to diameter enlargement. Consequently, the
from 350 nm to 1050 nm formed at 50 °C concentration of silver ions leading to the overly intense catalytic process where the light-trapping ability of NP-Si began to weaken because of long-term time etching from 3 min to 7 min, the reflectivity was lower than 9.00% overall at 50 °C. However, when reaction temperature reached 60 °C, the weight loss continued to increase with time and reached 0.65 g for 7 min. Too small weight loss showed mechanical damage layer on the surface of silicon wafers not be completely removed, which cause the minority carrier lifetime remains low [29], meanwhile too much weight loss may result in the risk of silicon wafers becoming thin and brittle, which is not conducive to further forming surface structure. Figure 2(b) shows the average reflectivity at different treatment temperatures for different time. The definition of \( R_{\text{avg}} \) is as follows [30]:

\[
R_{\text{avg}} = \frac{\int_{350}^{1050} R(\lambda) N(\lambda) d\lambda}{\int_{350}^{1050} N(\lambda) d\lambda}
\]

where \( R(\lambda) \) and \( N(\lambda) \) represent total reflectance and the solar flux under AM 1.5, respectively. With time prolong from 3 min to 7 min, the reflectance continuously decreases from 12.00% to 9.60% at 40 °C. As shown in figure 2(b), the average reflectance first decreased and then increased was observed at 45 °C, and above, especially the reflectance was lower than 9.00% overall at 50 °C, which can be explained by the nanopores being formed first and then dissolved and combined. Therefore, the etching temperature of 50 °C and reaction time for 5 min as the well conditions to explore relationship between formation nanopores and concentration of silver ions.

Metal-assisted chemical etching technology (MACE) was the key to achieve nanopores that silver ions deposited on the surface of silicon wafers were reduced to silver particles as a metal catalyst additive to accelerate HF dissolving silicon nanopores [31]. Too low or too high silver ion concentration had a certain effect on solar cell’s electric performances [32], which needed to be studied detailedly. SEM images in figures 3(a)–(e) show the morphologies of NP-Si after MACE technology with different silver ion concentrations. Figure 3(a) shows that the diameter of NP-Si less than 20 nm was available with 2 ppm silver ion concentration. The diameter of NP-Si enlarged gradually as increasing silver ion concentration, which about 80 nm with 4 ppm, about 150 nm with 6 ppm and about 200 nm with 8 ppm silver ion concentration was shown in figures 3(b)–(d). Further adding silver ion concentration to 10 ppm, the diameter of NP-Si about 210 nm was observed in figure 3(e). However, the diameter of nanopores become abnormally large and the number decreased instead, which could be understood as the difficulty in controlling the nanopores expansion process owing to the excessively high concentration of silver ions leading to the overly intense catalytic process [33]. Figure 3(f) shows reflectivity spectra of NP-Si formed with different silver ion concentrations. The reflectivity first decreased and then risen as

![Figure 2](image-url)
concentration of silver ions increased. The reflectivity was 10.04% with 2 ppm silver ion concentration, then increasing silver ion concentration to 6 ppm, the reflectance reached a minimum of 7.90%. Majority of nanopores with larger diameter obtained were conducive to enhance light trapping ability to promote light absorption thereby reduce reflectivity. When concentration further increased from 8 ppm to 10 ppm, the reflectivity increases from 8.41% to 10.33%, which can be explained that big size and reduced number of nanopores weaken light trapping ability of silicon wafers surface. Therefore, we propose 6 ppm silver ion concentration at 50 °C for 5 min to achieve nanopores.

The nanopores-forming process is based on electrochemical corrosion reaction [34], which the reaction between silicon and hydrofluoric acid is extremely slow, hydrogen peroxide as oxidant can oxidize silicon to silicon dioxide and provide holes [35]. The surface silicon and its compounds are rapidly dissolved to form nanopores by hydrofluoric acid under catalysis of metal silver ions. The relationship between HF concentration and the diameter and vertical depth of nanopores was studied to fabricate suitable NP-Si after taking into account optimal etching time, temperature and silver ion concentrations. Figures 4(a)–(e) show the SEM morphology of NP-Si surface and cross section after MACE technology. Figure 4(a) shows the diameter of NP-Si about 180 nm, and the vertical depth about 1 μm (cross-section in figure 4(a)) obtained in 1.84 M HF concentration. However, the diameter of nanopores changed more smaller and smaller and the vertical depth get deeper and deeper as shown in figures 4(b)–(e). The diameter of nanopores was significantly reduced from about 150 nm to about 120 nm while the vertical depth added from about 2 μm to about 4 μm (cross-section in figures 4(b), (c)) when HF concentration was expanded from 5.52 M to 9.2 M. As shown in figures 4(d), (e), with HF concentration up to 12.88 M even 16.56 M, the diameter of nanopores was less than about 100 nm and the vertical depth raised to above 5 μm inconceivably (cross section in figures 4(d), (e)). The etching effect of HF on the silicon wafer is reflected in the horizontal and vertical etching. Among them, the low concentration of HF was mainly lateral etching to enlarge the diameter of nanopores, and the shallow large nanopores acquired had weak light trapping ability, which is disadvantage to reduce reflectivity. As HF concentration was increased, the high concentration of HF mainly embodied longitudinal etching to prepare deep small nanopores lines. However, the NP-Si was etched too deeply leading to some disadvantages, which not only brought great difficulties to NSR treatment to fabricate IPMS, but also lead to the degradation of performances of sc-Si solar cells [32]. In summary, considering the balance between nanopores diameter and vertical depth, relatively uniform microscopical NP-Si with diameter of about 120 nm and vertical depth about 4 μm were achieved by selecting 9.20 M HF concentration, which facilitate preparation of IPMS.

Based of obtained nanopores with aid of MACE technology, the IPMS were achieved by NSR treatment with mixed solutions of H2O2, NH4HF2 [36, 37]and additive B, which plays similar role as conventional sc-Si texturing surfactants with alkaline solutions [38]. The oxidation rate of H2O2 on each crystal plane of silicon was different, which made etching rate of NH4HF2 different on each crystal orientation of silicon showing as anisotropic etching, especially when oxidizing ability was promoted. Thus, inverted pyramid structures with
strong light trapping ability were fabricated. The morphological changes of IPMS were explored according to variation of different reaction texturing times controlling temperature at 55 °C in NSR treatment process. Figure 5(a) shows NP-Si structure, and figures 5(b)–(f) show IPMS fabricated for texturing time 2, 4, 6, 8 and 10 min, respectively. As shown in figure 5(b), the diameter of NP-Si structure was further enlarged to about 400 nm for processing 2 min without forming IPMS. With the extension of NSR treatment time, the NP-Si structure was gradually anisotropically etched into IPMS shown in figures 5(c)–(f). The diameter of big IPMS was 700 nm for texturing 4 min, but the convex structure at the edge of inverted pyramid was observed in figure 5(c), which increased carrier recombination and reduce lifetime of minority carriers [24, 26]. The smooth IPMS with diameter about 1 μm and depth about 1.5 μm were prepared with etching 6 min. As shown in figures 5(e), (f), further adding texturing time to 8 min even 10 min, the diameter of big IPMS was about 1.5 μm and 2 μm. However, the difference in diameter of IPMS fabricated was further amplified, meanwhile the bottom
IPMS began to dissolve to form platform. The bottom of IPMS with pits crippled trap-light ability and reduced light absorption due to too long etching, which would affect electrical performance of sc-Si solar cells. Figure 6 shows reflectivity spectra of IPMS by NSR treatment process with different time for 2, 4, 6, 8 and 10 min, respectively. As shown in figure 6, the reflectivity of nanopores with enlarged diameter was 9.20% for etching 2 min. With texturing time extending, the reflectivity reduced, especially in the middle and long wavelength band [39], which can be explained that IPMS formed by anisotropic etching of nanopore structures performed excellent light trapping ability. The reflectivity reached a minimum of only 8.62% when reaction time was 6 min, and then increased to 11.20% for texturing 10 min. The reflectivity instead increased in that prolonging NSR treatment time caused the diameter of IPMS enlarge and the bottom etched into pits, which prevented multiple reflections of absorbed light inside IPMS. Therefore, we made full use of NH4HF2 solutions containing particular buffering agent to fabricate IPMS with diameter of about 1 μm, depth of 1.5 μm and reflectivity of 8.62%, which texturing time was selected for 6 min and temperature was controlled at 55 °C.

Considering the rigor of comparison, we selected 400 pieces original silicon wafers by the odd–even sharding method, of which 200 pieces with inverted pyramid structure were fabricated in the laboratory and 200 pieces with upright pyramid structure were fabricated in the actual production line, and then assembled into solar cells in the same production line. Figure 7 shows the reflectivity, external quantum efficiency (EQE) and internal quantum efficiency (IQE) of IPMS and UPMS sc-Si solar cells, respectively. As shown in figure 7(a), after SiNx layers coating [40], the reflectivity of IPMS sc-Si solar cells was only 2.10% slightly lower than that of UPMS one. Meanwhile, the EQE of IPMS sc-Si solar cells was mildly higher than that of the UPMS sc-Si solar cells in the range below about 440 nm (near ultraviolet region) and above about 700 nm (near near-infrared region). The short-wavelength and long-wavelength spectral response of IPMS sc-Si solar cells improved compared with the UPMS one, which could be interpreted that low reflectivity was beneficial to enhance light trapping ability and improve short-circuit current density [41, 42]. In addition, the appearance color of UPMS sc-Si solar cells appeared blue-black, while IPMS sc-Si solar cells looked dark blue observed in figure 7(a). This could be understood that the UPMS sc-Si solar cells exhibited strong light-capturing ability to fully absorb visible light. The IQE of IPMS sc-Si solar cells was basically the same as that of the UPMS one except superior below about
380 nm indicating that it had lower surface recombination in short wavelength. According to the above discussion of parameters, the IPMS sc-Si solar cells were promising in terms of conversion efficiency beneficial to strengthen sunlight absorption capacity to increase short-circuit current.

The main electrical performance parameters are shown in table 1. The open-circuit voltage of IPMS sc-Si solar cells was 3.8 mV lower than that of the UPMS sc-Si solar cells, which the inverted pyramid structure cannot match traditional PECVD technology contributed to poor surface passivation [43]. In addition, the process of converting nanopores to inverted pyramids were often imperfect, they were not ideal inverted pyramids due to the residual pores, resulting in higher surface enhancement. As shown in table 1, the short-circuit current of IPMS sc-Si solar cells was 9.918 mA higher than the UPMS one thanks to low reflectivity enhancing light absorption capacity [44]. At the same time, the IPMS sc-Si solar cells of fill factor was 0.04% extra indicating that quality was also superior. Then, the average photoelectric conversion efficiency of IPMS sc-Si solar cells actually was 22.06% appreciably higher than the UPMS one by 0.05%, which showed the promising improvement in conversion efficiency. In summary, the further improvement of photoelectric conversion efficiency could be come true by optimizing surface passivation to increase open-circuit voltage.

4. Conclusions

In summary, the IPMS sc-Si solar cells with diameter of 1 μm and reflectivity of 8.62% were fabricated through Ag-MACE technology combined with NSR treatment of NH₄HF₂ solution. The diameter and depth of NP-Si were easily controlled by Ag ions and HF concentration, respectively, and then the formation of IPMS was changed by the NSR treatment process. the efficiency of the IPMS sc-Si solar cells was 22.06% and the short-circuit current was 71 mA higher, which expressed better cells performance compared with traditional UPMS sc-Si solar cells. Hopefully, the dilemma of current conventional UPMS sc-Si solar cells efficiency further improvement would be solved assuming that passivation technology could be optimized to match IPMS sc-Si solar cells. As expected, this technology is promising in the near future.

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Author contributions

Hongcheng Gu designed and performed the experiments and wrote the paper; Zisheng Guan modified the manuscript writing; Yuchen Liu check the expression grammar of language.

Conflict of interest statement

The authors state that they have no conflicts of interest in this work.

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Table 1. Comparison of main electrical parameters of UPMS and IPMS sc-Si solar cells.

| Groups               | Voc (mV) | Isc (A) | Rs (Ω)  | FF (%) | Eff (%) |
|----------------------|----------|---------|---------|--------|---------|
| UPMS sc-Si solar cells | 678.5    | 9.847   | 0.002 24 | 80.49 | 22.01  |
| IPMS sc-Si solar cells | 674.7    | 9.918   | 0.002 14 | 80.53 | 22.06  |
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