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PHOTOELECTROCHEMICAL PROPERTIES OF NANOSTRUCTURED SILICON FOR SOLAR WATER SPLITTING

Abstract

Silicon, one of the most abundant and cost-effective materials on Earth, holds significant promise for applications in water splitting and photovoltaics due to its suitable bandgap energy of approximately 1.12 eV, which allows absorption of ultraviolet, visible, and infrared light. However, the high reflectivity (~25%) of flat silicon surfaces limits its conversion efficiency, making it less efficient for photoelectrochemical (PEC) processes. To address this, nanostructured silicon has emerged as a solution to enhance light absorption, reduce substrate resistance, and improve hydrogen production efficiency. In this study, we fabricated nanostructured silicon photoelectrodes using the metal-assisted chemical etching (MACE) method. The resulting black silicon (b-Si) electrodes demonstrated superior light-harvesting capabilities, leading to significantly enhanced photocurrent densities. Notably, the b-Si photoelectrodes achieved a photocurrent density of 800 μA/cm² at 0V vs RHE (reversible hydrogen electrode), compared to 200 μA/cm² for planar silicon. Furthermore, the b-Si electrodes exhibited excellent long-term stability under continuous illumination for 16 hours. These results highlight the potential of nanostructured silicon as an efficient and stable material for solar-driven PEC water splitting and related renewable energy applications.

Key words: nanostructured silicon, solar water splitting, black silicon, metal-assisted chemical etching.

Introduction

Water splitting is the process of separating water into hydrogen and oxygen gasses through electrochemical reactions [1, 2]. This process holds great potential for clean and sustainable hydrogen production, which can be used as a renewable energy source [3, 4]. Currently, the most common method for water splitting is through electrochemical water splitting using free and inexhaustible solar radiation. This production route has several advantages, such as the use of abundant water, no greenhouse gas emissions, high hydrogen production efficiency, and high-purity products [5–7]. However, this technology also faces challenges, particularly in terms of cost and efficiency. To address these challenges, researchers have been exploring different catalysts and materials to improve the efficiency of photoelectrochemical water splitting. Recent studies have investigated the utilization of various materials and photocatalysts in combination with traditional silicon wafer in order to enhance the efficiency of photoelectrochemical water splitting [8–10].

Silicon is one of the most earth-abundant elements, production cost is not expensive [11]. It meets specified criteria as a substrate for water-splitting and also demonstrates high potential in photovoltaics [12, 13]. Moreover, band gap energy of approximately 1.12eV (300-1200 nm), allowing it to absorb ultraviolet, visible light, and a wide range of IR light make them a prospective for PV-PEC device. However, flat surface of silicon is that it reflects approximately 25% of incoming photons, which has a detrimental impact on conversion efficiency. Utilizing nanostructured silicon presents a solution for enhancing light absorption, reducing substrate resistance, and consequently boosting hydrogen production [14–16]. Many studies have focused on improving the PEC performance of black silicon photoelectrodes, including efforts to maximize light absorption, optimize doping, and surface passivation strategies [17, 18] to enhance photoresponse and conversion efficiency. As a result, substantial progress has been made in achieving higher photocurrents and improved hydrogen production efficiency. While significant attention has been given to efficiency, much less emphasis has been placed on the long-term stability of black silicon under PEC operating conditions.

In this work, we successfully fabricated nanostructured silicon (Si) photoelectrodes using the metal-assisted chemical etching (MACE) method. The fabricated nanostructured silicon photoelectrodes demonstrated excellent light-harvesting capability due to the increased surface area and reduced reflectivity of the black silicon. This enhanced light absorption contributed to higher photocurrent densities, indicating improved efficiency in the PEC process. A key advancement in this work was the demonstration of the long-term stability of the nanostructured Si photoelectrodes.

Materials and methodology

Electrode preparation

Nanotextured Si (NSi) surfaces were obtained using the selective chemical etching method initiated by metallic silver nanoclusters. The polished p-type semiconductor Si wafers with a resistance of 10 $\Omega*cm^{-1}$ were chosen as a starting material. The heated to 80°C solution of $NH_4OH:H_2O_2:H_2O$ with a volume ratio (1:1:4) was used for cleaning procedure of substrates. A two-step chemical treatment was used to create the textured surface. At the first stage, the plates were immersed in an aqueous solution of $AgNO_3:HF:H_2O$ for 10 seconds, while a layer of silver nanoparticles was deposited on the substrate. Experiments were carried out with $HF:H_2O$ solutions in a ratio of 1:4 and $AgNO_3$ concentrations of 4 mM. Then Si wafers were etched in a solution of $H_2O_2:HF:H_2O$ with a volume ratio of 1:2:10 for 30 sec, after which the wafers were thoroughly washed in deionized water. At the final stage, to obtain a clean textured surface, residual silver nanoparticles were removed by boiling for 10 minutes in a solution of $NH_4OH:H_2O_2:H_2O$ in a volume ratio of 1:1, followed by washing in deionized water.

Photoelectrochemical measurements

The photoelectrochemical measurements were carried out in quartz cell using three electrode configurations on an electrochemical workstation (Corrtest CS310, China). The prepared nanostructures silicon, Pt mesh and Ag/AgCl electrode were used as working, counter and reference respectively. The

Nernst equation is used to convert potentials: $V_{RHE}=V_{(Ag/AgCl)}+0.0591\times pH+ V_{(0Ag/AgCl)}$, where $V_{Ag/AgCl}$ is the applied potential, $V_{0Ag/AgCl}$ is the standard potential of the Ag/AgCl reference electrode and pH is basicity or acidity of the electrolyte. The pH of the electrolyte is 1.2. A linear voltammetry was provided at a scan rate of 10mV/sec in 0.5M H_2SO_4 electrolyte. The xenon lamp (PLS-SXE300+, Perfect light, China) with AM 1.5G filter was used as a light source. A 50 nm Al layer was deposited onto the front side of silicon to get ohmic contact, subsequently, electrode was annealed in the Ar atmosphere at 500^0C for 5 min. Then Cu wire was connected to the Al layer using an ultrasonic soldering machine (OPVSO600, OPVTech, China). The working electrode was illuminated with a light intensity of 100mW/cm^2 (PL-MW2000 Optical Power Meter) chopped each 2 sec.

Hydrogen evolution testing

Hydrogen evolution test was provided using three electrode configuration quarts hermetic cell, Pt mesh and Ag/AgCl electrode were used as counter and reference respectively. Th hermetic Teflon cell with quartz window was connected to the gas chromatography (Fuli instrument GC9790II) and purged with argon gas to remove dissolved oxygen and air from the cell. Amperometry technique was used to test hydrogen evolution onto the photoelectrode, scanning at 0V vs RHE potential. After irradiation, every 30 minutes the valve from the cell was opened so that the gas from the cell could enter to the gas chromatograph.

Material characterization

The film morphology was studied using scanning electron microscopy scanning electron microscope (SEM, ZEISS Crossbeam 540) equipped with an energy dispersive X-ray spectroscopy (EDX) system. The crystal structure of the films was studied using a MiniFlex X-ray diffractometer (Rigaku, Tokyo, Japan). Raman spectra were obtained using Ntegra Spectra (NT-MDT) spectrometer with 473 nm excitation. The UV-Vis spectroscopy (Cary 5000 UV-Vis-NIR) was used to study optical properties.

Results and discussion

The SEM image was obtained using a scanning electron microscope. Figure 1 presents a typical top and cross-sectional SEM image of nanostructured Si. It is obvious that Si nanowires are formed vertically oriented to the surface. The length of nanowires is 520 nm, while diameter ranged from 10 to 20 nm.

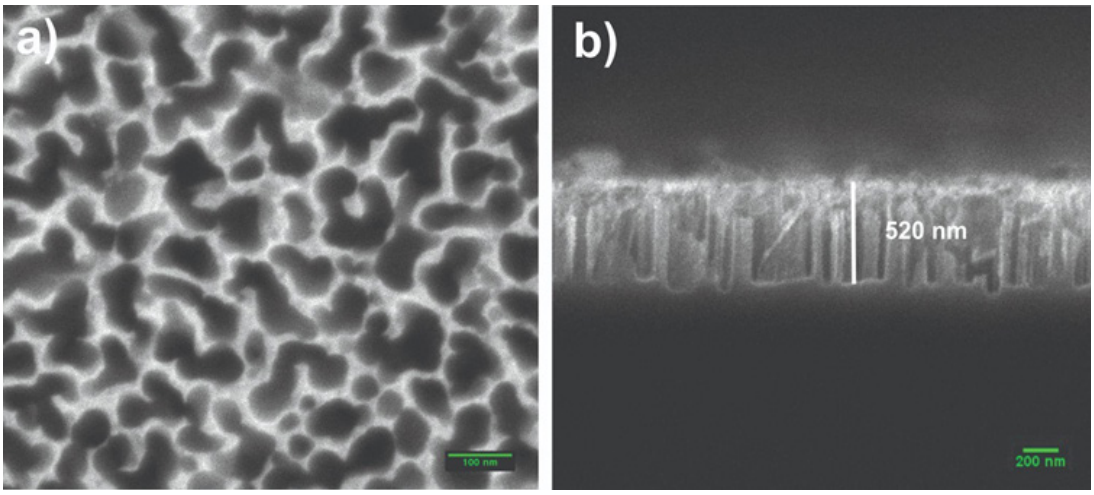


Figure 1 – SEM image of nanostructured Si top (a) and cross view (b)

Energy dispersive spectroscopy (EDS) results confirmed the silicon peak as shown in Fig.2. The structure of Si was studied using Raman spectroscopy. The Figure 3a demonstrates the Raman spectra for nanostructured Si (b-Si) and planar Si for comparison. As we can see, a strong peak located at 520 cm^{-1} and 960 cm^{-1} are in good agreement with other works. If we compare black and

planar silicon, the peaks are identical, there is a difference in intensity of the peaks. After etching the Raman signal is increased tenfold due to the nanostructured surface and geometry [19]. In our previous work [20] and in other works, a strong Raman scattering, enhanced by the surface (SERS effect) effect was found for nanostructured silicon [21].

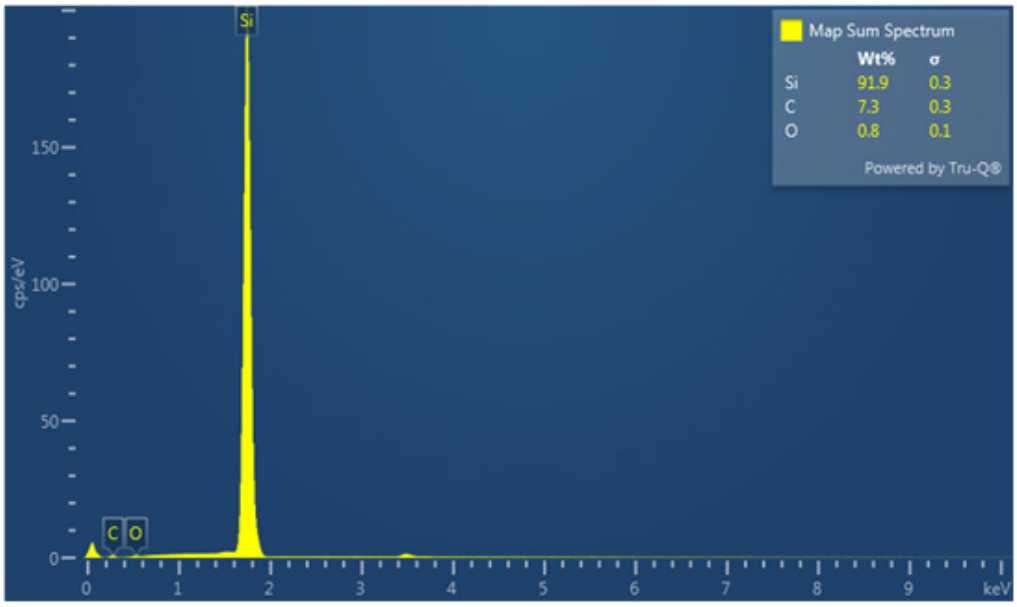


Figure 2 – EDS spectrum of black-Si

The UV-Vis spectroscopy (Cary 5000 UV-Vis-NIR) was performed to determine the optical reflectance of the silicon electrodes, and PTFE was used as a reference standard. The optical reflectance spectrum is presented in Fig.3b for planar and nanostructured silicon. As displayed in fig.3b the nanostructured surface of silicon decreased reflectance of light to 5%. This graph highlights how black silicon, due to its micro/nanostructures, absorbs more light than planar silicon, making it a more effective material for applications like solar water splitting or photovoltaics.

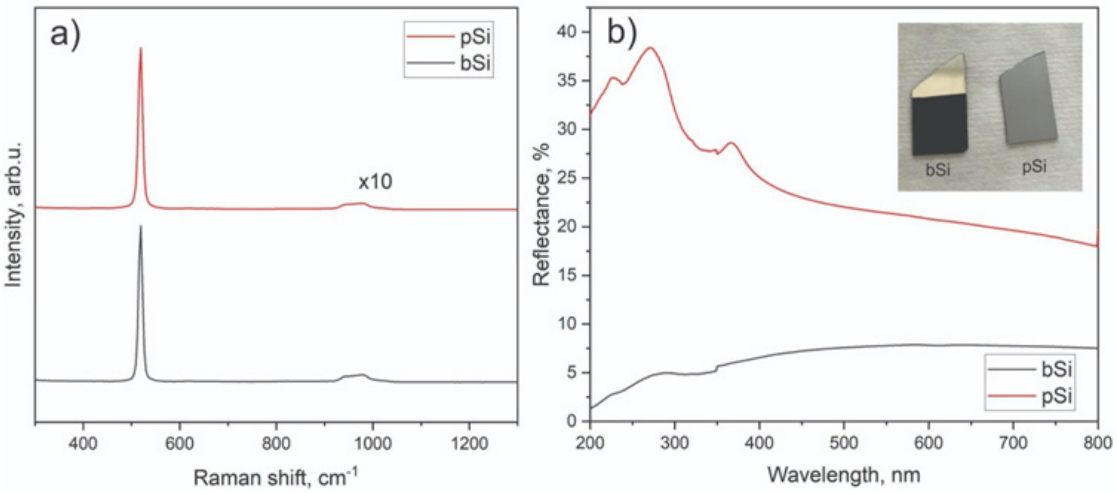


Figure 3 – Raman and reflectance spectra of black silicon and planar silicon

PEC water splitting properties of NSi are tested in three electrode configuration where the black silicon was used as a photocathode material. The figure 4a shows current-potential I-V curves chopped illumination each 2 sec are demonstrated. As can be seen, the negligible current is observed

in the dark. Under illumination the sharp rise of photocurrent beyond the potential indicates charge transfer and enhanced efficiency. The photocurrent density is increased by potential in case of black silicon, while planar silicon shows stable photoresponse at the entire potential region. The black silicon exhibits photocurrent density of 800 A/cm^2 while planar silicon shows 200 μA/cm^2 at 0V vs RHE. The enhancement of photoelectrochemical performances might be due to increasing surface area which provides more sides for water reduction at the electrode-electrolyte interface.

Stability is a critical parameter for practical applications, as photoelectrodes in PEC cells are exposed to harsh environments, including strong acidic or alkaline electrolytes. In this work, stability test was provided using chronoamperometry technique at 0 V vs RHE during 16 hours under continuous illumination (AM 1.5G) for nanostructured silicon (Fig 4b). It is seen that the SiNW electrode demonstrates good stability. The hydrogen evolution was tested using gas chromatography method as seen in Fig.5a. As can be seen from the figure, the first 30 minutes of the spectra contain three peaks corresponding to hydrogen, oxygen and nitrogen. At first oxygen and nitrogen are residual gases from the air, over time we see that the residual gas is gradually replaced by hydrogen, which is intensively released on the surface of working electrode. The hydrogen bubbles onto the nanostructured silicon under illumination was also demonstrated in figure 5b.

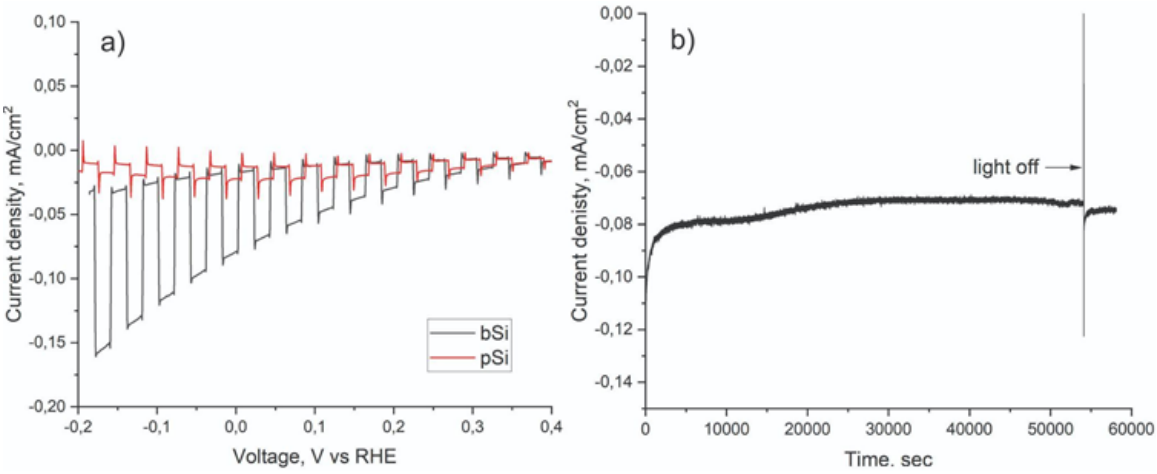
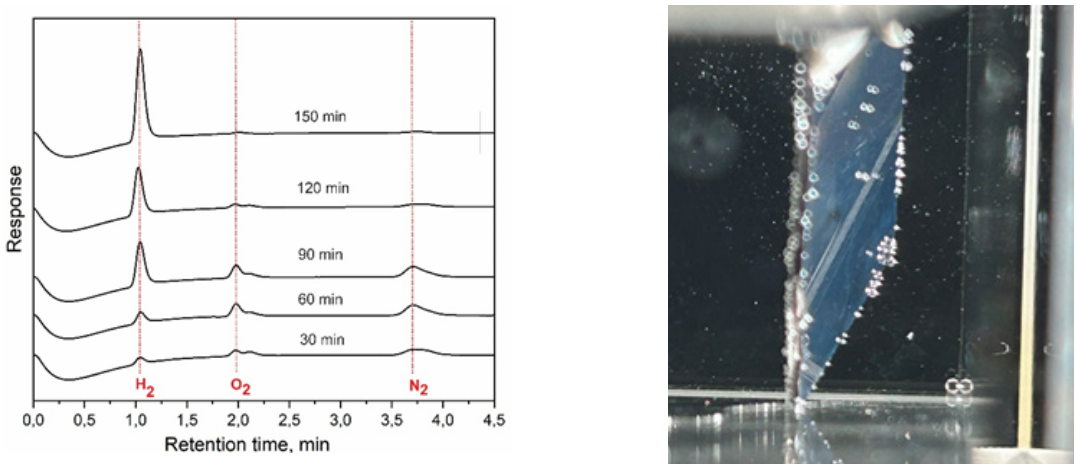


Figure 4 – I-V curves of black-Si and planar-Si photoelectrode under chopped illumination each 2 sec in 0.5M H_2SO_4 electrolyte (a), I-t curves of black-Si photoelectrode at 0V vs RHE during 60000 sec (16 h) in 0.5M H_2SO_4 electrolyte



a b

Figure 5 – Gas evolution onto the black-Si photoelectrode at -0.2V RHE in three electrode system in 0.5M H_2SO_4 electrolyte

Conclusion

In conclusion, our study demonstrates the superior performance of nanostructured silicon (b-Si) compared to planar silicon in terms of optical and photoelectrochemical properties. In photoelectrochemical water splitting experiments, black silicon exhibited a substantial improvement in photocurrent density, achieving 800 μA/cm² at 0V vs RHE, compared to 200 μA/cm² for planar silicon. Additionally, the nanostructured silicon electrode demonstrated excellent stability over 16 hours of continuous illumination. The enhanced performance of black silicon can be attributed to its increased surface area and effective light absorption, making it a promising material for solar-driven water splitting and related renewable energy applications.

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КҮН СӘУЛЕСІМЕН СУДЫ ЫДЫРАТУҒА АРНАЛҒАН НАНОҚҰРЫЛЫМДЫ КРЕМНИЙДІҢ ФОТОЭЛЕКТРОХИМИЯЛЫҚ ҚАСИЕТІ

Аңдатпа

Кремний – жер бетіндегі кең таралған және салыстырмалы түрде арзан материалдардың бірі. Оның энергетикалық жолақ аралығы 1,12 эВ болғандықтан, ол ультракүлгін, көрінетін және инфрақызыл сәулелерді тиімді жұтады. Бұл қасиеті кремнийді күн энергиясын басқа энергия көздеріне түрлендіру саласында перспективті материал етеді. Алайда тегіс кремний беттерінің жарықты шағылысу қабілеті (~25%) оның түрлендіру тиімділігін шектейді, бұл оны фотоэлектрохимиялық (РЕС) процестер үшін тиімсіз етеді. Осы мәселені шешу, атап айтқанда жарық жұтуын жақсарту, төсеніш кедергісін азайту және сутегі өндірісінің тиімділігін арттыру үшін кремнийдің бетін өңдеу арқылы наноқұрылымды кремний алуға болады. Бұл зерттеуде біз металл көмегімен химиялық өңдеу (MACE) әдісін қолдана отырып, наноқұрылымды

кремний фотоэлектродтарын жасадық. Алынған қара кремний (b-Si) электродтары жоғары жарық жұту қабілетін көрсетті, бұл фототок тығыздығының айтарлықтай жоғарылауына әкелді. Атап айтқанда, b-Si фотоэлектродтары 0 В (RHE) кернеуде 800 мкА/см² фототок тығыздығына жетті, бұл жазық кремний үшін 200 мкА/см² мәнімен салыстырғанда айтарлықтай жоғары. Сонымен қатар b-Si электродтары 16 сағат бойы үздіксіз жарықтандыру кезінде ұзақ мерзімді тұрақтылығын сақтады. Бұл нәтижелер наноқұрылымды кремнийдің күн энергиясымен көмегімен суды бөлу процестері үшін тиімді және тұрақты материал ретінде әлеуетін көрсетеді. Наноқұрылымды кремний қолдану жаңартылатын энергия көздерін дамыту және суды бөлу процестерінде маңызды рөл атқаруы мүмкін.

Тірек сөздер: наноқұрылымды кремний, суды күн энергиясының көмегімен бөлу, қара кремний, металдың көмегімен химиялық өңдеу әдісі.

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ФОТОЭЛЕКТРОХИМИЧЕСКИЕ СВОЙСТВА НАНОСТРУКТУРИРОВАННОГО КРЕМНИЯ ДЛЯ СОЛНЕЧНОГО РАЗЛОЖЕНИЯ ВОДЫ

Аннотация

Кремний, один из самых распространенных и экономически эффективных материалов на Земле, имеет значительные перспективы для применения в расщеплении воды и фотоэлектричестве благодаря своей подходящей ширине запрещенной зоны приблизительно 1,12 эВ, что позволяет поглощать ультрафиолетовый, видимый и инфракрасный свет. Однако высокая отражательная способность (~25%) плоских кремниевых пластин ограничивает его эффективность преобразования, что делает его менее

эффективным для фотоэлектрохимических (ПЭК) процессов. Наноструктурирование поверхности кремния позволяет улучшить коэффициент поглощения света, снижает его сопротивление и повышает эффективность производства водорода. В этом исследовании мы изготовили наноструктурированные кремниевые фотоэлектроды с использованием метода химического травления с использованием металла (MACE). Полученные черные кремниевые (b-Si) электроды продемонстрировали превосходные возможности поглощения света, что привело к значительному повышению плотности фототока. Примечательно, что b-Si фотоэлектроды достигли плотности фототока 800 мкА/см² при 0 В против RHE по сравнению с 200 мкА/см² для плоского кремния. Кроме того, b-Si электроды продемонстрировали превосходную долговременную стабильность при непрерывном освещении в течение 16 часов. Эти результаты подчеркивают потенциал наноструктурированного кремния как эффективного и стабильного материала для расщепления воды с помощью солнечной энергии и связанных с этим возобновляемых источников энергии.

Ключевые слова: наноструктурированный кремний, расщепление воды с помощью солнечной энергии, черный кремний, химическое травление с использованием металла.

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