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OPTIMIZATION OF THE OPTICAL PROPERTIES OF BLACK SILICON SOLAR CELL

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Black silicon (BSi) is an important texturized form of a semiconducting material used in photovoltaic solar cell technology. It is characterized by surface structuration of silicon with very low reflectance. In this paper, we study the optical properties of black silicon in the visible-near infrared wavelength range. Our work focuses on texturing the silicon surface using cryogenic etching in an inductively coupled plasma (ICP) system. The surface structure of black silicon is formed by varying several parameters of the cryo-etching process, like wafer temperature, SF_6/O_2 ratio and bias voltage. The microstructure surfaces of BSi can be formed in various shapes (Pyramids, Columns, and Cones forms). The optical properties of the micro-structures were studied by spectrophotometer measurements. The results obtained show that columnar microstructures (CMS) exhibit different texturing shapes under different plasma etching process conditions. The CMS obtained without HF chemical treatment process have a reflectance value as high as about 14%. However, the surface reflectance is reduced to less than 2% in the VIS-NIR range by processing the samples in HF solution.
Keywords: *Black silicon; Surface texturing; Vis-NIR reflectance; Solar cell; HF process*
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I. INTRODUCTION

Silicon is the second-most-abundant element on Earth, after oxygen. Its abundance and ease of processing make it a cost-effective, readily available material for various semiconductor applications. Crystalline silicon (c-Si) solar cells dominate more than 90% of the photovoltaic (PV) industry [1]. And its flat surface has a high natural reflectance around 35% in 300-1100nm spectral region [1]. Using Black silicon (BSi) as a surface texturing approach has shown promising results in improving the performance of c-Si solar cells and holds great potential for enhancing the efficiency of solar energy conversion. Black silicon (BSi) is an active candidate in the renewable energy area due to its potential applications in solar cells, its applications cover different fields such as photonic sensors and biosensors [2]. Black silicon (BSi) can be produced by several methods, such as electrochemical etching of macro porous silicon (macP-Si) [3, 4], metal assisted chemical etching (MACE) [5-6], femto-second laser [7], and inductively coupled plasma reactive ion etching (ICP-RIE) [8]. A Harvard University group has developed a process (Mazur's Method) in which Black silicon (BSi)is produced by irradiating Si with femto-second laser pulses. The reflectance of Black silicon (BSi), obtained by Mazur's process, is less than 5% and the absorbance is about 97%in the visible region [7]. Black silicon (BSi) is a surface modification of silicon where a nanoscale surface structure is formed bysilicon plasma etching. The resulting nanoscale structure provides an extremely low reflectivity. Because the Black silicon (BSi) surface nanostructure exhibits high absorption over a wide spectral range (250–2500 nm) [9], it offers an ideal solution as an Anti-reflective coating (ARC) for solar cells [9-10], as well as applications in photodetectors. The nanoscale structure may be in the form of inverted nanoscale cones (i.e. with the tip of the cones pointing upward away from the silicon surface) or a series of nanoscale pores of varying depths and diameters extending into the surface. Both types of nanoscale structures are distributed randomly over the silicon surface [11].

In this study, we used an inductively coupled plasma reactive ion etching (ICP-RIE) system. Which can be used to form Columns of different dimensions on a mask less silicon.

II. EXPERIMENT

II.1 Experimental device

Experiments and optimization settings are conducted using a commercial ICP reactor (Alcatel 601E). The source plasma is generated by an inductively coupled coil supplied by a 13.56 MHz R.F. generator. A 13.56 MHz generator powers the substrate holder electrode, allowing independent control of the bias potential. The substrate chuck is cooled with liquid nitrogen and maintained at a very low temperature (~-100°C) using resistances. To enhance the thermal conductivity between the substrate and the chuck, the wafer is electrostatically clamped to the chuck, and helium gas is injected under the wafer.

The standard cryogenic etching process uses SF_6/O_2 gas mixture to achieve deep silicon etching. For the experiments presented in this paper, the SF_6 flow rate was set to 200 sccm, and the total pressure was about 9 Pa.

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The Black silicon was obtained by the DRIE process with varying parameters at cryogenic temperature. The black silicon samples were produced on 6-inch-diameter maskless (100) silicon wafers with a thickness of 400 μm.

II.2. Black silicon formation mechanism:

Figure 1 shows the effect of O_2on the Etch profiles of maskless silicon after 10 min of process (source power 1500W, bias -50V, SF_6= 300 sccm, pressure 9 Pa, T: -100°C, Ar: 75sccm, at O_2flow rate of: 00, 30, 42, 60 sccm, respectively).

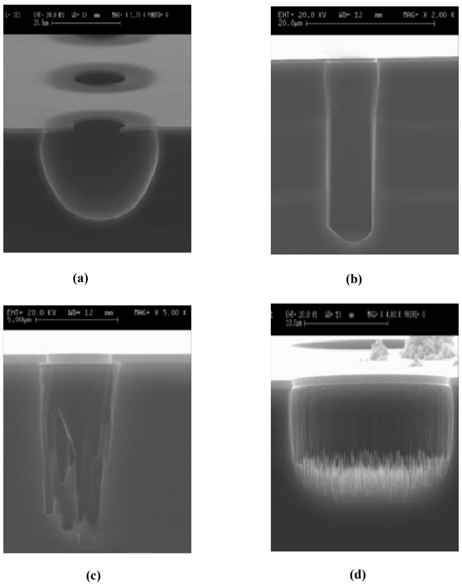

Figure 1. Etch profiles of mask-less silicon at different O_2 flow rate
(a): 00sccm, (b): 30sccm, (c): 42sccm, (d): 60sccm [13].

Black silicon, which is a grasslike structure, appears only at the bottom of the trench for high oxygen content as shown in Figure 1 (d), for an O_2/SF_6 ratio of 20%, indicating that we have reached the overpassivating regime, in this case, the increase of the oxygen flow rate tends to reinforce the passivation layer$S_iO_xF_y$, and the etching rate tends to a minimum value around 0.8 µm/min. In Figure 1 (a) dark areas correspond to the bottom of the structure and the white part corresponds to the top of the hole. [12].

The cryogenic etching process uses SF_6 / O_2 gas mixture to achieve a deep silicon etching at a very low temperature. The average diameter and height of black silicon (BSi) structures can be determined through precise measurements using advanced imaging and analysis techniques. To measure the diameter and height of black silicon structures, we first need high-resolution images of the sample surface. In our study, this is done using SEM. SEM provides a detailed view of BSi structures at micro- or nanometer scales. Structures are usually imaged from the top view to measure their base diameters or from a tilted angle to measure varying diameters along their height.

III- Results and discussion

1. Columnar Microstructures of Silicon (CMS)

The wafer temperature, O_2/SF_6 The flow rate ratio and bias voltage are the main parameters that play an important role in the formation of the black silicon. Basically, the microstructure appears in an overpassivating regime, i.e., for high O_2 / SF_6 ratios and at cryogenic temperature. Pressure process and voltage bias also play a role in its appearance [14]. The evolution of the typical microstructural dimensions (diameter and mean height) is studied as a function of wafer temperature and bias voltage. The images SEM of the textured silicon samples are shown in Figure 2.

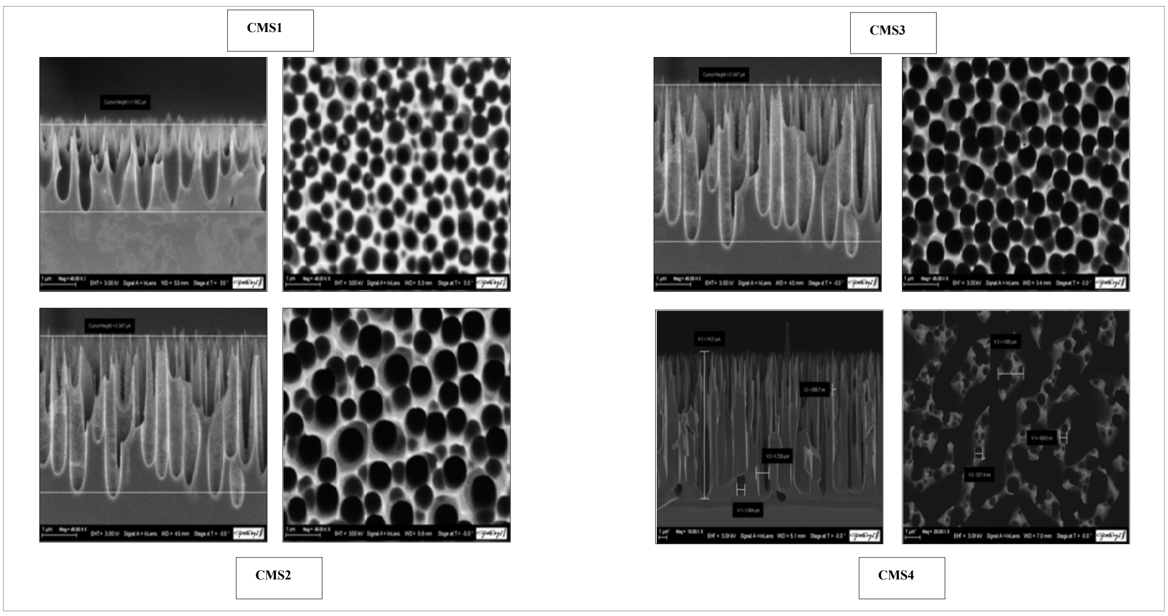

Figure 2. Image SEM with Top and Side views of samples with different
Bias voltages (CMS1: -10V, CMS2: -15V, CMS3: -20V, CMS4: -25V [13]

A. Effect of Bias voltage on columnar formation

The influence of the bias voltage on the formation of Black silicon will be studied by varying its value from-10V to -25V (Table 1) under the following fixed conditions: ICP power 1000 W, SF_6 flow rate = 200 sccm, T = -120°C, pressure = 3 Pa, T = 10 min, O_2 flow rate = 16 sccm. Figure 2 summarizes the SEM observations of these samples showing different types of BSi structure.

Table 1. Variable parameters for the cryogenic process

Samples	V Bias (V)	T(°C)
CMS 1	-10	-120
CMS 2	-15	-120
CMS 3	-20	-120
CMS 4	-25	-120
CMS 5	-20	-105
CMS 6	-20	-110
CMS 7	-20	-115

From −10V to −20V of bias voltage, the mean height slightly increases from less than 1.8μm to about 3.47μm as shown in Figure 3, at the bias voltage of -25V; a significant increase in the average column height is obtained around 6.21µm. Thus, it seems that for such bias value and beyond, the ion energy is efficient enough to avoid the silicon passivation by a $S_iO_xF_y$layer [12]. In cryogenic etching process, the bias voltage controls the energy and direction of the ions which arrive at the silicon surface. The BSi is formed for a bias voltage value between -10V and -25V. Figure 3 and Figure 4 show the morphological evolution of black silicon structures when the bias voltage varied from -10V to -25V.

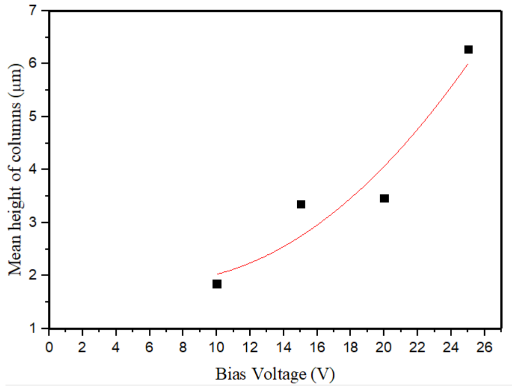

Figure 3. Mean column height versus bias voltage

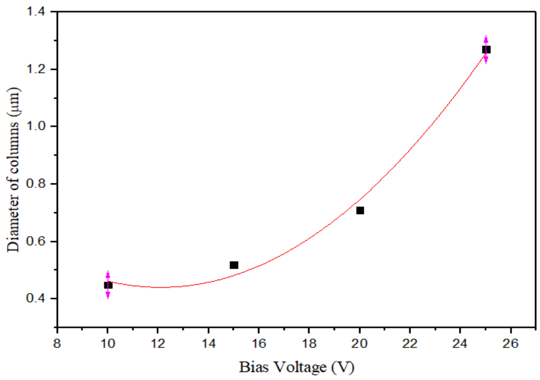

Figure 4. Mean column Diameter versus bias voltage

B. Effect of wafer temperature on column formation

We study the effect of wafer temperature on column formation under etching conditions of: power=1000W, SF_6flow rate =200sccm, O_2flow rate = 16sccm, pressure=3Pa, etching time=10 min, Bias voltage =-20V, and wafer temperature range from [-105°to-115°C] as shown on Table1. Figure 5 summarizes the SEM observations of samples showing different types of BSi structure.The formation of black silicon is very sensitive to the temperature gradient.

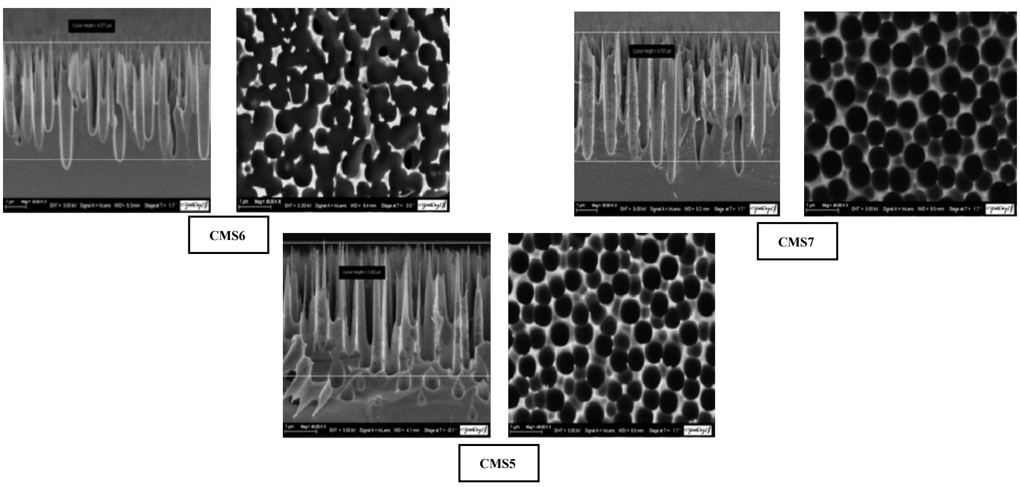

Figure 5. SEM with Top and Side view of samples with Wafer temperature (CMS5: -105°C, CM6: -110°C, CMS7: -115°C [13]

The cryogenic temperature also has very significant effects on the slope of the etching profile. For a temperature of -115°C (CMS7), we obtain a large increase of mean height column around 4.7µm as shown in Figure 6.

Figure 7 shows the morphological evolution of black silicon structures when the wafer temperature ranges from -105°C to-115°C.

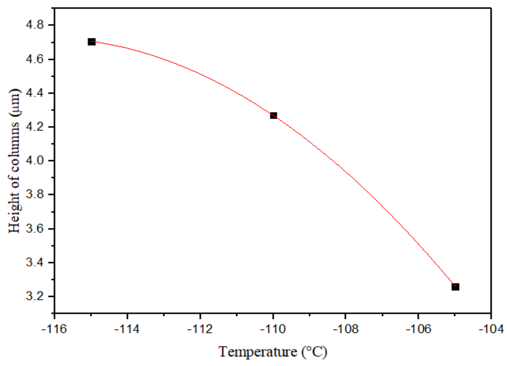

Figure 6. Column Mean height versus wafer Temperature

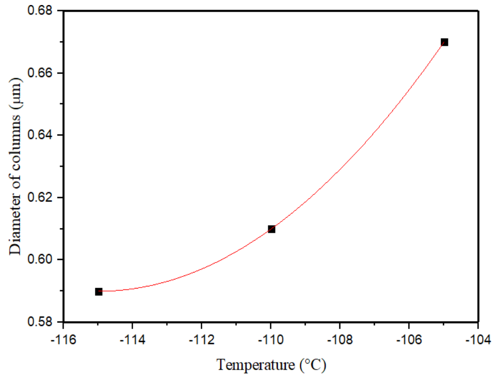

Figure 7. Column Mean diameter versus Wafer Temperature

2. OPTICAL PROPERTIES OF BLACK SILICON

We have started our work to characterize the optical properties for all black silicon samples by measuring their reflectance in the Vis-NIR range [300, 1100] nm. Spectrum acquisition is provided by the software Win ASPECT PLUS. The software Aspect Plus is windows-based and offers a variety of options for data acquisition and analysis. In our case we choose the curve which analyzes the reflectance as a function of the wavelength. The reflectance spectrum of black silicon samples is shown in Figure 8 for silicon process etching parameters of bias voltage. All reflectance measurements were performed using a Specord 210 spectrophotometer**,** without an integrating sphere. The measurements were performed using air as the reference standard, and the reported values represent the average reflectance over the **3**00-1100 nm spectral range**.**

Several samples have been studied to determine the influence of different parameters on the etching process on optical properties; certain parameters were fixed for all measurements, such as ICP power, gas pressure, and SF_6,O_2 gas flow, to 1000 W, 3 Pa, 200 sccm, and 16 sccm, respectively. The variable parameters are the bias voltage and the temperature; all these parameters are illustrated in theTable 1.

After the silicon DRIE etching, the samples are etched dipped in polypropylene beaker containing HF:$H_2O_2$with 1:5 volume ratio aqueous solution for 60s at room temperature. H_2O_2 participates in the oxidation reaction, and can be added to control of aqueous concentration of HF. Reflectance measurements have been performed in the VIS-NIR range for wavelengths between 300nm and 1100nm using the spectrophotometer SPECORD 210.

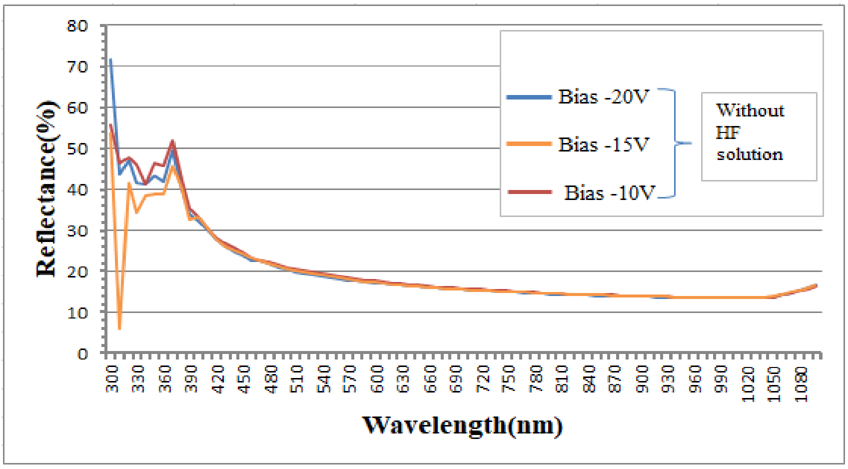

Figure 8. Reflectance of BSi at different Bias voltage value without HF solution

Figure 8 and Figure 9 shows the reflectance spectrum of the black silicon obtained by DRIE process etching before and after HF process etching. We choose three samples of BSi (CMS1, CMS2, and CMS3) for study the reflectance by variation parameters of bias voltage. In the first one, samples that are not processed in HF hydrofluoric acid bath have a decrease reflectance value of 14% from 600nm to 1000nm. The low reflectance of black silicon surface using mask-less DRIE etching is due to dense columns, which improves light trapping. [15]. Figure 8 shows the evolution of the reflectance spectrum of DRIE etched black silicon before HF etching which has almost the same curve by changing the value of bias voltage from -10V to-20V. In second step, the DRIE etched black silicon of all samples (CMS1, CMS2, and CMS3) is then etched in HF solution process with volume ratio of 1:5.

Figure 9 shows a decrease of the reflectance of the black silicon textured using HF solution process etching, the average of reflectivity is around 1% in the wavelength range of 300nm to 1100nm. After Black silicon HF etching, the reflectance of the black silicon wafers decreases dramatically with the plasma etching conditions from CMS1 to CMS3. The Black silicon wafer etched at a -20V Bias voltage in an HF solution process has the lowest reflectance compared to other Bias voltages, such as -10V and -15V, in the VIS-NIR range. The average reflectance of the samples etched in an HF solution decreases to 1% as the Bias voltage increases from -10V (CMS1) to -20V (CMS3). The decreased reflectance of black silicon is a result of its unique structure. The unique microstructure causes multiple reflections of incoming light [16]. The bias voltage used during plasma etching significantly influences the reflectance of the resulting surface; first the high Bias voltage leads to deeper, sharper, and denser nanostructures [17]. These features enhance light trapping by increasing multiple scattering and absorption, reducing reflectance. In the other side the low bias voltage produces shallow and less pronounced nanostructures. These structures may not trap light efficiently, resulting in higher reflectance.

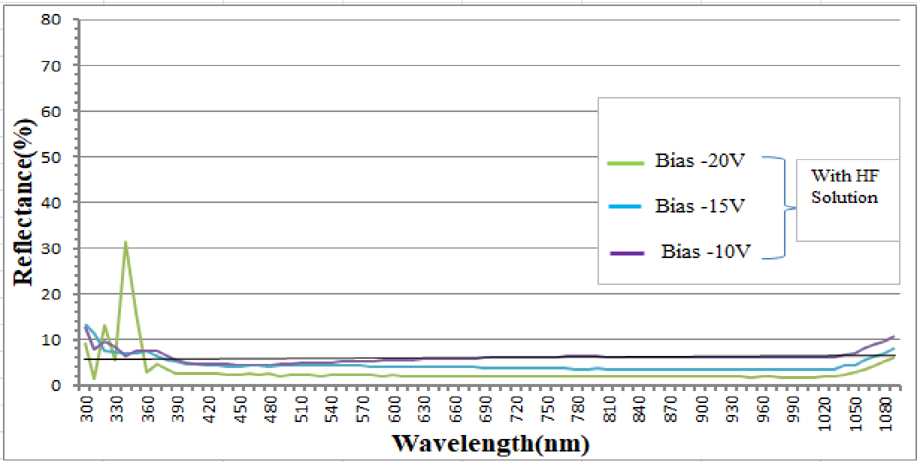

Figure 9. Reflectance of BSi at different Bias voltage value with HF solution

CONCLUSIONS

In this paper, we present a process of measuring of reflectance of black silicon. The process involves using ICP (Inductively Coupled plasma) plasma cryogenic etching and (HF/H_2O_2) process etching. We investigated the influence of various parameters such as wafer temperature and bias voltage on both of surface morphology and optical properties of the black silicon samples.

The combination of black silicon produced by plasma etching and HF/H_2O_2 treatment results in a surface texturing that significantly enhances light absorption, the nanostructures (plasma) and micropores (HF/H_2O_2) work synergistically to create a surface that traps light through multiple internal reflections, this effectively reduces reflectance and ensures that more light is absorbed by the silicon.

The results indicate that black silicon samples obtained through ICP plasma etching technique had a reflectance of approximately of 14% in the visible to near-infrared (VIS-NIR) range, however, the black silicon samples created using their proposed structure with (HF/H_2O_2) process etching exhibited superior anti-reflective and light-trapping properties, leading to a significantly lower reflectance of around of 1.75%in the same of range.

Author Contributions:
All authors contributed to the study.
N. Mekkakia conceived of the presented idea.
M. Azouza, N. Mekkakia, and R. Dussart, T. Tillocher, P. Lefaucheux carried out the experiment
M. Azouza, N. Mekkakia wrote the manuscript with support from R. Dussart.
R. Dussart author contributed to the final version of the manuscript.

Data Availability:
All the data and information mentioned in this article are related to this research.
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ОПТИМІЗАЦІЯ ОПТИЧНИХ ВЛАСТИВОСТЕЙ ЧОРНОГО КРЕМНІЄВОГО СОНЯЧНОГО ЕЛЕМЕНТА

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Чорний кремній (BSi) – це важлива текстурована форма напівпровідникового матеріалу, що використовується в технології фотоелектричних сонячних елементів. Він характеризується поверхневою структурою кремнію з дуже низьким коефіцієнтом відбиття. У цій статті ми вивчаємо оптичні властивості чорного кремнію у діапазоні довжин хвиль від видимого до ближнього інфрачервоного. Наша робота зосереджена на текстуруванні поверхні кремнію за допомогою кріогенного травлення в системі індуктивно зв'язаної плазми (ICP). Поверхнева структура чорного кремнію формується шляхом зміни кількох параметрів процесу кріотравлення, таких як температура пластини, співвідношення {SF}_6/O_2 та напруга зміщення. Мікроструктурні поверхні BSi можуть бути сформовані в різних формах (піраміди, колони та конуси). Оптичні властивості мікроструктур вивчали за допомогою спектрофотометричних вимірювань. Отримані результати показують, що стовпчасті мікроструктури (CMS) демонструють різні форми текстурування за різних умов процесу плазмового травлення. CMS, отримані без процесу хімічної обробки HF, мають значення коефіцієнта відбиття до 14%. Однак, поверхневе відбиття знижується до менш ніж 2% у діапазоні VIS-NIR шляхом обробки зразків у розчині HF.

Ключові слова: *чорний кремній; текстурування поверхні; відбивна здатність у видимому та ближньому інфрачервоному діапазонах; сонячний елемент; ВЧ-процес*