

Research Article

Change of Optical Properties of Carbon-Doped Silicon Nanostructures under the Influence of a Pulsed Electron Beam

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In the work, porous silicon with observed photoluminescence was made from a p-type silicon substrate doped with boron and crystallographic orientation (100) using the method of electrochemical etching in a solution containing $H_2(SiF_6)$ (silicon hydrofluoric acid) and ethyl alcohol. Thin carbon films were sprayed by high-frequency magnetron sputtering at room temperature onto the surface of porous silicon. The resulting carbon-doped thin films of porous silicon were irradiated on a pulsed electron booster and comparisons were made with nonirradiated films of porous silicon. To understand the effect of carbon on the properties of porous silicon films samples were analyzed by Raman spectroscopy, spectrophotometry, and scanning probe microscopy (SPM). The results of the SPM showed that the roughness of the samples increases after carbon doping on the surface of porous silicon. Thus, for the first time, experimental results were obtained on the effect of irradiation on carbon-doped porous silicon obtained in a solution containing hydrogen hexafluorosilicate $H_2(SiF_6)$ and a significant change in its optical properties was shown. The results of the study showed that irradiated samples of carbon-doped porous silicon have better photoluminescence compared to nonirradiated samples.

1. Introduction

Porous silicon (PS) is a promising material due to its high mechanical and thermal properties, its obvious compatibility with silicon microelectronics and luminescence efficiency at room temperature and low cost. The creation of modern nanoelectronics devices is realized by simplifying the technological intensity of the process, in which functional elements of the order of tens of nanometers are formed, in which the physical properties may differ significantly from the properties of the bulk material. The difference in properties is due to a greater share of the contribution of surface energy, which in particular is characterized by a discrete structure of energy levels as well as the appearing of the quantum-dimensional effect [1, 2].

Due to the possibility of creating porous structures with desired optical properties, PS is used in the manufacture of solar cells as antireflection textured coatings,

which are called “black silicon” [3–5]. Light-emitting properties arising from the effects of quantum confinement, biodegradability, and biological activity of nanostructured silicon are used in many areas of biomedical and pharmaceutical research, in particular as biomarkers and agents for targeted drug delivery, including prolonged action [6, 7].

At present, it has become possible to manufacture light-emitting structures [8], photoelectric solar energy converters [9], humidity sensors [10], electronic gas sensors [11, 12], chemical sensors [13], and other semiconductor devices based on PS layers. The principle of operation of such sensors is based on the influence of external molecules on the electronic state of the surface. In the case of porous structures, due to the high specific surface, this effect becomes more effective and the sensors are highly sensitive. Typically, such sensors detect changes in the capacitive, conductive, and luminescent properties of PS in a controlled

environment in the presence of specified molecules and chemical compounds.

To date, one of the available and inexpensive methods for obtaining porous structures is the electrochemical anodization of a crystalline silicon wafer in electrolytes containing hydrofluoric acid (HF)/silicon hydrofluoric acid ($\text{H}_2(\text{SiF}_6)$) and ethyl alcohol [14, 15].

It is known from the literature that the integration of nanomaterials and metallic nanoparticles such as gold (Au) [16], silver (Ag) [17], copper (Cu) [18], aluminum (Al) [19], and nickel (Ni) [20] into the PS matrix change its optical properties, in particular, the intensity of photoluminescence (PL) increases. In practice, there are various methods for integrating a metal or metal oxide onto the PS surface, such as electroplating [21], sputtering [22], electrochemical or electrodeless chemical deposition [23], and electron beam evaporation [24].

Doping of nanostructured silicon with carbon-containing materials is in demand from the point of view of creating wide-gap materials. In addition, the influence of the energy levels of defect centers and dopants can significantly improve the spectral dependences, both in the visible and in the infrared range, which in total will significantly increase the absorption band of photovoltaic structures [25, 26].

The thermal carbonization (TC) method can be used to stabilize the PL in PS. The purpose of this method is to replace the hydrogen endings with other, more stable species. TC, which is based on the replacement of existing Si-H terminations with Si-C varieties, can be considered a breakthrough in improving the stability of PS structures. The unique characteristics of silicon carbide (SiC), such as chemical inertness and thermal stability, are the main motivations for PS stabilization using the TC method. The formation of a SiC layer on PS is possible under the action of gaseous acetylene at high temperatures [27–29]. Despite certain advantages, there are some disadvantages of this method. First, a thin layer of oxide on the PS surface can prevent the diffusion of carbon atoms into the Si skeleton, which leads to a decrease in the coverage of the processing surface or the formation of pyrolytic carbon. Secondly, the structure and properties of carbonized PS are highly dependent on the processing temperature.

Recently, porous silicon materials have been treated with various types of irradiation, such as electron beam, γ -ray, neutron irradiation, since it is believed that irradiation can improve the properties of porous silicon. A small number of publications are devoted to studies of the structural and optical properties of irradiated porous silicon [30, 31].

Radio frequency (RF) magnetron sputtering is a convenient method for depositing uniform thin films of carbon on a PS surface without structural defects. This method can be very easily controlled by changing physical parameters such as substrate temperature, operating pressure, spray power, and deposition time. These parameters can be optimized individually as they are independent of each other [32].

Many previous studies have used various transition metals and metal oxides to improve the optical properties of PS. The aim of our research was to improve the optical

properties of PS obtained by electrochemical anodization of crystalline silicon in an electrolyte containing silicon, hydrofluoric acid ($\text{H}_2(\text{SiF}_6)$) and ethyl alcohol. For this purpose, thin films of carbon were deposited using RF magnetron sputtering at room temperature on the PS surface, then the resulting samples were irradiated in a pulsed electron booster. The effect of radiation on the optical properties and morphology of the PS samples obtained was investigated.

2. Materials and Methods

For the manufacture of PS, monocrystal p-type silicon substrates doped with boron with a phosphorus concentration of 10^{15} cm^{-3} and a crystallographic orientation (100) were used.

Before obtaining PS, silicon wafers were degreased in hydrofluoric acid, washed in deionized water, then etched for 10 minutes in a mixture of $\text{H}_2(\text{SiF}_6)$ and thoroughly washed in deionized water. PS samples were obtained at anodizing current densities $J = 20 \text{ mA/cm}^2$ and etching duration $t = 10$ minutes, the power supply voltage was 10 V. The etching process was carried out in an electrolyte containing silicon hydrofluoric acid ($\text{H}_2(\text{SiF}_6)$) and ethyl alcohol in the ratios 3 : 1. Ethyl alcohol is important for wetting the PS surface and also improves the lateral uniformity of its substrate. To determine the reproducibility, 3 groups of samples were prepared. As in the case of obtaining porous silicon in a traditional solution containing hydrofluoric acid and ethyl alcohol, the level of reproducibility is quite high.

Carbon films were deposited onto the surface of the PS obtained by a DC magnetron using a Vacuum Universal Post-5 setup. A magnetron cooled by water with a graphite target was placed in the chamber. The distance between the substrate and the graphite target is 40 mm. A DC power supply with a negative electrode was connected to the magnetron cathode. The voltage and current between the anode and cathode were 500 V and 100 mA, respectively. The spraying time was 20 minutes. The flow of pure Ar (99.999%) was used as the working gas. The Ar flow rate was $6 \text{ cm}^3/\text{min}$ and was controlled by using an MCV-500SCCM gas flow regulator.

The resulting carbon-doped PS thin films were irradiated on a TEA 500 pulsed electron booster with a dose rate of 5 Gy/min at a beam voltage of 20 kV and a beam current of 1 mA. The duration of exposure was 5 minutes.

The group of samples that passed all stages of processing showed good reproducibility. The paper presents the average values of the experimental data.

3. Results and Discussion

The band gap of PS increases due to the presence of nanosized silicon clusters that form near the pore walls, and this is a clear evidence of photoluminescence (PL) in PS. The PL spectra were measured at room temperature using an NT-MDT NtegraSpectra spectrometer under excitation with a 20 mW laser at a wavelength of 477 nm. Figure 1 shows the PL spectra for two types of PS samples. As can be seen from

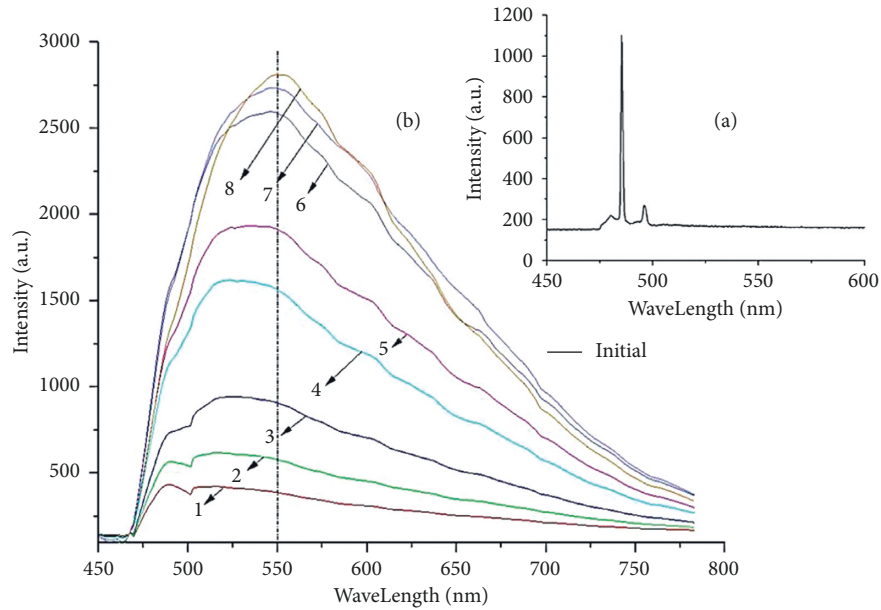


FIGURE 1: Photoluminescence spectra of PS samples obtained at $J = 20 \text{ mA/cm}^2$, $t = 10 \text{ min}$, and $U = 10 \text{ V}$. (a) Initial PS. (b) Irradiated PS doped with carbon at different delay times of the moment of registration relative to the laser pulse maximum t_d , μs : 0 (1), 5 (2), 10 (3), 15 (4), 20 (5), 25 (6), 30 (7), and 35 (8) μs .

Figure 1(a), the PL spectra of the initial nanoporous silicon were typical for this material. Figure 1(b) shows the PL spectra of irradiated PS doped with carbon at various delay times of the moment of registration. With an increase in the delay time of the moment of detection relative to the laser pulse maximum t_d from 0 to 35 μs , the maximum of time-resolved PL spectra shifts to the long wavelength region, being located in the wavelength range of 550–600 nm. The observed dynamics of changes in the time-resolved PL spectra in the microsecond range can be explained by the quantum size effect in a system of nanocrystallites of different sizes, as well as by the influence of localized states in the PS band gap. In addition, such a shift is possible as a result of the size quantization effect at sufficiently small sizes of silicon carbide (SiC) crystallites formed. The emission maxima of the sample in Figure 1(b) show a noticeable peak at 550 nm, which corresponds to a photon energy of $\sim 2.2 \text{ eV}$. Such intense behavior of the PL of the prepared porous sample is the result of quantum confinement of charge carriers in nanocrystals. Based on the above experimental data, it can be argued that PS irradiation leads to an increase in the PL intensity and a shift of its maximum to the long wavelength region of the spectrum. The presented data indicate a higher radiation resistance of PS doped with carbon compared to the original PS.

The reason for the increased radiation resistance of PS is its extremely developed surface, which can act as a region of effective sink and subsequent annihilation of radiation defects. In addition, when high-energy ions interact with elements of a porous structure, energy can be transferred not only to individual atoms but also to clusters or parts of silicon nanocrystals. Such a mechanism for receiving energy is possible due to the change in the phonon spectrum in nanometer silicon structures. The energy received by groups

of atoms in PS nanostructures is less than the energy received by individual atoms, which reduces the destructive effect of the ion beam. Thus, radiating PS optoelectronic structures will have a high radiation resistance.

It is important to note that for the first time experimental results were presented on the effect of irradiation on carbon-doped porous silicon obtained in a solution containing hydrogen hexafluorosilicate $\text{H}_2(\text{SiF}_6)$ and a significant change in the photoluminescence spectra was shown.

The Raman spectra of the obtained samples were recorded using an NT-MDT-Ntegra Spectra spectrometer and are shown in Figure 2. Raman spectroscopy is known to be a nondestructive method for characterizing changes in crystallinity and quantum size effects in silicon microstructures. Most studies of Raman spectroscopy usually focus on the c-Si peak at 520 cm^{-1} associated with Si–Si first-order optical phonon vibration modes. The shape of this peak for PS irradiated with doped carbon differs from bulk Si. There are various factors contributing to the decrease or expansion of the peak of the Raman spectrum of c-Si. Such factors include crystallinity, degree of doping, amorphization, dimension, temperature, and dissolution of the material [33, 34]. It is important to note that when analyzing the Raman spectra of silicon micro- and nanostructures, one also pays attention to the presence of another visible broad peak from 900 to 1000 cm^{-1} for PS doped with carbon. The presence of these peaks is associated with a second-order transverse acoustic system at a transverse optical point at point L for the 900 cm^{-1} peak. The Raman spectra of samples of irradiated PS doped with carbon demonstrate two intense peaks at 521 cm^{-1} and 970 cm^{-1} , which are characteristic of a c-Si monocrystal (Figure 2). These two peaks are superimposed on similar peaks observed in PS and PS doped with carbon. The Raman spectra of PS and PS doped with carbon

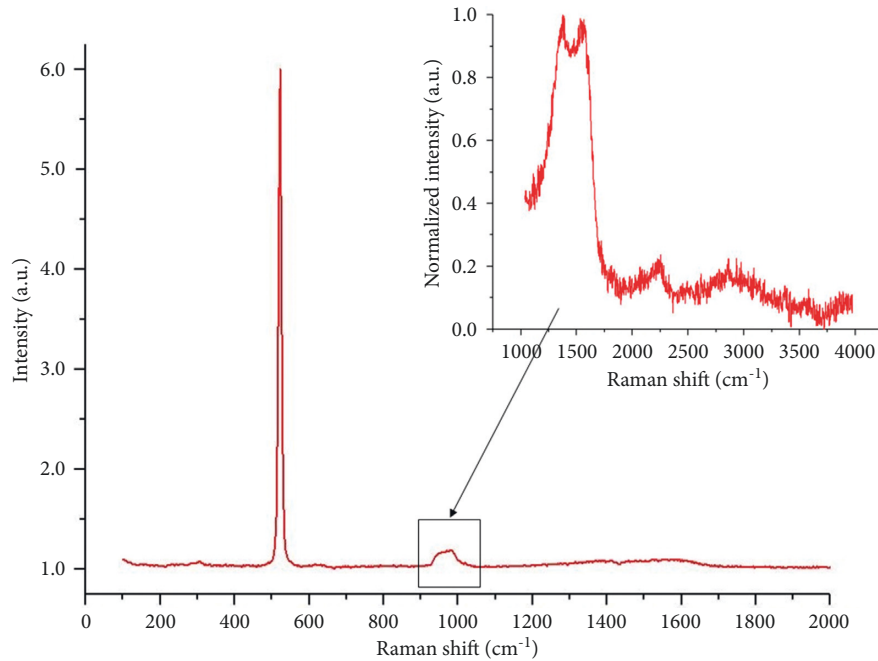


FIGURE 2: Raman spectra of irradiated PC doped with carbon.

show an increased intensity of peaks in the region of $950:1000\text{ cm}^{-1}$ (two-phonon scattering on lateral acoustic modes). This observation indicates a decrease in the size of nc-Si nanocrystallites in PS and carbon-doped PS samples, which causes more intense phonon scattering on these nanocrystals.

The reflection spectra for all groups of samples were obtained on a Shimadzu UV-3600 spectrophotometer in the wavelength range from 200 nm to 800 nm and are presented in Figure 3. From a comparative analysis of the reflection spectra, it follows that the total reflection of carbon-doped PS samples that were irradiated is significantly lower than the characteristic original substrate. The average reflection coefficient in the ultraviolet region of the spectrum for the original PS substrate is 12%, and in the visible region of the spectrum 18%, when applying carbon nanoparticles, the average reflection value in the ultraviolet region of the spectrum decreases to 9%, and in the visible region to 12%.

It should be noted that the total reflection in the ultraviolet region of the spectrum for all PS samples remains low, and in the visible region, an increase is observed due to an increase in the diffuse reflection component. This is due to the effect of light localization due to multiple reflection from PS walls and further absorption in the structure. PS irradiation did not affect the position of the peak of the samples doped with carbon, but only changed the intensity of this peak. The lowest effective reflectivity was recorded for the sample of irradiated PS doped with carbon, which indicates that the reflection decreased during irradiation. The high light capture ability and low reflectivity of the carbon-doped PS sample that were irradiated, which is due to the increased surface roughness and porosity of this sample, is the main motivation for the manufacture of photodetectors based on this substrate.

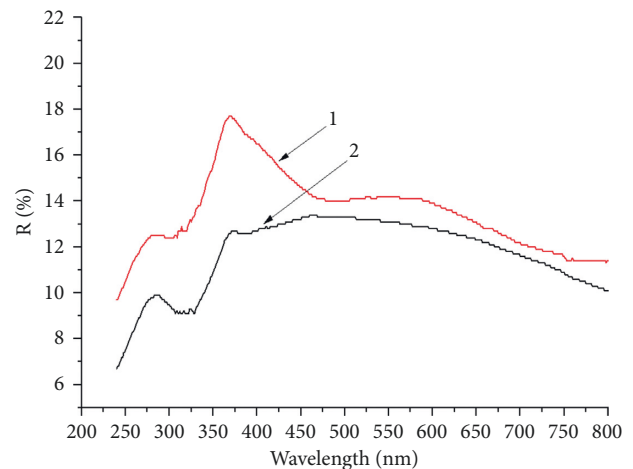


FIGURE 3: Spectral dependences of the reflection coefficient of PS samples: (1) initial PS; (2) irradiated PS doped with carbon.

The morphology of the film surface of the carbon-doped PS sample that was irradiated was studied using an NT-MDT NtegraTherma scanning probe microscope (SPM). The results of the surface morphology study are presented in Figures 4(a) and 4(b).

The sectional profile along the center line is shown in Figure 4(c). According to the 3D image, a strong inhomogeneity is observed on the surface of the sample (Figure 4(b)). It can be seen from this image that PS has a columnar structure with dense pores, in which irregular and randomly distributed nanocrystalline silicon columns and voids are visible throughout the surface. According to the 2D image, the approximate degree of porosity is 80%, and the crystallite height reaches 160 nm. If we assume that carbon

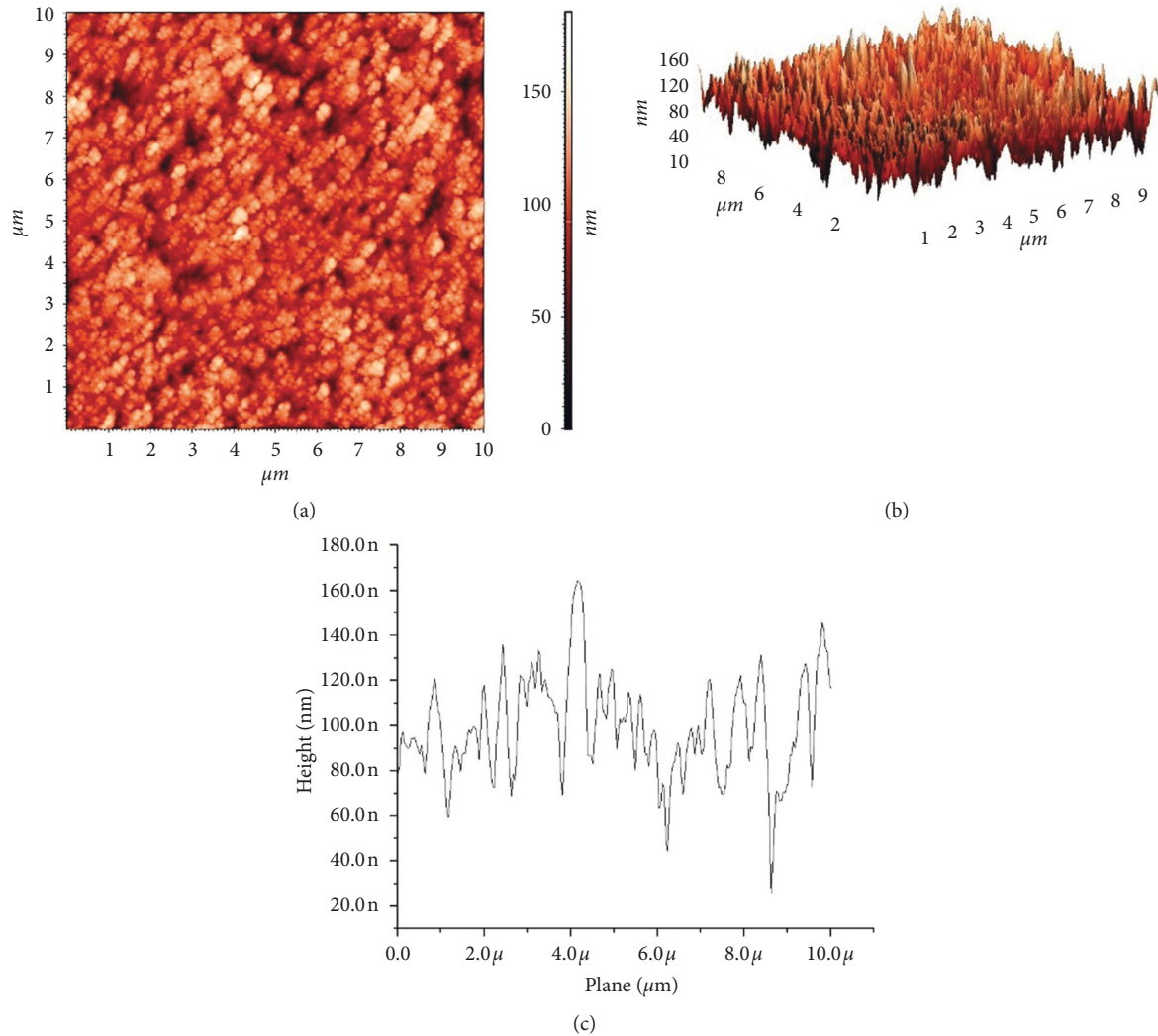


FIGURE 4: SPM images of samples of irradiated PS doped with carbon: (a) 2D images; (b) 3D images; (c) profiles of the cross sections along the central lines.

atoms form a uniform Si-C layer over the entire surface of the porous structure, the thickness of the carbon layer can be estimated at 5 nm. This insignificant thickness of the carbon layer somewhat reduced the specific surface area of the porous substrate, which does not affect the PS morphology. The presence of an ultrathin Si-C layer on the surface prevents the surface diffusion of silicon atoms, even if the surface area changes slightly. Therefore, this Si-C structure can act as a protective layer for silicon walls during electron irradiation processes and prevent serious changes in the PS structure from occurring.

4. Conclusion

In conclusion, groups of porous silicon films were obtained by electrochemical etching of p-type monocrystal silicon in an electrolyte containing ($\text{H}_2(\text{SiF}_6)$) and ethanol in a ratio of 3:1. After that, carbon films were sputtered onto the PS surface at room temperature by magnetron sputtering. The resulting thin films of PS doped with carbon were irradiated

in a pulsed electron booster and comparisons were made with unirradiated films of porous silicon. The high-energy electron irradiation changes the resistivity of the plate along the trajectory. This can change the structural and optical properties of porous silicon. From a comparative analysis of the reflection spectra, it follows that the total reflection of the carbon-doped PS samples that were irradiated is significantly lower than the characteristics of the original substrate. The obtained new experimental data revealed that, PS irradiation did not affect the position of the peak of carbon-doped samples, but only changed the intensity of this peak and leads to a decrease in the reflectance of the visible region of the spectrum from 18% to 12% for a wavelength of 375 nm. It is established that PS irradiation leads to an increase in the photoluminescence intensity and a shift of its maximum to the long wavelength region of the spectrum. The maximum intensity is achieved after irradiation of photoluminescence samples at 550 nm, which corresponds to a photon energy of ~ 2.2 eV. The results of the study of surface morphology revealed that PS doped with carbon has

a columnar structure with dense pores, in which irregular and randomly distributed nanocrystalline silicon columns and voids are visible over the entire surface. The carbon structure on the PS surface can act as a protective layer for silicon walls during electron irradiation processes and prevent serious changes in the PS structure from occurring.

Data Availability

The data obtained as part of the work are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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