Influence of current density, anodization time and illumination on the thickness of porous silicon in wafers with built-in p-n junction and its photoluminescence

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Abstract

The formation of a ***porous silicon*** (**PS**) layer in a thin p-type layer epitaxially grown on n-type silicon at *two anodizing current densities* and different anodizing times is studied and a comparison is made of transverse cleavages, surface morphology, reflection spectra, and photoluminescence spectra. The *minimum duration* of anodizing (**15** and **10 minutes**) at current densities of **10 mA/cm²** and **20 mA/cm²**, at which a single-layer PS structure is formed, is established. With an *increase* in the anodization time, regardless of the current density, a ***two-layer structure*** is formed with an ***internal tree-like porous silicon layer***, whose contribution to **photoluminescence** is ***minimal***, and the reflection coefficient ***drops strongly*** due to irretrievable losses in the ***porous tree-like layer***.

Introduction

For anodizing n-type silicon wafers, regardless of its electrical conductivity, it is necessary to ensure the generation of holes, which are minority carriers. This can be done in two ways [1]: (1) by applying a critical electric field to effect electrical breakdown, and (2) by illuminating with radiation sufficient to generate holes due to the photoelectric effect. It is known that IR illumination from the reverse side of the substrate provides the generation of electron-hole phases, their separation, and the diffusion of holes to the front side of the substrate, which is under the action of a negative potential, which leads to the formation of macroporous structures during anodization [1,2]. However, under conditions of illumination from the front side of the silicon substrate [3], depending on the parameters of anodization and the degree of its doping, the formation of a two-layer structure was observed, consisting of a thin nanoporous layer and a thicker macroporous layer.

However, complex studies of anodization in silicon with a built-in p-n junction, to our knowledge, have not been previously carried out.

Experimental

For anodizing, single-crystal silicon Si(100) wafers of n+-type conductivity with an epitaxial layer of p-type conductivity were used. Layers of porous silicon (PS) were formed at two anodizing current densities (**10** and **20 mA/cm^2**) and etching durations from **10** to **30 minutes**, as well as using illumination with a **150 W** tungsten lamp during the anodizing process.

A home-made Teflon attachment with a platinum wire cathode was used for anodizing and a copper anode, which was pressed through a layer of conductive silver paste to the reverse side of the silicon sample with the burnt ***Au-Sb*** contact. The edges of the front surface of the sample with an area of up to **1 cm^2** were protected with a special varnish. After anodizing, the samples were washed in deionized water and dried in a flow of dry nitrogen.

After mechanical removal of varnish residues and wiping with isopropyl alcohol, a porous silicon layer was studied by optical reflectance and photoluminescent (**PL**) spectroscopies, as well as by scanning electron microscopy (**SEM**), including cross-section images.

Results and Discussion

10 mA/cm^2

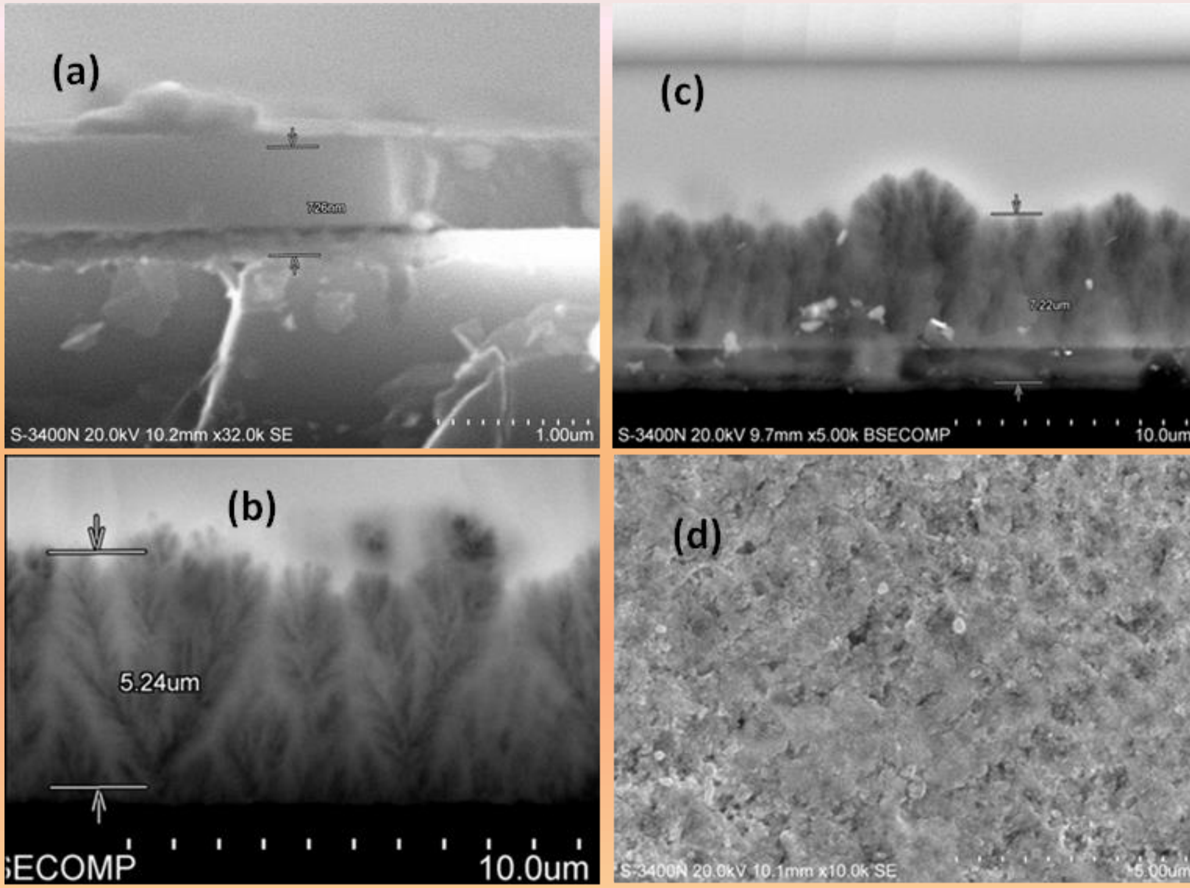
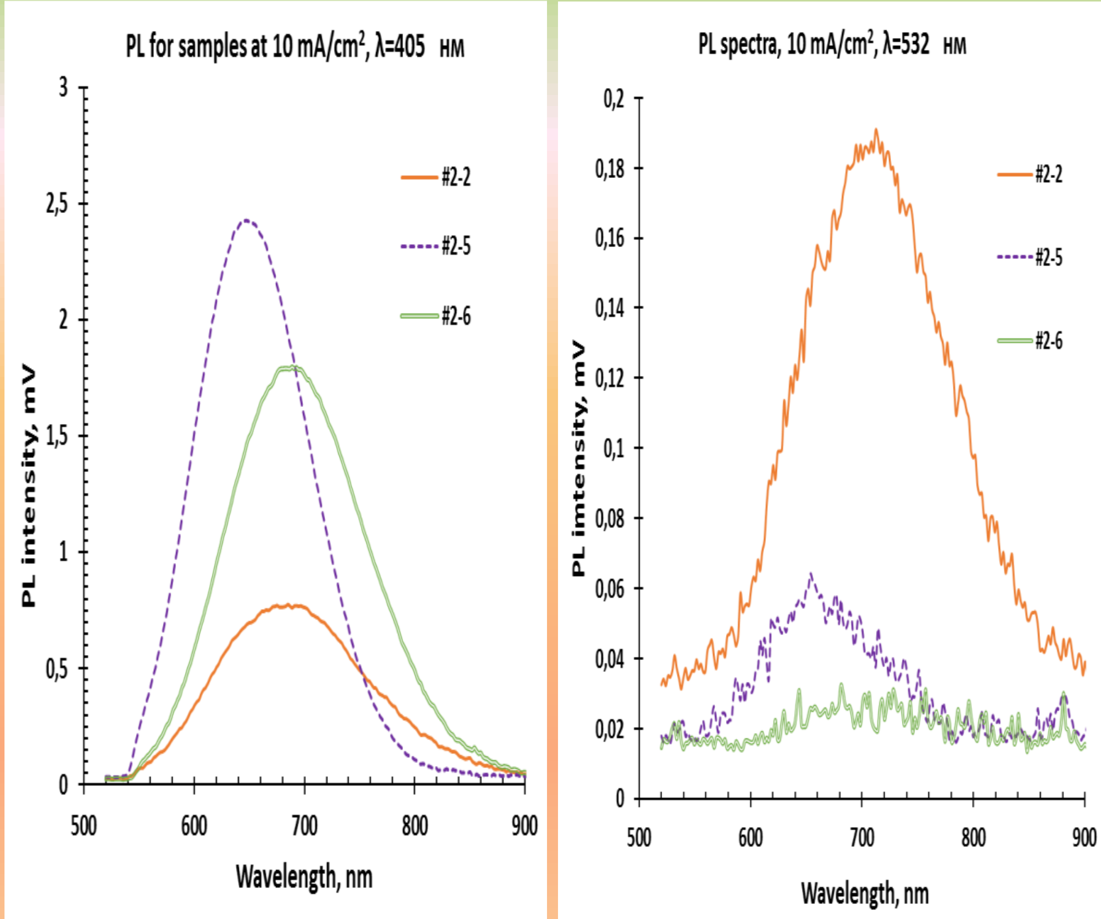


Fig. 1. SEM cross-section images of porous silicon (PS) layers on Si substrate with p-n junction formed at anodizing current density of 10 mA/cm^2 and different anodizing times: (a) 15 minutes (sample #2-5); (b) 25 minutes (sample #2-6) and (c) 30 minutes (sample #2-2). Surface image of PS layer (sample #2-6) after 25 minutes of anodizing (d)

When anodizing with a current density of **10 mA/cm^2** for up to **15 minutes**, the formation of a **relatively homogeneous PS layer** with a low density of punctures is observed. The thickness of PS layer is about 0.72 micron (Fig. 1 a). Increasing the anodization time to **25 minutes** and then to **30 minutes** led to the formation of a ***PC tree structure*** inside silicon with an increase in the **total PC thickness** from **5.2 µm** to **7.2 µm** (Fig. 1b, c). At the same time, a layer with *small punctures* and a **developed structure** was formed on the surface of the PS layer (Fig. 1в).

The **photoluminescence (PL) spectra** from the formed samples with porous silicon were studied at **room temperature** and two laser lines: **λ=405 nm** and **λ=532 nm**.



The maximum intensity of the PL spectrum at **λ=405 nm** was observed for the PC layer with an anodizing time of **15 minutes** (sample 2-5) (Fig. 2 a), that indicates its high porosity [**3**] and the generation of the **PL signal** only in a thin PS layer. With an increase in the anodizing time (samples **#2-6** and **#2-2**), the ***PL spectra decreased in intensity*** and **red shifted**. With an increase in the wavelength **(λ=532 nm**, Fig. 2 b)) and hence the depth of penetration into the PS layer, the intensity of the PL spectra ***strongly decreased*** (Fig. 2b). At the same time, the maximum intensity and shift to the red side of the spectrum was shown by a sample with an anodizing time of 30 minutes. This indicates the main contribution to the PL spectrum from the near-surface PS layer.

Fig. 2. Photoluminescence (PL) spectra of PS layers at different laser wavelengths: (a) 405 nm and (b) 532 nm for samples formed at anodizing current density of 10 mA/cm² and different anodizing times: *15 minutes* (sample #2-5), *25 minutes* (sample #2-6) and *30 minutes* (sample #2-2)

Registration of reflectance spectra in the UV-VIS range showed (Fig. 3) that in layers with a ***tree-like structure***, a sharp decrease in reflection is observed over the entire range, which indicates **an increase** in ***irretrievable light losses*** in such layers. **Noticeable interference features** in the **reflection spectra** (Fig. 3) for samples **#2-2** and **#2-6** indicate that the **PC layer** remains sufficiently flat. At the same time, the high reflectance for sample **#2-5** (***15 minutes***) corresponds to the preservation of the ***flat surface*** of the **PS layer** with a *high porosity* **[3]** and ***small sizes of Si nanocrystals***, taking into account the noticeable *blue shift of the PL spectrum* (Fig. 1a).

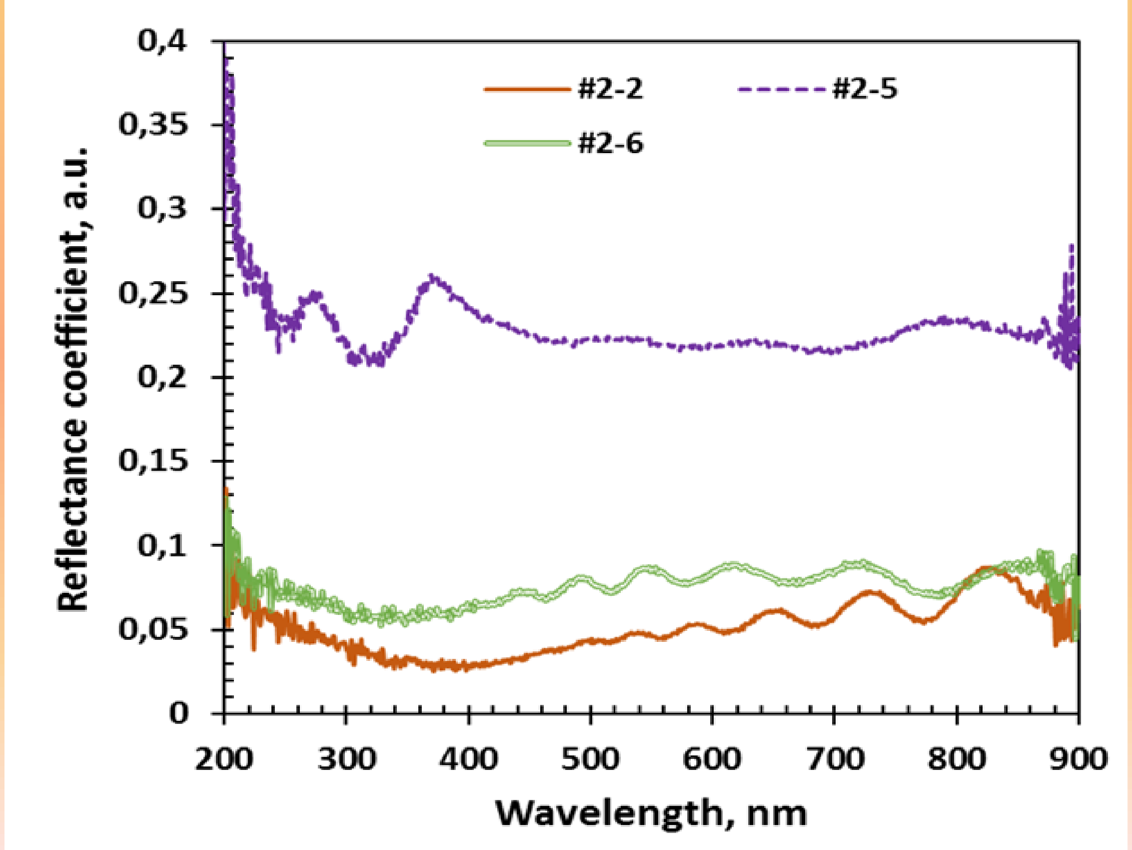


Fig. 3. Reflection spectra of PS layers formed at anodizing current density of **10 mA/cm2** and different anodizing times: ***15 minutes*** (sample **#2-5**), ***25 minutes*** (sample **#2-6**) and ***30 minutes*** (sample **#2-2**)

20 mA/cm^2

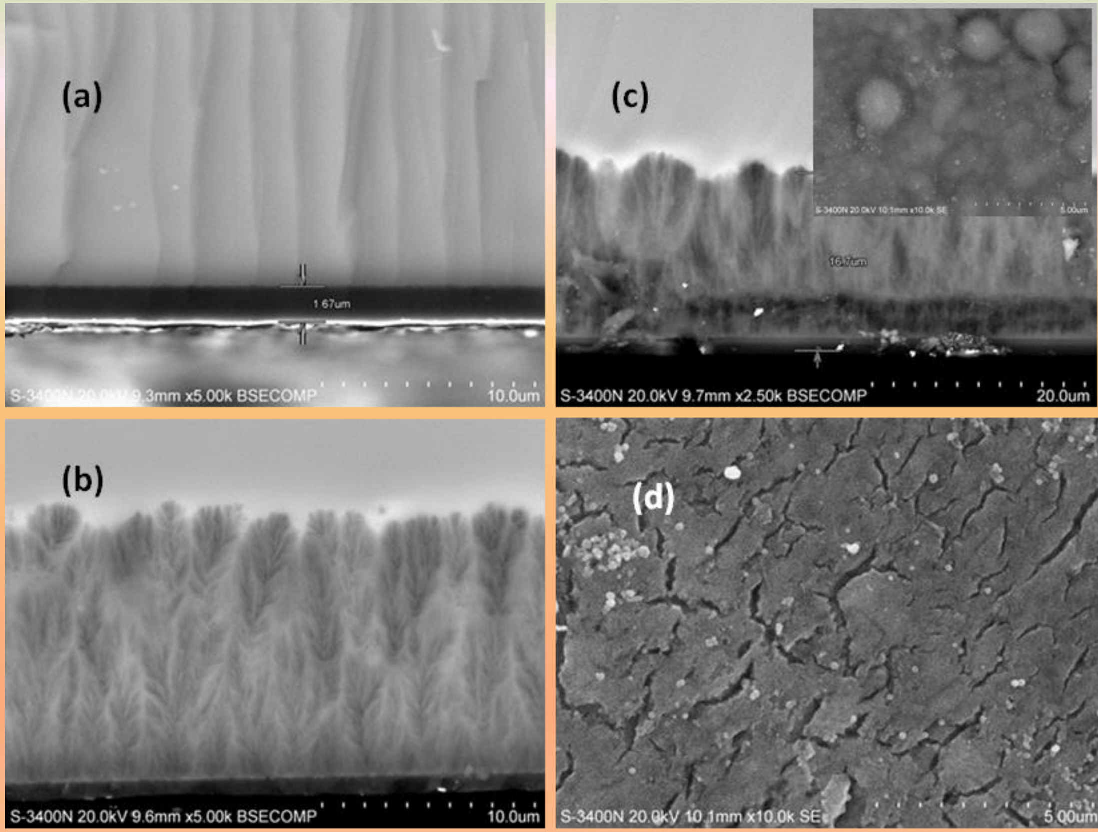
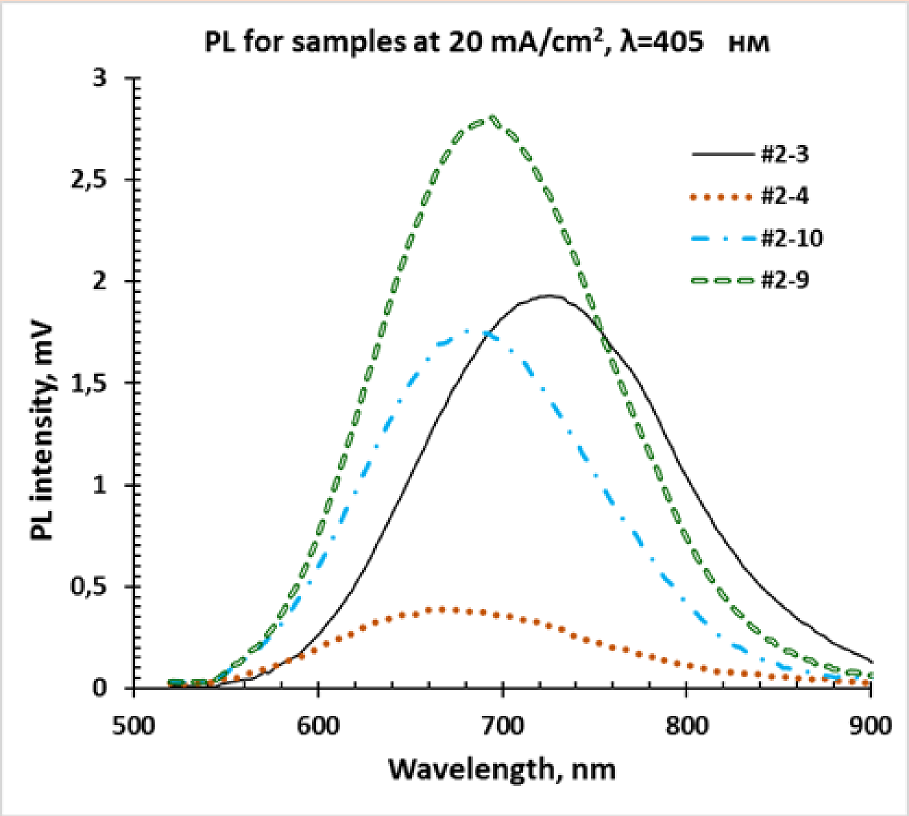


Fig. 4. SEM cross-section images of porous silicon (**PS**) layers on Si substrate with ***p-n junction*** formed at ***anodizing current density*** of **20 mA/cm^2** and ***different anodizing times***: (a) ***10 minutes*** (sample **#2-3**); (b) ***15 minutes*** (sample **#2-10**) and (c) ***30 minutes*** (sample **#2-4**). Surface image of PS layer (sample **#2-10**) after ***15 minutes*** of anodizing (**d**). On the ***insert of image*** (b) the sample's surface is shown.

An increase in the anodization current density to **20 mA/cm^2** led to the formation of a ***two-layer PS structure***, which includes a ***thin homogeneous PS layer*** and then a ***tree-like structure***. The thickness of the entire PS layer increased with the etching time (***10, 15, 30 min***) from **1.67 µm** to **16.7 µm**.

The ***position of the maximum in the PL spectra*** practically did not depend on the ***anodizing time***, and the ***intensity of the PL*** peak **decreased** with **increasing** ***anodizing time***, which is confirmed by a ***decrease in the PL intensity*** upon going from **λ=405 nm** to **λ=532 nm** and **PL localization** mainly in the ***upper PS layer***, but with variable porosity. Porosity ***turned out to be maximum*** for the **minimum** of ***anodizing time*** (see samples **#2-**3 and **#2-9** with ***10 minutes of anodizing time***).



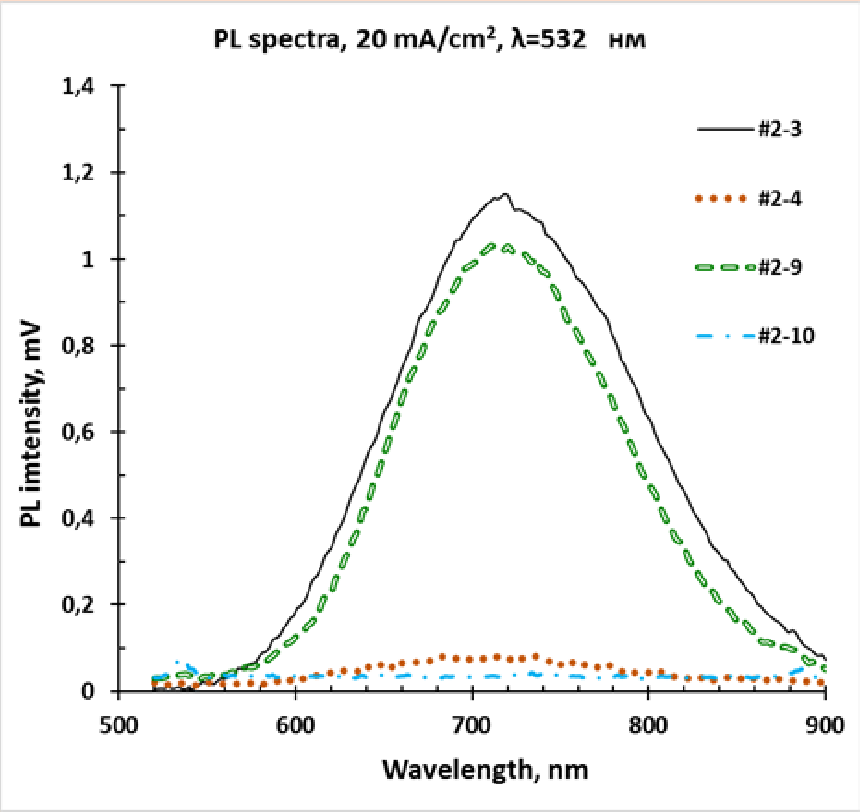


Fig. 5. Photoluminescence (PL) spectra of PS layers at different laser wavelengths: (a) **λ=405 nm** and (b) **λ=532 nm** for samples formed at anodizing current density of **20 mA/cm^2** and different anodizing times: ***10 minutes*** (samples **#2-3** and **#2-9**), ***15 minutes*** (sample **#2-10**) and ***30 minutes*** (sample **#2-4**).

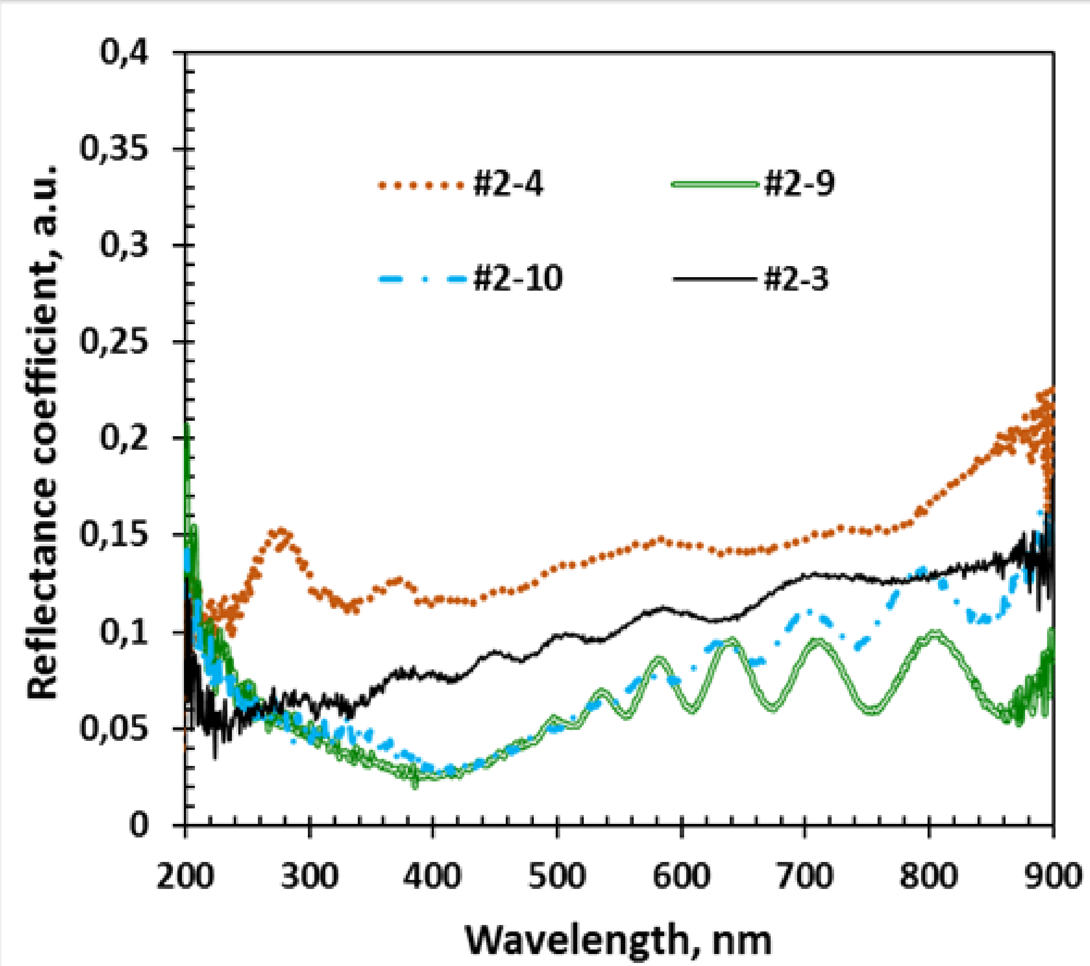


Fig. 6. Reflection spectra of PS layers formed at anodizing current density of 20 mA/cm² and different anodizing times: 10 minutes (samples #2-3 and #2-9), 15 minutes (sample #2-10) and 30 minutes (sample #2-4)

The ***reflection spectra*** of the samples of the ***second series*** with an anodizing current of **20 mA/cm²** showed that in the samples with the ***maximum PL intensity*** (**#2-3** and **#2-9**) with a ***minimum anodizing time*** (***10 minutes***), intense ***interference peaks*** are observed (Fig. 6), despite the *small thickness of the upper PS layer* (Fig. 4a). This means that a certain contribution is made by the ***inner layer with a tree structure*** and different porosity. In samples **#2-4** and **#2-10** with a **greater thickness** of the ***inner tree-like layer***, the ***upper flat layer with low porosity*** is retained, which determines a ***greater reflection coefficient***. This is confirmed by the presence of peaks from **single crystal silicon** at wavelengths of **270 nm** and **370 nm**.

Conclusions

The effect of anodizing modes (***anodizing current*** and ***duration***) on the formation of porous silicon layers in a Si-p/Si-n epitaxial structure under conditions of ***white light illumination*** has been studied. It has been established that at ***short anodization times*** of ***10 minutes*** at **10 mA/cm^2** and ***15 minutes*** at **20 mA/cm^2**, an ***upper porous layer*** with a ***noticeable porosity*** is formed, which ensures **strong photoluminescence (PL)**, which is ***more pronounced*** at a laser excitation length of **λ=405 nm** and **decreases noticeably** at **λ = 532 nm**. With an **increase** in ***anodizing time*** (***20-30 minutes***), ***a tree-like porous layer*** is formed inside under the first layer of PS, the ***thickness*** of which ***increases*** with an ***increase*** in ***anodizing time*** and ***anodizing current density***. The **photoluminescence** in the ***double porous structure*** **decreases** with an **increase** in the ***anodizing time***. In this case, the **PL intensity** from ***thick tree-like layers*** is minimal at a laser radiation length λ=532 **nm**. The **reflection coefficient** in samples with a *double porous structure* becomes ***less than 0.1*** if the *upper layer* of the PS is highly porous and does not retain the contribution from single-crystal silicon.

References

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