

VARIABLE ANGLE AND SPECTRAL ELLIPSOMETRY STUDY OF POROUS SILICON: INTERNAL STRUCTURE OF THE LAYERS

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For a set of porous silicon on monocrystalline substrate samples, variable-angle and spectral ellipsometry has been carried out within $40\ldots 80^\circ$ angle of incidence range (at 625 nm), and at the angles of 60° , 65° and 70° (within 1000...2000 nm range of wavelength). Angular dependencies of ellipsometric parameters were analysed according to several models of the reflective structure: 1) large-thickness single layer of porous material (which acts as semi-infinite medium in comparison to the wavelength); 2) two-layer model incorporating the same structure, just with additional finite layer on top of it. Experimental curves demonstrate better agreement namely with this two-layer model. Refractive indices of the media incorporated in the models were found, as well as the thickness of top finite layer. It was found that the porosity of the samples tends to be slightly higher near the surface than in the depth. Presence of surface layers of about 1 and 4 μm thickness was revealed by spectral ellipsometry and scanning electron microscopy on a few of the samples. These layers demonstrate blurred boundary with the underlying porous material.

Keywords: Porous silicon, spectral ellipsometry, thin films.

1. INTRODUCTION

Porous silicon (PS) is usually obtained by monocrystalline silicon etching in a hydrofluoric acid-based electrolyte at relatively low current densities. As a result of electrochemical reactions occurring within

the material, a region containing more or less branched pore system is formed. The pores are predominantly oriented along electrical current direction, in particular, normally to the surface of the sample. The

overall depth of this region can amount from a few units to a few tens of micrometer [1]–[2], depending on the conditions of electrochemical process technology.

Works [3]–[7] contain certain information on internal structure of porous silicon layers, which turns out to be complex in many cases, dependently on reaction conditions.

During continuous storage of porous silicon in air atmosphere, its near-surface region (meaning pore walls) can partly or

completely oxidize, which results in surface film of a few tens or even hundreds nanometer effective thickness. Properties of such films were studied in [8]–[10].

The aim of this work is to investigate the deep structure of porous silicon layers, including determination of their optical properties. Studies of porous silicon remain relevant today, in terms of its possible applications in sensors, polarizing elements, energy storage devices, and medico-biological area [11]–[14].

2. SAMPLE DESCRIPTION

Porous silicon layers were obtained by monocrystalline silicon (100 crystal lattice orientation, with 0.5 arcminute precision) electrochemical etching. The initial material had 0.01–0.02 $\Omega\cdot\text{cm}$ resistivity, conductivity of p-type due to boron doping, 525 μm wafer thickness and 100 mm diameter. The top side of the wafers had been polished during manufacturing.

The electrolyte was composed of 1:2 volumes of 48 % HF and ethyl alcohol $\text{C}_2\text{H}_5\text{OH}$ for Sample 5 (see below), and for the rest of the samples – the same with 1:1 volume ratio. Porosity of the layer formed was varied by current density, and the depth of material modification – by etch-

ing time. Current density has the impact on porous layer depth as well – the more current density, the faster etching goes. Resulting porosity was determined by gravimetric method. After the etching process, the wafers were rinsed three times in alcohol and distilled water. Technologically, according to preparation conditions, our material can be classified as mesoporous silicon type with 7–20 nm pore diameter. A set of five samples with different porosity and approximately comparable ($\sim 30\ \mu\text{m}$, except for one) porous layer thickness was prepared for the studies. The information on their preparation conditions and resulting mechanical parameters is shown in Table 1.

Table 1. Sample Preparation Parameters

Sample	Current density, mA/cm^2	Etching duration, min	Porous layer thickness, μm	Porosity P, %
1	20	14.3	24.5	40
2	40	12	32.3	48
3	130	4.5	29.6	60
4	200	3.26	28	71
5	133	14.3	85	77

After fabrication, Samples 1–4 were stored in dark place for about two years,

and Sample 5 – for about four years.

3. EXPERIMENTAL METHODS

Using automated polarimetric installation built on the base of G5 goniometer (which provides angular scale readings accuracy of 5 arcseconds), variable angle ellipsometry was carried out at the wavelength of 625 nm ($\Delta\lambda = 10$ nm) within wide angular range 40° – 80° , where ellipsometric function shows fully its shape. At each experimental point, the intensity of light reflected from the samples was measured with different combinations of polarizer's and analyzer's azimuths, according to specific algorithm. From these intensities, conventional ellipsometric parameters (EP) Δ and $\tan \psi$ were calculated (i.e., phase shift between p- and s-components of light wave after reflection, and the ratio of corresponding energy reflectivities). When angular dependencies of EP are known, one can compute the parameters of porous layers (refractive indices and thicknesses) according to a selected model of the sample structure. This was done with a program, which implements computational approach described in [16].

Spectral ellipsometry has also been conducted in NIR 1–2 micrometers range, which gives EP dependencies on radiation wavelength at 60° , 65° , and 70° incidence angles. For these measurements, SE-2000 SEMILAB ellipsometer with rotating compensator was utilised. This instrument covers wide spectral range from NIR to UV, has probe beam dimensions 470×0.365 μm and angle of incidence range available from 15° to 90° .

Experimental data processing and analysis were done with the use of WVASE software for ellipsometry (J.A Woollam Co.) [15]. In the framework of inverse problem of ellipsometry, a few layered medium optical models were fitted to the experimental spectra to find their optimal parameters (such as refractive index n , extinction coefficient κ , layer thickness, and porosity).

Visualisation of samples internal structure was done by scanning electron microscopy (SEM), using FEI Inspect S instrument.

4. EXPERIMENTAL DATA AND THEIR PROCESSING

4.1. Multi-Angle Measurements in the Visible Range

The results of variable-angle ellipsometric measurements are shown in Figs. 1 and 2.

As one can see from the measurements, angular dependencies of $\cos \Delta$ and $\tan \psi$ in their majority do not contain any pronounced structure, except for Sample 4 (Fig. 2).

At the beginning of analysis of the reflective system, selection of a proper

mathematical model is crucial to be able to provide adequate experimental results interpretation. With such thicknesses of the porous layers formed (Table 1), at least some of the samples can be enough transparent to enable light reflection from the back interface with the substrate, which leads to formation of an interference pattern. Such a pattern is experimentally observed only on the ellipsometric curves of Sample 4 (see

Fig. 2). For the rest of the samples, one can confidently say that the input light wave does not reach that back interface; porous

layer than can be considered as semi-infinite at calculations.

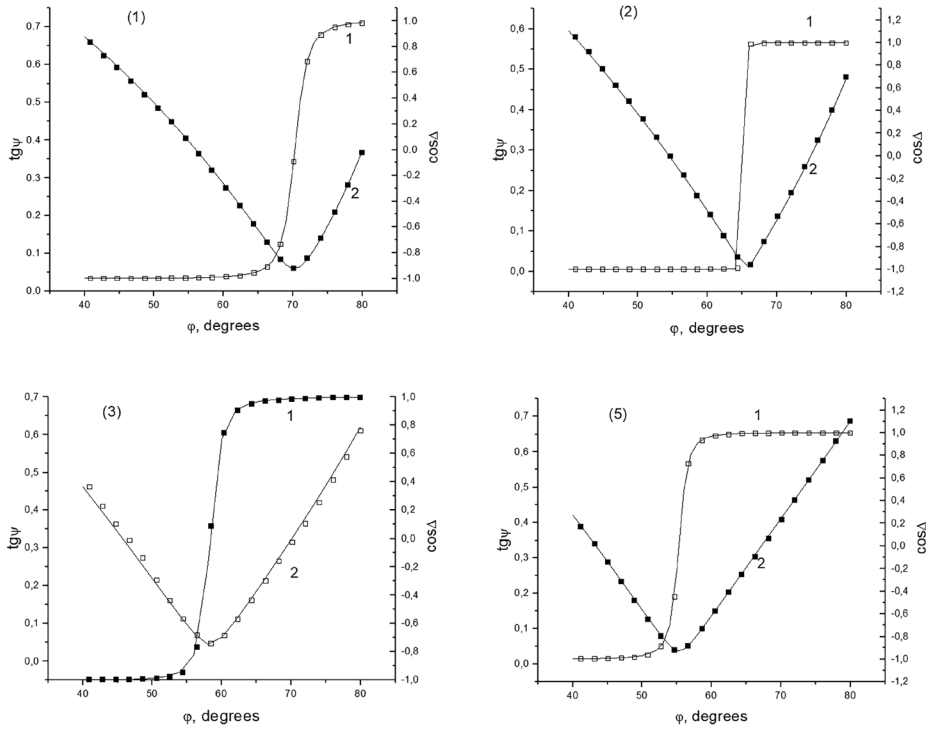


Fig. 1. $\cos \Delta$ (1) and $\tan \psi$ (2) dependencies on the angle of incidence ϕ for Samples 1, 2, 3, and 5. The graphs are arranged in the order of porosity ascending. Solid curves are computed using the parameters from Table 5.

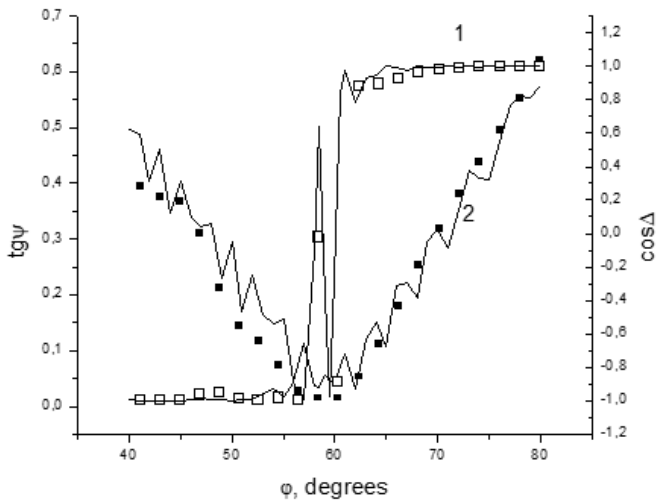


Fig. 2. $\cos \Delta$ (1) and $\tan \psi$ (2) dependencies on the angle of incidence ϕ for Sample 4. Solid curves are computed using the parameters from Table 5.

4.1.1. Single Layer Model

Initially, it is natural to assume that the output wave coming after the interaction with the samples is formed only as a result of reflection from semi-infinite porous material, since, as we mentioned above, the refracted wave decays before reaching the substrate. In this case, the system has only refractive index n and extinction coefficient κ as parameters to determine. At each of the incidence angles during the measurements, two ellipsometric parameters are determined, and two medium parameters are obtained from them.

The model of semi-infinite medium is illustrated in Fig. 3. Output wave here is only formed by upper interface reflection; the refracted part is fully absorbed in porous medium.

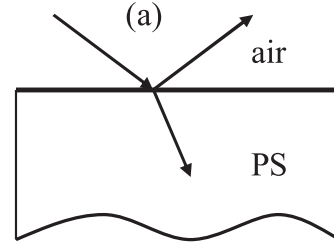


Fig. 3. Semi-infinite medium model (PS – porous silicon layer).

The results of calculation optical parameters of the medium using this model are presented in Table 2. It can be mentioned that optical constants n and κ have shown monotonic dependencies on incidence angle.

Table 2. Ranges of Porous Silicon Refractive Index (Δn) and Extinction Coefficient ($\Delta \kappa$) Variations for 40°–80° Angle of Incidence Change

Sample	Δn	$\Delta \kappa$	Φ	G
1	2.82–2.76	0.164–0.372	0.000053	0.000057
2	2.24–2.180	0.12–0.03	0.000055	0.001085
3	1.67–1.61–1.665	0.09–0.12	0.000182	0.003408
4	1.455–1.79–1.636	0.13–0.05	0.214	1.28
5	1.45–1.425	0.11–0.03	0.000038	0.001038

Table 2 also contains evaluation of the scattering between experimental points and theoretical values (calculated from average

optical constants). A simple function Φ was utilised for this purpose:

$$\Phi = \frac{1}{2N} \sum_{i=1}^N \left\{ (\operatorname{tg} \psi_{iE} - \operatorname{tg} \psi_{iT})^2 + (\cos \Delta_{iE} - \cos \Delta_{iT})^2 \right\}, \quad (1)$$

where N – the number of measurement points (in this case, angles of incidence), and indices T, E mark theoretical and experimental EP, respectively. The function (1) was numerically minimised using its gradient G . The minimal function value characterises deviation between modelled and measured data, and G approaching zero means successful result of minimisation.

From our previous experience in porous silicon studies, both the function and the gradient can typically amount a value of 10^{-6} order, but in the present case, Φ and G are significantly away from zero.

As the next step, refractive index n and extinction coefficient κ of semi-infinite

porous layer were computed in the framework of Bruggeman's effective medium approach. Porous material was represented as a mixture of monocrystalline silicon ($n=3.92$, $\kappa=0.03$) and air. The results are presented in Table 3.

Table 3. Optical Constants n_B and κ_B of Silicon with Different Porosity P , Calculated and Measured Principal Angle F , and Ellipticity $\tan\psi_m$

Sample	P, %	n_B	κ_B	Substrate, calculated		Material, experiment	
				F, deg.	$\tan\psi_m$	F, deg.	$\tan\psi_m$
1	40	2.017	0.0178	69.6891	0.0037	70.4	0.0614
2	48	2.4163	0.0145	67.5180	0.0035	65.27	0.0125
3	60	1.976	0.009	63.1576	0.0029	58.33	0.0504
4	71	1.6011	0.0044	57.9965	0.0019	61.5	0.0169
5	77	1.4282	0.0026	55.0009	0.0014	55.6	0.0378

Table 3 also contains the comparison of calculated and measured values of the principal angle F (at which $\Delta = 90^\circ$) and ellipticity in the minimum $\tan\psi_m$. The former parameter determines horizontal shift of Δ curve along the angle of incidence axis, and the latter one – the deviation of $\tan\psi$ curve.

From Table 2 and Table 3, one can see significant difference between expected parameters for porous silicon with known porosity and the values obtained from

4.1.2. Double-Layer Model

In the case of infinite porous silicon thickness, EP angular dependencies possess just simple shapes – $\cos\Delta$ increases monotonically from -1 to +1 (with zero transition at the principal angle), and $\tan\psi$ curve has a corresponding minimum. Real experimental curves are shifted in comparison with the simulated ones; particularly, they demonstrate lower principal angle F and higher ellipticity of polarization (Table 3). A probable reason for such behaviour may be the existence of some another layer with properties different from the substrate. These optical properties were calculated using double-layer model, which is illustrated in

experiment. Particularly, experimental extinction ratios κ (Table 2) are significantly higher than the modelled ones κ_B (Table 3). In accordance with this, parameters F and $\tan\psi_m$ of the ellipsometric curves also differ. These facts indicate actual differences of real reflective system from the model of semi-infinite medium. For further calculations, more advanced models should be taken instead.

Fig. 4. Within this model, parameters of the reflected wave are determined by multiple reflections in the top layer.

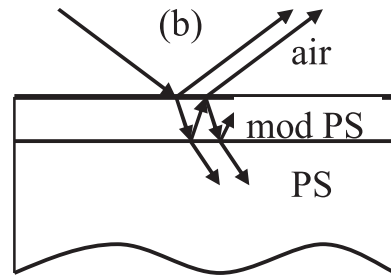


Fig. 4. Double-layer model (PS – porous silicon layer, mod PS – modified porous silicon layer).

Parameters n_1 i d_1 of the modified porous silicon layer were obtained by objective function (2) minimisation, assuming that the substrate consists of porous silicon with

porosity P and optical constants matching the values we had in Table 3. The results of such modelling are shown in Table 4.

Table 4. Refractive Index n_1 and Thickness d_1 of Modified Porous Silicon Layer, Calculated for 40°–80° Angle of Incidence Change

Sample	n_1	d_1 , nm	Φ	G
1	3.364	89*, 186*	0.000055	0.0117
2	2.306	50–70*, 90–75*	0.000208	0.01042
3	1.810–1.78	79*, 117*	0.000886	0.18192
5	1.520–1.535	78–120	0.000249	0.009474

*As the ellipsometric parameters depend periodically on layer thickness, the two least values from the possible are presented here.

This model describes better experimental curves – we obtain for the first two samples almost constant refractive index over the full angle of incidence range (at least, down to statistical data scatter). However, we still have angular dependence for the thickness. This means that the model considered still has some difference from real system; refractive index n_{ps} of the substrate should also be fitted together with top layer

parameters n_1 , d_1 . Their values are not far from substrate porous silicon parameters; we assume so that top layer is composed of modified porous silicon.

The results of optimisation n_1 , d_1 and n_{ps} all together are presented in Table 5. Corresponding extinction parameters were neglected during the optimisation, as they are too small to have any noticeable influence on the results.

Table 5. Modified Top Layer's Refractive Index n_1 and Thickness d_1 , and Porous Substrate Refractive Index n_{ps}

Sample	n_1	d_1 , nm	n_{ps}	Φ	G
1	2.322	134.5	2.861	0.000006	0.000003
2	2.316	222.4	2.427	0.000007	0.000002
3	1.775	82	1.963	0.000104	0.000018
4	1.715	23395	1.847	0.007666	30
5	1.549	140.1	1.673	0.000008	0.000004

The objective function Φ and its gradient G turned to be much higher for Sample 4. This can be easily explained by large EP scattering on its graph (Fig. 2) due to interference existing in this sample, and relatively large step of experimental points in comparison with the oscillations. It also should be noted that the thickness of interference layer in this sample (23.4 μm , Table 5) is significantly less than overall

porous silicon thickness (28 μm , Table 1).

In Table 5, we can see the lowest values of the objective function and gradient, for most of the samples. One can say that double-layer model incorporating modified top layer of porous silicon has the best agreement to experimental data considered in this work. Modified layer possesses slightly higher porosity than the main volume of porous silicon. We have estimated porosity

values according to Bruggeman's effective medium model (which represents material as a mixture of monocrystalline silicon and

air), corresponding results are presented in Table 6.

Table 6. Porosities of Surface Layer and the Main Volume of Porous Silicon

Sample	Surface layer		Main volume	
	n_1	P, %	n_{ps}	P, %
1	2.322	51	2.861	35
2	2.316	50.7	2.427	47.7
3	1.775	65.6	1.963	60.3
4	1.715	66.4	1.847	63.5
5	1.549	73	1.673	68.6

4.2. NIR Spectral Ellipsometry

Spectral ellipsometry measurements for some of the samples mentioned above are shown in Fig. 5 as parameter ψ dependencies on wavelength. The curves demonstrate typical interference structure with a sequence of maximums and minimums.

The first thing should be considered is the quantity of interference maximums for different samples, which is mainly deter-

mined by the thickness of surface layer. Within 1200–1500 nm range, one can see more than 20 maximums for Sample 2 (Fig. 5c). They have well pronounced shapes, which witness sharp interfaces of the layer. Their amount corresponds to nearly 30 μm layer thickness. Samples 1 and 3 show similar spectra, so there is no need to present them here.

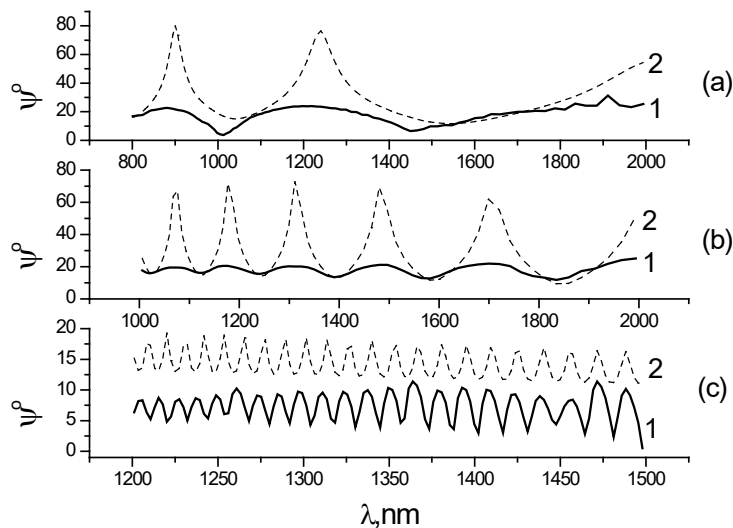


Fig. 5. Experimental (1) and theoretical (2) parameter ψ spectral dependencies of porous silicon samples 4 (a), 5 (b), 2 (c).

Sample 5 demonstrates just 6 maximums in 1000–2000 nm range (Fig. 5, b), which can mean much less interference layer thickness. Sample 4 shows only 3 maximums within 800–2000 nm range. It also can be noted that the last two samples possess blurred interference patterns in comparison with the simulated curves. It is naturally to expect not so perfect edges in porous layer obtained by common methodology of anode etching.

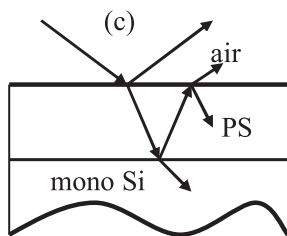


Fig. 6. Double-medium model (PS – porous silicon layer, mono Si – bulk monocrystalline silicon).

To provide spectral ellipsometry data interpretation for Sample 2, one more model has been proposed, which considers multiple reflections in thick porous silicon layer on monocrystalline substrate resulting in interference pattern formation. This double-medium model is illustrated in Fig. 6.

For spectral ellipsometry data interpretation, J.A. Woollam’s WVASE software [15] was utilised. In Table 7, one can see the parameters of PS interference layer, obtained out of analysis. Here we maintain using n_1 and κ_1 notations, the same as in Section 4.1.2, because top modified layer manifested itself in NIR spectra. It happens that ellipsometric spectra for Samples 4 and 5 can be described with the model (Fig. 4), while for the rest of the samples model (Fig. 6) is fulfilled.

Table 7. Refractive Index n_1 , Extinction Coefficient κ_1 , Thickness d_1 and Porosity P for the Top Layer of Porous Silicon, Obtained from Spectral Ellipsometry

Sample	P, %	n_1	κ_1	d_1 , nm
2	48	2.4163	0.0145	32039
4	71	1.6011	0.0044	1214
5	70	1.6324	0.0048	4383

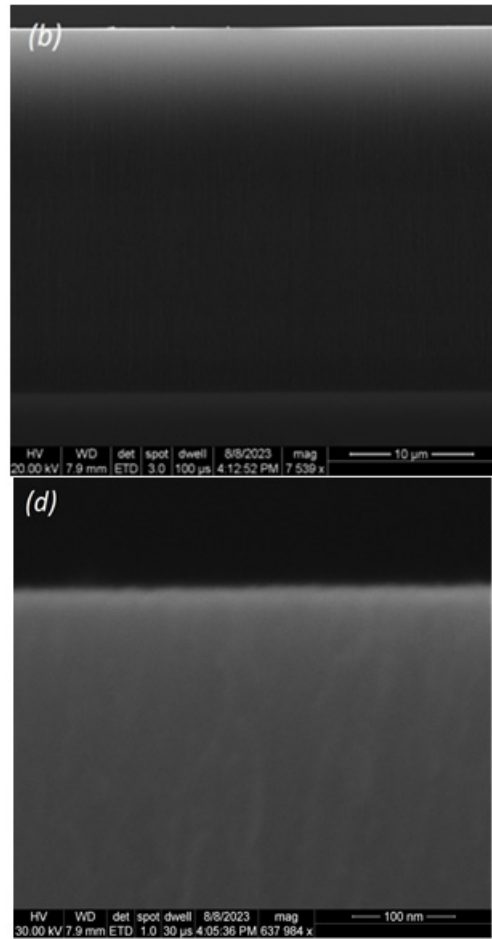
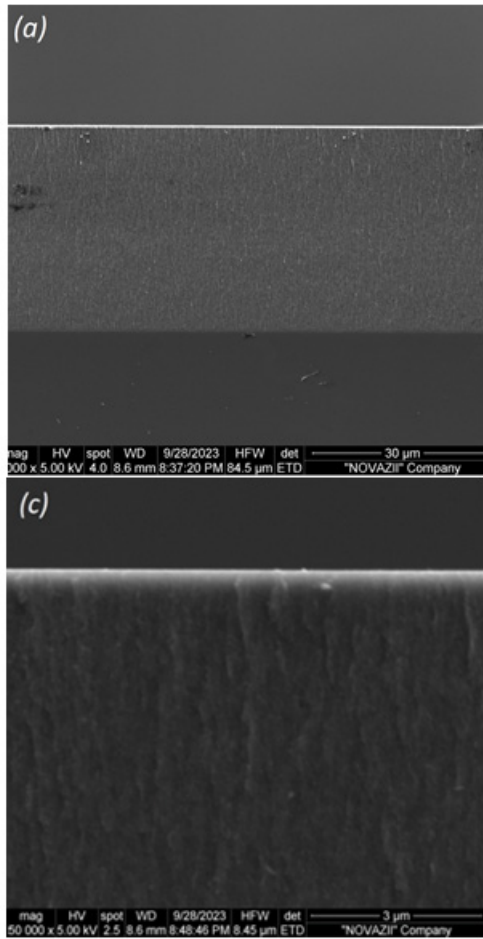
These results indicate existence of a surface layer with the thickness of 1200 nm and 4400 nm in Samples 4 and 5, respectively. For Samples 1, 2, and 3, interference occurs in overall porous layer; no sublay-

ers are detected. In addition, the porosity found for the sample 5 (70 %) resulted in a lower value than “technological” 77 % from Table 2.

4.3. Scanning Electron Microscopy Studies

In Fig. 7, an internal structure of a few samples is illustrated, as obtained from

scanning electron microscopy (SEM) data.



In Fig. 7a, b, one can see the edges between porous silicon and monocrystalline substrate, which lies at about 30 μm deep from the surface of Samples 2 and 4. Besides, an additional transient layer up to few hundreds nm thick can be observed for

Sample 2 in the magnified image (Fig. 7c). For Sample 4, some blurred region of about 4–5 μm thick is present, but in the magnified image (Fig. 7d) no thin surface layer is clearly seen.

5. RESULTS AND DISCUSSION

In this work, we observe by different experimental methods two types of layers in porous silicon samples. One of them is a thin near-surface layer up to 200 nm, another much thicker and can reach up to 4400 nm depth.

Multi-angular ellipsometric investiga-

tions allows detecting thin surface layers from 80 nm (Sample 3) to 220 nm (Sample 2). We identify them as consisting of modified porous silicon. These layers are slightly different by their optical parameters from the substrate material (also porous). Absence of an interference structure on

angular ellipsometric curves for Samples 1, 2, 3 and 5 (Fig. 1) can be explained by small thickness of corresponding layers. For interference occurrence, layer thickness must be equal to a few spatial periods determined by Eq. (2) below (as it comes out from the main equation of ellipsometry for a transparent material that ellipsometric parameters are periodical functions of the thickness):

$$d_{\Pi} = \frac{\lambda}{2\sqrt{n^2 - \sin^2 \varphi}}. \quad (2)$$

In our case, this period amounts between 149 nm (Sample 1) and 220 nm (Sample 5), which does not exceed observed thicknesses. For the visible range, that is not enough to form an interference pattern. This takes place only for Sample 4 (Fig. 2), which has modified porous layer estimated as 23400 nm thick.

In SEM images, modified layers can possibly be assigned to some region near the surface of Sample 2, which is much brighter than deep material (Fig. 7a, c).

From spectral ellipsometry graphs, it is difficult to notice this layer, because infrared interference pattern is mostly formed in overall porous layer. Presence of another, much thinner layer, just shifts a bit the extrema along wavelength axis, which is hard to separate and detect during data processing.

Thick modified layers have leaved their trace in spectral ellipsometry results for Samples 4 and 5 (Table 7). The former gives an interference pattern, which can be described by the model containing 1200 nm thick layer with “technological” optical parameters from Table 2. It possibly can correspond to the light blurred region in Fig. 7b. In case of Sample 5, the thickness obtained from modelling for such a layer is 4400 nm.

Multi-angular ellipsometric measure-

ments in a visible range show one more layer (23400 nm) in Sample 4, thickness of which is equal to the overall depth of porous material. This became possible due to high transparency of porous silicon at 625 nm, which was the wavelength of experiment.

We assume that such behaviour of ψ parameter for tSamples 4 and 5 (Fig. 5) is caused by 1200 nm and 4400 nm thick, porous silicon areas, respectively, which are transparent enough in 1200–1500 nm range. The underlaying material can be porous silicon saturated with certain impurities, such as OH-groups formed during electrochemical etching and washing. This contaminated part of material will absorb light more effectively, so that electromagnetic wave will not reach real bottom of overall porous material. Such situation has agreement with double-layer model from Fig. 4.

Numerous studies [1]–[3] have established that porous silicon is saturated with OH groups immediately after its preparation. These groups are localized at pores surfaces, being then gradually replaced by oxygen during storage in normal ambient conditions. Relatively thick layers, which manifest themselves in IR spectral ellipsometry (Samples 4 and 5), are therefore built of partially oxidized porous silicon. At the same time, the deeper material is less oxidized and has a residue of OH groups (or another impurities). The oxidized areas can be located at different depths, due to different storage time for Sample 4 and 5 (2 and 4 years in air atmosphere).

Since the porosity has the highest degree for these two samples, they possess a bigger surface area and consequently have higher number of impurities. This can explain why thick, porous layers could only be detected in Samples 4 and 5.

Spectral ellipsometry studies carried out by other authors also indicate complex few-layered structure of porous material. For

instance, in [4]–[7], the authors describe IR ellipsometry spectra by a two-layer model, or a model with continuously distributed parameters over the depth.

It is possible that the almost transpar-

ent layer observed in multi-angular ellipsometric studies [8]–[10] can consist of significantly oxidized material, similar to that what we refer here as modified porous silicon.

6. CONCLUSIONS

Porous silicon obtained by electrochemical etching of monocrystalline material can have a non-uniform structure incorporating modified porous silicon sublayers.

Variable-angle ellipsometry curves for such material in the visible range can be described by a model which contains modified layer on the surface (about 100–200 nm for Samples 1, 2, 3, 5, or 23400 nm for Sample 4).

The porosity of modified layers slightly differs (by 5–7 %) from the porosity of underlaying material (Table 6).

An interference pattern observed in IR spectral ellipsometry data indicates presence of sublayers in overall porous mate-

rial. Their thickness was estimated as 1200 nm (Sample 4) and 4400 nm (Sample 5). We assume that their bottom area is contaminated with certain impurities (probably OH groups), which absorb the light before reaching monocrystalline silicon substrate.

SEM images of porous silicon cross-section confirm a complex structure of the material in several cases.

Such inhomogeneity of porous silicon appears as the result of electrochemical reactions during wafer etching, as well as of subsequent long-term storage in ambient air. Apparently, the material in top area has a more loosened structure than in depth.

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