Measurement of optical loss in oxidized multilayer planar porous silicon waveguides

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There are presented the fabrication and characterization of planar waveguides in porous silicon. These results are extended to the visible spectrum by oxidation of the silicon to silica. Porous silicon is a novel material with potential applications in photonics due to its relatively easy manufacture. Its optical performance however, still needs to be improved before commercial applications become possible. An attempt is made to add new knowledge on this material. For this purpose, waveguides of 1.5 cm long were prepared in p-type silicon substrates by a double step anodization process. The influence of thickness of core and cladding layers on guiding properties of oxidized porous silicon waveguides was studied. The thickness of all samples was checked by measuring their porosity. The refractive index of the samples has been calculated before and after oxidation by the Bruggeman method. Optical losses were measured in the visible range (λ = 633 nm) by end fire coupling method. The results have shown that by increasing the guiding and cladding layer thickness, optical loss decrease. The reason could be the current density fluctuations during anodization process and light scattering from rough interfacial surfaces, which exist between guiding and cladding layers. The multilayer planar waveguides were made by careful modification of the anodization time. By this method, significant improvements of the waveguide preparation and waveguide characteristics were obtained.

(Received August 6, 2007; accepted August 30, 2007)

Keywords: Porous silicon, Waveguide, Oxidation, Optical loss, Multilayer planar waveguide

1. Introduction

In recent years, there has been an increasing interest in optoelectronic properties of porous silicon (PS). Besides its visible photoluminescence which was discovered by Canham in 1990 [1], PS became an attractive material for optoelectronic integrated circuits like light emitting diodes [2], photodetectors [3] and waveguides [4, 5]. Application of this material for the fabrication of waveguides is due to easy modulation of its porosity and, therefore, its refractive index by changing the current density during anodization. Also oxidizing as-prepared PS waveguides extend their guiding region into the visible range.

Several methods for fabricating porous silicon waveguides (PSWG) have been investigated and they can be divided into two groups. In the first group, PS acts only as a cladding layer and in the second group, PS acts as both guiding and cladding layers.

In the first case, the guiding layer is made by epitaxial growth or ion implantation of silicon on PS cladding layer. Waveguides fabricated by these methods have been described in Ref. [6] and Ref. [7], respectively.

In the second case, a PS guiding layer has been produced by changing the current density during the anodization process. In this way, it is possible to design multilayer and graded index planar waveguides in which the porosity is varied through the vertical cross section of the structure and they were called controlled index porous silicon (CLIPS) waveguides. Also the operational wavelength of as-prepared CLIPS waveguides can be extended to the visible region by oxidation of PS that leads to significant reduction in the optical losses [8].

Another case is the densified oxidized porous silicon waveguides (DOPS) which are formed by high temperature oxidation of a uniform layer of PS to obtain a densified layer of oxidized PS (SiO_2) on a porous oxide layer [9]. Both planar and pre-defined waveguides can be fabricated by this method. Of course, pre-defined waveguides were found to be multi-mode and have an optical loss of order 5-6 dB/cm at 633 nm [10].

Lerondel et al [11] studied the light scattering for the first time and they have shown that the main source of light scattering in porous silicon layers is the PS/silicon interface. In another paper, Lerondel et al [12] have shown that layer thickness fluctuations are at the origin of the light scattering. Two types of layer thickness fluctuations were observed. They occur at the micrometer scale and leads to isotropic roughness. The second one occurs at a few hundred of micrometer scale and leads to anisotropic waving. Both types of layers thickness are due to current density fluctuations but their origins are different. The waviness is the results of radial resistivity fluctuations and thus mainly concern the highly doped wafer. The roughness is the results of the initial wafer roughness, which, for nanoporous silicon obtained with low doped wafer as the one used by the authors, can lead to rms roughness of the order of a few tens of nanometers i. e. strong light scattering efficiency. These layer thickness fluctuations were found to depend strongly on the current density and the electrolyte viscosity but not on HF concentration.

Setzu et al [13] have shown how the roughness can be decreased by increasing the viscosity, i. e. lowering the electrolyte temperature. This leads to a smoothing of the interfaces and hence a drastic improvement of multilayered structure quality. Reece et al [14] have shown that not only the roughness but although the waviness are the main source of losses in case of highly doped multilayered structures. Also, losses can be decreased leading to subnanometer linewidth microcavities.

The paper describes the fabrication of oxidized multilayer planar porous silicon waveguides. Oxidized porous silicon waveguides were made with various porosities. Refractive indices of the layers and differences between them were measured. Optical losses were measured for the different thicknesses at a wavelength of 633 nm.

2. Sample preparation

Multilayer planar waveguide structures were made by electrochemically anodizing (111) oriented p-type silicon with 0.98 - 1.1 Ω-cm resistivity in an electrolyte mixture composed of 40 % hydrofluoric acid and ethanol (1:1). Current densities were varied between 25 to 40 mA/cm^2 and anodization time between 10 to 20 minutes. In this way, we obtained planar waveguides with two PS layers and arbitrary layer thickness. In the structures, the top layer (guiding layer) has low porosity and subsequently high refractive index that separated from bulk silicon by a cladding layer with high porosity and low refractive index. Details are given in Table 1 in order to understand how the studied structures were produced and how they looked like. A typical SEM image (cross section) of the produced multilayer planar porous silicon is shown in Fig. 1.

Table 1. Characterization of fabricated waveguides before oxidation.

PSWG Sample No.	J(mA/cm^2)	t (min)	Porosity (%)	Thickness (μm)
1	25-30	10-10	45-59	8.8-9.1
2	25-35	10-10	45-73	8.8-11.3
3	25-40	10-10	45-80	8.8-14.3
4	30-40	10-10	59-80	9.1-11.3
5	35-40	10-10	73-80	11.3-14.3
6	25-30	10-20	45-74	8.8-21.4
7	25-35	10-20	45-75	8.8-28
8	25-40	10-20	45-82	8.8-30
9	30-40	10-20	59-82	9.1-30
10	35-40	10-20	73-82	11.3-30
11	25-30	20-20	65-74	14-21.4
12	25-35	20-20	65-75	14-28
13	25-40	20-20	65-82	14-30
14	30-40	20-20	74-82	21.4-30
15	35-40	20-20	75-82	28-30

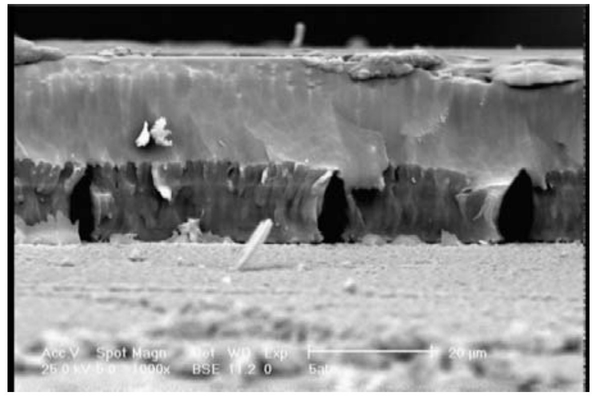

Fig. 1. SEM image of a typical multilayer planar porous silicon. Images were obtained with a Philips XL30 scanning electron microscope.

As can be seen from the Table 1, the multilayer planar waveguides were prepared in three groups. In first group, anodization time for both cladding and guiding layers were kept constant at 10 min, but with mixture of current density from 25 mA/cm^2 to 40 mA/cm^2. PSWG samples No. 1 to No. 5 from Table 1 are in this group. In second group, thickness of cladding layer enhanced by increasing anodization time to 20 min but anodization time of guiding layer were kept constant at 10 min with mixture of current density from 25 mA/cm^2 to 40 mA/cm^2. PSWG samples No. 6 to No. 10 from Table 1 are in this group. In third group, thickness of guiding layer enhanced by increasing anodization time to 20 min but anodization time of cladding layer kept constant at 20 min with mixture of current density from 25 mA/cm^2 to 40 mA/cm^2. PSWG samples No. 11 to No. 15 from Table 1 are in this group. The porosity data were obtained by gravimetric measurement and was performed in single PS layers.

Then, thermal oxidation of the structures was performed in a furnace by a three step process [15] in order to extend guiding region into the visible range. In the process, firstly samples were oxidized at 300 °C for 1 hour in dry O_2 to stabilize the porous material against the heat treatment at higher temperatures [16], then oxidation at 1000 °C for 25 minutes was performed in wet O_2, and finally the samples were oxidized at 1100 °C for 1 hour in dry O_2. In this way we made oxidized multilayer planar PS waveguides (OPSWG).

In all measurements, the waveguides were excited by end fire coupling method. A scheme of the experimental set-up for the optical loss measurements is presented in Fig. 2. The light source was a He-Ne laser beam with a wavelength of 633 nm. The coupling was performed with an optical fiber (5μm diameter) to focus the laser beam on the input of the waveguide. For this purpose the input part of the sample was cleaved by a diamond scribe. The edge looks like rectangular shape and width of 0.2 mm. The cleaved part designed the way that light scattering remains minimum and coupling efficiency becomes maximum. The cleaved part was close to natural plane and perpendicular to its surface. The diameter of laser beam was about 2 mm which was greater than cleaved part. For reduction of the edge effect on the measurement spot, the 5μm diameter optical fiber has been used. This would prevent injection loss. Illumination of background light for waveguide was fully covered. The output current was detected by a silicon photocell and measured with a Keithley model 485 Autoranging Pico Ampermeter. We studied optical loss in waveguides with various guiding and cladding thicknesses with the same lengths (1.5 cm).

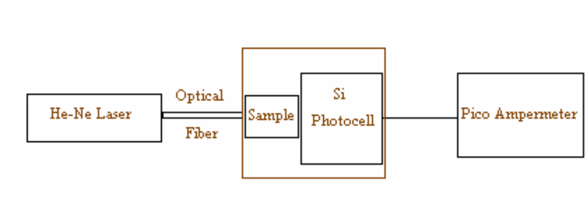

Fig. 2. Schematic of the experimental arrangement for measuring optical loss.

3. Results and discussion

Our waveguides were prepared from three layers, so characteristics of each porous layer separately should be explained to clarify the property of waveguides. The characteristics included such as porosity, thickness, and refractive index before and after oxidation. The porosity and thickness measurements were performed on single layers by gravimetric method. The method was done by weighting the samples before and after anodization (m_1 and m_2 respectively) and after dissolving the porous layer in 1 molar of NaOH solution (m_3). Then, the porosity and thickness of layers was determined by the following equations [17]:

$$P = \frac{m_1 - m_2}{m_1 - m_3} \tag{1}$$

$$d = \frac{m_1 - m_3}{\rho S} \tag{2}$$

where ρ is density of Si and S is surface area of porous sample. The porosity and thickness measurements are given in Table 1. As can be seen, porosity and thickness of each sample increase by increasing anodization time and current density. By this method, it is possible to prepare two porous layers planar waveguides with different thickness of cladding and guiding layers. For example, current density was kept constant at 25 mA/cm^2, and anodization time was increased from 10 to 20 min. Then porosity and thickness increase from 45 to 65% and from 8.8 to 14 μm, respectively (Table 1). Also at anodization time of 10 min, by increasing the current density from 25 to 40 mA/cm^2, porosity raised from 45 to 80% and thickness from 8.8 to 14.3 μm. Therefore, by combination of various thicknesses, two layer planar porous silicon waveguide were made (Table 1).

The refractive index of single porous structures for cladding and guiding layers can be determined by the Bruggeman model (Effective Medium Approximation). By knowing the porosity of PS layer in the model, we can determine the refractive index of the layer by the following equation [18]:

$$(1-P)\frac{n_{Si}^2 - n^2}{n_{Si}^2 + 2n^2} + P\frac{1-n^2}{1+2n^2} = 0 \tag{3}$$

where n is the refractive index and P is the porosity of PS sample. By substituting n_{Si} at used wavelength (633 nm) and porosity of each layer, refractive index of PS sample before and after oxidation were measured. Results are given in Table 2. As it can be seen, the refractive index decreases by increasing porosity. The reason could be due to the substitution of air with lower refractive index instead of Si with higher refractive index in PS nanocolumnar structure at high porosity [17].

Our light source was He-Ne laser (633 nm) which is in visible range. For this reason, the variation of porosity and refractive index should be studied after oxidation. The thermal oxidation process was explained in section 2. Also, the Bruggeman model was used for calculation of porosity (P') and refractive index (n') after oxidation.

The refractive index and porosity of oxidized porous silicon (OPS) layers are shown in Table 2. It is assumed that oxidation expands the volume of Si branches in size by 50%. So, the porosity after oxidation is calculated by the following equation [10]:

$$P' = 1 - \frac{3}{2}(1-P) = \frac{3}{2}P - \frac{1}{2} \tag{4}$$

Then, we can calculate the refractive index of OPS layers (n′) by substituting the porosity after oxidation (P′) into the Bruggeman equation (Eq. 3). It can be seen from Table 2, after oxidation, porosity and refractive index were reduced. It could be two reasons for this behavior. Firstly, the volume of porous material increased. Secondly, oxidation causes Si branches substituted by SiO_2 and it is clear that SiO_2 has a lower refractive index than Si [17].

Table 2. Refractive index of porous layers calculated by Bruggeman model before (n) and after (n′) oxidation. P and P′ are porosities before and after oxidation, respectively.

J (mA/cm^2)	t (min)	P (%)	n	P′ (%)	n′
25	10	45	2.5	17.5	1.38
30	10	59	1.99	38.5	1.28
35	10	73	1.46	59.5	1.18
40	10	80	1.35	70	1.13
25	20	65	1.79	47.5	1.23
30	20	74	1.5	61	1.17
35	20	75	1.48	62.5	1.16
40	20	82	1.3	73	1.12

Waveguide optical loss measurements

Optical losses were determined by coupling light from a He-Ne laser (λ=633nm) into the cleaved samples. The output signal was measured by a photocell. The measurements were performed for several waveguide structures. The optical loss measurements in our samples were normalized by using following formula

$$\alpha = -10 \log (I/I_0) \quad (5)$$

where α is optical loss in dB, I is output current intensity from waveguide, and I_0 is output current intensity without waveguide. By inserting the length of waveguide which is 1.5 cm α was converted to optical loss per unit length (dB/cm). The measurement represents total optical loss which includes loss inside waveguide and coupling loss in output and input of waveguide. Usually measuring of coupling loss is difficult because the samples were fabricated on (111) planes and it is negligible. Results for OPSWG samples are given in Table 3, the fabrication characteristics are given in Table 1.

This calculation ignores multiple reflections (Fabry-Perot resonances), which is justified by the relatively high waveguide loss, and the excitation loss caused by the difference in profile between the exciting beam and guided mode which implies the above value of α is an upper limit to the actual waveguide loss.

The measured optical losses (< 10 dB/cm) are comparable to other reported data (<several dB/cm). In our suggestion, the optical losses are mainly coming from attenuation losses and coupling losses have no sufficient effect. Attenuation losses are caused by absorption, interface scattering and volume scattering.

Dependence of the optical loss versus cladding layer thickness is reported in Fig. 3. In the figure, the guiding layer thickness kept constant at 14 μm and cladding layer thickness increased from 21.4 to 30 μm. As seen from Fig. 3, by increasing the thickness of cladding layer, propagation loss is decreased. The reason could be due to the reduction of optical radiation into the Si substrate [19].

Table 3. Optical loss measured in oxidized PS waveguides (OPSWG).

OPSWG sample No.	Optical Loss (dB/cm)
8	7.9
9	7.7
10	7.8
11	7.77
12	7.7
13	7.6
14	7.35
15	8

In the next step, the thickness of cladding layer kept constant at 30 μm and the thickness of guiding layer increased from 8.8 to 28 μm. Optical losses are reported in Fig. 4. Similar to the previous case, we succeed to reduce the propagation loss by increasing the thickness of guiding layer.

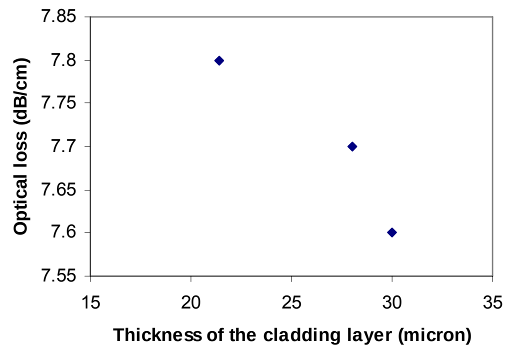

Fig. 3. Optical loss measured in OPSWG with 14 μm guiding layer at 633 nm.

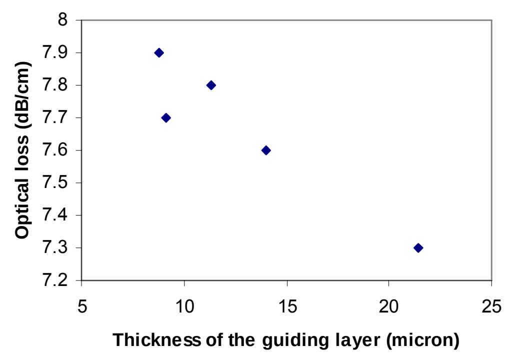

Fig. 4. Measured loss in OPSWG with 30 μm cladding layers at 633 nm.

In general, optical loss could be due to three mechanisms: scattering, absorption, and radiation. With increasing the thickness of cladding layer light transmission reduces through Si body. Also, since the waveguides are oxidized they do not exhibit high absorption. Therefore, loss in our waveguides mainly comes from scattering at interface surfaces between porous layers and scattering of nanostructures inside pores between air and SiO_2 walls.

Loss due to nanostructures (volume loss) directly depends on ratio of pore size and wavelength which has been used and it is important for shorter wavelengths (Rayleigh scattering). However, since we have used only one wavelength (633 nm) it is not possible to msake more comments on this loss.

Nevertheless, in our samples scattering loss from rough interface surface could be considered. Fluctuations in PS layers cause a roughness in these layers. This roughness is origin of light scattering in our waveguides that causes scattering from rough interface surfaces.

Variation of PS thickness is due to fluctuations of current density which for low doped Si wafer has the root mean square of several 10 nm. Therefore, it is a strong light scattering. If, ideally, the roughness of interface layer is assumed as sinusoidal, loss from rough interface, relative to difference of square refractive indices, is [20]

$$\alpha\ (\mathrm{dB/cm}) \approx (n_i^2 - n_j^2) \qquad (6)$$

If difference of refractive index between layers becomes less, loss should decrease. Also, this loss for higher mode increases. If difference of refractive index diminishes, the number of emission modes become less and therefore the loss decreases as well [21]. The results are given in Table 4. As seen from the Table 4, by decreasing the difference of refractive index between guiding/cladding and guiding/air layers loss become less. This shows that interfacial roughness plays a major role in PS waveguides so that by increasing the guiding layer thickness, the interfacial roughness and attenuation losses decreases [10].

Table 4. Relationship between the difference of the refractive index of waveguide layers and losses. n_g is guiding index, n_c is cladding index, and n_{air} is air index.

OPSWG No.	$n_g^2 - n_c^2$	$n_g^2 - n_{air}^2$	α (dB/cm)
8	0.75	0.9	7.9
9	0.39	0.64	7.7
10	0.14	0.39	7.8
13	0.26	0.51	7.6
14	0.12	0.37	7.35

The optical losses decrease with the increasing in thickness of the guiding layer. However, the roughness increases with thickness, so the surface scattering losses increase at the same time, the number of propagated modes increases with thickness, and, therefore, the intermodal losses also increase.

With the dimensions and refractive indices of guiding and cladding layers, one should have several TE and TM modes. Usually, when trying to couple light from outside to a multimode waveguide a superposition of different modes is excited. Each excited mode has its own losses because of the different confinement factor.

So, loss measurements depend on excited modes and according to light coupling, the excited modes could be different and the loss difference does not correspond to the same excited modes. This induces errors in loss measurement.

Loni et al [5] have made strip waveguides with 0.5 and 2 μm guiding layer thicknesses. They found that by increasing guiding layer thickness from 0.5 to 2 μm, optical loss decreased from 14 to 6.7 dB/cm at 633 nm. Also, Balucani et al. [19] have prepared straight waveguides with different cladding layer thicknesses. They showed that by increasing buffer layer thicknesses from 0.8 to 2.5 μm, attenuation losses would decrease from 3 to 0.4 dB/cm at 633 nm. In an other work [15] Balucani et al. mentioned that for fabricating waveguides, double step anodization gives more homogeneous buffer layer and lower optical loss than single step anodization.

Our results show consistency with them. However, we prepared planar waveguides from PS multilayer and we showed that optical loss can be reduced in planar waveguides similar to strip waveguides. Also, our planar waveguides were prepared by focusing on variation of anodization time and their method was by variation of current density. We showed that variation of anodization time gives wide range of cladding and guiding layers thicknesses for fabrication of planar waveguides. This method improves significantly the waveguide preparation and characteristics.

4. Conclusions

Oxidized multilayer planar porous silicon waveguides have been fabricated. Optical losses were measured in the visible range (λ=633 nm) for different thicknesses of the guiding and cladding layers. It has been shown that by increasing the guiding and cladding thicknesses, optical loss decreases but still remains high at 7.35. As to our knowledge the approach of reducing the optical losses in the described multilayer waveguides by variation of the oxidation time of the different layers is a new topic.

Losses may be explained by several mechanisms. Part of the losses is due to the radiation towards the substrate and it diminishes by increasing the cladding layer thickness. There could be some rests of silicon that absorbs part of the light. The rest of loss is related to the Rayleigh scattering from the roughness that exists at different interfaces between guiding and cladding layers and it could be due to the current density fluctuations. By better controlling the current density during anodization, reduction of difference square of refractive index between guiding and cladding layers, reduction of emission modes numbers in waveguide, we can minimize the fluctuations and subsequently have reliable reduction in losses. Another proposal for loss reduction is anodization at low temperature. In this case the interface surface roughness of PS layers will diminish [13].

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