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# Thermo-optic properties of oxidised porous silicon impregnated with index-matching liquids for active optical components

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Oxidised porous layers (OPS) were impregnated with index-matching liquids with high thermo-optical coefficient. In this paper, we will present some results on the evolution of the refractive index of impregnated OPS, as a function of the temperature. Refractive index linearly varies with temperature. The thermo-optical coefficients of OPS/liquid composite material have been measured. They directly depend on the OPS porosity. We have shown that the experimental results are in agreement with the temperature variations of the refractive index calculated using the effective medium Bruggeman model. The existence of a switching temperature  $T_s$  in impregnated two-layer plane waveguides has been demonstrated and the possibility for obtaining thermo-optical switches is discussed.

**1 Introduction** During these last few years, different technologies have been developed to control the refractive index of porous silicon in order to achieve porous silicon passive optical waveguides [1–2]. These devices are based on planar or buried optical waveguides whose structure consists of a low porosity waveguiding layer between air above and a higher porosity cladding layer below. These waveguides are patterned using a micro-lithography technique. The core is typically less than 10  $\mu\text{m}$  across. It was previously demonstrated that porous silicon can be easily oxidised [3] and that from the loss viewpoint, the oxidised porous silicon (OPS) waveguides give the best results [2].

An inherent feature of porous silicon material is its open pore network. In principle, this can be used to modify the waveguide property in-situ, by filling the pores with other materials in. Depending on their properties, such materials could impart active function that could be used in optical components (switches, modulators...) or in sensors [4].

In this paper, the pores were filled with index-matching liquids in order to obtain a “composite” material. The thermo-optic coefficient (TOC),  $dn/dT$ , of these silicone-based liquids was previously measured [5] and is equal to  $-4.5 \times 10^{-4} \text{K}^{-1}$ . It is much lower than that of silica material ( $+8 \times 10^{-6} \text{K}^{-1}$ ). Consequently, it is reasonable to predict that TOC of the composite depends on volume fractions of the two components, i.e. liquid and porous silica. We will present the evolution of the optical properties of “composite” material as function of the temperature for different porosities. These variations are simulated by the Bruggeman model [6–7] in order to demonstrate the potentiality of these impregnated OPS materials for optical switch applications.

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**2 Experiment and model** Mesoporous Si films were fabricated by conventional anodisation of low resistivity (0.005  $\Omega$  cm) p-type (100)-oriented Si wafers in a HF(50%):H<sub>2</sub>O:C<sub>2</sub>H<sub>5</sub>OH (2:1:2) mixture. Anodisation conditions and characteristics of the films are shown in Table 1. The thickness (about 5  $\mu$ m) of each PS layer was controlled by the anodisation time. Si porosity has been measured by gravimetry. Then, these films were pre-oxidised at 300  $^{\circ}$ C for one hour in dry O<sub>2</sub> followed by a wet oxidation step at 900  $^{\circ}$ C for one hour. Refractive index of all the OPS layers, impregnated with index-matching liquid or not, were measured by “m-lines” method at  $\lambda = 633$  nm. The refractive index values were accurately measured (error less than 0.001). Sample temperature was regulated between 20  $^{\circ}$ C–70  $^{\circ}$ C during the m-lines measurements. We verified that the variation of the m-lines prism refractive index with temperature was negligible and that it did not affect measurements of the composite material refractive index. Moreover, the m-lines method is sensitive to the optical path, which includes both the refractive index and the physical layer thickness. The thermal expansion coefficient of SiO<sub>2</sub> has a low value. So in the range of considered temperature, the variation of the thickness is negligible in these measurements.

Porosities of not-impregnated OPS layers were deduced from refractive index measurements using the Bruggeman model [8]. There are several effective medium models that correspond to different possible microstructures of the random medium. The choice of the Bruggeman model gives a reasonably good estimate of the refractive index of not-impregnated and impregnated OPS layers.

The OPS layers were impregnated with three different index-matching liquid based silicones whose refractive index at room temperature and TOC are shown in Table 2. The pore filling was carried out by thermo-mechanic stresses. Liquids were deposited by a micro-syringe onto the surface of OPS films. Then, these samples were pressed between two slides and heated at 150  $^{\circ}$ C for one hour.

For comparison purposes, composite material refractive indices were calculated by means of the Bruggeman effective medium model [6–8]. This model symmetrically takes into account the two components according to the following equation:

$$C_{\text{SiO}_2} \frac{n_{\text{SiO}_2}^2 - n^2}{n_{\text{SiO}_2}^2 + 2n^2} + C_{\text{liq}} \frac{n_{\text{liq}}^2 - n^2}{n_{\text{liq}}^2 + 2n^2} = 0,$$

where  $n_{\text{liq}}$ ,  $n_{\text{SiO}_2}$  and  $n$  are the refractive indexes of the liquid, SiO<sub>2</sub> and “composite material” respectively;  $C_{\text{SiO}_2}$  and  $C_{\text{liq}}$  are the volume fractions and  $C_{\text{SiO}_2} + C_{\text{liq}} = 1$ .

In order to take into account the TOC of each component, the refractive index is equal to:

–  $n_{\text{liq}} = -4.5 \times 10^{-4} (T - 25) + n_{\text{amb}}$  where  $n_{\text{amb}}$  is refractive index of this liquid at 25  $^{\circ}$ C for an index-matching liquids.

–  $n_{\text{SiO}_2} = 8 \times 10^{-6} (T - 25) + n_{\text{SiO}_2\text{amb}}$  where  $n_{\text{SiO}_2\text{amb}}$  which is equal to 1.460 at  $\lambda = 633$  nm, is the refractive index at 25  $^{\circ}$ C for silica.

**3 Results and discussion** We measured the refractive index of sample F55 impregnated by the index matching liquid L1.504 at room temperature. The refractive index of oxidised porous silicon without impregnation is equal to 1.20. After impregnating by L1.504, its refractive index increases and is equal to 1.480. So the index-matching liquid has infiltrated the pores because the refractive index of composite material is higher than the refractive index of silica. The theoretical value deduced from the Bruggeman model, for 55% porosity silica layer totally impregnated with this liquid is 1.481. So we can consider that the pores are nearly completely filled.

**Table 1** Anodisation conditions and film properties.

film number	anodisation current density (mA cm <sup>-2</sup> )	porosity of porous-Si film	refractive index of OPS film	porosity of OPS film*
F 39	50	73%	1.20	39%
F 55	100	80%	1.27	55%

\* deduced from effective medium model calculation.

**Table 2** Index-matching liquid properties.

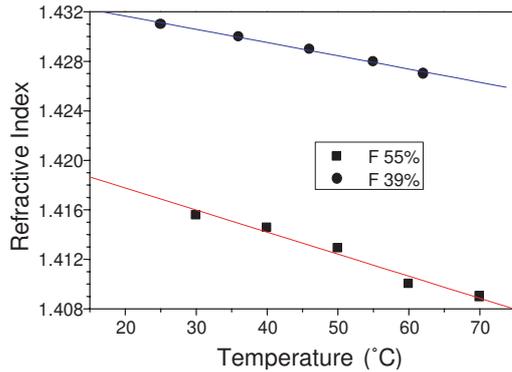
liquid number	refractive index at 25 °C	TOC (K <sup>-1</sup> ) [5]	switching temperature $T_s^*$ (°C)
L1.403	1.403	$-4.5 \times 10^{-4}$	-106
L1.467	1.467	$-4.5 \times 10^{-4}$	35
L1.504	1.504	$-4.5 \times 10^{-4}$	112

\* deduced from effective medium model calculation.

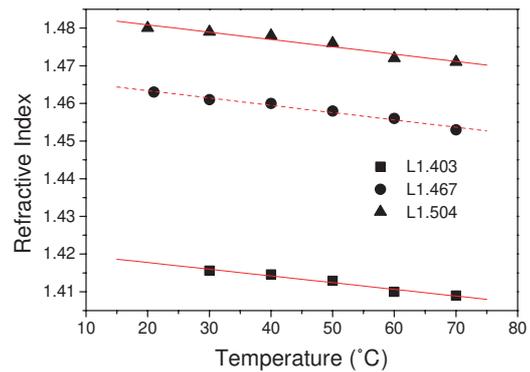
Then, we measured the refractive index of two films (F39 and F55), which have different porosities, impregnated with the same index-matching liquid L1.403. As shown in Fig. 1, the refractive index of the composite decreases linearly as function of the temperature in the considered temperature range. The slope, which corresponds to the composite TOC, is equal to  $-1.07 \times 10^{-4} \text{ K}^{-1}$  and to  $-1.98 \times 10^{-4} \text{ K}^{-1}$  respectively for F39 and F55 samples, which have porosities equal to 39% and 55%. So the composite material TOC depends on the porosity of the oxidised porous silicon layer. It rises as the porosity increases. Indeed, the thermo-optic coefficient of these liquids is equal to  $-4.5 \times 10^{-4} \text{ K}^{-1}$ . It is much lower than that of silica material ( $+8 \times 10^{-6} \text{ K}^{-1}$ ). Consequently the TOC of the composite mainly depends on the liquid volume fraction.

Figure 2 exhibits the evolution of the refractive index of composites as a function of the temperature for some samples that have the same porosity but the index-matching liquids are different. The liquids have the same TOC and the samples have the same porosity. We observe that the three curves have the same slope, i.e. the same TOC. It is equal to  $-1.94 \times 10^{-4} \text{ K}^{-1}$ . This value is lower than the index-matching liquids TOC because of the effect of oxidised porous silicon matrix.

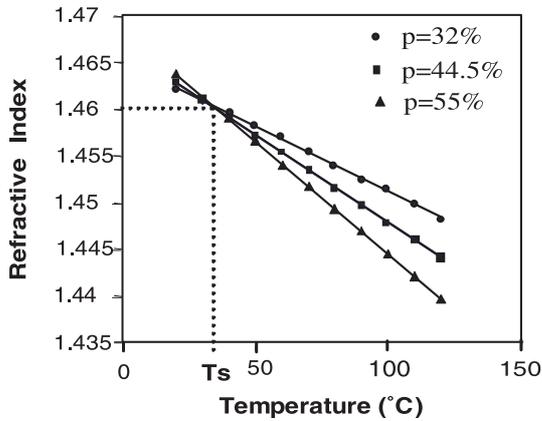
Variation of the calculated refractive index as a function of the temperature  $T$  in the oxidised porous silicon layer impregnated with the three liquids is reported in Fig. 3. It can be noticed that the curves have the same linear behaviour as the experimental data in the considered temperature interval. The “composite material” TOC (the slope) depends on the porosity of oxidised porous silicon layer. We can clearly see that the TOC rises when the porosity increases. Figure 3 also shows that the composite formed with the OPS having the highest porosity has the greatest refractive index value at low temperatures and the smallest one at high temperatures. These properties can be used for active optical



**Fig. 1** (online colour at: [www.interscience.wiley.com](http://www.interscience.wiley.com)) Evolution of the refractive index of OPS films impregnated with an index matching liquid L1.403, as a function of temperature for two values of OPS porosity: 39% and 55%. The refractive index was measured by m-lines at  $\lambda = 633 \text{ nm}$ . The measured TOC are  $-1.07 \times 10^{-4} \text{ K}^{-1}$  and to  $-1.98 \times 10^{-4} \text{ K}^{-1}$  for impregnated layers F39 and F55 respectively.



**Fig. 2** (online colour at: [www.interscience.wiley.com](http://www.interscience.wiley.com)) Evolution of the refractive index of oxidised porous silicon films F55 (55% porosity) impregnated with three different index-matching liquids (L1.403, L1.467 and L1.504) as a function of temperature. The refractive index was measured by m-lines at  $\lambda = 633 \text{ nm}$ .

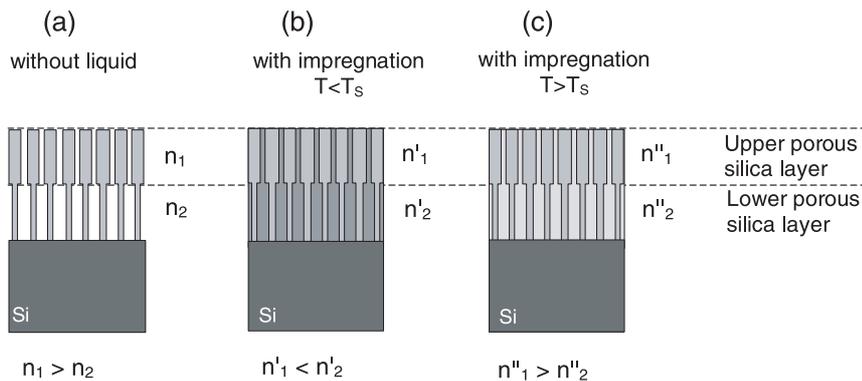


**Fig. 3** Calculated refractive index of OPS impregnated with an index-matching liquid vs. temperature for three different values of OPS porosity. In this case the refractive index of the liquid is 1.467 at 25 °C.

liquid refractive index is the same as the one of silica ( $n = 1.460$ ). In this example,  $T_s$  is reached at 35 °C. Switching temperature  $T_s$  is characteristic of each liquid, as is shown in Table 2.

The planar waveguide fabrication method is based on the modification of porous silicon refractive index by varying the current density during anodisation. Such a waveguide has a two-layer structure (Fig. 4). The low porosity layer is obtained by applying the lower current density, which results in a high refractive index whereas the higher porosity layer with a lower refractive index is obtained by applying a higher current density.

Without liquid infiltration, the index  $n_1$  of the upper OPS layer is higher than the one of the lower layer because of the difference in porosity. When the layers are impregnated with a liquid, whose index is higher than that of silica at temperature lower than  $T_s$ , the refractive index  $n'_1$  of the upper layer becomes lower than the index  $n'_2$  of the lower layer. In this case, the light guiding properties of the upper layer are lost. At temperatures higher than  $T_s$ , the refractive index of the upper layer once more increases. The light guiding properties of the planar waveguide are restored.



**Fig. 4** (online colour at: [www.interscience.wiley.com](http://www.interscience.wiley.com)) Changes in the refractive indexes in a two layer planar wave guide, before filling the pores with an index-matching liquid whose refractive index is higher than the one of silica at ambient temperature. a) Without liquid the upper layer, which has the lower porosity is the guiding layer. b) For the two impregnated layers at temperature lower than the switching temperature  $T_s$ , the refractive index of the upper layer becomes the lowest. The waveguiding properties of the upper layer are lost. c) At temperature higher than  $T_s$ , the refractive index of the upper layer once more is the highest. The waveguiding properties of the planar waveguides are restored.

**4 Conclusion** OPS layers infiltrated by index-matching liquids have been investigated by measuring their refractive indexes as a function of the temperature. We have shown that the pores are almost completely filled by comparing the refractive index of impregnated sample with the value calculated from Bruggeman effective medium model. The TOC of composite material mainly depends on the liquid volume fraction and increases with porosity. In the temperature range, the refractive index of the material composite decreases linearly as the temperature increases.

These results agree with the values predicted by the Bruggeman model. Moreover this model shows that the index difference between two layers of different porosities, which have been filled by the same liquid, varies with the temperature and that the sign of this difference is inverted at the critical temperature  $T_s$ . The modification of the refractive index profile in the waveguide by changing the temperature can induce a phase shift and/or variation in guided light intensity and even mode extinction. By this method, a thermo-optical switch could be achieved.

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