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# Protective coatings for interference filters made of porous silicon

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We have tested different kinds of protective coatings for interference filters made of porous silicon (PS). While thin $SiO_2$ films deposited by various kinds of chemical vapor deposition (CVD) provided reasonable protection against moisture, best results were obtained for Plexiglas coatings. They left the filter characteristics essentially unchanged and also delivered negligible ageing effects in a period of 180 days.

**1 Introduction** It is well known that multilayers can be made from porous silicon [1]. This allows the simple and cost effective fabrication of superlattices serving as optical interference filters in a wide spectral range. The remaining absorption of the Si limits these filters in the visible part of the spectrum (VIS) to reflectors which can be made e.g. as small band or laterally graded mirrors. In the infra-red (IR) regime where absorption of PS becomes negligible also transmission filters can be produced. Nevertheless they have to be made from free standing PS-films (or extremely thin Si-wafers) as the free charge carriers in the underlying highly doped substrate would prevent the transmission of light. The Research Center Jülich (FZJ) has set up a project (named ***PorSiLux***) which aims at the development of commercially available optical filters from PS. In this context we have routinely produced transmission filters with area as large as 3 $cm^2$ and minimum thickness as low as 30 μm.

For practical applications one has to consider that, due to its huge surface area, PS is vulnerable in particular to oxidation, humidity or other kinds of environmental influences which might change the filter characteristics. So an effective protection of the filters will be required.

The effect of oxidation on PS interference filters has already been reported in the literature [2]. After preparation of the superlattice a thin layer of native oxide readily forms at ambient air, typically 10 nm thick. This layer slowly grows thereby leading to an ageing of the filters. We have extended the investigation to almost 180 days. Fig. 1 shows the shift of the central wavelength of three different Bragg mirrors with time. There is a decrease of about 3.6% per year which seems to be negligible for many applications. For higher stability protective measures have to be taken. As suggested in Ref. [2] the thickness of the $SiO_2$ layer can be increased by raising the oxidation temperature and/or duration. Eventually this completely converts PS to porous $SiO_2$. Obviously this leads to stable filters. However, due to the considerably smaller index of refraction of $SiO_2$ compared to that of Si, they shift to smaller wavelengths. This then poses a design problem on the filters as the material parameters of the resulting multilayer system like layer thickness, porosity and the related index of refraction are not known initially. As a consequence a protection is required which essentially conserves the characteristics of the filters.

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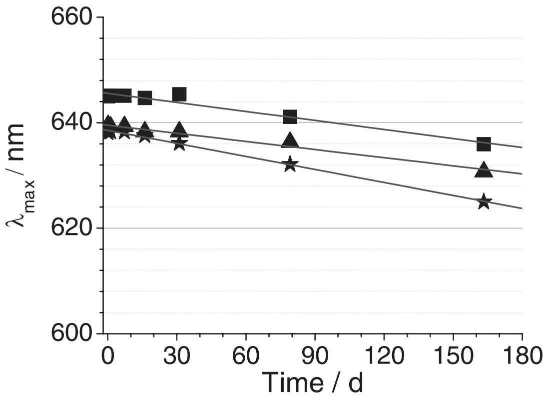


**Fig. 1** Influence of ambient air on three different Bragg filters. The filters are characterized by their maximum wavelengths $\lambda_{max}$.

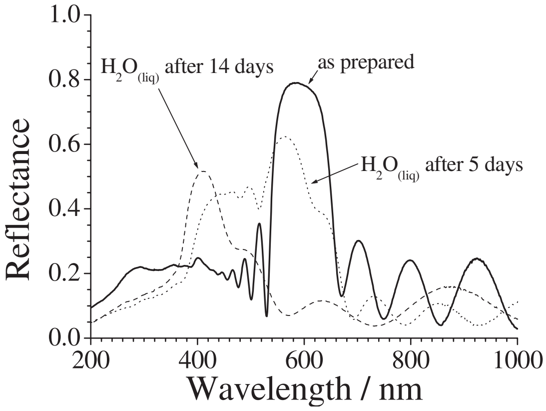


**Fig. 2** Influence of liquid water on the reflectance spectrum of an unprotected Bragg filter.

**2 Experimental details** The PS samples were formed on highly doped (10 mΩ cm) $p^+$-type substrates leading to mesoporous Si. Further details can be found in Ref. [2]. The optical spectra were recorded using a Perkin–Elmer Lambda900 spectrometer. In order to test environmental influences the samples were exposed to ambient air for long times (up to 180 days) or simply stored in de-ionized water. The latter served as an accelerated test for the influence of moisture.

Capping layers of $SiO_2$, i.e. layers which cover the top surfaces of the filter but do not penetrate into the pores were deposited in two different ways of CVD [3]: plasma enhanced chemical vapor deposition (PECVD) and low pressure chemical vapor deposition (LPCVD). In the latter case Tetraethylorthosilicate (TEOS) is used to form a silicon dioxide layer on the surface of the filter under low pressure and high temperature conditions. The deposition rate is a function of the partial pressure of TEOS and the deposition temperature. As a third technique spin-coating of Polymethylmethaacrylat (PMMA = Plexiglas) was employed. For the CVD techniques the layer thickness is controlled by the deposition rate and time, for the spin-coating by the spinning rotation speed.

**3 Experimental results** Figure 2 shows the influence of water on the reflectance spectrum of an unprotected Bragg mirror. After 5 days immersion into water the spectrum is severely disturbed and after 14 days it is almost completely corrupted. Obviously for applications in wet environment optical filters made of PS have to be protected.

**3.1 PECVD coating** As an example the effect of a 100 nm thick layer of $SiO_2$ deposited by PECVD on a Bragg reflector is shown in Fig. 3. Relative to the as prepared state the additional layer has only a minor effect on the central wavelength of the mirror. As a beneficial side effect the layer forms an antireflection coating [4] which considerably reduces the side lobes. This effect could be optimized by adopting the layer thickness. After storage of the filter for 14 days in water the spectrum remains essentially unchanged except for a slight shift to smaller wavelengths. This shift is continuous in time. Apparently the filter protection is not perfect. However, for most practical application in humid environment this type of coating seems to be sufficient, as first tests of filters in a relative humidity of 99% show. It should be noted that the PECVD technique is equally well applicable to free standing filters where both sides have to be coated. This is shown in Fig. 4 where the transmission spectrum of a free standing Bragg mirror designed at a wavelength of 1700 nm, i.e. in the near-IR, is plotted. Deposition of 150 nm PECVD-$SiO_2$ on each side does not influence the spectrum.

**3.2 LPCVD coating** The long term behavior of two different Bragg mirrors coated with LPCVD-$SiO_2$ is shown in Fig. 5 where the central wavelength of the reflection peak is plotted vs. time. Filter 1 (upper

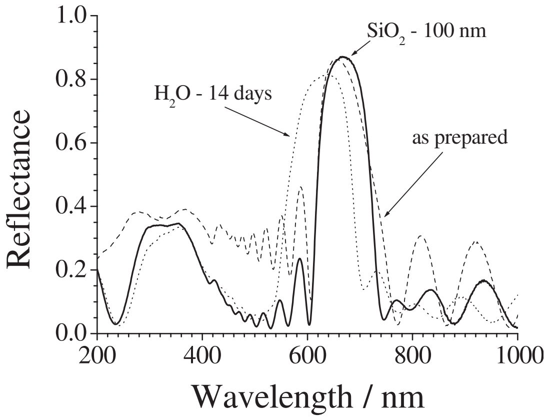


**Fig. 3** Influence of 100 nm $SiO_2$-coating (PECVD) on a Bragg filter and the protection against water.

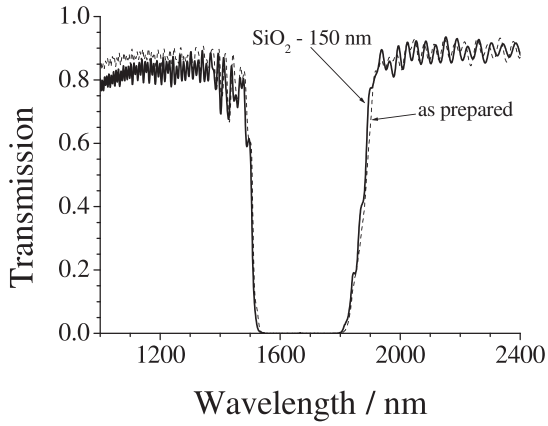


**Fig. 4** Transmission spectra of a free standing IR Bragg filter: As prepared and after PECVD coating with 150 nm $SiO_2$ on both surfaces.

curve) was stored in wet air (relative humidity 99%) at room temperature whereas filter 2 (lower curve) was stored at ambient air. The capping layer introduces a 5% reduction of the wavelength, which in the case of the wet environment recovers to a large extend within a few days. Apart from these initial effects both filters are then perfectly stable during the time of the investigation (170 days). A layer thickness as low as 50 nm is sufficient for the protection of the filters. Unfortunately this deposition technique is not applicable to thin free standing filters because of mechanical stresses introduced both by the elevated temperature and the low pressure.

**3.3 PMMA spin-coating** In order to test the effect of PMMA coatings a 300 nm thick PMMA layer (corresponding to a speed of 3000 rpm) was deposited onto a Bragg mirror. Figure 6 shows the reflectance spectrum of this sample in the as prepared state, immediately after deposition of the PMMA, and after storage in water for 14 days. One can see that PMMA coating has a negligible influence on the position of the spectrum and provides an effective protection against water.

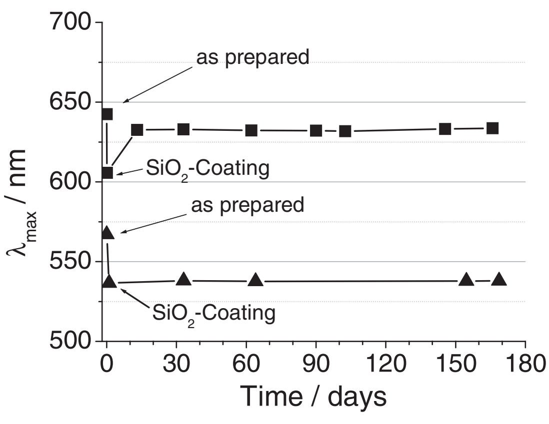


**Fig. 5** Long-time investigation of the shift of the maximum wavelength for two $SiO_2$-coated (LPCVD) Bragg filters. Filter 1 (upper curve) had a layer thickness of 50 nm and was stored in wet air of 99% relative humidity. Filter 2 (lower curve) had a layer thickness 100 nm and was stored in ambient air at room temperature.

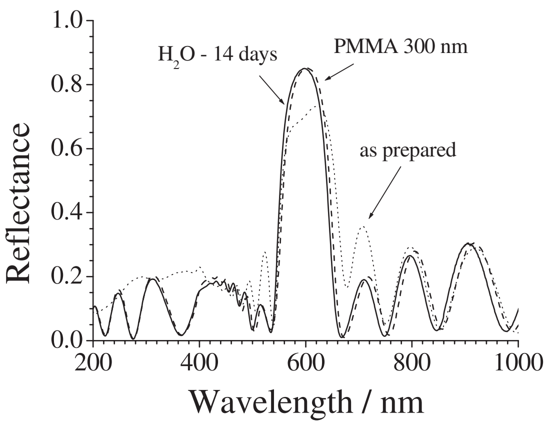


**Fig. 6** Influence of a 300 nm PMMA-coating on a Bragg filter and its protection against water.

**4 Conclusions** Aiming at the commercialization of optical filters made of porous Silicon both the long term stability of such filters as well as several types of protective layers for applications in wet environments have been explored.

The long term stability in ambient air turned out to be sufficient without any further measures except when high precision filters are required. The ageing due to surface oxidation was found to be as low as 3.6% per year.

In wet environments thin capping layers of $SiO_2$ or Plexiglas provide good protection. The filters are only slightly (30 nm for LPCVD coating) or even negligibly affected within the accuracy of the measurements (for PECVD and PMMA coatings). The long term stability after LPCVD coating is excellent. With respect to protection against moisture best results were obtained for PMMA coating.

For free standing filters only PECVD was found to be applicable. The application of PMMA spin-coating technique has not yet been tested.

As a beneficial side effect suitable choice of the layer thickness leads to an antireflection coating thereby improving the quality of the filters.

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