Porous silicon based optical multilayers

PhD thesis

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Premises

Due to the discovery (1990) of the photoluminescent properties, porous silicon (PS) has come to the front of the current research. Since that time, owing the huge effort it was also pointed out that PS is not only able to emit but even filter the light. For the latter purpose we have to realize optical multilayers having a proper refractive index profile. In case of PS it can be reached easily applying a programmed anodisation current control. Porous silicon multilayer (PSM) has promising applications in the field of chemical and biological sensing, where the porous nature and high specific surface are utilised.

The Research Institute for Technical Physics and Materials Sciences has been doing research in the field of porous silicon for more than 15 years, however, multilayer structures has been investigating only since 2000. Early results connecting to PSM-s are published in my diploma thesis [S9].

Targets

Our research institute (MFA) has set the goal towards the investigation of porous silicon multilayers. The long-range target is a lab-on-chip device, which enables to identify different chemical and biological substances.

My present work can be considered as a starting research towards the above-mentioned sensing purposes, where the refractive index of the pore filling liquid to be sensed is determined by the resonance peak position of a Fabry-Perot PSM interference filter.

The first task was to optimize the PSM structures by means of different optical characterization techniques and to survey the physical and practical limitations of the filter quality. The next step was the demonstration of the above-mentioned sensing principle.

Another goal of present research was to reveal novel research fields and preparation techniques in connection with PSM providing a solid base for further projects in the future.

Investigation methods

Sample preparation:

Varying anodiation current technique

Experimental techniques:

- Ex situ / in situ perpendicular spectroscopic reflectometry
- Spectroscopic ellipsometry (SE)
- Scanning electron microscopy (SEM)
- Gas ad-/desorption measurement (BET, BJH)

Mathematical methods:

Thin film calculation by means of scattering matrix method

New results

1. *I have developed a novel method for the optical modelling of porous silicon multilayer stacks, where the unknown parameters in case of less number of layers (e.g. $N \leq 16$ Bragg mirror) were determined by the direct evaluation of the spectroscopic ellipsometric spectra [S1, S8]. In case of higher number of layers the ellipsometric evaluation was supported by SEM image analysis [S2]. The optical model was rectified in all cases by an independent measurement method: spectroscopic reflectometry at normal angle of incidence (Chapter 6.1, 6.2).*

2. *First in the literature, I have carried out in situ spectroscopic reflectometry measurement on porous silicon multilayers during electrochemical preparation. The measurement data were fitted at first by an ideal than by an asymmetric model where the porosity changes in time. By the latter, refined version I have proved that the undesirable blueshift is caused by photoelectrochemical etching [S3]. The in situ monitoring supported by proper optical model enables the fine tuning of the optical element by direct feedback controlling of the technological parameters (etching time, current density) (Chapter 6.3).*

3. *Considering the theoretical and experimental aspects I have developed a novel optimization technique. By means of this method I have prepared Fabry-Perot interference filters with improved optical performance operating in the wavelength range of λ = 600-900 nm. The maximal porosity contrast of ΔP = 28 % was found at stable, room temperature environment (substrate resistivity: 0,005 Ωcm, HF concentration: 19,5 w%, etching current density function: j_H/j_L = 8 mAcm^{-2} / 350 mAcm^{-2}), which corresponds to a reflective index difference of Δn^D = 1,02. According to the in situ measurement introduced in the second thesis point the optimal optical structure for both simple and double FP microcavity consists of 24 layers ($H[LH]^5LL[HL]^6$, and $[HL]^3HLL[HL]^4HLL[HL]^6$, respectively). The realized simple microcavity interference filter can be described with a finesse of F = 268 and with a resonance peak of $\Delta\lambda_{FWHM}$= 3 nm [S4] (Chapter 6.4).*

4. *I have experimentally proved that there is a linear relationship in a relatively wide range (n=1,36-1,54) between the static response of the liquid filled porous structure (resonance peak position) and the unknown quantity (refractive index of the liquid). In case the volume of the liquid changes in the porous structure the in situ method described in the second thesis point is applicable for the investigation of the kinetics [S6]. By using three-component effective medium model, I have pointed out that the resolution of the porous silicon*

based refractometer is lower than expected ($\Delta n \approx 0{,}001$), which can be elucidated by the existence of interfacial layer (possibly SiOx) on the pore walls [S5] (Chapter 7).

5. *I have worked out a novel technique for preparation of 3D and lateral structures on silicon substrate. In this case the porous silicon multilayer was formed in 3D microgrooves having steep sidewalls. Applying the suggested technique I have attempted to fabricate air-core waveguide and high density optical grating (> 2000 lines/mm) [S7] (Chapter 8).*

Publications in connection with the PhD dissertation

[S1] J. Volk, M. Fried, O. Polgár, I. Bársony
Optimisation of porous silicon based passive optical elements by means of spectroscopic ellipsometry, Phys. Stat. Sol. (a), **197**, 208 (2003)

[S2] J. Volk, M. Fried, A. L. Tóth, I. Bársony
The ideal vehicle for optical model development: porous silicon multilayers Thin Solid Films, **455-456**, 535 (2004)

[S3] J. Volk, K. Ferencz, J. J. Ramsden, A. L. Tóth, I. Bársony
In situ observation of the evolution of porous silicon interference filter characteristics, to be published in Phys. Stat. Sol. (a)

[S4] Zs. Szabó, Gy. Kádár, J. Volk
Band gaps in photonic crystals with dispersion
COMPEL, **24**, No. 2, 521 (2005)

[S5] J. Volk, T. Le Grand, I. Bársony, J. Gombkötő, J. J. Ramsden
Porous silicon multilayer stack for sensitive refractive index determination of pure solvents, J. Phys. D: Appl. Phys., **38** 1313 (2005)

[S6] J. Volk, J. Balázs, A. L. Tóth, I. Bársony
Porous silicon multilayers for sensing by tuneable IR transmission filtering, Sensors and Actuators B, **100**, 163 (2004)

[S7] J. Volk, N. Nagy, I. Bársony
Laterally stacked porous silicon multilayers for subquart micron period UV gratings, to be published in Phys. Stat. Sol. (a).

[S8] Fried M., Lohner T., Petrik P., Polgár O., Volk J.:
Ellipszometria a vékonyréteg-technológiában,
Fizikai Szemle, 6, 200 (2003)

Publications not in connection with the PhD dissertation

[S9] Volk János: Pórusos szilíciumból készített Bragg-reflektorok optimalizálása (BME, 2001)

[S10] K. Molnár, T. Mohácsy, A.H. Abdulhadi, J. Volk, I. Bársony
On the nature of metal-porous Si-single crystal silicon (MPS) diodes, Phys. Stat. Sol. (a), **197**, 446 (2003)

[S11] P. Petrik, É. Vázsonyi, M. Fried, J. Volk, G. T. Andrews, A. L. Tóth, Cs. S. Daróczy, I. Bársony, J. Gyulai
Optical models for the ellipsometric characterisation of porous silicon structures, to be published in Phys. Stat. Sol. (a)

[S12] N. Nagy, J. Volk, A. Hámori, I. Bársony
Submicrometer period silicon diffraction gratings by porous etching, to be published in Phys. Stat. Sol. (a).

[S13] Zs. Vízváry, J. Volk, Cs. Dücső
Elasticity modulus measurement of silicon-nitride, Proceedings of the 4th Conference on Mechanical Engineering, pp. 215-219, Gépészet 2004, 2004., Budapest

Exploitation of the results

The success of the following projects was promoted by the work presented:

OTKA: Electroluminescent porous silicon structures (T033094)
Electromagnetic waves in artificial periodic structures (T046696)

Joint Project Grand (Royal Society of London):
Fabry-Perot multilayers for chemical sensing