

Maria Figurova - Peter Gaso - Dusan Pudis*

MICRO-OPTICS FOR LAB-ON-A-CHIP

Lab-on-a-chip (LOC) is nowadays one of the most interesting topics at the field of biomedicine and biosensing applications. Mainly devices that integrate a detection system right into a chip with microfluidic network are highly desirable. In this article we will present Mach-Zehnder interferometer created in IP-Dip photoresist by direct laser lithography system for laboratory on chip applications. Interferometer will be later used for biosensing application as integrated micro-optical device inside a microfluidic chip. Measurements of transmission characteristics of water solution with different sugar concentration show promising ability of the Mach-Zehnder interferometer to detect different liquids on principle of refractive index change.

Keywords: Mach-Zehnder interferometer, PDMS, DLW lithography, refractive index.

1. Introduction

In the field of biomedicine and health care is sustained effort to create compact and cheap device for a quick detection of a biologic sample. Lab-on-a-chip is a kind of device that can provide multiple operations on a small chip, similar to conventional laboratory working with biological samples. LOC works on various principles of detection, where optical detection is becoming more and more popular. Implementation of photonic sensing devices such as resonator based sensors [1], crystal-based sensors [2] or interferometers [3] enable us to detect a change in a concentration of biological samples. The detection principle of most of such photonic LOC is based on evanescent field where the biological sample is placed on the one top of resonator arms [4] based on interference change [5].

Unfortunately many sensing devices for LOC have been demonstrated as proof-of-concept experiments, only few work as biosensing applications so far. Usually LOC biosensors working on optical principles are using micro-optical components like silicon-based resonators. In this paper we present functionality of various Mach-Zehnder interferometers (MZI) made in IP-Dip photoresist, which is a polymer material. In general, for LOC biosensors are polymer materials advantageous solution because of their thermal and chemical stability and affordability. One of the most popular materials - polydimethylsiloxane (PDMS) is even biocompatible and appropriate for optical measurements because of its optical properties. A wide range of applications for this material is mainly focused on the process of replica molding for microfluidic parts of chip [6].

In this paper we present fabrication of specially designed MZI created in IP-Dip photoresist by direct laser writing (DLW) lithography system. IP negative photoresists designed for commercial platform of 3D laser lithography provide high resolution and mechanical stability of structures in the micrometer and submicrometer scale [7].

Specially designed MZI's (Figs. 2a, 3a, 4a) were created in IP-Dip photoresist for liquids with different concentrations and refractive indices.

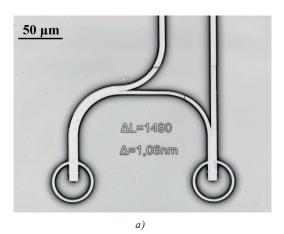
2. Microstructure fabrication

For the structure fabrications, commercial direct laser writing system Nanoscribe was used. This kind of laser lithography enables us to create 3D structures on a principle of two-photon absorption and polymerization in a volume of photoresist. By this method microfluidic and micro-optic component for LOC can be created in high resolution quality (down to 500 nm). Platform of Nanoscribe DLW system allows 3D structures patterning in negative IP-Dip photoresist. For creating our structures we used mode of Dill configuration [8].

We designed various kinds of MZI and the whole process of creating micro-optic structures involves few simple steps. Firstly IP-Dip photoresist was drop-cast on the top of cleaned glass substrate and then of MZI started. For the developing of printed structures, PGMA (polymer glycol monomethyl ether acetate) was used to dissolve unexposed parts. Finally, the substrate was rinsed in isopropyl and deionized water and dried by air. Structure morphology (in case of size parameter larger than 250 μm) and

Department of Physics, Faculty of Electrical Engineering, University of Zilina, Slovakia E-mail: figurova@fyzika.uniza.sk

^{*} Maria Figurova, Peter Gaso, Dusan Pudis



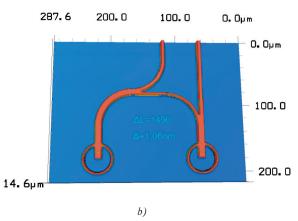


Fig. 1 a) Detail picture of MZI from laser mode of confocal microscope and b) 3D image for structure adhesion checking

proper adhesion of glass was checked by confocal microscope (Fig. 1). Optical properties were analysed by spectral transmission measurements using different surrounding cladding liquids.

3. Experiment and results

Optical properties of prepared MZI's were analysed by spectral transmission measurements. Experimental setup for spectral transmission measurements consists of optical spectral analyzer (OSA) Anritsu MS9710B with resolution of 0.1 nm coupled to the output of the MZI using single mode optical fiber (SMF). For light coupling the input and outputs of MZI were specially designed and created by inclined planes as 45° micromirrors. Guiding of the lights in these micro-mirrors works on the contrast of refractive indices of used photosensitive IP-Dip resist and surrounding air or liquids. The MZI structure has squared cross section with dimension of $8~\mu m$. The refractive index of

applied liquid changes the transmission characteristics of MZI. As the power source for transmission measurements, the light emission diode with central emission wavelength at 1550 nm was used.

The measured properties were compared with simple calculation of free spectral range λ_{FSR} , taking into account the parameters of prepared MZI. Interference spectral dips are distributed according to

$$\lambda_{FSR} = \frac{\lambda^2}{n\Delta L} \tag{1}$$

where ΔL represents arm difference of MZI and n = 1.52 is refractive index of IP-Dip photoresist (for $\lambda = 1550$ nm).

Theoretical value of λ_{FSR} for measured MZI is 1 nm and 1.06 nm. By implementation of such MZI in LOC device, we expect good detection properties of fluids based on refractive index change of cladding. Prepared MZI devices will be implemented in

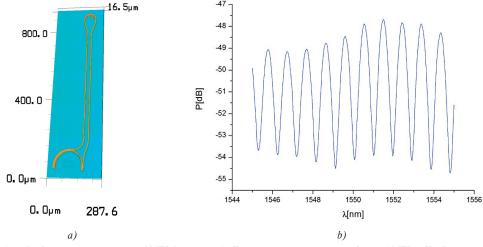


Fig. 2 a) Confocal microscope image of MZI 1 structure. b) Transmission characteristics of special MZI in IP - Dip photoresist for air as surrounding cladding, where $\Delta L = 1604 \mu m$.

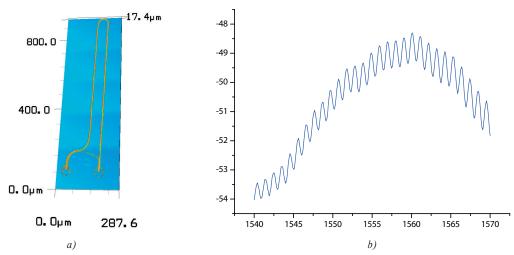


Fig. 3 a) Confocal microscope image of MZI 2 structure. b) Transmission characteristics of special MZI in IP-Dip photoresist for air as cladding surrounding, where $\Delta L = 1690 \mu m$

next microfluidic systems with the aim of optical characterization of assays in LOC.

We were focusing on the testing of three various designs of MZI's (MZI1, MZI2 and MZI3), where the surroundings are air, water and different concentrations of sugar dissolved in water. MZI1 with long detection arm was created for better contact of MZI with liquids during measurement (Fig. 2a). Transmission characteristic (Fig. 2b) of MZI1 in air surrounding shows 5 dB dips. During experiments we found applicability problems connected with hydrophilic surface of IP-Dip photoresist. Coupling mirrors cannot be sealed with water during application of liquids because of the lowering reflectance what leads to lost signal. Another design brings improvement by ring protection (Fig. 3a).

MZI2 with mirror ring protection showed to be better solution for liquid measurements. Drop-casting in this case was much easier. This structure shows smaller dips, less than 1 dB (Fig. 3b) caused probably by structure discontinuities in the long arm.

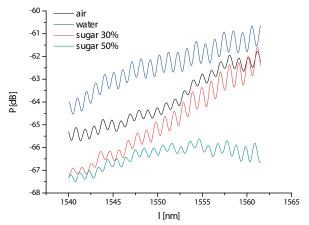


Fig. 4 Transmission characteristics of MZI2 for air, water and water solutions with different sugar concentrations

On the prepared MZI2, we were testing different water solutions of sugar with concentrations 0 %, 30 % and 50 % and air (Fig. 4). With increase of sugar concentration the refractive index of water/sugar solution increases together with free spectral range λ_{FSR} dips in the measured signal. λ_{FSR} from the measured transmission characteristics (Fig. 4) was estimated λ_{FSR} = 1.016 nm for air surrounding.

From the measured spectral transmission characteristics, we estimated the λ_{FSR} for the measured water/sugar solutions. The dependence of λ_{FSR} on sugar concentration is shown in the Fig. 5.

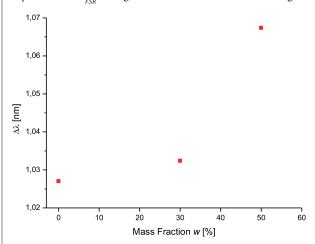


Fig. 5 Dependence of FSR of transmission peaks on mass fraction of aqueous mixture of sugar

The last tested design for MZI3 (Fig. 6a) keeps the mirror protection and has smoother curves mainly for the reference arm and shorter sensing arm. It was proved by transmission characteristic (Fig. 6b), that the dips for surrounding air are better, almost 15 dB. From the transmission characteristics we estimated $\lambda_{FSR} = 1.105$ nm.

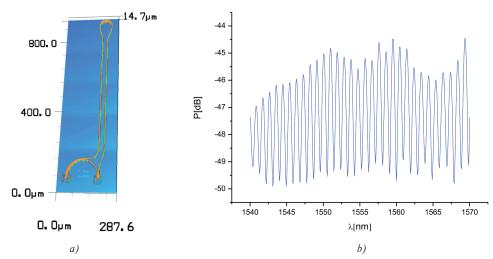


Fig. 6 a) Confocal microscope image of MZI3 structure. b) Transmission characteristics of special MZI3 in IP-Dip photoresist for air as cladding surrounding, where $\Delta L = 1604 \mu m$

According to the obtained results, we suppose the improvement of resolution of this sensing method for precise measurements of water solutions of different chemicals. However, it needs optimisation process of MZI design, especially the core dimensions.

4. Conclusions

In this paper we presented new concept of MZI prepared by laser lithography in IP-Dip photoresist. Designed and prepared MZI's show effective interference with significant dips in spectral transmission characteristics. For different designs we achieved

15 dB dips. Effect of different water/sugar solutions was tested for sensing properties of the prepared MZI's. In the present state, only high concentrations of sugar resolved the increase of free spectral range in measured transmission spectral characteristics. More precise measurements need to also optimize the core dimensions of MZI's.

Acknowledgement

This work was supported by the Slovak National Grant Agency under the projects No. VEGA 1/0491/14 and 1/0278/15 and the Slovak Research and Development Agency under the project No. APVV-16-0129.

References

- [1] WANG, J., YAO, J., LEI, T., POON, A. W.: Silicon Coupled-Resonator Optical-Waveguide-Based Biosensors Using Light-Scattering Pattern Recognition With Pixelized Mode-Field-Intensity Distributions. *Scientific Reports*, 4, 1-9, 2014.
- [2] YAN, H., ZOU, Y., CHAKRAVARTY, S., YANG, CH.-Y., WANG, Z., TANG, N., FAN, D., CHEN, R. T.: Silicon On-Chip Bandpass Filters for the Multiplexing of High Sensitivity Photonic Crystal Microcavity Biosensors. *Optic Express*, 20, 7195-7205, 2012.
- [3] DANTE, S., DUVAL, D., SEPULVEDA, Y., GONZALEZ-GUERRERO, A. B., SENDRA, J. R., LECHUGA, L. M.: Integrated Optical Devices for Lab-On-A-Chip Biosensing Applications. *Applied Physics Letters*, 106, 1-5, 2015.
- [4] ESTEVEZ, M. C., ALVAREZ, M., LECHUGA, L. M.: Integrated Optical Devices for Lab-On-A-Chip Biosensing Applications. *Laser Photonics Reviews*, 6, 463-487, 2011.
- [5] MARTINCEK, I., PUDIS, D.: A Theoretical Study of the Temperature Sensor Based on the LP₀₁- LP₀₂ Intermodal Interference in Optical Fiber with a Liquid Core. *Communications Scientific Letters of the University of Zilina*, 10(2), 10-13, 2010.
- [6] Sylgard® 184 Silicone Elastomer [online]. Date: 20/09/2016. Available: http://www.dowcorning.com/DataFiles/090276fe80190b08. pdf.
- [7] IP Photoresists, Nanoscribe [online]. Date: 29/09/2016. Available: http://www.nanoscribe.de/files/1814/0662/4393/IP-Resist_IP-Dip web.pdf.
- [8] GASO, P., JANDURA, D., PUDIS, D.: Fabrication of 2D and 3D Photonic Structures Using Laser Lithography. Proc. of SPIE, 1-5, 2016.