# Tuning refractive index sensing properties of micro-cavity in-line Mach-Zehnder interferometer with plasma etching

Monika Janik[a,*], Marcin Koba[b,c], Wojtek J. Bock[a], Mateusz Śmietana[b,**]
[a]Centre de recherche en photonique, Université du Québec en Outaouais, 101 rue Saint-Jean-Bosco, Gatineau, QC J8X 3X7, Canada; [b]Institute of Microelectronics and Optoelectronics, Warsaw University of Technology, Koszykowa 75, 00-662 Warsaw, Poland; [c]National Institute of Telecommunications, Szachowa 1, 04-894 Warsaw, Poland;

## ABSTRACT

This work presents an application of reactive ion etching (RIE) for an effective tuning of the spectral response and the refractive-index (RI) sensitivity of the micro-cavity in-line Mach-Zehnder interferometer (μIMZI). The μIMZIs were fabricated using femtosecond laser micromachining in a standard single-mode fiber as a form circular holes with a diameter of 54 μm. The application of RIE with $SF_6$ and $O_2$ used as reactive gas allows for an efficient and well-controlled etching of the fabricated structure. The process resulted in cleaning the bottom of the micro-cavity and smoothening of its sidewalls. In transmission measurements, the effect of the plasma processing was observed as an increase in both spectral depths of the minima and RI sensitivity of the structure, as well as improved wettability of the micro-cavity surface, which made the measurements faster and easier.

**Keywords:** optical fiber sensors, refractive index sensing, Mach-Zehnder interferometer (MZI), microstructure fabrication, femtosecond laser micromachining, refractive ion etching (RIE), plasma processing

## INTRODUCTION

The concept of Mach-Zehnder interferometer is very well known and there are many ways to implement it in a fiber [e.g. 1,2,3]. When the interferometer is supposed to be used for sensing purposes, especially for determination of properties of liquids, typically a cavity needs to be formed in a fiber. Out of many available methods for cavity fabrication, femtosecond (fs) laser micromachining where extremely short laser pulses with high peak power are applied, offers many advantages, which includes negligible heating of the beam-exposed area resulting in very little damage in the working area and ability to produce small, well-defined shape on and in the fiber. That is why a fs laser system was recently used to fabricate several micro-cavity in-line MZI (μIMZI) structures [4,5]. The light propagating through the fiber core splits at the cavity sidewall [6]. Both parts of the light, penetrating the cavity and the other, undisturbed in the core, interfere at the other sidewall. When the liquid sample fills the cavity, the sensing path leads through it. Depending on properties of liquid, i.e., its refractive index (RI), the interference take place differently. Besides advantages of μIMZI structure such as portability, high sensitivity and the possibility to measuring very small volumes of liquid, there are also some challenges. A very small size of the cavity makes its cleaning difficult. The small pieces of glass remain after the fabrication process and can reduce the volume of the cavity as well as the area of the sensing surface, which in turn can affect the RI sensitivity. What is more, there is a problem with a filling of the cavity, especially with high-density liquids. Any increase in wettability of the cavity surface would allow for faster, more repeatable and accurate measurements.

Etching of the optical fiber cladding has already been applied for enhancing the RI sensitivity of optical fiber sensors. There are two techniques which are widely used, namely those using liquid etchants (e.g., wet etching in HF acid [7, 8]) or vapor-based etchants, i.e., plasma etching [9, 10, 11]. In this work, we used reactive ion etching (RIE) where in addition to chemical etching, the particles are accelerated towards the sample surface and physically remove the material by an ion bombardment on material's surface [12].As etching reagents typically fluorides, e.g. $SF_6$ or $CF_4$, are used. In fluorine forming plasmas, the F atoms are principal etchant, but also is well known that the addition of oxygen can increase the etch rate [13]. Oxygen has a dual role in this process: enhances the production of the etchant, F atoms and occupies the active silicon etch site, which retards the etching reaction but significantly increases the hydrophilicity of the surface.

*monikaa.janik@gmail.com ** M.Smietana@elka.pw.edu.

25th International Conference on Optical Fiber Sensors, edited by Youngjoo Chung, Wei Jin, Byoungho Lee, John Canning, Kentaro Nakamura, Libo Yuan, Proc. of SPIE Vol. 10323, 103238M · © 2017 SPIE · CCC code: 0277-786X/17/$18 · doi: 10.1117/12.2265755

The plasma processing is also accurate, more precise than any wet etching process, especially when anrrow trenches are processed (e.g., [7]) and in contrast to chemical reagents, nontoxic and noncorrosive. Might be especially convenient for modification of as small cavities as a couple of picoliters in volume. In this paper, we exposed to RIE a μIMZI structure, which in comparison to earlier reported works, is fabricated as a circular hole (Fig.1). The structure was fabricated solely with a femtosecond laser, and then modified with $SF_6$ and $O_2$ plasma-based post-processing, aiming for improving the wettability of the structure sidewalls, clean and better define the flat bottom of the micro-cavity and in consequence increase the overall sensitivity of the sensor.

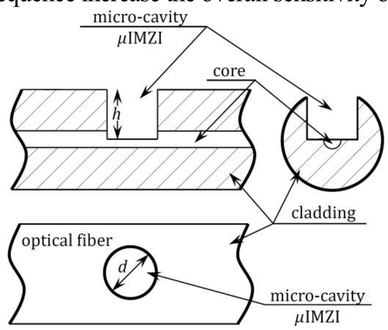

Figure 1. Schematic representation of the μIMZI.

# METHODOLOGY

### 2.1 Manufacturing of the μIMZI structures

Structures in the form of cylindrical holes were fabricated in standard Corning SMF28 fibers. The micromachining process was performed using a Solstice Ti:Sapphire fs laser operating at λ = 795 nm. The fiber was irradiated by 82 fs pulses. The system was working with a repetition rate of 10 kHz. In order to make the micro-cavity, the laser beam was directed into a suitably designed micromachining setup based on the Newport μFab system. The system was equipped with a 20x lens, with NA = 0.30. Fiber transmission was monitored during the process with an NKT Photonics SuperK COMPACT supercontinuum white light source and a Yokogawa AQ6370C optical spectrum analyzer. The fabrication process was controlled with software developed in-house.

### 2.2 Plasma etching

The RIE process has been performed using Oxford PlasmaPro NGP80 system. The plasma obtained with the $SF_6$ flow of 30 sccm and $O_2$ flow of 100 sccm, pressure 100 mTorr and power 250 W. The temperature during the processes was set to 20°C and each etching was 3 min.-long.

### 2.3 μIMZI analysis

The optical transmission of the μIMZI for the refractive index sensitivity measurement was, as well as in the case of micromachining, monitored in the spectral range of 1100-1700 nm using a broadband laser light source NKT Photonics SuperK COMPACT and an optical spectrum analyzer Yokogawa AQ6370C. The set of water/glycerin solutions whose $n_D$ varied in the range of 1.3330-1.4000 RIU was used to perform the sensitivity measurements of the μIMZIs. The $n$ of the solution was measured using a digital refractometer VEE GEE PDX-95 as a reference.

# RESULTS

The structure with diameter d = 54 μm was chosen to illustrate the effect of etching on properties of μIMZI. The sample underwent four consecutive 3-min.-long etching processes. The transmission spectra after each process when the micro-cavity was filled with water is shown in Fig. 2. Comparison of spectra reveals that the etching is well observed in the case of a μIMZI structure in terms of the depth of minimum in the spectrum (by over 10 dB), while barely noticeable in terms of minima shift. The transmission spectra of the samples for the $n$ varying from 1.3333 to 1.4000 RIU in the micro-cavities before and after plasma post-processing are shown in Fig. 3a and 4a respectively. In Fig. 3b and 4b, the corresponding spectral locations of minima are plotted for various $n$. The points corresponding to each minimum are linearly approximated with the least squares method and the values of sensitivity in different $n$ regions. The sensitivity is designated by a shift of minima in transmission spectra caused by aqueous solutions with various $n$ filling the micro-cavity. It can be seen that the etching process affected the spectra and had a significant impact on the overall RI sensitivity. As it is depicted in Fig. 4b, in comparison to Fig. 3b, the sensitivities after plasma post-processing in both the RI ranges, namely

1.3333 to 1.3500 RIU and $n$ = 1.3600 to 1.3900 RIU, are higher than before plasma treatment and reach over 12,000 and 15,000 nm/RIU, respectively. Also, the traced minima in the µIMZ spectrum look smoother and more pronounced (Fig. 4a).

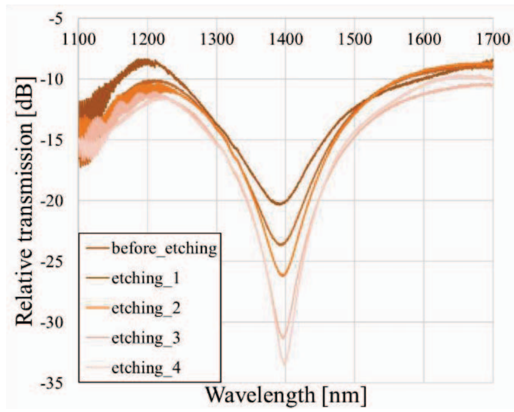

Fig. 2. The effect of each consecutive etching process on the µIMZI transmission spectra when water filled the micro-cavity.

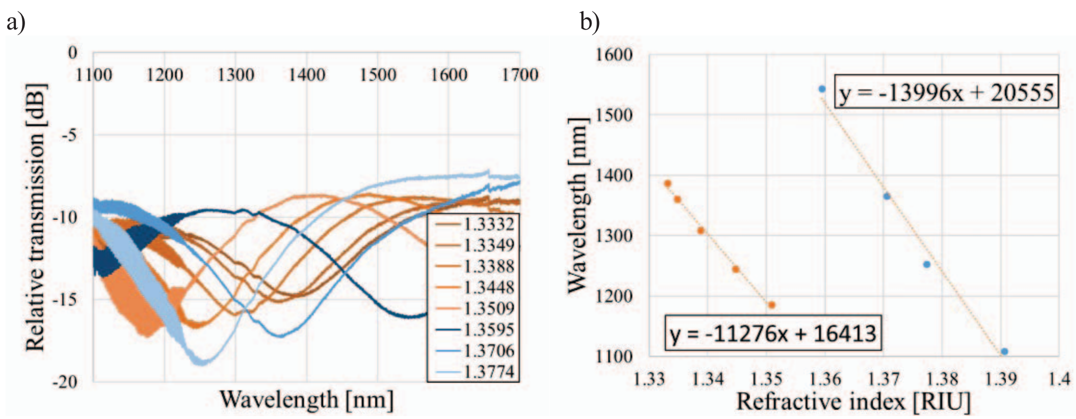

Fig. 3. Spectral response (a) and transmission minimum wavelength (b) of the µIMZI before the plasma etching for aqueous solutions in the micro-cavity with $n$ ranging from 1.33 to 1.40 RIU.

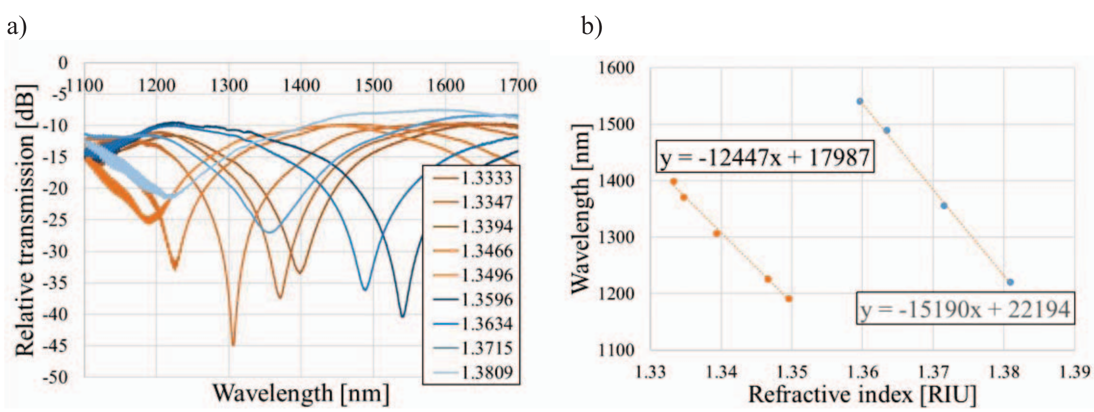

Fig. 4. Spectral response (a) and transmission minimum wavelength (b) of the µIMZI after all the plasma etching processes for aqueous solutions in the micro-cavity with $n$ ranging from 1.33 to 1.40 RIU.

In must be also noted that the surface after plasma treating was much more hydrophilic than before any processing. The effect enabled for easier introduction of the liquid into the micro-cavity, especially those with higher refractive indices. Furthermore, we have found that plasma-based etching used for micro-cavity processing is much more precise than earlier reported wet etching with HF acid [4]. The process described here did not cause any further uncontrolled etching of the surface and is strictly controlled by process time.

Additionally, we have obtained good cleaning the cavity surface which is very important, since the residual material in form of glass scarps does not disturb the further measurements. This issue might have a crucial impact on the accuracy and repeatability of the further measurements.

## CONCLUSIONS

In this paper, we have demonstrated a highly sensitive refractive index sensor, based on a micro-cavity in-fiber Mach-Zehnder interferometer. It was fabricated with a femtosecond laser and then underwent plasma post-processed. The processing resulted in a well-visible increase in the spectral depth of the interference minima, what made the minima wavelength highly defined for any measurements. The process also had an impact on refractive index sensitivity which was increased to over 15 000 nm/RIU. What is more an application of RIE for hydrophilization of the surface allows for the effective introduction of the liquid into the micro-cavity. The obtained improvements in functional properties of the interferometers allow for their future applications for investigations on sub-nanoliter volumes of liquids.

## ACKNOWLEDGEMENTS

This work was supported by the Polish National Science Centre (NCN) as a part of 2014/13/B/ST7/01742 project and Natural Sciences and Engineering Research Council of Canada (NSERC) under Industrial Research Chair program.

## REFERENCES

[1] Ngyuen, L.V.; Hwang, D.; Moon, S.; Moon, D.S.; Chung, Y.J. “High temperature fiber sensor with high sensitivity based on core diameter mismatch” Opt. Express, 16, 11369-11375 (2008)

[2] Frazao, O.; Silva, S.F.O.; Viegas, J.; Baptista, J.M.; Santos, J.L.; Kobelke, J.; Schuster, K. “All fiber Mach-Zehnder interferometer based on suspended twin-core fiber” IEEE Photon. Technol. Lett., 22, 1300-1302 (2010)

[3] Smietana M., Brabant D., Bock W.J., Mikulic P., Eftimov T., “Refractive-index Sensing with Inline Core-cladding Intermodal Interferometer Based on Silicon Nitride Nano-coated Photonic Crystal Fiber”, Journal of Lightwave Technology 30(8), 1185-1189 (2012)

[4] Hu T. Y., Wang Y., Liao C. R., and Wang D. N., “Miniaturized fiber in-line Mach-Zehnder interferometer based on inner air cavity for high-temperature sensing,” Opt. Lett. 37 (24), 5082-5084 (2012)

[5] Jiang L., Zhao L., Wang S., Yang J., and Xiao H., “Femtosecond laser fabricated all-optical fiber sensors with ultrahigh refractive index sensitivity: modeling and experiment”, Opt. Express 19 (18), 17591-17598, (2011)

[6] Dębowska A.K., Koba M., Janik M., Bock W.J., Śmietana M. “Increased sensitivity of femtosecond laser micro-machined in-fiber Mach-Zehnder Interferometer for small-scale refractive index sensing”, Proc. SPIE 9916, 99160Q (2016)

[7] Sun, X. Dong, Y. Hu, H. Li, D. Chu, J. Zhou, C. Wang, and J. Duan, “Highly sensitive refractive index fiber inline Mach-Zehnder interferometer fabricated by femtosecond laser micromachining and chemical etching,” Sensors Actuators A. Phys., 230,11–116 (2015)

[8] Myśliwiec M., Grochowski J., Krogulski K., Mikulic P., Bock W.J., Śmietana M., ”Effect of Wet Etching of Arc-Induced Long-Period Gratings on Their Refractive Index Sensitivity”, Acta Physica Polonica, 124, 3, 521-524 (2013)

[9] Śmietana M., Koba M., Mikulic P., and Bock W.J., "Measurements of reactive ion etching process effect using long-period fiber gratings," Opt. Express 22, 5986-5994 (2014)

[10] Śmietana M., Koba M., Mikulic P., Bock W.J., “Tuning properties of long-period gratings by plasma post-processing of their diamond-like carbon nano-overlays”, Measurement Science and Technology, 25 114001 (2014)

[11] Śmietana M., Koba M., Mikulic P., Bock W.J., “Combined Plasma-Based Fiber Etching and Diamond-Like Carbon Nano-overlay Deposition for Enhancing Sensitivity of Long-Period Gratings”, Journal of Lightwave Technology – in press

[12] Tzeng Y. and Lin T. H., “Dry etching of silicon materials in SF6 based plasmas. Roles of N2O and O2 gas additives,” J. Electrochem. Soc. 134, 2304–2309 (1987).

[13] Mogab C. J., Adams A. C., and Flamm D. L.,” Plasma etching of Si and $SiO_2$—The effect of oxygen additions to $CF_4$ plasmas” Journal of Applied Physics, 49, 3796-3803 (1978)