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Full length article

[INVITED] Sensing properties of micro-cavity in-line Mach-Zehnder interferometer enhanced by reactive ion etching ☆

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A B S T R A C T

In this work, we discuss an application of reactive ion etching (RIE) for enhancing the sensing properties of a micro-cavity in-line Mach-Zehnder interferometer (μIMZI). The μIMZI was fabricated using femtosecond laser micromachining in a standard single-mode fiber as a circular hole with a diameter of 54 μm. Next, the structures underwent two kinds of RIE using as reactive gases: sulfur hexafluoride (SF_6) and oxygen (O_2) mixtures (SF_6/O_2) or O_2 itself. When RIE with SF_6/O_2 was applied, it allowed for an efficient and well-controlled etching of the fabricated structure at nanometers level observed as an increase in spectral depths of the minima in the μIMZI transmission spectrum. A similar RIE process with O_2 alone was ineffective. The well-defined minima obtained with the SF_6/O_2 RIE significantly improved the resolution of measurements made with the μIMZI. The effect was demonstrated for high-resolution refractive index (RI) measurements of liquids in the cavity. The result of the RIE process was to clean the micro-cavity bottom, increase its depth, and smooth its sidewalls. As an additional effect, the wettability of the micro-cavity surface was improved, making the RI measurements faster and more repeatable. Moreover, we demonstrated that RIE with SF_6/O_2 results in more stable wettability improvement than when O_2 is applied as a reactive gas.

1. Introduction

The concept of the Mach-Zehnder interferometer (MZI) is very well known, and there are many ways to implement it in an optical fiber [1–3]. For sensing purposes, especially for determination of properties of liquids, a cavity may to be formed in a fiber and filled with an analyte. Among the many available methods for cavity fabrication, femtosecond (fs) laser micromachining, where extremely short laser pulses with high peak power are applied, offers numerous advantages, including negligible heating of the beam-exposed area. This low heating results in limited damage to the target area and makes it possible to fabricate small, well-defined shapes on and in the fiber. That is why fs laser micromachining systems have been used to fabricate various micro-cavity in-line MZI (μIMZI) structures, e.g., [4,5]. The concept of a μIMZI takes advantage of the fact that light propagating through the fiber core splits at the cavity's sidewall into two parts, one propagating in the core and the other penetrating the cavity. The cavity length is a sensing architecture that rarely exceeds 100 μm. At the far sidewall of the cavity, the two beams interfere resulting in an interference pattern observed at the fiber output [6]. When an investigated analyte fills the cavity, the sensing path leads through it, and the type of interference depends on the properties of the analyte. For refractive index (RI), for example, a change in the analyte's RI is observed as a spectral shift of the spectrum at the fiber output. It must be noted that to obtain a well-defined interference pattern on the fiber output, i.e., spectrally deep minima, a precisely adjusted micro-cavity depth is required. This condition mainly determines the light splitting rate.

Besides the certain advantages of the μIMZI structure, such as portability, high sensitivity, and the possibility of examining minimal volumes, there are also some challenges with this sensing concept. The petite size of the cavity makes it difficult to clean. Even tiny pieces of glass remaining in the cavity after the fabrication process can reduce its volume while also reducing the sensing area. Such residues can affect the sensitivity of the device and the repeatability of the measurements. Additionally, the glass residues can cause light scattering and optical signal distortion, which in turn increase the insertion loss of the μIMZI. Filling the cavity with a liquid may trigger further problems, especially in the case of high-density liquids with high viscosity. All of these issues indicate

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that for faster, more repeatable, and more accurate measurements, a high degree of control over the cavity depth is required. Efficient cleaning of the cavity, especially after fabrication of the structure, and improved wettability of the cavity's surface is also essential. The majority of the problems mentioned can be solved if the μIMZI is etched – a process which may be part of the fabrication post-processing.

Etching of optical fibers has already been applied as a means of enhancing the sensing properties of optical fiber sensors. Two techniques are widely employed, namely, those using liquid etchants, e.g., wet etching in hydrofluoric (HF) acid [7,8], or those based on dry etching, i.e., plasma-based etching [9–11], which includes reactive ion etching (RIE). In general, when RIE is applied, in addition to the chemical etching by plasma-activated ions, the ions are also accelerated towards the surface of the sample, and physically remove the material at nanometers level by a high-energy bombardment of the surface [12]. As etching reagents of silicon-based materials, fluorides such as sulfur hexafluoride (SF_6) or carbon tetrafluoride (CF_4), are typically used. In fluorine plasma, the F ions are the principal etchant, but addition of oxygen (O_2) might be used to increase the etching rate [13]. The application of O_2 has a dual role in this process: it enhances the production of the etchant, i.e., F ions, and it also occupies the active silicon etching sites, not only delaying the etching reaction but also significantly increasing the wettability of the etched surface [13]. It is worth mentioning that plasma processing is highly accurate (at nanometers level) and more precise than any other wet etching process, especially when narrow trenches are processed. In contrast to most wet chemical reagents, plasma is relatively nontoxic and noncorrosive [14]. Thus, RIE processes were found to be far superior to wet etching for modification of small cavities.

In this paper, we present the results for RIE of the μIMZI. The examined structure was fabricated with a fs laser, and then modified with SF_6/O_2 or O_2 based RIE post-processing, the aim is to improve the wettability of the structure's sidewalls, enable thorough cleaning and provide better definition of the flat bottom of the micro-cavity, thus enhancing the overall sensing properties of the device. We compared for the first time two RIE processes, namely SF_6/O_2 and O_2 alone as reactive gases, taking into consideration influence of the RIE process on transmission and μIMZI performance, as well as wettability and stability of the surface properties after the RIE treatment. What is more we compared for the first time the effect of RIE process with micromachining process itself, and RIE etching effect with wet etching using HF acid.

2. Experimental details

2.1. Manufacturing of the μIMZI structures

Structures in the form of cylindrical holes (d = 54 μm, h = 62 μm) were fabricated in standard Corning SMF28 fibers (Fig. 1A). 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. Fused silica glass has low absorption at 795 nm, and therefore linear absorption of the laser radiation does not occur when the glass is irradiated by the laser beam [15]. The system was operated with a repetition rate of 10 kHz. 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 20× lens (NA = 0.30). The laser pulse energy was equal to 6 nJ. Fiber transmission was monitored during the process with an NKT Photonics SuperK COMPACT supercontinuum white light source and a Yokogawa AQ6370C optical spectrum analyzer working with 10^{-2} nm resolution. The fabrication process was controlled by software developed in-house.

2.2. RIE processes

The RIE processes were performed using the Oxford PlasmaPro NGP80 system. During the experiment, the plasma was obtained at a pressure of 100 mTorr and RF power of 250 W. The flows of the SF_6 and O_2 gases were set to 30 and 100 sccm, respectively. The

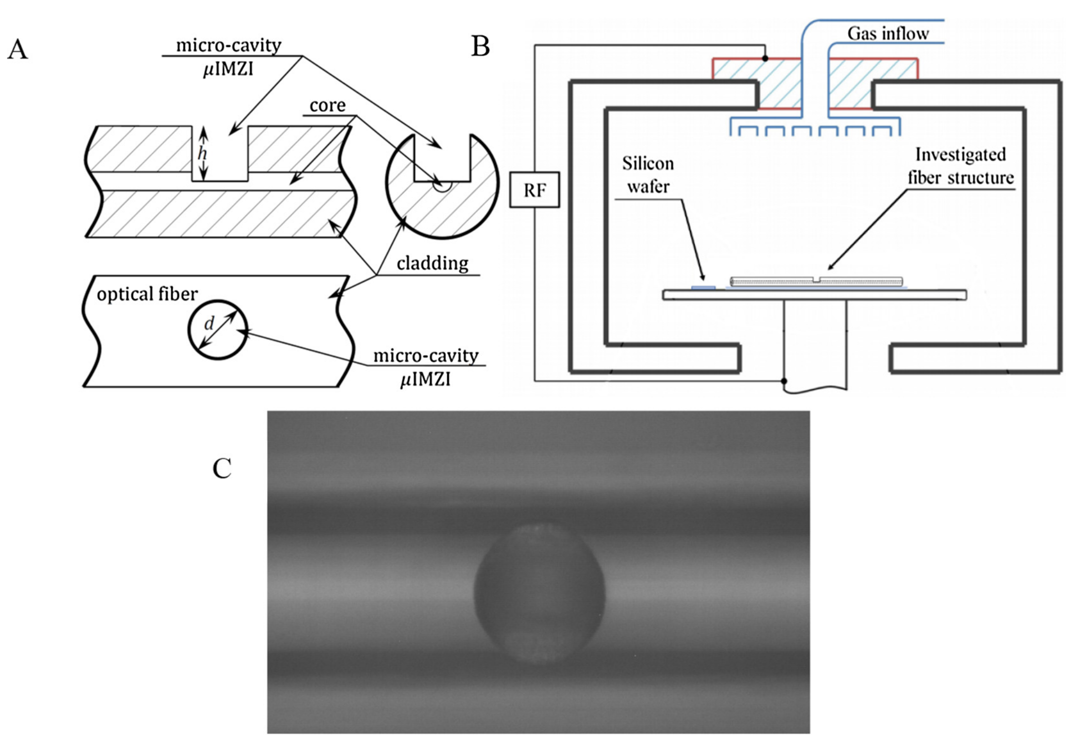

Fig. 1. (A) Schematic representation of the μIMZI structure. The diameter of the micro-cavity is indicated by *d*, depth by *h*. (B) The μIMZI and SiO_2/Si wafer placement in the RIE process chamber. (C) Microscope image of the top view of the microstructure.