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FIBER MACH-ZEHNDER INTERFEROMETER MICRO-CAVITY LENGTH ADJUSTMENT IN TENS OF NANOMETERS FOR SENSING SYSTEM MINIATURIZATION

Fiber Mach-Zehnder interferometer (FMZI) micro-cavity length adjustment in tens of nanometers by chemical etching for refractive index sensing system miniaturization was firstly reported. This etching process is capable of adjusting the length of micro-structure at the order of tens of nanometers, and achieving such high precision without involving complicated and expensive nano-fabrication facilities. The chemical etching opens up the fiber micro-cavity and redshifts its resonant wavelength at the rate of 3.6 nm/min, upon an estimated etching speed of 41.5 nm/min. The shift of the micro-cavity's resonant wavelength close to the LD's emitting wavelength is achieved by such precise length adjustment for sensing system miniaturization. The miniaturized FMZI is experimentally applied to the refractive index sensing for ethanol solutions. The measured transmission is in good linearity with regards to the solution index and the sensitivity is -12.8 dB for 0.0043 RIU (refractive index unit) difference in this study. The miniaturized FMZI index-sensing system can be portable and operating at a fast speed, which is well suited for practical field applications.

Keywords: fiber Mach-Zehnder interferometer, precise length adjustment, chemical etching, fiber sensor.

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ВОЛОКОННЫЙ МИКРОПОЛОНАТЕЛЬНЫЙ ИНТЕРФЕРОМЕТР МАХА – ЦЕНДЕРА С РЕГУЛИРОВКОЙ ДЛИНЫ В ДЕСЯТКАХ НМ ДЛЯ МИНИАТЮРНЫХ СИСТЕМ ДАТЧИКОВ

Впервые предложен способ регулирования длины микрорезонатора волоконного интерферометра Маха – Цендера (FMZI) в десятки нанометров путем химического травления для миниатюризации системы измерения показателя преломления. Этот процесс травления способен регулировать длину микроструктуры порядка десятков нанометров и достигать высокой точности без использования сложного и дорогостоящего нанопроизводства. Химическое травление открывает микрополость волокна и смещает его резонансную длину волны в красную область со скоростью 3,6 нм/мин при расчетной скорости травления 41,5 нм/мин. Смещение резонансной длины волны микрорезонатора, близкое к длине волны излучения ЛД, достигается за счет такой точной регулировки длины для миниатюризации чувствительной системы. Миниатюрный FMZI экспериментально применяется для измерения показателя преломления растворов этанола. Измеренная передача имеет хорошую линейность относительно показателя раствора, а чувствительность составляет -12,8 дБ для разницы 0,0043 RIU (единицы показателя преломления) в этом исследовании. Миниатюрная индексная система FMZI может быть портативной и работать с высокой скоростью, что хорошо подходит для практического применения в полевых условиях.

Ключевые слова: волоконный интерферометр Маха – Цендера, точная регулировка длины, химическое травление, волоконный датчик.

Introduction

Fiber sensing systems have attracted tremendous attention for their high sensitivity, immunity to electromagnetic disturbance, and accessible to hazardous situations to monitor different physical, chemical, and biologic parameters [1-5]. Among fiber sensors, the fiber Mach-Zehnder interferometer (FMZI) that is based on a fiber micro-cavity is particularly interesting for its high sensitivity of ~104 nm/RIU for index sensing as well as its ultra-small sensing zone that is just over several tens of μm2. Such properties are suitable to monitor the refractive index for micro-scale liquids (or parameters related to the refractive index) with high sensitivity. However, field application for the micro-cavity-based FMZI sensing is still impractical as the system operation is cumbersome, expensive, and slow. Based on the principle of resonance shift, the system mandates at least a broadband light source (BBS) and an optical spectrum analyzer (OSA) for spectral analysis [6]. Meanwhile, the sensing speed is slowed down by the process of spectral scanning. Therefore, it is important for index sensing systems to be miniature, portable, affordable, and fast for perspectives in practical field applications.

One solution is to convert from the spectrum to intensity monitoring [7-10], and to utilize the linear transmission part of spectra for sensing. However, for the micro-cavity-based FMZIs, the resonant wavelength cannot be pin-pointed at an exact wavelength due to the fabrication limits; while to use a commercial laser diode (LD) for intensity monitoring, the FMZI's resonant wavelength needs to closely match the LD's emission bandwidth. Therefore, it is critical to develop a process that can precisely adjust the resonant wavelength of micro-cavity, down to the nanometer scale, so that it can be in the near neighborhood to that of the LD. Such an ultra-precise adjustment for the FMZI device has never been reported to date.

In this work, we present a miniaturized FMZI sensor based on the precise fiber micro-cavity length adjustment by chemical etching, to the order of several tens of nanometers. The chemical etching expands the fiber micro-cavity length and red-shifts the resonant wavelength of transmission at the finest rate of 3.6 nm/min, corresponding to an estimated etching speed of 41.5 nm/min. Following the initial micro-cavity fabrication, this fine etching red-shifts the resonance dip close to the emitting wavelength of a LD, and the transmitted intensity at this operating wavelength serves as the signal for index sensing. The miniaturized FMZI sensor thus composes of one LD and a pair of PDs, instead of the common bulky equipment. In the experimental demonstration of the miniature FMZI sensor, the transmitted intensity is found in good linear relationship with respect to the solution index, and the sensitivity is -12.8 dB for 0.0043 RIU difference in the study.

1. Fine tuning the interference length of FMZI

1.1. The principle of resonant wavelength shift

The micro-cavity-based FMZI device is fabricated by a laser-induced micro-plasma process with a nanosecond pulsed fiber laser [6]. The micro-cavity structure of the FMZI is illustrated in Fig. 1. After the laser ablation, the micro-cavity is chemically etched by the HF solution for >30 minutes to remove all plasma processed area, the carbon residue on the cavity wall and smoothen the surface roughness. The FMZI's transmission spectrum is monitored by the BBS and OSA. The etching process and the transmission measurement are set up in a cleanroom with stable temperature and humidity control (25°C and 55%) to avoid environment disturbance.

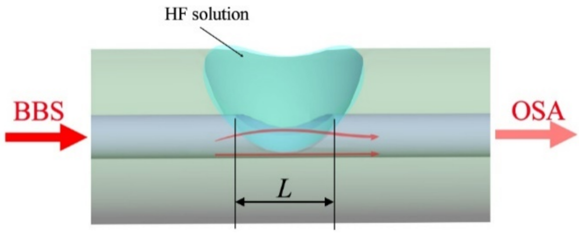


Fig. 1. Precise adjustment of the micro-cavity length by chemical etching

For the interference, assume the micro-cavity/interference length of the FMZI device is L and the resonant wavelength is thus expressed as [11]

$$\lambda_m = \frac{2\pi L \Delta n_{eff}}{(2m+1)\pi - \varphi_0}, \tag{1}$$

where λ_m is the resonant wavelength at the mth order interference minimum and φ_0 is the initial phase. For $\Delta n_{eff} = n_{core} - n_{ext}$, where n_{core} and n_{ext} are the effective indices of the core and the external material filling the micro-cavity, respectively. The length L can be calculated from the transmission spectra as

$$L = \frac{\lambda_m \lambda_{m+1}}{(\lambda_m - \lambda_{m+1})\Delta n_{eff}}. \tag{2}$$

Assume the increment of micro-cavity length is dL that leads to the resonance shift, the derivative of m^{th} order resonance wavelength regarding L is

$$\frac{d\lambda_m}{dL} = \frac{2\pi \Delta n_{eff}}{(2m+1)\pi - \varphi_0} = \frac{\lambda_m}{L}. \tag{3}$$

Also assume a chemical etching process gradually chips away the cavity surface and enlarge the interference length L, the etching speed v can be obtained from Eq. 3

$$v = \frac{dL}{dt} = \frac{dL}{d\lambda_m} \cdot \frac{d\lambda_m}{dt} = \frac{L}{\lambda_m} \cdot \frac{d\lambda_m}{dt}. \tag{4}$$

*Eq.*4 thus indicates that the etching speed can be derived from the FMZI's transmission spectra and the rate of resonance wavelength shift.

As stated earlier, the fabrication process of laser ablation and initial HF etching, in general, cannot precisely locate the FMZI's resonance wavelength to be within the LD's emission bandwidth. Thus, a follow-up fine-tuning process is required to precisely adjust the micro-cavity length L for its resonant wavelength shift. In fabrication processes with the femtosecond (fs) lasers, the FMZI devices usually end up with relatively large length variation [12, 13] and the capability for very fine length control hasn't been presented to date. Although sub-50 nm nanofluidic channels have been fabricated in silicate glasses by fs laser direct writing [14], it is still unproven if the fs lasers can stably achieve the fabrication accuracy down to the order of tens of nanometers. On the other hand, etching-assisted process in fabricating micro/nanostructures with hard materials offers the ability to precisely control the structural dimension at the nm-level [15-19]. Therefore, it points the direction to apply etching-assisted method in precise adjustment of the micro-cavity length of FMZI devices.

1.2. The micro-cavity length adjustment by chemical etching

Hereby in the follow-up process, a second HF etching has been executed to precisely adjust the micro-cavity length. As a small amount of HF solution is dripped into the fiber micro-cavity, it will gradually chip away the wall of micro-cavity and slowly expand the interference length, resulting in the resonant wavelength shifting accordingly.

In the first step, HF solution (10 μL, concentration of 8%) is dripped into the micro-cavity to etch for one minute, and then rinsed away with deionized water. The transmission spectrum is monitored after the FMZI sample dries out. Such an etching and testing process are repeated 5 times for the FMZI sample. To study how the HF concentration affects the etching speed, the HF solution concentration of 6%, 4% and 2% are selected to repeat the complete etching processing upon FMZI micro-cavity sample, respectively. The transmission spectra of FMZI samples processed with different HF concentrations and etching time are illustrated in Fig. 2. The FMZI transmission spectra are measured when the micro-cavities are filled by air.

As illustrated by Fig. 2(*a*) through 2(*d*), all FMZI samples have at least two resonance dips from 1300 to 1600 nm for air transmission, consistent with the previous study [6]. As the HF etching sustains, it is evident that the resonant wavelengths all red-shift in the direction indicated by the red arrows.

The dependence of wavelength shift and etching speed versus the etching time and HF concentration is plotted in Fig. 3.

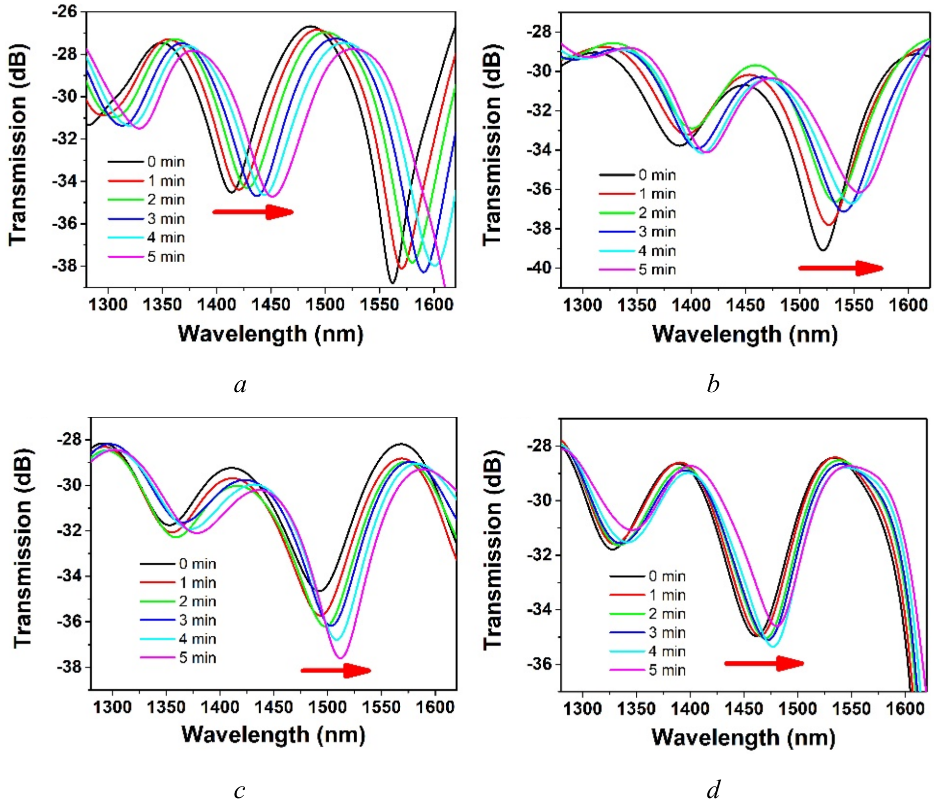
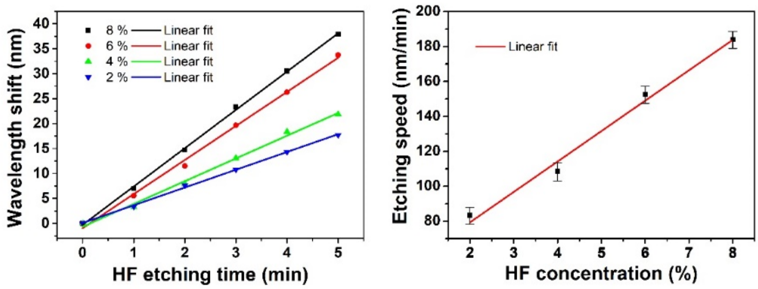


Fig. 2. The FMZI transmission spectra of various etching time and HF concentration. (*a*) – (*d*) HF solution of concentration 8%, 6%, 4% and 2%, respectively

By linear fitting presented in Fig. 3(*a*), the slope efficiencies ($d\lambda_m/dt$) are calculated to be 7.7, 6.8, 4.6 and 3.6 nm/min, respectively, indicating the dependence of wavelength shift rate upon the concentration of HF solution. The interference length of FMZI can also be obtained, based on Eq.2, from the transmission spectra in Fig. 2(*a*)-(*d*). Thus, the etching speed of various HF concentrations are calculated, based on Eq. 4, to be 183, 152, 106 and 83 nm/min, respectively, and plotted in Fig. 3(*b*). As chemical etching is equally applied on both sides of the micro-cavity wall, the etching speed of 41.5 nm/min per single wall is obtained for the 2% HF solution, which is on par with the resolution of fs laser direct writing in silicate glass [14], but with a much simpler and more accessible process.



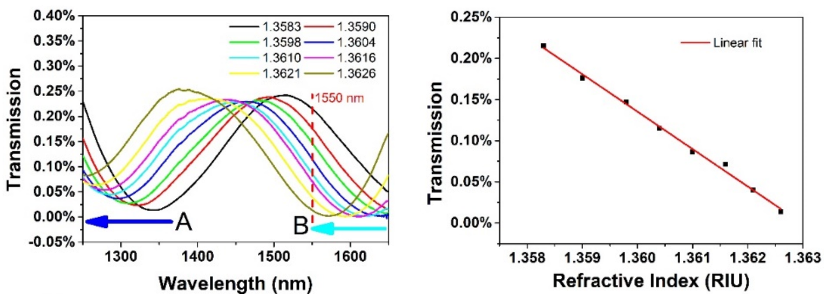
a *b*

Fig. 3. (*a*) The resonant wavelength shift of FMZI vs. the etching time; (*b*) The etching speed vs. the HF concentration

2. Miniaturization of the fmzi sensing system

2.1. From spectral analysis to intensity measurement

By the precise chemical etching process, the resonant wavelength of FMZI can thus be shifted to any desirable wavelength that a regular LD can offer. We therefore choose the common telecommunication wavelength of 1550 nm as the operation wavelength for the commercial availability of the off-the-shelf components. The ethanol solution is also selected as the testing sample for the index-sensing FMZI device. By the controlled etching process, the FMZI resonant wavelength can be shifted into the region around 1550 nm, as the dashed vertical line in Fig. 4(*a*), when the micro-cavity is immersed in ethanol solution of 54 vol.% concentration. When the concentration of ethanol solution varies from 40 to 54 vol.%, which has a corresponding index variation from 1.3583 to 1.3626, the transmission spectra of FMZI are monitored and recorded in Fig. 4(*a*).



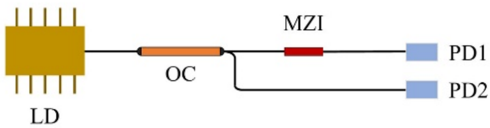
a *b*

Fig. 4. (*a*) The transmission spectra of FMZI in ethanol solutions of various concentrations and refractive indices; (*b*) The transmission at 1550 nm versus the index change

The resonant wavelength shift of FMZI has been demonstrated for index sensing of high sensitivity [6]. As marked in Fig. 4(*a*), there are two resonant wavelengths (zone A & B) and both blue-shift as the solution index increases. At the chosen wavelength of 1550 nm, the transmission values in ethanol solutions are plotted versus index change in Fig. 4(*b*). It is observed that in the index range from 1.3583 to 1.3626, the transmission intensity at 1550 nm decreases in a good linear relationship, which indicates the feasibility of the proposed conversion from spectrum analyzing to intensity monitoring for index sensing. We also note there exist two simultaneous resonant wavelengths in the transmission spectrum, and it is also possible to use the other resonant wavelength as the operating signal (~1350 nm) depending on the emitting LD's availability. The possibility of using two or more sensing signals simultaneously at various wavelengths can potentially improve the sensitivity and monitor different parameters at the same time [8].

2.2. The miniaturized FMZI sensor setup and measurement

To establish the relationship between the transmitted intensity at 1550 nm and the solution index for the fiber sensor calibration, the scheme of a FMZI-based index sensing system is proposed and illustrated in Fig. 5(*a*).



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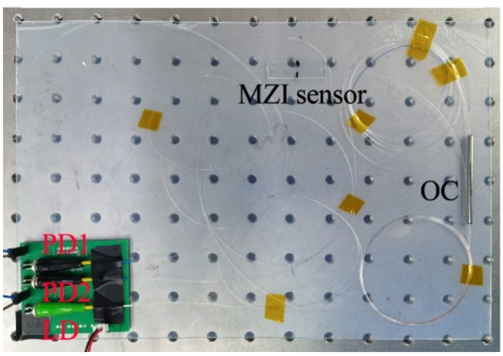

b

Fig. 5. The miniature FMZI index sensor: (*a*) the scheme; (*b*) the designed setup

Compared with the regular sensing system based on spectrum analysis, the BBS and OSA are substituted by a laser diode (LD, λ = 1550 nm) and two photodiodes (PDs), respectively. The utilization of two PDs is to eliminate the influence of LD intensity fluctuation. As the power of emitting LD is split by a 3-dB fiber optical coupler (OC), one portion of light is sent into the FMZI and the output is received at PD1, while the other portion is directly sent to PD2. The received signals at PD1 and PD2 are combined to offset the LD power fluctuation. The designed sensing setup is arranged and displayed in Fig. 5(*b*). As the LD and PDs are much more compact and affordable than BBS and OSA, the system is greatly miniaturized as well as cost effective. Furthermore, the test speed is also greatly accelerated as the intensity measurement is much faster than spectrum scanning.

In calibrating the index sensor, the power of emitting LD is set at 5.0 mW. When the FMZI is immerged in the ethanol solutions with refractive index ranging from 1.3583 to 1.3626, the 1550-nm transmission is recorded by PD1 and normalized by PD2. In each solution of different concentration, the measurement is repeated 5 times and the average transmission versus refractive index is plotted in Fig. 6. The transmission vs. index curve has a good linearity with the adjusted R2 = 0.99809, as the transmission decreases from 0.00244 to 0.000129 for index from 1.3583 to 1.3626, about -12.8 dB for 0.0043 RIU difference with an estimated mean-square-error of 0.0001. This confirms that our proposed miniaturized system is effective of refractive index sensing with considerable sensitivity. We note that the FMZI transmission could be adjusted higher for better signal-to-noise ratio if the spit of initial LD power has a larger portion entering the interference arm and PD1, instead of the 3-dB coupler used.

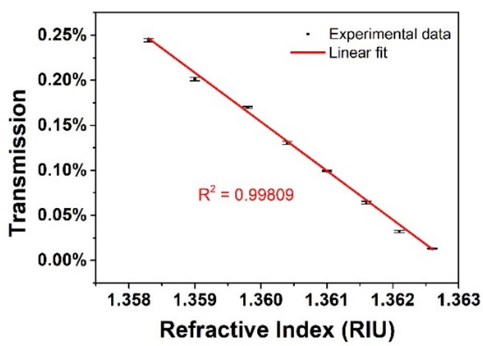

Fig. 6. The transmission vs. refractive index
of FMZI in the ethanol solutions

The proposed precise length control by chemical etching and the miniaturized FMZI sensing system may have much broader application fields than the simple index sensing of ethanol solution as the example in this study. First, the proposed precise length adjustment by chemical etching is not limited to the fiber micro-cavity length control, but might be beneficial for any microstructure fabrication process that needs a fine length (depth or diameter) adjustment in the follow-up tuning. Second, the intensity-measurement-based sensing system possesses very high sensing speed, it is thus applicable to high-speed sensing scenarios such as distributed salinity sensing of fast moving ocean currents. Lastly, although the detectable index range, only 0.0043 RIU from 1.3583 to 1.3626, is quite limited as demonstrated here, it may be applicable in biomedical or environmental sensing applications. For example, naturally, the refractive index of most liquids in living bodies are kept at a very small range, which can be used as indicators for body anomaly. Our proposed miniaturized sensing system may be applicable in these scenarios with further advancing. The other potential application field is to monitoring the salinity of seawater, which is ranging between 30‰ and 40‰ for the vast majority of ocean areas from NASA's global map of ocean salinity [20, 21]. The refractive index of ocean seawater is thus only 0.0017 RIU from 1.3383 to 1.34, a narrower index range applicable with the proposed system. The prototype high-speed seawater salinity monitoring sensor system based on the FMZI device is currently under investigation in the lab.

Conclusions

To conclude, we present the method of adjusting the micro-cavity length of a FMZI device to the order of tens of nanometers by a chemical etching process for the first time. This etching process is applied to shift the resonant wavelength of micro-cavity to the emitted wavelength of a LD, which is used as the light source for the index sensing system. By matching the micro-cavity's resonant wavelength to that of the LD's emission, the transmitted intensity of the FMZI device can be measured as an indicator of the refractive index of the medium filling the micro-cavity. The index-sensing fiber system is thus miniaturized based on the simple transmission monitoring. The constructed test setup has demonstrated its effectiveness by sensing the index of sampled ethanol solutions of different concentrations. The miniaturized fiber sensing system is not only simple, affordable, and portable, but operates much faster than the usual spectrum-analyzing type. The proposed fiber sensing system has the potential to be applicable for sensing the seawater salinity as well as liquids in living bodies.

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