

## Regular Articles

# A chitosan-coated humidity sensor based on Mach-Zehnder interferometer with waist-enlarged fusion bitapers



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## ABSTRACT

A novel humidity sensor, which adopts a Mach-Zehnder interferometer (MZI) in normal single mode fiber (SMF) modified by the deposition of chitosan (a moisture-sensitive natural polymer) on the cladding, is proposed and experimentally demonstrated. It is fabricated by the fusion splicing of a segment between the two SMF with waist-enlarged fusion bitapers. This all-fiber MZI based on SMF incorporates inter-modal interference between the core mode and the cladding mode. Due to the fact that it is sensitive to external refractive index and that the RI of the chitosan multi-layer film coat depends on the environmental humidity, the SMF-MZI with a chitosan coating layer of nanometer thickness is employed in humidity measuring. The sensitivity of  $\sim 119.6$  pm/RH (relative humidity unit) is achieved within the range from 10% to 90% on the experimental level. Moreover, the chitosan coat has good biocompatibility for in vivo biomedical applications like immunosensing and DNA hybridization detection in the near future.

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## 1. Introduction

Humidity measuring is important in medicine, biology, chemistry, agriculture, forestry, food science, environmental science, oceanography, and civil engineering. Although it's been a long time since electrical measuring methods were adopted for the first time, normal humidity electrodes (often relatively voluminous) in vivo is inconvenient and incompetent, especially in medical and clinical analysis, which has made miniature optical humidity sensor a pretty hot topic. Small-sized, flexible in designs, and non-metallic conductive, fiber-optic sensors are ideal for in situ and in vivo sensing, and have been fully demonstrated in past decades in the way of fiber Bragg gratings (FBGs) [1], long period gratings (LPGs) [2], tilted fiber Bragg gratings (TFBGs) [3], photonic crystal fibers (PCFs) [4], polymer fibers [5], hollow core fibers [6], tapered fibers [7] and fiber Fabry-Perot cavity [8]. However, all of these sensors have their own disadvantages. The fiber taper structures are very fragile due to poor mechanical strength of the small taper waist; fiber Fabry-Perot structures or TFBGs' fabrication are complicated in manufacturing; the cost of PCF should be considered; LPGs suffer from high dependence on temperature, so the measurement error is increased. The fiber-optical humidity sensors is based on the

morphology of nanostructured film known as swelling. Since the refractive index (RI) of a swelling film, like hydrogel [2] or polyelectrolyte-deposited nanostructured material [9], depends deeply on humidity, this sensor could be designed by the way of external RI measuring. Recently, the layer-by-layer (LbL) self-assembly technique, due to its precise control of thickness at the nanometer level and construction on non-flat surfaces [8], has emerged as a convenient and universally adopted method for the construction of ultrathin multilayer films. It is demonstrated in this paper that a novel miniature humidity sensor, which is based on robust all-fiber modal interference, is constructed by two waist-enlarged fusion bitapers sandwiched between the SMFs. The core mode and the cladding mode have been coupled and recombined, the latter sensitive to outside environment refractive index (RI) with chitosan nanocoated on the SMF-MZI; as for the chitosan film coat's RI which highly depends on the environmental humidity, this kind of SMF-MZI with a chitosan coating of nanometer thickness was fully explored for humidity measuring. Chitosan is a biopolymer, which recently emerged as a feasible material for biological sensing studies [13–14]. The chitosan has two features—hydrophilic and adhesion. Owing to these properties, Chitosan is most frequently preferred over chitin despite both having similar properties such as biocompatibility, biodegradability and nontoxicity. It has also gained popularity in research studies for its technological importance. Experimental results have shown that it has a

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special sensitivity responding slightly higher to external RI and much lower to temperature. Moreover, it has some advantages like robustness and lower fabrication costs when different types of fiber are spared.

## 2. Design and principle

Fig. 1 shows the schematic of a proposed MZI constructed by two cascaded waist-enlarged fiber bitapers. Unlike some conventional fiber tapers which have gradually decreased several micrometers in their waist diameters, this bitaper has enlarged its diameter with hundreds of micrometers [10], providing it with more robustness. In the first waist-enlarged bitaper region, having travelled a short optical path of SMF, the cladding mode is excited, recoupled back and interfered with the core mode by the other bitaper, thus consequentially forming the inter modal MZI. The transmission of the interferometer can be expressed in the equation stated below [11]:

$$I = I_{core} + I_{cladding} + 2\sqrt{I_{core}I_{cladding}} \cos(\delta) \quad (1)$$

where  $I$  is the intensity of the interference signal, and  $I_{core}$  and  $I_{cladding}$  are the intensities of the core and cladding modes;  $\delta$  is the phase difference of these two modes, defined as:

$$\delta = \frac{2\pi}{\lambda} \int_L (n_{cladding} - n_{core}) dz \quad (2)$$

where  $\lambda$  is the wavelength and  $n_{core}$  and  $n_{cladding}$  are the effective indices of the two modes respectively. If the SMF between these two bitapers is surrounded by a certain analyte ( $n_x$ ),  $n_{core}$  would be constant, because the core mode is isolated from the outside environment, while  $n_{cladding}$  would change with  $n_x$  owing to the analyte's effect upon the cladding. The refractive index sensitivity  $S$  is defined as the interference wavelength ( $\lambda_i$ ) shift divided by the corresponding  $n_x$  changes [12]:

$$S = \frac{d\lambda_i}{dn_x} = \frac{\lambda_i}{n_{cladding} - n_{core}} \frac{\partial(n_{cladding} - n_{core})}{\partial n_x} \quad (3)$$

When the analyte is deposited on the outer surface of the SMF, the equation " $\Delta n_{eff} = n_{cladding} - n_{core}$ " might be varied, after which the peaks in the transmission spectrum are shifted. Therefore, the refractive index measuring could be achieved by the way of corresponding wavelength shifting. The RI decreases while the swelling of the nanocoating increases, leading to the decrease of the transmission dip's wavelength. This is exactly why the SMF-MZI with LBL coating layer of nanometer thickness has been applied in humidity measuring.

## 3. Materials and methods

The MZI has been functionalized for the humidity detection, with 'chitosan' [13,14], a humidity sensitive polymeric material considered as a promising biopolymer for various applications like environmental sensors, coatings, and components of multilayer packaging, as it offers biocompatibility, high mechanical strength and excellent adhesion. Chitosan is composed of  $\beta(1 \rightarrow 4)$ -linked

glucosamine(X) and N-acetylglucosamine(Y), and could be synthesized from shellfish wastes like shrimps, crabs, and crawfishes. The two main unique properties, amino and hydroxy groups, are making chitosan the humidity sensing element, they are offering two distinctive features-hydrophilic and adhesive, and the former provides stability to the solvent in terms of its swelling ability in the presence of the acid or alkali solution, while the latter allows greater ease for chitosan to dissolve in low concentration of aqueous acetic acid and offers perfect film forming capability. Chitosan is a semi-crystalline polysaccharide [15] with stable chemical properties and has a highly porous structure as shown in Fig. 2. Moreover, it is selected as a humidity sensing element due to the feature of hygroscopicity.

### 3.1. SMF-MZI fabrication

The polymer coating of the SMF (Corning, SMF-28) must be stripped off before the process of splicing. The fiber ends of the SMF are cut by a high-precision cleaver and spliced to the SMF, by a conventional fiber splicer, in a manual operation program. (Fujikura FSM-40S). With precise manual adjustments, two prepared SMF stubs are butt-coupled and aligned along axis. To form a waist-enlarged fiber bitaper, the splicing parameters in the manual mode must be default but a large "overlap" of 80  $\mu\text{m}$  is chosen for fusion splicing correspondingly [10]. Here, "overlap" means the overlapping distance in which the two fibers are pushed further together for they have been softened as compared to being merely touched by each other in butt coupling. After arc discharges have been applied, the fiber tips are softened and pushed together, and their diameters are gradually enlarged due to the large overlap and pushing force, and a waist-enlarged fiber bitaper is constructed. Fig. 3 shows its side view of the formed fusion bitaper. The waist diameter and bitaper length are estimated to be around 12.7 and 257.7  $\mu\text{m}$  respectively, while the outer fiber diameter has been increased to be 143.8  $\mu\text{m}$  from 125  $\mu\text{m}$ . Then another fiber end of the SMF has been cleaved with fiber length control, and the total length of the SMF-MZI is about 40 mm. The next procedure would be the splicing of the second point under the same program as mentioned above. The insertion loss of the fabricated sensor is about 30 dB. To form a thin chitosan membrane on the cladding of the SMF, a dip-coating process has to be performed [14].

### 3.2. Chitosan film coat fabrication

Before the SMF-MZI with chitosan has functioned, it must be treated with piranha solution to achieve negatively charged sur-

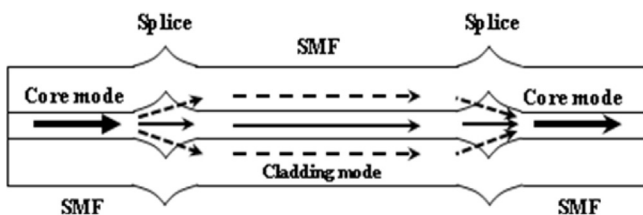


Fig. 1. Schematic diagram of the proposed humidity sensor.

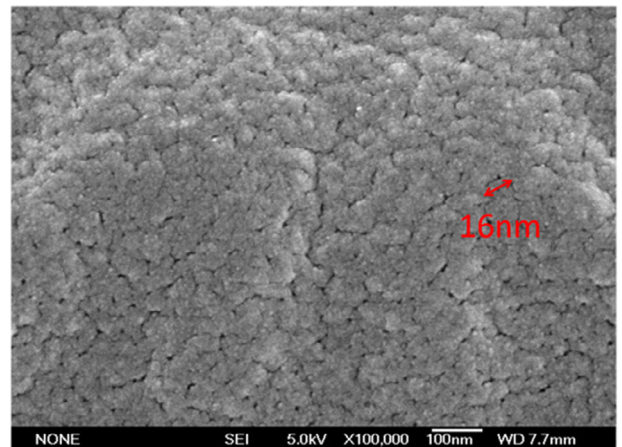


Fig. 2. SEM image of chitosan film coat surface.

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