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Surface plasmon cavities on optical fiber end-facets for biomolecule and ultrasound detection

Tian Yang [a,*], Xiaolong He [b], Xin Zhou [a], Zeyu Lei [a], Yalin Wang [b], Jie Yang [a], De Cai [a], Sung-Liang Chen [a], Xueding Wang [c]

[a] State Key Laboratory of Advanced Optical Communication Systems and Networks, Key Laboratory for Thin Film and Microfabrication of the Ministry of Education, UM-SJTU Joint Institute, Shanghai Jiao Tong University, Shanghai 200240, China
[b] Xu Yuan Biotechnology Company, 1180 Xingxian Road, Shanghai 201815, China
[c] Department of Biomedical Engineering, University of Michigan, 2200 Bonisteel Blvd, Ann Arbor, MI 48109, USA

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ABSTRACT

Integrating surface plasmon resonance (SPR) devices upon single-mode fiber (SMF) end facets renders label-free sensing systems that have a simple dip-and-read configuration, a small form factor, high compatibility with fiber-optic techniques, and invasive testing capability. Such devices are not only low cost replacement of current equipments in centralized laboratories, but also highly desirable for opening paths to new applications of label-free optical sensing technologies, such as point-of-care immunological tests and intravascular ultrasound imaging.

In this paper, we explain the requirements and challenges for such devices from the perspectives of biomolecule and ultrasound detection applications. In such a context, we review our recent work on SMF end-facet SPR cavities. This include a glue-and-strip fabrication method to transfer a nano-patterned thin gold film to the SMF end-facet with high yield, high quality and high alignment precision, the designs of distributed Bragg reflector (DBR) and distributed feedback (DFB) SPR cavities that couple efficiently with the SMF guided mode and reach quality factors of over 100, and the preliminary results for biomolecule interaction sensing and ultrasound detection. The particular advantages and potential values of these devices have been discussed, in terms of sensitivity, data reliability, reproducibility, bandwidth, etc.

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1. Introduction

Integrating sensors at the ends of optical fibers, which changes the intensity, spectrum and/or phase of the fiber-guided lightwaves upon detection of the targeted substances, is desired in a number of application scenarios, e.g., biomolecule interaction analysis, drug screening, immunological tests, agriculture produce inspection and ultrasound imaging. For they have the advantages of allowing optical access to the inside of opaque subjects, transmitting optical excitations and signals in flexible fibers and through long distances, taking advantages of the development of fiber-optic communication technologies, and providing more compact, simple and stable systems compared with the free-space optics counterparts. In addition, by placing the fiber ends right onto the targets and reading the outputs, the operation requires much less technical expertise and is much more time efficient than the alternative techniques, which is of high importance for emergent medical diagnosis, point-of-care testing (POCT), and on-site inspections.

An ideal fiber-end sensor device should satisfy the following four requirements. (1) The sensing performance, in terms of limit of detection (LOD), satisfies the targeted applications or even matches its free-space counterpart which couples to planewaves. (2) The fiber is a single-mode fiber (SMF) and the optical system is an all-fiber system. Although using a straight segment of multi-mode fiber (MMF) is an effective way to guide light into and out of the sample [1], it loses most advantages of using fiber-optics. In particular, when MMFs are applied, free-space optics setups are often involved in order to couple to the fundamental or the few lowest order fiber-guided modes, which makes the optical system complicated, bulky and incompatible with standard fiber-optic telecommunication techniques. (3) The sensor signal is collected by reflecting into and being guided through the same fiber. The end-reflection configuration is crucial for dip-and-read operation and insertion into tiny vessels. (4)

* Corresponding author.
E-mail address: tianyang@sjtu.edu.cn (T. Yang).

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Reproducibility between different devices is important for quantitative measurements and sensor arrays. This presents a challenge for device fabrication.

Despite the fact that fiber-end sensors have been investigated extensively for many years, there is limited success to achieve all of the above four requirements, which has significantly hindered progress for real applications and commercialization. In this paper, after introduction to the fabrication methods, we review our recent work on surface plasmon cavities on optical fiber end-facets for refractive index sensing, in the contexts of biomolecule detection and ultrasound detection [2–4]. In particular, we discuss the requirements on the sensor devices in further details from the application point of view, which seems to be elusive from an amount of recent device research reports. We believe these discussions are necessary to guide current device research towards producing really valuable products.

While this review focuses on fiber end-facet surface plasmon resonance (SPR) devices, reviews of a broad range of recent technical advances about lab-on-fiber nanostructures and fabrication technologies can be found in Ref. [5–9], which open many other opportunities for performance improvement and applications. In addition, there have been a number of reports on SPR devices on planar substrates whose sizes and numerical apertures match that of a SMF, which are prospective to be integrated upon SMF end-facets, e.g. SPP launching and shaping [10–14] and polarization conversion [15].

2. Device fabrication

The endeavor to fabricate nano-plasmonic structures on the tiny flat end-facets of optical fibers have been a subject under active exploration. Electron beam lithography, in spite of being the most efficient and reliable methods for patterning nanostructures on a planar substrate, becomes hugely challenging when being applied on the fiber end-facets, due to the difficulty to coat a thin layer of photoresist, the lack of a conductive substrate and the difficulty to align the nano-pattern to the core of fiber. Nonetheless, successful patterning of periodic nanodot arrays upon SMFs and concentric rings upon large core area MMFs have been successfully demonstrated [16,17]. The photoresist layers were coated by dipping the fiber tips into the resist and then shaking or blowing off the extra resist, by mounting the fiber end-facets onto flat surfaces, or by thermal evaporation [18]. Meanwhile, interference lithography has also been demonstrated, which also requires a photoresist layer [19]. On the other hand, focused ion beam milling is a much more straightforward method for fiber end-facet nano-patterning, as have been reported by several groups [20–23]. However, it is much more time consuming, and the injection of gallium ions is a severe concern for Raman spectroscopy measurements [24].

In view of the difficulties to directly pattern the fiber end-facet, a few transfer techniques have been developed in recent years, in which a nano-patterned metallic film is first fabricated on a planar substrate and then transferred to the fiber end-facet. In a nanoskiving approach, the metallic nanostructures embedded in epoxy are sectioned into thin slabs, then the slabs are floated flat on top of water and transferred to the fiber end-facets by pressing the fibers into the slabs [25]. In a decal transfer approach, the nanometallic structures are stripped from a planar substrate, and transferred to a thin sacrificial film and then the fiber end-facet in sequence [26]. This approach also enables transfer onto curved surfaces. At the same time, the transfer method proposed by Ref. [27], in which the gold nanostructures are directly stripped off silicon or SiO_2 substrates, has become a widely employed technique in nanoplasmonics device fabrication nowadays, due to the weak Van der Waals binding force between gold and the substrates, and the superior surface smoothness of the transferred gold. This transfer method has also been adopted for transferring gold nanostructures onto fiber end-facets by several groups, including us [2,28–30]. More recently, transfer by using hydrochloric acid to etch a sacrificial indium tin oxide layer has been demonstrated [31].

Fig. 1a shows the concept of our glue-and-strip fabrication approach. The details are as follows [2]. First, an amount of optical epoxy adhesive is applied on the tip of the fiber, with a careful control of the volume of the epoxy droplet. Then, the fiber is mounted on a multi-axis translation stage and moved towards the substrate which carries the gold film with the nano-patterns. The fiber is observed under a stereomicroscope and aligned with its mirror image in the gold film to be a straight line. Alignment marks on the gold film enables a coarse alignment of the fiber to the nano-pattern. Then precise alignment is achieved by sending a broadband light into the fiber, monitoring its reflection spectrum off the gold nanostructure in real-time, and optimizing the position of the fiber end. After alignment, the epoxy is either cured by a UV lamp or heating the substrate. At last, the fiber tip is moved off the substrate quickly to strip the gold film. For non-periodic nano-patterns such as a cavity, UV curing through a transparent substrate such as glass and quartz is preferred, since it avoids thermal expansion of the substrate which may severely affect the alignment. A front-view optical microscopy image of the resulting device is shown in Fig. 1b, where the irregular outer rim is the edge of the stripped and transferred thin gold film, the circular inner rim is the edge of the cladding of a bare fiber underneath the gold film, and the square-shaped shadow is a nanoslit array on the gold film. The four dark rectangles are alignment marks. In the center of the nanoslit array is an SPR cavity, which shows a lighter color than its surrounding and which is aligned to the core of fiber.

In addition, there are a plethora of other inventions to modify the fiber tips, including but not limited to the flat end-facets, e.g., direct laser writing [32–34], mechanical polishing [35–37], chemical etching and tapering [38–42], fiber-aligned photolithography [43], nanoimprinting [29], self-assembly of nanoparticles [44], and Micro-Electro-Mechanical System (MEMS) techniques [45]. A survey of these techniques can be found in Ref. [6].

3. SPR cavities on optical fiber end-facets for biomolecule sensing

3.1. Label-free biosensing: Promises, challenges and comments

Micro-optical resonators and interferometers, whose responses to external optical excitations shift with the changes in environmental refractive indices, have been widely and intensively investigated for label-free biomolecule sensing [46–48]. It has long been anticipated that the development of label-free sensing technologies, due to the fact that they significantly simply the biochemical experiments by eliminating multiple labeling steps, will revolutionize the traditional drug screening markets and bring new markets in POCT, food safety, environmental monitoring, etc., which are worth tens of billions of USDs [49–52]. In addition, SPR has been written in the pharmacopeia of the United States and Japan for the first time last year, which brings the anticipation of a fast growth of market in the near future [53]. However, more than ten years after these predictions, nowadays the applications of label-free biosensing are still mainly restricted in high-end biology laboratories and secondary drug screening in pharmacy [54,55]. The limited applications are due to the high costs and bulky sizes of the equipments, and the lengthy operation procedures which require a certain degree of professionalism. In this aspect, the successful integration of SPR at optical fiber end-facets has a great

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