Breath Figures onto Optical Fibers for Miniaturized Sensing Probes

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1 Introduction

Recently, the realization of miniaturized and advanced optical fiber devices and the consequent development of technological processes, specialized for the optical fibers, led to the definition of the "lab-on-fiber" concept [1], devoted to the realization of novel and highly functionalized technological platforms completely integrated in a single optical fiber for communication and sensing applications. In this scenario, the creation of micro- and nanostructures on the end facet of optical fibers is of great interest because it may yield versatile optical devices well suited to serve as miniaturized probes for remote sensing applications. Several approaches have been recently introduced to fabricate metallic and dielectric structures on the optical fiber end facet. Some approaches rely on the study of appropriate techniques to transfer planar nanoscale structures, fabricated on a planar wafer by means of standard lithographic techniques, onto the optical fiber end facet. These methods exploit well-assessed fabrication processes developed for planar substrates, but they are

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limited by the final transferring step that plays a fundamental role in determining both the fabrication yield and the performance of the final device [2]. Alternative approaches are based on direct-write patterning of the fiber tip. These methods, based on conventional lithographic techniques adapted to operate on unconventional substrates such as the optical fiber tip, are able to efficiently provide nano-structured devices on the optical fiber, but they require complex and expensive fabrication procedures with a relatively low throughput [2, 3].

In this work, we propose the creation of periodic metallo-dielectric structures on the optical fiber tip by using self-assembly techniques. Specifically, we selected the breath figure (BF) technique for the preparation of patterned polymeric films directly on the optical fiber tip. After this stage, we employ a simple evaporation technique for the conformal deposition of a thin metal layer of gold. Following this simple approach, we fabricated several prototypes of miniaturized sensing probes.

2 Fabrication Process

To build metallo-dielectric periodic structures directly on the end facet of a single-mode optical fiber in a simple and cost-effective way, we developed a fabrication procedure based on the BF technique. This self-assembly approach, in fact, allows for the preparation of honeycomb-structured films with a high degree of order, which are, at least in principle, suitable for our purpose. BF formation spontaneously takes place when a polymer solution is cast on a substrate, under humid atmosphere. If the solvent has a sufficiently low boiling point, its fast evaporation lowers the temperature of the system, which triggers the condensation of water droplets on the film which is forming. In the following stage, water droplets arrange themselves closely, producing a template for the porous pattern. Finally, once both solvent and water are completely evaporated, a honeycomb imprint is left on the film surface [4].

Our fabrication strategy consisted initially in the preparation of a highly ordered microporous film on the fiber tip and then in the vapor deposition of a thin layer of Au on top of this assembly (see Fig. 1). To reach this goal, we conveniently modified the standard setup which is normally utilized for BFs on a glass substrate. The main drawback of building patterns directly on the fiber is the restricted surface of its facet: 125 μm of diameter for a standard single mode. For that reason, the optical fiber was embedded in a ceramic ferule with diameter of 2.5 mm and then accurately polished. Then this assembly, much easier to handle as compared to the bare fiber, was mounted on an Al holder of 20×20×8 mm, so that the polymer solution

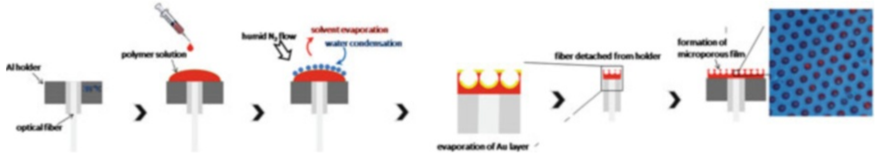

Fig. 1 Schematic view of the fabrication strategy. The detail of the microporous film is a real view taken by confocal microscope, in the central area of the fiber face

can be more easily drop cast on a surface of about 1 cm^2, much larger than that of the only fiber tip. In this way we overcame also the problem of poor pattern homogeneity, which is often encountered on the edges of BF films.

Films were prepared by drop casting on the holder with the fiber a 4 mg/mL CS_2 solution of a fluorinated fluorescent dye-terminated linear polystyrene, which we had already described as a good candidate for producing highly ordered BFs [4]. A flux of moist nitrogen (60 % R.H. at 25 °C) was directed on the Al surface. The solvent was completely evaporated within 20 s, living on holder and fiber surface an opaque film, which shined in bright iridescent colors when viewed in reflected light. By regulating the rate of the moist nitrogen flux between 1.5 and 2.5 L/min, i.e., by varying the solvent evaporation rate parameter, honeycomb films with cavities ranging from 2.5 to 0.9 μm of external diameter, respectively, were obtained. Once the optical fiber facet was covered with this polymeric pattern, it was placed in a vacuum evaporation chamber and 30–40 nm of Au were deposited on it. By this two-step procedure, prototypes consisting in a single-mode optical fiber end-coated with a double metal pattern, a micrometric Au mesh lying on top of the polymer film and an array of Au cups lying on the bottom of the cavities, were achieved.

3 Experimental Results

In this section the attention is principally focused on the experimental analysis of a representative sample fabricated by means of the fabrication process previously described. With regard to this sample, a complete morphological characterization has been carried out via scanning electron microscope (SEM) and atomic force microscope (AFM) analysis. Figure 2a shows an SEM top view image of the sample where can be appreciated the ceramic ferule with smoothed edge (diameter 2.5 mm). Magnified SEM image (here note reported) and AFM image (Fig. 2b) permit to measure statistic values of the cavity diameters and pitches of the patterned region. From images analyses following average (variance) values were retrieved: diameter of 0.95 μm (8.99×10^{-4} μm^2), pitch of 2.67 μm (0.0012 μm^2), cavities depth of 1.78 μm (0.0038 μm^2), and structure height of 2.5 μm (0.0032 μm^2). From these results it is evident that the breath figure technique enables the possibility to realize

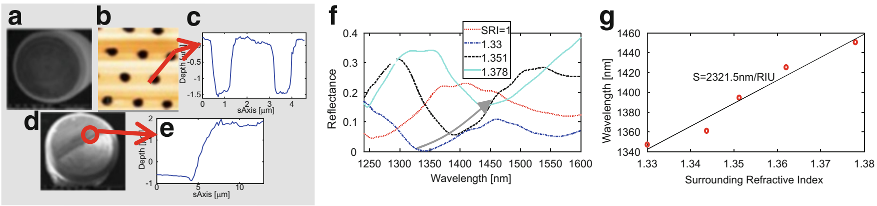

Fig. 2 Morphological characterization via SEM and AFM images. Sample: (**a**) view SEM image; (**b**) AFM image of patterned region; (**c**) AFM profile; (**d**) SEM top view image after excimer laser treatment; (**e**) AFM profile of the pattern edge; (**f**) SRI characterization; (**g**) wavelength of the reflection dip versus SRI

metallo-dielectric structure directly self-assembled on fiber optic tip with highly regular pattern. To show the potentiality of patterned metallo-dielectric lab-on-fiber structures realized via breath figures approach, in the following, we present some preliminary results demonstrating how the fabricated sample is able to sense the refractive index variations in the surrounding environment.

To investigate the surrounding refractive index (SRI) sensitivity, the reflectance spectra were measured while the fabricated samples were immersed in different liquid solutions (n in range 1.333–1.362). The experimental results are shown in Fig. 2f, in which a significant red shift of the curves with increasing values of the SRI is evident. Focusing the attention on the wavelength shift of spectral features, in Fig. 2g, we plot the wavelength of the reflection minimum (at 1,347 nm for SRI = 1.333) as a function of the SRI. The graph demonstrates a sensitivity (S) of ~2,300 nm/RIU for detecting changes in the bulk refractive indices of different chemicals surrounding the fiber-tip device.

The excellent sensitivities versus SRI changes as well as the properties of gold for the binding of suitable bioreceptors make these structures promising candidates for novel miniaturized affinity-based biological nanosensors with the ability of detecting few or even single nanoparticles.

4 Conclusions

In conclusion, the reported experimental results demonstrated the feasibility of the proposed fabrication approach to realize hybrid metallo-dielectric structures directly on the end facet of optical fibers. This enables the fabrication of micro- and nano-structured devices on fiber tip by means of simple and non-expensive fabrication procedures differently from conventional approaches. Also, BF technique could easily permit the realization of patterned structures with a relatively high throughput. The excellent sensitivities versus SRI changes as well as the properties of gold for the binding of suitable bioreceptors make these structures promising candidates for novel miniaturized affinity-based biological nanosensors with ability of detecting few or even single nanoparticles.

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