

Fiber Optic Sensing Probes using Self-assembly techniques

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Abstract – The “Lab on Fiber” technology has been recently proposed as a valuable route for the realization of novel and highly functionalized technological platforms completely integrated in a single optical fiber in communication and sensing applications. As a follow up of the proposed technological approach, here, we present recent results on the fabrication of metallo-dielectric structures on the optical fiber tip by using a self-assembly technique. The pursued approach basically consists in the preliminary preparation of a patterned polymeric film by the breath figure technique, directly on the optical fiber tip, and in the successive metal deposition by evaporation. The experimental results demonstrate the successful creation of a metallo-dielectric honeycomb pattern on the optical fiber tip. The sensing properties of the optical fiber probes have been successfully explored in terms of sensitivity to the surrounding refractive index changes demonstrating their potentialities for chemical and biological sensing applications. Finally, we report the preliminary results on the ongoing activities aimed to develop novel fabrication approaches of optical fiber sensing probes based on the assembly of microspheres into 2D colloidal crystals.

I. 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 nano-scale 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 limited by the final transferring step that plays a fundamental role in determining both the fabrication yield and the performance of the final device [1]. 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 nanostructured devices on the optical fiber, but they require complex and expensive fabrication procedures with a relatively low throughput [2]. In this work, we propose the creation of periodic metallo-dielectric structures on the optical fiber tip by using a 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. Here, we report details on the fabrication process employed to create honeycomb patterns on the optical fiber tip and some representative results of the numerical and experimental analysis. Finally, the functionality of the realized probes for sensing applications is demonstrated. Finally, we report the preliminary results on the ongoing activities aimed to develop novel

fabrication approaches of optical fiber sensing probes based on the assembly of microspheres into 2D colloidal crystals.

II. FABRICATION PROCESS

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 Figure 1). To reach this goal, we conveniently modified the standard setup which is normally utilized for BFs [3,4] 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 $20 \times 20 \times 8$ mm, so that the polymer solution 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. The Al holder was pre-cooled by placing it on a cold stage maintained at a temperature of 10°C , and removed from there just before starting the film deposition. Given the high thermal conductivity of the metal substrate, as compared to glass, such pre-cooling step is required to promote an efficient water condensation on the evaporating film and, consequently, the BF formation.

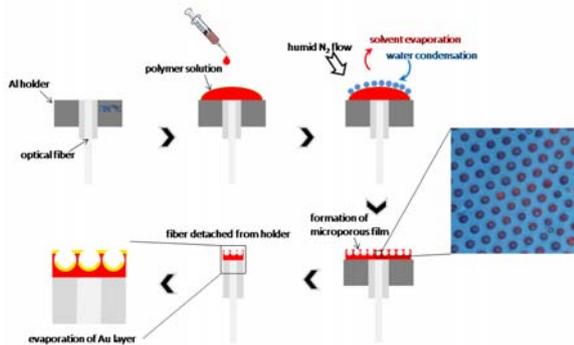


Fig. 1. Schematic overview of the fabrication process. The detail of the microporous film is a real view taken by a confocal microscope, in the central area of the fiber face.

III. EXPERIMENTAL AND NUMERICAL RESULTS

In this section the attention is principally focused on the experimental and numerical 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 2(a) shows a

SEM top view image of the sample where can be appreciate the ceramic ferule with smoothed edge (diameter 2.5 mm). Magnified SEM image (here not reported) and AFM image (fig. 2(b)) permit to measure statistic values of the cavity diameters and pitches of the patterned region. From images analysis following average (variance) values were retrieved: diameter of $0.95 \mu\text{m}$ ($8.99 \cdot 10^{-4} \mu\text{m}^2$) and pitch of $2.67 \mu\text{m}$ ($0.0012 \mu\text{m}^2$). From the analysis of the AFM profile (see fig. 2(c)) the average (variance) depth of the cavities was retrieved of $1.78 \mu\text{m}$ ($0.0038 \mu\text{m}^2$). Finally to estimate the patterned polystyrene height, we first removed the polystyrene layer on half area of the ceramic ferule by using an excimer laser (operating at the wavelength of 248 nm), and then measured the structure height with AFM analysis. A SEM top view after laser treatment is shown in fig. 2(d). To this aim laser fluence and repetition rate were opportunely selected in order to ablate polystyrene layer without damage the ceramic surface. AFM profile of the edge region is plotted in fig. 2(e) providing to measure the average (variance) structure height of $2.5 \mu\text{m}$ ($0.0032 \mu\text{m}^2$). This reveals that the polystyrene layer consists in a solid basement (approximately height $0.72 \mu\text{m}$) and a patterned region. From these results it is evident that the breath figure technique enables the possibility to realize metallo-dielectric structure directly self-assembled on fiber optic tip with highly regular pattern.

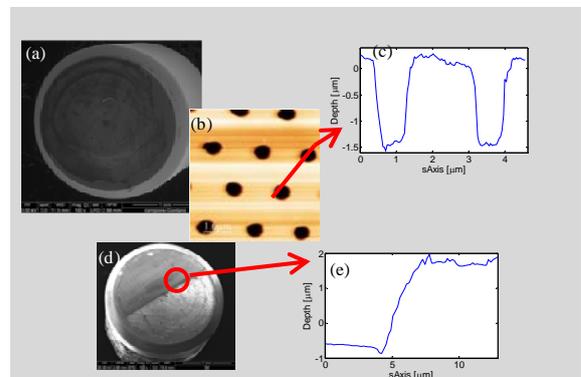


Fig. 2. Morphological characterization via SEM and AFM images: (a) view SEM image; (b) AFM image of patterned region; (c) AFM profile; (d) SEM top view image after excimer laser treatment; (e) AFM profile of the pattern edge

The performed morphological analysis supplied detailed information of the geometrical sizes of the realized samples and demonstrated the successful creation of self-assembled lattice structures with highly regular pattern directly on the fiber optic tip [5].

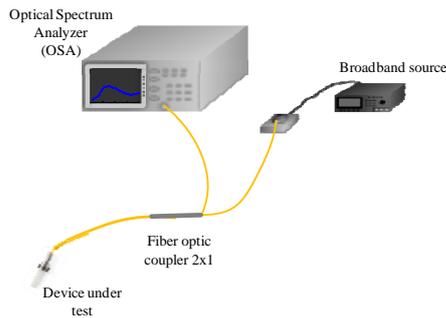


Fig. 3. Schematic optoelectronic setup.

Spectral reflectance measurements were carried out by means of an easy measurement setup involving a 3 dB directional coupler placed between a broadband light source (covering the wavelength range 1250-1650 nm) and the optical fiber probe, with one return end sent to the optical spectrum analyzer, as schematically plotted in figure 3.

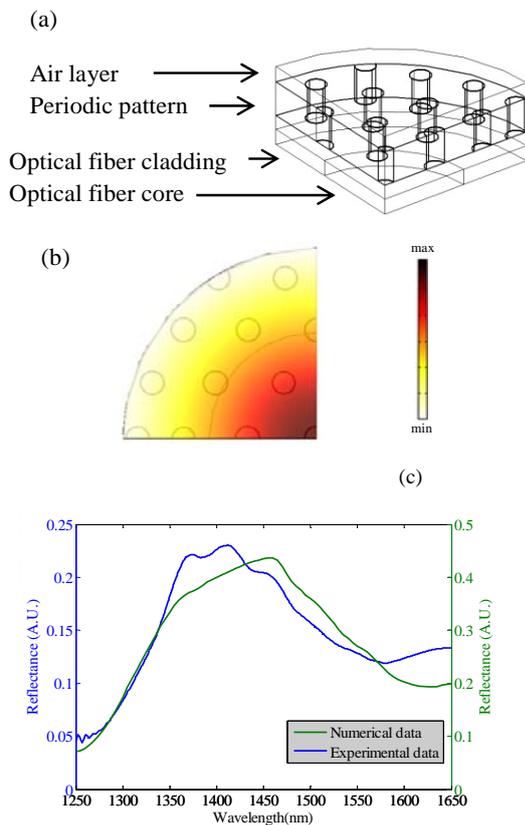


Fig. 4. (a) Computational domain of the optical fiber based model (b) Normalized electric field distribution of the input light source superimposed to the periodic pattern onto the optical fiber; (c) Experimental reflectance spectrum in comparison with the numerical reflectance retrieved by the optical fiber based model.

The sample reflectance was normalized by using a fiber-optic reference mirror, fabricated by depositing a 160 nm-

thick gold film on the tip of a standard single-mode fiber. In Figure 4(c) we show the experimental reflectance spectrum (blue line) of the fabricated sample. It can be noted that the maximum reflectance remains quite low (below 25%) in all the investigated spectral range. Also a broadband reflectance peak centered around at 1400nm can be appreciated. A dedicated numerical tool was developed to simulate arbitrary sub-wavelength structures integrated on the optical fiber tip in order to predict the spectral features of the fabricated fiber tip structures. The numerical model admits a Gaussian-like mode as input light source and it is characterized by a periodic pattern space-limited in order to take into account finite-size effects. The corresponding computational domain, shown in figure 4(a), involves a thin slice of optical fiber, composed by a core with a diameter of 9 μm and a reduced cladding with diameter of 16 μm terminated with perfect electric conductor (PEC) condition. The structure realized is numerically reconstructed in the form of a thin polystyrene uniform layer beneath the BF pattern and the entire structure is assumed to be covered by a conformal thin film of gold. From the comparison (figure 4(c)) it can be clearly appreciated the good agreement between numerical and experimental data. Having improved the numerical prediction capability, in figure 5 we show the electric field distribution at different wavelengths, representative of the investigated spectral range. At a first glance, the normalized electric field appears quite similar to that observed in a simple interferometric structure, featured by sinusoidal behavior along the propagation direction.

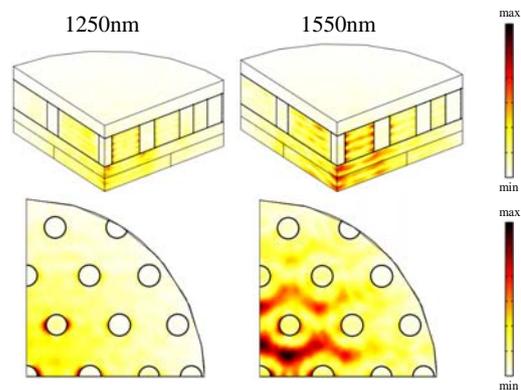


Fig.5. Normalized electric field distributions at the wavelengths 1250 and 1550nm and top view of the normalized electric field distributions in a middle slice at the same wavelengths

However, remarkable differences are evident in correspondence to the metal-dielectric interfaces and in correspondence to the holes pattern, where a significant field localization can be clearly appreciated. In this regard, the figure 5 also shows the top view of a slice positioned in the middle of the patterned region, where field localization can be easily noticed especially close to

the gold disks and at the interfaces between the thin layer of gold and the surrounding dielectric. These considerations lead us to envision the interplay taking place between the classical interference mechanism and the plasmonic excitation occurring at the metal interfaces.

IV. SENSING CHARACTERISTICS

To investigate the surrounding refractive index (SRI) sensitivity the reflectance spectra were measured while the fabricated samples were immersed in different liquid solutions with well-known refractive index such as water ($n= 1.333$), ethanol ($n = 1.362$), isopropyl alcohol ($n = 1.378$), and some solutions with different concentration of water and ethanol where the refractive indices were previously measured via commercial Abbe refractometer (n in range 1.333-1.362). The experimental results are shown in figure 6(a), in which a significant red-shift of the curves with increasing values of the SRI is evident. Also it can be noted that the maximum reflectance value keeps quasi unchanged when external medium changes from air to water while it monotonically increases with further increasing values of the SRI.

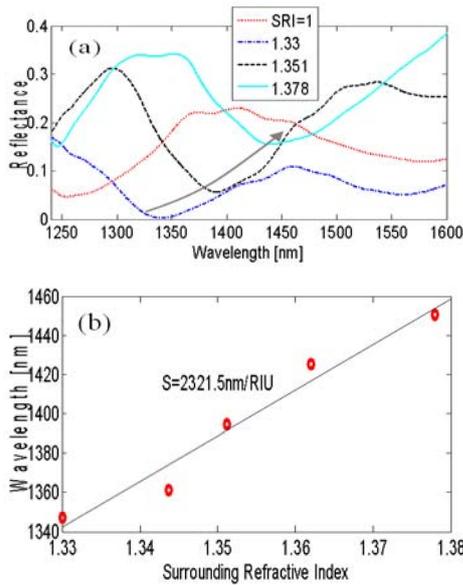


Fig. 6. SRI analysis: (a) experimental reflectance spectra as function of the surrounding refractive index; (b) wavelength of the reflection dip versus SRI.

Focusing the attention on the wavelength shift of spectral features, in figure 5(b) we plot the wavelength of the reflection minimum (at 1347nm for SRI=1.333) as a function of the SRI. The graph demonstrates a sensitivity (S) of ~ 2300 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 ability of detecting few or even single nanoparticles.

V. MICROSPHERES IN 2D COLLOIDAL CRYSTALS

Encouraged by the obtained promising results, we are currently exploring novel fabrication approaches enabling

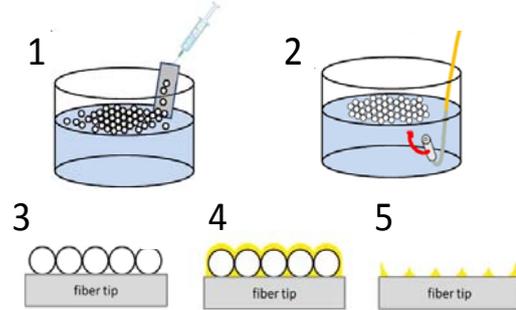


Fig. 7. Overview of the five-steps fabrication process: 1) assembly of PS microspheres at the air/water interface; 2) transferring of the assembled monolayer on the fiber tip; 3) slow drying; 4) Au layer deposition by thermal evaporation; 5) microsphere removal by sonication.

the realization of various nanostructures on the optical fiber tips. Specifically, we selected another kind of self-assembly approach, suitable for realizing periodic structures on the optical fiber, that is the assembly of microspheres into 2D colloidal crystals. We explored this possibility by covering the optical fiber tip with 500 nm polystyrene microspheres, following the fabrication scheme depicted in Fig.7.

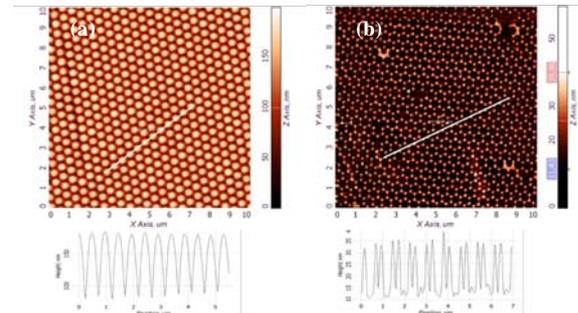


Fig. 8. (a) AFM height images with corresponding section profiles of the optical fiber tip: after fabrication step 4 of Fig. 7; (b) AFM height images with corresponding section profiles of the optical fiber tip after fabrication step 5 of Fig. 5.

The nanospheres are suspended in a 50% ethanol/water mixture, and then slowly spread on water surface through a nearly vertical Si conduit plate. The 2d colloidal crystal spontaneously forms at air/water interface, forming floating islands that can be easily transferred on the fiber for the following fabrication steps. As shown by the AFM images reported in Fig.8(a,b), once all the fabrication steps were optimized, we were able to obtain two different typologies of periodic structure: a 2d colloidal

crystal covered by a thin gold layer (Fig.8 (a)) and a periodic distribution of triangular gold islands attached directly to the optical fiber face (Fig.8 (b)). Such fiber prototypes are attractive as optical sensing probes because they are potentially able to trigger various plasmonic resonant phenomena, useful for label-free chemical and biological sensing applications. Optical characterizations and devices optimization are currently taking place.

VI. 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 enable the fabrication of micro- nano-structured devices on fiber tip by means of simple and non-expensive fabrication procedures differently from conventional approaches. 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. The successfully realization of the first prototypes encourage future work and design improvement. Specifically, we selected another kind of self-assembly approach, suitable for realizing periodic

structures on the optical fiber, that is the assembly of microspheres into 2D colloidal crystals.

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