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Optical Properties of the Refractive Index Sensor Based on Nanoparticles Composites

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Polymer microtips fabricated at the ends of multi-mode optical fibers can act as refractive index sensor transducers and as lenses. The aim of the studies was to test the possibility of improving the refractive index sensor sensitivity by adding gold nanoparticles (concentrations of 1% and 5%) and titanium dioxide (concentrations of 5% and 16.67%) to the monomer mixture. The presence of a nanoparticle composite in the mixture creates strongly scattering centers, which results in reduced reflected signal levels and a lower value of return losses. Shifts in return loss minima for the refractive index sensors were observed. After a microtip was manufactured from UV-cured pentaerythritol triacrylate mixture on multi-mode fiber with a 105 nm core diameter, the minimum reached a refractive index value of 1.44, while when Au 1% and Au 5% composites were added, the minimum shifted to 1.48. Doping the mixture with a 1% Au composite resulted in improved transmission properties in the 600–1200 nm spectral range. An improvement in transmission in the infrared range was obtained for a microtip doped with 16.67% TiO_2. Doping with titanium dioxide resulted in a significant decrease in signal in the wavelength range below 1200 nm.

topics: polymer microtips, optical fiber sensors, nanoparticles

1. Introduction

During the research, attention was paid to the possibility of producing microtips based on a *nanoparticle* (NP) composite, in which a selected mixture of monomers is used as a base, which allows for checking the optical properties of the element made in this way. NP composites are materials often used in studies combining the above properties [1–3]. Hence, after producing the appropriate materials, studies were conducted to characterize the effect of selected *nanoparticles* (NPs) on the geometry of microtip formation.

The importance of materials containing NPs was realized when it was discovered that size can affect the physicochemical properties of the substance, including optical properties [4]. The possibilities of their applications are widely described in the literature. In [5], the authors describe the use of various types of nanostructured materials (including mesoporous silica-based NPs) in medicine, including diagnostics and various types of therapy. Also, in this field, NPs are used as a contrast agent in magnetic resonance imaging and for transporting anticancer drugs directly to diseased cells [6]. A hydrogen sensor was made based on tin (IV) oxide (SnO_2) NPs [7]. On the other hand, lithium (Li) and zirconium (Zr)-doped silica NPs were successfully tested for CO_2 adsorption [8].

In recent decades, gold (Au) and titanium dioxide (TiO_2) NPs have attracted considerable interest due to their advantages, including biocompatibility, non-toxicity, corrosion resistance, and the possibility of functionalization and modification of optical and electronic properties [9]. Nanocomposite materials seem to be a promising and interesting solution from the point of view of designing and building a *refractive index* (RI) sensor that allows for measuring the refractive index. Adding NPs to the mixture with the monomer allows for modifying the reflective properties of the transducer, while maintaining geometric parameters such as size and shape of the microtip.

Gold reduced to nanometer size changes its physicochemical properties. The visible light absorption spectrum of Au NPs gives an absorption band of about 520 nm. The absorption band is sensitive to the size of the NPs and surface modifications. As a result of the reaction of the gold nanostructure with electromagnetic radiation, the localized surface plasmon resonance is observed, which is used in biomedicine. The optical sensitivity of Au NPs is crucial in surface-enhanced Raman

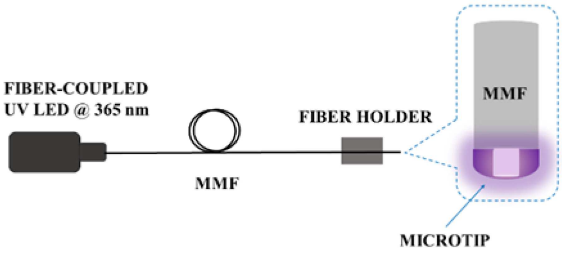

Fig. 1. The sketch of the experimental set-up for the microtip manufacturing.

scattering. Plasmonic NPs amplify the local electric field, which increases the strength of the Raman signal of molecules located in close proximity. This is particularly important in sensor applications [9]. Titanium dioxide (IV) NPs have versatile applications, including photovoltaic cells, solar filters, biodetectors, and drug delivery [10]. Moreover, due to their photocatalytic activity, these materials can be used to degrade organic pollutants from drinking water and industrial wastewater [11]. The advantages of photocatalysts are that they are self-regenerating, can be recycled, and do not require special oxidants.

2. Materials and methods

2.1. Nanoparticles used in experiment

The advantages of nanocomposites described above provided the basis for using selected NPs as additions to monomer mixtures. The following NPs were used in the studies: gold (courtesy of the Department of Materials Technology and Chemistry, Faculty of Chemistry, University of Łódź) and titanium dioxide (courtesy of the Institute of Optoelectronics, Military University of Technology (WAT)). For preliminary studies, the concentrations of admixtures calculated based on the weight proportions of all components were selected, and they were cured with UV light for the mixture: for Au 1% and 5% and for TiO_2 5% and 16.67%. Microtips manufactured on the *multi-mode fiber* (MMF) with 62.5 μm or 105 μm core diameter were analyzed in terms of geometry, surface structure, and optical properties.

2.2. Microtip manufacturing and sensor preparation

As described in the literature [12–15], the production of polymer microtips is based on the photopolymerization phenomenon. A monomer mixture drop was deposited on the fiber end face, and the other end was coupled to the light source (Fig. 1). Microtip was formed as the extension of the core thanks to the illumination of the hemispherical monomer drop. Such a microtip is ready as the sensor head for further testing.

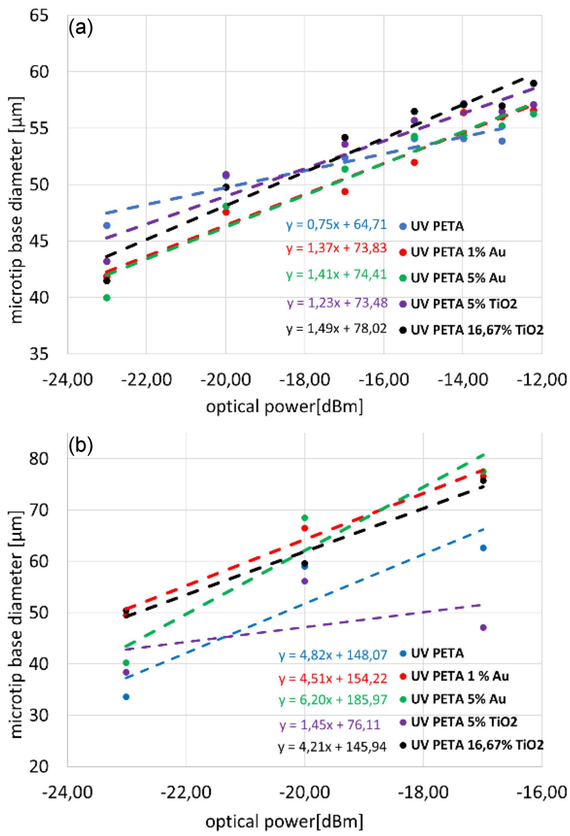

Fig. 2. Microtip base diameter vs optical power for UV PETA mixture and: (a) MMF 62.5 μm, (b) MMF 105 μm.

3. Experiment

3.1. Doping the polymer mixture with NPs

The dependence of the microtip base diameter on the optical power for the above-described selected concentrations of both dopants is shown in Fig. 2. The graphs show measurement data for MMF 62.5 (Fig. 2a) and MMF 105 (Fig. 2b), which were approximated by straight lines. All tested elements were dimensioned using *scanning electron microscope* (SEM) images and microscope software (Fig. 3).

Due to the different quality of optical fiber connections and the high attenuation of 365 nm wavelength, the optical power range for microtips forming was limited. Hence, the available optical power range for MMF 62.5 was from -23 to -12 dBm and for MMF 105 from -23 to -17 dBm.

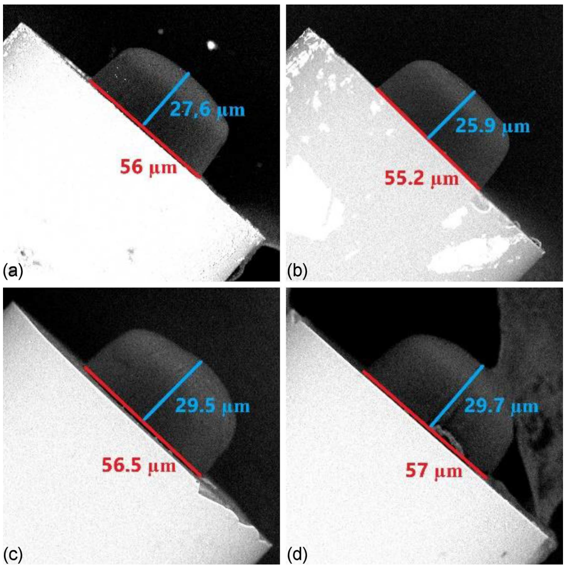

Fig. 3. SEM images with dimensions of microtips manufactured on MMF 62.5 μm using UV PETA with various admixtures: (a) Au 1%, height 27.6 μm, base diameter 56 μm; (b) Au 5%, height 25.9 μm, base diameter 55.2 μm; (c) TiO_2 5%, height 29.5 μm, base diameter 56.5 μm; (d) TiO_2 16.67%, height 29.7 μm, base diameter 57 μm. Optical power 50 μW, time 60 s.

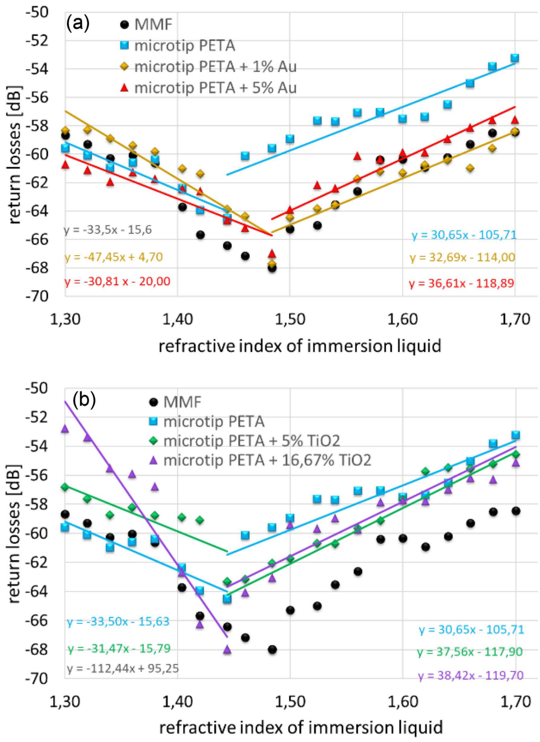

Fig. 4. Return losses of microtips vs refractive index MMF 105 UV PETA mixture with composites: (a) Au; (b) TiO_2.

With the use of both pure *pentaerythritol triacrylate* (PETA) mixture and nanocomposites, it can be noticed that microtip base diameter increases with the increase in optical power. The fastest increase was noted for microtips on the MMF 105 for the mixture doped with Au 5%.

However, no unequivocal effect of dopant concentration on the relation between the base diameter of the microtip and optical power was found. For all dopant concentrations, it has an increasing tendency, but it is not possible to determine a clear trend of these changes after adding dopants.

3.2. Microtips doped with NP materials as transducers for RI sensors

Polymer microtips can act as transducers for optical fiber RI sensors [16]. Nanocomposite materials added to the mixture make it possible to modify the optical properties of such a sensor.

This subsection presents tests using the OBR 4600 (Luna) device, which measures return losses [dB]. The tests were performed on microtips made on MMF 62.5 and MMF 105 using UV LED from a mixture without dopants and an NPs-doped mixture. Microtips were tested in the environment of immersion liquids with known refractive index values. As a reference, the return loss test of doped transducers in the air environment was used.

Figure 4 shows the return loss characteristics of sensors made on MMF 105, taking the characteristics of the fiber as a reference, i.e., without microtip and with microtip made of UV PETA material. Characteristics of sensors with nanocomposite microtip based on Au NPs and TiO_2 NPs were shown in Fig. 4a and Fig. 4b, respectively.

The return loss minimum for the sensor without a microtip was noted for a refractive index value of 1.48. Manufacturing a microtip from the *UV-cured pentaerythritol triacrylate* (UV PETA) mixture on this fiber caused a shift in this value to 1.44. In the case of Au 1% and Au 5% composites and TiO_2 5% and TiO_2 16.6%, the minimum occurred for the values of 1.48 (Fig. 4a) and 1.44 (Fig. 4b), respectively.

The presence of the NP composite in the mixture changes its refractive index and also creates strongly scattering centers, which changes reflected signal levels.

3.3. Transmission characteristics of microtips doped with NPs

The studies of polymer microtips manufactured at the end of selected optical fibers are supplemented by studies of light beam transmission through this type of optical elements.

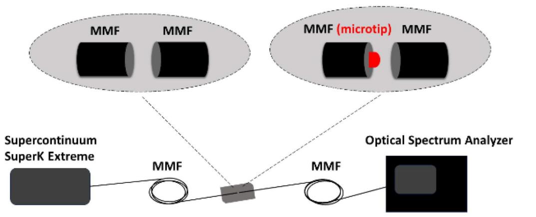

Fig. 5. The sketch of the experimental set up for testing the microtips transmission properties.

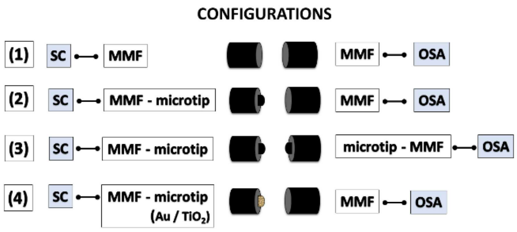

Fig. 6. Configurations of the microtip transmission properties testing system: (1) two MMF optical fibers, (2) a microtip on the transmitting fiber, (3) a microtip on the transmitting and receiving fibers, (4) a microtip doped with Au and TiO_2 on the transmitting fiber; SC — light source; OSA — spectrum analyzer.

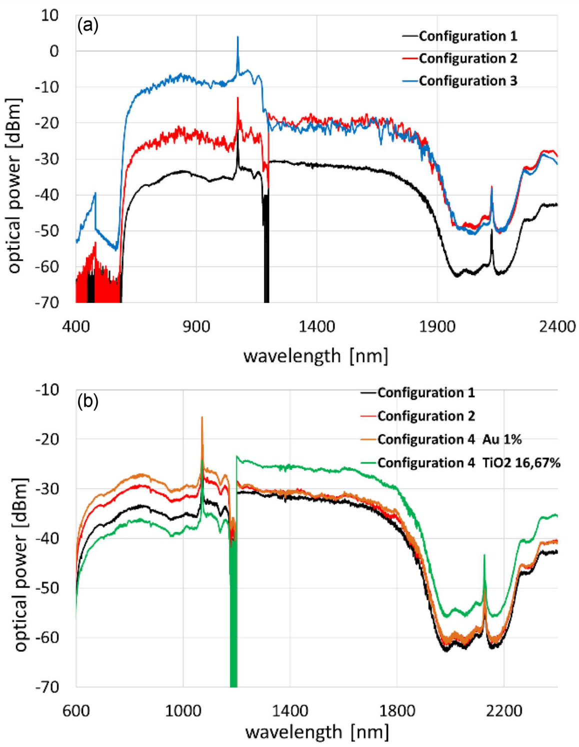

Fig. 7. Spectral characteristics in the transmission set-up — (a) configurations: 1 (black), 2 (red), 3 (blue); (b) configurations: 1 (black), 2 (red), 4 (1% Au yellow), 4 (16.67% TiO_2 green).

The experimental setup (Fig. 5) consisted of an MMF 105 or MMF 62.5 as a transmitting fiber coupled at one end to a broadband source SuperK EXTREME (NKT Photonics) with a spectral range of 400–2400 nm, and its other end, without or with a microtip, was directed to a receiving fiber, also MMF 105, which was connected to an *optical spectrum analyzer* (OSA) — AQ6373 (OSA, Yokogawa), in the range from 350 to 1200 nm, and AQ6375 (OSA, Yokogawa), in the range from 1200 to 2400 nm. The centering of these two fibers was possible thanks to the use of a linear stage, with the possibility of translation in three directions. The light transmission between axially centered fibers took place in free space.

Figure 6 shows all configurations in which transmission tests were performed. In the first experiment, two fibers without microtips were centered (configuration 1). Then, a microtip was produced on the transmitting fiber (configuration 2) and on the transmitting and receiving fibers (configuration 3). The last step was the measurement of MMF with a microtip made of a mixture doped with NPs: Au 1%, Au 5%, TiO_2 5%, and TiO_2 16.67% (configuration 4).

As one can see (Fig. 7a), creating a microtip on the transmitting fiber resulted in improved transmission in the spectral range of 600–2400 nm. Additionally, in the range of 400–1200 nm after the production of an additional microtip on the receiving fiber (configuration 3), higher values of the transmitted signal were obtained, which was not observed in the range above 1200 nm, for which the signal level for configurations 2, 3 was the same.

Transmission properties were changed after adding NPs to the polymer mixture. The manufacturing of a microtip doped with 1% Au on the transmitting fiber (Fig. 7b) resulted in transmission improvement (an increase in the transmitted signal level was in the range of 600–1200 nm). An improvement in the infrared transmission was obtained with a microtip doped with 16.67% TiO_2 (Fig. 7b). Interestingly, for the wavelength range below 1200 nm, doping with TiO_2 caused a significant decrease in the signal level.

4. Conclusions

Polymer microtips fabricated at the ends of MMF are used as transducers to create optical fiber sensors. Doping the mixture with NPs changes its optical properties, but does not change its shape and size. Microtip base diameter increases with the increase in optical power. However, the admixtures do not significantly improve this trend.

Adding NPs to the mixture causes reduced reflected signal levels and a lower value of return losses, especially visible in the range of 1.45–1.7

of RI. Moreover, shifts in return loss minima for the RI sensors were observed. The minimum was shifted from 1.44 to 1.48 after adding gold NPs (Au 1% and Au 5%) to the mixture.

Polymer microtips can act as scattered lenses at the ends of optical fibers. Doping the mixture with a 1% Au composite resulted in improved transmission properties in the 600–1200 nm spectral range. An improvement in transmission in the infrared range was obtained for the microtip doped with 16.67 % TiO_2. Doping with titanium dioxide resulted in a significant decrease in signal in the wavelength range below 1200 nm.

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