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D-shape polymer optical fibres for surface plasmon resonance sensing

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ABSTRACT

We experimentally studied three different D-shape polymer optical fibres with an exposed core for their applications as surface plasmon resonance sensors. The first one was a conventional D-shape fibre with no microstructure while in two others the fibre core was surrounded by two rings of air holes. In one of the microstructured fibres we introduced special absorbing inclusions placed outside the microstructure to attenuate leaky modes. We compared the performance of the surface plasmon resonance sensors based on the three fibres. We showed that the fibre bending enhances the resonance in all investigated fibres. The measured sensitivity of about 610 nm/RIU for the refractive index of glycerol solution around 1.350 is similar in all fabricated sensors. However, the spectral width of the resonance curve is significantly lower for the fibre with inclusions suppressing the leaky modes.

1. Introduction

Surface plasmon resonance (SPR) sensors are used in many fields of science, including chemistry and biology [1–3]. The operation principle of SPR sensors relies on a spectral shift of a resonant coupling between plasmon and incident light, observed in response to small variations in the refractive index of the medium covering a metal layer [4]. In the most frequently used prism coupling configuration, the displacement of resonance related to the change in a refractive index is determined either as a function of a wavelength at a fixed incidence angle or as a function of an incidence angle for a fixed wavelength. Fibre-optic SPR sensors have many advantages over traditional sensors based on prism coupling, such as simpler design, no demand for control of an incidence angle, possibility of remote sensing, and small size of a sensing head [5]. Different designs of fibre-optic SPR sensors have been reported in literature so far, including sensors based on single mode fibres [4–7] and multimode fibres [8–10]. In the latter case, the spectral width of resonance is broader due to many guided modes which results in less precise measurements of the resonance position [11]. On the other hand, the SPR sensors based on multimode fibres are characterized by a simple construction of the sensing head and allow for an easy coupling with broad band light sources and, thus, assuring convenient resonance detection [9].

So far, several techniques have been reported in literature to expose the core of multimode fibres to allow deposition of a metal layer, including the stripping of the fibre cladding [12], fabrication of a sensing tip [9], tapering [13,14] and side-polishing [8,15–20]. All these methods, however, require a laborious and time consuming fibre processing. Another approach discussed in literature for both multimode and single fibres is the use of specially designed microstructure. In 2008, Hautakorpi et al. theoretically analyzed an SPR sensor construction based on a single mode suspended core microstructured silica optical fibre [21]. The main practical difficulty related with the fabrication of such a sensor is the metal deposition onto the inner surface of air holes. The chemical methods were used for coating the inner surfaces of air channels of a microstructured cladding [22]. However, the SPR sensors using such a deposition technique have not been fabricated yet. One of the attempts to overcome the problems with metal deposition onto the inner surfaces of air-holes reported in Ref. [23] was a development of a sensing fibre-optic structure with metal nanowires.

A process of metal deposition is the simplest for fibre-optic structures with an exposed core such as D-shape fibres. A single-mode silica microstructured D-shape fibre formed by side-polishing was studied numerically in Ref. [24] for possible applications as a SPR sensor with increased sensitivity obtained by applying a phase sensitive detection method. In Ref. [25] the SPR sensor based on a few-mode side-hole polymer fibre was studied both numerically and experimentally. Opening one of the side-holes over a short fibre distance allowed to deposit a metal layer directly on the fibre core by a sputtering method. Investigations of the sensor based on multimode silica microstructured fibres with

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the core exposed along the whole fibre length was recently reported in Refs. [26,27]. A special fibre structure with a side-opened suspended core allowed for easy silver deposition on the fibre core by thermal evaporation [26] while in Ref. [27] a silver layer was deposited by an electro-less plating method.

Nowadays most of the SPR fibre optic sensors reported in literature are based on silica optical fibres. However, polymer optical fibres (POFs) can be more advantageous in some applications due to their low cost, flexibility and simplicity of SPR sensors fabrication. Therefore, in this work we have proposed and studied different designs of conventional and microstructured D-shape polymer optical fibres with an exposed core that can be used as SPR sensors without troublesome fibre preprocessing. A flat sidewall in the developed D-shape fibres forms a boundary of the fibre core, thus allowing for convenient deposition of a metallic layer which can directly interact with the light guided in the core. All the fibres are multimode which, additionally, facilitates the fabrication of SPR sensors. The first of the investigated D-shape fibres has no microstructure while in two others the core is surrounded by two rings of air holes. Additionally, special absorbing inclusions are placed in the cladding of one of the microstructured fibres to attenuate leaky modes. For the first time to our knowledge, we report experimental investigations of this type of SPR sensors made in polymer fibres. In particular, we have shown that there are no significant differences in the sensitivity of the sensors fabricated in different types of D-shape fibres. However, the spectral width of the SPR resonance can be decreased by a proper design of the fibre structure. We have also demonstrated that the fibre bending strengthens the surface plasmon resonance in the investigated fibres.

2. Fabricated D-shape fibres

In Fig. 1, we have shown the optical micrographs of multimode polymer D-shape optical fibres drawn by the Laboratory of Optical Fibre Technology, Maria Curie-Sklodowska University, Lublin, Poland. The fibre no. 1 has no microstructure while two other fibres have a microstructured cladding consisting of two rings of air holes. A primary material used for the fabrication of the investigated optical fibres is polymethyl methacrylate (PMMA) of a technical grade. The microstructured cladding was fabricated by drilling holes into the PMMA rod. Then the D-shape preforms were made by planar grinding/polishing the side of initially cylindrical PMMA rods. Owing to an early stage of development of the manufacturing technology, diameters and shapes of holes vary in the fibre cross-section. In consequence, we observed an increased leakage of light from the core of the microstructured fibres through PMMA bridges adjacent to the flat fibre surface as the dimensions of air holes decrease. Hence, in case of the fibre no. 3, additional inclusions in the solid part of the cladding were introduced to inhibit light propagation outside the microstructure. To fabricate the inclusions with a greater refractive index than surrounding PMMA, four holes were additionally drilled and filled with a liquid mixture of styrene, benzyl peroxide (initiator), thioglicolic acid (moderator), and a small (0.01%, w/w) amount of carbon micropowder.

From the micrographs shown in Fig. 1 we determined the geometrical parameters averaged in the fibre cross-sections which are gathered in Table 1. In this table we also show the loss of all the fibres measured at 0.8 µm using a cut-back method. The repeatability in manufacturing the polymer microstructured optical fibres is not yet satisfactory, therefore the diameters and shapes of holes vary in the fibre cross-sections. Moreover, we observed a few percent variations in geometrical parameters along the length of microstructured fibres. Nonetheless, the effect of bending on surface plasmon resonance studied in this work was qualitatively

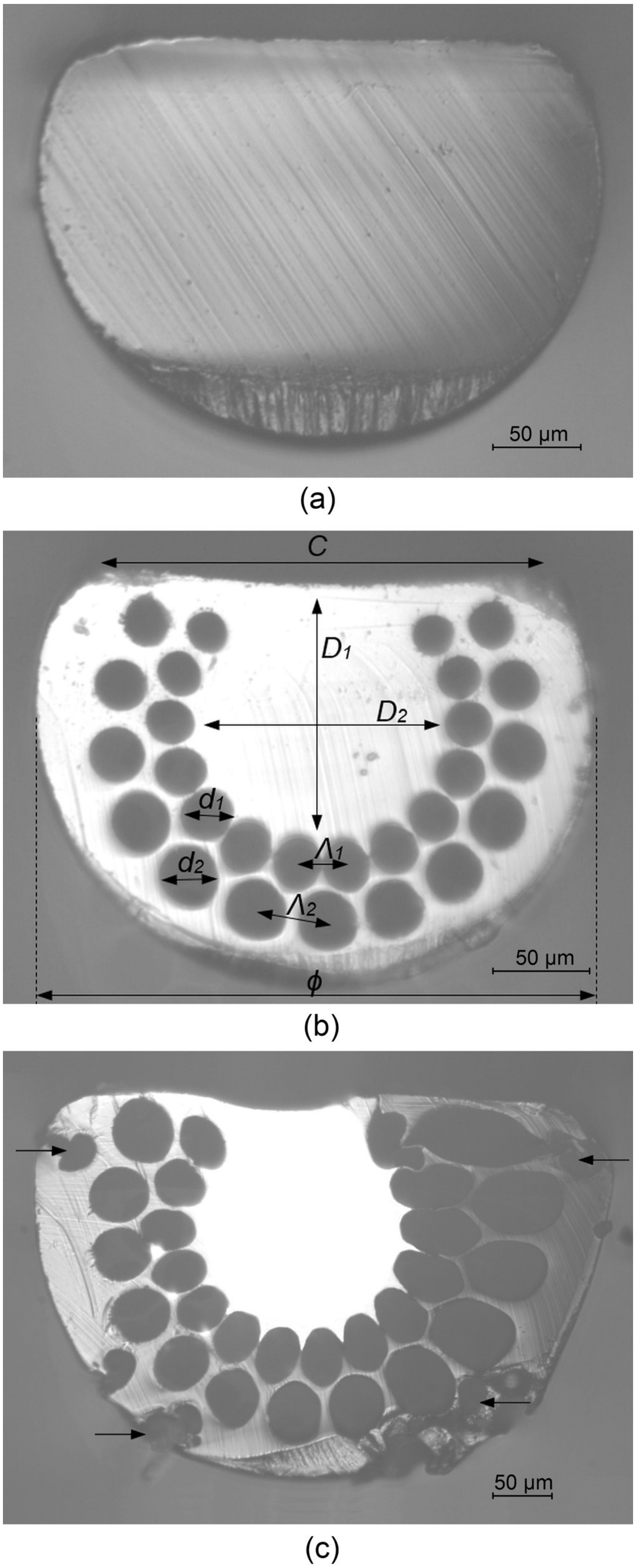

Fig. 1. Optical micrographs of fabricated D-shape fibres: (a) fibre no. 1 – conventional fibre with no microstructure, (b) fibre no. 2 with the core surrounded by two rings of air holes, (c) fibre no. 3 with the core surrounded by two rings of air holes, and additional polystyrene inclusions containing carbon micropowder indicated by arrows. Micrographs of microstructured fibres show light distribution across the fibre cross-sections.

Table 1
Geometrical parameters and loss of the fabricated D-shape fibres.

	Fibre no. 1	Fibre no. 2	Fibre no. 3
Φ [μm]	291	286	456
D_1 [μm]	208	126	186
D_2 [μm]	291	121	170
C [μm]	249	225	365
d_1/Λ_1	–	0.81	0.95
d_2/Λ_2	–	0.78	0.85
Loss [dB/m]	4	7	14

repeatable for every investigated fibre and the same behaviour of the SPR sensors fabricated on different sections of each investigated fibre was observed.

3. Experimental results

The SPR sensors were made by covering the flat core surface over a distance of 5 mm with a gold layer of a thickness of 40 nm deposited in a plasma sputtering machine (Q150T ES, Quorum Technologies). The process was conducted in a vacuum chamber at a pressure of 5×10^{-5} mbar. The thickness of the deposited gold layer was controlled by a standard film thickness monitor based on quartz crystal. We fabricated and studied both straight and U-bent SPR sensing probes. In the second case, the fibre with a deposited gold layer was bent and fixed in a v-groove of a half-circular shape made in a PMMA plate (Fig. 2). To study the effect of fibre curvature on the performance of the U-bent SPR sensors, we used the plates of different radii R = 10, 20, 30, and 40 mm, respectively.

The performance of the SPR sensors was investigated using an experimental set-up based on a wavelength interrogation method. A halogen lamp was applied as a light source, while the spectrum of transmitted light was measured at the fibre output by means of a miniaturized spectrometer (Ocean Optics USB4000). As a test solution we used glycerol-water mixtures with different refractive indices measured by an Abbe refractometer at the wavelength of 589 nm. The SPR transmission spectra registered after the application of glycerol solutions were normalized with respect to the air spectrum used as a reference.

We first investigated the effect of different fibre designs on the resonance characteristics. For this purpose, in each of the investigated fibres we made the SPR sensor with the same length of the sensing section equal to L = 5 mm. In Fig. 3 we present the resonance curves obtained for the three sensors for the refractive index of glycerol solution n_s = 1.332 and different bending radii R. The resonance occurs at around 600 nm for all the investigated sensors and it shifts towards longer wavelengths in response to a curvature increase. Similar effect was observed for internally tilted long-period gratings [29]. We clearly see in Fig. 3 a significant increase in the resonance depth *vs*. the fibre curvature 1/R. In the short-wavelength range, the sensors transmission decreased to 90% which is most probably caused by the fibre loss related to the presence of a glycerol solution on the fibre cladding and has nothing to do with the SPR effect. Consequently, the resonance depth was determined with respect to the baseline obtained by a linear approximation of transmission data in the spectral intervals of 485–500 and 750–850 nm. The resonance width was determined at half of its minimum (FWHM) measured with respect to the baseline.

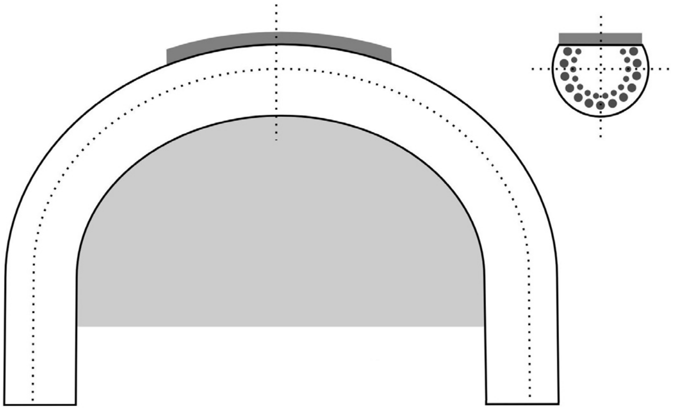

Fig. 2. Schematic view of the U-bent SPR sensing probe made of a D-shape microstructured fibre.

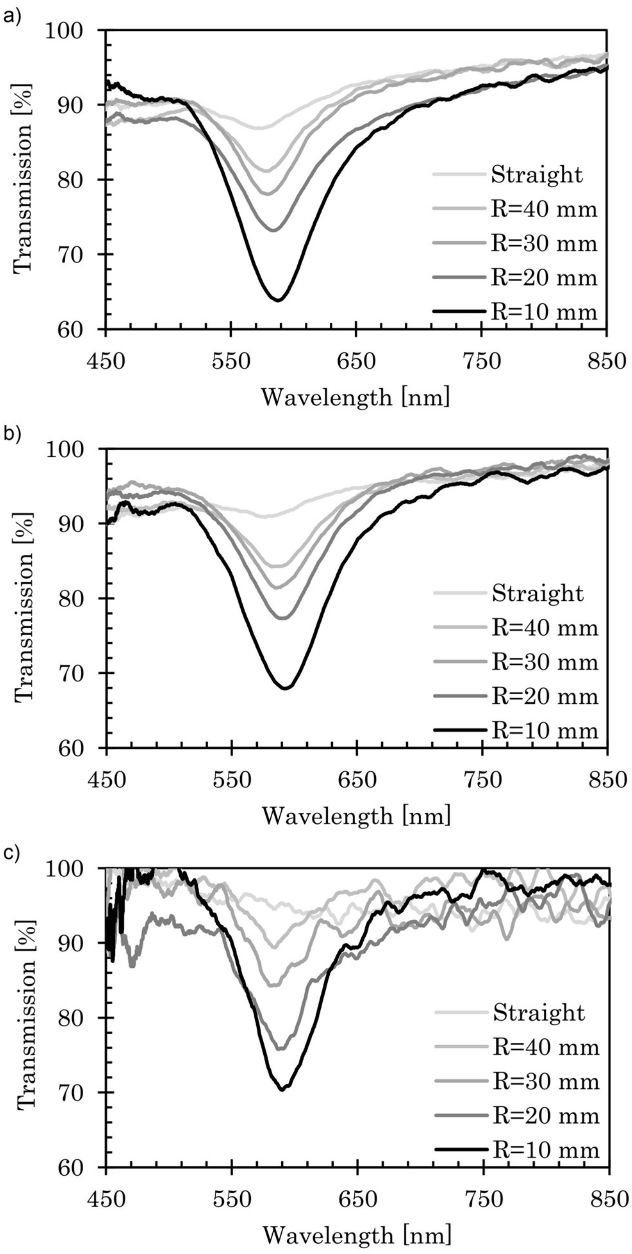

Fig. 3. SPR transmission spectra registered for the refractive index of the glycerol solution n_s = 1.332 and various bending radii of the sensing probes made on fibre no. 1 (a), fibre no. 2 (b), and fibre no. 3 (c).

The results of measurements of the resonance depth ΔT and FWHM *vs*. the fibre curvature 1/R are presented in Fig. 4. To verify the repeatability of the sensor response to bending, the fibre bending procedure was repeated 5 times. In the conducted series of experiments, the measured values for the resonance depth ΔT and

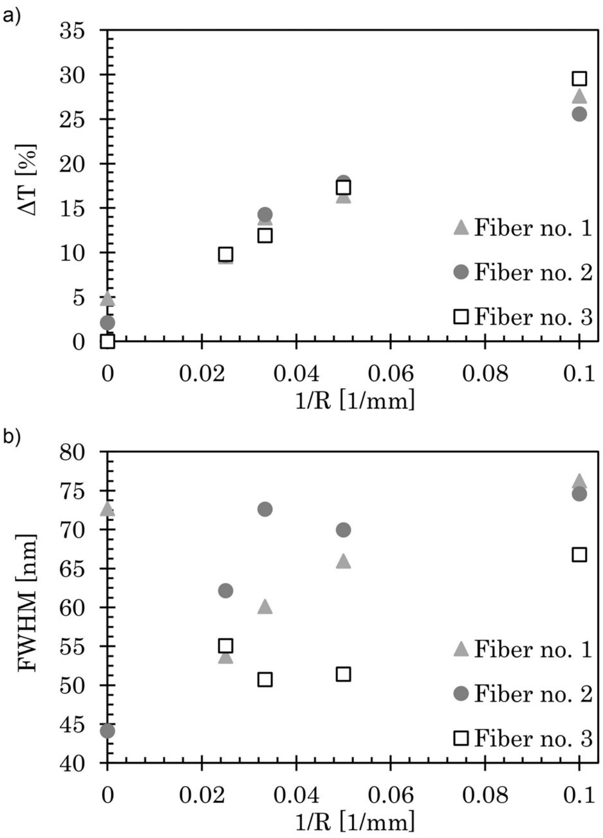

Fig. 4. Resonance depth ΔT (a) and resonance width FWHM (b) measured as a function of the fibre curvature $1/R$ for sensing probes made on different fibres.

FWHM were scattered depending on the fibre curvature within the margin of 1–3% and 4–7 nm, respectively. For the smallest radius of curvature $R = 10$ mm, the resonance depth ΔT reaches 28, 26 and 30%, respectively for the fibre no. 1, 2 and 3 [Fig. 4(a)]. On the other hand, for $R = 10$ mm the lowest value of the FWHM equal to 67 nm is achieved for the fibre no. 3, while for other fibres the FWHM is similar and equals 76 and 75 nm, respectively for the fibre no. 1 and 2 [Fig. 4(b)]. Therefore, the obtained results show that the SPR resonances in the fibres no. 1 and 2 do not differ significantly because the addition of two rings of air holes is not sufficient to keep the guided light in the fibre core. For the fibre no. 1, the light distribution is uniform in the whole cross-section as there is no microstructure. In the fibre no. 2, the core is surrounded by two rings of air holes but, as it is shown in Fig. 1(b), the modes guided in the core leak out of the microstructured cladding, especially through the region between the air holes and the flat surface of the fibre. In consequence, the numerical aperture and the number of modes guided in the fibre no. 1 and 2 do not differ significantly. As a result, the characteristics of the SPR resonance obtained for these two fibres are similar.

To suppress the light leaking from the core, we have added polystyrene inclusions containing carbon micropowder in the solid part of the cladding of the fibre no. 3. As it is shown in Fig. 1(c), in this fibre the light is guided mostly in the core, which confirms that leaky modes are highly attenuated. It means that the effective numerical aperture of the fibre no. 3 is lower and consequently lower is the number of guided modes. As a result, we observed the decrease in the resonance FWHM. Our observations are in agreement with the results reported in Ref. [27] for the SPR sensor based on a fibre with a side-opened suspended core. When a light was launched into the cladding of such a fibre supporting multimode propagation, a broadening of the SPR resonance was visible.

Table 2
Parameters of the SPR sensors based on multimode microstructured fibres investigated in this work compared to literature data.

	S [nm/RIU]	FWHM
Fibre no. 1, $R = 10$ mm	730 ($n_s = 1.410$)	76 ($n_s = 1.332$)
Fibre no. 2, $R = 10$ mm	735 ($n_s = 1.410$)	75 ($n_s = 1.332$)
Fibre no. 3, $R = 10$ mm	728 ($n_s = 1.410$)	67 ($n_s = 1.332$)
Side-hole fibre [25]	2000 ($n_s = 1.38$–1.41)	170 ($n_s = 1.38$)
Fibre with exposed core [26]	2344 ($n_s = 1.330$–1.386)	44
Fibre with exposed core [27]	1753 ($n_s = 1.33$–1.37)	77

The proposed construction of the fibre no. 3 results in a decrease in the SPR resonance FWHM which potentially allows for more precise measurements of the resonance wavelength. As it is seen in Fig. 3(c), due to an increased noise level related to high attenuation of this fibre, the exploitation of this feature is currently limited to the smallest bending radius ($R = 10$ mm) yielding the deepest resonance and, therefore, a good signal to the noise ratio.

It should be also stressed that despite the usage of highly multimode fibres we observed a significant reduction in the resonance width (FWHM = 67 nm) compared to the SPR sensor made on the side-hole fibre guiding a few spatial modes (FWHM of about 170 nm) [25]. Most probably, the reduction in the SPR resonance width in our sensors is caused by improved smoothness of the metal layer which was deposited on the flat fibre surface obtained by polishing the preform. For other SPR sensors made on multimode microstructured optical fibres with an exposed core, the resonance FWHM was about 44 nm [26] and 77 nm [27] (see Table 2), but the SPR spectrum was additionally modulated due to intermodal interference, which obstructs precise measurements of the resonance wavelength.

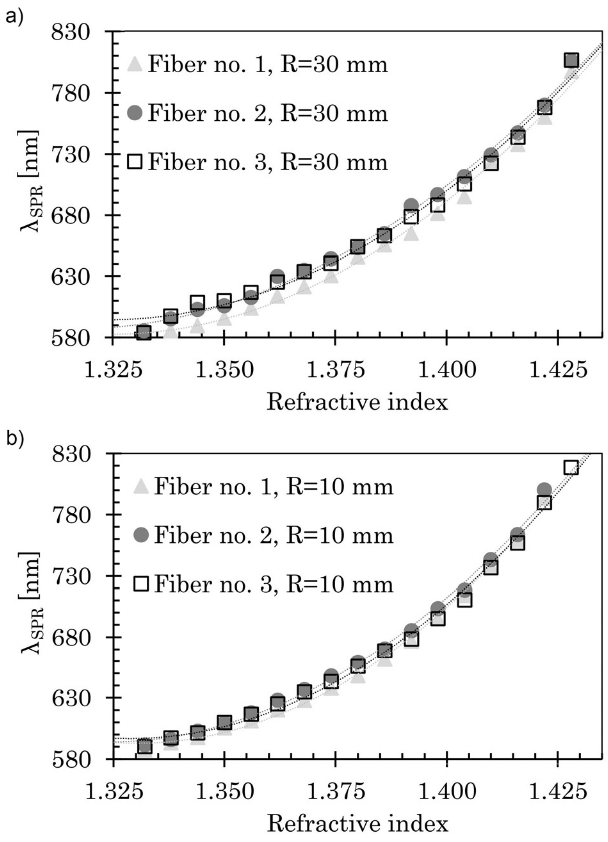

Fig. 5. Resonance wavelength λ_{SPR} measured as a function of the refractive index of glycerol solution for U-bent sensing probes made on different fibres: $R = 30$ mm (a) and $R = 10$ mm (b).

Finally, we measured the change in the resonance wavelength λ_{SPR} in response to the variation of the refractive index of the glycerol solution n_s for U-bent sensing probes. The results obtained for the probes with $R = 30$ and 10 mm are shown in Fig. 5. The dependence of the resonance wavelength upon the refractive index change is nonlinear. Thus, for $R = 30$ mm and $n_s = 1.350$, the sensitivity ($S = d\lambda_{SPR}/dn_s$) calculated by polynomial fitting of the experimental data equals $S = 603$, 611 and 616 nm/RIU, respectively for the fibre no. 1, 2 and 3, while for $n_s = 1.410$ it increases correspondingly to 730, 735 and 728 nm/RIU. The sensitivities measured for the lower bending radius $R = 10$ mm take similar values as for $R = 30$ mm. In our previous work [28], a small dependence of the sensitivity upon a bending radius was reported. This discrepancy is related to a greater diameter of the fibre used for the fabrication of sensors reported in Ref. [28] which was equal to 980 μm. Consequently, the bending induced deformation of the external layer of the cladding, which scales up with the fibre diameter, is a few times lower in the fibres reported in this work compared to Ref. [28], thus giving a practically null impact on the sensors sensitivity.

4. Conclusions

We have fabricated low cost multimode polymer optical fibres designed for SPR sensing applications. A D-shape of the fibres allows for an easy fabrication of SPR sensing probes in straight or U-bent configurations. The experimental studies of the SPR sensors made on different fibres showed that a significant enhancement of the surface plasmon resonance can be achieved by bending the sensing probe covered with a metal layer. For the bending radius equal to 10 mm, we obtained the resonance of about 30% deep for 5 mm long sensing probes made on different fibres. We also demonstrated that introducing absorbing inclusions in the solid part of the cladding outside the microstructure attenuates the leaky modes and in consequence narrows the resonance of FWHM to 67 nm. Finally, we measured the sensitivity of U-bent sensing probes to refractive index changes of glycerol solution. Although bending of a sensing probe clearly amplified the resonance depth, we did not observe a significant effect of bending on of the SPR sensors sensitivity. This parameter was practically the same for the sensors fabricated in different D-shape fibres and was equal to about $S = 610$ nm/RIU for $n_s = 1.350$ and $S = 730$ nm/RIU for $n_s = 1.410$.

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