Sensitivity of a PMMA polymer capillary microresonator for measuring relative humidity.

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Abstract. This paper studied experimentally the behavior of a capillary microresonator made from PMMA polymer as a sensor for measuring relative humidity. In the manufactured device, the WGMs modes within the microcavity are excited by the proximity of an optical fiber Taper made from the stretching of a standard optical fiber of silica by the method of scanning flame with waist of the order of 3-5 µm. In the device, the field from a tunable laser system TLS is coupled into the capillary along its cross section where the resonant modes were observed WGMs into the cavity. When the system is subjected to change of the relative humidity of the external environment, the wavelengths of peaks resonances of WGMs modes of the resonant system experience a spectral shift, so that a sensitivity of the microresonator is observed at changes in humidity of the external environment. During the experiment, it was manufactured capillaries with different diameters and different wall thickness obtaining a sensitivity of the order of 0.07 nm/% RH for a capillary thickness 42.1 µm.

1. Introducción

The optical resonators play an important role in modern optics, being not only important for the manufacture of Laser, but are constituted as indispensable devices for performing accurate measurements, experiments nonlinear optics and other applications. One of the objectives most important in manufacturing such devices, is the miniaturization of optical resonators made from different materials, the most important materials that have been reported, Silice, Silica and some types of polymers, which have enabled the interconnection with integrated optical systems used for optical telecommunications. These devices have great attention due to their ability to confinement of light inside, which has led to breakthroughs in the area of sensors applied to biology, physics, chemistry and the study of nonlinear optical effects.

Currently, these structures are designed with dimensions on the order of microns to adapt easily to dielectric structures of waveguides and high symmetry to take advantage of the features and their properties, including their relative ease for mathematical modeling.

In the manufacture of microcapillaries silica and polymers, has led to the development of sensors for applications in measuring physical variables, chemical and biological [1-4]. Some measurement techniques reported in the literature have used the microcapillary as cavity for confining the field within it through the excitation of the WGMs modes within the cavity using different techniques, such as coupling technique through optical fibers Taper [1-3].

The capillaries thin wall used as optical microcavities are important in the development of sensors to study properties microfluidic due to their small size, low modal volume and relatively high Q factor, so that, due to its structural design can be used as a channel for the transit of small amounts of liquids

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and gases, which depending on the type of study being performed can modify the characteristics of the optical field is confined inside.

2. WGMs modes within a cylindrical dielectric cavity.

The dielectric structures having a circular symmetry, can confine the field inside by total internal reflection within its surface, whose propagation modes are commonly called Whispering Gallery Modes WGMs. Indeed, in the literature is reported the excitation of WGMs modes within cavities with rotational symmetry in the form of spheres, cylinders, toroids, rings and discs made from different materials, which constitute a resonant structure where a field external is introduced into the cavity. Therefore, in such structures is of importance to study the wavelengths/frequencies of resonance, the Q factor of the oscillations within the cavity, the free spectral range FSR and the maximum width at half maximum FWHM.

In the case of a resonant structure in the form of capillary, the light is trapped by the outer wall by total internal reflection when this flows along of the circumference of the capillary, where the study of the wavelengths of the resonant modes WGMs allows its use as an optical sensor.

The cylindrical cavities, present a set of analogous resonances the case of structures in the form of spheres, where the main difference is supported by the fact that the cavities have a capillary not support the polar field component having spherical cavities, as it assumes that the field confinement occurs through the cross section of a two-dimensional cylinder. In this sense, the expressions used to determine the resonances in both resonators is the same with the difference that in the case of a cylindrical structure the functions of cylindrical Bessel is used while in a spherical structure Bessel functions spherical used as the solution of Maxwell's equations with boundary conditions.

Some studies report the analytical and experimental study of the optical microcavities as capillaries to very specific practical situations, such as refractometer sensor [3] and hydrostatic pressure sensor in microfluidics [1]. In the case of this research we are interested in the study of the sensitivity of the cavity to changes in relative humidity of the medium, which is determined from shifts in the wavelength of peak resonances obtained experimentally. In such systems a theoretical analysis based on Maxwell's equations with boundary conditions can determine the sensitivity of the devices, the Q factor of the oscillations and the FSR of the cavity.

The excitement of the WGMs modes inside a microcapillary could contain losses curvatures when the diameter of the microcapillary is the order of the micron low and may have differences in index of major refraction between the internal or external environment with the material capillary. Similarly, the resonances obtained in different modes can achieve important factor Q enabling improved signal/noise ratio in measurement applications and sensitivity.

With respect to coupling of the field within this type of microcavities, the power transferred from the optical fiber tapered to the microcavity is optimal when there is significant overlap of the field between the modes of the coupled structures, wherein the coupling length depends on the proximity of the fiber Taper. Therefore, it is possible to achieve a process of efficient power transfer when the optical modes have equal phase, which depends on the geometry of the resonant structure and the type of material used in its manufacturing process. For coupling the field within this type of resonant dielectric structures such as microcavities as spheres, discs, toroids, rings, bottles, bubbles etc., have specifically reported two types of techniques used for coupling of the field from a source coherent light known as coupling prism and optical fiber Taper coupling.

In the case of the technique of coupling prism, this has been used to couple the field into microspheres and microtubes through total internal reflection in the microsphere-prism interface obtaining an efficient coupling of the field into the microcavity, however, this technique has not been widely used because of the complexity of the resonant system related to the effect of displacement Goos-Hanchen and the precision of the instruments used [5]. In the case of the microresonators in the form of toroids, discs, bottles, bubbles and even into bottles and rings they have been reported various studies where the field confinement is achieved through an optical fiber taper, which can reach easily required phase equalization between the modes of the cavity and the optical fiber taper. This technique last coupling of the field has proven efficient results for two fundamentals reasons: The first one, is related to the manufacturing techniques used to design of fiber taper with specific characteristics in the fibers, which are relatively easy and the other reason is related with its ease of interconnection with standard telecommunication systems through optical fibers [1,3,6]. For a possible theoretical study of
the problem, it is proposed to study the structure as a system composed of three layers where Maxwell’s equations apply with the boundary conditions appropriate to determine the wave equation in the microcapillary which can obtain the wavelengths resonances and the field distribution of the corresponding modes with different radial orders WGMS. To understand the model, Figure (1) illustrates the general scheme of the dielectric structure designed to confine the field inside.

Figure 1. a) General outline of the microcavity for confinement of modes WGMS inside. b) three-layer system in a cylindrical shape with internal and external radii \( b \) and \( a \) respectively and with different refractive indices.

For the theoretical analysis, the symmetry of the cavity is exploited and cylindrical coordinates \((r, \theta, z)\) whose origin is the center of the capillary are chosen. In the microcavity, the internal radius \( b \) has a refractive index \( n_1 \), the cavity wall has a thickness \( a - b \), where \( a \) is the outer radius having a refractive index \( n_2 \) and the external medium has a refractive index \( n_3 \). In the device, the field confinement is realized in the medium with refractive index \( n_2 \) which is physically wall structure. In the study of the field distribution of the problem, it is required to know the states of polarization of the field within the cavity. These polarization states are classified according to how electric and magnetic fields propagate with respect to the plane of propagation of the WGMS modes and which are known as TE and TM modes.

3. Experimental setup.
For the resonant structure can be used as an optical measuring system sensitive to changes in humidity, it is required to couple the field inside the microcavity. For this, the technique used is through a taper optical fiber, which allows the excitation of resonant modes WGMS within the cavity. In the manufacturing process of the taper optical fiber, it was used flame brushing technique, which is suitable due to its high flexibility for controlling the movement of the flame, the length and speed of fiber drawing.

During manufacture was used an optical fiber commercial step index SMF 20 Corning, where a small portion of the fiber in the order of millimeters is selected to remove the coating and then subjected to the heat produced by a flame that heats fiber at a temperature of 1500 °C, while simultaneously the fiber is attached from the ends until achieving stretching and thus obtain the desired profile. During this process, fibers are obtained with neck diameters of 2-5 µm, sufficient to ensure optimal coupling of the field inside the cavity. In the test transmission fiber, is used a tunable laser system TLS with operating wavelengths in the range of 1500-1600 nm and optical spectrum analyzer OSA, mounted BraggMeter FS 2100 model Fibersensing with a resolution of 10 pm. The diagram of experimental setup for obtaining transmission spectrum of manufactured fibers is shown in Figure 2a, while in Figure 2b the spectrum of transmission obtained for a taper fiber of 4 µm in diameter is observed in the range of 1500-1600 nm of wavelength. In the same figure, a close is performed on the spectral response of the fiber in the range of 1562-1572 nm wavelength.

For the manufacture of the microcavity, they were manufactured microcapillaries PMMA by stretching a preform tube from a tower PMMA optical fiber design polymer. For stretching the preform tube, it requires heating the tube to a temperature of 210 °C and then you stretch to reach diameters and the desired wall thickness. In Figure 3, microscope images of some of the samples manufactured are observed. For the experimental setup performed, it was necessary to manufacture different capillary samples with different geometrical parameters.
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The general diagram of the experimental setup is shown in Figure 4a and the general structure of the system for coupling the field to the cavity shown in Figure 4b.
In the figure it shows that the taper–capillary fiber system is coupled within the humidity chamber. The humidity inside the chamber is controlled electronically and the tapered fiber is connected to the TLS / OSA for the analysis of the displacement of the peaks of resonances observed at the output of the taper, which verifies the resonant characteristics of the system. In Table 1, the geometrical parameters and experimental sensitivities of each of the samples obtained in the laboratory are observed.

| SAMPLE      | External diameter (µm) | Wall thickness (µm) | Humidity up (nm/% RH) | Humidity down (nm/% RH) |
|-------------|------------------------|--------------------|-----------------------|-------------------------|
| Sample Nº 1 | 2300,22                | 42,10              | Test A: 0,05247       | Test A: 0,07139         |
| Sample Nº 2 | 306,81                 | 78,00              | Test A: 0,05265       | Test A: 0,06485         |
| Sample Nº 3 | 1143,59                | 101,00             | Test A: 0,02661       | Test A: 0,03336         |
| Sample Nº 4 | 2180,37                | 50,70              | Test A: 0,04548       | Test A: 0,0608          |
| Sample Nº 5 | 1231,07                | 109,73             | Test A: 0,02008       | Test A: 0,02989         |

The output of the taper, we allowed to determine the transmission spectrum of each of the samples produced through the OSA, obtaining peaks characteristic resonances of resonant systems. In Figure 5, the transmission spectrum of the capillary sample No. 2 is shown in the range of 1530 to 1535 nm wavelength to a fixed humidity value of 65% RH. With the information provided by the resonance peak of Figure 5b, we obtain a factor $Q \approx 34 \times 10^3$ which is approximately equal to the case of silica capillaries reported in the literature [3].

In order to determine the sensitivity of the device to changes in humidity medium, it was necessary a humidity chamber in an operating range between 53-78% RH. For measuring humidity it was used a PCE-555 hygrometer PCE Intruments with an accuracy of 0.1% RH.

Figure 6 shows the displacement of the resonance peaks for different values of the relative humidity in the range from 60.6 to 64.0% RH. The curves were obtained with relative humidity up to higher values, for which a shift of the resonance peak of black color is initially centered at a wavelength of 1534.3248 nm at a relative humidity of 60,6 % RH is observed, however, by changing the relative humidity of the medium, the central wavelength of the peak is shifted to longer wavelengths.

Figure 5. Peaks resonances of the sample No. 2. a) Curves resonances in the range of wavelengths 1530 to 1535 nm. Figure 5. b) Resonance peak centered at 1530.68 nm.
Figure 6 shows the displacement of the resonance peaks for different values of the relative humidity in the range from 60.6 to 64.0% RH. The curves were obtained with relative humidity up to higher values, for which a shift of the resonance peak of black color is initially centered at a wavelength of 1534.3248 nm at a relative humidity of 60.6 % RH is observed, however, by changing the relative humidity of the medium, the central wavelength of the peak is shifted to longer wavelengths.

![Displacement of the resonance peaks.](image)

**Figure 6.** Graph of intensity Vs wavelength peak resonances at different values of relative humidity.

The peak shifts of resonances in Figure 6, corresponds to the capillary sample No. 1, whose geometric parameters are summarized in Table 1. During the experiment, was given the experimental sensitivity of the sensors manufactured for several times to determine the reproducibility of the experiment.

![Graph of sensitivity for sample No. 1 obtained from two different tests (Test A and Test B) with increasing and decreasing humidity.](image)

**Figure 7.** Graph of sensitivity for sample No. 1 obtained from two different tests (Test A and Test B) with increasing and decreasing humidity.

Figure 7 shows the results of the reproducibility in the experiment for the sensitivity of sample No. 1. The experiment was initially developed with humidity ascending and then descending in a controlled manner. In the results, it was analyzed hysteresis in sensitivity measures for changing a relative humidity of up and down manner, which results are shown in Figure 8.
4. Analysis and conclusions

Hysteresis in the analysis performed, it is evidenced that the sensitivity of the different samples analyzed for humidity measurement is not completely linear. However, there are ranges where humidity sensor response can be considered linear. During the hysteresis test, it is remarkable to note that there are slight differences in the sensitivity of the device to measure humidity when this increases or decreases gradually. During the experimental results it can be seen that the sensitivity is slightly higher when the humidity is down for all samples tested. The measurement sensitivity experimentally for each of the samples analyzed is summarized in Table 1. During the analysis of the results can be determined that the best sensitivity was obtained for the sample No. 1 with a sensitivity 0.071 nm/% RH, whose sample had a wall thickness of 42.1 µm. The analysis of samples, show an increase in sensitivity when the samples analyzed lower wall thickness.

As a result of the experiments, we found that the microcavity as manufactured cylinder from PMMA has certain sensitivity to humidity changes in the medium, unlike those made from the silica as it was found in some experiments above. The sensitivity depends on the wall thickness of the microcapillary, which improvement microcapillaries with thin wall. In our experiment the sensitivity was achieved in the range 0.027-0.071 nm/% RH measure through different microcapillaries with different wall thickness.

5. References

[1] Gouveia M A, Duber-Avila P, Marques T H R, Cesar Torres M. C and Cordeiro C M B 2015 Morphology dependent polymeric capillary optical resonator hydrostatic pressure sensor Opt. Express 23 pp 10643-10652
[2] Avila D A, Gouveia M A, Marques T H R, Torres C O and Cordeiro C M B 2014 Polymeric Capillary Optical Resonator in Latin America Sensors J. Opt. Soc. Am. LTu4A.38
[3] Zamora V, Díez A, Andrés M V, and Gimeno B 2007 Refractometric sensor based on whispering-gallery modes of thin capillaries Opt. Express 15, 12011-12016
[4] Matthew S. Luchansky and Ryan C. 2012 Bailey High-Q Optical Sensors for Chemical and Biological Analysis Anal. Chem. 84 (2) pp 793–821
[5] Tao Ling and L. Jay Guo A. 2007 Unique resonance mode observed in a prism-coupled microtube resonator sensor with superior index sensitivity Opt. Express 15 17424-17432
[6] Yang Y, Saurabh S, Ward J, and Chormaic S N, 2015 Coupled-mode-induced transparency in aerostatically tuned microbubble whispering-gallery resonators Opt. Lett. 40, 1834-1837