2012

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IEEE SENSORS JOURNAL, PISCATAWAY, v. 12, n. 1, p. 195-200, JAN, 2012
http://www.producao.usp.br/handle/BDPI/35482

Temperature Sensing Using Colloidal-Core Photonic Crystal Fiber

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***Abstract*—We report on a temperature sensor based on the monitoring of the luminescence spectrum of CdSe/ZnS nanocrystals, dispersed in mineral oil and inserted into the core of a photonic crystal fiber. The high overlap between the pump light and the nanocrystals as well as the luminescence guiding provided by the fiber geometry resulted in relatively high luminescence powers and improved optical signal-to-noise ratio (OSNR). Also, both core end interfaces were sealed so as to generate a more stable and robust waveguide structure. Temperature sensitivity experiments indicated a 70 $\mathrm{pm}/^{\circ}\mathrm{C}$ spectral shift over the 5 °C to 90 °C range.**

***Index Terms*—Colloidal quantum dots (QDs), luminescence, optical fiber sensors, photonic crystal fiber, semiconductor nanostructures.**

I. Introduction

COLLOIDAL nanocrystal quantum dots (QDs) are chemically synthesized semiconductor nanoparticles with efficient photoluminescence, whose spectral position can be controlled by tailoring the particle size and shape [1], [2]. Several applications using QDs have been reported, including photovoltaics [3], imaging [4], telecommunications [5], and sensing [6]–[13]. Within the latter application field, chemical and biological sensors have been extensively studied [6], [7]. In addition, both the intensity and the emission spectrum of quantum dots are temperature dependent [14], [15] making the use of these nanostructures also attractive for temperature sensing. As a consequence, temperature sensors [8]–[13] and, in particular, optical fiber temperature sensors [9]–[13] have been demonstrated.

Among the available fibers, photonic crystal fibers [16] are particularly attractive for the development of QD-based sensors because the array of micro-holes that runs along their axis allows the material to be directly and straightforwardly inserted into the waveguide structure, thus enabling efficient light-material interaction. Indeed, PCFs filled with gases [17], liquids [18]–[20], and colloids [21] have been reported for a wide range of applications. In the specific case of QD-based temperature fiber sensors [11], the inner walls of one solid-core PCF were coated with CdSe nanocrystals. Pump light was launched into the microstructured cladding with a multimode fiber so that excitation was maximized, achieving a spectral shift of the luminescent peak (temperature sensitivity) of 145 $\mathrm{pm}/^{\circ}\mathrm{C}$ [11]. Another multimode fiber at the output collected the luminescence.

A similar approach was reported using capillary fibers [10], the inner walls of which were coated with CdTe QDs. In this case, lateral UV excitation was used. Note that in all these approaches light-material overlap is relatively weak and interaction occurs via the evanescent field. Also, the photoluminescence is not guided by a core and, therefore, gradually leaks out of the waveguide [22].

Higher light-QD overlap and luminescence guidance were achieved in setups (so far not employed for temperature sensing) that used polymer [23] or silica [24]–[26] microstructured fibers, the hollow cores of which were filled with QD-doped polymers [23], [26] or sol gels [24], [25]. The resulting fiber structures guided light by total internal reflection within a solid QD-doped material. However, the insertion and subsequent solidification of a material into the fiber structure can lead to the formation of cracks and bubbles at the core-cladding interface, thereby inducing scattering and, consequently, affecting light guidance. Also, refractive index fluctuations can arise from a nonhomogeneous cure of the polymer.

These interface problems can be avoided if a liquid core is employed, in which the QDs are in suspension. Therefore, in the present work, we demonstrate and characterize a temperature sensor based on the luminescence generated in a QD colloid inserted into the hollow core of a PCF. Both the pump and the luminescence are index guided within the core, thus allowing for the luminescence to build up along the fiber. We also demonstrate that the colloid can be sealed within the hollow core by adding droplets of a UV-curable optical adhesive to the core input in both fiber tips. This procedure increases the applicability of the device by avoiding evaporation and core emptying.

II. Fiber Preparation and Experimental Setup

The QD colloid consisted of a suspension in mineral oil (an apolar liquid) of commercially available CdSe/ZnS core/shell nanocrystals (5.8 nm in diameter), from Evident Technologies, which presented a luminescence peak at $\sim 612\,\mathrm{nm}$. The choice of mineral oil is dictated by some of its attractive properties, which include a refractive index of ~ 1.45, very low volatility at room temperature (allowing fiber samples to remain stable for at least three weeks) and transparency in the visible portion of the optical spectrum. The concentration of CdSe/ZnS nanocrystals dispersed in mineral oil was 250 $\mu\mathrm{g}/\mathrm{ml}$, which was optimized to

Manuscript received March 28, 2011; revised April 19, 2011; accepted April 19, 2011. Date of publication April 25, 2011; date of current version December 01, 2011. The associate editor coordinating the review of this paper and approving it for publication was Prof. Ignacio Matias.

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Digital Object Identifier 10.1109/JSEN.2011.2146771

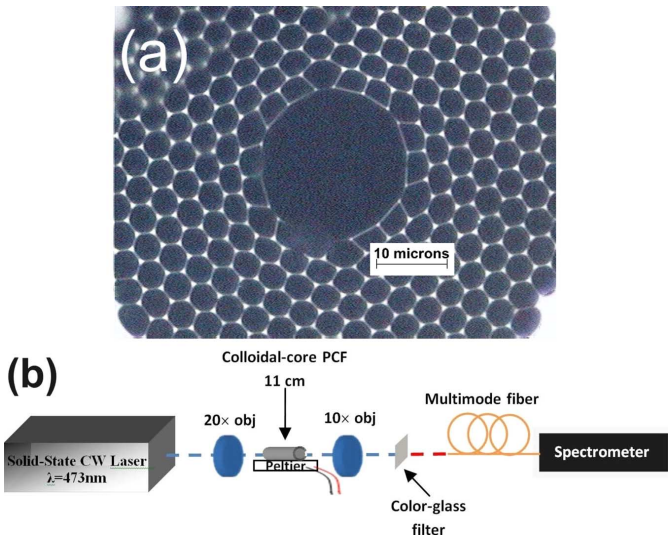

Fig. 1. (a) Optical micrograph image of the cross section of the hollow-core PCF (HC19-1550-01 from NKT Photonics). (b) Experimental setup of the temperature sensor based on a colloidal-core PCF.

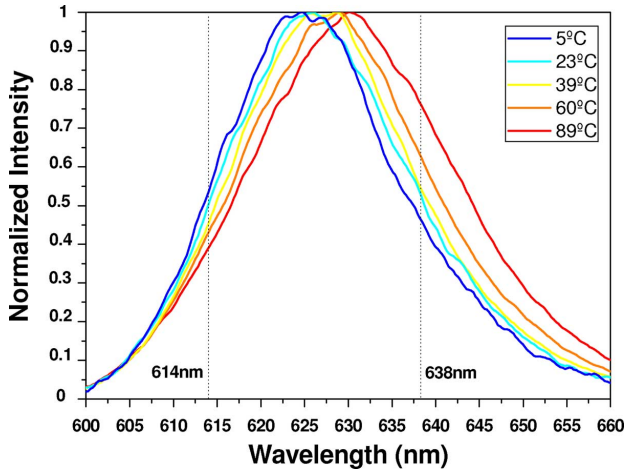

Fig. 2. Luminescence output spectra for several applied temperatures (color inset).

furnish maximum luminescence power within our experimental conditions.

The colloid was selectively inserted into the core of the PCF, shown in Fig. 1(a), using the method described in [27]. The method consists of collapsing the microstructured cladding of the PCF through the application of an electric arc produced by a fiber fusion splicer, so that only the core hole remains open. The colloid is then inserted by pressure using a syringe. After the filling process, the tip of the PCF is cleaved to remove the collapsed cladding region. The resulting waveguide consists of a cladding microstructure that is left filled with air and a core that is filled with the colloid. This arrangement allowed for total internal reflection guidance both for the pump and for the emission. The hollow-core PCF had a 20 μm diameter core and a cladding microstructure with a pitch of $\sim 4\ \mu$m and a hole diameter of $\sim 4\ \mu$m. From the oil and cladding refractive indices and the core diameter, ~ 2800 index-guided transverse modes are estimated at the QD emission wavelength. The overall coupling and propagation losses along a core-filled PCF, measured in a sample whose core was filled with oil only, was about 6 dB.

The experimental setup used to characterize the proposed temperature sensor is shown in Fig. 1(b) and consists of a solid-state cw laser at 473 nm with ~ 3.5 mW power, coupled into 11-cm-long samples of the colloidal-core PCF with a 20 × objective lens. The luminescence was then extracted at the fiber output and coupled, with a 10 × objective lens, into a multimode fiber connected to a spectrometer (Ocean Optics HR-4000-CG). These lenses can, in principle, be replaced with butt-coupled fibers to insert and extract light from the PCF. For output luminescence power measurements, an optical long-pass filter (RG-610) was used to remove the remaining pump.

To obtain a 5 °C to 90 °C temperature variation, a Peltier device (50 mm × 50 mm × 5 mm in spatial dimensions) was used. Two fiber Bragg gratings written on a conventional fiber, which was placed next to the PCF, were employed to monitor the temperature and to confirm its spatial homogeneity along the PCF length. Also, two microscope slides were placed on the top of the two fibers to ensure mechanical contact with the Peltier and to provide thermal insulation from the external environment.

III. Results and Discussion

Fig. 2 shows the normalized measured emission spectra for several temperatures. The emission peak at room temperature (23 °C) was located at 625 nm, presenting an associated FWHM of ~ 24 nm. At a fixed temperature, we observed a red-shift of the emission spectrum when compared with the spectrum obtained with a droplet of the colloid on a microscope slide, which peaked at ~ 612 nm. This shift can be understood considering that in the fiber the luminescence propagates along a longer path within the colloid, thus favoring re-absorption followed by re-emission by unpumped quantum dots. When the temperature was decreased a blue spectral shift was observed, as expected [14], with a corresponding red shift observed for temperature increases. The obtained luminescence exited the PCF via the 20-μm core only, which would allow an easy butt-coupling to a multimode fiber. The luminescence output power was $\sim 9\ \mu$W, with a power decrease of 10% observed under continuous illumination for 4 h. This decrease is similar to that reported in [10] (6,3%).

To put the measured luminescence power in proper context, a comparison was made with samples of a solid-core PCF (SC-5.0-1065 from NKT Photonics), the inner walls of which were covered with the same QDs. This approach is similar to that reported in [11]. The maximum emission power achieved in this case was 0.28 μW, which is 32 times lower than possible by using our colloidal-core PCF. As a consequence of this lower power, the resulting spectra presented a much worse signal-to-noise ratio, impacting the performance of the optical sensor.

Also, in principle, both the luminescence intensity and the luminescence spectral shift can be exploited for temperature sensing. However, wavelength measurements offer the advantage of being immune to loss variations and pump intensity fluctuations. Fig. 3 shows the luminescence peak shift with temperature. The linear fit (solid line) corresponds to a sensitivity of

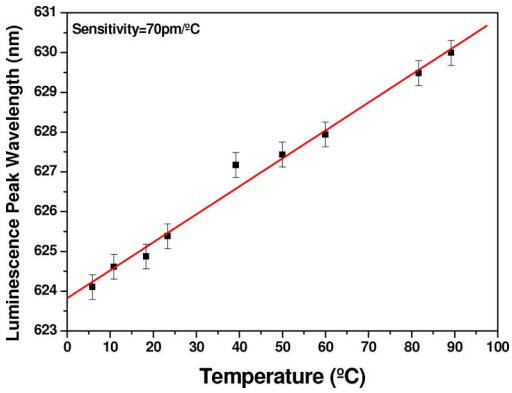

Fig. 3. Luminescence peak wavelength dependence on temperature.

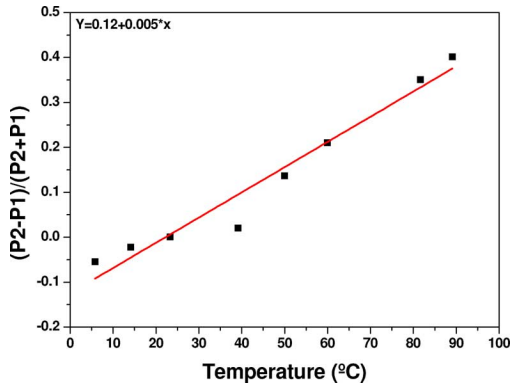

Fig. 4. Normalized power difference between the emission at 638 and 614 nm as a function of applied temperature.

70 $\mathrm{pm}/^{\circ}\mathrm{C}$, which is $\sim 5\times$ larger than that of fiber Bragg gratings ($\sim$ 13 $\mathrm{pm}/^{\circ}\mathrm{C}$) and similar to that measured in a control experiment ($\sim$ 68 $\mathrm{pm}/^{\circ}\mathrm{C}$) undertaken with a droplet of colloid outside the fiber. All sensing experiments were carried out at least four times, showing a maximum sensitivity variation of 4 $\mathrm{pm}/^{\circ}\mathrm{C}$. It is important to note that the investigated temperature range was limited by the available experimental apparatus. Semiconductor nanocrystals have been already tested for temperatures up to 627 °C [28] and are expected to exhibit a linear spectral shift with temperature for temperatures well above the material Debye temperature [29], [30] (-33 °C for CdSe [31]).

It should be stressed, that the value of sensitivity achieved in our work is not directly comparable to those reported in previous experiments available in the literature. This is because the QD composition varies in each case. The value of sensitivity is strongly dependent on the specific composition of the semiconductor QDs used since the main sensing mechanism is the QD bandgap variation with temperature, dependence which is different for each semiconductor QD material. Therefore, our goal here is not the demonstrate a record-breaking sensitivity but rather assure a stable setup with adequate output optical power for sensing applications.

In practical applications, it is also desirable to replace the spectrometer with a simpler and more cost-effective interrogation setup, based on photodetectors. Such a scheme can be made immune to power oscillations, while directly probing the fluorescence spectral shift, if the powers, P1 and P2, at two different wavelengths within the luminescent spectrum are measured and a normalized power difference is obtained through $(\mathrm{P2}-\mathrm{P1})/(\mathrm{P2}+\mathrm{P1})$ [13], [32]. This parameter is plotted as a function of temperature in Fig. 4, with $\mathrm{P1} = 614$ nm and $\mathrm{P2} = 638$ nm, which were chosen due to the high slope of the room temperature spectrum at these wavelengths. To emulate the use of 1-nm bandpass filters, while obtaining P1 and P2, adjacent averaging was applied to the spectra of Fig. 2. The results again show a linear increase with temperature, with a slope of $5 \times 10^{-3} {}^{\circ}\mathrm{C}^{-1}$.

IV. Sealed Colloidal-Core Photonic Crystal Fiber

To increase the usability of the device, a technique was developed to selectively seal a liquid-filled hole of a photonic crystal fiber [33]. This technique consists of using a micropipette with a tip with external and inner diameters of $\sim$ 4 and $\sim$ 1 μm,

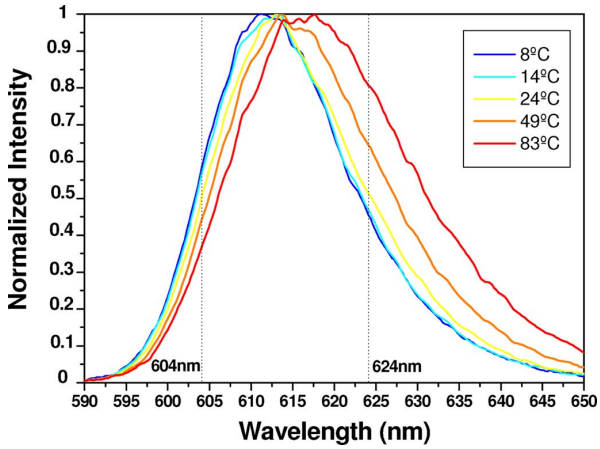

Fig. 5. Luminescence output spectra for several applied temperatures in a sealed colloidal-core PCF (color inset).

respectively, to deploy a microdroplet of a UV curable optical adhesive (NOA-61, refractive index $\sim$ 1.56) in both core inputs. After cured, the adhesive creates polymer plugs with an estimated length of tens of microns. During the insertion process an optical microscope was used to guide the micropipete, which was attached to a triaxial translation stage. To reduce the amount of air trapped between the colloid and the adhesive, the pipette tip was pressed against the inner wall of the core, thus allowing the remaining air to exit. Adhesive deploying was stopped when its level slightly passed the fiber tip surface, yielding a convex surface which was observed to yield lower insertion losses (estimated to be $\sim$ 2 dB for each plug). The sealing procedure prevents evaporation with minimal impact to the fiber ends. In particular, it is noted that, as the adhesive is not deployed in the cladding microstructure, index guidance is maintained all the way to the fiber tip.

After this sealing procedure, the sensing experiments were repeated and Fig. 5 shows the normalized measured emission spectra for several temperatures with a sealed colloidal-core PCF sample. The luminescence output power obtained was up to 2 μW, which is lower than in the unsealed sample because the insertion loss of the polymer plugs affects both the pump power launched into the PCF and the luminescence extracted from it. Nevertheless, the obtained emission power still compares favorably with the measured results from the solid-core PCF.

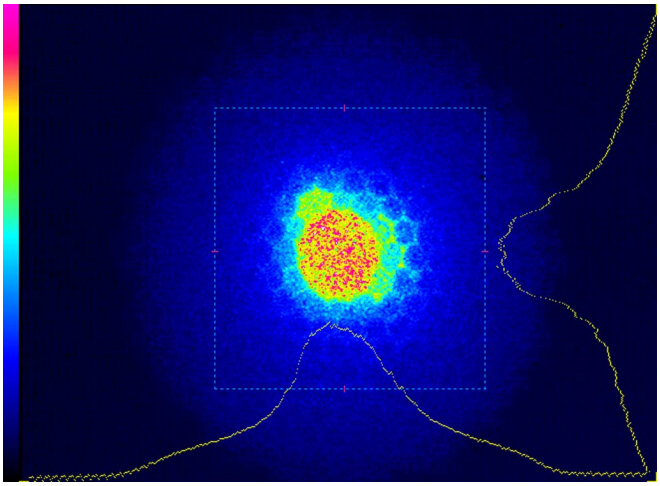

Fig. 6. Intensity distribution of the luminescence exiting a sealed colloidal-core PCF.

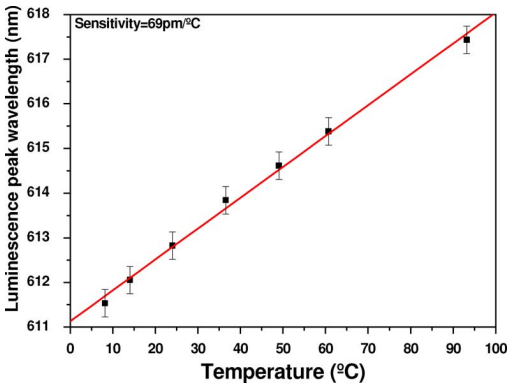

Fig. 7. Luminescence peak wavelength as a function of temperature.

Note that the luminescence at room temperature peaks at 613 nm, which is closer to the value obtained with the colloid droplet. The measured spectral width is also narrower (20 nm) than that of Fig. 2. These features may be explained by the lensing effect caused by the polymer plug, which was observed (via imaging of the output of a sealed liquid-core PCF) to induce the excitation of higher order transverse modes. When the unsealed fiber is used, coupling a Gaussian beam into the fiber preferentially excites the fundamental core mode, leaving unpumped a larger portion of the QDs close to the core-cladding border. This, in turn, increases the absorption and re-emission rate, thereby producing a red shift and spectral broadening. In contrast, in the sealed fiber, the lensing effect effectively increases the number of luminescence QDs across the core and, thus, reduces absorption and re-emission.

Fig. 6 shows the intensity distribution of the luminescence exiting the PCF, which was obtained by imaging the fiber output on a beam profiler. It can be seen that a large fraction of the emission leaves the fiber via the core. Also, the emission homogeneously fills the core area.

Fig. 7 shows the luminescence peak spectral shift with temperature. A linear fit was obtained with a sensitivity of 69 $\mathrm{pm}/^{\circ}\mathrm{C}$, which is equal to the result previously observed with the unsealed colloidal-core PCF, within the achievable reproducibility.

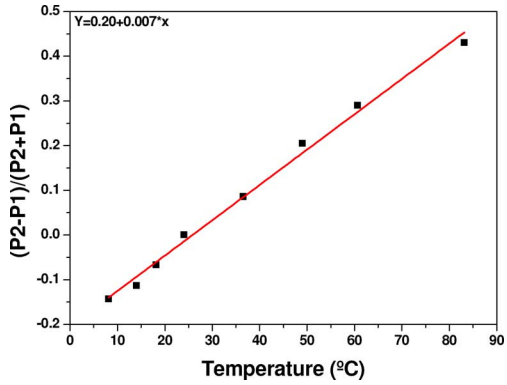

Fig. 8. Normalized power difference between the emission at 624 and 604 nm as a function of applied temperature.

Fig. 8 shows the normalized power difference between the intensities measured at 624 and 604 nm for the sealed colloidal-core PCF, which exhibits a slope of $7 \times 10^{-3\circ}\mathrm{C}^{-1}$. The higher slope obtained with the sealed fiber is a direct consequence of the narrower luminescence spectral width.

V. Conclusion

An optical fiber temperature sensor based on colloidal quantum dot luminescence was successfully demonstrated and tested, indicating the suitability of colloidal-core PCFs for optical sensing applications. The value of sensitivity is strongly dependent on the specific composition of the QDs used because the main sensing mechanism is the QD bandgap variation with temperature, dependence which is different for each semiconductor material.

In our particular configuration, a sensor sensitivity of 70 $\mathrm{pm}/^{\circ}\mathrm{C}$ was measured in the 5 °C to 90 °C range, which is ~ 5 times higher than that of fiber Bragg gratings. Most important than the sensitivity itself, which can probably be increased by a change of QD material, was the recording of a luminescence power of 9 μW, measured at the core output, allowing for improved signal-to-noise ratios.

A method to seal the colloidal core was also demonstrated. The use of our sealing process allows increased applicability of the proposed approach by avoiding colloid evaporation and core emptying. The polymer seal was also found to improve the homogeneity of the quantum dot excitation across the core, which increases the sensor sensitivity by narrowing the emission spectrum.

Acknowledgment

The authors would like to thank the Laboratório de Sensores Fibra Óptica (LSFO), PUC-Rio, Brazil, for providing the fiber Bragg gratings.

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