Temperature sensor based on colloidal Quantum Dots-PMMA nanocomposite waveguides

Antonio Bueno, Isaac Suárez, Rafael Abargues, Salvador Sales and Juan P. Martínez Pastor

***Abstract*—In this work integrated temperature sensors based on active nanocomposite planar waveguides are presented. The nanocomposites consist of cadmium selenide (CdSe) and cadmium telluride (CdTe) quantum dots (QDs) embedded in a polymethylmethacrylate (PMMA) matrix. When the samples are heated in a temperature range from 25 ºC to 50 ºC the waveguided photoluminescence of QDs suffers from a strong intensity decrease, which is approximately quadratic dependent with temperature. Moreover, the wavelength peak of the waveguided emission spectrum of CdTe-PMMA shows a blue shift of 0.25 nm/ºC, whereas remains constant in the case of CdSe-PMMA. A temperature resolution of 0.05 ºC is obtained. QDs-waveguides have great potential for the development of photonic sensors because of their integration, multiplexing and roll-to-roll fabrication capabilities.**

***Index Terms*—Temperature sensor, colloidal quantum dots, nanocomposites, PMMA.**

I. INTRODUCTION

RECENTLY, there is a huge interest in developing a great variety of integrated photonic devices because of their application in fields like telecommunication and sensing. For this purpose, colloidal quantum dots (QDs) are a very attractive material as an active medium to be integrated in photonic devices. These nanostructures join the three-dimensional confinement of the wave function, characteristic from QDs, with the feasibility of chemical methods, able to achieve nanocrystal radii from 1 nm to 10 nm with size dispersion as low as 6 % [1], [2]. Since the size of the QDs is very small, the separation of the electronic states is much greater than thermal energy and the emission of the QDs in colloidal solution turns to be temperature independent. Moreover, QDs allow the possibility of tuning their emission wavelength by changing the size, but also the base material, without modifying the surface chemistry. Then, the appropriate choice of two or more different QDs provides multi-wavelength (or multi-'color') operation, which is a very potential and attractive feature since it allows the possibility of multiplexing in wavelength several optical sensors [3].

During the last decade, many researchers have studied the use of fluorescent QDs in biological applications (as labels in bioanalysis and diagnostics, tags for proteins and DNA immunoassays or compatible labels for in-vivo imaging studies [4]-[7]) since they can modify their surface by conjugation with the appropriate biomolecules. Moreover, chemical sensors had been also developed since a desired selectivity can be obtained by chemically tailoring the outer surface of the QDs. For instance, a selective ion probe was developed for sensing Zn^{2+} and Cu^{2+} cations [8] and a sensor for explosive molecules such as TNT or nitrobenzene was presented [9].

In order to achieve the integration of the properties of QDs into optoelectronic devices, a common approach is to incorporate them into a host material. These multicomponent materials are known as nanocomposites. Walker et al. first characterized the temperature response of the photo-luminescence (PL) of colloidal QDs immobilized inside a polylaurylmethacrylate (PLMA) matrix [10] proving the suitability of QDs as temperature references for PL-based sensing applications. Other authors have also reported the use of different host polymers such as polystyrene (PS) [11] or polymethylmethacrylate (PMMA) [12] to embed QDs, and even coupling the light with the aid of optical fibers [13]. All these works use the PL of QDs as the sensing parameter, but none of them integrate the PL response into an optical waveguide, which is the key step to develop a temperature sensor into a photonic device.

In this paper, we present a temperature sensor based on the temperature-dependent response of the waveguided PL of CdSe and CdTe QDs embedded into PMMA thin films. We describe the fabrication process of planar waveguides based on QDs and present the experimental setup carried out in order to perform temperature tests. Finally we show the temperature characterization of waveguided PL in both CdSe-PMMA and CdTe-PMMA waveguides from 25 to 50 ºC. Peak intensity of the waveguided PL shows a decreasing quadratic behavior in both waveguides. Between both nanocomposites, CdTe-PMMA

Manuscript received XXX, 2012. This work was supported through the Spanish MICINN and Generalitat Valenciana Grants Nos. TEC-2011-06756-C03-03 and PROMETEO/2009/074 and through the Plan Nacional I + D projects TEC2011-29120-C05-01 and TEC2011-29120-C05-05, and also through "Infraestructura FEDER UPVOV08-3E-008".

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waveguides showed the largest response to temperature.

II. FABRICATION

The active waveguide materials chosen for this study were CdSe and CdTe QDs incorporated into a PMMA matrix. CdSe was synthesized following the procedure developed by Peng's group [14]. CdTe was synthesized in a similar way, from a solution of tellurium-trioctylphosphine. Once QDs have been prepared, CdSe-PMMA and CdTe-PMMA nanocomposite solutions were obtained by mixing PMMA and QDs, both diluted in toluene. The concentration of QDs in the polymer was chosen according to a previous study [15], where filling factors around $5 \cdot 10^{-4}$ had been found to be optimum for waveguiding. In order to implement the waveguides, nanocomposite solutions were spin-coated on SiO_2/Si substrates (as it is illustrated in Fig. 1) and baked at 80 ºC and 150 ºC for 2 minutes. The SiO_2 layer is prepared by a sol-gel method [16] resulting in a film of 600 nm thick, acting as a good low index cladding layer for the optical modes of the core at the wavelengths of interest (400-600 nm). Finally, the edges of the samples were cleaved for end fire coupling purposes, being the edge-to-edge distance of around 5 mm in width and 10 mm in length.

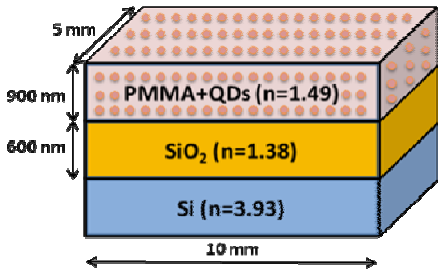

Fig. 1. Structure of the planar waveguides fabricated (not in scale).

The absorbance spectra of solutions containing CdSe and CdTe QDs are depicted in Fig. 2. The absorbance spectrum of CdSe QDs (blue line) has an absorbance peak corresponding to the ground state exciton transition at 580 nm, whereas an excited state transition is observed at around 473 nm. The absorbance is negligible beyond 650 nm and increases continuously for shorter wavelengths. Similarly, the ground state exciton peak is observed at 537 nm for CdTe QDs (green line) and two excited state transitions are resolved at 490 nm and 430 nm. The absorbance is negligible for wavelengths longer than 570 nm and increases continuously for shorter wavelengths. According to the absorption data published by other authors, the exciton peaks at 580 nm for CdSe and at 537 nm for CdTe QDs should correspond to nanocrystal radius of around 2.5 nm and 1.5 nm respectively [17], [18].

Fig. 3 shows the PL spectra at 25 ºC of CdSe (blue lines) and CdTe (green lines) QDs measured in the colloidal solution of toluene (solid lines) and in the nanocomposite of PMMA (dotted lines). They were measured by optical pumping with a GaN laser diode (404 nm) by back-scattering. The PL line of CdSe QDs in the colloidal solution is centered at 620 nm and has a full width at half maximum (FWHM) of 40 nm, whereas the PL line of the CdTe QDs in the colloidal solution is centered at 560 nm and has a FWHM of 30 nm. The red shift of the PL peak with respect to its corresponding absorption resonance is known as the Stokes shift and it is typical of semiconductor nanostructures [19]. When the QDs are embedded in the polymer, the absorbance curves are similar to the ones depicted in Fig. 2, but a smaller Stokes shift is measured due to the fact that a certain proportion of nanoparticles can agglomerate. The PL lines of CdSe and CdTe QDs in the nanocomposite are centered at 600 nm and 545 nm respectively, being shifted to shorter wavelengths. These shifts are a visible sign of the surrounding influence in the emission of QDs.

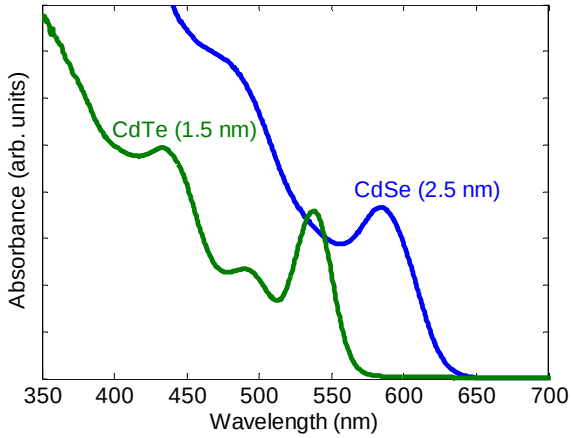

Fig. 2. Absorbance spectra for CdSe and CdTe QDs in the colloidal solution.

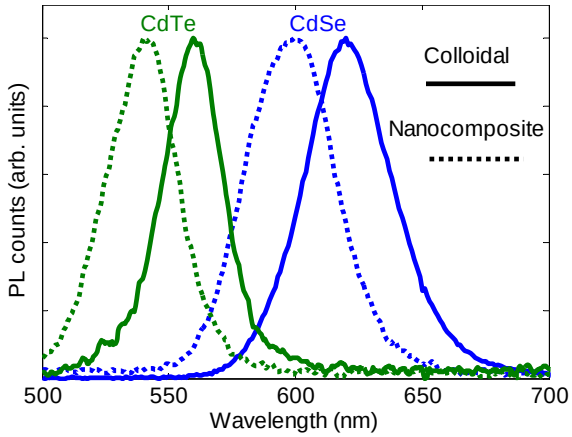

Fig. 3. Photoluminescence spectra of CdSe and CdTe QDs in the colloidal solution and incorporated in the PMMA nanocomposite.

III. EXPERIMENTAL SETUP

The experimental setup is depicted in Fig. 4. The PL wave-guiding has been characterized by coupling light from a GaN laser diode centered at 404 nm to both CdSe-PMMA and CdTe-PMMA planar waveguides. The laser beam was focused on the surface of the planar waveguides from the vertical direction in normal incidence with the aid of a semi-cylindrical lens. As a result, the beam was focused along the length of the waveguide in a thin line. Under this condition, QDs are equally pumped in the whole length of the waveguide, being its PL coupled to a waveguide mode as it has been discussed in [15]. The waveguides were placed on the top of a ceramic resistor in order to increase the temperature by Joule effect. The ceramic

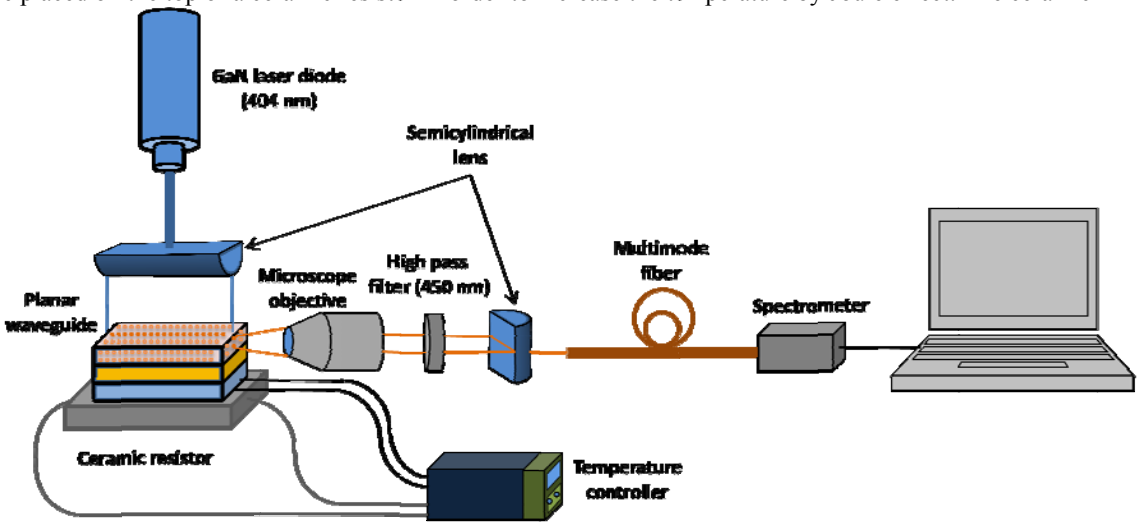

Fig. 4. Experimental setup carried out in order to perform temperature experiments with CdSe-PMMA and CdTe-PMMA waveguides.

resistor used had an electrical resistance of 22 KΩ and dimensions of 5 mm x 15 mm . The temperature on the top of the resistor was obtained by a PT100 electrical sensor with a resolution of 0.1 ºC and controlled by a CAL9300 (CAL Controls) temperature controller.

A microscope objective collects the light at the output of the waveguide and focuses it to the detection system. It was mounted in a three-dimensional positioning system with micrometric resolution in order to improve the light coupling from the waveguides. Collimated PL light is finally focused with a semi-cylindrical lens to a multimode optical fiber attached to the entrance slit of a spectrometer (StellarNet EPP2000). This spectrometer is capable to acquire a spectrum in the range from 256 nm to 1100 nm with a resolution of 0.5 nm. The PL spectra were recorded by the spectrometer was recorded by a specific control software running in a personal computer.

IV. RESULTS AND DISCUSSION

In a first step, a reference PL spectrum was recorded at room temperature (25 ºC) in the QDs-PMMA waveguides. The waveguided PL spectrum exhibit some differences compared to the PL spectrum of the nanocomposite due to the guiding effect through the sample. The propagating light in the waveguide can experience a reabsorption phenomenon, leading to a blue shift and a broadening of the waveguided PL spectrum [15]. In the CdSe-PMMA waveguide a 30 nm blue shift in the PL peak and a FWHM of 52 nm is measured. In CdTe-PMMA the waveguided PL spectrum was centered at 540 nm and had a FWHM of 38 nm.

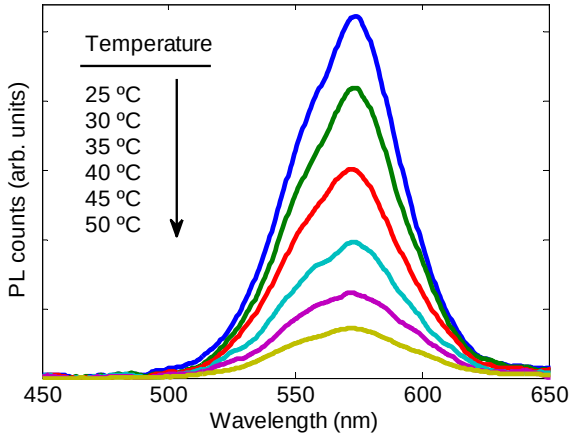

Fig. 5. Photoluminescence spectra recorded from the CdSe-PMMA waveguide at each setting temperature.

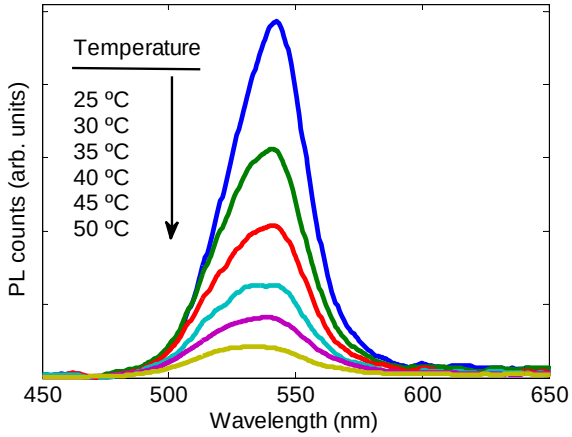

Fig. 6. Photoluminescence spectra recorded from the CdTe-PMMA waveguide at each setting temperature.

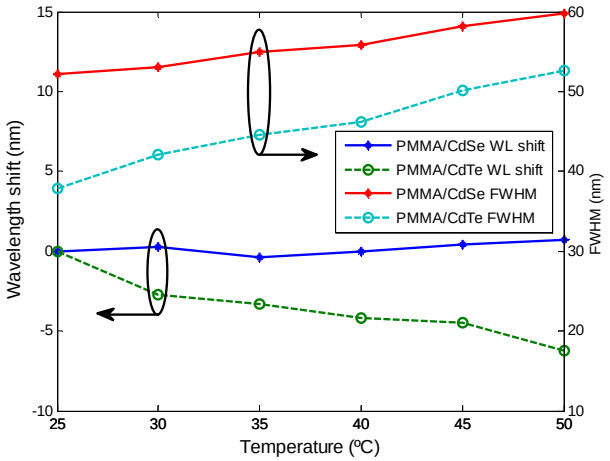

Fig. 7. Temperature dependence of wavelength shift and FWHM of PL spectrum.

Then, the waveguides were placed on the top of the ceramic resistor and the samples were heated in steps of 5 ºC up to a maximum temperature of 50 ºC. The PL spectra from the CdSe-PMMA waveguide at each setting temperature are depicted in Fig. 5. As it can be seen, the PL intensity strongly decreases with increasing temperature of the sample. There is no appreciable shift in the PL peak wavelength and a clear broadening of 7.6 nm from 25 ºC to 50 ºC, as it can be observed in Fig. 7. The CdTe-PMMA waveguide was placed on top of the ceramic resistor in an identical procedure as the previous sample. A similar strong decrease of the PL intensity is observed when the sample is heated from 25 ºC to 50 ºC, as shown in Fig. 6. Nevertheless, the PL spectrum of the CdTe-PMMA waveguide experiences a broadening of 14.9 nm and a blue shift of 6.2 nm (Fig. 7).

The CdSe-PMMA waveguide shows a negligible shift (within a range of ± 0.5 nm) whereas in the CdTe-PMMA waveguide an overall blue shift of 6.2 nm is measured, which means a shift rate of -0.25 nm/ºC. Regarding to the FWHM of the PL spectrum, both materials exhibit an increase. In the case of the CdSe-PMMA waveguide the broadening increase is 7.6 nm and nearly double, 14.9 nm, in the case of the CdTe-PMMA waveguide, corresponding to broadening rates of 0.3 nm/ºC and 0.6 nm/ºC respectively. Since colloidal QDs (dropped cast layers) exhibit typically a smooth wavelength red shift with temperature, the different waveguided PL measured from 25 ºC to 50 ºC can be mainly attributed to the variations of the optical (refraction index) and morphological (film roughness) properties of PMMA matrix depending on the working temperature [20]. The PMMA stability is assured since there is not weight loss at temperatures below 165 ºC and its elastic regime is up to 195 ºC [21]. Moreover, any degradation of the material through the temperature cycles repeated during several days has not been observed. Concerning the cross-sensitivity with humidity, it is well known that both oleate-capped QDs and PMMA are hydrophobic. Therefore, water absorption by the nanocomposite is estimated to be very low. Thus, it is expected that humidity does not influence the measurements under the conditions of our experiment.

Although the temperature range studied is far away from the glass transition temperature of PMMA (around 105 ºC), heating can provoke the migration of the nanocrystals or even the modification of the QD's surface, leading to a decrease of the PL intensity. In fact, it has already been demonstrated the decrease of PL in CdSe-PMMA [22], [23] and in CdSe/ZnS polystyrene composites [11] upon heating. This is explained by the strain induced in the QDs due to the thermal expansion of the polymer chains. Indeed, in these works the decrease of PL is accompanied by a broader FWHM and a red shift of the emission peak. In this case, CdSe-PMMA does not have any shift, whereas CdTe-PMMA shows a blue shift. However, in this case the waveguided PL is influenced as well by the waveguide properties (thickness, refractive index, losses…).

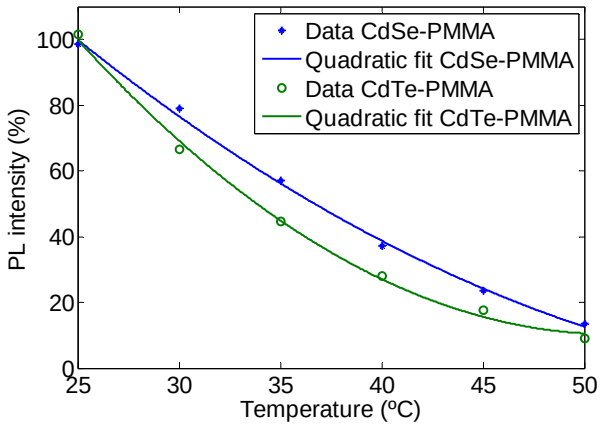

Fig. 8. Experimental data and quadratic fit in CdSe-PMMA and CdTe-PMMA planar waveguides.

The refractive index of polymers decreases with temperature at a rate of 10^{-4}/ºC [20], [22], thus when the sample is heated long wavelength modes are to be expected at cut-off, dealing to a blue shift in the spectra. Also, since a waveguide can propagate more quantity of modes at shorter wavelengths, it is reasonable that this effect should be stronger in the CdTe-PMMA waveguide.

Fig. 8 shows the experimental data of the PL peak intensity versus temperature for both CdSe-PMMA and CdTe-PMMA waveguides. Quadratic fits of these data have been applied, obtaining polynomial expressions of $PL(\%) = 0.058 \cdot T\ (ºC)^2 - 7.87 \cdot T\ (ºC) + 260.33$ for the CdSe-PMMA waveguide and $PL(\%) = 0.13 \cdot T\ (ºC)^2 - 13.18 \cdot T\ (ºC) + 349.53$ for the CdTe-PMMA waveguide. The correlation coefficients obtained are 0.9978 for the CdSe-PMMA waveguide and 0.9973 for the CdTe-PMMA waveguide, showing an excellent fit of the experimental data. These polynomial coefficients state a higher temperature sensitivity of the CdTe-PMMA waveguide. The sensitivity in both waveguides depends on the working temperature due to the decreasing quadratic dependence of the PL intensity. In the lower temperatures range (25-40º) the highest performance is achieved. In this case , the curve can be approximated by a linear function with slopes of -4.9 % per ºC and -6.7 % per ºC in the CdSe-PMMA and CdTe-PMMA waveguides respectively. On the other hand, the lowest performance is achieved in the highest temperature range (40-55 ºC), where the curve shows a quadratic decrease. The sensitivities obtained in this region are -2.1 % per ºC and -0.5 % per ºC in the CdSe-PMMA and CdTe-PMMA waveguides respectively. The best temperature resolution is achieved in the lower temperature range, where the sensitivity of the sensor is higher. Taking into account this parameter and the precision of the spectrometer, the best temperature resolution of this set-up is estimated as 0.05 ºC.

V. CONCLUSION

In this work, the feasibility of a temperature sensor based on nanocomposite waveguides is demonstrated. The recent advances in colloidal QDs fabrication make the photonic integration of these nanostructures in polymer hosts possible. As the surrounding media varied from the colloidal solution to a polymer matrix, the PL spectrum is temperature dependent due to guiding and reabsorption effects of light travelling through the waveguide.

Temperature sensors based on CdSe and CdTe QDs incorporated in PMMA were characterized. The waveguides were heated in the range from 25 ºC to 50 ºC and the temperature characterization of the waveguided PL shows a decrease in its intensity with quadratic temperature dependence. It is interesting to note that CdTe-PMMA waveguide presents more sensitivity and also its wavelength peak suffers from a temperature dependence of 0.25nm/ºC, whereas remains constant for the CdSe-PMMA. The possibility of wavelength multiplexing these sorts of integrated sensors allows a great number of temperature measurement applications.

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launched the “Unit of Opotelectronic Materials and Devices” at the Institute of Material Science (University of Valencia), now with 10 researchers and 8 PhD students. His research interests include Semiconductor Physics, particularly optical properties of quantum heterostructures and nanostructures based on III-V semiconductors, but more recently he has opened and leaded new research lines in nanotechnology: development of nanomaterials (colloidal metal and semiconductor nanocrystals, nanocomposites based on these nanocrystals embedded in polymers and oxides) and applications to photonics/plasmonics for telecom, sensing and photovoltaics