

Avoiding induced heating in optical trap

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ABSTRACT

Luminescence of a single upconverting particle ($\text{NaYF}_4:\text{Er}^{3+}, \text{Yb}^{3+}$) can be used to determine the optical trap temperature due to the partial absorption of the trapping beam either by the medium (water) or the optically trapped particle itself. This fact is an important drawback can be reduced by shifting the trapping wavelength out of the water absorption band, or by using time-modulated laser trapping beams. Both approaches have been studied and the results have shown that the thermal loading due to the trapping radiation can be minimized.

Keywords: temperature sensing, $\text{NaYF}_4:\text{Er}^{3+}, \text{Yb}^{3+}$, Optical Trapping, upconversion

1. INTRODUCTION

In recent months, the interest in the development of new materials and techniques for measuring temperature at the single cell level has experienced a spectacular growth. One of these approach is based on upconverting particles (UCPs) with an excellent ratiometric thermal sensitivity, allowing for subdegree resolution thermal measurement.[1-4] Multipoint thermal sensing was made possible by scanning this thermal sensing unit by means of a single beam optical tweezer. The wavelength of trapping radiation is normally chosen to match the absorption peak of the $\text{NaYF}_4:\text{Er}^{3+}, \text{Yb}^{3+}$ particles (typically 980 nm), allowing for simultaneous three dimensional manipulation and thermal sensing, i.e. allowing for single beam thermal scanning.

However, there is an important drawback concerning the possible appearance of additional thermal gradients/contributions due to the partial absorption of the trapping beam either by the medium (water) or by the optically trapped particle itself. This is not a negligible effect and, indeed, it has been the subject of study of a number of papers appearing in the field of optical trapping and manipulation.[5-9] In those cases, alternatives should be found to minimize this effect.

Shifting the trapping wavelength out of the water absorption band, or using time-modulated laser trapping beams are different ways to reduce the laser-induced trap heating. Both approaches have been studied showing that the thermal loading due to the trapping radiation can be minimized.[2] This work introduces to the scientific community a novel and simple approach for high resolution thermal sensing avoiding the heating effect.

2. EXPERIMENTAL

2.1 Trapped microparticles

The NaYF₄:Er,Yb UCPs were synthesized by a hydrothermal reaction. Typically, aqueous solutions of sodium citrate (1.33 mL, 0.3 M) and Y(NO₃)₃ (2 mL, 0.2 M, Yb:Er = 5:0.5 mol%) were mixed under vigorous stirring to form a milky suspension, into which an aqueous solution of NaF (6.4 mL, 0.5 M) was added to form a transparent colloidal. The resulted colloidal was then transferred to a 14 mL Teflon vessel and heated to 220 °C for 12 h. **Figure 1(a)** includes the Scanning Electron Microscopy (SEM) images of the different UCPs used, as well as their size histograms. NaYF₄:Er,Yb UCPs present a quasi-hexagonal shape with dimensions 1.6 x 3 μm (thickness, diameter). The sample showed a good colloidal stability in water.

2.2 Optical trapping setup

To carry out the optical trapping (OT) experiments, the UCPs were dispersed in distilled water (0.01% in weight) and sonicated for 30 min in order to break possible agglomerates between UCPs. The UCPs dispersion was placed into a 100 μm height microchannel (Ibidi Inc., μ-Slide I, catalog number: 80166.) and excited with 980 nm radiation using a single-mode fiber-coupled laser diode (LUMICS). The laser source was controlled by a DSRC0102 (OsTech) laser controller, which allows for working on continuous and modulated (pulsed wave) mode. The laser radiation coming out from the single-mode fiber (SMF) was collimated by a fiber-port (Thorlabs PAF-X-7-B). The collimated beam was expanded by a 2X beam expander (Thorlabs BE02M) resulting in a laser beam of 2 mm in diameter. This matches the back aperture of the microscope objective used to focus the trapping beam into the microchannel. Optical images were recorded using a CCD camera. A schematic representation of the setup is shown in **Figure 1(b)**.

2.3 Temperature measurements

Luminescence spectra of an optically trapped UCP was spectrally analyzed by a high sensitivity Si CCD camera (Synapse, Horiba) attached to a monochromator (iH320, Horiba), all it optically coupled to the single-beam optical tweezers setup. Calibration curves were acquired by varying the temperature of the aqueous solution contained the optically trapped UCP while performing simultaneous spectrum acquisition and analysis. Thermal resolution had been estimated to be ±1°C from the signal/noise ratio of the upconverting spectra. In order to avoid the laser-induced local heating, temperature calibration has been carried out with the minimum laser power (1 mW) needed to trap and detect the luminescence ($0.3 \times 10^5 \text{ Wcm}^{-2}$ power density).

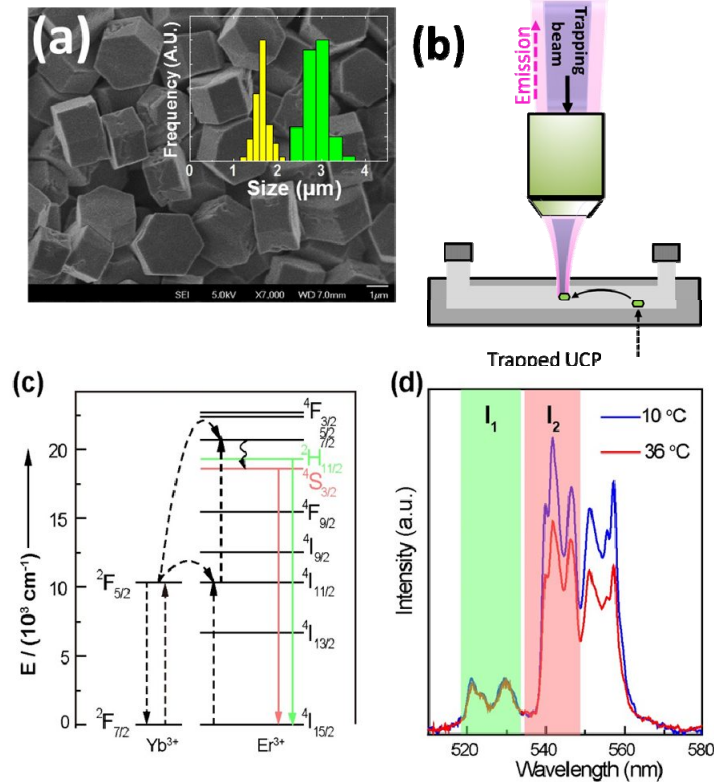


Figure 1. (a) Scanning electron microscopic image of UCPs, showing a clear hexagonal morphology. The inset shows the size distribution of the UCPs used. (b) Schematic representation of the experimental setup used in this work for thermal sensing from an optically trapped UCP. (c) Simplified energy level diagram showing the temperature sensitive transition levels (in highlighted colors), $^2H_{11/2}$ and $^4S_{3/2}$, and corresponding energy transition processes. (d) Upconverting emission spectra obtained from a single optically trapped UCP at two different temperatures. The intensity ratio I_1/I_2 is strongly dependent on the temperature. Reprinted with permission from *Adv. Mater.* 2016, 28, 2421–2426. Copyright 2016 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim.

3. RESULTS

3.1 Optical trapping and temperature measurements

Figure 1(b) schematically represents the experimental setup used for OT of a single UCP. A solution containing the UCPs was introduced in a microchannel, into which a 980 nm laser beam was focused in order to achieve OT. A single microscope objective was used. When an UCP is optically trapped, a strong green emission is generated from the trap. This green emission is generated by Er^{3+} ions as a consequence of the $Yb^{3+} \rightarrow Er^{3+}$ energy transfer that is activated after the absorption of 980 nm laser radiation by Yb^{3+} ions (see the schematic energy level diagram of **Figure 1(c)**).^[10-13] Although we were able to detect several emission bands generated by Er^{3+} ions, in the following we will focus our attention on those bands lying within the 500-570 nm spectral range, as they are known to be especially sensitive to temperature changes.^[1-3, 14-17] In particular, as it can be observed in **Figure 1(d)**, Er^{3+} ions show two intense bands centered at 530 and 550 nm which correspond to the $^2H_{11/2} \rightarrow ^4I_{15/2}$ and $^4S_{3/2} \rightarrow ^4I_{15/2}$ transitions, respectively (see level diagram in **Figure 1(c)**). As these emission bands are generated from two thermally coupled excited states ($^2H_{11/2}$ and $^4S_{3/2}$) the ratio between their emitted intensities is strongly temperature dependent, thus allowing for accurate thermal

sensing.[10, 13] The thermal sensitivity of an optically trapped UCP has been explored by measuring their two-photon excited luminescence spectrum at different temperatures. **Figure 1(d)** shows the luminescence spectra generated from an optically trapped UCP as obtained when the liquid solution temperature was set to 10 and 36 °C. As can be observed, a rise in temperature causes an increment in the intensity ratio, R , between the 540 and 525 nm bands ($R=I_1/I_2$ ratio, according to **Figure 1(d)**), due to the temperature-induced increase (decrease) of the population of the ${}^2H_{11/2}$ (${}^4S_{3/2}$) excited state. Indeed, according to the Boltzmann thermal statistics, the emission intensity ratio R should be proportional to $\exp(-\Delta E/k_B T)$, where ΔE is the energy separation between the ${}^2H_{11/2}$ and ${}^4S_{3/2}$ emitting states, k_B is the Boltzmann constant and T is the absolute temperature.[10]

Figure 2 shows the dependence of the $\ln(R)$ on the inverse temperature, $1/T$, as obtained after systematic variation of medium (UCPs in water) temperature while keeping the UCP optically trapped. In accordance with the Boltzmann statistics, a linear behavior is obtained. Such a linear relation evidences that the local temperature can be accurately measured from the analysis of the single optically trapped UCP in the 10-50°C temperature range. Thermal sensitivity is defined as:

$$S = R_{RT}^{-1} \cdot \frac{dR}{dT} \quad (1)$$

where R_{RT} is the emission intensity ratio at room temperature. In our case, we have obtained a thermal sensitivity close to $1.6 \times 10^{-2} \text{ }^\circ\text{C}^{-1}$, which is in good agreement with previous reports.[1, 10] Once the temperature calibration from a trapped UCP had been achieved, we applied this to elucidate the open question regarding the thermal gradient created by the trapping laser.

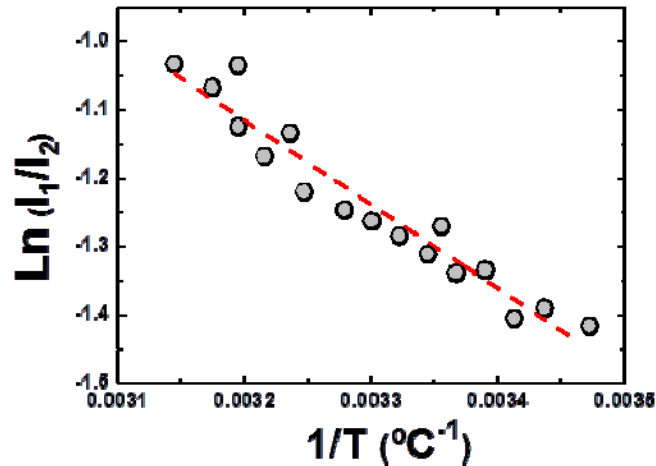


Figure 2. Dependence of the $\ln(R)$ on the inverse of temperature, $1/T$, as obtained from a systematic variation of the medium temperature while keeping the UCP optically trapped. Dots are experimental data and dashed line is the best linear fit. *Reprinted with permission from Adv. Mater. 2016, 28, 2421–2426. Copyright 2016 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim.*

3.2 Effect of the trapping laser

An evaluation of the possible presence of laser-induced local heating induced by the 980 nm laser radiation has been performed. For this purpose, an UCP was trapped and the temperature change obtained from the previous calibrations

through luminescence measurement. **Figure 3(a)** shows the trap temperature increment (trap heating) as a function of the 980 nm trapping laser power. The temperature of the optically trapped UCP was found to increase linearly with the laser power at a rate of 0.16 °C/mW. Comparing to previous works and numerical models, we can conclude that such heating is mainly caused by the residual water (medium) absorption, since other contributions, such as the UCP intrinsic absorption, can be considered negligible. However, data included in **Figure 3(a)** reveal that laser-induced trap heating cannot be neglected, and strategies for keeping it at a minimum must be used, such as reducing the laser power (which is not desirable as it would imply a reduction in the trapping force), performing the thermal scanning in non-absorbent media such as heavy water (not feasible in biological applications), shifting the trapping wavelength out of the water absorption band, or using time-modulated laser trapping beams. **Figure 3(a)** shows the heating curve obtained from a single UCP optically trapped by an 808 nm laser beam. The absorption of the aqueous medium at this wavelength is low, (see **Figure 3(b)**), but the upconversion luminescence can also be excited at 808 nm due to the characteristic Erbium absorption band located around 800 nm. As can be observed, when the trapping wavelength is shifted from 980 nm to 808 nm, the laser-induced thermal loading experiments a drastic reduction and becomes almost negligible.

Additionally, laser-induced thermal loading can be reduced by using time-modulated laser beams. It has recently been demonstrated that, in OT setups similar to those used in this work, the characteristic time of laser-induced heating events is in the order of $t_h \approx 100$ ms. Thus, if OT is achieved by laser pulses shorter than 100 ms, then thermal loading is expected to be significantly reduced as the heating source (laser pulse) is applied during a period shorter than that required for thermal stabilization. This possibility has been experimentally corroborated by modulating the 980 nm laser beam. **Figure 3(c)** shows the laser-induced trap heating produced by a time-modulated 980 nm laser beam (130 mW) as a function of the pulse width (time separation between pulses was kept constant and equal to 40 ms). As can be observed, a drastic reduction in trap temperature can be achieved by using laser pulses shorter than t_h . Indeed, for pulse widths similar to 100 ms (i.e. close to t_h) the laser-induced heating is found to be the same as that obtained under continuous wave (CW) trapping. Laser-induced thermal loading can also be significantly reduced by varying the time separation between pulses. When it is shorter than t_h , a complete thermal relaxation between consecutive pulses does not take place and thermal accumulation between consecutive pulses is produced. As a consequence, laser-induced thermal loading is expected to decrease as the separation between pulses is increased. This alternative route for thermal loading minimization has also been experimentally corroborated. **Figure 3(d)** shows the laser-induced trap heating produced by 980 nm, 20 ms long pulses (130 mW in power) as a function of the time separation between consecutive pulses. As expected, trap heating is strongly reduced as the time interval between pulses is increased, as a consequence of the minimization of thermal accumulation. Thus, **Figures 3(c)-(d)** evidence that an appropriate time modulation of laser trapping beams constitute a promising approach for the minimization of laser-induced thermal effects. Obviously, the characteristic trapping and escaping times should always be taken into account when modulating the trapping beam, so as to prevent the particle from escaping from the trap.

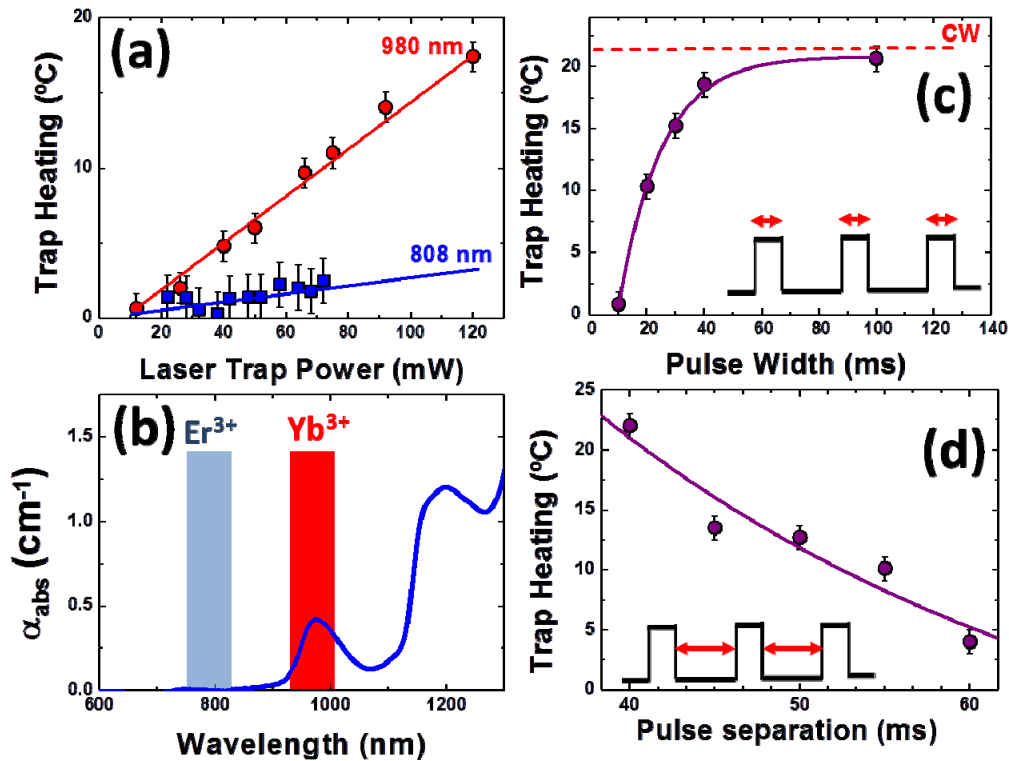


Figure 3. (a) Laser-induced thermal loading of an optically trapped UCP using trapping wavelengths of 980 and 808 nm. Dots are experimental data and solid lines are the best linear fits obtained in each case. (b) Room temperature absorption coefficient of water. Blue and red colored regions indicate the spectral ranges used for optical excitation of Ytterbium and Erbium ions. (c) Laser-induced thermal loading of an UCP when it is optically trapped by a pulsed (modulated) 980 nm laser beam (130 mW power) as obtained for different pulse widths. Dots are experimental data and solid line is a guide for eyes. Dashed horizontal line indicates the laser-induced heating obtained under continuous wave illumination. (d) Laser-induced thermal loading of an UCP when it is optically trapped by a pulsed (modulated) 980 nm laser beam (130 mW power) as obtained for different time between pulses. Dots are experimental data and solid line is a guide for eyes. *Reprinted with permission from Adv. Mater.* 2016, 28, 2421–2426. Copyright 2016 WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim.

4. CONCLUSIONS

In summary, results reveal that laser-induced thermal loading is not negligible in optically trapped fluorescent particles experiments. This has been demonstrated by optical trapping of an NaYF₄:Er,Yb particle by a 980 nm laser beam. This beam allows for simultaneous three-dimensional manipulation and fluorescence ratiometric thermal sensing by using simple experimental designs. However, the thermal loading due to the partial absorption of the trapping beam either by the medium (water) or by the optically trapped particle itself is not negligible. This fact is an important drawback that can be reduced by shifting the trapping wavelength out of the water absorption band, or using time-modulated laser trapping beams. Both approaches have proven to substantially reduce the laser-induced heating at the optical trap

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