Comprehensive Study on the Combined Effect of Laser-Induced Heating and Laser Power Dependence on Luminescence Ratiometric Thermometry

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ABSTRACT: Luminescence ratiometric thermometry, on the basis of nonthermally linked states of lanthanides, became a hot research issue recently because of its several attractive features. Here, the $^5F_4$/$^5S_2$/$^5F_5$ transitions of Ho$^{3+}$ embedded in calcium tungstate host are taken as an example to show the influence of laser pump power on this temperature detection technology. The luminescence intensity ratio between the $^5F_4$/$^5S_2$ and $^5F_5$ upconversion emission lines was found to respond monotonously to the temperature between 303 and 603 K and could be fitted well with the use of an empirical function. It suggested that this ratio might be suitable for temperature measurement. However, at 303 K, the temperature readout derived from this ratio decreased from 303 to 248 K on increasing the laser pump power from 35 to 205 mW (the irradiated spot’s area: ca. 2 mm$^2$). This uncommon phenomenon differs from the conventional laser-induced heating effect. With the help of the Boltzmann distribution based on the two Stark components of the $^5F_4$ state of Ho$^{3+}$, the laser-induced heating was calculated to be ca. 20 K when the excitation power was 205 mW. Thus, this suggested that there should be a mechanism responsible for the gradually decreasing temperature readout. It was then confirmed that this mechanism was the different dependences for the $^5F_4$/$^5S_2$/$^5F_5$ transitions on laser pump power, which was much stronger than the laser-induced heating effect. A calibration method to eliminate the influence of laser power dependence on luminescence ratiometric thermometry was then proposed.

INTRODUCTION

As is well known, temperature plays a key role in many fields, ranging from the biological domain to electronic devices.1−4 In general, temperature is expected to provide us much useful information based on which we can monitor the real time state of the object of interest and make a decision promptly. Because of the significance of temperature, recent years have witnessed the boom of thermometers, among which the luminescence thermometry based on nonTCELs are much more than those based on TCELs. However, the important point, that is, whether nonTCELs can be applied or not, is rarely studied in the field.

Generally, the processes for the populations of TCELs are the same because the gap between the two levels of TCELs is less than the threshold 2000 cm$^{-1}$.10 Therefore, it is unnecessary to consider the effect of laser pump power on the LIR between this pair of TCELs. Nonetheless, the energy gap between nonTCELs (ascribed to one kind of rare earth ion) is much larger than 2000 cm$^{-1}$. Thus, the channels to populate the two levels of nonTCELs may be different.11 In this case, it becomes meaningful to consider and correct the effect of laser pump power on the LIR between this pair of nonTCELs. Here, the $^5F_4$/$^5S_2$ and $^5F_4$ levels of Ho$^{3+}$ are taken as a typical example. These two levels are far from each other, and their space is ca. 3000 cm$^{-1}$ in various hosts. Therefore, they are not in thermal equilibrium. In this case, the ways to populate the $^5F_4$/$^5S_2$ and $^5F_4$ levels are likely to possess the same dependence on laser pump power, being an indication that the LIR between these two levels probably depends on laser pump power, to some extent.
bands studying the double logarithmic plot of the UC emission from 303 to 603 K, with a step increase of 20 K. When only the temperature of the prepared phosphors was changed, the latter was heated by a homemade heating chamber with an accuracy of ±0.3 K in the range of 303–783 K. In the stage of detecting the temperature-dependent UC luminescence of phosphors, only the temperature of the prepared phosphors was changed from 303 to 603 K, with a step increase of 20 K. When studying the double logarithmic plot of the UC emission bands’ intensities as a function of laser pump power, other experimental setups were kept unchanged except for the laser pump power of the irradiated point of phosphors.

**EXPERIMENTAL SECTION**

CaO, WO₃, Yb₂O₃, and Ho₂O₃ particles (>99.99%) were all of analytical purity. All involved chemicals were used as purchased without any further purification. CaWO₄:10% Yb³⁺–1%Ho³⁺ phosphors were prepared via the traditional high temperature solid state method, the specific procedure for which can be found anywhere.

Powder X-ray diffraction (XRD) of the samples was carried out (Rigaku D/MAX-2600/PC with Cu Kα radiation, λ = 1.5406 Å) at room temperature. A continuous wave 980 nm laser diode, whose current and output were controlled by the adjustor (ITC-4005, Thorlabs), was used as the excitation source. The spot of the samples irradiated by the laser diode was kept unchanged throughout the whole experiment, and its area was estimated to be 2 mm². The UC luminescence of phosphors was first focused by a lens with f = 3.8 cm, followed by the collection of this luminescence with the use of a photomultiplier tube (PMTH-3005, Zolix Instruments Co., Ltd) coupled with a commonly used photomultiplier tube (PMTH-1525, Zolix Instruments Co., Ltd). The slit width of the monochromator was set to be 2 mm. The voltage imposed on S1-CR131, Zolix Instruments Co., Ltd, was set to be 600 V. The sample was heated by a homemade heating chamber with an accuracy of ±0.3 K in the range of 303–783 K. In the stage of detecting the temperature-dependent UC luminescence of phosphors, only the temperature of the prepared phosphors was changed from 303 to 603 K, with a step increase of 20 K. When studying the double logarithmic plot of the UC emission bands’ intensities as a function of laser pump power, other experimental setups were kept unchanged except for the laser pump power of the irradiated point of phosphors.

**RESULTS AND DISCUSSION**

The XRD patterns of the prepared phosphors are presented in the inset in Figure 1a. It is found that the diffraction peaks of the samples are in good accordance with the reference date with number 41-1431 from the Joint Committee on Powder Diffraction Standards, indicating that CaWO₄:Yb³⁺–Ho³⁺ phosphors were prepared successfully and crystalized well, with the scheelite structure. Figure 1a presents the room temperature UC emission spectrum of CaWO₄:10%Yb³⁺–1%Ho³⁺ phosphors following the NIR excitation by a 980 nm laser diode; the inset shows the XRD patterns of the samples. (b) Energy level diagram of the Yb³⁺–Ho³⁺ system and the possible UC processes.

Following the NIR excitation by a 980 nm laser diode, Yb³⁺ at the 2F⁵/₂ level absorb these NIR photons and jump to the higher 2F⁷/₂ level. An energy transfer (ET) from Yb³⁺ to Ho³⁺ occurs, exciting the latter ions to the 5I₇ level from the 3I₈ level. In the next step, there are two possible routes. For one thing, the excited Ho³⁺ relax quickly to the lower 3I₇ level. Another ET from Yb³⁺ to Ho³⁺ excites the latter ions to the 5I₅ level from where the 5I₅→3I₇ transition takes place and leads to the 658 nm emission line. For another, Ho³⁺ at the 3I₈ level are further excited to the 5I₄ level by an ET process from Yb³⁺ to Ho³⁺, which is followed by the 5I₄→3I₈ and 5F⁵/₂→3I₈ transitions.

With the increase of temperature from 303 to 603 K, by integrating the emission line, and thus the LIR between the 540 and 658 nm spectral regions, the emission degree. This is, obviously, not good for the practical application of nonTCELs. This problem may be serious but is easily neglected. Chen’s group did the pioneering work concerning this issue. However, laser-induced heating was rarely considered. Moreover, related work is scarcely reported. To thoroughly investigate this issue and to push the LIR method based on nonTCELs as being more applicable, here, the 5F⁴→5S₂/5F⁵→3I₈ transitions of Ho³⁺ embedded in calcium tungstate host are taken as the object to be studied. CaWO₄:Yb³⁺–Ho³⁺ phosphors were prepared according to a simple high temperature thermal quenching (UC) emission spectra of the as-synthesized phosphors, following the near-infrared (NIR) laser diode excitation at 980 nm, were investigated in detail, aiming to show that the 5F⁴→5S₂/5F⁵→3I₈ transitions responded sensitively to temperature. In the next step, laser pump power was found to have an influence on the LIR between the 5F⁴→5S₂/5F⁵→3I₈ transitions and thus on the temperature readings derived from the LIR. What’s more, a calibration method to eliminate the influence of laser power dependence on luminescence thermometric thermometry was proposed.
intensities of the 540 and 658 nm lines can be obtained, respectively. One can see from Figure 2c that although both emission lines decrease gradually, there is a minor difference between them. Compared with the 658 nm emission line, the 540 nm one exhibits a faster decrease rate. Owing to this rate difference, LIR_{540/658} can be regarded as a temperature indicator. Figure 2d shows this ratio as a function of temperature in the range between 303 and 603 K. Considering that there is no physically clear model or theory to describe LIR_{540/658}, it is thus fitted by an empirical expression. This method had been used several times in previous literature. The empirical expression used here is

$$ LIR = \frac{1}{A + B \exp \left( -\frac{C}{T} \right)} $$

where LIR is the emission intensity ratio between the 540 and 658 nm emission lines and A, B, and C are constants. As can be observed from Figure 2d, these experimental data points are in good accordance with eq 1 (with $r^2 > 0.999$). The fit results for the parameters are $A = 2.8$, $B = 50.2$, and $C = 1086$ K. It thus suggests, undoubtedly, that the 540 and 658 nm emission lines can be used for ratiometric thermal sensing.

It is known that the relative sensitivity, $S_r$, is a key parameter to evaluate the performance of thermometers. According to the definition, $S_r$ can be written as

$$ S_r = \left| \frac{dLIR}{dT} \right| \frac{1}{LIR} $$

Based on eq 2, the relative sensitivity for the 540 and 658 nm emission lines was achieved, as shown in the inset in Figure 2d. As can be observed, $S_r$ decreases gradually from 0.45 to 0.27 K\textsuperscript{-1} on increasing the temperature from 303 to 603 K. The average $S_r$ was calculated to be 0.33 K\textsuperscript{-1} over the whole experimental temperature range.

It can be known from Figure 2a that the 540 and 658 nm emission bands are separated by a gap of ca. 3300 cm\textsuperscript{-1} which is much larger than 2000 cm\textsuperscript{-1}. Therefore, the 540 and 658 nm emission bands are nonthermally linked. Because of this large gap and the complex UC processes, the $^5\Phi_4, ^5\Phi_2$ and $^5\Phi_3$ levels might possess different dependences on laser pump power. It further indicates that the LIR between the $^5\Phi_4, ^5\Phi_2$ and $^5\Phi_3$ levels is probably sensitive to laser pump power, which is then investigated. Figure 3a presents LIR_{540/658} as a function of laser pump power at 303 K. As can be observed, LIR_{540/658} increases.
gradually on increasing the laser pump power. From eq 1, we have

\[
T_{\text{cal}} = \frac{-C}{\ln(1/LIR - A)}
\]  

(3)

where \(T_{\text{cal}}\) is the calculated temperature when the value of LIR\(_{540/658}\) is known. Substituting LIR\(_{540/658}\) obtained under the different excitation conditions presented in Figure 3a into eq 3, the temperature, \(T_{\text{cal}}\) was obtained, as depicted in the top panel in Figure 3b. It can be seen that \(T_{\text{cal}}\) decreases markedly on increasing the laser pump power. By using the expression \(T_{\text{err}} = T_{\text{cal}} - T_{\text{mea}}\), where \(T_{\text{mea}}\) is the practical temperature measured by a thermocouple and it is 303 K here, the temperature error, \(T_{\text{err}}\), was obtained, as presented in the bottom panel in Figure 3b. As can be observed, as the laser pump power is increased, \(T_{\text{err}}\) reaches \(-55\) K at the 205 mW excitation condition. At this point, it must be stressed that according to conventional recognition, that is, laser-induced heating, \(T_{\text{cal}}\) is expected to increase gradually on increasing the laser pump power. Therefore, the laser-induced temperature decreasing phenomenon, shown in Figure 3b, indicates that in addition to laser-induced heating, \(T_{\text{cal}}\) is expected to increase gradually on increasing the laser pump power. In this work, this mechanism is confirmed to be the dependence of LIR\(_{540/658}\) on the laser pump power. Obviously, it is necessary to distinguish the laser-induced heating first.

As can be observed from the inset in the top right of Figure 2b, the 642 nm emission peak increases gradually with temperature, which can be explained as follows. The 642 and 657 nm emission bands are ascribed to the transitions from the two Stark components of the \(5F_5\) level to the \(5I_8\) level of Ho\(^{3+}\). The gap between the two \(5F_5\) Stark sublevels is calculated to be ca. 355 cm\(^{-1}\), which is much smaller than the threshold 2000 cm\(^{-1}\) that ensures a thermal equilibrium between two closely spaced levels, indicating that the two \(5F_5\) Stark sublevels are thermally linked. Therefore, according to the Boltzmann distribution, the upper level will be populated at the expense of the depopulation of the lower one. On the basis of this theory, the LIR between the 642 and 657 nm emission bands, marked as LIR\(_{642/657}\), can be expressed as

\[
\text{LIR}_{642/657} = D \exp\left(-\frac{\Delta E}{kT}\right)
\]  

(4)

where \(D\) is a pre-exponential factor and \(\Delta E\) is the gap between the TCELS.

As confirmed by Quintanilla et al., an error will be introduced by the effect of laser heating when obtaining the thermal calibration curve in ratiometric thermometry as during the calibration the samples are always illuminated with the laser. Therefore, the laser heating effect should be considered first. As can be seen from Figure 4a, LIR\(_{642/657}\) increases gradually on increasing the laser power from 35 to 205 mW because of the laser-induced heating. At relatively high laser powers, LIR\(_{642/657}\) changes markedly. In contrast, in the power range below 35 mW, LIR\(_{642/657}\) keeps nearly unchanged. This phenomenon is similar to previous literature. It means that there is little heat generated by laser irradiation in the low pump power limit. Therefore, the laser power was set to be 35 mW during the achievement of the calibration curve for the emission bands of Ho\(^{3+}\), aiming to eliminate the laser heating in maximal degree. Figure 4b shows LIR\(_{642/657}\) as a function of temperature between 303 and 603 K under the excitation of 35 mW. As can be observed, LIR\(_{642/657}\) increases from 0.80 to 1.02 on increasing the temperature from 303 to 603 K. These experimental points are in good agreement with eq 4, and the fit results are \(A = 1.312(3)\) and \(\Delta E/k = 148(1)\) K. Equation 4 can be further expressed as

\[
T = -\frac{\Delta E/k}{\ln(\text{LIR}_{642/657}/D)}
\]  

(5)

On the basis of this equation, the temperatures under different laser pump excitations were calculated, as presented in Figure 4c. One can see that the temperature increases from 303 to ca. 323 K on increasing the laser pump power from 35 to 205 mW.

As mentioned above and as shown in the bottom panel in Figure 3b, LIR\(_{540/658}\) decreases markedly on increasing the
laser pump power. For comparison, it is also shown in Figure 5, labeled as Σ. It is obvious that this phenomenon is caused by two reasons, laser-induced heating and laser power dependence of LIR540/658. The laser-induced heating is also depicted in Figure 5 for comparison and is marked by Λ. Therefore, the pure power dependence of LIR540/658, Ω, should be equal to Σ − Λ. As can be observed from Figure 5, Ω decreases gradually from 0 to −75 K on increasing the laser power from 35 to 205 mW.

As demonstrated previously, the UC luminescence intensity, $I_{\text{em}}$, can be expressed as

$$I_{\text{em}} = Q P^n$$

where $Q$ is a constant, $P$ is the laser pump power, and $n$ is the photon number needed to excite the emitting levels involved. The 540 and 658 nm emission lines’ intensities are shown in Figure 6a as a function of laser pump power, with the double logarithmic plot. It can be seen that the two slopes of the linear fits for the 540 and 658 nm emission bands are 1.867(5) and 1.707(7), respectively. It suggests that both the emission lines are probably from two-photon UC processes, as depicted in Figure 1b. The two slopes were, however, found to be slightly different from each other. It suggests that the 540 and 658 nm emission lines come from different two-photon UC mechanisms, although the slopes are both close to 2. From Figure 6a, we have

$$I_{540} = Q_{540} P^{1.867}$$

$$I_{658} = Q_{658} P^{1.707}$$

where $Q_{540}$ and $Q_{658}$ are two constants. Based on eqs 7 and 8, the following can be obtained

$$\ln \left( \frac{I_{540}}{I_{658}} \right) = \ln \left( \frac{Q_{540}}{Q_{658}} \right) + 0.16 \ln(P)$$

(9)

It means that LIR540/658 is proportional to the laser power term $P^{0.16}$. Figure 6b presents LIR540/658 as a function of laser pump power, with the double logarithmic plot. As can be observed, LIR540/658 increases markedly with the increase of laser pump power. It is obvious that LIR540/658 is dependent on laser pump power. The slope of the linear fit was calculated to 0.159(3), being quite close to the expected value of 0.160(7). This fact indicates that the 540 and 658 nm emission lines, although both are from a two-photon UC mechanism, exhibit different dependence on laser pump power. It means that there are two different mechanisms or routes responsible for the $^3\text{F}_4$, $^5\text{S}_2$ and $^3\text{F}_4$ levels, and this is the fundamental cause for the dependence of LIR540/658 on laser pump power.

As there is power dependence of LIR540/658 on laser power, using the 540 and 658 nm emission lines for ratiometric thermal sensing becomes unreliable, to some degree. This has been demonstrated in Figure 3. Specifically, at a certain temperature, if two different laser pump powers are used, the two temperatures calculated from LIR540/658 values may be different, thus leading to an error. To overcome the limitation of the thermometer based on these two nonthermally linked bands, here, we propose a possible valid calibration method with the use of eqs 1 and 9. Equation 9 can be further written as

$$\ln \left( \frac{I_{540}}{I_{658}} \right) = \ln \left( \frac{Q_{540}}{Q_{658}} \right) + 0.16 \ln(35)$$

(10)

If we take LIR540/658 obtained under the excitation of 35 mW laser power (labeled as LIR540/658(35)) as a reference, we have

$$LIR_{540/658}(X) = \frac{Q_{540}}{Q_{658}} X^{0.16}$$

(11)

Therefore, LIR540/658 obtained under a certain excitation of X mW, LIR540/658(X), can be expressed as

$$LIR_{540/658}(X) = \frac{Q_{540}}{Q_{658}} X^{0.16}$$

(12)

On the basis of eqs 11 and 12, we have

$$LIR_{540/658}(X) \times \left( \frac{35}{X} \right)^{0.16} = LIR_{540/658}(35)$$

(13)

Combining eqs 1 and 13, we have

Figure 5. Temperature errors generated by (Λ) laser-induced heating, (Ω) laser power dependence, and (Σ) the combination of these two mechanisms as a function of laser pump power.

Figure 6. (a) 540 and 658 nm emission lines’ intensities and (b) LIR540/658 as a function of laser pump power, with the double logarithmic plot.
Based on eq 14, the LIR$_{540/658}(X)$ obtained at different laser pump powers has been corrected, and the results are presented in Figure 7 labeled as the green rhombuses. As can be observed, the corrected LIR$_{540/658}(X)$ keeps nearly unchanged at different laser pump powers, suggesting that the LIR$_{540/658}(X)$, after calibration, is immune to laser pump power. Therefore, the influence of laser pump power on LIR$_{540/658}$ for ratiometric thermal sensing is eliminated. It indicates that the proposed calibration method is valid.

**CONCLUSIONS**

In a word, LIR$_{540/658}$ was demonstrated to exhibit a monotonous and sensitive response to the temperature in the range of 303–603 K and it could be fitted well via an empirical formula. However, the temperature readout derived from this ratio decreased markedly from 303 to 248 K on increasing the laser pump power from 35 to 205 mW, with a maximum error of −55 K being different from the expectation of laser heating effect. The different dependences for the $^{1}P_{1/2}^{0}$S$_{3/2}/^{1}P_{1/2}$ transitions on laser pump power were confirmed to be responsible for this phenomenon. A calibration method to diminish the influence of laser pump power was then proposed and verified for CaWO$_4$:10%Yb$^{3+}$−1%Ho$^{3+}$ phosphors. It must be mentioned, however, that the feasibility and universality of this strategy should be checked in more compounds doped with different kinds of rare earth ions in the future.

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**Notes**

The authors declare no competing financial interest.

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