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Tingxing Shi,[1] Feng Chen,[1] Xiyu Zhao,[1] Jiahua Zhang,[2] Xiao-Jun Wang,[3] and Feng Liu[1,a)]

AFFILIATIONS

[1]Key Laboratory for UV-Emitting Materials and Technology of Ministry of Education, Northeast Normal University, Changchun 130024, China
[2]State Key Laboratory of Luminescence and Applications, Changchun Institute of Optics, Fine Mechanics and Physics, Chinese Academy of Sciences, Changchun 130033, China
[3]Department of Physics, Georgia Southern University, Statesboro, Georgia 30460, USA

[a)]Author to whom correspondence should addressed: fengliu@nenu.edu.cn

ABSTRACT

As an emerging approach to charge storage phosphors, upconversion charging (UCC) is attracting increased attention owing to its fundamental and practical perspectives. Despite the potential, further development of the UCC technology is restricted by the limited types of excitation light sources. Here, we use a white flashlight as excitation to investigate the UCC performance of storage phosphors. We demonstrate, as an example, that a $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor exhibits long-lasting emissions in the ultraviolet and visible regions after intense illumination from the flashlight. Thermoluminescence investigations reveal that both excited-state absorption and energy-transfer upconversion are involved in the UCC process. Based on the luminescence performance of the white-light charged phosphor, a conceptual thermometry approach is introduced, which can remotely sense the local temperature by monitoring the afterglow intensity ratio. Considering the wide use of flashlight, such a white-light excitability and the associated glow emission may potentially revolutionize the way to utilize storage phosphors.

Storage phosphors are a kind of special luminescent materials that can store excitation energy by trapping electrons or holes and then delay emissions after ceasing the excitation.[1–4] Generally, storage phosphors can be charged in three ways, namely, high-energy interband excitation, direct charge-transfer excitation, or upconversion charging (UCC).[2–4] In contrast to the progress of the first two excitation methods, research on the UCC is relatively lacking.

As a promising approach to charge storage phosphor using visible or infrared illumination, UCC technology has been considered to be of great academic and practical significance.[2–12] In a typical UCC process, it is now generally accepted that the trap in phosphor is filled after a two-step ionization of activator upon illumination. In addition, when the UCC of phosphor is mentioned, people also think of conventional upconversion luminescence (UCL), which is generated under a real-time excitation with a longer wavelength than that of the emission.[13] Previous studies on the UCC or UCL primarily focused on the luminescence performance under excitation by monochromatic light, such as lasers.[5–17] Taking into account the practical operations, however, it would be great if the phosphors are easily chargeable/excitable by more convenient light sources. Accordingly, a white light source, such as a white flashlight, which is much more readily available and less harmful than coherent lasers, is desired to charge/excite the phosphors.

Trivalent praseodymium ion (Pr^{3+}), which is characterized by $4f^2$ or 4f5d configuration, has been extensively studied as activator in phosphors. According to the electron structure and energy-level scheme of Pr^{3+} in phosphors, UCC and UCL processes upon illumination with non-coherent white light are predictable. In fact, UCL under excitation with a white flashlight has been reported in Pr^{3+}-doped garnet phosphors (e.g., $Lu_3Al_5O_{12}:Pr^{3+}$) in which only ultraviolet emission can be detected.[18] The absence of other emission wavelengths of Pr^{3+} beyond the ultraviolet is due to the presence of real-time excitation, which submerges the UCL signal in the visible region. Since emissions of storage phosphors (e.g., persistent luminescence) may be exempted from a real-time excitation, we can envisage to combine the multi-wavelength-emitting persistent luminescence and the white-light excitability in Pr^{3+}-doped garnet phosphors. Such a combination may not only provide insights to the understanding of the UCC mechanism but also enable some interesting applications,

such as luminescence temperature sensing by monitoring the afterglow emissions of the white-light charged phosphor.

In this work, we report the UCC process of Pr^{3+}-doped garnet phosphor, $Y_3Al_2Ga_3O_{12}$, using a white flashlight as the excitation source. We demonstrate that the intense flashlight illumination can produce ultraviolet and visible persistent luminescence in the phosphor. We also investigate the upconversion mechanism underlying the white-light charging and reveal the existence of both excited-state absorption and energy-transfer upconversion processes during the UCC excitation. Moreover, the white-light excitability of the phosphor as well as the multi-wavelength persistent luminescence is expected to serve as a conceptual luminescence thermometry in which the local temperature can be remotely sensed by monitoring the intensity ratio of two afterglow emission peaks after the end of the flashlight illumination.

The method for preparing Pr^{3+} doped garnet phosphor has been described previously.[9,10] A phosphor composition with a chemical formula of $Y_{2.995}Pr_{0.005}Al_2Ga_3O_{12}$ (hereafter referred to as $Y_3Al_2Ga_3O_{12}:Pr^{3+}$) was prepared by mixing Y_2O_3, Al_2O_3, Ga_2O_3, and Pr_6O_{11} powders and firing the mixture at 1350 °C for 3 h. To validate the concentration dependence of UCC, we also synthesized a series of phosphors with compositions of $Y_{3-x}Pr_xAl_2Ga_3O_{12}$ ($x = 0.0005$–0.05). Any dopant concentration of these phosphors can achieve UCC upon illumination with an intense flashlight by means of the flashlight beam transmitted subsequently through a lens combination (supplementary material, Fig. S1). Emission spectra for persistent luminescence were measured by a modular spectrofluorometer (QuantaMaster 8075–11, PTI) in which a CMOS detector was equipped (iStar-sCMOS-18U-73, Andor). Conventional persistent luminescence excitation spectrum was measured after illumination with filtered xenon light, while the UCC excitation spectrum was recorded after illumination with a tunable laser, whose wavelength was adjusted by using an optical parametric oscillator (Continuum SureLite). Before each measurement, the phosphor was bleached by heating it at 420 °C, which had been experimentally determined as the optimal temperature for thermal emptying of the traps in the phosphor. Thermoluminescence curves were measured by a TL Reader (SL08-L, Rongfan) with a heating rate of 4 °C s^{-1}. In the measurements of temperature dependent afterglow emissions, the temperature of sample was varied by mounting it in Cryo-77 cryostat (Orient Koji Scientific).

Conventional persistent luminescence performance of $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor has been reported.[19,20] That is, after ultraviolet irradiation, the phosphor exhibits characterized emission of Pr^{3+}, peaking at ultraviolet ($4f5d \rightarrow 4f^2$ transitions) and visible ($4f^2 \rightarrow 4f^2$) spectral regions (supplementary material, Fig. S2). The corresponding excitation spectrum is an indication of the illumination energy for directly filling the trap[21,22] (supplementary material, Fig. S2). In addition to the direct charging by ultraviolet irradiation, the ladderlike energy levels of Pr^{3+} in $Y_3Al_2Ga_3O_{12}$ allow us to deduce a UCC process upon illumination with a monochromatic light. Accordingly, we further measure the persistent luminescence after illuminating the phosphor with a 450 nm laser (supplementary material, Fig. S3). During such a UCC process, the illumination (e.g., 450 nm laser) excites the ground state of Pr^{3+} to a delocalized 4f5d state via an upconversion process, followed by filling of traps. In studying the excitability of UCC in the $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor, we further measure UCC excitation spectrum by recording the afterglow decay curves after excitation by a pulsed laser with tunable wavelengths over 440–500 nm. After laser excitations with different wavelengths, the phosphor exhibits varied afterglow intensities (supplementary material, Fig. S3). Accordingly, the UCC excitation spectrum is plotted by recording the afterglow intensity as a function of the laser wavelength [Fig. 1(a)]. This excitation spectrum exhibits four peaks at 450, 458,

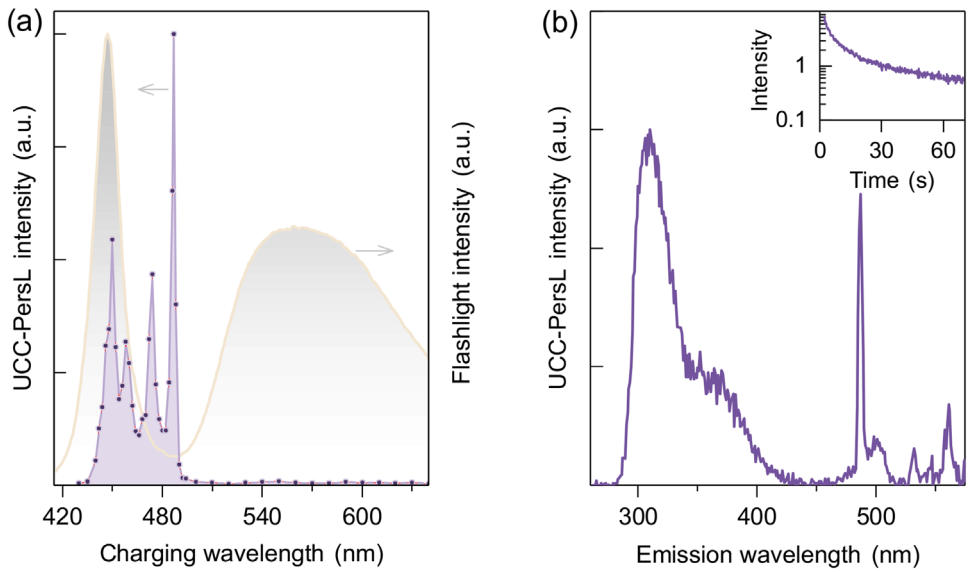

FIG. 1. Upconversion charging-induced persistent luminescence (UCC-PersL) of $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor. (a) UCC-PersL excitation spectrum recorded after charging the phosphor with a pulsed laser, whose output wavelength is tunable. The excitation spectrum is obtained by recording the afterglow decay intensity vs the charging wavelength. Emission spectrum of the flashlight is also presented. (b) UCC-PersL emission spectrum recorded at 10 s after ceasing flashlight illumination (power density, 1.77 W cm^{-2}) at room temperature. Inset shows the UCC-PersL decay curve of the white-light charged phosphor. The decay intensity is scaled in unit of $\mu W\ sr^{-1}\ m^{-2}$.

474, and 487 nm, indicating that the $^3H_4 \rightarrow {}^3P_2$, $^1I_6$, $^3P_1$, and $^3P_0$ excitation transitions may result in population of the upconversion intermediate state of Pr^{3+} ion.

Subsequently, to validate the feasibility of white-light illumination as excitation to charge the $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor, we record persistent luminescence emission spectrum and decay curve of the phosphor using an intense flashlight as the excitation source [output spectrum of the flashlight has been presented in Fig. 1(a)]. As shown in Fig. 1(b), after illumination with the flashlight (power density, 1.77 W cm^{-2}), the phosphor exhibits UCC-induced persistent luminescence, whose emission spectrum involves a structural broadband emission from the $4f5d \rightarrow (4f^2)$ ^3H_J,^3F_J inter-configurational transitions as well as sharp peaks mainly from the $(4f^2)$ $^3P_0$ state. The corresponding afterglow decay curves are recorded by measuring the afterglow intensities vs the decay times, as depicted in the inset of Fig. 1(b). Notably, although the UCC-induced persistent luminescence is detectable by the detector, the decayed emission intensity is too weak to be observed by the naked eye in the dark.

In the study of storage phosphors, thermoluminescence measurement is an alternative approach, which may evaluate the charging performance.[23] Accordingly, we measure the thermoluminescence curves of the $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor by monitoring at 310 nm with different excitation parameters of the flashlight, including varied power densities (0.065–1.77 W cm^{-2}) but a fixed illumination duration of 10 s. The resulting curves show varied intensities with similar spectral shapes, whose glow maximum peaks at around 130 °C (inset of Fig. 2). Figure 2 shows the thermoluminescence intensity (I) as a function of the flashlight excitation power (P). The straight line in Fig. 2 is an indication of quadratic relationship between the I and P at double-logarithmic coordination ($I \propto P^{1.81}$), providing evidence that a two-step ionization is involved in the UCC process (supplementary material, Fig. S4).[9–11]

Subsequently, to gain insight into the UCC of the $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor, we carry out an extensive thermoluminescence measurement and study the mechanism that accounts for the upconversion excitation. According to the typical upconversion excitation mechanisms, after excitation in the intermediate state of Pr^{3+}, the system may be further excited into the 4f5d state by excited-state absorption or energy transfer upconversion. As a matter of fact, excited-state absorption scheme often dominates upconversion in low-doped phosphor, whereas upconversion in highly doped phosphor is often dominated by energy-transfer scheme.[13]

During the UCC in $Y_3Al_2Ga_3O_{12}:Pr^{3+}$, the $^3P_0/^3P_1$ thermally coupled state can be directly populated upon illumination with the flashlight. If an excited-state absorption process contributes to the UCC, it is expected that the polychromatic light from the intense flashlight can promote the system from the $^3P_0/^3P_1$ state upward to the delocalized 4f5d state. To evaluate the explicit contribution of the white-light excitation during the UCC, we conduct thermoluminescence measurements upon illumination by a combined excitation involving the flashlight (0.45 W cm^{-2}) and a 488 nm laser (0.03 W cm^{-2}). In this measurement, a low laser power density (i.e., 0.03 W cm^{-2}) is applied, which is close to the upconversion excitation threshold of the phosphor, so that the thermoluminescence curve recorded after the laser illumination alone shows a barely detectable intensity [Fig. 3(a)]. Upon the flashlight illumination alone, the phosphor gives a distinct thermoluminescence signal [Fig. 3(a)], while the thermoluminescence intensity is further enhanced after exposing the phosphor to the combined excitation (the flashlight and the 488 nm laser), as shown in Fig. 3(a). The enhancement of thermoluminescence intensity results from a two-step excitation. Schematic representation of the UCC upon illumination with the combined excitation is illustrated in Fig. 3(b). From the above results, it can be concluded that the flashlight illumination accounts not only for the ground-state absorption but also for the excited-state absorption. Moreover, we also illuminate the phosphor using varied excitation

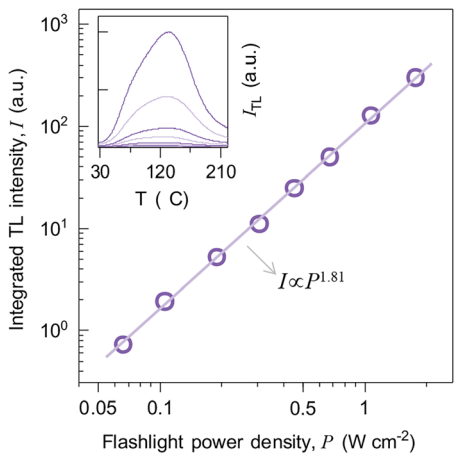

FIG. 2. Flashlight excitation power vs thermoluminescence intensity of $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor. Inset shows thermoluminescence (TL) curves of the phosphor recorded after the flashlight illumination with different power densities but a fixed exposure duration of 10 s. The integrated TL intensity (I) is plotted against the flashlight excitation power density (P). Fitting the data to a quadratic function ($I \propto P^{1.81}$) indicates that a two-step ionization is involved in the UCC process.

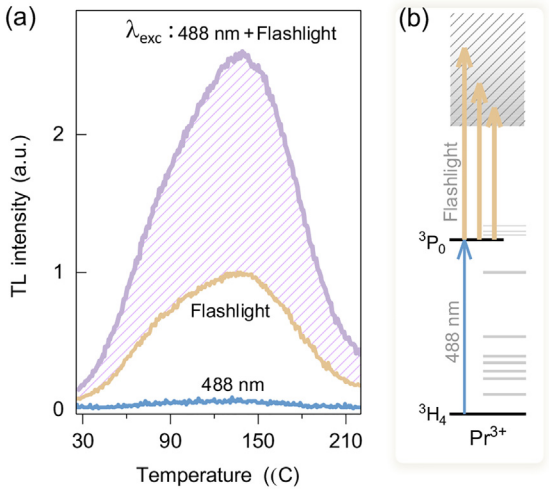

FIG. 3. UCC of $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor upon illumination with a combined excitation with a 488 nm laser and the flashlight. (a) Thermoluminescence (TL) curves recorded after illuminating the phosphor by the combined excitation. The power density of the 488 nm laser has been fixed as 0.03 W cm^{-2}, which is close to the threshold of the upconversion excitation. The output power density of the flashlight is 0.45 W cm^{-2}. (b) Schematic representation of the excited-state absorption process, which accounts for the shadow area in (a).

combinations, involving (1) a combined excitation of 450 and 532 nm lasers (supplementary material, Fig. S5) and (2) a combined excitation of 593 and 532 nm lasers (supplementary material, Fig. S6). For each combination, enhancement of thermoluminescence intensity is achieved via a two-step excitation, further verifying the excited-state absorption mechanism in the UCC excitation.

Although the excited-state absorption process is involved in the UCC of the $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor, it cannot be excluded that an energy-transfer upconversion contributes to the UCC excitation. Subsequently, to testify the energy-transfer upconversion, we prepare samples with different Pr^{3+} contents, changing the value of x from 0.001 to 0.1 (here x is defined by the formula $Y_{3-x}Pr_xAl_2Ga_3O_{12}$). First, we illuminate these phosphors under a 280 nm ultraviolet excitation and record the thermoluminescence curves of the phosphors with different Pr^{3+} contents [Fig. 4(a)]. Figure 4(b) shows that the thermoluminescence intensity reaches a maximum value for $x = 0.002$ and then decreases for higher Pr^{3+} concentration due to concentration quenching. Subsequently, we use the flashlight (1.77 W cm^{-2}) to excite each sample and record the thermoluminescence curves [Fig. 4(a)]. If an energy-transfer mechanism dominates the UCC excitation, the more efficient energy transfer is predictable along with higher Pr^{3+} concentration.[13] We plot the thermoluminescence intensity vs the Pr^{3+} content for the system $Y_{3-x}Pr_xAl_2Ga_3O_{12}$ after illumination with the flashlight. Figure 4(b) shows that the optimum content is at $x = 0.005$. Such a content results from a competition between the energy-transfer upconversion and the concentration quenching of emission. The comparison of concentration dependence in Fig. 4 indicates that the UCC excitation in $Y_{3-x}Pr_xAl_2Ga_3O_{12}$ involves an energy-transfer step. From these results presented in Figs. 3 and 4, we conclude that, upon illuminating the $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor with the flashlight, both excited-state absorption and energy-transfer upconversion processes are present in the UCC excitation.

The unique spectroscopic performance, involving the multi-wavelength afterglow emission [see Fig. 1(b)] and the white-light excitability, makes the flashlight-chargeable $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor a potential candidate as a ratiometric luminescence thermometry based on an afterglow intensity ratio (*AIR*) approach.

In previous studies, ratiometric luminescence thermometry based on afterglow intensity ratio (*AIR*) can only be achieved upon illumination with ultraviolet light.[24–26] Taking into account the fact that the ultraviolet irradiation may be harmful or not suitable for some practical applications, UCC approach may offer an alternative solution to the problem. That is, after the end of an upconversion excitation, the local temperature may be evaluated by means of the relative intensity ratio of two afterglow emission peaks of the employed phosphor. Moreover, if the phosphor is chargeable by white-light illumination, such an *AIR* thermometry will be inexpensive and convenient for practical applications.

In our study on the $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor, we define the *AIR* for thermometry as ratio of the integrated 4f5d→$(4f^2)$ $^3H_J/^3F_J$ transition intensity (290–410 nm) to the integrated $(4f^2)$ $^3P_0/^3P_1$ → $(4f^2)$ $^3H_4$ transition intensity (465–515 nm). After charging the phosphor using the flashlight at a certain temperature (for 10 s illumination at 1.77 W cm^{-2}), we record the persistent luminescence emission spectra at the corresponding temperature. Figure 5(a) shows that the afterglow intensity of the ultraviolet emission band decreases faster than that of the emission from the $(4f^2)$ $^3P_0/^3P_1$ state with increasing temperature. The change of the *AIR* along with temperature can be attributed to the thermal quenching mechanisms in which the lowest 4f5d state may be quenched by thermally activated crossover or thermal ionization process,[27] while the effect of thermal quenching in a certain temperature range is nearly negligible to the $^3P_0/^3P_1$ emission.[28]

Figure 5(b) shows the *AIR* of the $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor as a function of the sample temperature. The *AIR* decreases with temperature following a monotonical trend and so allowing its use for radiometric temperature measurement. We subsequently fit the *AIR* along with the temperature (T, unit in Kelvin) using the following formula: $AIR = AIR_0/(1 + \alpha e^{-\Delta E/kT})$, where AIR_0 is the afterglow intensity ratio at absolute zero temperature, ΔE is the thermally activated energy, k is the Boltzmann constant.[29] Constant α is related to the

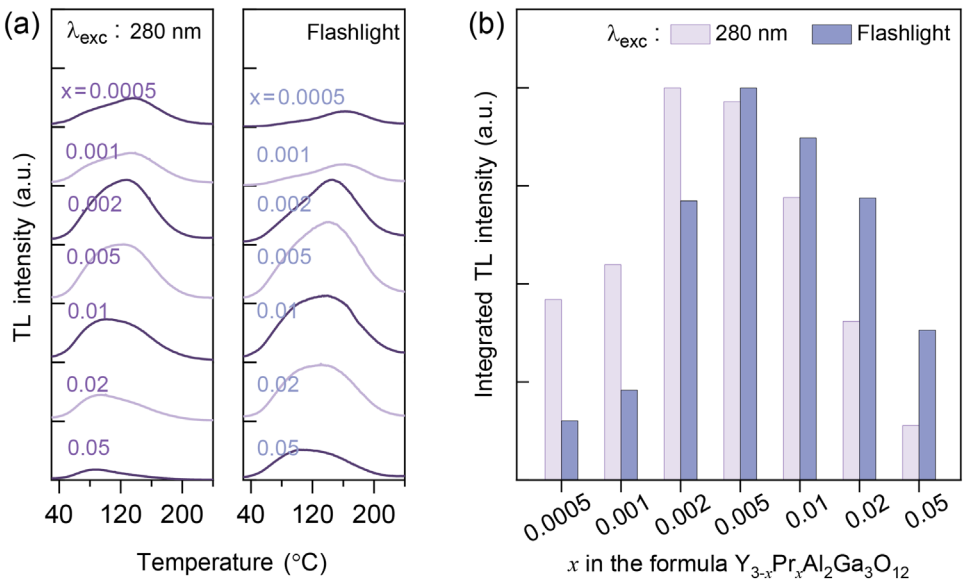

FIG. 4. Charging $Y_{3-x}Pr_xAl_2Ga_3O_{12}$ ($x = 0.0005$–0.05) phosphors using 280 nm xenon light irradiation or the flashlight. **(a)** Thermoluminescence (TL) curves of these phosphors, which have been charged by the 280 nm xenon light irradiation (left-hand side) and by the flashlight (right-hand side). (b) TL intensities recorded as a function of the Pr^{3+} content ($x = 0.0005$–0.05). The comparison of the TL intensities implies that an energy-transfer upconversion mechanism contributes to the charging process upon the flashlight illumination.

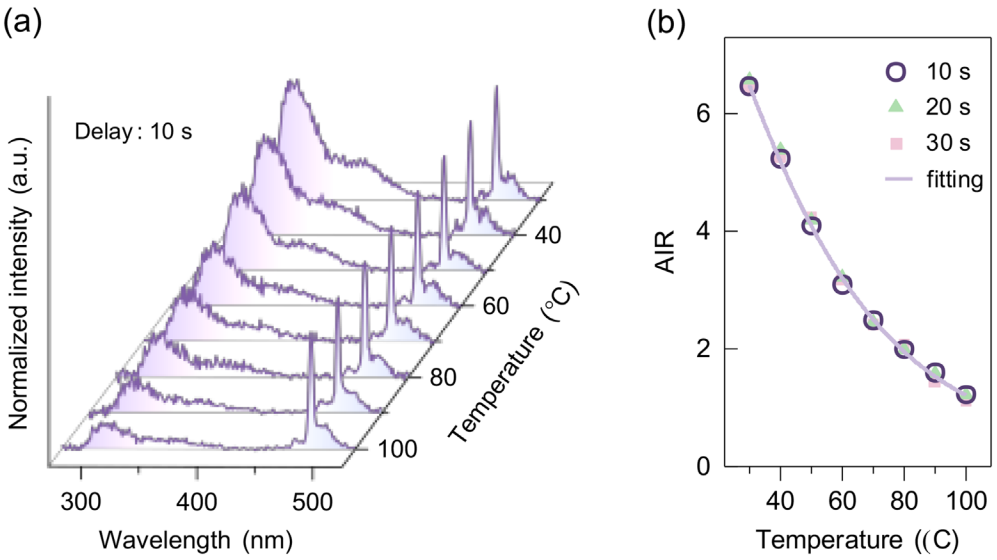

FIG. 5. Temperature dependence of persistent luminescence of $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor upon illumination with the flashlight. (a) Persistent luminescence emission spectra recorded at different temperatures over 30–100 °C. Before each measurement, the phosphor has been illuminated by the flashlight (1.77 W cm^{-2}) for 10 s at the corresponding temperature. Each spectrum is measured at 10 s after ceasing the flashlight illumination. All the spectra are normalized to the $^3P_0$ emission peaking at 487 nm. **(b)** Temperature dependence of the afterglow intensity ratio (*AIR*) of the emissions from the transitions of 4f5d→$(4f^2)$ $^3H_J/^3F_J$ and $(4f^2)$ $^3P_0$→$(4f^2)$ $^3H_4$.

thermal quenching of the 4f5d emitting state (supplementary material, Fig. S7). Moreover, the temperature dependence of *AIR* is also recorded at different times (10, 20, and 30 s) after ceasing the flashlight illumination (supplementary material, Fig. S7). As shown in Fig. 5(b), the *AIR* is nearly independent of delay time at a fixed temperature, implying that the present approach has a potential to serve as a radiometric luminescence thermometry. Notably, however, although the conceptual *AIR* thermometry possesses some excellent features over the conventional luminescence thermometry, there seems two intrinsic limitations on the *AIR* approach. The first is due to the poor signal-to-noise ratio of the persistent luminescence after charging by the flashlight, leading to a large temperature uncertainty.[30] The second limitation originates from the nature of trap, whose releasing is the result of temperature rise. The *AIR* approach, therefore, may monitor the thermal evolution of the phosphor from low to high temperature but does not work in the reverse way.

In summary, we have studied the UCC performance of $Y_3Al_2Ga_3O_{12}:Pr^{3+}$ phosphor upon charging with a white flashlight. Spectroscopic measurements indicate that various spectral compositions of the white-light illumination contribute to the excited-state absorption for charging the phosphor. Moreover, thermoluminescence investigation on the concentration dependence of UCC validates that an energy-transfer upconversion scheme is also involved in the white-light excitation. Based on the luminescence performance, we present an interesting demonstration that the flashlight-charged phosphor may serve for temperature sensing. Temperature dependence of persistent luminescence emissions indicates that the concept of *AIR* thermometry is feasible. Understanding and insights gained from these results will provoke the developments of UCC technology and the application upon white-light illumination. In addition to the present phosphor, we have explored other phosphor systems, including different ions activated phosphors, such as $LaMgGa_{11}O_{19}:Cr^{3+}$ and $LaMgGa_{11}O_{19}:Mn^{2+}$. All the phosphors exhibit UCC performance upon illumination with the white flashlight (supplementary material, Fig. S8).

See the supplementary material for the persistent luminescence spectra, UCC-persistent luminescence upon illumination by a combined excitation with 450 and 532 nm lasers, UCC-persistent luminescence emission spectra recorded at different temperatures, and UCC-persistent luminescence of various phosphors with white-light chargeability.

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AUTHOR DECLARATIONS

Conflict of Interest

The authors have no conflicts to disclose.

Author Contributions

Tingxing Shi: Data curation (equal); Investigation (equal); Writing – original draft (equal). **Feng Chen:** Data curation (equal); Investigation (equal); Writing – original draft (equal). **Xiyu Zhao:** Formal analysis (equal); Investigation (equal); Validation (equal). **Jiahua Zhang:** Formal analysis (equal); Funding acquisition (equal); Validation (equal). **Xiao-Jun Wang:** Formal analysis (equal); Methodology (equal). **Feng Liu:** Conceptualization (lead); Funding acquisition (equal); Project administration (lead); Supervision (lead); Writing – review & editing (lead).

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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