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White-light-emitting properties of SrTiO₃:Pr³⁺ nanoparticles

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Single-phase $SfTiO_3$: $mPr^3+(m=0.01-0.1)$ nanoparticles have been synthesized by hydrothermal method. Different from reported $Pr³⁺$ -based phosphors which were all dominated by red emissions, considerably strong and overwhelming blue-green emission centered at 491 nm has been achieved together with minor green and red emissions. The absolute and relative intensities of these emissions can be modified by $Pr³⁺$ concentrations and annealing temperatures in a controlled manner, giving rise to varied emission colors. In particular, owing to the appropriate combination of the blue to red emissions, desirable white-light emission has been realized from the as-grown SrTiO₃:0.01Pr³⁺ and the annealed SrTiO₃:0.06Pr³⁺ samples at 350 $^{\circ}$ C with CIE chromaticity coordinates of (0.33, 0.35) and (0.35, 0.37) and a correlated color temperature of 5601 K and 4883 K, respectively, indicating the promising application potential of $STIO_3:Pr^{3+}$ as single-phase phosphors for white light-emitting-diodes. This work may provide a fresh route to acquire white emission from a single-composition phosphor.

1 Introduction

As solid-state light sources, white light-emitting-diodes (WLED) have become specifically appealing due to their remarkable superiorities over conventional incandescent or white fluorescent illuminants, such as high efficiency, long lifetime, and low thermal resistance.¹⁻⁵ Considerable efforts have been paid on WLED design following different strategies. So far, the alliance of blue LED and yellow phosphors has been the most popular way to achieve WLEDs, in spite of obvious disadvantages of input power-dependent emitting color and deficiency in the red-light region. $6-9$ Combination of multiphosphors, including red, green and blue light-emitting phosphors, with a near ultraviolet (UV) excitation source could be another option. However, this method not only has high cost but also involves complex interactions among the emission and excitation lights. 10^{-12} Therefore, UV-excited white-emitting phosphor from a single host is preferred, which has been successfully fabricated by either doping a single rare earth ion Sm^{3+} , Eu³⁺ or Dy³⁺),¹³⁻¹⁵ or co-doping ion pairs (Ce³⁺-Mn²⁺, $Eu^{2+}-Mn^{2+}$, $Ce^{3+}-Eu^{2+}$, $Ce^{3+}-Tb^{3+}$, *etc.*) based on the energy transfer mechanism,16-19 or multi-doping ions emitting different colors, such as $Ce^{3+}/Li^{+}/Mn^{2+}$, $Eu^{3+}/Tb^{3+}/Tm^{3+}$ and $Eu^{2+}/Tb^{3+}/Eu^{3+}, ^{20-22}$ into oxides or fluorides.

 $Pr³⁺$ -doped inorganic materials are well-known for the promising emission properties, in particular as red phosphors. The three red emissions are from different origins of the $4f^2-4f^2$

transitions, *i.e.* ¹D₂-³H₄ (603 nm), ³P₀-³H₆ (617 nm) and ³P₀-³F₂ (650 nm).²³⁻²⁵ Among them, the ${}^{1}D_{2}$ -³H₄ emission was studied most extensively, and its optimized emission efficiency was obtained at a Pr^{3+} doping concentration of 0.2-1.0%.²⁶⁻²⁹ For a higher doping, concentration quenching effect will reduce the emission, because the odds of nonradiative energy transfer between Pr^{3+} ions can be increased with Pr^{3+} concentration.³⁰ Recently, a red phosphor based on the ${}^{3}P_{0}$ - ${}^{3}F_{2}$ emission from $BaMoO₄:Pr³⁺$ was reported, and the strongest emission was produced at a Pr^{3+} concentration of 2% ³¹ In addition to the red emissions, $Pr³⁺$ -doped materials also have two green emissions corresponding to ${}^{3}P_{1}$ - ${}^{3}H_{5}$ (530 nm) and ${}^{3}P_{0}$ - ${}^{3}H_{5}$ (547 nm) together with one blue-green emission $({}^{3}P_{0}^{-3}H_{4}$ (491) nm)),^{24,26,32} which were all usually neglected for phosphors application due to the rather low intensity compared to that of the red emissions at room temperature. Apparently, white-light emission of $Pr³⁺$ has not been reported. However, previous work on Pr^{3+} -doped ABO₃ perovskite-type oxide delivered interesting result that Pr^{3+} ions occupied on the A- and B-sites are associated with the ${}^{1}D_{2}$ - ${}^{3}H_{4}$ and ${}^{3}P_{0}$ -related emissions, respectively.33 This finding implies that the emission properties, especially the relative intensities of different emissions are likely to be modified by tuning the accommodation of $Pr³⁺$ on the two sites. Additionally, the compositions of the perovskitetype hosts have been confirmed to remarkably influence the intensity of the ${}^{3}P_{0}$ -related emissions at room temperature.³² If one can regulate the combination of the blue to red emissions in

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a controlled manner, white emission from a single host may be realized from $Pr³⁺$ -doped materials.

In this work, quasi-spherical $SrTiO₃:Pr³⁺$ nanoparticles with a Pr^{3+} concentration in the range of 1-10% have been synthesized by a hydrothermal method. Different from reported $Pr³⁺$ -based phosphors mentioned above, considerably strong and dominated ${}^{3}P_{0}$ -related blue-green emission centered at 491 nm has been achieved together with minor green and red emissions. Varied doping concentration and annealing temperatures have been employed to modify the absolute and relative intensities of these emissions, aiming on a proper combination of the blue to red emissions. As a result, adequate white emission has been successfully accomplished from $SrTiO₃:Pr³⁺ products.$

2 Experimental

A series of $SrTiO₃:mPr³⁺$ ($m=0.01-0.10$) nanoparticles were synthesized by a hydrothermal method. First, 40 ml aqueous solution of praseodymium nitrate hexahydrate (1.25×*m* mol/L, 99%, Aladdin) and strontium nitrate (1.25×(1-*m*) mol/L, 99%, Aladdin) were mixed with 23 ml ethanol solution of tetrabutyl titanate (1.125mol/L, 99%, Aladdin). The pH value of the mixed solution was set to be 13.5 by adding NaOH. The reactive solution was then sealed in a Teflon autoclave at 160 C for 48 h. After cooling down to room temperature, the obtained product was thoroughly washed by deionized water and ethanol, eventually dried overnight in air.

The crystallization nature and morphology of the samples were characterized using powder X-ray diffraction (XRD; Bruker, Advanced D8, with Cu Kα radiation) and scanning electron microscopy (SEM; JEOL 6700F). Photoluminescence (PL) and photoluminescence excitation (PLE) spectra of the samples were measured using a spectrofluorometer (HORIBA, Fluoromax-4).

3 Results and discussion

To modify and optimize the emission properties, we tuned the concentration of Pr^{3+} and synthesized a series of $SrTiO₃: mPr^{3+}$ samples, where *m*=0, 0.01, 0.03, 0.05, 0.06, 0.07, 0.08 and 0.10. The crystallization nature of the samples was characterized by XRD, as shown in Figure 1. All peaks in the spectra can be assigned to the SrTiO₃ host structure (ICSD files no. 27045), and no phase related to impurities was observed. Apparent peak shifts with Pr3+ concentrations can be identified in Figure 1. The unit-cell parameters refined with Pm3m space group using MDI Jade 5.0 software are depicted in Table S1. With *m* increases from 0 to 0.1, the refined lattice parameters and unitcell volumes decrease firstly and reach the minimum values at *m*=0.03, and then increase gradually. Given that the ionic radius of Sr^{2+} , Ti^{4+} and Pr^{3+} are 1.44 Å, 0.61 Å and 1.13 Å respectively, such variation with m indicates $Pr³⁺$ ions prefer to occupy A-sites when *m*≤0.03, and B-sites when *m*>0.03. Despite the varied $Pr³⁺$ concentration, all nanoparticles emerge quasi-spherical morphology with a similar size. A SEM image

Fig.1 XRD patterns of $SrTiO₃: mPr³⁺$ ($m=0$, 0.01, 0.03, 0.05, 0.06, 0.07, 0.08 and 0.10) powers. SEM image of the SrTiO₃:0.06Pr³⁺ sample is presented in the inset.

Fig.2 PL spectrum of the as-grown $SrTiO₃:0.06Pr³⁺$ nanoparticles with an excitation of 325 nm and its PLE spectra monitored at 491 nm and 603 nm.

Figure 2 shows the PL and PLE spectra of the $SrTiO₃:0.06Pr³⁺$ powders, normalized respectively by their own maximum intensity. It is noted that, different from the overwhelming red emissions reported in the literature, PL spectrum of the $SrTiO_3:0.06Pr^{3+}$ sample under the excitation of 325 nm is dominated by a very strong blue-green emission at 491 nm together with minor peaks, including two green emissions (530 nm and 547 nm) and three red emissions (603

The PLE spectrum monitored at 491 nm was measured in an excitation wavelength range < 400 nm, showing a clear absorption edge in the vicinity of 370 nm corresponding to the band gap of $SrTiO₃$. Although the three red emissions are stepped from totally different origins, their PLE spectra do exhibit a rather similar profile (See Figure S2), showing an absorption edge of the matrix $SrTiO₃$ together with three sharp peaks centered at 447 nm, 474 nm and 488 nm, which are attributed to ${}^{3}H_{4}{}^{3}P_{2}$, ${}^{3}H_{4}{}^{3}P_{1}$ and ${}^{3}H_{4}{}^{3}P_{0}$ of the 4f²-4f² Pr³⁺ transitions, respectively.²⁷ It was found that the 447 nm excitation (See Figure S3) can also excite the strong blue-green emission as well as the green and red emissions from $SrTiO₃:0.06Pr³⁺$, implying both UV and blue excitation can be used for promising application of $SrTiO₃:Pr³⁺$ as a single-host phosphor.

under 325 nm excitation. The inset shows the $Pr³⁺$ concentration dependence of the emission intensity of $SrTiO₃:mPr³⁺$.

Can white emission be realized by tuning the $Pr³⁺$ concentration then? Figure 3 presents PL of $SrTiO₃: mPr³⁺$ samples with *m* in the range of 0.01–0.1 and an excitation of 325 nm. Dependences of the 491 nm, 603 nm and 617 nm integrated peak-area intensities $(I_{491}, I_{603}$ and I_{617}) on *m* are shown in the inset. It is revealed that the ${}^{3}P_{0}$ -related I_{491} and I_{617} both have an inflexion at *m*=0.06, *i.e*., increased with *m* when *m* < 0.06 and reduced when $m > 0.06$. This means that the concentration quenching effect for ${}^{3}P_{0}$ - ${}^{3}H_{4}$ (491 nm) and ${}^{3}P_{0}$ - ${}^{3}H_{6}$ (617 nm) emissions occurs at a Pr³⁺concentration above 6%, different from the case of ${}^{1}D_{2}$ - ${}^{3}H_{4}$ (603 nm) emissions, where the strongest emission is from a 0.2-1.0% doping. Moreover, I_{491} and I_{617} of the SrTiO₃:0.06Pr³⁺ sample are, respectively, enhanced about 60% and 20% compared to those of *m*=0.01. While for the I_{603} , the quenching effect has occurred when $m \geq$ 0.01, consistent with previous results.²⁶⁻²⁹ Interestingly, I_{603} is not monotonically decreased with *m* as anticipated, but shows a

small peak at $m=0.06$, similar to I_{491} and I_{617} . This elucidates that a nonradiative transition from ${}^{3}P_{0}$ to ${}^{1}D_{2}$ occurs. Considering increase of $Pr³⁺$ ion occupations on the A- and Bsites with increasing *m*, the doping concentration (*i.e. m*) dependent emission properties and unit cell parameters of the $SrTiO₃: mPr³⁺$ samples are consistent with Reference 33.

The varied emission properties for different $Pr³⁺$ concentrations, in particular the changes of the relative intensity of different emissions, directly indicate that the emission color of $SrTiO_3$: $mPr³⁺$ products is tunable in a controlled manner. In order to visually confirm the white emission, the CIE coordinates (x, y) for as-grown SrTiO₃: mPr^{3+} phosphors were measured, obtaining (0.33, 0.35), (0.31, 0.37), (0.30, 0.37), (0.30, 0.38), (0.29, 0.36), (0.31, 0.37) and (0.31, 0.38) for *m*=0.01, 0.03, 0.05, 0.06, 0.07, 0.08, and 0.1, respectively, which means that all these $SrTiO_3$: $Pr³⁺$ samples deliver white emission. Comparing these samples, $SrTiO₃:0.06Pr³⁺$ presents the highest emission efficiency, and the CIE (0.33, 0.35) of $SrTiO_3:0.01Pr^{3+}$ is revealed to be the nearest to the standard CIE (0.33, 0.33) coordinates of white light (see in Figure 4) with a calculated correlated color temperature (CCT) of 5601 K, within the range of 5500-6000 K for typical daylight.

Fig. 4 CIE chromaticity diagram and chromaticity coordinates (x, y) of emission from as-grown SrTiO₃:0.01Pr³⁺ and SrTiO₃:0.06Pr³⁺ samples before and after annealing at different temperatures.

Annealing treatment has been widely confirmed as another effective means to enhance and/or modify emission properties of rare-earth ions-doped oxides, which may affect various aspects of the products, such as the crystallinity, crystal field and oxygen vacancies.^{27,34,35} Post-annealing treatments in argon for 2 h at different temperatures were performed on the $SrTiO₃:0.06Pr³⁺$ samples. The annealed samples present the

similar morphology to that of the as-grown sample as shown by the SEM image in Figure S4, while their emission properties have been significantly modified. The PL results reveal that an annealing below 750 °C can barely change I_{491} , I_{530} , I_{547} and I_{650} , but remarkably enhance $I_{603+617}$, confirmed by the PLE spectra of $SrTiO_3:0.06Pr³⁺$ sample annealed at 750 °C (monitored at 491 nm and 617 nm, Figures S5 and S6). Interestingly, annealing at 1350 °C can enhance all emissions especially the red emissions of $I_{603+617}$. Figure 5 presents PL spectra of SrTiO₃:0.06Pr³⁺ samples annealed at different temperatures, normalized by their I_{491} . It is found that the relative intensity of red emission can be enhanced with temperature, which shifts the CIE coordinate from (0.30, 0.38) at room temperature to (0.35, 0.37), (0.43, 0.36), (0.53, 0.35) annealed at 350 ºC, 550 ºC, and 750 ºC, respectively. The SrTiO₃:0.06Pr³⁺ annealed at 350 °C possesses a much improved white light emission with CIE coordinates of (0.35, 0.37) and CCT of 4883 K, which is close to 5000 K, *i.e*., the value of horizon daylight. We conclude that post-annealing is an efficient way to further tune the emission color of the products besides varying $Pr³⁺$ concentration, and both the $SrTiO₃:0.06Pr³⁺$ annealed at 350 °C and the as-grown $SrTiO₃:0.01Pr³⁺$ can be desirable white emitting phosphors from a single host to function as WLEDs.

Fig.5 PL of $SrTiO₃:0.06Pr³⁺$ nanoparticles annealed at different temperatures.

We notice that post-annealing at 750 ºC can modify the emission color of $SrTiO_3:0.06Pr^{3+}$ from white to red, while increased annealing temperature to 1350 ºC can further enhance the red emission, with $I_{603+617}$ 14 times as that of the as-grown sample, resulting in CIE coordinates of (0.56, 0.35). It was confirmed that 490 nm can efficiently excite the annealed $SrTiO₃:0.06Pr³⁺$ samples to get red-color dominated emission (See Figure S7). It suggests that the annealed $SrTiO₃:0.06Pr³⁺$ can also combine with widely used commercial blue LED chips at 490 nm to achieve WLEDs with a blue LED plus blue-red phosphor strategy (See Figure S8). By adjusting the output power of the 490 nm LED chips, the ideal CIE coordinates of

(0.33, 0.33) with correlated color temperature of 5612 K can be achieved.

To further understand the influence of annealing treatment on the emission properties, XRD patterns of as-grown and annealed $SrTiO_3:0.06Pr³⁺$ samples (at an annealing temperature of 750 °C and 1350 °C) have been examined (Figure S9). The profile of the results looks quite similar with all the peaks assigned to the $SrTiO₃$ -host structure and evident peak shift at different annealing temperature. Analysis based on the XRD results reveals that the relative refined unit-cell parameters are reduced with annealing temperature increasing (Table S2). According to the emission results, the reduced A-site and increased B-site occupation of $Pr³⁺$ can enhance the emission intensity of $SrTiO₃:0.06Pr³⁺$. And such changes of $Pr³⁺$ occupations are supposed to induce increased unit-cell parameters, which are apparently conflicted with our XRD results

Formation of oxygen vacancies can also lead to decreased unit-call parameters. It was pointed out in Reference 35 that oxygen vacancies resulted from the vacuum annealing at $700\,^{\circ}\text{C}$ can create a defect level in the band gap of $SrTiO₃ \cdot Pr³⁺$ sample, and benefit energy transfer to Pr^{3+} to enhance the band at ~350 nm in the PLE spectra monitored at the ${}^{1}D_{2}$ -3H₄ red emission, showing remarkably enhanced ${}^{1}D_{2}$ - ${}^{3}H_{4}$ red emission and barely changed ${}^{3}P_{0}$ related blue emission at ~491 nm. Those results are consistent well with both the PL and the XRD results obtained from the $SrTiO₃:0.06Pr³⁺$ samples annealed in argon at 750 °C in this work. Therefore, the different behaviours of the blue and red emission upon annealing is likely due to the much weaker energy transfer from the oxygen deficiency level to ${}^{3}P_{0}$ level than that to the ${}^{1}D_{0}$ level times that the ${}^{3}D_{0}$ level than the ${}^{1}D_{0}$ level (Figure D_2 level given that the ³P₀ level is higher than the ¹D₂ level (Figure S10). The simultaneous enhancement of both the blue and the red emission of the SrTiO₃:0.06Pr³⁺ annealed in argon at 1350 °C can be understood by considering the significantly modified crystallinity and crystal field of the $SrTiO₃:Pr³⁺$ sample because the high annealing temperature of 1350 °C is closed to the widely employed sintering temperature of $SrTiO_3$: $Pr^{3+34-37}$

Conclusions

Different from reported $Pr³⁺$ -based phosphors, which were all dominated by red emissions, considerably strong and overwhelming blue-green emission corresponding to ${}^{3}P_{0}$ - ${}^{3}H_{4}$ transition has been achieved from a series of single-phase $SrTiO₃:Pr³⁺$ nanoparticles, with a $Pr³⁺$ -doping concentration in the range of 1-10%. The emission properties can be modified controllably and efficiently by both the $Pr³⁺$ concentration and the annealing treatments. Owing to the appropriate combination of the blue to red emissions, desirable white-light emission has been realized from the as-grown $SrTiO₃:0.01Pr³⁺$ samples (CIE chromaticity coordinates of (0.33, 0.35) and CCT of 5601 K) and the annealed $SrTiO₃:0.06Pr³⁺$ samples (CIE chromaticity coordinates of (0.35, 0.37) and CCT of 4883 K), revealing the promising potential of $SrTiO₃:Pr³⁺$ for WLED applications. Our work also provides a fresh route to acquire white emission from a single-composition phosphor, *i.e*., by modifying and controlling relative intensity of different emissions of the single product, which may be applied on diverse light-emitting materials.

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Notes and references

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c Department of Mathematics and Materials Research Institute, The Pennsylvania State University, University Park, Pennsylvania16802, USA. Electronic Supplementary Information (ESI) available: Size distribution of the $SrTiO₃:0.06Pr³⁺$ Nanoparticles, PLE spectra of the as-grown $SrTiO₃:0.06Pr³⁺$ monitored at 603 nm 617 nm and 650 nm, PL of the asgrown $SrTiO₃:0.06Pr³⁺$ sample under 447 nm excitation, PL of the 1350°C annealed SrTiO₃:0.06Pr³⁺ sample under 490 nm excitation, and the energy level diagram of $Pr³⁺$ ions.

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Graphical abstract

White-Light-Emitting Properties of SrTiO3:Pr3+ Nanoparticles

Wei Tang, Ye Sun,* Miao Yu,* Xiao Liu, Yongqi Yin, Bin Yang, Limei Zheng, Feng Qin, Zhiguo Zhang, Wenwu Cao

White-light emission from $SrTiO₃:Pr³⁺$ nanoparticles has been achieved by tuning combination of the ${}^{3}P_0$ and ${}^{1}D_2$ -related blue to red emissions.

