Thermally enhanced photoluminescence and temperature sensing properties of $\text{Sc}_2\text{W}_3\text{O}_{12}$$:\text{Eu}^{3+}$ phosphors

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Abstract

Currently, lanthanide ions doped luminescence materials applying as optical thermometers have arose much concern. Basing on the different responses of two emissions to temperature, the fluorescence intensity ratio (FIR) technique can be executed and further estimate the sensitivities to assess the optical thermometry performances. In this study, we introduce different doping concentrations of Eu\(^{3+}\) ions into negative expansion material Sc\(_2\)W\(_3\)O\(_{12}\), accessing to the thermal enhanced luminescence from 373 to 548 K, and investigate the temperature sensing properties in detail. All samples exhibit good thermally enhanced luminescence behavior. The emission intensity of Sc\(_2\)W\(_3\)O\(_{12}\) : 6 mol\% Eu\(^{3+}\) phosphors reaches at 147.81\% of initial intensity at 473 K. As the Eu doping concentration increases, the resistance of the samples to thermal quenching decreases. The FIR technique based on the transitions \(^5\)D\(_0\)\(\rightarrow\)\(^7\)F\(_1\) (592 nm) and \(^5\)D\(_0\)\(\rightarrow\)\(^7\)F\(_2\) (613 nm) of Eu\(^{3+}\) ions demonstrate a maximum relative temperature sensitivity of 3.063 K\(^{-1}\) at 298 K for Sc\(_2\)W\(_3\)O\(_{12}\) : 6 mol\% Eu\(^{3+}\) phosphors. The sensitivity of sample decreases with the increase of Eu\(^{3+}\) concentration. Benefiting from the thermal enhanced luminescence performance and good temperature sensing properties, the Sc\(_2\)W\(_3\)O\(_{12}\) : Eu\(^{3+}\) phosphors can be applies as optical thermometers.

Keywords: photoluminescence, Sc\(_2\)W\(_3\)O\(_{12}\) : Eu\(^{3+}\), negative lattice expansion, thermal enhanced luminescence,
1. Introduction

Currently, lanthanide ions doped luminescence phosphors have been widely applied in displays,\(^1\) lighting,\(^2\) and sensors\(^3\). Therein, optical thermometry based on luminescence materials have arose many attentions due to the fast response, non-contact and high sensitivity.\(^4\),\(^5\) By employing the fluorescence intensity ratio (FIR) of thermally coupled energy levels (TCELs) or non-thermally coupled energy levels (NTCELs), the temperature sensing properties can be obtained.\(^6\),\(^7\) The potential of luminescence materials for applied as optical thermometers is evaluated by absolute sensitivity \((S_a)\) and relative sensitivity \((S_r)\).\(^7\),\(^8\) For Eu\(^{3+}\)-doped materials, several pairs of emissions have been executed for FIR technique due to the abundant energy levels of Eu\(^{3+}\) ions.\(^9\),\(^10\) Liang et al. revealed that \(5\text{D}_1/5\text{D}_0\rightarrow7\text{F}_1\) TCELs (541 nm/590 nm) and \((5\text{D}_0\rightarrow7\text{F}_2)/(5\text{D}_1\rightarrow7\text{F}_1)\) NTCELs (625 nm/541 nm) of Eu\(^{3+}\) ions in LiNbO\(_3\) single crystals can be devoted to the optical temperature sensor based on FIR method, and the maximum \(S_a\) values are \(7\times10^{-4}\text{K}^{-1}\) and \(24\times10^{-4}\text{K}^{-1}\), respectively.\(^11\) In addition, Nikolić et al. developed \((5\text{D}_1\rightarrow7\text{F}_1)/(5\text{D}_0\rightarrow7\text{F}_2)\) NTCELs (533 nm/611 nm) in Gd\(_2\)O\(_3\):Eu phosphors for thermography, and the maximum \(S_a\) is \(7\times10^{-4}\text{K}^{-1}\) at 800 K.\(^12\)

Generally, the performance of luminescence materials for applications, including optical thermometry, is usually restricted by inevitable debasement of luminescence intensities with elevated temperatures, namely thermal quenching.\(^13\) So far, many efforts have been devoted to resist thermal quenching.\(^14\)-\(^16\) Negative thermal expansion (NTE) luminescence materials have triggered much concern due to the resistance of volume expansion with increasing temperature, which is a distinct advantage to practical applications.\(^17\) In particular, the energy collection by activators can be promoted by the reversible lattice shrinkage and deformation, resulting the thermal enhanced emissions.\(^17\),\(^18\) For instance, Zou et al. reported NTE up-conversion Yb\(_2\)W\(_3\)O\(_{12}\):Er crystal with more than 12 times luminescence enhancement when elevating temperature to 573 K.\(^18\) Besides, Zhou et al. demonstrated that the NTE effect of Zr(WO\(_4\))\(_2\):Eu phosphors can effectively inhibit the emission loss of thermal quenching. Compared with room temperature, the luminescence intensity can be enhanced by 130% at 373 K.\(^19\) Furthermore, the investigation on luminescence
properties of lanthanide ions doped NTE Sc$_2$W$_3$O$_{12}$ material was carried out by researchers. Li et al reported Eu$^{3+}$-doped Sc$_2$W$_3$O$_{12}$ phosphors with good thermal quenching resistance at low temperature (97-280 K). Recently, Wang et al. demonstrated that the emission intensity of Sc$_2$W$_3$O$_{12}$:Eu$^{3+}$ (x = 0.01–0.10) are abnormally enhanced with increasing temperatures up to 473 K.

In this work, we have successfully synthesized Sc$_2$W$_3$O$_{12}$ phosphors with high Eu$^{3+}$ concentration (6-34 mol%). The luminescent thermal properties of Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ with various concentrations and their application in the field of optical temperature measurement were systematically investigated. Under the excitation at 264 nm, the bright red emission of Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ phosphors exhibit good thermally enhanced luminescence from 373 to 548 K. Specifically, the Sc$_2$W$_3$O$_{12}$: 6 mol% Eu$^{3+}$ phosphors presents 147.81% of initial intensity at 473 K. Besides, owing to the different responses of emissions at 592 nm ($^5$D$_0$→$^7$F$_1$) and 613 nm ($^5$D$_0$→$^7$F$_2$) to temperature, Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ phosphors show good temperature sensing properties based on FIR technique. The estimated maximum S$_a$ and S$_r$ are 6.872×10^{-3} K$^{-1}$ and 3.063% K$^{-1}$ at 298 K, respectively. These results verify the availability of lanthanide ions doped in NTE luminescence materials against thermal quenching, and demonstrate the application in optical thermometers.

2. Experimental

2.1. Sample preparation

Sc$_2$W$_3$O$_{12}$ phosphors were prepared by high-temperature solid-state reaction. Sc$_2$O$_3$ (Scandium oxide, 99.99%, Aladdin), WO$_3$ (Tungsten trioxide, 99.99%, Aladdin) and Eu$_2$O$_3$ (Europium oxide, 99.99%, Aladdin) were used as raw materials. After weighed according to stoichiometry ratio, the raw materials were stirred in planetary ball mill for 12 h. After drying the obtained slurry, the mixture was pressed into thin sheets with a diameter of 15 mm and a thickness of 2 mm, and sintered in a box furnace at 1100 °C for 4 h. The sintered samples can be used for subsequent tests after being carefully ground in an agate mortar.

2.2. Characterization
The X-ray diffraction (XRD) of all samples were identified by the D8 ADVANCE X-ray diffractometer of the Bruker corporation with Cu-Kα radiation ($\lambda=1.5406$Å). Morphologies were characterized on a field-emission scanning electron microscope (FE-SEM, JSM-6700 F). Photoluminescence (PL) and PL excitation (PLE) spectra were measured using a fluorescence spectrophotometer (F-7000, Hitachi) equipped with a high-temperature fluorescence controller (TAP-02).

3. Results and discussion

3.1 Phase and morphology analysis

Fig. 1(a) shows the XRD results of Sc$_2$W$_3$O$_{12}$: x mol% Eu$^{3+}$ phosphors ($x=6, 10, 14, 18, 22, 26, 30, 34$). The diffraction peaks of samples are well matched with the standard patterns of Sc$_2$W$_3$O$_{12}$ (PDF#89-4690), demonstrating the successful synthesis of Sc$_2$W$_3$O$_{12}$ pure phase.$^{22}$ Fig. 1(b) depicts the schematic of the crystal structure of Sc$_2$W$_3$O$_{12}$, consisting of tetrahedral WO$_4$ and octahedral ScO$_6$ units connected by oxygen. The Rietveld refinement results indicate the orthorhombic structure with space group of Pnca (No. 60) of Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ phosphors at room temperature, as shown in Figs. 1(c) and S1. According to the refinement, we calculated the lattice parameters and corresponding volumes of various Eu$^{3+}$-doped Sc$_2$W$_3$O$_{12}$ phosphors and presented in Figs. 1(d) and S2, respectively. Obviously, the volume of the crystal gradually increases with increasing the doping concentration of Eu$^{3+}$ ions. This can be attributed to that Eu$^{3+}$ (0.95 Å) with larger radius occupies the position of Sc$^{3+}$ (0.885 Å).$^{23}$
Fig. 1 (a) the XRD pattern of Sc$_2$W$_3$O$_{12}$: $x$ mol\% Eu$^{3+}$ ($x = 6, 10, 14, 18, 22, 26, 30, 34$) at room temperature. (b) Schematic of the crystal structure of orthorhombic Sc$_2$W$_3$O$_{12}$ and Eu$^{3+}$ ions substitution diagram. (c) Results of the Rietveld refinement of the XRD pattern of Sc$_2$W$_3$O$_{12}$: 6 mol\% Eu$^{3+}$ phosphors. (d) Cell volume of Sc$_2$W$_3$O$_{12}$ with various Eu$^{3+}$ doping concentration.

The morphologies are recorded by SEM images in Fig. 2(a). The average size of Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ particles is 1 $\mu$m to 5 $\mu$m. Fig. 2(c-f) shows the element mappings of Sc, W, O and Eu$^{3+}$ in Sc$_2$W$_3$O$_{12}$:22 mol\% Eu$^{3+}$ phosphors snatched in Fig. 2(b), respectively. It can be observed that the elements are uniformly distributed in Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ phosphors.

Fig. 2 (a) and (b) are SEM images of Sc$_2$W$_3$O$_{12}$:22 mol\% Eu$^{3+}$ phosphors. (c-f) The element
3.2 Photoluminescence (PL) properties at room temperature

In order to study the luminescence properties of Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ phosphors, we measured the PL excitation (PLE) spectra of samples with different doping concentration of Eu$^{3+}$ ions at room temperature. The measurements were monitored at 613 nm emission, and the results are presented in Fig. 3(a). In general, the excitation spectrum of samples can be mainly divided into two parts. The broadband in the ultraviolet region (200-350 nm) derive from the charge transfer band (CTB) of tungstate ions.$^{19,20}$ And the other sharp weak peaks at 318 nm, 360 nm, 384 nm, 393 nm and 464 nm are owing to transitions$^7F_0 \rightarrow ^5D_4$, $^7F_0 \rightarrow ^5L_7$, $^7F_0 \rightarrow ^5L_6$, $^7F_0 \rightarrow ^5D_3$ and $^7F_0 \rightarrow ^5D_2$ of Eu$^{3+}$ ions, respectively. Fig. 3(b) illustrates the emission peaks of samples with different Eu$^{3+}$ concentrations under 264 nm excitation. And the peaks located at 592 nm, 613 nm and 653 nm result from the $^5D_0 \rightarrow ^7F_1$, $^5D_0 \rightarrow ^7F_2$ and $^5D_0 \rightarrow ^7F_3$ transitions of Eu$^{3+}$ ions respectively. With increasing Eu$^{3+}$ concentration, the emission intensities initially increase and reach a maximum when $x = 22$, and then decrease due to the concentration quenching effect. The emission intensities of 592 nm and 613 nm are normalized to the emission of Sc$_2$W$_3$O$_{12}$: 22 mol% Eu$^{3+}$ phosphors, and their variation with Eu$^{3+}$ concentrations is depicted in Fig. 3(c). It can be observed that the emission intensity of 592 nm varying with Eu$^{3+}$ concentration is basically consistent with that of 613 nm. To explore the interaction type of the activators in Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ phosphors, Dexter theory is employed in Fig. 3(d)$^{24-26}$.

$$\ln\left(\frac{I}{x}\right) = A - \frac{\theta}{3} \ln(x)$$

where $I$ is the integral fluorescence intensity, $x$ represents the doping concentration of Eu$^{3+}$, $A$ is a constant, and $\theta$ refers to the eigenvalue related to the interaction type ($\theta = 3, 6, 8, 10$ corresponding to Exchange, dipole-dipole, dipole-quadrupole, quadrupole-quadrupole interactions, respectively)$^{26}$. According to the fitting result, the $\theta$ value is about 3.0199, suggesting concentration quenching of activators in Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ phosphors derived from exchange interaction.
3.3 Temperature-dependent PL performances

Taking the Sc$_2$W$_3$O$_{12}$: 6 mol% Eu$^{3+}$ sample as an example to demonstrate the negative thermal expansion behavior of the Sc$_2$W$_3$O$_{12}$ matrix. The Rietveld refinement results (Fig. S2) of variable temperature XRD patterns presented in Fig. 4(a) verify the NTE of sample, and the refined lattice parameters as well as the cell volume values are illustrated in Fig. 4(b) and (c), respectively. Notably, the in-situ XRD patterns demonstrate the stable orthorhombic phase of Sc$_2$W$_3$O$_{12}$ at variable temperature. And the lattice of the sample shrinks in the direction of a-axis and c-axis, and expands slightly in the direction of b-axis from room temperature to 573 K. Consequently, the cell volume expands until 373 K and further contracts with rising temperature, which indicates the NTE of Sc$_2$W$_3$O$_{12}$: 6 mol% Eu$^{3+}$ phosphors ranging from 373 K to 573 K. As is known to all, changes in the crystal structure of materials will inevitably affect its luminescent properties. The temperature dependent emission spectra of Sc$_2$W$_3$O$_{12}$: 6
mol% Eu\(^{3+}\) phosphors under the excitation at 264 nm is delineated in Fig. 4(d). Fig. 4(e) shows that the emission intensity at 613 nm maintains 86.61% of initial intensity at 373 K, then abnormally rapidly enhanced with increasing temperatures up to 548 K and the highest emission intensity reaches at 147.81% of initial intensity at 473 K. Moreover, the emissions at 592 nm present similar trends with 613 nm. It is noteworthy that the attenuate luminescence intensity from RT to 373 K can be ascribed to the thermal quenching, and the enhanced luminescence is due to the NTE effect from 373 to 548 K. In general, the bridging oxygen of Sc-O-W undergo transverse vibrations, leading to the coupled tilting of framework polyhedra.\(^{22}\) As a result, the crystal structure of Sc\(_2\)W\(_3\)O\(_{12}\): Eu\(^{3+}\) phosphors grows denser and the energy collection of activator is promoted, demonstrating the stronger thermal quenching resistance of luminescence.\(^{18, 21, 27}\) In addition, we measured the PL spectra of Sc\(_2\)W\(_3\)O\(_{12}\): x mol% Eu\(^{3+}\) (x=10, 14, 18, 22, 26, 30) phosphors (Fig. S4). As shown in Fig. 4(f), it is obvious that the thermal quenching resistance of Sc\(_2\)W\(_3\)O\(_{12}\): Eu\(^{3+}\) decreases with the increase of Eu\(^{3+}\) concentration at 298-548 K. At low Eu\(^{3+}\) concentration (6-10 mol%), the PL intensity of the sample can maintain about 85% of initial intensity (in the green dashed box of Fig. 4(f)). However, at medium Eu\(^{3+}\) concentration (14-18 mol%, in the orange dashed box) and high doping concentration (26-30 mol%, in the blue dashed box), the PL intensity of the sample can be reduced to about 80% and 67% of initial intensity, respectively. On the other hand, the onset temperature at which luminescence enhancement begins to appear also tends to increase with the increase of Eu\(^{3+}\) doping concentration.
Fig. 4 (a) Temperature dependent XRD patterns of Sc$_2$W$_3$O$_{12}$: 6 mol% Eu$^{3+}$ phosphors. (b) The lattice constants and (c) cell volume values of sample changing with temperature. (d) Temperature dependent PL spectra of Sc$_2$W$_3$O$_{12}$: 6 mol% Eu$^{3+}$ with 264 nm excitation. (e) Relative intensity at 592 nm and 613 nm emissions of Sc$_2$W$_3$O$_{12}$: 6 at% Eu$^{3+}$ varying with temperature. (f) Relative Intensity at 613 nm of Sc$_2$W$_3$O$_{12}$: x mol% Eu$^{3+}$ (x=6, 10, 14, 18, 22, 26, 30) varying with temperature.

To investigate the temperature sensing properties of Sc$_2$W$_3$O$_{12}$: x mol% Eu$^{3+}$ phosphors, the TCELs emissions at 592 nm and 613 nm are contributed to FIR technique. The FIR value R is defined as $R = \frac{I_{592\text{ nm}}}{I_{613\text{ nm}}}$, and depicted in Fig. 5(a) and Fig. S5. After fitted by a single-exponential function, the calculation of absolute sensitivity and relative sensitivity are executed. Generally, these two indicators are employed to evaluate the temperature sensing properties of luminescence materials. They can be estimated by equations $^{30, 31}$

$$S_a = \frac{dR(T)}{dT}$$
$$S_r = \frac{1}{R} \frac{dR(T)}{dT}$$

The calculated sensitivities are shown in Fig. 5(b) and Fig. S5. Comparing the maximum $S_a$ and $S_r$ values of samples under different Eu$^{3+}$ concentrations in Fig. 5(c), we can see that the sensitivity of the sample decreases with the increase of Eu$^{3+}$ concentration. When the concentration reaches 22%, the maximum sensitivity begins
to increase again. Among all samples, the sensitivity of the sample with 6 mol% Eu$^{3+}$ is the highest, and its maximum $S_a$ and $S_r$ values can reach $6.872 \times 10^{-3}$ K$^{-1}$ and 3.063% K$^{-1}$ at 298 K, respectively. In addition, the $S_a$ and $S_r$ values maintain the downward trends with increasing temperature, which implies possible preferable temperature sensing properties in low-temperature region.

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**Fig.5** Temperature dependent (a) PL intensity ratio of 593 nm and 613 nm with fitting results and (b) absolute sensitivity ($S_a$) and relative sensitivity ($S_r$) values of Sc$_2$W$_3$O$_{12}$: 22 mol% Eu$^{3+}$ phosphors. The maximum $S_a$ and $S_r$ values varying with doping concentration.

### 4. Conclusion

In summary, we synthesized negative thermal expansion Sc$_2$W$_3$O$_{12}$:Eu$^{3+}$ phosphors by conventional solid-state method. XRD results reveal that the pure orthorhombic phase can be maintained when doping with various concentration of Eu$^{3+}$ ions or elevating temperature. Furthermore, the SEM images and elemental mappings confirm the uniform distribution of Sc, W, O and Eu ions in samples. Under 264 nm irradiation, the bright red emission can be observed and the quenching concentration of Eu$^{3+}$ ions is 22 mol% due to the exchange interaction. When increasing temperature from 298 K, the bridging oxygen of Sc-O-W undergo transverse vibrations, leading to the coupled tilting of framework polyhedra. And the lattices of Sc$_2$W$_3$O$_{12}$:Eu$^{3+}$ phosphors expand until 373 K and then contract up to 548 K. The emission intensity present abnormally rapidly enhanced with increasing temperatures derived from the enhanced energy collection of activators by lattice shrinkage and deformation. The ability of anti-thermal quenching decreases with increasing Eu$^{3+}$ doping concentration. In addition, resulting from the different responses of emissions at 592 nm ($^4D_0\rightarrow^7F_1$)
and 613 nm ($^5D_0\rightarrow^7F_2$) to temperature, Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ phosphors can be used as optical thermometers based on FIR technique. The estimated maximum $S_a$ and $S_r$ values of Sc$_2$W$_3$O$_{12}$: 6 mol% Eu$^{3+}$ phosphors are $6.872\times10^{-3}$ K$^{-1}$ and 3.063% K$^{-1}$ at 298 K, respectively. These results demonstrate that the Sc$_2$W$_3$O$_{12}$: Eu$^{3+}$ phosphors can be applies as optical thermometers due to the good temperature sensing properties and thermal enhanced luminescence performance.

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Supporting Information

Thermally enhanced photoluminescence and temperature sensing properties of Sc$_2$W$_3$O$_{12}$:Eu$^{3+}$ phosphors

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Fig S1 (a-h) Results of the Rietveld refinement of the XRD pattern of Sc$_2$W$_3$O$_{12} : x$ mol\% Eu$^{3+}$ ($x =$ 6, 10, 14, 18, 22, 26, 30, 34 ) at RT.
Fig. S2 Lattice parameters of Sc$_2$W$_3$O$_{12}$ changes with the doping concentration of Eu$^{3+}$ ions.
Fig. S3 Results of the Rietveld refinement of the XRD pattern of Sc$_2$W$_3$O$_{12}$ : 6 mol% Eu$^{3+}$ at different temperatures.
Fig. S4 (a-f) Temperature dependent XRD patterns of Sc$_2$W$_3$O$_{12}$: x at% Eu$^{3+}$ (x=10, 14, 18, 22, 26, 30) phosphors.
Fig. S5 Temperature dependent PL intensity ratio of 593nm and 613nm with fitting results and sensitivity values of Sc$_2$W$_x$O$_{12}$: $x$ mol% Eu$^{3+}$ ($x=6, 14, 18, 26, 30$).