Tunable Yellow-Red Emission via Energy Transfer in Dy^{3+} , Mn^{4+} : NaLaMgWO_6 Phosphors for UV-Excited LED

yonghua wu (✉ wuyonghua@fjixu.edu.cn)

fujian Jiangxia University <https://orcid.org/0000-0002-5203-0573>

Fugui Yang

Jiangxia University

Fengpo Yan

Jiangxia University

Ruijuan Zuo

Fujian Normal University

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Abstract

The Dy^{3+} , Mn^{4+} : NaLaMgWO_6 phosphors are synthesized successfully using the technique of solid-state reaction at 1150 °C. The XRD and SEM-EDS are measured to analyze the phase purity. The concentrations of Dy^{3+} , Mn^{4+} , Mg^{2+} , Na^+ , La^{3+} and W^{6+} are measured by ICP. The absorption and excited spectra are presented, which indicate the 390 nm is suitable for the tunable yellow-red light excitation. Using the 390 nm LED as the excitation source, the emission spectra with different doped concentrations ratios of Dy^{3+} to Mn^{4+} are obtained, which shows good tunable yellow-red emission performance.

1. Introduction

As well known, the LED technology has been applied in many fields such as display, lighting, medical, health, transportation, etc. And the commercial LED sources can provide almost all wavelengths in the visible bands from 360–650 nm and the power of per-chip is up to Watt level, which indicates the LED can act as the excitation source like Xe-lamp to generate the needed green-yellow-red emission. Namely, one economical and compact white LED solution might be that using LED source to excite phosphor to generate white light.

In the phosphor family, the rare-earth phosphors occupy the vast majority such as $\text{YAG}:\text{Ce}^{3+}$ [1–2], $\text{SrAl}_{12}\text{O}_{19}:\text{Eu}^{2+}$ [3]...Among them, the Dy^{3+} has been widely used to generate the blue, yellow and red light, corresponding to the transition of $^4\text{F}_{9/2} \rightarrow ^6\text{H}_{15/2}$, $^6\text{H}_{13/2}$, $^6\text{H}_{11/2}$ [4–5] because of its plentiful absorption and emission spectra.. Whereas, the emission intensity of red band of Dy^{3+} ions is weak, which cannot contribute to the warm-white LED enough. And so, for the warm white LED application, the Dy^{3+} needs the help of other ions.

The Mn^{4+} activated phosphors have been widely investigated recently because of the good far-red emission bands of 650–735 nm [6–7]. The $3d^3$ electronic structures make the absorption band is very broad and the emission band is narrow due to the octahedral crystal field. Particularly, in the Mn^{4+} : NaLaMgWO_6 , the Mn^{4+} puts up excellent efficiency, which is up to 60% [8]. The host of NaLaMgWO_6 , a novel double perovskite tungstate compound, has attracted extensive attention for the applications in white LED. The Er^{3+} , Sm^{3+} : NaLaMgWO_6 [9], Eu^{3+} : NaLaMgWO_6 [10], Yb^{3+} , Er^{3+} : NaLaMgWO_6 [11], Dy^{3+} , Tm^{3+} : NaLaMgWO_6 [12]...indicate the host of NaLaMgWO_6 is suitable for the dopant of rare earth or transition ions.

Considering of the character of Dy^{3+} and Mn^{4+} , in the work, we attempt to investigate the fluorescence performance of Dy^{3+} , Mn^{4+} : NaLaMgWO_6 phosphors and discuss the mechanism of tunable yellow-red emission. Using the host of NaLaMgWO_6 , the Dy^{3+} (0.091 nm) can easily replace the La^{3+} (0.106 nm) due to the similar electronic structure (4f), and the Mn^{4+} (0.054 nm) can also occupy the site of W^{6+} (0.041 nm). More important, some of the absorption peaks of Dy^{3+} and Mn^{4+} are overlapped, which make the

excitation source is single and simple. And so, we can expect the Dy^{3+} , Mn^{4+} : NaLaMgWO_6 phosphors have good fluorescence performance.

2. Experimental

2.1 Synthesis of Dy^{3+} , Mn^{4+} : NaLaMgWO_6 phosphors

The $x\text{Dy}^{3+}$, $y\text{Mn}^{4+}$: NaLaMgWO_6 ($x = 0.5-10$ at%; $y = 0.5$ at%) phosphors were synthesized by a solid state reaction method[13–17]. The raw materials are Na_2CO_3 (99.99 %, 0.5 μm), MgO (99.99 %, 1 μm), La_2O_3 (99.99 %, 1 μm), Dy_2O_3 (99.99 %, 0.5 μm), MnO_2 (99.99 %, 1 μm), WO_3 (99.99 %, 0.5 μm). The reaction equation is as follows:

The raw materials were mixed homogeneous by an agate mortar and placed in an Al_2O_3 crucible. The mixture was sintered in a tube furnace at $1150 \pm (5)^\circ\text{C}$ for 4 h in air. Finally, the samples were cooled down to room temperature.

2.2 Characterization

The phase purity was identified by Rigaku Miniflex 600 XRD analysis ($\text{Cu-K}\alpha$, $\lambda = 0.1540$ nm, $5^\circ\text{C}-80^\circ\text{C}$). The concentrations of Na^+ , La^{3+} , Dy^{3+} , Mn^{4+} , W^{6+} , Mg^{2+} ions were measured by inductively coupled plasma atomic emission spectrometry method (ICP-AES instrument, Agilent ICPOES730). The micro-structure was measured by SEM-EDS (Energy Dispersive Spectrometer). The absorption spectra were recorded by Shimadzu UV-2600 UV-VIS-NIR spectrophotometer. The excitation was measured by Edinburgh Instruments FLS980 fluorescence spectrometer. Excited with 390 nm LED, the emission spectra were measured by USB2000 + spectrometer (Ocean Optics Ltd.).

3. Results And Discussion

3.1 The micro-crystal structure and morphology of Dy^{3+} , Mn^{4+} : NaLaMgWO_6 phosphor

The NaLaMgWO_6 , monoclinic with space group of $\text{P}2_1$ (4), and $a = 0.55239$ nm, $b = 0.55253$ nm, $c = 0.78939$ nm (JCPDS # 88-1761). Figure 1 shows the comparison of X-ray diffraction pattern between the 10% Dy^{3+} , 0.5% Mn^{4+} : NaLaMgWO_6 and the standard NaLaMgWO_6 .

In the figure, the XRD pattern of 10% Dy^{3+} , 0.5% Mn^{4+} : NaLaMgWO_6 is identical well with the standard pattern of JCPDS # 88-1761. The concentration of Dy^{3+} is up to 10 at %, the highest doped concentration of the samples. From the figure, we can conclude that all of the samples $x\text{Dy}^{3+}, y\text{Mn}^{4+}$: NaLaMgWO_6

($x=0.5-10\text{at\%}$, $y=0.5\text{at\%}$) will keep single phase and the doping process of Dy^{3+} , Mn^{4+} never destroy the single phase structure of pure NaLaMgWO_6 .

For the purpose to investigating the micro-structure deep, the line-scanning SEM –EDS was measured, shown in Figure 2 . The line-EDS spectra were operated with the maximum length of $40\text{ }\mu\text{m}$ and maximum voltage of 15 KeV . In Fig2.(a), the total length is $40\text{ }\mu\text{m}$ and the diameter of cell particles is $0.5-1\text{ }\mu\text{m}$. The Fig.2 (b) shows the counts per-second vs scanning energy. From the figure, in general, the sum counts keep nearly uniform with the scanning energy of $0-10\text{ KeV}$. The distributions of Dy^{3+} and Mn^{4+} always appear simultaneously, which are mainly distributed in $1-3\text{ KeV}$ and $7-8\text{ KeV}$. According to the data, we can conclude that the Dy^{3+} and Mn^{4+} evenly replace the sites of NaLaMgWO_6 and the micro-structure keeps periodicity and integrity.

The concentrations of Na^+ , Mg^{2+} , La^{3+} , Dy^{3+} , Mn^{4+} , W^{6+} ions were measured by ICP (the sample is $10\text{at\%Dy}^{3+}, 0.5\text{at\%Mn}^{4+}$: NaLaMgWO_6). The used mass is 0.0557 g and the constant volume is 50 ml . The 20 ml aqua regia was added in the samples, and then heated them to $180\text{ }^\circ\text{C}$ to chemical reaction. After complete reaction, the solution was cooled down and prepared to test. The measured amount of Na^+ , Mg^{2+} , La^{3+} , Dy^{3+} , Mn^{4+} , W^{6+} were shown in the following (table 1). The molecular weight of $10\text{at\%Dy}^{3+}, 0.5\text{at\%Mn}^{4+}$: NaLaMgWO_6 is 466.7 g/mol ($1\text{Kg}-2.1427\text{mol}$). The ICP measurement has the process of liquid to solid, namely recrystallization process, and then the segregation coefficient can be used to estimate the doped complexity. From the table 1, we can clearly find that all of the segregation coefficients are near 1, which implies the doped process is very easy, also represents 100% replacement, no waste of the involved ions, and significantly reduces the defect structure caused by doping. The actual concentration ratio of Dy^{3+} to Mn^{4+} is $0.11:0.005=22$, near the preparation ratio of 20.

Table 1: the measured concentrations of $10\text{at\%Dy}^{3+}$, $0.5\text{at\%Mn}^{4+}$: NaLaMgWO_6 sample.

ion	Dy^{3+}	Mn^{4+}	Na^+	La^{3+}	Mg^{2+}	W^{6+}
Preparation concentration (mol)	0.10	0.005	1	0.90	1.005	0.995
Measured value (mg/Kg)	39769.0	810.2	49281.1	272269.7	52266.6	400637.8
Actual concentration (mol)	0.110	0.005	1.000	0.914	1.003	1.017
Segregation coefficient	1.1	1.0	1.0	1.01	0.998	1.02

3.2 The absorption, excitation and emission spectra of Dy^{3+} , Mn^{4+} : NaLaMgWO_6 phosphors

The absorption spectrum of the $x\text{Dy}^{3+}, y\text{Mn}^{4+}$: NaLaMgWO_6 ($x=5\text{ at\%}$, $y=0.5\text{ at\%}$) phosphors is shown in Fig.3 from $300-700\text{ nm}$. In the figure, there are many absorption peaks observed, which are centered at 315 nm (Dy^{3+}), $320-323\text{ nm}$ (Dy^{3+} , Mn^{4+}), $336-339\text{ nm}$ (Dy^{3+} , Mn^{4+}), 347 nm (Dy^{3+}), $360-363\text{ nm}$ (Dy^{3+} , Mn^{4+}), $370-373\text{ nm}$ (Dy^{3+} , Mn^{4+}), $389-390\text{ nm}$ (Dy^{3+} , Mn^{4+}), 465 nm (Dy^{3+} , Mn^{4+}), $475-515\text{ nm}$ (Mn^{4+}). Many peaks of Dy^{3+} and Mn^{4+} are overlapped. The absorption spectrum of Mn^{4+} is always broad, but the Dy^{3+} has relatively sharp absorption

peaks. Under 575 nm emissions, the excitation spectrum was also measured, shown in the right upper of Fig.3, where six peaks of Dy³⁺ centered at 352 nm, 365 nm, 390 nm, 427 nm, 454 nm and 476 nm were observed. Three strong excitation intensities are found in 352 nm, 390 nm and 454 nm. Compared with the absorption spectrum, the 390 nm is more suitable than 352 nm and 454 nm.

Using the 390 nm LED as the excitation source, the emission spectra are shown in Fig.4. In Fig.4, Three emission peaks centered at 575 nm (⁴F_{9/2}→⁶H_{13/2} (Dy³⁺)), 698 nm and 712 nm (²E_g→⁴A_{2g}, ²T₁→⁴A_{2g} (Mn⁴⁺)) are observed. With the increasing doped concentration of Dy³⁺, the emission intensity of 575 nm becomes stronger. When the ratio of Dy³⁺to Mn⁴⁺ is up to 5at%:0.5at%=10:1, the peak intensity of 575 nm is equal to that of 698 nm. When the ratio of Dy³⁺to Mn⁴⁺ is 10at%:0.5at%=20:1, the peak intensity of 575 nm is near double to 698 nm (15300:7950=1.92), but the fluorescence integral intensity is only half of the 698-712 nm (670825 a.u*nm). And so, we can obtain the tunable yellow-red emission with the concentration ratio of Dy³⁺to Mn⁴⁺, which contributes to the broad application field such as display, health and plant growth [18].

3.3 The influence of Dy³⁺ concentrations on the Correlated Color Temperature (CCT)

For the purpose of investigating the color temperature of LED, we calculated the total relative spectrum energy by using the following equations, which are stated in CIE 1931 and CIE 1964 [19]. Tristimulus values X (R=700 nm), Y (G=546.1 nm), Z (B=435.8 m) are defined:

$$\begin{aligned} X &= k \int_{380}^{780} P(\lambda) x(\lambda) d\lambda, \\ Y &= k \int_{380}^{780} P(\lambda) y(\lambda) d\lambda, \\ Z &= k \int_{380}^{780} P(\lambda) z(\lambda) d\lambda, \end{aligned}$$

The P(λ) is the relative spectrum energy distribution and x, y, z are the three basic colors. The λ is the color light wavelength and the k is the normalized coefficient. And so, the CIE coordination (x, y) can be obtained:

$$x=X/(X+Y+Z); y=Y/(X+Y+Z)$$

According to the emission spectra shown in Fig. 4, we can get the CIE coordination (x, y) with the different ratio of Dy³⁺ to Mn⁴⁺, shown in Table 2.

Table 2 The CIE coordination and the Correlated Color Temperature (CCT) of xDy³⁺, 0.5at%Mn⁴⁺: NaLaMgWO₆ (x=0.5at%-10 at %) phosphors excited with 390 nm LED

sample	0.5at%Dy ³⁺	2at%Dy ³⁺	4at%Dy ³⁺	5at%Dy ³⁺	10at%Dy ³⁺
CIE coordination	0.403,0.346	0.401,0.350	0.400,0.362	0.402,0.370	0.405,0.385

Excitation with 390 nm, the white light performance has been investigated. The white light photos and the Correlated Color Temperature (CCT) also have been calculated. The Fig.5 shows the CIE coordination and fluorescence photos of $x\text{Dy}^{3+}$, $0.5\text{at}\%\text{Mn}^{4+}$: NaLaMgWO_6 ($x=0.5\text{at}\%-10\text{ at } \%$) phosphors excited with 390 nm LED source. Results indicate that the Correlated Color Temperature (CCT) is in the region of 3000K-4000K, which shows it's the warm white-LED and can be useful for many applications.

3.4 The photoluminescence mechanism of $x\text{Dy}^{3+}$, $0.5\text{at}\%\text{Mn}^{4+}$: NaLaMgWO_6 ($x=0.5\text{at}\%-10\text{ at } \%$) phosphors[20-25]

The emission spectra indicate that the Dy^{3+} , Mn^{4+} co-doped in NaLaMgWO_6 have meaningful influence on the tunable yellow-red LED. The energy levels of Dy^{3+} , Mn^{4+} are presented in Fig.6. Excited with 390 nm, the electrons are inspired to $^4\text{M}_{21/2}$ (Dy^{3+}), and then transit from the $^4\text{M}_{21/2}$ (Dy^{3+}) to $^4\text{G}_{11/2}$ (Dy^{3+}), $^4\text{I}_{15/2}$ (Dy^{3+}), $^4\text{F}_{9/2}$ (Dy^{3+}) by the non-radiation transition. Finally, the electrons will transit from $^4\text{F}_{9/2}$ (Dy^{3+}) to $^6\text{H}_{13/2}$ (Dy^{3+}) to generate the 575 nm yellow light. Similarly, about the Mn^{4+} ions, the electrons will excited from $^4\text{A}_{2g}$ to $^4\text{T}_1$, and then transit from $^4\text{T}_1$ to $^4\text{T}_{2g}$, $^2\text{E}_g$. The red emission 698-712 nm can be obtained corresponding to the transition of $^2\text{E}_g \rightarrow ^4\text{A}_{2g}$. The energy level $^4\text{M}_{21/2}$ (Dy^{3+}) is close to the $^4\text{T}_1$ (Mn^{4+}), which make the energy level band become the share poll. The energy level $^4\text{F}_{9/2}$ (Dy^{3+}) is higher than the $^4\text{T}_{2g}$ (Mn^{4+}), which make some of the electrons of Dy^{3+} transit from the $^4\text{F}_{9/2}$ (Dy^{3+}) to $^4\text{T}_{2g}$ (Mn^{4+}) by the cross relaxation. Fortunately, the doped concentration of Dy^{3+} can be up to 10at% or more, which can guarantee the emission intensity of 575 nm enough.

4. Conclusions

In the work, the $x\text{Dy}^{3+}$, $0.5\text{at}\%\text{Mn}^{4+}$: NaLaMgWO_6 ($x=0.5\text{at}\%-10\text{ at } \%$) phosphors were successfully synthesized with the solid state reaction method. The XRD pattern is measured, which indicates the pattern is well identical with the standard JCPDS # 88-1761. The line-scanning SEM-EDS is measured to check the homogeneity and result indicates that the doped ions of Dy^{3+} and Mn^{4+} are evenly distributed in the micro-crystal. The absorption and excitation spectra are presented, which indicates the 390 nm suitable for the excitation source. Excited with 390 nm LED, the emission spectra are measured with different concentrations ratio of Dy^{3+} to Mn^{4+} . Results indicate that the 575 nm yellow emission can be tunable with different concentrations of Dy^{3+} . And then, the 390 nm, 575 nm and 689-712 nm can be mixed to near white light. The CIE coordinate and CCT are also measured. The CCT is in the region of 3000-4000K, which indicates it's the warm white-LED. The fluorescence mechanism is also presented, which imply that share poll and cross relaxation exist in the $x\text{Dy}^{3+}$, $0.5\text{at}\%\text{Mn}^{4+}$: NaLaMgWO_6 ($x=0.5\text{at}\%-10\text{ at } \%$) phosphors. And the high doped concentration of Dy^{3+} can guarantee the emission intensity of 575 nm enough. In this paper, commercial LED 390nm light source is used for excitation, which makes the products easy to be commercialized, while most other products are fired by Xe-lamp, which is not conducive to commercial applications.

Declarations

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Figures

Figure 1

The powder X-ray diffraction pattern for $\text{Dy}^{3+}, \text{Mn}^{4+}:\text{NaLaMgWO}_6$.

Figure 2

The line-scanning SEM-EDS spectra with length of 40 μm and 15 KeV voltage (Dy^{3+} , Mn^{4+} : NaLaMgWO_6 sample); (a) is the line-SEM photo; (b) is the counts per-second (cps/eV) vs energy (KeV).

Figure 3

The absorption and excitation spectra of Dy^{3+} , Mn^{4+} : NaLaMgWO_6 phosphor; the upper is the excitation spectrum with 575 nm emission.

Figure 4

The emission spectra of $x\text{Dy}^{3+}, 0.5\text{at}\%\text{Mn}^{4+}:\text{NaLaMgWO}_6$ ($x=0.5\text{at}\%-10\text{ at}\%$) phosphors excited with 390 nm LED.

Figure 5

The CIE and fluorescence photos of $x\text{Dy}^{3+}$, 0.5at%Mn $^{4+}$: NaLaMgWO $_6$ ($x=0.5\text{at}\%-10\text{ at}\%$) phosphors excited with 390 nm LED.

Figure 6

Schematic illustration of Dy^{3+} and Mn^{4+} energy levels in $x\text{Dy}^{3+}$, $0.5\text{at}\%\text{Mn}^{4+}$: NaLaMgWO_6 ($x=0.5\text{at}\%-10 \text{ at}\%$) phosphors excited by 390 nm LED.