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Spectroscopic parameters of Ce^{3+} ion doped $Na_2CaMg(PO_4)_2$ phosphor

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ABSTRACT

Ce^{3+}-doped $Na_2CaMg(PO_4)_2$ was prepared by high temperature solid-state reaction. The phosphor was confirmed to be pure crystalline phase of $Na_2CaMg(PO_4)_2$. The spectroscopic properties of the Ce^{3+} ions were investigated by vacuum ultraviolet spectroscopy. The energy transfer from the host to Ce^{3+} ions was demonstrated. The phosphor could be excited by VUV light and showed a broad purplish blue emission with the maximum wavelength at 385 nm. The five 5d levels corresponding to the $4f^1 \rightarrow 4f^0 5d^1$ transition of Ce^{3+} ions were identified. The spectroscopic parameters, e.g., the barycenter, host absorption bands, crystal field splitting and stokes shift were discussed.

1. Introduction

In recent years, rare earth (RE) activated inorganic compounds have attracted the attention due to their potential applications, e.g., flat panel displays [1], long-lasting phosphorescent materials [2], thermoluminescent dosimeters [3], electroluminescence devices [4], white light-emitting diodes [5], scintillators [6] and non-destructive test [7]. For example, the luminescence spectra of Ce^{3+} ion doped in many hosts have been intensively investigated, including silicates [6,8], aluminates [9,10], sulfides [3,11], borates [7,12], phosphates [13], etc.

Phosphates usually show a rather short wavelength of optical absorption edge. This makes them suitable as hosts to accommodate active RE ions. The luminescence of RE-doped phosphates usually has a high thermal stability. As a member of orthophosphates, $Na_2CaMg(PO_4)$ was first described by Fuchs et al. [14]. Moore proposed that $Na_2CaMg(PO_4)_2$ was a phosphate analog of merwinite $Ca_3Mg(SiO_4)_2$ [15]. Alkemper and Fuess [16] reported the structure of $Na_2CaMg(PO_4)_2$ to be related to the arrangement of cations and phosphate tetrahedra in the glaserite structure. It has a monoclinic structure and a space group of $P2_{1/c}$ with lattice constants of $a = 13.36$ Å, $b = 5.23$ Å, $c = 9.13$ Å [16]. However, up to now, there are no reports to investigate the luminescence of RE ions doped in this host. The luminescence and spectroscopic parameters of Ce^{3+}-doped in $Na_2CaMg(PO_4)_2$ under excitation of vacuum ultraviolet (VUV) and ultraviolet (UV) were investigated for the following reasons.

Firstly, the 5d level spectroscopy of Ce^{3+} ion is very simple, which has only one electron in the 4f shell and exhibits strong absorption and efficient fluorescence from the allowed interconfigurational $4f^1 \rightarrow 4f^0 5d^1$ transition. It has been reported that the spectroscopic parameters of Ce^{3+}, e.g., the positions of 5d level, can be used to predict the 5d energies of other lanthanides, since the influence of the crystal field and covalence of the host lattices on the 4f5d levels are approximately equal for all the RE ions [17].

Secondly, efficient energy transfer from the Ce^{3+} ions (as an energy donor) to acceptors (the other RE or transition metal ions) can be realized in many inorganic materials excited by the VUV–UV. For example, energy transfer was confirmed from Ce^{3+} to Tb^{3+} ions in the $(La,Ce)PO_4$:Tb green phosphors used in fluorescent lamps [18], Ce^{3+} to Nd^{3+} in pentaphosphate crystals [19], Ce^{3+} to Mn^{2+} ions in fluoride single crystals [20], Ce^{3+} to Eu^{3+} ions in aluminates used as a red phosphor in light-emitting diodes (LEDs) [21] and Ce^{3+} to Eu^{2+} ions in silicates for the application in plasma display panels (PDP) display under VUV excitation [22], etc. Consequentially, the investigations on the energy transfer from Ce^{3+} to other activators have been attracted much attention for its practical applications in optical devices.

Thirdly, Ce^{3+}-doped materials are especially attractive for numerous applications, such as PDP display. The excitation source of PDP, viz., the discharge of noble gas, is mainly at vacuum ultraviolet region (VUV, wavelength <200 nm). For phosphors with highly efficient luminescence in PDPs, the host lattice or the activators must have strong absorption in the range of 120–180 nm. Usually, the mono-phosphate compounds with PO_4 groups show a strong absorption in the VUV region.

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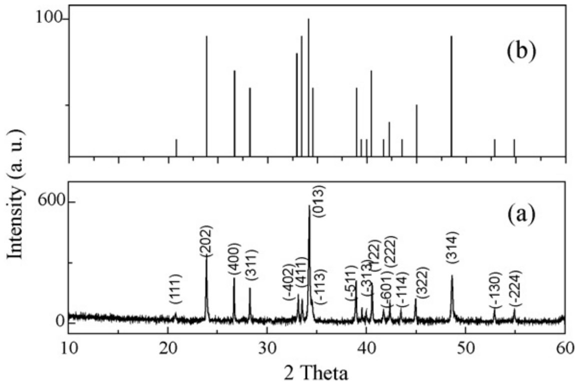

Fig. 1. XRD patterns of $Na_2CaMg(PO_4)_2$:Ce^{3+} (a) and JCPDs 29-1192 card (b).

In this study, Ce^{3+}-doped $Na_2CaMg(PO_4)_2$ was prepared by high temperature solid-state reaction. Its structure was studied by XRD measurement. The luminescence properties of the Ce^{3+} ion were investigated under VUV–UV synchrotron radiation. The spectroscopic parameters such as emission bands, 5d components, stokes shift, barycenter of Ce^{3+} ion doped in $Na_2CaMg(PO_4)_2$ lattice were discussed. So this research can be referential for the luminescence investigation of other RE ions doped in this phosphate.

2. Experimental details

The $Na_2CaMg(PO_4)_2$:Ce^{3+} powders were prepared by the high temperature solid-state reaction. The raw materials were high-purity Na_2CO_3, $CaCO_3$, $4(MgCO_3)\cdot Mg(OH)_2\cdot 5H_2O$ (magnesium carbonate basic pentahydrate) and CeO_2. The doping concentration of CeO_2 was 1.0 mol% of $CaCO_3$. After being weighed, they were mixed and ground in a carnelian mortar. The mixture was firstly heated up to 300 °C and kept at this temperature for 5 h. The obtained powder was thoroughly mixed in acetone and then heated up to 750 °C and kept at this temperature for 10 h in air. After that, the sample was thoroughly mixed and heated in the reductive atmosphere at 950 °C for 10 h.

The room temperature VUV–UV excitation and VUV-excited emission spectra were measured in Beijing Synchrotron Radiation Facility of Institute of High Energy Physics. A 1 m Seya-Namioka monochromator and an Acton Spectra-308 monochromator were used for the measurement of excitation and emission spectra. The signal was detected by a Hamamatsu H5920-01 photon counter. In all measurements, the 280 nm cut-off filter was used to prevent the incident light entering into the secondary monochromator. The pressure of the sample chamber was lower than 1×10^{-3} Pa. The VUV excitation intensities of the samples were corrected by dividing the measured excitation intensity of the sample with that of sodium salicylate measured at the same condition.

3. Results and discussion

Fig. 1 shows the XRD patterns of $Na_2CaMg(PO_4)_2$:Ce^{3+} (a) and the JCPDs card no. 29-1192 (b). It can be found that the sample and the standard card almost have the same position. This shows the orthophosphate structure of $Na_2CaMg(PO_4)_2$ with $P2_{1/c}$ (14) crystal group and $a = 13.3884$ Å, $b = 5.21544$ Å, $c = 9.13609$ Å. These data are in agreement with the reported results for $Na_2CaMg(PO_4)_2$ [16].

$Na_2CaMg(PO_4)_2$ doped with Ce^{3+} ions was chosen for the investigations of energy transfer from the host excitations to the impurity ion. It is well known that the radiative lifetime of the excited state $4f^05d^1$ of Ce^{3+} is much shorter (tens of ns) than that corresponding to forbidden 4f–4f transitions in RE ions resulting in a high emission intensity of Ce^{3+}. The emission spectra of undoped and Ce^{3+}-doped $Na_2CaMg(PO_4)_2$ (1 mol%) excited at 174 nm are shown in Fig. 2. The undoped sample exhibits the luminescence from the host excitations centered at 270 nm (Fig. 2a). The emission of the Ce^{3+}-doped sample is dominated by the $4f^05d^1 \rightarrow 4f^1(^2F_{5/2})$ transition of Ce^{3+} (385 nm), whereas the host excitation emission is quenched.

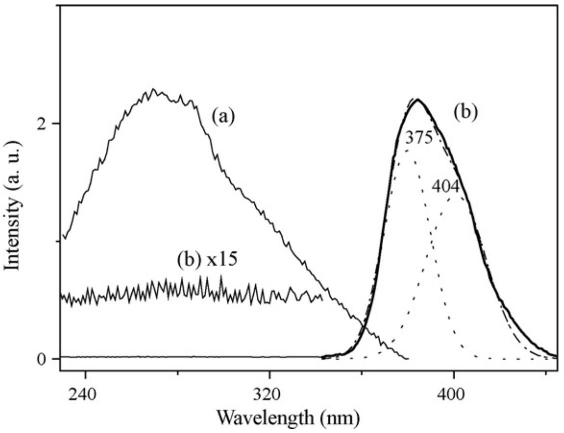

Fig. 2. The emission spectra of undoped $Na_2CaMg(PO_4)_2$ (a) and Ce^{3+}-doped $Na_2CaMg(PO_4)_2$ (b) under the excitation of 147 nm at room temperature. The dot lines are the two decomposed Gaussian components of the curve (b).

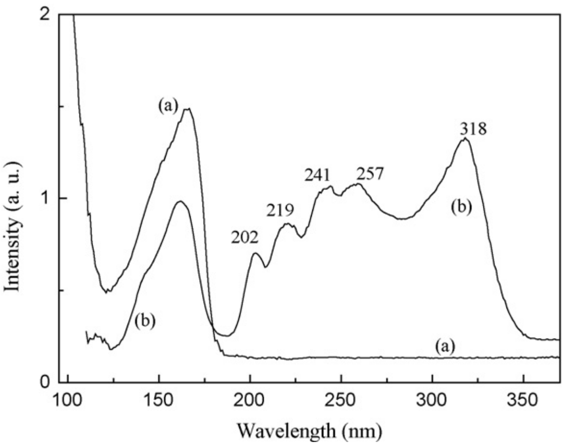

Fig. 3. The room temperature VUV–UV excitation spectra of undoped $Na_2CaMg(PO_4)_2$ (a, by monitoring the host emission at 270 nm) and Ce^{3+}-doped $Na_2CaMg(PO_4)_2$ (b, by monitoring the 5d → 4f emissions at 385 nm of Ce^{3+} ions).

Fig. 3 shows the VUV–UV excitation spectra for the undoped $Na_2CaMg(PO_4)_2$ (a) and Ce^{3+}-doped $Na_2CaMg(PO_4)_2$ (b). There is a broad absorption band between 120 and 180 nm on the excitation spectrum of undoped $Na_2CaMg(PO_4)_2$. This band is from the electronic excitation of $PO_4{}^{3-}$ anions, which is similar to the results observed in other phosphates, fluorophosphates [23–25]. Theoretical calculation has confirmed that the lowest intra-molecular $2t_2 \rightarrow 2a$, $3t_2$ transition energy of the tetrahedral $PO_4{}^{3-}$ molecule is around 7–10 eV (177–124 nm) [26]. The onset of host absorption at about 165 nm implies that the band gap of $Na_2CaMg(PO_4)_2$ hosts is about 7.52 eV, which is similar to that of $REPO_4$ and $A_3RE(PO_4)_2$-type (RE-rare earth, A-alkali metal ions) mono-phosphates [27].

The VUV–UV excitation spectrum of Ce^{3+}-doped $Na_2CaMg(PO_4)_2$ (Fig. 3b) shows two broad absorption bands, i.e., 120–180 and 180–450 nm. Based on the fact that the Ce^{3+} 5d manifold undergoes an energetic depression on the basis of the nephelauxetic effect and a large splitting due to the crystal field. It is expected that the absorption in the wavelength of 180–450 nm are from direct $4f^1(^2F_{5/2}) \rightarrow 4f^05d^1$ absorption of Ce^{3+}. This absorption overlaps the host emission very well (see Fig. 2a).

The quenching of the host emission and the overlapping of the Ce^{3+} excitation spectra on the host absorption band in Ce^{3+}-doped $Na_2CaMg(PO_4)_2$ indicate that the conditions of Forster–Dexter energy transfer are well satisfied [28]. Usually, there are two different mechanisms for energy transfer between sensitizer and

Table 1
The spectroscopic data of Ce^{3+}-doped $Na_2CaMg(PO_4)_2$.

	(nm)	$\times 10^3$ cm^{-1}
First 5d component	318	31.446
Second 5d component	257	38.911
Third 5d component	241	41.494
Fourth 5d component	219	45.662
Fifth 5d component	202	49.505
A_{cfs}		18.059
5d barycenter		41.404
$D(A)$		17.894
Emission bands	375, 404	26.667, 24.753
Stokes shift (ΔS)		4.991
L–S splitting		1.914

Stokes shift (ΔS) is defined as the energy difference between the lowest excitation band and the highest emission band; $D(A)$ is defined as the value of first 5d component relative to the free ion (49,340 cm^{-1}) energy value of Ce^{3+} ions. The crystal field splitting value A_{cfs}, is defined as the energy difference between 5d(1) and 5d(5).

activator: radiative transfer through emission of sensitizer and reabsorption by activator and non-radiative transfer associated with resonance between absorber and emitter. The efficiency of radiative transfer depends on how efficiently the fluorescence of the activator is excited by the emission of the sensitizer. For the non-radiative transfer, electric multiple interactions and those by exchange interactions are important [29]. At present, it is not easy to distinguish between transfer by exchange and electric multiple interactions. However, the probability of total energy transfer is proportional to the energy overlap between the emission of the sensitizer and the absorption of the activator whether transfer is caused by radiative decay, exchange or electric multiple interaction of non-radiative transfer. Thus the energy overlap plays a decisive role in energy transfer from sensitizer to activator [28]. As mentioned above, the emission of undoped $Na_2CaMg(PO_4)_2$ centered at 275 nm (Fig. 2a) well overlaps the 4f–5d absorption band of Ce^{3+} at 200–310 nm (Fig. 3b). Thus, the efficient energy transfer from the host excitations to Ce^{3+} could take place in Ce^{3+}-doped $Na_2CaMg(PO_4)_2$ sample. Since the samples were not optimized for Ce^{3+} concentration, the higher efficiencies may be achieved.

It is well known that the 4f configuration of Ce^{3+} ion has only one electron, and a VUV–UV photon can excite the 4f electron into the 5d orbital, leaving the 4f shell empty. So, the excitation spectrum of Ce^{3+} shows the direct splitting information of 5d orbital in the crystal field. It has been reported that the f–d transition of Ce^{3+} ions will exhibit a subtle structure due to the influence of crystal field splitting and spin–orbit coupling [23]. VUV–UV excitation spectrum can give the information such as 5d levels of Ce^{3+}, host absorption and the energy transfer process from the host to Ce^{3+}. When Ce^{3+} ion is doped in a crystalline host, the mean energy of the 5d configuration shift downward relative to the free ion value and the 5d levels split into five different crystal field components.

In the VUV–UV excitation spectra, the narrow peaks are assigned as 4f–5d transition. In Fig. 3, the five bands corresponding to Ce^{3+} intra-ion 4f → 5d(i) (i = 1, 2, 3, 4, 5) transitions in the range of 190–330 nm show that the crystal field of Ce^{3+} 5d level fully split into five levels 5d(1), 5d(2) and 5d(3) and two higher-lying of 5d(4) and 5d(5) as shown in Table 1.

It is well known that from the lowest 5d energy level of Ce^{3+} in a specific compound, the 5d energy levels of other rare earth ions in the same compound can be predicted well. Dorenbos gathered and analyzed the 4f–5d transition energies of triply ionized lanthanides in various compounds and proposed that the crystal field depression $D(\mathrm{Ln, A})$ of the energy of lowest $4f^{n-1}5d$ levels of a lanthanide ion in compound "A" relative to the level energies in the free ion is almost independent of the type of lanthanide ion [30,31]:

$$D(\mathrm{Ln, A}) = E(\mathrm{Ln, free}) - E(\mathrm{Ln, A}) \qquad (1)$$

where $E(\mathrm{Ln, free})$ is the energy of the first f–d transition of Ln^{3+} (Ce^{3+}) in a free ion and $E(\mathrm{Ln, A})$ the energy of $4f^{n-1}5d$ levels of a lanthanide ion in compound A. The f–d excitation transition of Ce^{3+}, $E(\mathrm{Ce}, Na_2CaMg(PO_4)_2)$, is found at 318 nm (i.e., 31,446 cm^{-1}) and the reported d level energy of free Ce^{3+} is 49,340 cm^{-1} [32,33]. It could be calculated that the f–d transition energy of Ce^{3+} in $Na_2CaMg(PO_4)_2$ ($D(Ce^{3+}, Na_2CaMg(PO_4)_2)$ is depressed by 17,894 cm^{-1}, compared to that of free Ce^{3+} ions.

Furthermore, the approximate positions of Ce^{3+} 5d sublevels, emission bands and stokes shift values are reported in Table 1. The first 5d component of Ce^{3+} in the $Na_2CaMg(PO_4)_2$ is observed at 31,446 cm^{-1}. The gravity centre of the 5d bands of Ce^{3+} is 41,404 cm^{-1}. $D(\mathrm{Ln, A})$ of the Ce^{3+} ions in the $Na_2CaMg(PO_4)_2$ is 17,894 cm^{-1}, which is similar to that of other mono-phosphate, for example, Ce^{3+}-doped $Sr_3(PO_4)_2$ with a $D(\mathrm{Ln, A})$ of 15,210 cm^{-1} [34].

Under VUV excitation at 147 nm, the emission spectrum in Fig. 2 shows a broad band located at about 385 nm. Generally, the emission spectra of Ce^{3+} ions have a doublet character due to the spin–orbit splitting of ground state ($^2F_{5/2}$ and $^2F_{7/2}$). Under the inter-band excitation of 147 nm in $Na_2CaMg(PO_4)_2$:Ce^{3+}, the two 5d(1) → ^2F_J (J = 5/2, 7/2) sub-bands can be well resolved by using the deconvolution method based on their curve shape shown in Fig. 2. The emission curve may contain two peaks at 375 and 404 nm and the Gaussian peak-fitting suggests that the L–S splitting value of Ce^{3+} 4f level is 1914 cm^{-1}, which is a bit smaller than the normal ground state splitting of Ce^{3+} ions (2000 cm^{-1}) [30,31].

$Na_2CaMg(PO_4)_2$ crystallizes in a monoclinic system with the space group of $P2_{1/c}$. The calcium is coordinated octahedrally [16]. Based on the effective ionic radii of cations with different coordination numbers, it could be proposed that Ce^{3+} ions occupy preferably the Ca^{2+} site. The required charge compensations for the occupation of Ce^{3+} ions could be more complicated due to three kinds of cations in the lattices. This can most probably be achieved by the possible mechanism: the positive charge due to Ce^{3+} ion substitute for Ca^{2+} may be combined with the cation vacancy to form the dipole complexes of $[2({Ce_{Ca}}^{3+})^{\bullet} - V''(V''_{mg}, \text{ or } V'_{Na})]$. Such a charge compensation mechanism is very common in other components, e.g., RE^{3+} doped apatite structure phosphate [35]. The possible negative charge compensation related to interstitial oxygen O''_i here is difficult because this mechanism usually occurs in the case of the high RE-doping. And usually, in oxides, the reaction energy to create the O''_i is higher than that of cation vacancy [36]. However, the defect compensation does not exert any obvious influence on the luminescence of Ce^{3+} in this sample because of the low doping level. This is in agreement with the emission spectra of Ce^{3+} ions in Fig. 2, which presents that there is one Ce^{3+} crystallographic site in $Na_2CaMg(PO_4)_2$ lattice.

The lifetime of Ce^{3+} was investigated by luminescence decay curve as shown in Fig. 4. The fluorescence decay curve could be nearly fitted using the exponential equation:

$$y(x) = A\ \exp\left(-\frac{x}{t}\right) \qquad (2)$$

where $y(x)$ is the emission intensity at time x, A is constant and t is lifetime. The lifetime was fitted to be 89 ns.

Until recently, many phosphates doped by lanthanide ions have been reported to the development of new and efficient VUV-excited PDP phosphors, for example, RE-activated lanthanide phosphates $REPO_4$ (RE = La, Y, Gd, Tb) [24,37,38], alkaline–earth metal lanthanide phosphates $M_3RE(PO_4)_3$ (M = Ca, Sr, Ba, RE = La, Y, Gd, Tb) [39], alkaline lanthanide phosphates $AREP_2O_7$ (A = Na, K, Rb, Cs; RE = Y, Lu) [40]. Compared with the presently reported VUV phosphors, RE-doped $Na_2CaMg(PO_4)_2$ phosphors have some advantages such as easy preparation, low preparation cost and high efficiency at the wavelength of the PDP excitation source.

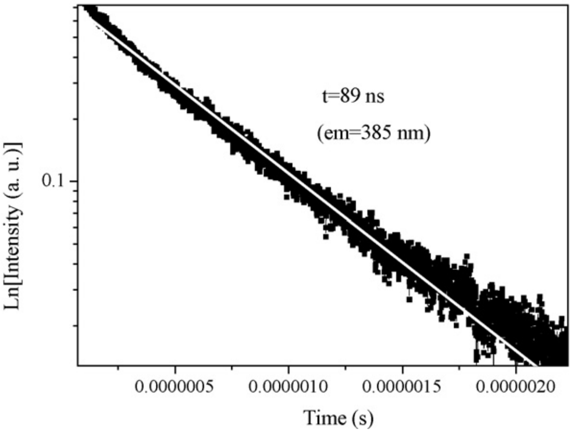

Fig. 4. The decay curve of the luminescence at 385 nm of Ce^{3+}-doped in $Na_2CaMg(PO_4)_2$. The excitation source was the fourth harmonic of a Nd:YAG laser (266 nm).

Phosphors for application in PDP are required to have high conversion efficiency and rich color and pure purity under the VUV radiation. Taking into account of the emission properties (in purplish blue region) of Ce^{3+} ion doped in $Na_2CaMg(PO_4)_2$ and the possible energy transfer from Ce^{3+} ions, high efficient phosphors could be obtained by co-doping Ce^{3+} ions with the other RE ions, for example, co-doping of Tb^{3+}, $Eu^{3+,2+}$, Mn^{2+}, ions in this host to get green, red and deep red phosphors, respectively [18,20,21].

4. Conclusions

The VUV synchrotron radiation spectroscopy technique was used to study the 5d energy levels of Ce^{3+}-doped in $Na_2CaMg(PO_4)_2$ phosphor. The strongest host absorption maximized at 147 nm occurs in the region of 120–180 nm, which is in good coincidence with the PDP excitation source, viz., the discharge of noble gas. Due to the crystal field effect, the Ce^{3+} 5d level splits into five levels of 31,446, 38,911, 41,494, 45,662, 49,505 cm^{-1} for the 5d(1), 5d(2), 5d(3), 5d (4) and 5d(5), respectively. Under the excitation at 147 nm, the $Na_2CaMg(PO_4)_2$:Ce^{3+} shows a typical doublet character on the emission band due to the spin–orbit splitting of ground state ($^2F_{5/2}$ and $^2F_{7/2}$). Potential material for PDP phosphors could be obtained by co-doping Ce^{3+} ions with other RE ions. This is the subject of further studies.

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