New Yellow-Emitting Whitlockite-type Structure
Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$:Eu$^{2+}$ Phosphor for Near-UV Pumped White Light-Emitting Devices

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ABSTRACT: New compound discovery is of interest in the field of inorganic solid-state chemistry. In this work, a whitlockite-type structure Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$ newly found by composition design in the Sr$_3$(PO$_4$)$_2$−Ca$_3$(PO$_4$)$_2$ join was reported. Crystal structure and luminescence properties of Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$:Eu$^{2+}$ were investigated, and the yellow-emitting phosphor was further employed in fabricating near-ultraviolet-pumped white light-emitting diodes (w-LEDs). The structure and crystallographic site occupancy of Eu$^{2+}$ in the host were identified via X-ray powder diffraction refinement using Rietveld method. The Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$:Eu$^{2+}$ phosphors absorb in the UV−vis spectral region of 250−430 nm and exhibit an intense asymmetric broadband emission peaking at 518 nm under $\lambda_{ex} = 365$ nm which is ascribed to the 5d−4f allowed transition of Eu$^{2+}$. The luminescence properties and mechanism are also investigated as a function of Eu$^{2+}$ concentration. A white LED device which is obtained by combining a 370 nm UV chip with commercial blue phosphor and the present yellow phosphor has been fabricated and exhibit good application properties.

1. INTRODUCTION

White light emitting diodes (w-LEDs) are regarded as the next generation lighting source due to its outstanding merits of high energy efficiency, long lifetime, and environmental friendliness, in comparison to the conventional incandescent or fluorescence lamps. In recent decades, solid-state lighting which relies on combination of phosphors and indium gallium nitride (InGaN) chip has attracted significant attention; red, green, and blue (RGB) phosphors play a key role in fabricating phosphor-converted (pc) w-LEDs devices. Currently, the mostly used pc w-LEDs are produced by combining a blue LED chip with Y$_3$Al$_5$O$_{12}$:Ce$^{3+}$ (YAG:Ce) yellow phosphor. In addition to the famous YAG:Ce, the Eu$^{2+}$/Ce$^{3+}$ doped yellow emitting phosphors have also been developed in the host systems including aluminates (La$_2$Sr$_{6}$Al$_{12}$O$_{25}$.Ce$^{3+}$)$_6$, silicates (Sr$_2$SiO$_4$:Eu$^{2+}$)$_2$, phosphates (Ba$_2$Mg(PO$_4$)$_2$:Eu$^{2+}$)$_8$, borates (Ba$_2$Gd(BO$_3$)$_2$:Cl:Eu$^{2+}$)$_9$, and nitriles (Ca$\alpha$-SiAlON: Eu$^{2+}$)$_{10}$. The effort to develop suitable phosphor materials for application in w-LEDs is still ongoing.

Orthophosphate doped with rare earth ions is widely explored as RGB phosphors owing to advantages of relatively low sintering temperature, good chemical stability, and satisfactory absorption in the ultraviolet (UV)−near UV region. For example, the end members and some solid solution compositions in the Sr$_3$(PO$_4$)$_2$−Ca$_3$(PO$_4$)$_2$ join have been reported to be hosts for Eu$^{2+}$/Ce$^{3+}$ activators. Both Sr$_3$(PO$_4$)$_2$ and Ca$_3$(PO$_4$)$_2$ are known to exhibit polymorphism on heating and cooling. Sarver et al. reported the polymorphic nature of Sr$_3$(PO$_4$)$_2$: the $\alpha$ phase undergoes a rapid, reversible transition to the $\beta$ modification at 1305 °C. The $\alpha$ phase crystallizes with monoclinic unit cell, and the $\beta$ polymorph forms in a whitlockite-type structure of rhombohedral unit cell. As for Ca$_3$(PO$_4$)$_2$, $\alpha$-Ca$_3$(PO$_4$)$_2$ stable at room temperature (RT). $\alpha$-Ca$_3$(PO$_4$)$_2$, stable between 1135 and 1430 °C, is quenchable to RT, whereas $\alpha'$-Ca$_3$(PO$_4$)$_2$, stable

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above 1430 °C, is not quenchable.15 The polymorphic transition that resulted from sintering temperature or cation impurity stabilizing phenomenon may induce tunable color emission of Eu2+ in the solid solution phases.

In our attempt to explore the luminescence of Eu2+ in compositions of the Sr3(PO4)2−Ca3(PO4)2 join, it is interestingly observed that, under an identical sintering procedure (holding temperature at 1250 °C and cooling rate of ∼10 °C/min), the end members Sr3(PO4)2 (α type with space group of R3̅m) and Ca3(PO4)2 (β type with space group of R3c) doped with Eu2+ ions emit bluish-purple light peaking at 416 nm, whereas the selected compositions with a proper amount of Ca2+ substituting Sr2+ show yellow light emitting; the corresponding emission peak even gets red-shifted to be 534 nm. The distinctive luminescence evolution of Eu2+ suggests the formation of a new phase that may have a similar space group with either of the end members but with different cell parameters. New compound discovery is of great interest to a board range of research fields including materials, crystal physics, and chemistry.16,17 Particularly, among the Sr3−xCa3(PO4)2 string compositions with interval x of 0.25, the Sr1.75Ca1.25(PO4)2:Eu2+ attracts much our attention because of its yellow-emitting merit and relatively excellent luminescence property.

The aim of this study is the observation of spectroscopic properties of Sr1.75Ca1.25(PO4)2:Eu2+ in relation with its crystal structure information and practical application in w-LEDs. The X-ray diffraction (XRD) pattern of SrTiO3 (the Sr1.75Ca1.25(PO4)2:Eu2+ attracts much our attention because of its yellow-emitting merit and relatively excellent luminescence property, this work is also aimed to investigate the structural property of Sr1.75Ca1.25(PO4)2:Eu2+ by XRD refinement. Furthermore, a near-UV w-LED lamp using the yellow emitting Sr1.75Ca1.25(PO4)2:Eu2+ and commercial blue-emitting BaMgAl10O17:Eu2+ (BAM:Eu) in combination with a 370 nm UV LED chip is successfully fabricated, and the optical properties of the device are examined. The results indicate that the reported phosphor can serve as a yellow component for application in the near-UV w-LEDs.

2. EXPERIMENTAL SECTION

2.1. Materials and Synthesis. The Sr1.75Ca1.25(PO4)2:xEu2+ (x = 0–0.15) powder samples were synthesized by the high temperature solid-state reaction method. The starting materials, SrCO3 (A.R. grade), CaCO3 (A.R. grade), NH4H2PO4 (A.R. grade), and Eu2O3 (99.99% purity), were stoichiometrically weighed and thoroughly mixed by an agate mortar and pestle. First, the mixtures were preheated at 700 °C for 2 h in a mull furnace in air to release NH3, CO2, and H2O. Then, the precursor was reground and heated at 1250 °C for 5 h in a tube furnace under flowing 5% H2−95% N2 reducing atmosphere. Finally, the samples were furnace-cooled to room temperature with cooling rate of ∼10 °C/min. A near-UV w-LED lamp was fabricated using a mixture of silicon resin and phosphor blend of the as-prepared yellow-emitting Sr1.75Ca1.25(PO4)2:Eu2+ and commercial blue-emitting BaMgAl10O17:Eu2+, which were dropped onto a 370 nm UV chip.

2.2. Characterization. The elemental composition of the phosphor was quantitatively analyzed by the inductively coupled plasma atomic emission spectroscopy (ICP-OES; IRIS Intrepid II XSP, Thermo Scientific). The powder diffraction pattern of Sr1.75Ca1.25(PO4)2:Eu2+, for Rietveld analysis was collected at room temperature with a Bruker D8-ADVANCE powder diffractometer (Cu Kα radiation) and a linear VANTEC detector. The step size of 0.02° was 0.016°, and the counting time was 1 s per step. Rietveld refinement was performed by using TOPAS 4.2.23 Room temperature excitation and emission spectra were measured on a fluorescence spectrophotometer (F-4600, HITACHI, Japan) with a photomultiplier tube operating at 400 V, and a 150 W Xe lamp used as the excitation source. The morphology was observed using scanning electron microscopy (SEM; JSM-6460LV, JEOL, Japan) and high-resolution transmission electron microscopy (HRTEM; JEM-2010, JEOL, Japan). The energy dispersive X-ray spectroscopy (EDS, INCA, Oxford instruments, U.K.) linked with the HRTEM was used to assist the composition analysis.
determination. The diffuse reflection spectra were measured using a UV–vis–NIR spectrophotometer (SHIMADZU UV-3600) attached with an integrating sphere. BaSO$_4$ was used as a reference for 100% reflectance. The optical properties, including the luminescent spectrum, $R_m$, CCT, and CIE value of the fabricated w-LED lamp, were measured by a HAAS-2000 light and radiation measuring instrument (Everfine, China).

### 3. RESULTS AND DISCUSSION

#### 3.1. Crystal Structure Refinement and Micromorphology of Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$Eu$^{2+}$

Under the present sintering process, the end members Sr$_3$(PO$_4$)$_2$ and Ca$_3$(PO$_4$)$_2$ are found to crystallize in space groups $R3m$ and $R3c$, respectively. Thus, it is expected that the samples with composition falling between the two end members would prefer to form partial solid solutions rather than complete solid solution, indicating that pure-phase formation of Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$ is really necessary. However, no Inorganic Crystal Structure Database (ICSD) or Joint Committee on Powder Diffraction Standards (JCPDS) file for the Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$ is available. The selected sample Sr$_{1.75}$Ca$_{1.25}$Eu$_{0.1}$(PO$_4$)$_2$ is then refined using the Rietveld method. The single crystal structure data of Ca$_{2.83}$Sr$_{0.17}$(PO$_4$)$_2$ (ICSD No. 161330; whitlockite-type structure)$^{24}$ is used as a starting model to refine the crystal structure. The observed (red), calculated (black), and difference (gray) XRD profiles for the refinement are shown in Figure 1. There are five independent Ca positions in the structure. In our model all of these positions were occupied by Ca and Sr, and occupation was refined; after refinement the Ca5/Sr5 position has Ca ion occupation equaling 1.00(1) and Sr ion occupation 0.0(1). Analysis of Ca5/Sr5–O bond lengths shows that this site is very small for Sr and Eu ions; therefore, it was decided to occupy this position by Ca ion only. The other four sites can be occupied by Sr and Eu ions. Eu ions were equally distributed between the four Ca/Sr positions, and their concentrations were fixed in these sites. The structure refinement was stable and ended with low $R$-factors (shown in Table 1, Figure 1).

#### Table 1. Crystallographic Data and Details in the Data Collection and Refinement Parameters for the Sr$_{1.75}$Ca$_{1.15}$Eu$_{0.10}$(PO$_4$)$_2$ Sample

<table>
<thead>
<tr>
<th>Sr$<em>{1.75}$Ca$</em>{1.15}$Eu$_{0.10}$(PO$_4$)$_2$</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>space group</strong></td>
<td>$R3c$</td>
</tr>
<tr>
<td>$a$, Å</td>
<td>10.6498(3)</td>
</tr>
<tr>
<td>$c$, Å</td>
<td>38.787(1)</td>
</tr>
<tr>
<td>$V$, Å$^3$</td>
<td>3809.8(2)</td>
</tr>
<tr>
<td>$Z$</td>
<td>21</td>
</tr>
<tr>
<td>2$\theta$-interval, deg</td>
<td>5–110</td>
</tr>
<tr>
<td>no. params of refinement</td>
<td>84</td>
</tr>
</tbody>
</table>

When doped with 0.10 mol Eu$^{2+}$ per mol Ca$^{2+}$ ions, the Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$ host persists the single-phase structure. The refinement shows that Sr$_{1.75}$Ca$_{1.15}$Eu$_{0.10}$(PO$_4$)$_2$ crystallizes in a rhombohedral unit cell with space group $R3c$ and lattice constants of $a = 10.6498(3)$ Å and $c = 38.787(1)$ Å, and cell volume $V = 3809.8(2)$ Å$^3$. The refinement is finally converged to $R_{exp} = 6.22$, $R_{wp} = 8.30$, and $R_p = 6.26$ (Table 1). The crystal structure is shown in Figure 2. Figure 2 also presents that the Sr1/Ca1, Sr2/Ca2, Sr3/Ca3, and Sr4/Ca4 positions are coordinated with seven, eight, eight, and six oxygen atoms, and the Ca5 position is coordinated with six oxygen atoms. Crystallographic information file (CIF) of Sr$_{1.75}$Ca$_{1.15}$Eu$_{0.10}$(PO$_4$)$_2$ is given in Supporting Information.

The elemental composition of sample with nominal formula Sr$_{1.75}$Ca$_{1.15}$Eu$_{0.10}$(PO$_4$)$_2$ verified by ICP-AES shows (wt %) Sr 38.05%, Ca 11.43%, Eu 3.690%, and P 51.560%. The stoichiometric weight ratio of each element in Sr$_{1.75}$Ca$_{1.15}$Eu$_{0.10}$(PO$_4$)$_2$ is Sr 37.772%, Ca 11.354%, Eu 3.743%, and P 51.26%. The composition suggested by ICP-AES is consistent with the stoichiometric weight ratio with reasonable relative error. Coexistence and the element distribution of Sr, Ca, P, O, and Eu in the solid-solution sample was also verified by the HRTEM and corresponding EDS analysis, as shown in Figure 3. Figure 3a shows the TEM image obtained from the selected Sr$_{1.75}$Ca$_{1.15}$Eu$_{0.10}$(PO$_4$)$_2$ crystal, and all the elements (including the C and Cu from the sample holder) were detected from one complete microcrystal (Figure 3d). The fine structures are further examined by HRTEM, and the fast Fourier transform (FFT) images suggest the highly single-crystalline nature, as shown in Figure 3b,c. It could be seen that the continuous lattice fringe measurements with $d$ spacing of 0.308 and 0.283 nm are in line with the solved structure of the compound by refinement, and could be assigned to the corresponding (030) and (042) planes, respectively.

![Figure 2](dx.doi.org/10.1021/ic500230v)
The micromorphology of the crystalline Sr\(_{1.75}\)Ca\(_{1.24}\)\((PO_4)_{2}:0.01\)Eu\(^{2+}\) phosphor sample observed by SEM is shown in Figure 4. It is observed that the particles have smooth morphology and the diameters are ranging from 3 to 8 \(\mu\)m. Some of the primary crystals reunited to yield agglomerates, and the observed “sintering necks” are suggesting that grain growth has occurred during the synthesis procedure. Without further hard crush, the as-prepared microphosphor particles can be used for w-LED fabrication.

3.2. Photoluminescence Properties of Sr\(_{1.75}\)Ca\(_{1.25}\)\((PO_4)_{2}:0.01\)Eu\(^{2+}\). Figure 5 illustrates the photoluminescence excitation and emission spectra of Sr\(_{1.75}\)Ca\(_{1.24}\)(PO\(_4\))\(_2\):0.01Eu\(^{2+}\). The broad asymmetric emission spectrum peaking at 514 nm shows that Eu\(^{2+}\) has more than one emission center in Sr\(_{1.75}\)Ca\(_{1.25}\)(PO\(_4\))\(_2\) which belongs to the typical emission of Eu\(^{2+}\) ions ascribed to 5d\(^{-}\)4f transitions.\(^{25}\) The excitation spectrum monitoring at 514 nm is composed of three absorption peaks located at 279, 320, and 364 nm in the spectral range from 200 to 430 nm. Since phosphate hosts exhibit absorption in the vacuum ultraviolet region (100–200 nm),\(^{26}\) the excitation band in the range of 230–430 nm is originated from the 4f\(^{5}\)5d\(^{1}\) excited state transition of Eu\(^{2+}\) ions. Under the excitation at 279 and 365 nm, the phosphor exhibited similar broad yellow emission band peaking at 514 nm. No dependence of the emission spectra on the excitation wavelengths was found. The broadband excitation character in 200 and 430 nm of the excitation spectrum verifies the possibility of the operation as a yellow-emitting phosphor combining with the near-UV LEDs chip (\(\lambda_{\text{em}}\) 350–410 nm), especially the excitation wavelength at 365 nm that can be found from typical commercial chips.

By Gaussian deconvolution, the emission spectrum of Sr\(_{1.75}\)Ca\(_{1.24}\)(PO\(_4\))\(_2\):0.01Eu\(^{2+}\) can be well-decomposed into four Gaussian profiles peaking at 449 nm (22 271 cm\(^{-1}\)), 500 nm (20 000 cm\(^{-1}\)), 534 nm (18 726 cm\(^{-1}\)), and 589 nm (16 978 cm\(^{-1}\)) which can be ascribed to four different Ca\(^{2+}\) sites occupied by Eu\(^{2+}\) ions. The refinement shows that the structure of Sr\(_{1.75}\)Ca\(_{1.25}\)(PO\(_4\))\(_2\) contains four crystallographically distinct Ca\(^{2+}\) sites that can be occupied by Eu\(^{2+}\) ions with average Ca–O bond distance as follows: Ca(1)–O 2.621 Å, Ca(2)–O 2.555 Å, Ca(3)–O 2.604 Å, and Ca(4)–O 2.655 Å. The Eu\(^{2+}\) ions substituting Ca\(^{2+}\) ions in the site which has shorter Ca–O bond distance are expected to experience stronger crystal field strength corresponding to a longer wavelength emission with larger Stoke shift, when the crystal environments are analogous. Figure 5 illustrates four kinds of polyhedra with different average Ca–O bond distance, corresponding to the four Gaussian profiles in the emission spectrum. Moreover, due to the straightforward symmetry behavior of the energy levels,
Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$ doped with Eu$^{3+}$ was prepared following the identical sintering procedure in air, to use Eu$^{3+}$ as a probe to determine the local structure symmetry. The PL spectrum of Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$:Eu$^{3+}$ (Supporting Information Figure S1) shows strong emission at 616 nm along with a relatively weak emission at 592 nm, suggesting that the symmetry around the Eu$^{3+}$ ions in the host is low, a fact confirming the crystallographic sites occupancy of Eu$^{2+}$ in Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$ identified by the refinement.

Figure 6 exhibits the UV–vis reflection spectra of undoped and Eu$^{2+}$-doped Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$ samples. The Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$ host demonstrates an absorption edge in the ≤420 nm region. With the introduction of Eu$^{3+}$ ions in Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$, a broad absorption appears in the range 300–460 nm which originated from the $4f^{-4}f^65d^1$ transition of Eu$^{3+}$, which matches well with near-UV chips for applications in near-UV w-LEDs. Also, the absorption edge gradually extends to longer wavelengths, and the absorption gets enhanced with higher Eu$^{2+}$ concentration.

Figure 7 presents the room temperature emission spectra of Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$:Eu$^{2+}$ ($x = 0.005–0.15$) under 365 nm UV light excitation. With an increasing Eu$^{2+}$ doping level, the emission intensity of Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$:Eu$^{3+}$ initially increased, reached the maximum at $x = 0.01$, and then decreased, resulting from the concentration quenching effect. The inset of Figure 7a shows the dependence of the emission intensity on the concentration of Eu$^{2+}$, suggesting that the quenching in Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$:Eu$^{2+}$ ($x = 0.005–0.15$) phosphors under 365 nm UV light excitation. The inset shows the dependence of the emission intensity on the concentration of Eu$^{3+}$. Although the intrinsic explanation is still unknown, it suggests that Eu$^{2+}$ prefers to occupy some specific sites at high doping level. It is known that the interaction type between sensitizers or between sensitizer and activator can be calculated by the following eq 1:

$$I/x = k[1 + \beta(x)^{0/3}]^{-1}$$

Here, $x$ is the activator concentration, $I/x$ is the emission intensity (I) per activator concentration ($x$), $k$ and $\beta$ are constants for the same excitation condition, and $\theta$ is a function of multipole–multipole interaction. When the value of $\theta$ is 6, 8, or 10, the form of the interaction corresponds to dipole–dipole ($d=d$), dipole–quadrupole ($d=q$), or quadrupole–quadrupole ($q=q$), respectively. The value of $\theta$ is calculated to be 4.1073, which is mostly close to 6, suggesting that the quenching in Sr$_{1.75}$Ca$_{1.25}$(PO$_4$)$_2$:Eu$^{2+}$ phosphors most likely results from dipole–dipole interactions.
3.3. Performances of the Fabricated Near-UV w-LEDs.

The CIE chromaticity diagram for Sr_{1.75}Ca_{1.24}(PO_4)_2:0.01Eu^{2+} under 365 nm light excitation is shown in Figure 9a. The color coordinates are calculated to be (x = 0.374, y = 0.588), indicating this phosphor may serve as a yellow emitting component for w-LED application. The inset in Figure 9a shows the digital photograph of Sr_{1.75}Ca_{1.24}(PO_4)_2:0.01Eu^{2+}, the intense yellow emission of which can be observed. To evaluate the potential application of Sr_{1.75}Ca_{1.25}(PO_4)_2:Eu^{2+} in w-LED, we combined mixtures of this phosphor and commercial blue-emitting BaMgAl_{10}O_{17}:Eu^{2+} (BAM:Eu) with 370 nm UV chip to realize white light. Figure 9b shows the electroluminescent (EL) spectrum of the fabricated w-LED prototype. The blue emission band is ascribed to BAM:Eu, and the yellow emission band corresponds to Sr_{1.75}Ca_{1.25}(PO_4)_2:Eu^{2+}; the full-color white light emission is achieved. The color coordinate is calculated to be (0.368, 0.414) with the white light emission correlated color temperature (CCT) of 4529 K and good CRI value (R_a) determined as 85.1.

4. CONCLUSIONS

On the basis of the composition design in the Sr_{3}(PO_4)_2−Ca_{3}(PO_4)_2 join, a new yellow-emitting whitlockite-type structure phosphor Sr_{1.75}Ca_{1.25}(PO_4)_2:Eu^{2+} has been discovered, and its crystal structure is refined to be rhombohedral unit cell with space group R3c and lattice constants a = 10.6498(3) Å and c = 38.787(1) Å, and cell volume = 3809.8(2) Å³ using the Rietveld method. It exhibits a broad emission band peaking at 514 nm which is ascribed to the 5d−4f transition of Eu^{2+}. The dipole−dipole interaction plays a major role in the explanation of the concentration quenching effect. The w-LED is encapsulated with a 370 nm-emitting chip, and the as-prepared Sr_{1.75}Ca_{1.25}(PO_4)_2:Eu^{2+} and commercial blue-emitting BaMgAl_{10}O_{17}:Eu^{2+} exhibit bright white emission, indicating that Sr_{1.75}Ca_{1.25}(PO_4)_2:Eu^{2+} can serve as a potential yellow component candidate for near-UV w-LED fabrication.
providing a doctoral scholarship for his Ph.D. study at the University of Auckland.

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