A Novel Narrow-Band Green-Emitting Phosphor CsKNaLi(Li3SiO4)4 with the UCr4C4-Related Type Structure

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Abstract

We successfully synthesized CsKNaLi(Li3SiO4)4:Eu2+ (CKNLLSO:Eu2+) as a new member of the UCr4C4-type structural oxide family. The crystal structure and band structure of the host compound of this phosphor were analyzed and calculated through Rietveld refinement method and density functional theory, respectively. Owing to the highly compressed and rigid anionic framework, Eu2+ substituting the Cs site emits bright green fluorescence under excitation by InGaN-based UV LEDs, with a full width at half maximum of 55 nm. The stability of CKNLLSO:Eu2+ remains relatively good when the temperature is elevated to 190°C. The LED device fabricated using concentration-optimized CKNLLSO:4%Eu2+ emits green light with color coordinates of (0.2320, 0.6016) and a correlated color temperature of 7314 K. The analysis results demonstrate that this phosphor is a promising green-emitting phosphor for white LEDs.

Full Text

Preamble

A Novel Narrow-Band Green-Emitting Phosphor CsKNaLi(Li3SiO4)4 with the UCr4C4-Related Type Structure

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CsKNaLi(Li3SiO4)4:Eu2+ (CKNLLSO:Eu2+) has been successfully synthesized as a new member of the oxide-based family with a UCr4C4-related type structure. The crystal and band structure of the host compound were

characterized and analyzed through Rietveld refinement and density functional theory, respectively. As a result of the highly condensed and rigid anion framework, Eu2+ substituting the Cs site yields intense green emission with a narrow full width at half maximum of 55 nm when excited by InGaN-based near-UV LEDs. The CKNLLSO:Eu2+ phosphor exhibits relatively high thermal stability even when the temperature is raised to 190 °C. The LED fabricated using the optimized CKNLLSO:4%Eu2+ phosphor demonstrated bright and narrow green light with chromaticity coordinates (0.2320, 0.6016) and a correlated color temperature of 7314 K, implying its great potential for applications as a green component in white LEDs.

1. Introduction

Phosphor-converted white light-emitting diodes (pc-wLEDs) have been widely used in general lighting, liquid crystal display (LCD) backlights, and automotive headlights. Conventional pc-wLEDs are composed of a blue LED chip and yellow YAG:Ce phosphor, where the latter governs the color characteristics of white LEDs. However, due to the lack of red emission, high color rendering index (CRI) white LEDs cannot be realized using YAG:Ce alone. Simultaneously, the low color saturation of such white LEDs makes it difficult to achieve the National Television System Committee (NTSC) standard because of their broad full-width at half-maximum (FWHM). An alternative approach to generating white light involves pumping red-green-blue (RGB) phosphors with a near-UV LED chip.

Current commercial green phosphors such as (Ba,Sr)2SiO4:Eu2+ suffer from poor chemical stability and severe thermal quenching. Promising candidates including Si6−zAlzOzN8−z:Eu2+, Ba2LiSi7AlN12:Eu2+, and BaLi2(Al2Si2)N6:Eu2+ exhibit high quantum efficiency, narrow FWHM, and excellent thermal stability [**?**]. However, the preparation conditions for these nitride or oxynitride phosphors are harsh, typically requiring high nitrogen gas pressure, which restricts intensive study of their luminescent mechanisms and hampers further development and applications. Therefore, novel green phosphors are urgently needed for pc-wLEDs.

Recently, Schnick and co-workers discovered a series of nitride phosphors with UCr4C4-related type structures [**?**]. These compounds, including Ca(LiAl3)N4, Sr(LiAl3)N4, Sr(Mg3Si)N4, Ca(Mg3Si)N4, M(Mg2Al2)N4 (M=Ca, Sr, Ba), and Ba(Mg2Ga2N4), have the general formula ABC3X4 or AB2C2X4 and have proven to be promising red phosphors for pc-wLEDs with narrow emissions. Building on the discovery that the structure types of Ca(LiAl3)N4 and Sr(Mg3Si)N4 are shared by NaLi3SiO4, and that Sr(LiAl3)N4 crystallizes in the KLi3SiO4 structure, Prof. Hubert Huppertz and co-workers described a dozen additional narrow-band representatives [**?**, **?**]. Three lithosilicate phosphors—NaLi3SiO4:Eu2+, KLi3SiO4:Eu2+, and NaK7(Li3SiO4)8:Eu2+—were reported to exhibit ultra-narrow blue, broad near-warm-white, and yellow-blue emissions under near-UV or blue light excitation. All these alkali lithosilicates

feature a novel structure type with a highly condensed network of LiO4 and SiO4 tetrahedra, where monovalent cations occupy vierer-ring channels along [001]. Eu2+ ions substitute these alkali metal cations within the channels, and the highly diverse luminescence was attributed to the complex extended coordination rather than the first coordination sphere of the substituted sites [**?**].

From last year to early this year, Prof. Zhiguo Xia and co-workers reported several phosphors with UCr4C4-related type structures [**?**]. Among them, RbNa3(Li3SiO4)4:Eu2+ and RbLi(Li3SiO4)2:Eu2+ exhibit narrow-band blue and green emissions at 471 nm (FWHM=22.4 nm) and 530 nm (FWHM=42 nm), respectively [**?**, **?**]. These two host compounds originate from Na4(Li3SiO4)4, where one-quarter of Na atoms are replaced by Rb to form RbNa3(Li3SiO4)4 belonging to the tetragonal (I4/m) space group. Similarly, all Na atoms in Na2(Li3SiO4)2 are substituted by Rb and Li atoms in equal amounts to obtain RbLi(Li3SiO4)2. These works proposed a mineral-inspired phosphor design principle. Inspired by this concept, two additional phosphors —RbNa2K(Li3SiO4)4:Eu2+ (RNKLSO:Eu2+) and CsNa2K(Li3SiO4)4:Eu2+ (CNKLSO:Eu2+)—were obtained through cation substitution. In RNKLSO, one-third of Na atoms in RbNa3(Li3SiO4)4 were replaced by K while keeping the Rb substitution, and in CNKLSO, the Rb atom was further substituted by Cs. The photoluminescent (PL) properties of RNKLSO:Eu2+ and CNKLSO:Eu2+ phosphors are similar.

M4(Li3SiO4)4 (where M denotes one, two, or more alkali metal ions from Li, Na, K, Rb, Cs with a total atom number of 4) represents the general formula of alkali lithosilicates with UCr4C4-type structure. Since different M cations exhibit different luminescent properties, this work synthesized the CKNLLSO:Eu2+ phosphor through a cation substitution strategy. Starting from CNKLSO, half of the Na atoms were replaced by Li to synthesize CKNLLSO. Under 395 nm UV LED excitation, the PL spectra of CKNLLSO:Eu2+ consist of a dominant band peaking at 19027 cm−1 (525 nm) with FWHM=55 nm and two minor shoulder bands peaking at 21365 cm−1 (468 nm) and 20813 cm−1 (480 nm). Unlike the blue-emitting property of CNKLSO:Eu2+, CKNLLSO:Eu2+ produces green emission. Additionally, its thermal quenching behavior was analyzed using a configuration coordination diagram, and the optical properties of fabricated LED devices were discussed. All results indicate that CKNLLSO:Eu2+ is a promising green-emitting candidate for near-UV LED devices.

2. Results and Discussion

Na4(Li3SiO4)4 crystallizes in the tetragonal system with space group I41/a (No. 88) and lattice parameters a=1078.4(1) and c=1263.3(1) pm. Its crystal structure consists of a highly condensed network constructed from LiO4 and SiO4 tetrahedra in a 3:1 ratio, leading to a high degree of condensation =1 (atomic ratio of (Li,Si)/O). These tetrahedra form vierer-ring channels along the [001] direction by sharing corners, as shown in Figure 1(b). The channel

contains one crystallographic Na1 site surrounded by six LiO4 and two SiO4 tetrahedra. The structure also features a 41 screw axis at its center that is not aligned with the Na strands. This asymmetry leads to variation in Na–O bond lengths from 243.81 to 273.42 pm.

Figure 1 illustrates Na strands viewed along [001] in Na4(Li3SiO4)4 (a) and its crystal structure (b, c). A schematic shows the composition transformation from Na4(Li3SiO4)4 to CsKNaLi(Li3SiO4)4 (from b to d). Na–Li strands and Cs–K strands viewed along [001] in CsKNaLi(Li3SiO4)4 are shown (e), along with its crystal structure (d, f).

Starting from Na4(Li3SiO4)4, three-fourths of the Na atoms were substituted by Cs, K, and Li in equal amounts, resulting in a new phase CKNLLSO. This compound crystallizes in the tetragonal system with space group I4/m (No. 87) and lattice parameters a=1102.0(6) and c=637.9(5) pm. Similar to the parent structure, it is composed of LiO4 and SiO4 tetrahedra in a 3:1 ratio with a high degree of condensation (=1, i.e., atomic ratio (Li,Si)/O=1). The unit cell contains two types of channels designated CH1 and CH2 (Figure 1(e)), which can be identified by their central cations. Na and Li atoms occupy the centers of the first channel type (CH1), while Cs and K atoms occupy the central positions of CH2. As illustrated in Figure 1(e), Na and Li atoms alternate and are surrounded by equal numbers of SiO4 and LiO4 tetrahedra sharing oxygen vertices. In CH2, alternating Cs and K atoms are surrounded exclusively by LiO4 tetrahedra. Both CH1 and CH2 contain two crystallographic sites: Li3 and Na1 sites in CH1, and Cs1 and K1 sites in CH2, respectively. The symmetry of these channels differs from that in Na4(Li3SiO4)4. All cation sites in CKNLLSO are aligned along a 4 inversion axis, as evident from the Na–Li and Cs–K strands shown in Figures 1(e) and 1(f). CH2 is constructed solely from LiO4 tetrahedra, which can be attributed to the larger size of Cs and K cations compared to Na.

Figure 2(a) shows the X-ray diffraction (XRD) patterns of CKNLLSO:x%Eu2+ (x=0.1, 0.5, 1, 2, 5, 6) alongside the reference ICSD-74863. All diffraction peak positions match well with those of the single crystal reference. Eu2+ doping did not introduce any impurities or cause significant structural transformation even at doping concentrations as high as 6%. To determine the actual crystal structure of the synthesized samples, Rietveld refinement of CKNLLSO:4%Eu2+ was performed using the GSAS program with ICSD-74863 as the starting model. The experimental spectrum and calculated data are represented by black crosses and a red line, respectively. A swelling appears in the range of 16° to 37°, caused by the Si substrate of the X-ray diffractometer and deducted as part of the background. Comparison of experimental and calculated spectra shows that each diffraction peak aligns with Bragg reflection positions, and no impurity phase was detected, confirming single-phase crystallization. The calculated reliability factors are Rp=3.26%, wRp=4.78%, and ^{2}=3.251. Detailed refinement procedures and the crystallographic information file (CIF) for CKNLLSO are provided in the Supporting Information.

To understand the light absorption capability, the band structures of CKN-

LLSO and EuKNaLi(Li3SiO4)4 (EKNLLSO) host compounds were calculated using density functional theory with generalized gradient approximations (DFT-GGA), as shown in Figures 2(b) and 2(e). The calculations predict an indirect band gap (Eg) of 5.015 eV for the CKNLLSO host, with the conduction band minimum and valence band maximum located at k-point G and near k-point G, respectively. Figures 2(c) and 2(f) illustrate the calculated total and orbital-projected densities of states (DOS) for CKNLLSO and EKNLLSO. The valence band maximum is primarily composed of O 2p orbitals, while the conduction band bottom is dominated by Li 1s, Na 2s, K 3s and 3p, and Cs 5p orbitals. Figure 2(e) depicts the band structure of the EKNLLSO host; a unit cell was used for electronic structure calculation to reduce computational cost. Therefore, only Cs atoms were replaced by Eu2+ ions in the unit cell, and the band gap decreased to 4.395 eV after doping. Eu introduction did not noticeably spin-polarize the valence band but triggered spin polarization of the conduction band. The corresponding spin-polarized DOS in Figure 2(f) shows that O 2p orbitals form the main components of the valence band top, similar to the EKNLLSO host. However, the conduction band minimum primarily consists of majority-spin Eu 5s, 5p, 4d and minority-spin Eu 4d, 5p, 4f orbitals, indicating that electron transitions from O 2p to Eu 4f orbitals will occur under UV radiation, forming O–Eu charge transfer bands.

Figure 3(a) presents the 7Li solid-state NMR spectrum of CKNLLSO:4%Eu2+, showing an asymmetric broad peak with a Li chemical shift at 1.29 ppm, confirming Li incorporation into the structure. Scanning electron microscopy (SEM) images in Figure 3(b) reveal that CKNLLSO:4%Eu2+ phosphor particles are approximately 3–8 m in size with smooth surfaces and irregular shapes. Figure 3(d) shows energy-dispersive X-ray spectrometer (EDS) mapping images of CKNLLSO:4%Eu2+ particles. Combined with the 7Li NMR spectrum, these images confirm that the sample contains Cs, Si, Eu, K, Na, O, and Li, with all elements distributed uniformly throughout the synthesized material.

Figure 4(a) displays the photoluminescence (PL) spectra of CKNLLSO:x%Eu2+ (x=0.1, 0.5, 1, 2, 4, 5, 6) under 397 nm excitation, demonstrating the optimization of Eu2+ doping concentration. The PL spectrum shows an asymmetric emission band centered at 525 nm with a shoulder peak near 482 nm. All emission profiles are identical except for intensity variations. In Figure 4(d), the PL spectrum of CKNLLSO:4%Eu2+ is fitted and deconvoluted into three Gaussian peaks at 21364.76 cm−1 (468 nm), 20812.83 cm−1 (480.5 nm), and 19027.30 cm−1 (525.6 nm). Monovalent cations in lithosilicates are known to be suitable for Eu2+ doping [**?**, **?**]. Therefore, in the CKNLLSO host, three cation sites are available for Eu2+ incorporation: Na+ in the first channel type (CH1), and K+ and Cs+ in the second channel type (CH2). The environments of these three potential sites show great similarities, with all exhibiting eight-fold oxygen coordination in the first coordination sphere. The Na+, K+, and Cs+ sites display cuboid (K+, Cs+) or slightly deformed cuboid (Na+) coordination with respect to oxygen (Figure S1). Na–O bond lengths range from 2.5019 to 2.6745 Å, providing a slightly small space for Eu2+ cations. Na+ is surrounded by four LiO4

and four SiO4 tetrahedra, while both Cs+ and K+ show octahedral coordination by eight LiO4 tetrahedra. Compared to lithium, Si4+ cations have higher partial charges and thus exhibit more rigid positioning within the anionic framework, leading to stronger interactions between central cation positions and SiO4 tetrahedra. Consequently, the narrow-band weak emission of CKNLLSO:Eu2+ peaking at 480.5 nm should originate from Eu2+ occupying Na+ sites. Unlike Na+, K+ and Cs+ provide symmetric cuboid coordination toward oxygen (Figure S1), with K-O and Cs-O bond lengths of 2.8521 and 3.0917 Å, respectively. The KO8 and CsO8 octahedra in the CKNLLSO host provide adequate space for Eu2+ ions. Figure 4(a) illustrates that the intensity changes of the main and shoulder emission bands differ with increasing Eu2+ concentration. As Eu2+ concentration increases from 0.1% to 6%, the intensity of the 525 nm emission band rises sharply initially, reaching maximum at 4% doping. In contrast, the shoulder band intensity also increases initially but then decreases, with maximum intensity occurring at 1% doping. Based on this behavior, the main and shoulder emission bands should be attributed to 4f-5d transitions of Eu2+ at Cs and K sites, respectively, because the KO8 octahedron provides smaller space than CsO8, making luminescence concentration quenching more probable.

Figure 4(b) illustrates the dependence of normalized integrated emission intensity and FWHM values of the main emission band on Eu2+ concentration. FWHM values are expressed in wavenumber units; for CKNLLSO:4%Eu2+, the FWHM is 1995 cm−1 (from 500 to 555 nm), confirming it as a very narrow-band green-emitting phosphor. Digital photographs of all samples under natural and UV light in Figure 4(c) show bright emission. Figure 4(a) presents decay curves of CKNLLSO:x%Eu2+ (x=2, 4, 5) monitoring 525 nm green light under 397 nm excitation. These curves can be fitted using a first-order exponential decay formula [**?**]:

$$I(t) = I_0 + A\exp(-t/\tau)$$

where I(t) and I0 are luminescence intensities at time t and t , A is a constant, and is the decay time for an exponential component. Using this formula, the lifetime values for CKNLLSO:x%Eu2+ (x=2, 4, 5) phosphors are 0.907, 0.927, and 0.905 s, respectively, which parallels the trend in PL intensity. Photoluminescence excitation (PLE) and diffuse reflection spectra of CKNLLSO:x%Eu2+ (x=0.1, 0.5, 1, 2, 4, 5, 6) are depicted in Figures 4(e) and 4(f). The PLE spectra monitored at 525 nm show broad bands from 250 to 500 nm, demonstrating a wide spectral response range and compatibility with UV or blue excitation. The diffuse reflection spectra also reflect the light absorption capability of the samples. As shown in Figure 4(f), absorption band intensity increases with Eu2+ content, confirming that concentration quenching causes the emission intensity decrease when Eu2+ concentration exceeds 4%.

Figure 5(a) presents a schematic energy level diagram for Eu2+ ions in the CKNLLSO crystal. Eu2+ enters Cs and K sites in CsO8 and KO8 cuboid

coordination environments, where 5d energy splitting occurs under the influence of a cubic crystal field. Due to different shapes and orientations of the 5d orbitals, the cubic crystal field of EuO8 affects the five orbitals differently. The maxima of dxy, dyz, and dxz orbitals face the centers of cubic edges, while those of $d_{x^2-y^2}$ and d_{z^2} point toward the centers of cubic faces. Consequently, dxy, dyz, and dxz orbitals are closer to oxygen ligands than $d_{x^2-y^2}$ and d_{z^2}, causing the 5d orbitals to split into two groups as shown in Figure 5. Transitions from 4f to dxy, dyz, dxz and to $d_{x^2-y^2}$, d_{z^2} orbitals generate two absorption bands at 35211 cm−1 and 27382 cm−1, respectively. The barycenter energy of 4f 5d can be calculated as (35211×3 + 27382×2)/5 = 32079 cm−1. Combined with the emission band in Figure 4(a), the Stokes shift is calculated to be 8355 cm−1.

Thermal stability is a critical parameter for high-power applications. Temperature-dependent PL spectra of CKNLLSO:4%Eu2+ under 397 nm radiation are illustrated in Figures 6(a) and 6(c). The sample exhibits relatively good thermal stability, with integrated PL and peak (525 nm) intensities at 190 °C retaining approximately 61.2% and 60.6% of their values at 30 °C, respectively. Additionally, the emission spectra blue-shift with increasing temperature, which can be attributed to thermally activated phonon-assisted tunneling from lower-energy to higher-energy emission bands in the excited states of Eu2+.

The temperature-dependent PL intensity can be fitted using the Arrhenius equation [**?**]:

$$I_T = \frac{I_0}{1 + A\exp(-\Delta E/kT)}$$

where IT and I0 are PL intensities at temperature T and room temperature, respectively, A is a constant, ΔE is the activation energy for thermal quenching, and k is the Boltzmann constant. As shown in Figure 6(d), ΔE is calculated to be 0.2328 eV based on PL intensity variation with temperature, indicating that thermal quenching in CKNLLSO:4%Eu2+ is more likely to occur than in RNKLSO:Eu2+. However, compared with CNKLSO:Eu2+, the thermal stability is slightly better [**?**].

To further evaluate the potential of CKNLLSO:Eu2+ in lighting and display applications, the optimized phosphor was mixed with epoxy and coated on near-UV chips (=395 nm). The emission spectra of the fabricated single-color LED consist of two bands in the blue and green regions, producing intense green light visible to the naked eye. The chromaticity coordinates are (0.2320, 0.6016) with a correlated color temperature of 7314 K. These phosphors are suitable candidates as green components in WLEDs for full-spectrum emission.

In conclusion, a novel UCr4C4-related type silicate-based CKNLLSO:Eu2+ phosphor was prepared, exhibiting narrow-band green emission at 525 nm with FWHM = 55 nm. The structure features a highly condensed network

of LiO4 and SiO4 tetrahedra forming an anionic framework, with cation sites located in vierer-ring channels along the [001] direction. The narrow-band green emission originates from 5d→4f transitions of Eu2+ at Cs sites and is related to the high degree of condensation (=1) and cubic coordination sites for Eu2+ ions. The 5d energy levels of Eu2+ split into two groups in the cubic crystal field, with a barycenter energy of 4f 5d calculated to be 32079 cm−1. The CKNLLSO:Eu2+ phosphor demonstrates relatively good thermal stability and is a suitable candidate as a green component in backlighting LEDs for full-spectrum emission.

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Note: Figure translations are in progress. See original paper for figures.