1. Introduction

Temperature sensing and measuring are crucial for various scientific investigations and technological developments [1–4]. According to the contact between the sensor and the object, the measurement methods can be classified into three categories of invasive, semi-invasive, and noninvasive [2]. The invasive contact thermometry shows great potential when spatial resolution decreases to the submicron scale and cannot meet the increasing sensing demands in newly emerging areas of nanomedicine, microoptics, microelectronics, photonics, and microfluidics [2,3]. The non-contact optical thermometers show unique features of noninvasive operation, rapid response, and high-spatial resolutions. The noninvasive thermometers are favorable candidates for the measurement of fast moving targets and are capable of operating in chemically, electromagnetically, and thermally harsh surroundings [2,5]. The limitations of invasive thermometers and the merits of the non-contact thermometers spur the development of new semi-invasive and noninvasive (noncontact) accurate sensing systems with micrometric and nanometric spatial resolution [1,2]. Among non-contact spectroscopic methods sensing temperature via the thermal dependence of phosphor luminescence is one of the most promising techniques. The thermographic phosphor thermometry senses temperature based on the change of peak shape, peak energy and intensity, peak shift, luminescence lifetimes, and fluorescence intensity ratio (FIR) of the phosphors against temperature [2]. The method has the advantages of remote observation, high sensitivity, good spatial resolution, and short acquisition periods [6]. In phosphor-based thermometry, the phosphors are key component and decisively determine the properties of the thermometry. Ln3+ -based phosphors are versatile, stable, and in general with high emission quantum yields. Various systems have been investigated as optical thermometers such as SrY2O4:Bi3+,Sr3+Eu3+ [6], Gd4O3F6: Ho3+,Yb3+ [7], Sr3Y(PO4)2:Yb3+ /Ln3+ [8], Sr3Ce(PO4)2:Tb3+/Mn2+ [9], Li4CaLa3Nb2O12:Yb/Er [10], Ce3+/ Eu3+: in LnSr4(BO3)3 [11], Er:Yb:NaY2F5O [12], etc. It was found that phosphors have good thermal stability tend to show favorable sensing properties. Rare earth double tungstate NaLn(WO4)2 (Figure S1) beneficially delays the concentration quenching process of activator as most rare earth are well isolated by [WO4] tetrahedron and have large RE:RE distance of 5.2483 Å (Figure S1 (a)). Although some rare earth are adjacent
2. Experimental procedure

2.1. Reagents and sample synthesis

The starting chemicals of Ln(NO$_3$)$_3$·6H$_2$O (>99.9% pure; Ln = Gd, Eu, Er, and Yb respectively), (NH$_4$)$_2$SO$_4$ (>99.5% pure), NH$_3$·H$_2$O (analytical grade), and Na$_2$WO$_4$·2H$_2$O (>99.5% pure) were purchased from Kanto Chemical Co., Inc. (Tokyo, Japan) and used without further purification. Milli-Q filtered water (resistivity >18 MΩ·cm) was used throughout the experiments. According to previously references 5% doping already made the host show good photoluminescence properties [18,19] thus in this work the Eu$^{3+}$ doping level was chose to be 5 at%. The synthesis of (Gd$_{0.95}$Eu$_{0.05}$)$_2$(OH)SO$_4$·2H$_2$O and (Gd$_{0.97}$Yb$_{0.03}$)$_2$(OH)SO$_4$·2H$_2$O precursors (Ln-241) typically involves hydrothermal reaction of an aqueous solution (60 ml) containing 6 mmol of Ln nitrates and 6 mmol of (NH$_4$)$_2$SO$_4$ under 100°C and pH = 7 for 24 h, and the details of which can be found elsewhere [24]. For phase conversion synthesis of double tungstate, the Ln-241 precursor was dispersed in 60 ml of Na$_2$WO$_4$ solution (WO$_4$$^2$·Ln$^{3+}$ molar ratio = 2.5) by 30 min of magnetic stirring, followed by hydrothermal reaction in a Teflon-lined stainless steel autoclave at a predetermined temperature (100–200°C) for 24 h. After natural cooling, the hydrothermal product was collected via centrifugation, washed with water three times and absolute ethanol once, and then dried in air at 70°C for 24 h.

2.2. Characterization

Phase identification was performed via X-ray diffractometry (XRD; Model RINT2200, Rigaku, Tokyo, Japan) under 40 kV/40 mA using nickel-filtered Cu–Kα radiation ($\lambda$ = 0.15406 nm) and a scanning speed of 1 °/min for 2θ = 5–70°. Structure parameters of the product were obtained via Rietveld refinement of XRD pattern using the TOPAS software [25]. Morphology analysis was performed via field emission scanning electron microscopy (FE-SEM; Model S-5000, Hitachi, Tokyo) under an acceleration voltage of 10 kV. Fourier transform infrared spectroscopy (FT-IR) was conducted by the standard KBr pellet method on a Model 4200 instrument (JASCO, Tokyo). Room–temperature down conversion (DC) photoluminescence spectra were measured with an FLS 1000 fluorospectrophotometer (Edinburgh Instruments Ltd., Herrsching am Ammersee, Germany) equipped with a 450 W xenon lamp for excitation. Temperature-dependent PL was measured in the range of 25–275°C using the same fluorospectrophotometer equipped with a TAP-02 high temperature controller. The room temperature and temperature-dependent upconversion (UC) luminescence spectra were recorded using the FLS 100 under 978 nm continuous wavelength (CW) laser excitation (Model MDL-III-980-2 W-18,050,833). Fluorescence decay kinetics of DC and UC emissions were both analyzed with the lifetime testing unit of the FLS 1000 equipment.
3. Results and discussion

3.1. Synthesis of Na(Gd, Eu)(WO₄)₂ via layered hydroxide precursor

Figure 1(a) shows the products converted from the 241-L(Gd, Eu)H precursor at temperatures ranging from 100 to 200°C with the WO₄²⁻/RE⁺⁺ molar ratio and reaction time were fixed at R = 2.5 and 24 h, respectively. It was found that the 100 and 120°C products show the diffractions of LRHs precursor (black dots, Figure 1b) and these of unknown phase (red dots, Figure 1b). It is encouraging to find out that target Na(Gd, Eu)(WO₄)₂ has become the main phase of the product (blue dots, Figure 1b) when the temperature was increased to 150°C, although the presence of unknown phase (red dots, Figure 1b) and the precursor (black dots, Figure 1b) can still be found. The 180 and 200°C reaction successfully produced phase pure Na(Gd, Eu)(WO₄)₂. Rietveld refinement was carried out for 241-L(Gd, Eu)H precursor and the 200°C product and well acceptable reliability factors (Figure S2 and Table S1) were obtained which further confirm the purity of the product. The derived cell dimensions are slightly bigger than those of pure precursor and NaGd(WO₄)₂ (Table S1), which is owing to the doping of bigger Eu³⁺ into the host [26].

Figure 2 shows the micro-morphology of the 241-L(Gd, Eu)H precursor and the products obtained via 24 h of reaction under WO₄²⁻/Ln³⁺ ratio R = 2.5 and different temperatures. Figure 2a shows the aggregated sphere morphology of the 241-L(Gd, Eu)H precursor, with microplate the basic unit (the insert in Figure 2a). The 100 and 120°C products mainly have the microplates morphology, which corresponding well with the observed XRD patterns. The 150°C product obviously has the mixed morphologies of microsquares and unconsumed microplates precursor. The 180°C product mainly contains microsquares. The 200°C product solely contains microsquares similar with 180°C product only with bigger sizes, and the results correspond well with those of the XRD analysis shown in Figure 1 that phase-pure Na(Gd, Eu)(WO₄)₂ has been formed at 180 and 200 °C.

To further elucidate chemical and phase purity, the FT-IR spectra of the L(Gd, Eu)H-241 precursor and the phase conversion product of Na(Gd, Eu)(WO₄)₂ were comparatively studied and showed in Figure S3. The precursor (Figure S3a) shows the vibrations of OH⁻ (~3620 and 3491 cm⁻¹), SO₄²⁻ (ν₅, ν₁, ν₄, and ν₃), and hydration water (~3491 and 1645 cm⁻¹) [27], which conform to the chemical formula of (Gdₓ,Eu₁₋ₓ)₂(OH)₄SO₄·2H₂O. The conversion product (Figure S3b) exhibits strong [WO₄]²⁻ vibrations at 445 cm⁻¹ (ν₄ vibration, symmetric bending), ~953 (ν₁ vibration, symmetric stretching) and ~713/795/853 cm⁻¹ (ν₃ vibrations).
vibration, asymmetric stretching mode) [28]. The $SO_4^{2-}$ and hydroxyl vibrations of the precursor were hardly observed in the conversion product. The above results further confirmed that phase conversion is complete at 200°C and the high phase and chemical purity of the resultant Na(Gd,Eu)(WO$_4$)$_2$.

### 3.2. Lifetime based luminescent thermometry of Na(Gd,Eu)(WO$_4$)$_2$

Figure 3a shows the excitation spectra for the Na(Gd,Eu)(WO$_4$)$_2$ phosphors measured at different temperatures. It can be found that the spectra consist of a weak broad band in short UV region (roughly 240-320 nm) and a series of sharp peaks in the ~350-500 nm region. The relatively weak broad band is overlapped from excitation of the tungstate ligand ($^1A_{1g} \rightarrow T_{1g}$ transition) [29], and $O^{2-} \rightarrow $ Eu$^{3+}$ charge transfer (CT) with former the dominant [30]. The sharp strong peaks in 350 to 500 nm regions belong to the $4f^6$ transitions of Eu$^{3+}$, as labeled in the figure, with the 394 nm ($^7F_{0.1} \rightarrow ^5L_6$) and the 464 nm ($^7F_{0.1} \rightarrow ^5D_2$) the predominately stronger two excitations. This indicates that the phosphors can be effectively excited by both UV light of 394 nm and blue light of 464 nm. The intensities of $4f^6$ transitions monotonously decreased with increasing temperatures, the tendency was shown in Figure 3b. The 394 ($^7F_{0.1} \rightarrow ^5L_6$) and 464 ($^7F_{0.1} \rightarrow ^5D_2$) nm peaks retain their ~45% original intensity at 150°C. Further analysis find that with increasing temperature the $I_{464}/I_{396}$ ratio increased in a single exponential mode (Figure 3c). This indicates that the phosphors can be more efficiently excited with blue light at higher temperature. This can be explained from positioning of the excited states of Eu$^{3+}$ in the band-gap of the NaGd(WO$_4$)$_2$ host lattice. The bottom of the conduction band of NaGd(WO$_4$)$_2$ lie closer to the $^5L_6$ excited state than $^5D_2$ state. The $^5L_6$ electrons may be activated to the conduction band under thermal fluctuation, leading to decreased $^7F_{0.1} \rightarrow ^5L_6$ (394 nm) peak intensity with increasing temperature.

Figure 4 presents the effects of temperature on the photoluminescence emission of Na(Gd,Eu)(WO$_4$)$_2$ phosphors. Figure 4a shows the emission spectra recorded at representative temperatures of RT, 150, and 275°C, and all the spectra measured with a temperature interval of 25°C in 25–275°C range were shown in Figure 54. Under 394 nm excitation, characteristic $^5D_1 \rightarrow ^7F_j$ (j = 1-4) emissions of Eu$^{3+}$ activator were obtained as labeled in the figure. The $^5D_0 \rightarrow ^7F_2$ (~615 nm) forced electric dipole transition is remarkably stronger than the $^5D_0 \rightarrow ^7F_1$ (~591 nm) parity-allowed magnetic dipole transition, since Eu$^{3+}$ would reside at the noncentrosymmetric site in the triclinic NaGd(WO$_4$)$_2$ lattice. The International de L’Eclairage (CIE) chromaticity coordinates estimated from the luminescence spectra shift from (0.67, 0.33) to (0.65,0.35) via (0.66, 0.34) with increasing temperatures (Figure 55). No new emission band and peak shift were observed at a higher measurement temperature. The emission intensity was gradually weakened as commonly observed in the phosphor field with the increasing temperatures. At 150°C and the highest measurement temperature of 275°C, the intensities of the main emission (615 nm) dropped to ~35 and 4% of the initial values, respectively (Figure 4b). It is also noted in Figure 4b that the emission peaks basically showed same intensity loss rates with increasing
temperature (Figure 4b), which indicated that the current DC phosphor cannot be employed as a probe for optical thermometry via fluorescence intensity ratio (FIR) mode. The activation energy (ΔE) of thermal quenching can be calculated from the Arrhenius Equation shown below [31,32]:

\[ S_R = \frac{d(FIR)}{d(T)} = \frac{FIR - C}{FIR} \times \frac{\Delta E}{kT^2} \]

In the equation, \( I_0 \) and \( I \) represents the emission intensities of the Na(Gd,Eu)(WO₄)₂ phosphor at room temperature and the testing temperature, respectively, \( k \) is the Boltzmann constant (8.617 × 10⁻⁵ eV), \( c \) a pre-exponential constant, \( T \) the absolute temperature in Kelvin, and \( \Delta E \) the activation energy of thermal quenching. Figure 4c displays the \( \ln([I_0/I]-1) \) versus 1/kT plots converted from the Arrhenius Equation and the linear fitting of the data yielded \( \Delta E \) values of 0.352, 0.312, 0.299, and 0.294 eV for the emissions of 655, 615, 702, and 591 nm, respectively.

Temperature-dependent (RT–275°C) fluorescence decay analysis were taken for the dominantly strong red emission of Eu³⁺ (⁴D₀ → ⁵F₂) transition, ~615 nm; \( \lambda_{ex} = 395 \) nm. Fitted decay curve measured at 275°C is shown in Figure 5a as example and all the other curves measured with a temperature interval of 25°C in 25–275°C range were shown in Figure 5b. It was found that the decay curves can be well fitted with the second-order exponential equation \( I(t) = A_1 \exp(-t/\tau_1) + A_2 \exp(-t/\tau_2) \), where \( \tau_1 \) and \( \tau_2 \) are the decay time of exponential components, \( I(t) \) is the fluorescence intensity, and \( A_1 \) and \( A_2 \) are pre-exponential constants. Table S2 summarized the derived \( \tau_1, \tau_2, A_1, A_2 \), and the chi-square values of the fittings. The decay curves were fitted with two exponential which means that two types of luminescent centers participate in the luminescence process. The crystal structure of NaGd(WO₄)₂ host material only has a single S4 site for Eu³⁺ doping [33]. The long lifetime components (\( \tau_2 \)) can be assigned to the decay time of the Eu³⁺ ions located in the particle bulk in S4 site, which were less affected by surface effects, and the shorter lifetime components (\( \tau_1 \)) are associated with the decay time of the active ions located near the particle surface, where the presence of impurities led to the luminescence quenching through multiphononic processes [17,34]. The average lifetime (\( \tau^* \)) of the phosphors can be calculated with the formula \( \tau^* = \frac{A_1\tau_1 + A_2\tau_2}{A_1 + A_2} \) [35] and results are shown in Table S2. The derived average lifetimes decreased from 683.2721 μs to 527.9371 μs over temperature range from RT to 275°C. Figure 5b shows the tendency of average lifetime of the 615 nm emission versus temperatures. It was encouraging to find that the lifetime of the ⁴D₀ → ⁵F₂ emission in the studied temperature range following an exponential decrease mode with increased temperature as shown in Figure 5b. A 0.62134 μs/°C decrease on average was found. It thus indicates that the Na(Gd,Eu)(WO₄)₂ phosphors obtained in this work can served as luminescence lifetime-based nanothermometry i.e. using the luminescence lifetime of Eu³⁺ instead of the emission intensity ratios.
3.3. Fluorescence intensity ratio (FIR)-based luminescent thermometry of Na(Gd, Yb, Er)(WO₄)₂

The Na(Gd₀.⁹⁷Yb₀.⁰²Er₀.⁰₁)(WO₄)₂ up-conversion (UC) phosphor was obtained from (Gd₀.⁹⁷Yb₀.⁰²Er₀.⁰₁)₂-241 precursor via 24 h of reaction at 200°C under WO₄³⁻/Ln³⁺ molar ratio R = 2.5 following the synthesized procedure of its DC counterpart Na(La, Eu)(WO₄)₂ and the XRD patterns and the Rietveld refinement results of the precursor and the product are shown in Figure 6. The Rietveld refinement yielded stable results and acceptable reliability factors which indicate the purity of the products and further confirm the proposed sacrificial template method was repeatable and applicable to the synthesis of UC phosphors. The derived structure parameters are tabulated in Table 1, from where it can be seen that the Yb-Er co-doped products have smaller cell dimensions and volumes than corresponding pure Gd–241 [24] and NaGd(WO₄)₂ (JCPDS card 00-025-0829). The results prove the doping of smaller Yb³⁺-Er³⁺ into the host and the completion of phase conversion process, since Gd³⁺, Er³⁺ and Yb³⁺ have the successively smaller ionic radii of 1.053, 1.004, and 0.985 Å (coordination number, CN = 8), respectively [26].

Figure 7 shows the micro-images of the Yb³⁺-Er³⁺ pair co-doped layered precursor and its derived rare earth double tungstate. It is seen that compared with the morphologies of Eu³⁺ doped samples (Figures 2a and 2f), Yb³⁺-Er³⁺ doping doesn’t appreciably change the micro-morphology. Similar aggregated sphere morphology of the precursor with microplate the basic unit (Figure 7b) and micro-squared Na(Gd₀.⁹⁷Yb₀.⁰²Er₀.⁰₁)(WO₄)₂ were observed.

Figure 6. Observed (black), calculated (red), and difference (gray) XRD profiles for the (Gd₀.⁹⁷Yb₀.⁰²Er₀.⁰₁)-LHₙ (a) and Na(Gd₀.⁹⁷Yb₀.⁰₂Er₀.⁰₁)(WO₄)₂ up-conversion phosphors (b) synthesized via 24 h of reaction at 200°C under WO₄³⁻/Ln³⁺ molar ratio R = 2.5. The green tick marks denote the positions of Bragg reflection.

The up-conversion spectra of Na(Gd₀.⁹⁷Yb₀.⁰₂Er₀.⁰₁)(WO₄)₂ taken under 978 nm excitation with the

**Table 1.** Structure parameters of the (Gd₀.⁹⁷Er₀.⁰₁Yb₀.⁰₂)-241 precursor and the Na(Gd₀.⁹⁷Er₀.⁰₁Yb₀.⁰₂)(WO₄)₂ product, with those of Gd–241 [24] and NaGd(WO₄)₂ [JCPDS card 00-025-0829] included for comparison.

| Sample                  | Sp.Gr. | a, Å  | b, Å  | c, Å  | β, ° | V, Å³ |
|------------------------|--------|-------|-------|-------|------|-------|
| Gd-241                 | C2/m   |       |       |       |      |       |
| (Gd₀.⁹⁷Er₀.⁰₁Yb₀.⁰₂)-241| C2/m   | 16.6362(8) | 3.7312(1) | 6.2530(3) | 90.064(8) | 388.14(3) |
| NaGd(WO₄)₂             | I41/a  | 5.243 |       | 11.368 |      | 312.5 |
| Na(Gd₀.⁹³Er₀.⁰₁Yb₀.⁰₂)(WO₄)₂ | I41/a  | 5.2375(4) | -     | 11.3598(5) | -    | 311.61(5) |
excitation power varying from 0.7 to 1.5 w and the logarithmic fitting of the UC intensity against excitation power were shown in Figure S7. The up-conversion spectra (Figure S7a) observed in this work are similar to NaGd(WO₄)₂:Yb³⁺/Er³⁺ micro octahedral particle [14] and NaGd(WO₄)₂:Yb³⁺/Er³⁺ micro discs [35]. Sharp and splitting emissions originate from ²H₁₁/₂ → ⁴I₁₅/₂ (520/524/530 nm), ⁵S₅/₂ → ⁴I₁₅/₂ (543/552 nm), and ⁴F₉/₂ → ⁴I₁₅/₂ (656/670 nm) transitions of Er³⁺ were observed, with the peak at 552 nm (⁵S₅/₂ → ⁴I₁₅/₂) being the strongest [21,36]. However, the spectral features are different with nanorods of NaY/WO₄):Er³⁺:Yb³⁺ with ²H₁₁/₂ → ⁴I₁₅/₂ emissions the stronger group under 978 nm excitation. It was suggested that the spectra difference related to the morphological of the products. The phosphor shows very stable color coordinates at (0.26, 0.72) typical of a vivid green color (Figure S7a, Figure S8), with varying excitation power. The number of photons required to excite electrons from the ground state to emitting state (n) can be determined from the slope of the log–log plot of I_em and P (Figure S7b). The n values (slopes) of about 2.21–2.50 suggest that a two-photon mechanism is basically responsible for the observed UC luminescence.

By referring other Yb/Er codoped tungstate systems [8,10,13,37], the energy level diagram and up-conversion process of NaGd(WO₄)₂:Yb₀.₀₂Er₀.₀₁(WO₄)₂ were constructed and shown in Figure 8. The UC process involves excited state absorption (ESA), energy transfer (ET), and non-radiative (NR) relaxation. The following reactions happened during the process: (1) Excited state absorption (ESA): [ESA; ²F₇/₂(Yb³⁺) + hv (978 nm) → ²F₅/₂(Yb³⁺)]; or [ESA; ⁴I₁₅/₂(Er³⁺) + hv (978 nm) → ⁴I₁₁/₂(Er³⁺)]. Upon excitation with 978 nm laser, the ground state electrons of Yb³⁺(²F₇/₂) and Er³⁺(⁴I₁₁/₂) are pumped to the excited state ²F₅/₂ for Yb³⁺ and ⁴I₁₁/₂ for Er³⁺; (2) Via the following energy transfer process [ET₁; ²F₅/₂(Yb³⁺) + ⁴I₁₁/₂(Er³⁺) → ²F₇/₂(Yb³⁺)] + ⁴I₁₁/₂(Er³⁺), the ⁴I₁₁/₂ ground state electrons of Er³⁺ can also be excited to ⁴I₁₅/₂ level. In fact, compared with the ESA process, the energy transfer from Yb³⁺ to Er³⁺ dominates the real excitation process since the absorption cross section of Er³⁺ is much smaller than that of Yb³⁺ at ~980 nm [38]; (3) excitation of Er³⁺ electrons from ⁴I₁₁/₂ to ²F₇/₂ with energy transfer of a second laser photon [ET₂; ²F₅/₂(Yb³⁺) + ⁴I₁₁/₂(Er³⁺) → ²F₇/₂(Yb³⁺) + ²F₇/₂(Er³⁺)]; (4) NR relaxation of most of the ²F₇/₂ electrons to the ⁴H₁₁/₂, ⁴S₅/₂ and ⁴F₉/₂ levels (NR; ²F₇/₂(Er³⁺) → ⁴H₁₁/₂(Er³⁺); ²F₅/₂(Er³⁺) → ⁴S₅/₂(Er³⁺); ²F₇/₂(Er³⁺) → ⁴F₉/₂(Er³⁺)); (5) [²H₁₁/₂ → ⁴I₁₅/₂ + hv (520/524/530 nm) ⁵S₅/₂ → ⁴I₁₅/₂ + hv (543/552 nm) ²F₉/₂ → ⁴I₁₅/₂ + hv (656/670 nm)], back-jumping of the ²H₁₁/₂, ⁴S₅/₂, and ²F₉/₂ electrons to the ⁴I₁₅/₂ ground state produce UC luminescence.

Figure 9 presents the effects of temperature on the photoluminescence up–conversion emission of NaGd(WO₄)₂:Yb₀.₀₂Er₀.₀₁(WO₄)₂ phosphors. Figure 9a shows the emission spectra recorded at representative temperatures of RT, 150, and 275°C, and all the spectra measured with a temperature interval of 25°C in 25–275°C range were shown in Figure S9. New emission peak and peak position shift were not observed at a higher measurement temperature. The International de L’Eclairage (CIE) chromaticity coordinates estimated from the luminescence spectra shift from (0.26, 0.72) to (0.19, 0.76) with increasing temperatures (Figure S10), which indicates that more green ingredients can be observed in the phosphors. This corresponding well with the fact that the green to red emission ratio trends to increase with increasing temperature. It is seen from Figure 9b that the ²H₁₁/₂→⁴I₁₅/₂ green emissions (520/524/530 nm) gain intensity with initial increasing temperature and reach their maxima till 373 K. Intensity decrease was observed after 373 K and the intensity of 524, 520, 530 nm drop to their initial values at around 489 K, 523 K, 473 K, respectively. At the highest measurement temperature of 548 K (275°C), the intensity of 524, 520, 530 nm drops to 67.7%, 87.9%, and 58.6% of the initial value, respectively. While the ⁵S₅/₂→⁴I₁₅/₂ (543/552 nm) green and ²F₉/₂→⁴I₁₅/₂ (656/670 nm) red emissions lose intensity.
right after the temperature increased and the intensity loss of the emission is mostly due to enhanced non-radiative relaxation by intensified lattice vibration at a higher temperature [8,39]. At the highest measurement temperature of 548 K (275°C), the intensity of 543, 552, 656, and 670 nm drops to 17.1%, 14.4%, 28.8%, and 26.3% of the initial value, respectively. It is also seen that the $^4\text{H}_{11/2} \rightarrow ^4\text{I}_{15/2}$ emission of the Na(Gd$_{0.97}$Yb$_{0.02}$Er$_{0.01}$)(WO$_4$)$_2$ microsquares show quite different intensity loss rates compared with $^4\text{S}_{3/2} \rightarrow ^4\text{I}_{15/2}$ and $^4\text{F}_{9/2} \rightarrow ^4\text{I}_{15/2}$ emissions with increasing temperature (Figure 9b) which indicates that the UC phosphors may thus be employed as a probe for luminescent thermometry via fluorescence intensity ratio (FIR) mode.

The thermally coupled $^2\text{H}_{11/2}$ and $^4\text{S}_{3/2}$ levels were analyzed to check the temperature-sensing performance. Figure 10a displays the trend of $I_{520}/I_{552}$ FIR with measurement temperature. It was found that the $I_{520}/I_{552}$ FIR continuously increases with increasing temperature in single–exponential mode by equation of FIR ($I_{520}/I_{552}$) = 8.379exp($-1873.45/5 + 0.41$ (T = 298-548 K). The fitting equation corresponding well with the Boltzmann distribution, which can be described as FIR = Nexp($-\Delta E/kT$) + C for thermally coupled $^2\text{H}_{11/2}/^4\text{S}_{3/2}$ levels, where N is a proportionality constant, $\Delta E$ is the energy gap between the $^2\text{H}_{11/2}$ and $^4\text{S}_{3/2}$ levels, k is 0.695 K$^{-1}$ cm$^{-1}$ (the Boltzmann constant), T is the absolute temperature, and C is the constant. This indicates that the Na(Gd$_{0.97}$Yb$_{0.02}$Er$_{0.01}$)(WO$_4$)$_2$ microsquares can indeed be employed as a luminescent thermometer for temperature sensing via FIR mode. The $I_{520}/I_{552}$ ratios almost keep unchanged when the material is excited by 980 nm source of different power at the room temperature (Figure S11). This further indicated that the phosphor can be used for luminescence thermometry. The quality of optical thermometry can be quantified via absolute sensitivity ($S_A$) and relative sensitivity ($S_R$) and Figure 10b shows the results of $S_A/S_R$ derived from the following equations [8,9]:

$$S_A = \left| \frac{d(FIR)}{d(T)} \right| = (FIR - C) \times \frac{\Delta E}{kT^2} \tag{1}$$

$$S_R = \left| \frac{d(FIR)}{d(T)} \right| = \frac{FIR - C}{FIR} \times \frac{\Delta E}{kT^2} \tag{2}$$

It is clear from the results shown in Figure 10b that both $S_A$ and $S_R$ values remain on the same order across the temperature range of measurement. $S_A$ and $S_R$ have their respectively maximum value of $\sim 174 \times 10^{-4}$ K$^{-1}$ and $80 \times 10^{-4}$ K$^{-1}$ at 548 and 373 K. The maximum $S_A$ value obtained this work is higher than similar tungstate systems [13,44–46] and much higher than other systems [8,44–47] as shown in Table 2.

The minimum detectable temperature change of an optical thermometer was quantified by temperature
Table 2. A summary of $S_A$ and $S_R$ values, electronic transitions and temperature sensitivity ranges for some typical temperature sensing phosphors.

| Ion pair | Host          | Transition/wavelength         | Range (K) | $S_A(K^{-1}) \times 10^{-4}$ | $S_R(K^{-1}) \times 10^{-4}$ | Ref |
|----------|---------------|-----------------------------|-----------|-------------------------------|-----------------------------|-----|
| Yb$^{3+}$-Er$^{3+}$ | NaGd(WO$_4$)$_2$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 300-550 | 174 (548 K) | 80 (373 K) | This work |
| Yb$^{3+}$-Er$^{3+}$ | NaGd(WO$_4$)$_2$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 300-550 | 260 (300-550 K) | 96 (298 K) | This work |
| Yb$^{3+}$-Er$^{3+}$ | NaY(WO$_4$)$_2$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 133-773 | 112 (515 K) | - | [13] |
| Yb$^{3+}$-Er$^{3+}$ | NaGd(WO$_4$)$_2$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 298-383 | - | 120 (310 K) | [31] |
| Yb$^{3+}$-Er$^{3+}$ | NaGd(WO$_4$)$_2$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 293-573 | 114.96 (453 K) | - | [35] |
| Er$^{3+}$ | NaGd(WO$_4$)$_2$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 298-573 | 126 (510 K) | - | [36] |
| Yb$^{3+}$-Er$^{3+}$ | NaY(WO$_4$)$_2$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 303-523 | 97 (493 K) | - | [37] |
| Yb$^{3+}$-Er$^{3+}$ | NaY(WO$_4$)$_2$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 30-300 | 61 (300 K) | - | [38] |
| Yb$^{3+}$-Er$^{3+}$ | KY(WO$_4$)$_2$, KWO$_4$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 83-663 | 147 (463 K) | - | [39] |
| Yb$^{3+}$-Er$^{3+}$ | Sr$_2$YPO$_4$, | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 298-573 | 50.7523 K | 1026.5 T$^2$ | [8] |
| Er$^{3+}$ | Na(WO$_4$)$_2$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 300-550 | 97 (510 K) | - | [14] |
| Er$^{3+}$ | Y$_2$O$_3$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 93-613 | 44 (427 K) | 886.08 T$^2$ | [40] |
| Er$^{3+}$ | CaLa$_2$Si$_2$O$_8$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 293-573 | 39.5100 K | 1008 T$^2$ | [41] |
| Er$^{3+}$ | β-NaYF$_4$ | $^{2}H_{11/2} \rightarrow ^{2}S_{5/2}$ | 303-573 | 48.4515 K | 1025.8 T$^2$ | [42] |

resolution ($\delta T$ or temperature uncertainty) and can be assayed via the following equation:

$$\delta T = \frac{1}{S_R} \frac{\delta \text{FIR}}{\text{FIR}}$$

(3)

where $\delta \text{FIR}/\text{FIR}$ is the relative uncertainty of FIR. It can be seen from the equation that the value of $\delta T$ mainly depends on relative sensitivity $S_R$ and experimental setup. The $S_R$ value has been derived from Figure 10b and for experimental setup, the $\delta \text{FIR}/\text{FIR}$ reaches the typical value of 0.03% for the FLS 1000 spectrophotometer used in this work [2], [9]. The minimum $\delta T$ of $I_{520}/I_{552}$ FIR was thus estimated to be $\sim$0.037 K at 373 K, which is significantly superior to reported thermometric phosphor systems such as Sr$_2$Ce(Po$_4$)$_2$:0.4Tb$^{3+}$ (0.078 K) [9], Sc$_2$O$_3$:Eu$^{2+}$/Eu$^{3+}$ (0.08 K) [48] and GdVO$_4$:Sm$^{3+}$ (0.14 K) [49].

The non-thermally coupled $^4H_{11/2}$ and $^4F_{9/2}$ levels were also analyzed to check the temperature-sensing performance. Figure 11(a) displays the dependence of $I_{520}/I_{670}$ FIR on absolute temperature. The $I_{520}/I_{670}$ ratios of the phosphors show linear fitting at different temperatures. The excitation power dependence of the ratios was also checked at room temperature and it was found that unlike the above discussed $I_{520}/I_{552}$ ratio the green to red emission ratio of $I_{520}/I_{670}$ fluctuated slightly in the measured power range (Figure S11). This indicates that the $I_{520}/I_{670}$ ratio is less stable than $I_{520}/I_{552}$ ratio when used for luminescence thermometry. According to the linear fitting in Figure 11(a) and equation (1), the $^3H_{11/2}$ and $^4F_{9/2}$ levels have an $S_A$ of $260 \times 10^{-4}$ K$^{-1}$, which is higher than the maximum $S_A$ of $I_{520}/I_{552}$ FIR (254 $\times 10^{-4}$ K$^{-1}$, Figure 10(b)). The $S_A$ values were obtained via equation (2) and it was found that $S_R$ decreased with increasing temperature and has maximum value of $96 \times 10^{-4}$ K$^{-1}$ at 298 K. The minimum temperature resolution $\delta T$ of $I_{520}/I_{670}$ FIR was calculated to be $\sim$0.037 K at 298 K from equation (3), which is nearly the same with $\sim$0.037 K at 373 K for thermally coupled $^4H_{11/2}$ and $^4S_{3/2}$ levels.

3.4. Fluorescence lifetime-based luminescent thermometry of Na(Gd,Yb,Er)(WO$_4$)$_2$

Temperature-dependent (RT–275° C) fluorescence decay analysis were taken for the strongest $^4S_{3/2} \rightarrow ^4I_{15/2}$ transition ($\sim$552 nm; $\lambda_{\text{ex}} = 980$ nm) of Na(Gd$_{0.97}$Yb$_{0.02}$Er$_{0.01}$)(WO$_4$)$_2$ phosphors. Fitted decay curve measured at 275°C is shown in Figure 12a as example and all the other curves measured with a temperature interval of 25°C in 25–275°C range were shown in Figure S12. It was found that the $^4S_{3/2} \rightarrow ^4I_{15/2}$ transition decay in second-order exponential mode by equation (2) ($t = A_1 \exp(-t/T_1) + A_2 \exp(-t/T_2)$). The shorter ($T_1$) and longer ($T_2$) lifetime components was assigned to luminescent center of S4 site in the particle bulk and active ions located near the particle surface, respectively, as analyzed before in Figure 5a. The derived $T_1$, $T_2$, $A_1$, and $A_2$ values and the chi-square factor of fitting are tabulated in Table S3. The average lifetime ($\tau^*$) was

Figure 11. The $I_{520}/I_{670}$ FIR (a) and the $S_A$ and $S_R$ of the $I_{520}/I_{670}$ FIR (b) as function of measurement temperature for Na(Gd$_{0.97}$Yb$_{0.02}$Er$_{0.01}$)(WO$_4$)$_2$ phosphors.
calculated with the formula $\tau^* = \frac{A_1 \tau_1^0 + A_2 \tau_2^0}{A_1 + A_2}$. Figure 12b shows the tendency of average lifetime of the 552 nm emission ($^4S_{3/2} \rightarrow ^4I_{15/2}$ transition) versus temperatures (25-275°C). It can be found in Figure 12b that the fluorescence lifetimes of 552 nm emission decrease almost linearly from 77.42409 μs (RT) to 70.3833 μs (275°C) with increasing temperature, which is different with the exponential decreased tendency of average lifetime for 615 nm emission of NaGd0.97Yb0.02Er0.01(WO4)2 (Figure 4). In this case, the thermal sensitivity of the probes can be roughly estimated by lifetime thermal coefficient ($\alpha_\tau$) which was defined as the slope of the normalized lifetime vs temperature linear curve [12]. From the linear fit (Figure 12b), lifetime thermal coefficient ($\alpha_\tau$) of 3.80°C⁻¹ was obtained. It thus proved that the Yb-Er codoped NaGd(WO4)2 can be applied as thermal probe via the mode of FIR or LF.

**4. Conclusions**

Rare earth double tungstate NaGd(RE)(WO4)2 (RE = Eu/Yb-Er) were successfully obtained from layered rare earth hydroxide precursor via sacrificial reaction. The temperature-sensing performances of NaGd(RE)(WO4)2 with both fluorescence intensity ratio (FIR) and fluorescence lifetime (FL) mode were systematically investigated by analyzing the temperature-dependent down-conversion (DC) and up-conversion (UC) luminescence properties. The main conclusions are summarized as follows:

1. Phase-pure NaGd(RE)(WO4)2 (RE = Eu/Yb–Er) can be directly produced in the absence of any organic additive by hydrothermally reacting an aqueous mixture of (Gd(RE)(OH))2SO42H2O precursors and Na3WO4 at 180–200°C for 24 h under a WO42-/Ln3+ molar ratio of 2.5.

2. The NaGd0.95Eu0.05(WO4)2 DC phosphors were found to be well capable of sensing temperature via fluorescence lifetime mode, and a 0.62134 μs/°C decrease on average was found over the measured temperature range (RT–275°C). The DC phosphor cannot sense temperature via fluorescence intensity ratio (FIR) mode.

3. The NaGd(RE)(WO4)2 UC phosphors were capable of sensing temperature via both FL mode and FIR mode. The NaGd(RE)(WO4)2 has maximum absolute sensitivity ($S_\tau$) and relative sensitivity ($S_\phi$) of 174 × 10⁻⁴ K⁻¹ (548 K) and 80 × 10⁻⁴ K⁻¹ (298 K) for the thermally coupled $^2H_{11/2}$ $^4S_{3/2}$ levels ($I_{520}/I_{552}$) and maximum $S_\phi$ of ~260 × 10⁻⁴ K⁻¹ (300–550 K) and 96 × 10⁻⁴ K⁻¹ (298 K) for the non thermally coupled $^2H_{11/2}$ $^4S_{3/2}$ energy levels ($I_{520}/I_{570}$). Lifetime thermal coefficient ($\alpha_\tau$) of 3.80°C⁻¹ was obtained for NaGd(RE)(WO4)2 when sensing temperature via FL mode.

**Disclosure statement**

No potential conflict of interest was reported by the author(s).

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