Multi-Color Luminescence Transition of Upconversion Nanocrystals via Crystal Phase Control with SiO₂ for High Temperature Thermal Labels

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Upconversion nanocrystals (UCNs)-embedded microarchitectures with luminescence color transition capability and enhanced luminescence intensity under extreme conditions are suitable for developing a robust labeling system in a high-temperature thermal industrial process. However, most UCNs based labeling systems are limited by the loss of luminescence owing to the destruction of the crystalline phase or by a predetermined luminescence color without color transition capability. Herein, an unusual crystal phase transition of UCNs to a hexagonal apatite phase in the presence of SiO₂ nanoparticles is reported with the enhancements of 130-fold green luminescence and 52-fold luminance as compared to that of the SiO₂-free counterpart. By rationally combining this strategy with an additive color mixing method using a mask-less flow lithography technique, single to multiple luminescence color transition, scalable labeling systems with hidden letters-, and multi-luminescence colored microparticles are demonstrated for a UCNs luminescence color change-based high temperature labeling system.

Spectrally tunable microarchitectures are effective information media because they act as carriers of luminescent materials that change their properties in response to external stimuli. However, it is challenging to meet the stringent requirements of practical applications, such as in optical labeling systems under high-temperature conditions. Thus, robust optical system with high thermal stability is required to reliably ensure product identification and the valuable information under high-temperature thermal processes such as sterilization and cleansing of the steel, ceramic, and aerospace industries. The required temperature for labeling such products ranges from 600 up to 1600 °C, and the optical labeling system should survive these conditions for several hours. Downconversion ceramic materials are actively researched for practical applications due to their robust luminescence properties, but the ultra-high temperature (1000–1550 °C) pre-annealing process for the production of application systems is not suitable for actual industrial applications, and the system production cost is also very high because they require multiple optical filters to selectively observe the emission colors.

Recently, taking advantage of the technological advances in microfabrication techniques and the luminescence properties of upconversion nanocrystals (UCNs; e.g., anti-Stokes effect, high photochemical stability, and the absence of background autofluorescence), UCNs-based labeling systems that can deliver the desired information in micro-sized solid architectures have been developed in a scalable manner. Examples of such systems include latent fingerprinting, quick response codes, anti-counterfeiting systems, and luminescence displays. The use of upconversion microparticles as a multiplexed labeling system overcomes many of the current limitations, such as a low information storage capacity, the requirement for costly decoders, and precise loading of optical dyes. Many researches have been conducted to improve the inefficient upconversion process of UCNs by using surface plasmon resonance (SPR) of metal nanostructure and by increasing the radiative decay rate of the emitters. However, a majority of UCNs systems have a critical limitation in that they lose their luminescence property and change their multiple luminescence colors to weak red upon exposure to high temperature environments owing to the collapse of their inherent crystal structures. Thus, a facile multiple luminescence color transition strategy that shows enhanced luminescence intensities and solid reading ability in a high temperature range would be desirable for the practical application.

Herein, we report a strategy to modulate the predetermined luminescence color of lanthanide-ion doped UCNs with large enhancement in luminescence intensity upon heating with silica nanoparticles (SiO₂ NPs). The key idea behind this work is...
incorporating SiO$_2$ NPs into the crystal lattice of lanthanide-ion doped UCNs at an elevated temperature to create a new crystal phase, hexagonal apatite, which has higher upconversion efficiency. The resulting hexagonal apatite UCNs show enhanced emission intensity, ideally higher than that of the SiO$_2$ NPs-free counterpart. Extending the use of this optical property changing principle, we propose an optical labeling system with cryptographic multiple luminescence color, patterns, and letters for commercial high-temperature thermal processes by combining an additive color mixing method and a digital micro-mirror device (DMD)-based mask-less lithography technique.

Figure 1 illustrates the phase-transition strategy for developing lanthanide-doped UCNs with enhanced multi-spectral luminescence intensity via a SiO$_2$ NPs involved annealing process. In this study, we prepared hexagonal rod-shaped yellow emissive UCNs by a conventional hydrothermal method$^{[13]}$ (see Experimental Section for details and Note S1, Supporting Information). We studied three different phase transition processes: hexagonal NaREF$_4$ to cubic NaREF$_4$ phase with low upconversion efficiency (yellow to red), hexagonal NaREF$_4$ to hexagonal apatite for achieving enhanced luminescence intensity (yellow to green), and cubic NaREF$_4$ to hexagonal apatite to change the low-efficiency red to high-efficiency green luminescence. Note that RE indicates rare earth elements (e.g., Y, Gd, Yb, Er, and Tm). In the case of yellow emissive UCNs, the hexagonal-to-cubic phase transition results in a decrease of the upconversion luminescence at 550 nm (color from yellow to red) and lowering of the luminescence efficiency (Figure 1a), which is consistent with previous results.$^{[38,39]}$ In contrast, UCNs including SiO$_2$ NPs converts to hexagonal apatite phase with large enhancement of the luminescence intensity at 550 nm compared to that of the annealed cubic UCNs (Figure 1b). Therefore, in the case of cubic to hexagonal apatite phase transition, we reason that SiO$_2$ NPs might enable the recuperation of the cubic phase UCNs to the hexagonal apatite phase accompanied by enhanced luminescence efficiency (Figure 1c). We studied various types of metalloid oxides (Al$_2$O$_3$, Sb$_2$O$_3$, As$_2$O$_3$, GeO$_2$, and SiO$_2$) and they exhibited enhanced luminescence intensity upon the same thermal process (Figure S2, Supporting Information). SiO$_2$ NPs are the most cost-effective, easy-to-use material and showed most uniform dispersion property in the photocurable resin used in this study. Therefore, for a precise quantitative optical analysis of the investigated unique crystal phase transition, all further experiments were conducted using SiO$_2$ NPs.

To explore the efficiency of upconversion luminescence process, we prepared yellow ($\beta$-NaYF$_4$:Gd$^{3+}$, Yb$^{3+}$, Er$^{3+}$ (30/30/2 mol%)) and blue ($\beta$-NaYF$_4$:Gd$^{3+}$, Yb$^{3+}$, Tm$^{3+}$ (30/18/0.2 mol%)) emissive UCNs-embedded polyurethane acrylate (PUA) microparticles using an automated DMD-based mask-less flow lithography technique with adjusting the presence or absence of SiO$_2$ NPs (Figure S3, Supporting Information) and annealed them at different temperature (see Experimental Section for details). For the convenience of quantitative analysis of the effect of SiO$_2$ NPs on the luminescent properties of UCNs, photocurable PUA (Mins-311) was used to synthesize a desired
shape of microstructure with good dispersion and transmittance property and to quantitatively measure the luminescence characteristics after thermal process (Figure S4, Supporting Information). Upon annealing at 300 °C, the luminescence color of the UCNs-embedded microparticles with/without SiO2 NPs changed to orange (Figure 2a,b and TGA in Note S2, Supporting Information). As the annealing temperature exceeded 700 °C, a weak red luminescence was observed for the system without SiO2 NPs. However, surprisingly, the UCNs-embedded PUA microparticles containing SiO2 NPs exhibited bright green luminescence when the annealing temperature was increased to 900 °C, and the intensity of the emission band at 520 and 550 nm wavelength increased more than 130 times as compared to that of the annealed SiO2-free counterpart (Figure 2d). Moreover, the ratio of 550 and 660 nm luminescence intensities was increased in the UCNs with SiO2 NPs after the annealing process.
Figure 3. Change in the crystal structure and morphology of UCNs with and without SiO2 NPs in PUA microparticles as a function of the annealing temperature. XRD patterns of a) pristine UCNs and b) UCNs with SiO2 NPs at different annealing temperatures. SEM images, cross-sectional TEM images, and SAED patterns of c) pristine UCNs, d) pristine UCNs with SiO2 NPs, e) UCNs annealed at 900 °C, and f) UCNs with SiO2 NPs annealed at 900 °C.

Figure S9. Supporting Information) and white-light-emitting microparticles of YBS, GWS, and BWS were successfully synthesized (Figure 2g). As in Figure 2g, the white luminescence of YBS, GWS, and BWS microparticles were changed to a broad range of multiple colored particles of red, green, and blue with strong luminescence intensity under thermal processing (Luminescence spectra in Figures S10 and S11, RGB values in Table S4, and Note S3, Supporting Information).

To quantify the effect of the annealing temperature on UCNs with/without SiO2 NPs, the morphologies of the annealed microparticles containing hexagonal NaREF4 UCNs (β-NaYF4:Gd3+, Yb3+, Er3+ (30/30/2 mol%), JCPDS file number 01-072-4799) with and without SiO2 NPs were analyzed at different temperatures. Similar reflections were observed in the X-ray diffraction (XRD) patterns for both systems (with/without SiO2 NPs) annealed at temperatures below 600 °C, indicating that annealing at a low temperature is ineffective for changing the crystal phase of the UCNs (XRD data in Figures S12 and S13, Supporting Information). Upon increasing the annealing temperature to >700 °C (Figure 3a), the crystal phase transitioned to the typical cubic NaREF4 phase (JCPDS file number 01-072-4799) in the system without SiO2 NPs. However, the UCNs in the system containing SiO2 NPs transformed to a new crystal phase of hexagonal apatite phase (JCPDS file number 00-035-0404) after annealing at 900 °C (Figure 3b). Energy-dispersive X-ray spectroscopy (EDS) indicated the Na:F ratio to be ≈1:1, which is consistent with the X-ray photoelectron spectroscopy (XPS) results (see EDS and XPS data in Figure S14, Supporting Information). Therefore, the stoichiometric composition of
the UCNs in the system with SiO₂ NPs that underwent phase transition was assumed to be NaₓYₓ(SiO₂)ₙF₂. The difference in the surface morphology depending on the presence or absence of SiO₂ NPs therefore indicated the evolution of new crystal phases after annealing at 900 °C (Figure 3c-f, and SEM in Figure S15, Supporting Information). Note that PUA has no influence on the morphology, crystal structure, and luminescent property of UCNs with or without SiO₂ NPs before and after thermal decomposition by the annealing process (Figure S16, Supporting Information). In particular, there exists a large morphological difference at the surface of annealed microparticles with only UCNs and a mixture UCNs and SiO₂ NPs (Figure 3d,f). The surface of the specimen with SiO₂ NPs is much smoother than that without them. The smooth morphology of the annealed system with UCNs and SiO₂ NPs can be ascribed to the enhanced coarsening of grains with diffusional interaction between SiO₂ NPs and UCNs through Ostwald ripening, whereby Si and O diffuse into the crystal lattice of the UCNs. In general, the diffusional interaction occurs in order to reduce the total energy of the system by reducing the total interfacial area of the particles. Further, the diffusional interaction increases the rate of coarsening through Ostwald ripening, whereby larger particles grow at the expense of smaller ones. According to the Gibbs–Thomson relation, the atomic diffusion occurs from small particles to a larger one, resulting in the growth of the larger particle at the expense of the smaller one (Note S4, Supporting Information). These results are consistent with the results of Fourier transform infrared spectroscopy (FT-IR) investigations (FT-IR in Figure S17, Supporting Information). We also confirmed that the cubic UCNs transformed to the hexagonal apatite UCNs as the weight ratio of the SiO₂ NPs to UCNs (SiO₂/UCNs) is increased from 0.05 to 0.6 (see XRD data in Figure S18, Supporting Information).

Another important aspect of our finding is that the luminescence of cubic UCNs recovered by transforming crystal phase to the hexagonal apatite phase through annealing process with SiO₂ NPs (see XRD data in Figure S19 and upconversion luminescence data in Figure S20, Supporting Information). Further, it is clear that the hexagonal apatite UCNs phase is formed regardless of the initial phase of the UCNs (hexagonal NaREF₄, or cubic NaREF₄) and two factors, annealing temperature and SiO₂ NPs, play crucial roles in this process. The SiO₂ NPs-involved annealing process described herein is promising for regenerating the lanthanide luminescence at a higher photon energy with higher intensity from cubic UCNs that emit luminescence at lower intensities.

To understand the luminescence color change of the UCNs corresponding to the crystal phase transition, we considered four factors: crystal field, cross relaxation, nonradiative relaxation, and the electronic properties of the hexagonal NaREF₄, cubic NaREF₄, and hexagonal apatite phases of the lanthanide UCNs (Figure 4a). First, the effect of the crystal field on the energy levels of the Er³⁺ activator was investigated (see effective-operator Hamiltonian calculation in the Experimental Section) and the observed energy level splitting was marginal (10 cm⁻¹–100 cm⁻¹, Figure S22, Supporting Information). This result indicates that the effect of the crystal field on the color transition is negligible that is consistent with previous reports.[40,41] Second, cross relaxation, which could be caused by close distances between the activators (e.g., Er³⁺ or Tm³⁺).[25,38,42,43] was verified by comparing the interionic distances between the RE³⁺ ions through the calculated number density of RE³⁺ ions using ab initio molecular dynamics simulation (Figure 4b, density functional theory (DFT) calculation in Experimental Section). We speculate that the cross relaxation of Er³⁺ doped in the host likely occurred more in the cubic NaREF₄ phase than in the other phases. Third, we found that the cubic phase has higher phonon energy than the hexagonal phase, which implies that nonradiative relaxation is more prevalent in the cubic phase than in the hexagonal phase. Meanwhile, the hexagonal apatite phase showed weaker nonradiative relaxation owing to its low phonon energy compared to that of the hexagonal phase (Figure S25, Supporting Information).

Lastly, we studied the electronic properties of the three Er³⁺-doped crystal phases to trace the energy levels of Er³⁺ between the valence band maximum (VBM) and conduction band minimum (CBM)[44] (Figure 4c). In particular, the hexagonal apatite phase showed the lowest energy level of Er³⁺ between the VBM and CBM as compared with those of the other phases, owing to the coordination of the 2p state of O²⁻ with Er³⁺, implying that the photons could be more easily excited to the energy state of Er³⁺.

To elucidate the luminescence color changes of the cubic NaREF₄ and hexagonal apatite phases, energy transfer mechanisms of each UCNs phase are proposed (Figure 4d). One note that the ground 4I₁₅/₂ energy state of Er³⁺ does not have the zero-energy position because Er³⁺ ion is doped into the crystal structure and the unfilled energy state of 4f orbital of Er³⁺ is located between VBM and CBM.[45] The location of ground energy state of Er³⁺ (i.e., 4I₁₅/₂) can be estimated from the energy difference between the maximum of Er³⁺ 4f states near the VBM and the minimum of the F⁻ 2p or O²⁻ 2p states, which are coordinated to the Er³⁺ ion in each crystal phase, in the valence band. For the cubic phase, cross relaxation (i.e., close interionic distance of Er³⁺) and nonradiative relaxation (i.e., high phonon energy of Er³⁺) induced the red luminescence color. Wang et al. also reported similar cross relaxation induced by the shorter interionic distance of Er³⁺,[38] Additionally, the nonradiative relaxation (i.e., 4I₁₅/₂ → 4I₁₃/₂ level) owing to the high phonon energy of Er³⁺ could be another factor for the red luminescence color (Cubic NaREF₄, Figure 4d). Evidently, the upconversion luminescence spectrum of the cubic NaREF₄ indicates the occurrence of cross relaxation through the disappearance of green luminescence between 500 and 600 nm (Figure 2d). Therefore, it is reasonable to consider that cross relaxation and nonradiative relaxation are the major factors determining the yellow-to-red luminescence color change. For the hexagonal apatite phase, the green color is primarily induced by the large number of excited photons (i.e., 4I₁₅/₂ level → F₇/₂ level), followed by weak nonradiative relaxations (i.e., 4I₁₁/₂ → 4I₉/₂, 6S₅/₂ → 4F₉/₂, 6F₁/₂ → 4I₁₁/₂, and 4H₁₁/₂ → 4S₁₅/₂) owing to the low phonon energy of Er³⁺ (Hexagonal apatite in Figure 4d). In addition, the upconversion luminescence spectrum also indicates that the ratio between the green and red luminescence intensities for the hexagonal apatite phase increased as compared to that for the hexagonal NaREF₄ (Figure 2b). Therefore, we conclude that the low energy state and nonradiative relaxations of Er³⁺ in the hexagonal apatite phase are the major factors for yellow-to-green luminescence color change. The low luminescence of UCNs changed to green in the presence of SiO₂ NPs, whereas the luminescence color of blue UCNs did not change; instead, the intensity of blue luminescence increased. The blue UCNs (i.e., Tm³⁺-doped UCNs) with/without SiO₂ NPs
Figure 4. Structural and electronic properties, and the proposed energy transfer mechanism of three crystal phases. a) Schematic of the three types of crystal phases: hexagonal NaREF₄, cubic NaREF₄, and hexagonal Na₂RE₈(SiO₄)₆F₂ (apatite). b) Number density of rare earth (RE³⁺) ions within the same radial distance from the reference RE³⁺ ion for the three types of crystal phases. c) Projected density of state (PDOS) and band structure of the three crystal phases system doped with Er³⁺ ions. Here, 2p states were analyzed for F⁻ and O²⁻ ions coordinated to the Er³⁺ ion so as to calculate the lowest energy level of the Er³⁺ ion. The location of the lowest unfilled energy level of Er³⁺ ion was estimated from the energy difference between the maximum of Er³⁺ 4f states near the valence band maximum (VBM) and the maximum of F⁻ 2p or O²⁻ 2p states in the valence band. Each colored box indicates the position of the energy level of Er³⁺ ion in each crystal phase. d) Proposed energy transfer mechanism of Er³⁺ according to the type of phase (hexagonal NaREF₄, cubic NaREF₄, or hexagonal apatite). Note that the thickness of the arrows qualitatively indicates the strength of photon excitation, nonradiative relaxation, and emission.

followed the same crystal phase transition (hexagonal to hexagonal apatite or cubic phase) as that of the yellow UCNs (i.e., Er³⁺-doped UCNs) after annealing at 900 °C (Note S7, Supporting Information).

To provide a proof of concept demonstration of multiple color encrypted particle system, we rationally interweaved the crystal phase transition based luminescence color change and DMD-based maskless flow lithography technique (details in Note S8, Supporting Information). The utility of our luminescence color transition mechanism was expanded to synthesize binary color-displaying microparticles (Figure S28, Supporting Information). As shown in Figure 5a, yellow or green luminescent microparticles changed to orange and yellow at 300 °C, respectively. When the temperature reached 900 °C, the color of the area containing SiO₂ NPs changed to bright green while that of the counterpart changed to red (Figure 5a, bottom).
We further applied the aforementioned multiple luminescence color changing strategy to a potential high temperature optical labeling system displaying hidden patterns and letters (for details, see Experimental Section and Figure S29, Supporting Information). As in Figure 5b, by selectively localizing SiO$_2$ NPs in the yellow micro-post, the red triangle pattern was revealed after the thermal process. In addition, by selectively arraying the yellow micro-posts with/without SiO$_2$ NPs using our automated DMD-based lithography system, a scalable hidden letter encryption system was also realized (Figure 5c).

Lastly, we applied the investigated UCNs luminescence color and intensity changing strategy to conventional thermal processes such as a high-temperature glass forming and ceramic glazing process (Figure 5d–f). After the glass forming process, the SiO$_2$ NPs included yellow luminescence micro-post array (Figure 5d, left) exhibited bright green luminescence but SiO$_2$-free yellow luminescence micro-post array emitted almost invisible weak red luminescence after a glass forming process (Figure 5d (middle and right)). White cross and yellow pentagon shaped microparticles without/with SiO$_2$ NPs attached to traditional ceramic products such as a pottery bird (Figure 5e) and a pottery spoon rest (Figure 5f). Note that, the white luminescent microparticles with a mixing ratio of Y:B:S (1:2:1) was used, as shown in Figure 2g. After the thermal process, sky blue (hexagonalapatite) cross and bright green (hexagonal-apatite) pentagon were appeared but SiO$_2$-free counterparts lost their luminescence intensity. Based on the improved luminescent color change with the help of SiO$_2$ NPs, we may confirm that the ceramics are the right products that have undergone the proper glazing process at a designed temperature.

Our facile synthetic method can effectively manipulate or tailor the luminescence color of UCNs with high luminescent intensity by involving SiO$_2$ NPs to be used in the commercial high temperature thermal processes. We envision that the strategy of SiO$_2$ NPs-promoted luminescence color change of UCNs combined with the described additive color mixing method is a particularly effective means to label information through stepwise alteration of the luminescence color in a high temperature industrial thermal process.

**Experimental Section**

**Synthesis of rod-shaped $\beta$-NaREF$_4$ (RE = Gd, Y, Yb, Er, and Tm) UCNs:** Hexagonal phase rod-shaped NaREF$_4$ UCNs were synthesized through a hydrothermal method.$^{[13]}$ Briefly, NaOH (0.2 g mL$^{-1}$), ethanol (10 mL), oleic acid (10 mL), a solution of a mixture of lanthanide chlorides (0.2 M RECl$_3$•6H$_2$O, RE = Gd, Y, Yb, Er, and Tm, 4 mL), and 2 M ammonium fluoride (NH$_4$F, 2 mL) were mixed by stirring for 10 min and then transferred to a Teflon-lined hydrothermal device (lanthanide ion concentrations in Table S1, Supplementary Information). The mixture was
then heated at 200 °C for 3 h. Subsequently, the hydrothermal device was cooled naturally to room temperature. The resulting product was collected by centrifugation and washed several times with ethanol and water. The resultant white powder was finally dispersed in cyclohexane.

Fabrication of UCNs embedded microparticles with/without SiO$_2$ NPs: UCNs embedded microparticles with/without SiO$_2$ NPs were synthesized by a stop-flow lithography technique. UCNs with/without SiO$_2$ NPs dispersed in a photocurable resin (PUA and photo-initiator at 9:1 ratio) were flown into a polydimethylsiloxane (PDMS) microfluidic channel and polymerized with patterned UV (365 nm) illumination at 2 mW power through a ×20 objective. Digital micro-mirror device (DMD)-based mask-less lithography was used to control the pattern of UV light. The synthesized microparticles were washed five times with ethanol.

Annealing process: PUA microparticles embedded with UCNs and SiO$_2$ NPs and covert UCNs microstructures were annealed on sapphire windows (1.5 × 1.5 mm$^2$) at a desired temperature (300, 500, 700, or 900 °C) under continuous air flow (mixture of 80% N$_2$ and 20% O$_2$; 20 sccm) for 1 h in a tube furnace. After the reaction, the samples were cooled to room temperature. To characterize the crystal phase of cubic NaREF$_4$ and hexagonalapatite UCNs, the PUA film embedded with UCNs and SiO$_2$ NPs was annealed on an alumina boat.

Fabrication of microstructures with hidden pattern and letters: Microstructures with hidden pattern and letters were fabricated by controlling the photo-crosslinking location of SiO$_2$ NPs. UCNs with/without SiO$_2$ NPs dispersed in a photocurable PUA resin (PUA and photo-initiator at 9:1 ratio) were coated on an acylated substrate followed by microparticle fabrication through selective DMD-patterned UV irradiation using LabVIEW program and an inverted microscope (Nikon Ti-E). The unpolymerized monomer was rinsed with ethanol. Note that microstructure fabrication was first performed in the presence of SiO$_2$ NPs (triangle of Figure 5b, background of Figure 5c) and then in the absence of SiO$_2$ NPs (circle of Figure 5b and letters (UNIST) in Figure 5c).

Effective-operator Hamiltonian calculation: To calculate the energy level diagram of the Er$^{3+}$ ion, effective-operator Hamiltonian calculations were performed. Briefly, the effective-operator Hamiltonian calculation can be used to construct the energy level diagram of the rare earth (RE) $^{3+}$ ion in the free ion form and under the influence of a crystal field. In this study, the energy level diagram of the Er$^{3+}$ ion was built according to the three crystal phases of the host matrix. The details of the calculation are presented in Note S5, Supporting Information.

DFT calculation: DFT-based ab initio molecular dynamics simulation and geometry optimization were carried out using the CASTEP program. The details of DFT calculations and model systems are presented in Note S6, Supporting Information.

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

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