Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

Growth Phase Diagram and Upconversion Luminescence Property of $NaLuF_4:Yb^{3+}/Tm^{3+}/Gd^{3+}$ Nanocrystals

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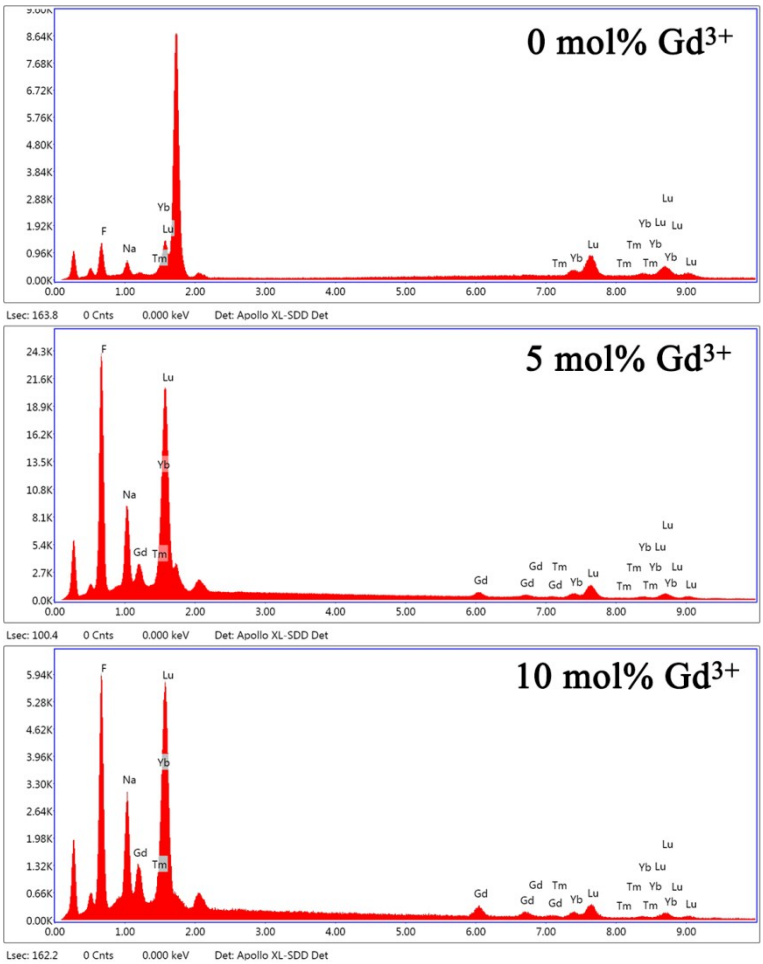

Fig. S1 EDX spectra of $NaLuF_4$:20%Yb^{3+}, 0.5%Tm^{3+}, x%Gd^{3+} (x=0, 5, 10).

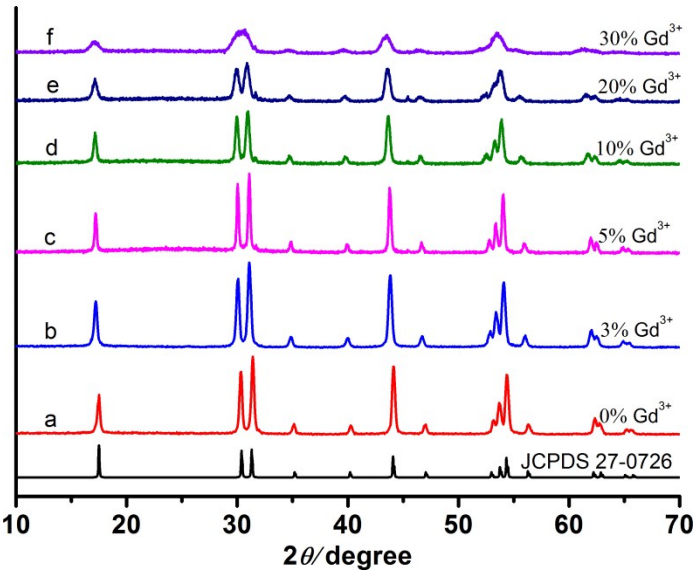

Fig. S2 XRD patterns of $NaLuF_4$:20%Yb^{3+}, 0.5%Tm^{3+}, x%Gd^{3+} with (a) x=0, (b) x=3, (c) x=5, (d) x=10, (e) x=20, (f) x=30 and the standard diffraction pattern for the β-phase $NaLuF_4$ (JCPDS 27-0726).

It can be observed from Fig. S2 that all the patterns match well with the standard hexagonal

$NaLuF_4$ pattern of JCPDS 27-0726, indicating the samples of different Gd^{3+} concentrations are pure β-phase. As shown in Fig. S2b-f, with the doping of Gd^{3+} ions in the host lattice, Lu^{3+} ions are substituted by larger Gd^{3+} ions as a result of expansion in unit-cell volume leading to the diffraction peaks shift towards lower diffraction angles along the horizontal axis.[1] For Figure 3e and f, the peaks showed significantly wider than that of the samples with lower doping concentrations of Gd^{3+} ions, indicating the size of crystal decreases with the increase of impurity ion concentration.

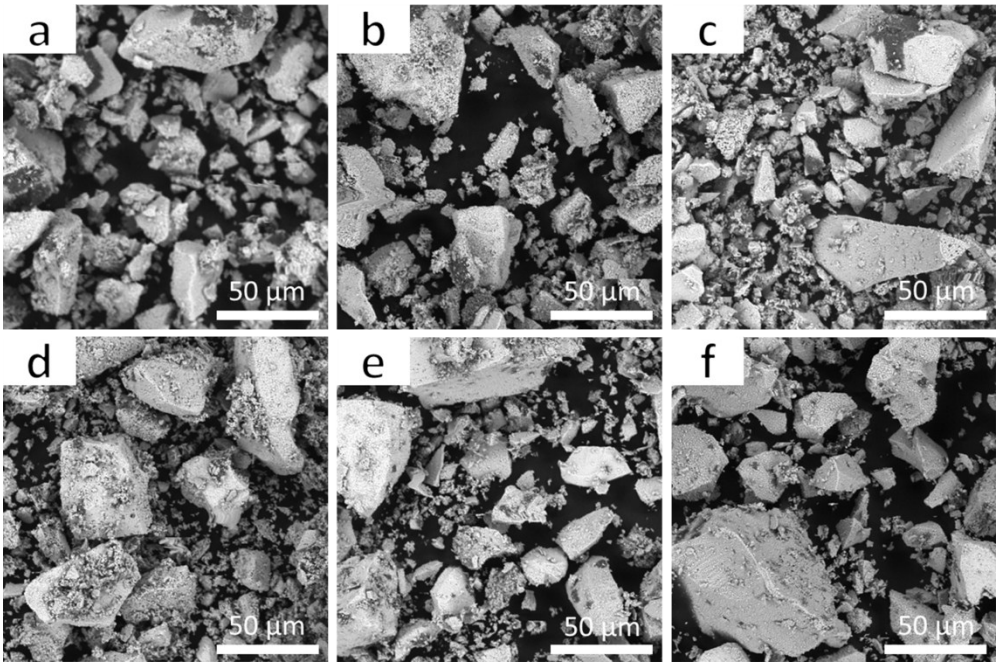

Fig. S3 SEM images of annealed $NaLuF_4$:20%Yb^{3+}, 0.5%Tm^{3+}, x%Gd^{3+} with (a) x=0, (b) x=3, (c) x=5, (d) x=10, (e) x=20, (f) x=30.

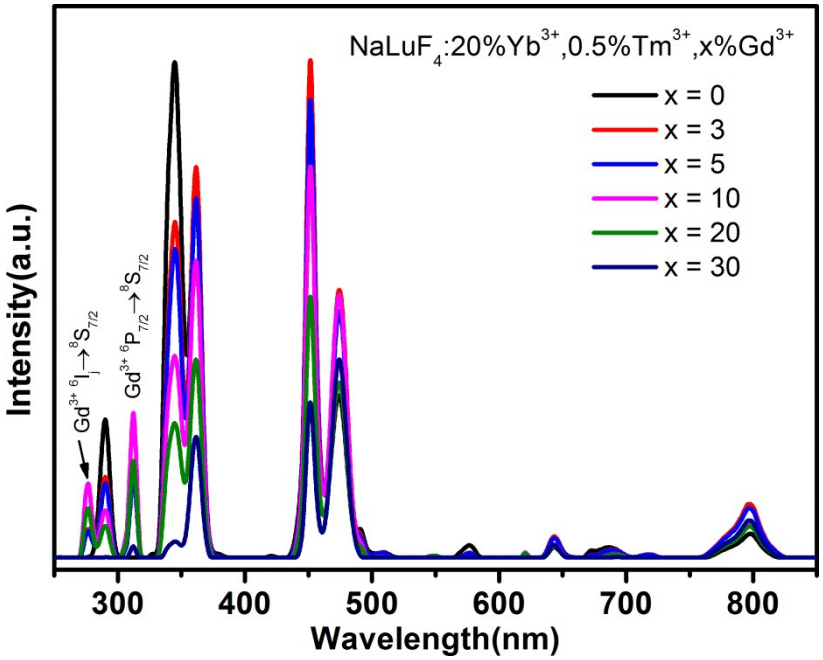

Fig. S4 Upconversion emission spectra of annealed $NaLuF_4$:20%Yb^{3+}, 0.5%Tm^{3+}, x%Gd^{3+} with (a) x=0, (b) x=3, (c)

x=5, (d) x=10, (e) x=20, (f) x=30.

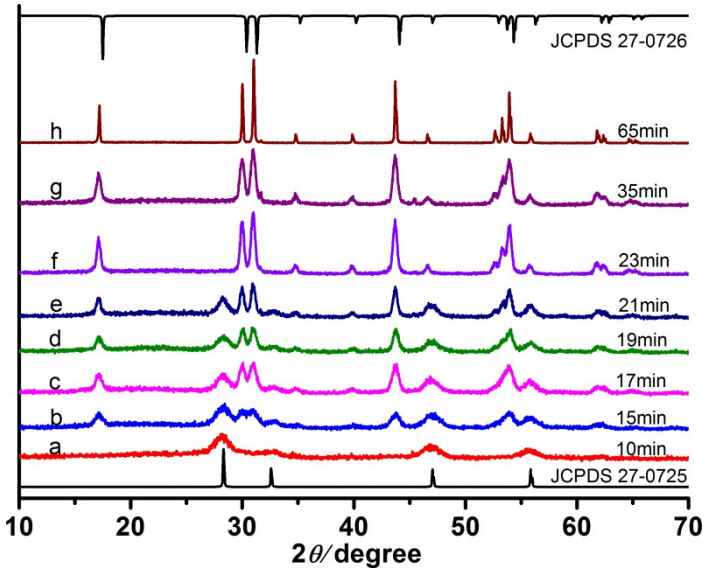

Fig. S5 XRD patterns of $NaLuF_4$:20%Yb^{3+}, 0.5%Tm^{3+}, 10%Gd^{3+} synthesized in different reaction time of (a) 10 min, (b) 15 min, (c) 17 min, (d) 19 min, (e) 21 min, (f) 23 min, (g) 35 min, (h) 65 min and the standard diffraction patterns for the α-phase $NaLuF_4$ (JCPDS 27-0725) and β-phase $NaLuF_4$ (JCPDS 27-0726).

Fig. S5 reveals the α→β phase transition and growth processes of $NaLuF_4$ NCs in detail through XRD analysis. With increasing the reaction time, α-$NaLuF_4$ transformed gradually into β-$NaLuF_4$ with the three main stages: pure α-$NaLuF_4$ at 10 min, coexistence of α-$NaLuF_4$ and β-$NaLuF_4$ from 15 to 21 min, and pure β-$NaLuF_4$ after 23 min. Notably in figure 5h, obvious narrowing of the diffraction peaks was observed which indicates the size of nanoparticles is relatively large, comparing well with the TEM image in Fig. 3g.

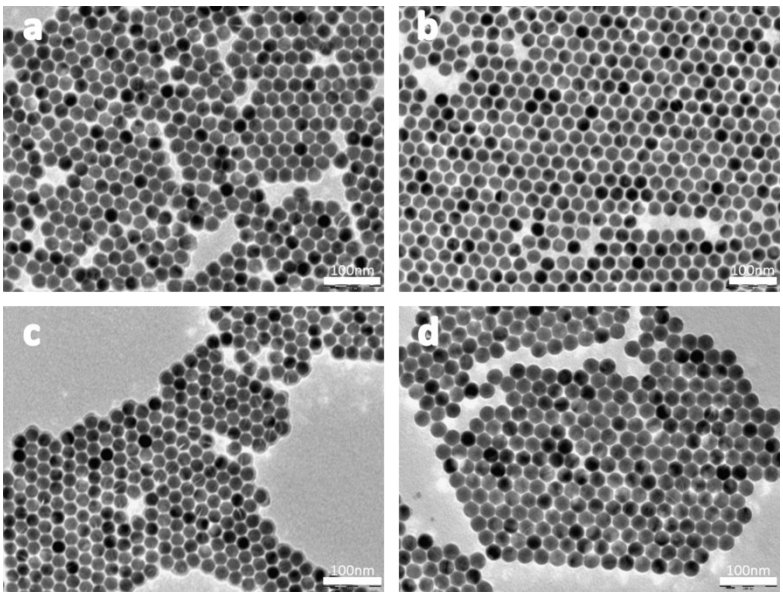

Fig. S6 TEM images of $NaLuF_4$:20%Yb^{3+}, 0.5%Tm^{3+}, 10%Gd^{3+} synthesized in different reaction time of (a) 25 min, (b) 35 min, (c) 45 min, (d) 55 min.

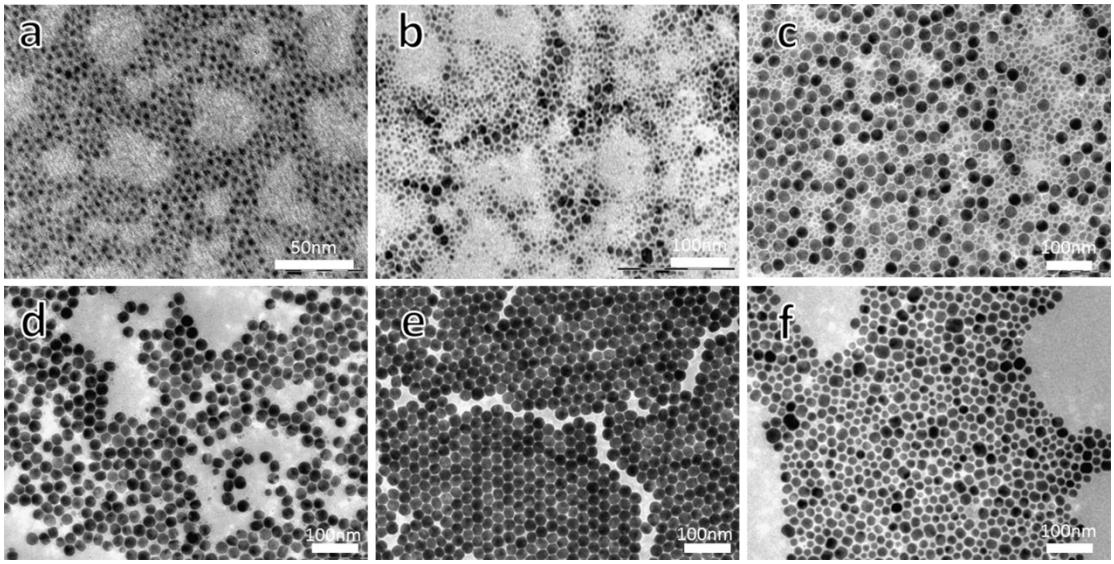

Fig. S7 TEM images of $NaLuF_4$:20%Yb^{3+}, 0.5%Tm^{3+}, 10%Gd^{3+} synthesized at 285 °C in different reaction time of (a) 20 min, (b) 40 min, (c) 80 min, (d) 100 min, (e) 110 min, (f) 300 min.

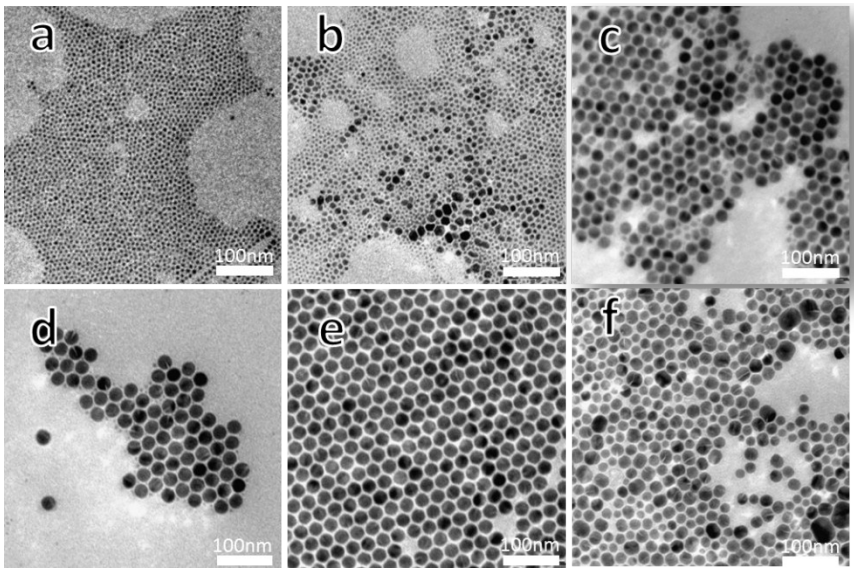

Fig. S8 TEM images of $NaLuF_4$:20%Yb^{3+}, 0.5%Tm^{3+}, 10%Gd^{3+} synthesized at 295 °C in different reaction time of (a) 15 min, (b) 25 min, (c) 40 min, (d) 50 min, (e) 60 min, (f) 170 min.

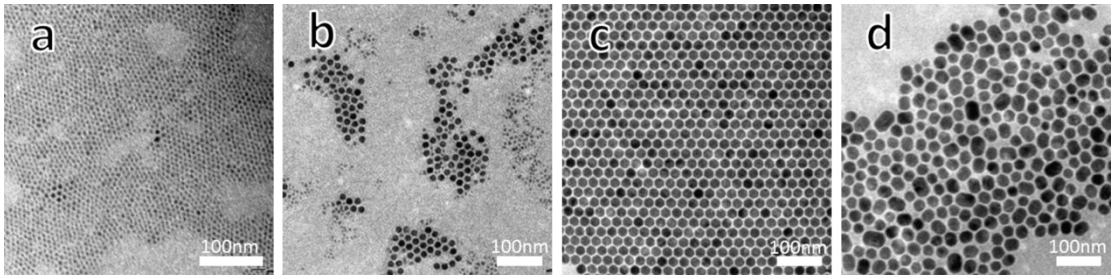

Fig. S9 TEM images of $NaLuF_4$:20%Yb^{3+}, 0.5%Tm^{3+}, 10%Gd^{3+} synthesized at 315 °C in different reaction time of (a) 5 min, (b) 8 min, (c) 12 min, (d) 40 min.

Reference

S1. F. Wang, Y. Han, C. S. Lim, Y. Lu, J. Wang, J. Xu, H. Chen, C. Zhang, M. Hong and X. Liu, *Nature*, 2010, **463**, 1061-1065.