## **Supporting Information:**



SI.Fig.1: Normalized upconversion luminescence decay kinetics of the  $Er^{3+}$  emission bands G1 and G2 of UCNP<sub>AS</sub>.



Fig.SI-2: SEM-image of annealed UCNP<sub>400</sub> with micro domains, which is representative for all UCNP<sub>x</sub> with  $x > 400^{\circ}$ C.



Fig. SI-3: X-ray diffraction patterns of UCNP<sub>x</sub> after annealing between  $200^{\circ}C < T_{an} < 280^{\circ}C$  for 5h.

SI-Tab.2: Crystallite size of UCNP <sub>x</sub> nanoparticles after different thermal treatment calculated
with Debye-Scherrer from typical a-phase XRD pattern.

UCNPx	Millersche Indices					
	111	200	220	311		
T <sub>an</sub> [°C]	Crystallite size [nm]					
200	36.8	38.2	27.7	26.2		
220	38.7	39.7	28.9	26.1		
240	38.9	43.1	29.0	26.3		
280	39.6	39.9	28.6	25.8		
300	39.6	39.6	29.2	25.5		
700	47.7	48.6	34.1	25.2		

SI-Tab.3: Crystallite size of UCNP<sub>x</sub> nanoparticles after different thermal treatment calculated with Debye-Scherrer from typical  $\beta$ -phase XRD pattern.

UCNPx	Millersche Indices					
	100	110	101	201	300	
T <sub>an</sub> [°C]	Crystallite size [nm]					
400	64.4	59.8	58.2	46.4	41.3	
500	95.1	75.4	73.6	58.1	53.4	
600	105.3	78.7	69.6	53.6	53.0	



Fig. SI-4: Enlarged representation in G1-related TRANES for UCNP<sub>300</sub> showing the isoemissive points in detail.