

Supporting Information for:

**Selective Synthesis, Polymorphism, Reversible Phase
Transition and Structure-Dependent Optical Functionalities of
Gadolinium Oxyfluorides**

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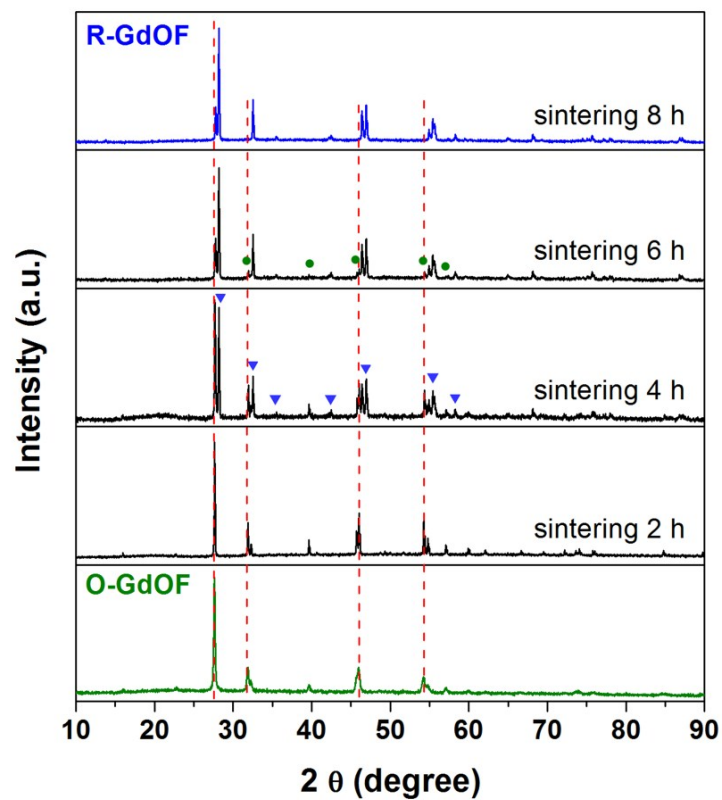


Figure S1. Ex situ powder XRD patterns of O-GdOF show its transformation to R-GdOF at 700 °C with different sintering time.

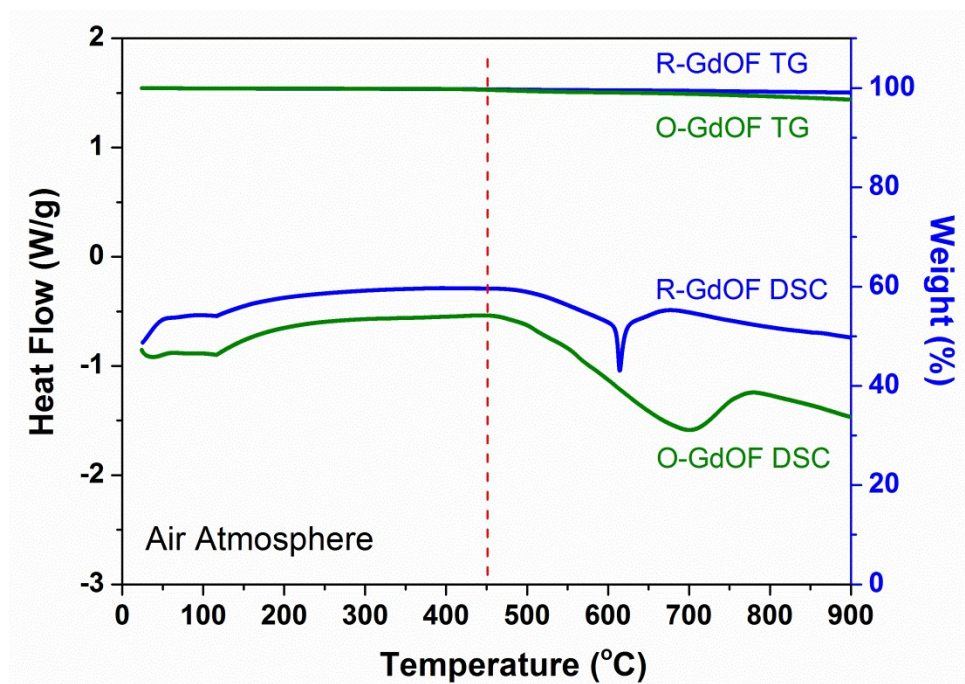


Figure S2. DSC and TG analysis of R- and O-GdOF samples in air.

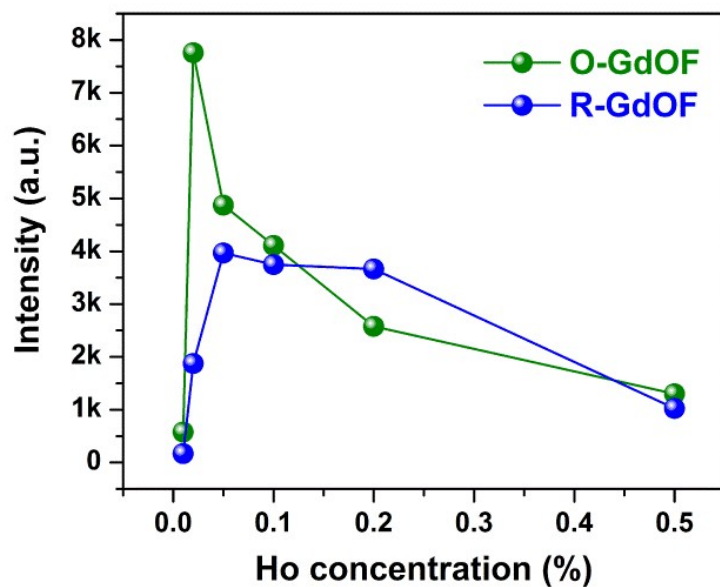


Figure S3. The net area of band at 670 nm of O- and R-GdOF against Ho^{3+} doping concentration.

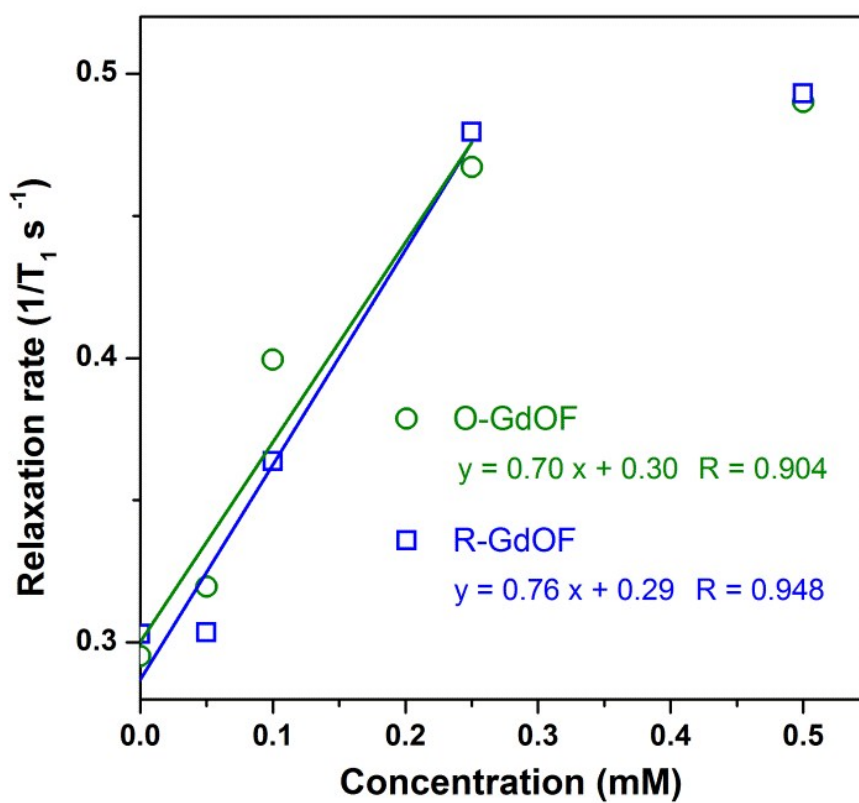


Figure S4. Plot of relaxation rate ($1/T_1$) versus different Gd concentrations of O- and R-GdOF samples. (O-GdOF: Yb 20%, Er 0.2%; R-GdOF: Yb 20%, Er 0.1%)

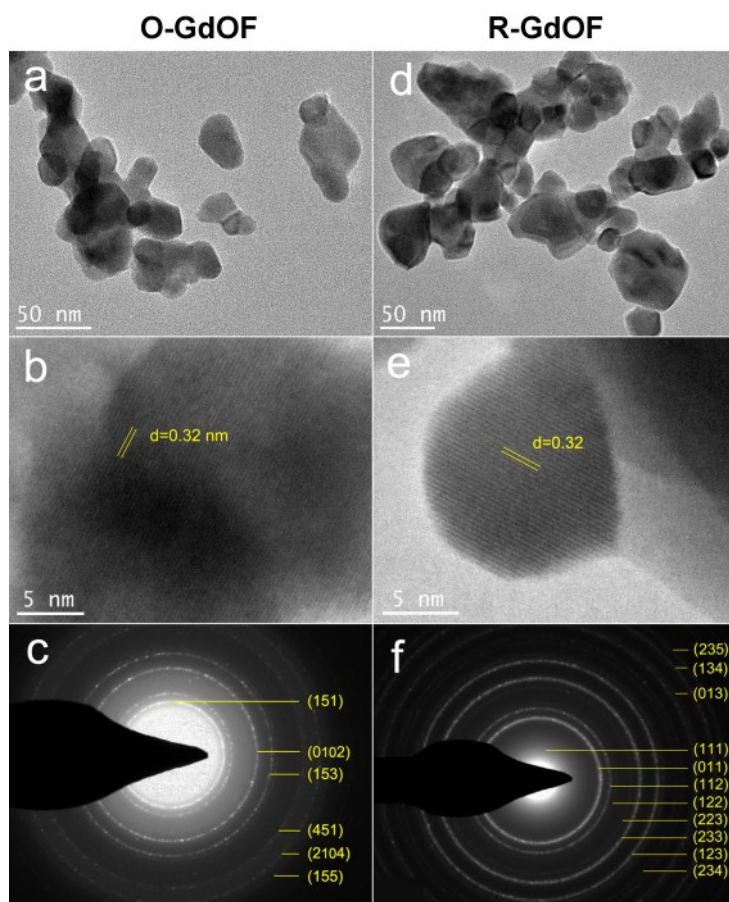


Figure S5. TEM, high-resolution TEM images and SAED patterns of O- and R-GdOF samples. The HRTEM images in (b), the lattice fringes of $d = 0.32$ nm is consistent with the distance of 151 facets of O-GdOF, and (e) the lattice fringes of $d = 0.32$ nm consists with the distance of 011 facets of R-GdOF.