

## Supporting Information

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Nanoscale Porosity of High Surface Area Gadolinium Oxide Nanofoam Obtained With Combustion Synthesis

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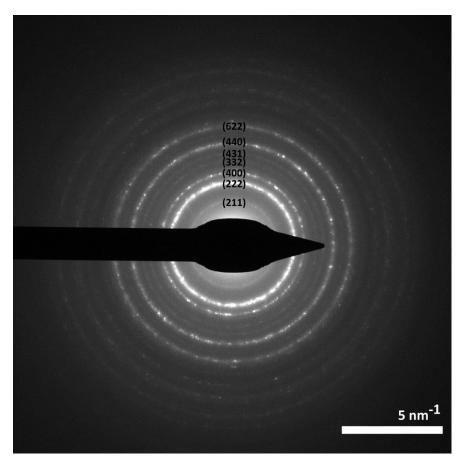


Figure S1: Indexed electron diffraction pattern indicating the cubic  $Gd_2O_3$  phase.

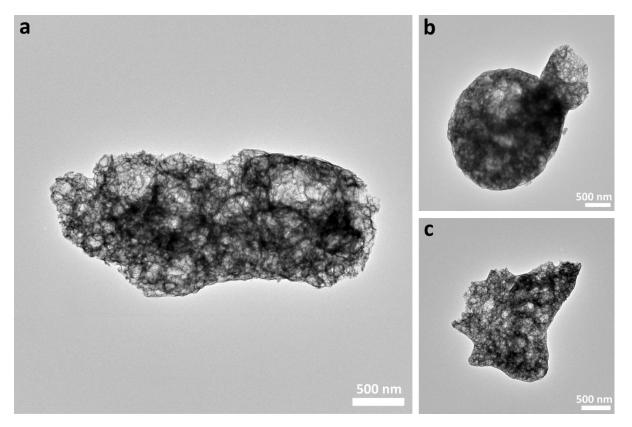


Figure S2: TEM images of different Gd<sub>2</sub>O<sub>3</sub> morphologies showing a cylindrical (a), spherical (b), and irregular structure (c).

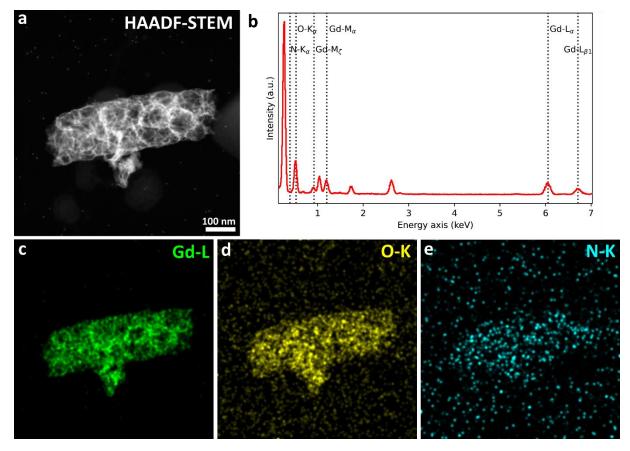


Figure S3: EDS analysis of the  $Gd_2O_3$  grain shown in the HAADF-STEM image (a). The EDS spectrum in (b) shows peaks corresponding to the elements Gd and O, and the absence of a N peak. Elemental maps shown in (c) and (d) indicate that Gd and O are uniformly presented throughout the sample, whereas (e) shows that the presence of N is negligible.

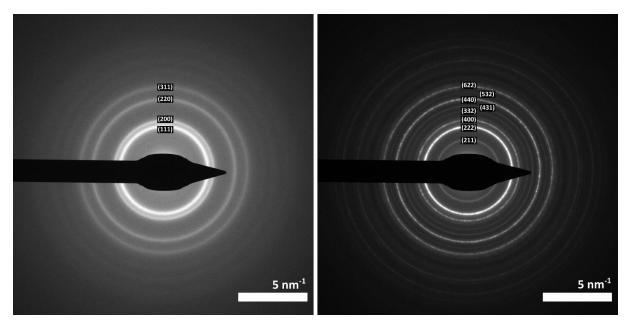


Figure S4: Indexed diffraction patterns of the in-situ heating to 600  $^{\circ}C$  corresponding to GdO (left) and the ex-situ heating to 900  $^{\circ}C$  corresponding to Gd<sub>2</sub>O<sub>3</sub> (right).

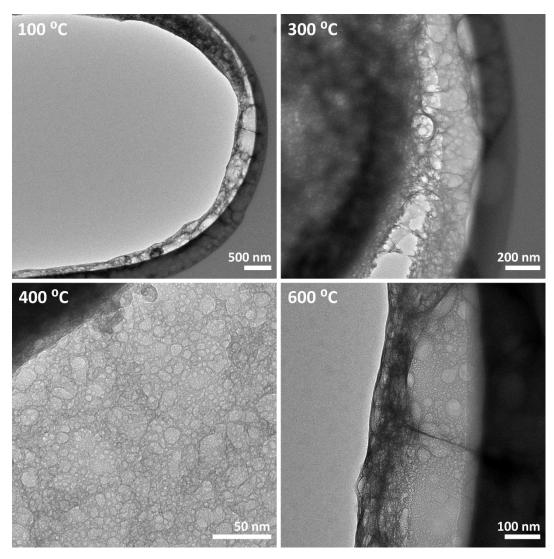


Figure S5: TEM images of Gd gel during in-situ heating to 600  $^{\rm o}{\rm C}.$ 

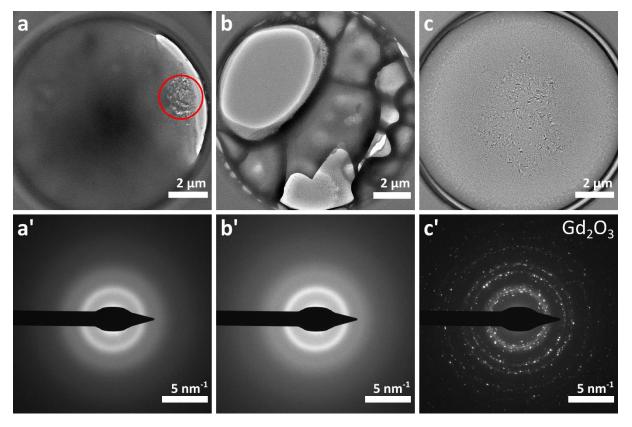


Figure S6: TEM window overview image of 600 °C in-situ sample after 6 weeks (a), 600 °C ex-situ sample after 6 weeks (b), and 900 °C ex-situ sample after 7 weeks (c) with corresponding diffraction patterns ('). The red circle indicates the area that abruptly changed when illuminated with the electron beam.

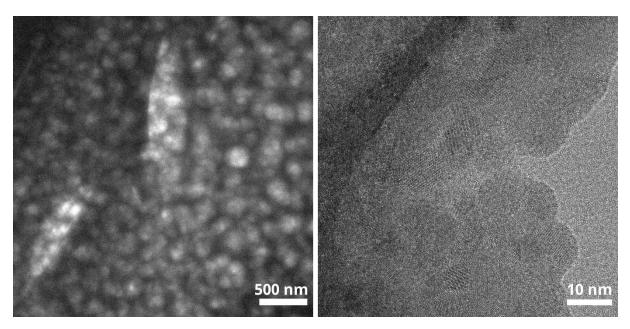


Figure S7: TEM image of the precursor heated to 100 °C showing large scale porosity (left) and HRTEM image of the precursor heated to 900 °C, which is not showing small scale porosity (right).

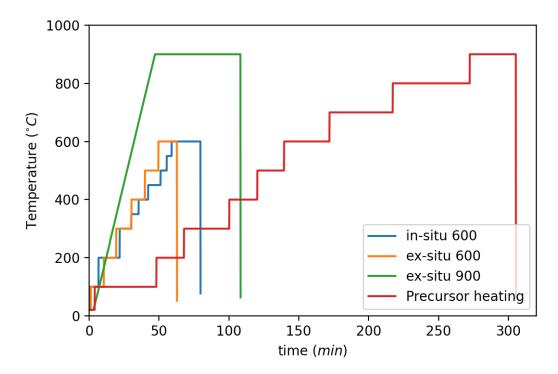


Figure S8: Heating profiles for the various in-situ and ex-situ experiments.

**Supporting Video 1:** Aligned tilt series of a large Gd<sub>2</sub>O<sub>3</sub> piece tilted from -73° to 80° in TEM mode.

**Supporting Video 2:** Aligned tilt series of a large Gd<sub>2</sub>O<sub>3</sub> piece tilted from -73° to 80° in STEM mode.

**Supporting Video 3:** Tomographic reconstruction of the large  $Gd_2O_3$  piece acquired in STEM mode made using the weighted back projection algorithm.

**Supporting Video 4:** Animation of the 3D reconstruction of the large  $Gd_2O_3$  piece, first showing the surface of the particle and then slicing through it.

**Supporting Video 5:** Aligned tilt series of a small Gd2O3 piece tilted from -65° to 78° in STEM mode.

**Supporting Video 6:** Tomographic reconstruction of the small Gd<sub>2</sub>O<sub>3</sub> piece acquired in STEM mode made using the weighted back projection algorithm.

**Supporting Video 7:** Animation of the 3D reconstruction of the small  $Gd_2O_3$  piece, first showing the surface of the particle and then slicing through it.