Supporting Information

for Adv. Mater. Interfaces, DOI: 10.1002/admi.202300060

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$_2$O$_3$ phase.
Figure S2: TEM images of different Gd₂O₃ morphologies showing a cylindrical (a), spherical (b), and irregular structure (c).
Figure S3: EDS analysis of the Gd$_2$O$_3$ 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 °C corresponding to GdO (left) and the ex-situ heating to 900 °C corresponding to Gd$_2$O$_3$ (right).
Figure S5: TEM images of Gd gel during in-situ heating to 600 °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$_2$O$_3$ piece tilted from -73° to 80° in TEM mode.

Supporting Video 2: Aligned tilt series of a large Gd$_2$O$_3$ piece tilted from -73° to 80° in STEM mode.

Supporting Video 3: Tomographic reconstruction of the large Gd$_2$O$_3$ piece acquired in STEM mode made using the weighted back projection algorithm.

Supporting Video 4: Animation of the 3D reconstruction of the large Gd$_2$O$_3$ piece, first showing the surface of the particle and then slicing through it.

Supporting Video 5: Aligned tilt series of a small Gd$_2$O$_3$ piece tilted from -65° to 78° in STEM mode.

Supporting Video 6: Tomographic reconstruction of the small Gd$_2$O$_3$ piece acquired in STEM mode made using the weighted back projection algorithm.

Supporting Video 7: Animation of the 3D reconstruction of the small Gd$_2$O$_3$ piece, first showing the surface of the particle and then slicing through it.