***Electronic Supplementary Information***

# Hierarchical γ-$Al_2O_3$ Monoliths with Highly Ordered 2D hexagonal Mesopores in Macroporous Walls

Le-Le Li, Wen-Tao Duan, Quan Yuan, Zhen-Xing Li, Hao-Hong Duan, and Chun-Hua Yan *

*Beijng National Laboratory for Molecular Science, State Key Laboratory of Rare Earth Materials Chemistry and Applications, PKU-HKU Joint Laboratory on Rare Earth Materials and Bioinorganic Chemistry, Peking University, Beijing, 100871, China. E-mail: yan@pku.edu.cn; Fax & Tel: +86 10 6275 4179*

## Experimental Section

***Materials***: Pluronic P123 ($M_{av}$ = 5800, $EO_{20}PO_{70}EO_{20}$) was purchased from Aldrich. Polyurethane (PU) foam with porosity of 45 ppi (pores per linear inch) was purchased from Shenzhen Xinweifa Company. Aluminum iso-propoxide, $Ni(NO_3)_2{\cdot}6H_2O$, and $HNO_3$ were purchased from Beijing Chemical Reagents. All of the chemicals were used as received without further purification.

***Synthesis***: In a typical synthesis, 1.0 g of Pluronic P123 was dissolved in 20 mL of ethanol at room temperature. Then 1.4–1.6 mL of 67 wt% nitric acid and 2.04 g (10 mmol) of aluminum iso-propoxide were added into the above solution with vigorous stirring. The mixture was covered with PE film, stirred at room temperature for 1 day. Afterward, the polyurethane (PU) foam was completely immersed into the sol and frequently squeezed in order to force the sol to migrate into all pores. Then the infused PU foam was put into a 40 °C drying oven to undergo the solvent evaporation process, which ensures that the self-assembled mesophase uniformly coated on the struts of the PU foams. After 2 days of aging, they were heat-treated at a slow heating rate of 1 °C/min to 550 °C and kept at that temperature for 4 h in air. High-temperature treatment was carried out in air for 1 h with a temperature ramp of 10 °C/min.

Al-based multicomponent mesoporous monoliths were prepared in the same procedure by using quantitative $Ni(NO_3)_2{\cdot}6H_2O$ and aluminum iso-propoxide (total amount of Ni plus Al is 10 mmol) instead of aluminum iso-propoxide as inorganic precesur.

***Characterization:*** X-ray powder diffraction (PXRD) patterns were recorded on a Rigaku DMAX-2000 diffractometer (Japan) using Cu Kα(λ = 1.5405 Å) radiation. Scanning electron microscopy (SEM) observations were carried out with DB-235 focused ion beam (FIB) system operated at an acceleration voltage of 15 kV. Transmission electronic microscopy (TEM) was taken on a Hitachi H-9000 NAR transmission electron microscope (Japan) under a working voltage of 300 kV. High-resolution transmission electron microscopy (HRTEM) was performed with a Philips Tecnai F30 FEG-TEM (USA) operated at 300 kV. The nitrogen adsorption and desorption isotherms at 78.3 K were measured using an ASAP 2010 analyzer (Micromeritics Co. Ltd., USA).

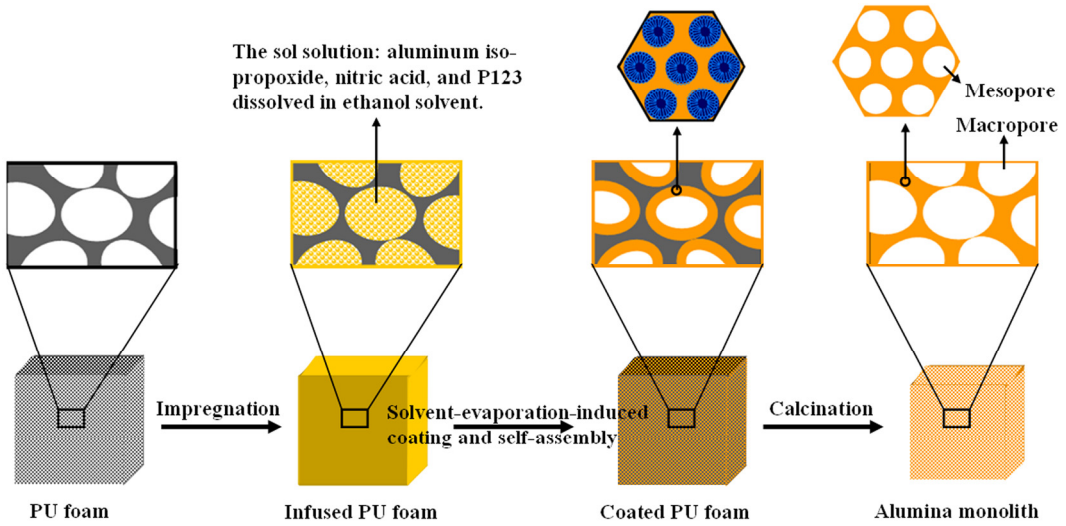


**Figure S1.** Illustration of the synthesis procedure of hierarchical porous $Al_2O_3$ monoliths by using nonionic triblock copolymer and polyurethane (PU) foam as cotemplates via a solvent-evaporation-induced coating and self-assembly method.

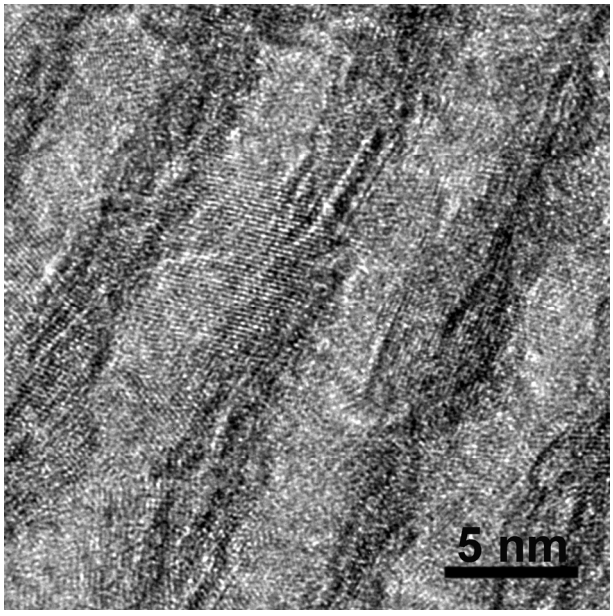


**Figure S2.** HRTEM image of alumina monolith calcined at 900 °C.

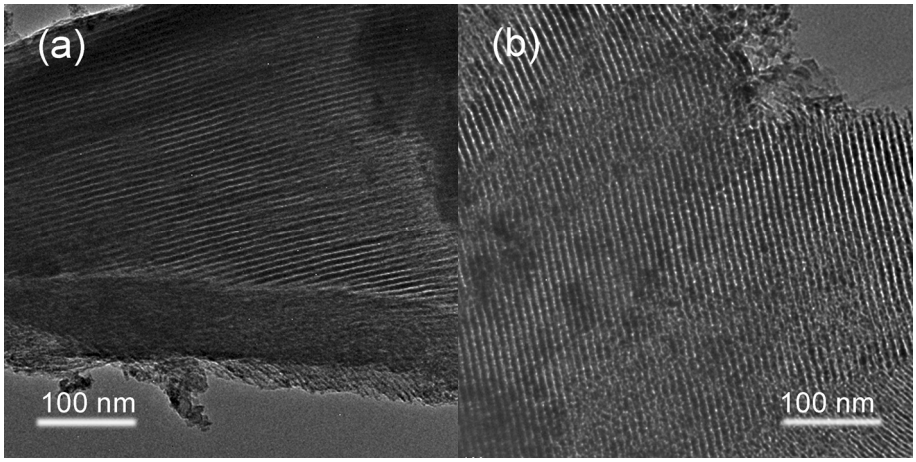


**Figure S3.** TEM images of the Ni-containing alumina monoliths (12 mol% of Ni) calcined at (a) 550 °C and (b) 900 °C.

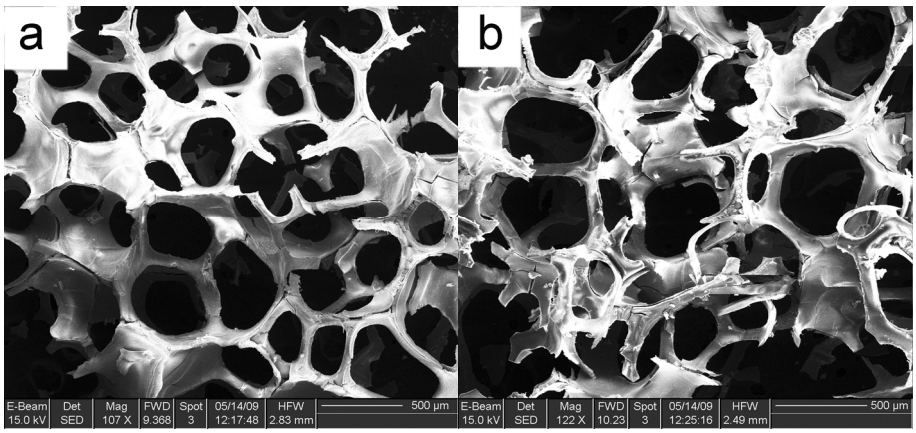


**Figure S4.** SEM images of the Ni-containing alumina monoliths (12 mol% of Ni) calcined at (a) 550 °C and (b) 900 °C.

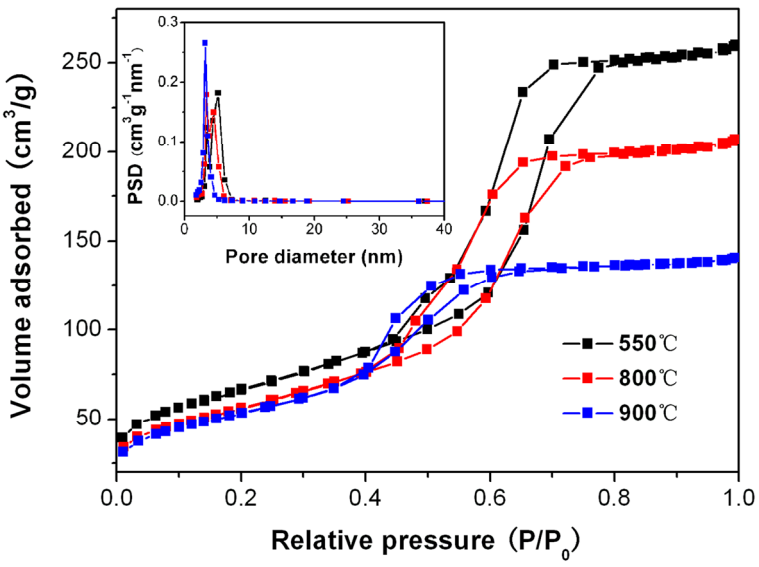


**Figure. S5** Nitrogen adsorption-desorption isotherms and the pore-size distributions of the mesoporous Ni-containing (12 mol%) alumina monoliths calcined at various temperatures.