

Supplementary Information

Magnetic Studies of Mesoporous Nanostructured Iron Oxide Materials Synthesized by One-Step Soft-Templating

Jing Jin,^a William A. Hines,^b Chung-Hao Kuo,^c David M. Perry,^b Altug S. Poyraz,^c Yan Xia,^d Taha Zaidi,^b Mu-Ping Nieh,^{ade} and Steven L. Suib^{†ac}

^a Institute of Materials Science, University of Connecticut, Storrs, CT 06269-3136.

^b Department of Physics, University of Connecticut, Storrs, CT 06269-3046.

^c Department of Chemistry, University of Connecticut, Storrs, CT 06269-3060.

^d Department of Chemical and Biomolecular Engineering, University of Connecticut, Storrs, CT 06269-3222.

^e Department of Biomedical Engineering, University of Connecticut, Storrs, CT 06269-3247.

[†] Corresponding Author Email: steven.suib@uconn.edu

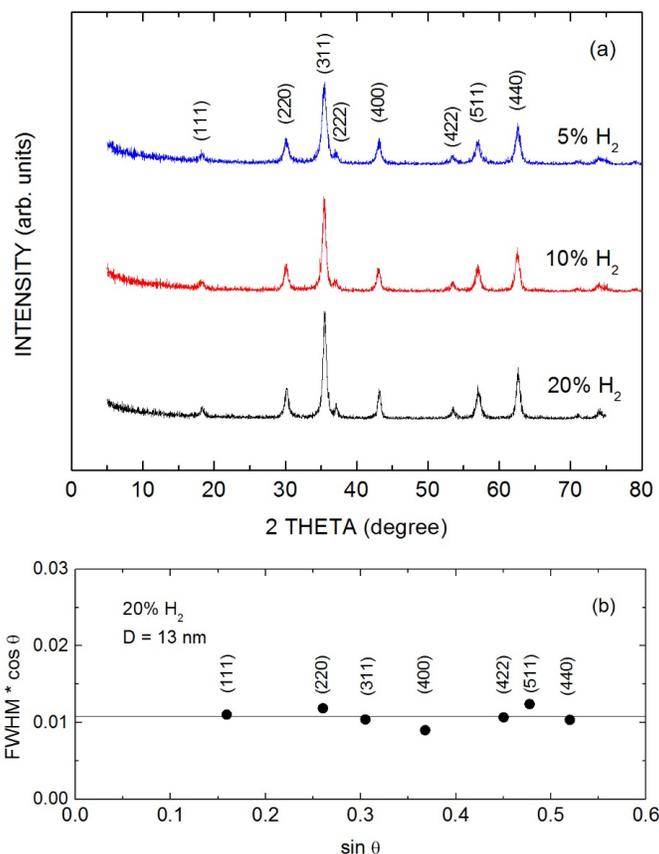


Figure S1. (Color Online) (a) Wide-angle PXRD patterns for the three (second series) mesoporous nanostructured samples heated at 250 °C for 3 hrs in 5% H₂, 10% H₂, and 20% H₂ atmospheres, and (b) (expanded) Scherrer equation analysis for the grain size in the 20% H₂ sample. The wide-angle pattern is consistent with the spinel Fe₃O₄ crystalline phase; cubic unit cell with Fd-3m space group (JCPDS no. 85-1436)²⁰.

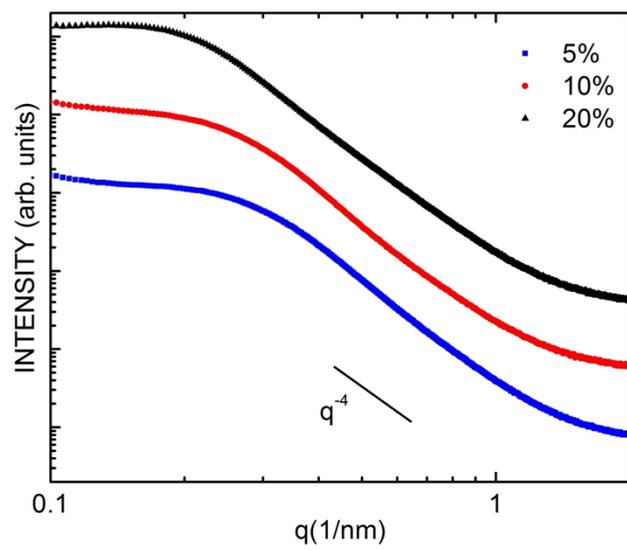


Figure S2. (Color Online) 1-D SAXS intensity profiles (log-log plots) obtained by integration of the 2-D data for the three (second series) mesoporous samples heated in 5% H₂, 10% H₂ and 20% H₂ atmospheres. The “knee”-like features yield estimates for the nanoparticle size (see text).

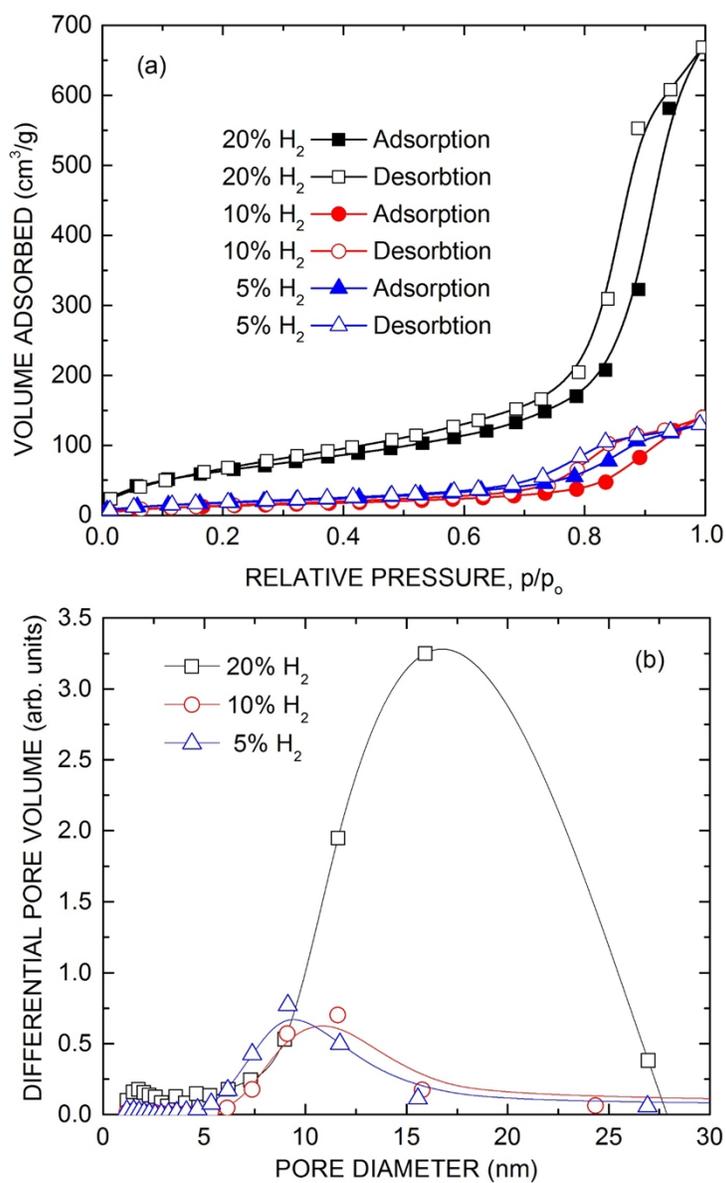


Figure S3. (Color Online) (a) Nitrogen sorption isotherms and (b) pore size distribution curves for the three (second) series mesoporous samples heated at 250 °C for 3 hrs in 5% H₂, 10% H₂, and 20% H₂ atmospheres. The BET surface areas and BJH pore sizes obtained from analysis of the isotherms are presented in **Table I**. Unlike traditional mesoporous materials, the UCT materials expand with heat treatment.

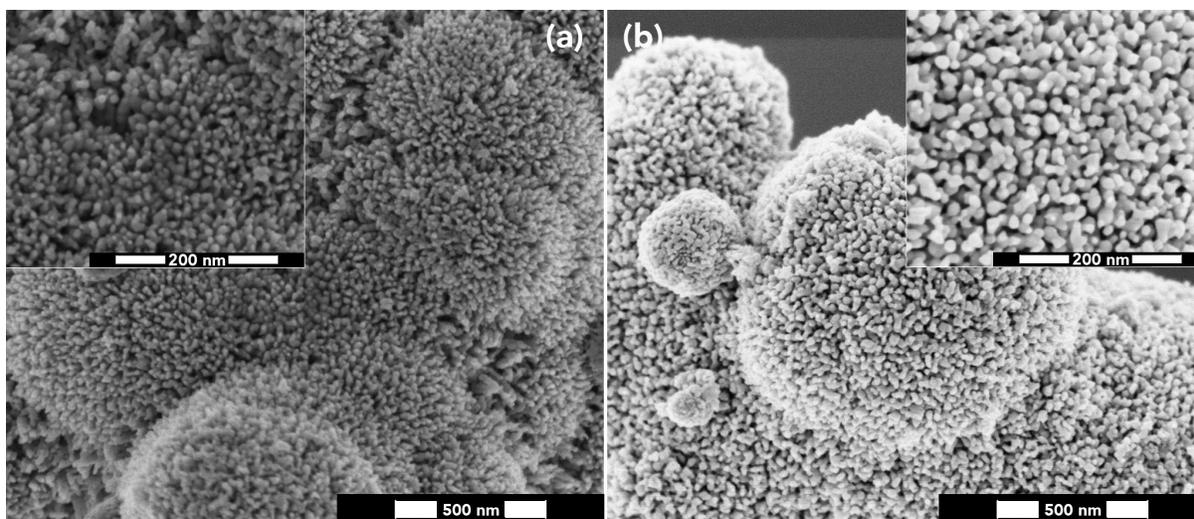


Figure S4. Scanning electron microscope images obtained from two of the three (second) series samples: (a) 5% H₂ sample - 500 nm scale bar (inset 200 nm) and (b) 20% H₂ sample - 500 nm scale bar (inset 200 nm). The overall sample morphology can be described as essentially spherical clusters with nanoscale segments.

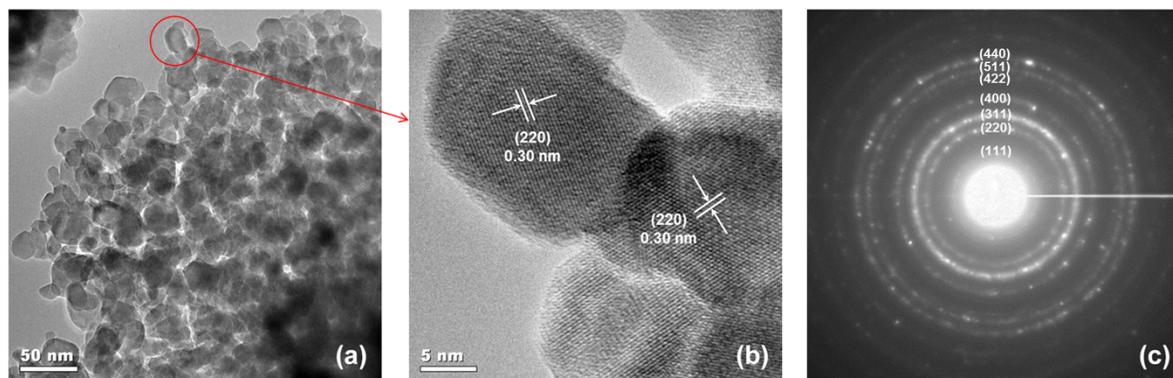


Figure S5. Transmission electron microscope images obtained from the sample heated at 250 °C for 3 hrs in a 20% H₂ atmosphere: (a) TEM, 50 nm scale bar, (b) HRTEM, 5 nm scale bar, and (c) SAED pattern with Debye-Scherrer-type rings which can be indexed to the spinel Fe₃O₄ structure. The mesostructure consists of an assembly of nanoparticles with diameters ranging from 17 nm to 24 nm (see **Figure S5a**). With high resolution (see **Figure S5b**), the d-spacing of the resolved fringes is 0.30 nm, consistent with (200) planes for Fe₃O₄.

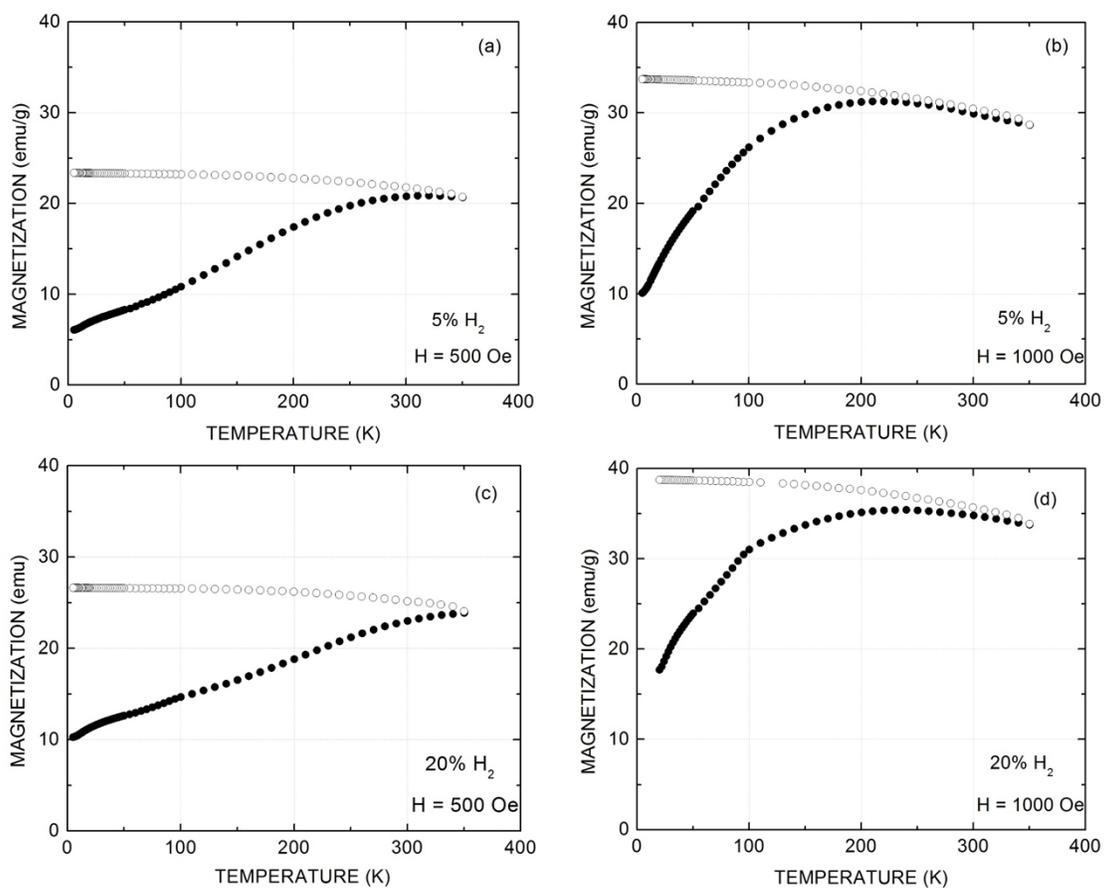


Figure S6. Zero-field-cooled (closed symbols) and field-cooled (open symbols) magnetization versus temperature for the sample heated in a 5% H₂ atmosphere at 250 °C for 3 hrs (a) H = 500 Oe and (b) H = 1,000 Oe. Zero-field-cooled (closed symbols) and field-cooled (open symbols) magnetization versus temperature for the sample heated in a 20% H₂ atmosphere at 250 °C for 3 hrs (c) H = 500 Oe and (d) H = 1,000 Oe. The decrease of the peak temperature with magnetic field indicates superparamagnetic behavior; the blocking temperatures T_B are indicated.

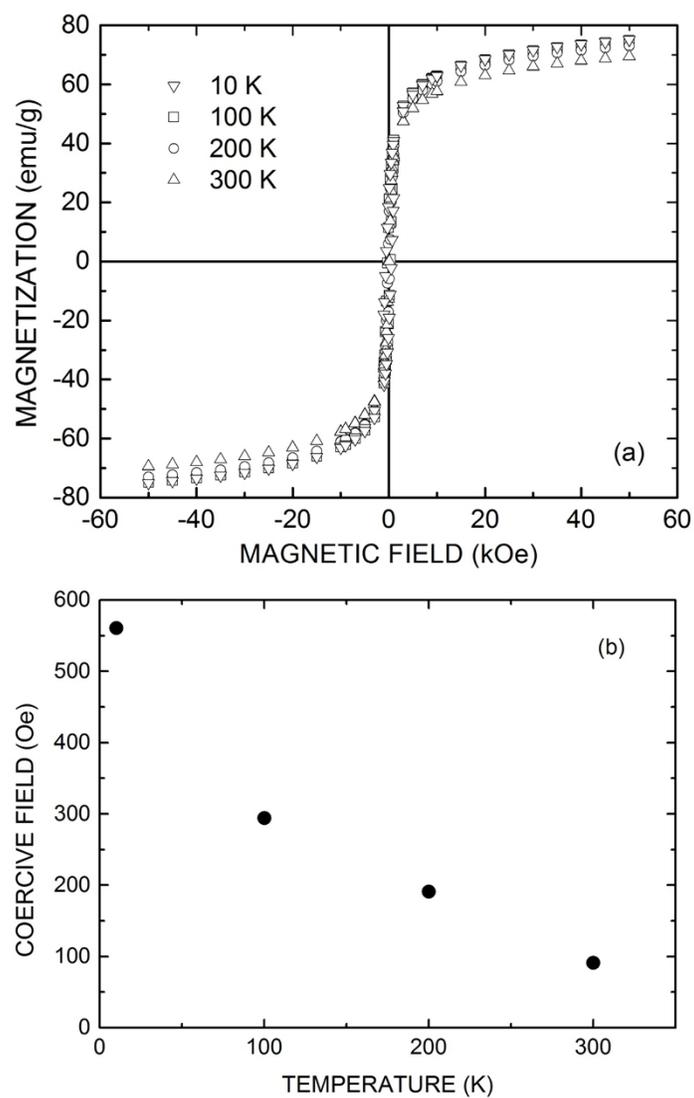


Figure S7. (a) Magnetization versus magnetic field (hysteresis loops) measured at temperatures ranging from 10 K to 300 K for the sample heated in a 5% H₂ atmosphere at 250 °C for 3 hrs and (b) temperature dependence of the coercive field values, H_c, obtained from the hysteresis loops.

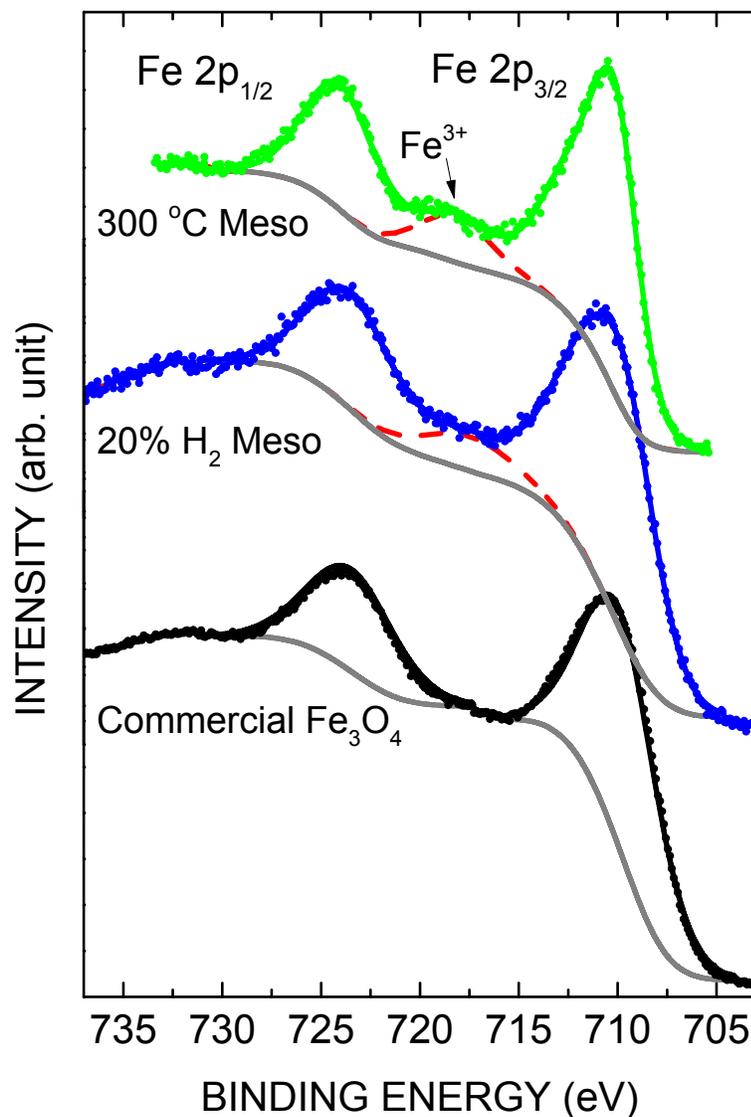


Figure S8. X-ray Photoemission Spectrum (XPS) for the 300 °C mesoporous sample (green dots are the data points and the green solid line is the fitting curve), 20% H₂ mesoporous sample (blue dots are the data points and the blue solid line is the fitting curve) and the commercial Fe₃O₄ (black dots are the data points and the black solid line is the fitting curve). The Shirley backgrounds for each sample are presenting as the solid gray lines. The Fe³⁺ satellite peak (red dash line) is clearly observed for the 300 °C mesoporous sample, consistent with the γ -Fe₂O₃ phase. The satellite structure of the commercial Fe₃O₄ is completely obscured by the overlapping Fe²⁺ and Fe³⁺ satellites. The Fe³⁺ satellite is only partially obscured by the Fe²⁺ satellite for the 20% H₂ mesoporous sample, which indicates a contribution from the γ -Fe₂O₃ phase.