SUPPLEMENTARY INFORMATION

Interplay between microstructure and magnetism in NiO nanoparticles: breakdown of the antiferromagnetic order

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Figure Sup1. Histograms (bars) of the NP diameter, extracted from the HRTEM images, together with log-normal distribution fits (lines), providing mean particle sizes and standard deviations, $D_{TEM}(\sigma)$, of 2.5(0.6), 4(1) and 9(2) nm for S1 (left), S2 (center) and S3 (right), respectively.



Figure Sup2. Normalized x-ray absorption spectra measured at the Ni K-edge at room temperature for S1 (blue), S2 (red), S3 (green) and a NiO standard (black).



Figure Sup3. Variation of the first (N_{Ni-O} , blue dots) and second (N_{Ni-Ni} , brown squares) shell coordination numbers due to cluster size (*D*) reduction. Theoretical values of *N* (lines) were estimated from Calvin's equation¹, while experimental ones (symbols) were extracted from the EXAFS fits of the recorded spectra of S1, S2 and S3.



Figure Sup4. M(T) curves in the ZFC (green circles) and FC (grey diamonds) regimes measured for S1 (a), S2 (b) and S3 (c) under an applied magnetic field of 0.1 kOe. Inset in (a) highlights a detail of the low temperature region, evidencing a peak in the ZFC curve. It should be noted that the low signal of S3 was severely affected by noise. Anyway, these curves presented the same features as those recorded at 1 kOe, being these latter ones clearer due to the higher measured signal.

1. S. Calvin, M. M. Miller, R. Goswami, S. F. Cheng, S. P. Mulvaney, L. J. Whitman, and V. G. Harris, *J. Appl. Phys.*, 2003, **94**, 778–783.