Electronic Supplementary Material (ESI) for Nanoscale Horizons.

Supporting Information

Thermally Driven Phase Transition of Cobalt Hydroxide Sheets via Cobalt Oxide to Co Particles

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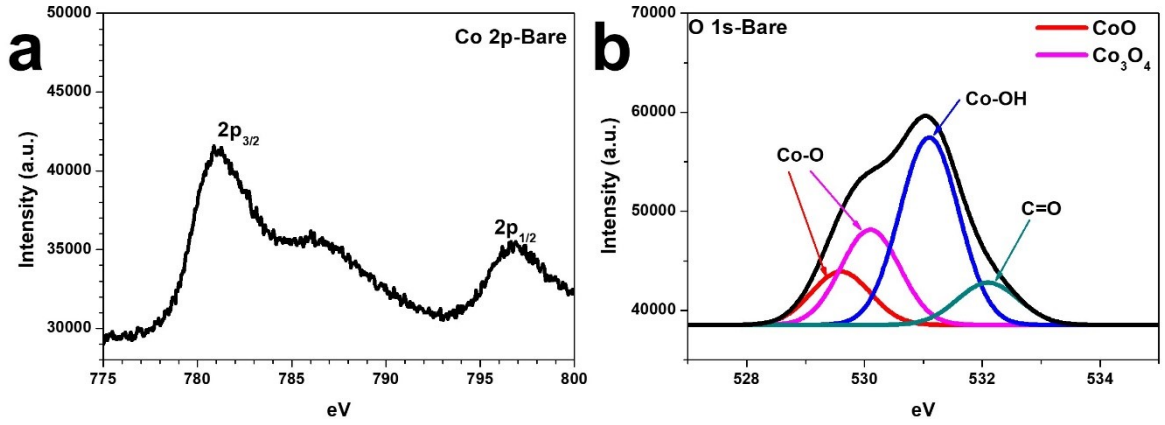

Fig. S1. XPS spectra of synthesized $Co(OH)_2$ nanosheet.

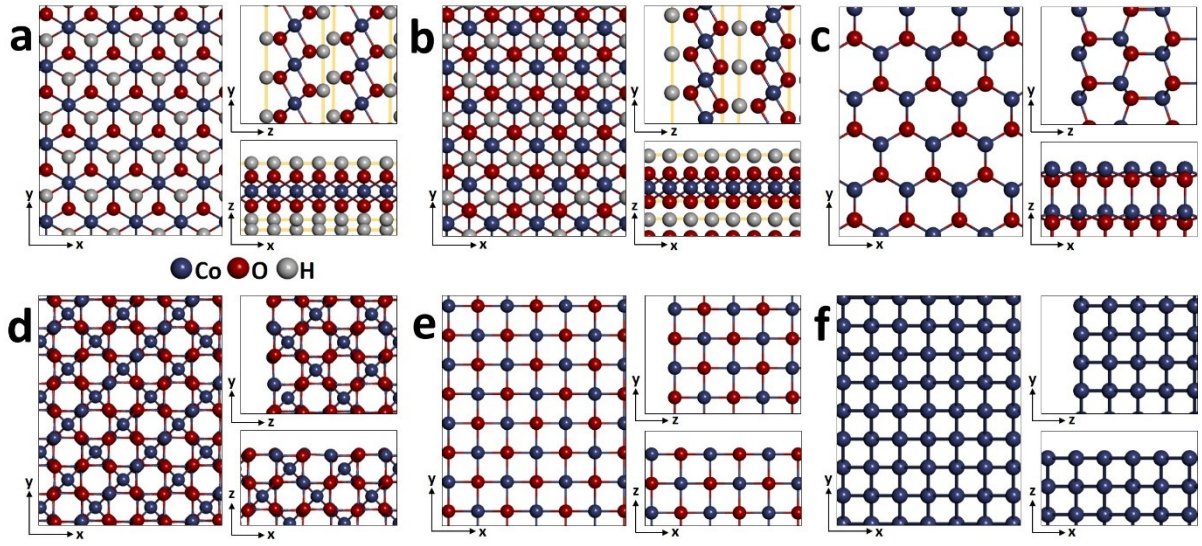

Fig. S2. Detailed atomic models (a) $Co(OH)_2$, (b) CoOOH, (c) h-CoO, (d) Co_3O_4, (e) c-CoO, and (f) Co, respectively.

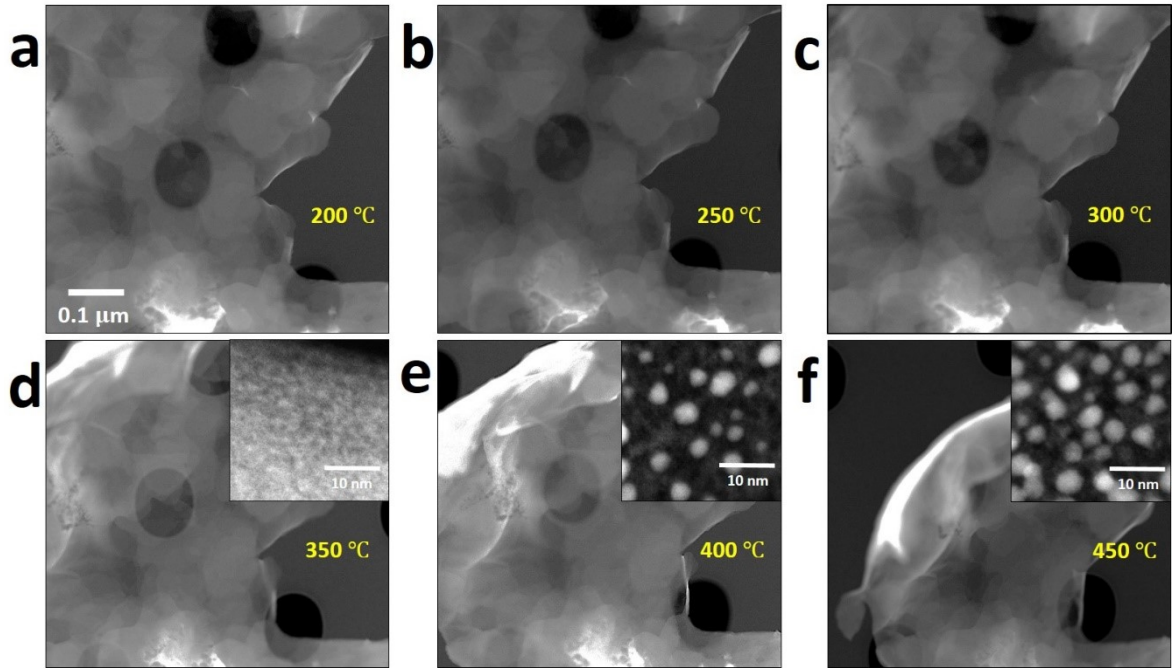

Fig. S3. HAADF-STEM images showing the phase transition according to heating pulse. The interval time at which each image was acquired is about 10 minutes.

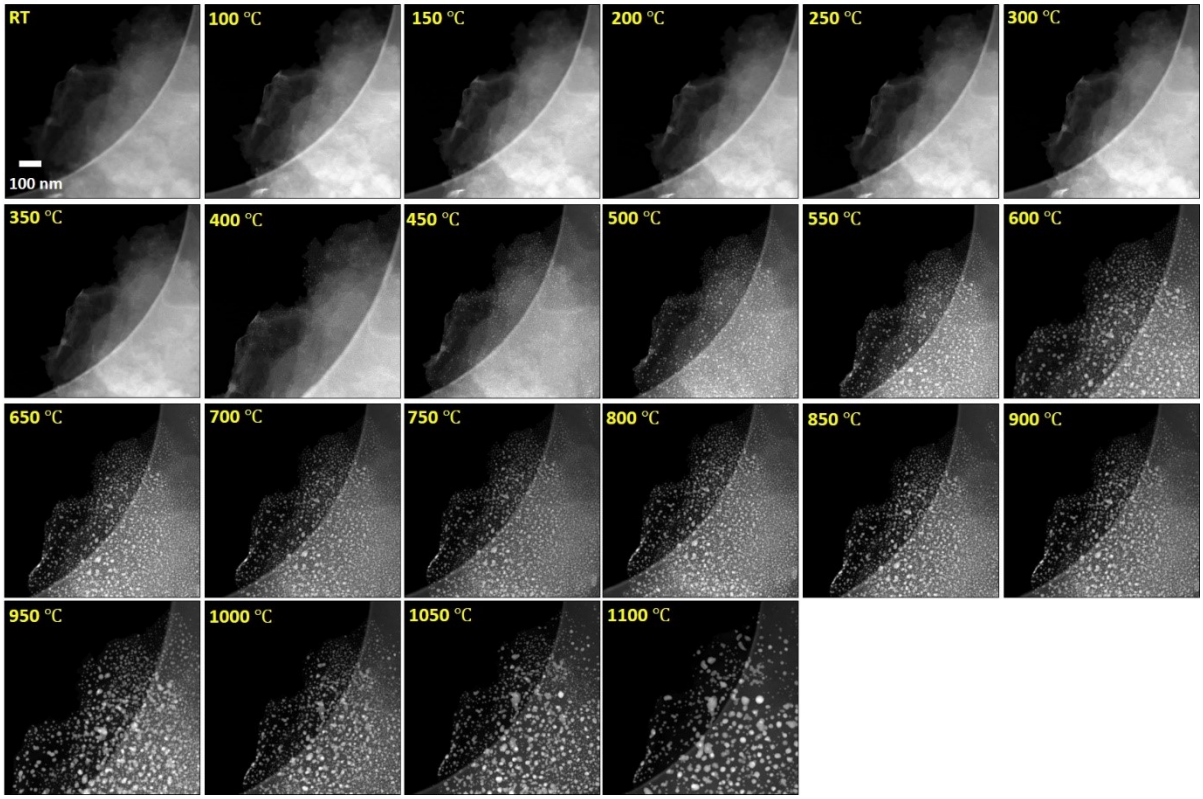

Fig. S4. Extended HAADF-STEM images showing the phase transition according to heating pulse from room temperature up to 1100 °C. The interval time at which each image was acquired is about 10 minutes.

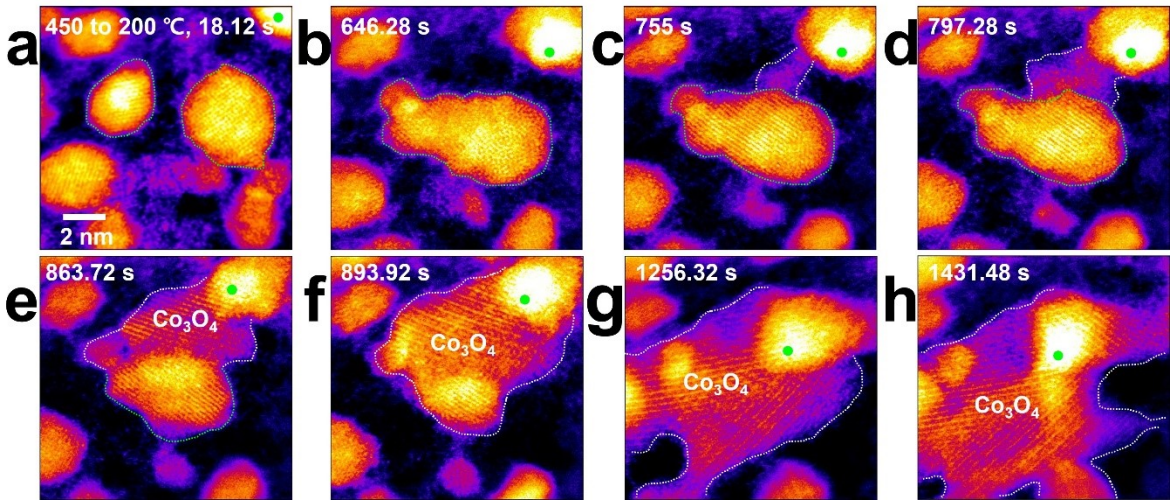

Fig. S5. Formation of Co_3O_4 from Co particles around Co and O atoms. (a)−(h) Successive HAADF-STEM images at the region based on a reference spot, which is colored green.

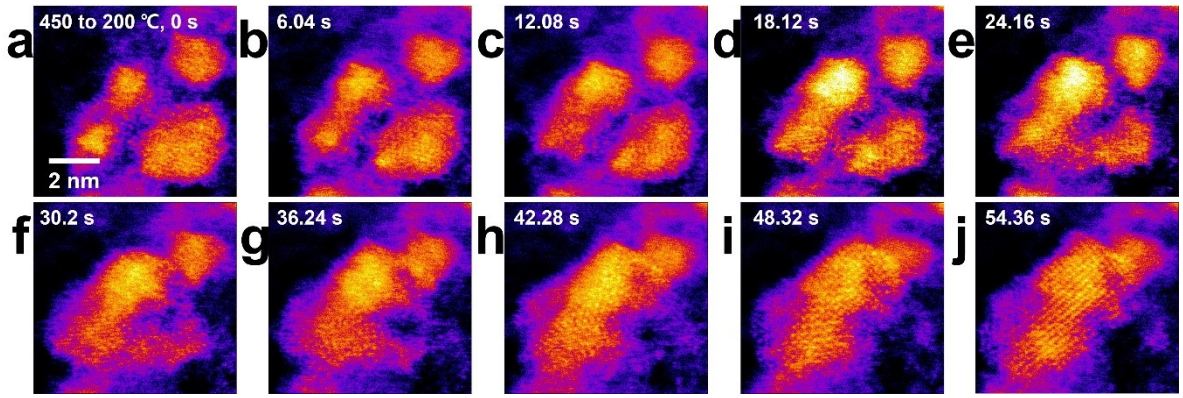

Fig. S6. Expansion of Co_3O_4. (a)−(j) Successive HAADF-STEM images at the same region.

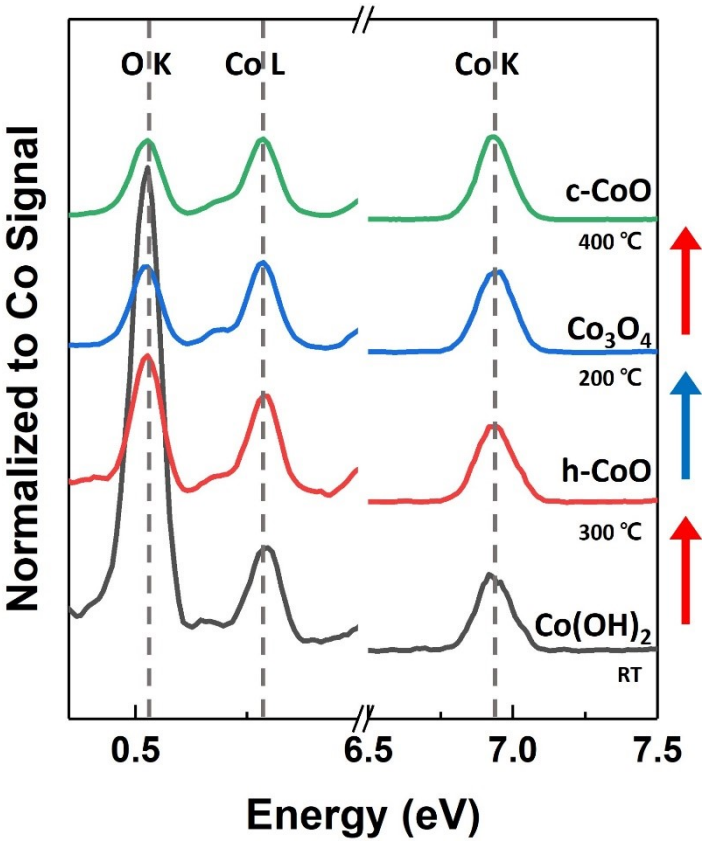

Fig. S7. EDS spectra taken from the $Co(OH)_2$, h-CoO, Co_3O_4, c-CoO by controlling the temperature. Red and blue arrow denote the annealing and cooling process. Each spectrum is normalized to Co signal for comparing the atomic ratio between Co and O.

Note. S1

We have confirmed the thermal transition of the $Co(OH)_2$ nanosheets that occurs in air using x-ray photoelectron spectroscopy (XPS). Basically, their chemical bonding states consist of mainly cobalt hydroxide with some cobalt oxides (Figure S7a). The peak of O1s at 531.1 eV indicates that the Co atoms are bonded with the OH^- group. The deconvolution of O 1s exhibits two clear peaks located at binding energy at 529.6 eV and 530.5 eV, which is attributed to oxygen in the Co-O of c-CoO crystal and Co_3O_4 crystal, respectively.[1,2] When heat is applied to the sample in an air atmosphere, as the temperature increases, the Co-O ratio increases, and the OH^- group ratio decreases (Figures S7b and c). However, when a temperature of 500 °C or more is applied, most of the sample is evaporated and the remaining sheet amount is significantly smaller, and further analysis is not possible. When the crystal structure is confirmed using HRTEM, the sample annealed at 200 °C is partially crystallized as shown in Figure S7d, and in the case of a higher temperature, the polycrystalline structure of the overall Co_3O_4 structure is confirmed (Figure S7e).

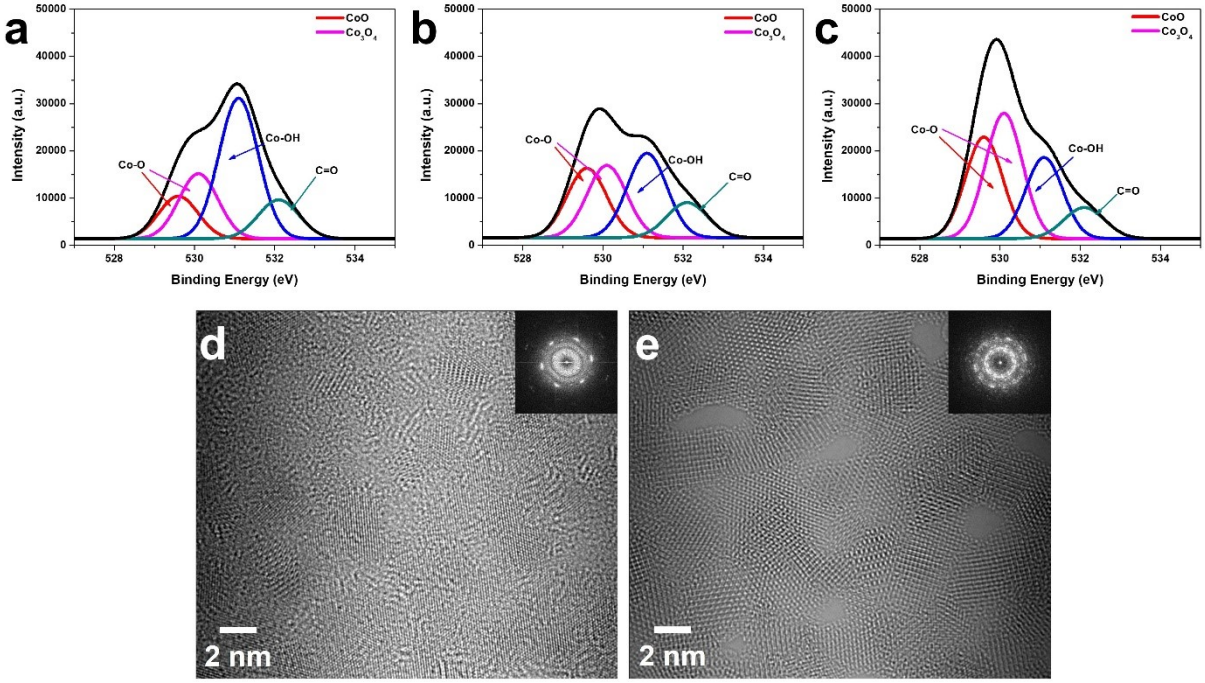

Fig. S8. XPS O 1s spectra of (a) the synthesized $Co(OH)_2$ nanosheets and the annealed nanosheets at (b) 200 °C and (c) 500 °C in air for 2 hours. HRTEM images show the annealed at (d) 200 °C and (e) 500°C.

References

1. S. C. Petitto, M. A. Langell. J. Vac. Sci. Technol. A 2004, **22**, 1690.
2. Y. Wang, Y. Shi, Z. Zhang, C. Carlos, C. Zhang, K. Bhawnani, J. Li, J. Wang, P. M. Voyles, I. Szlufarska, X. Wang. Chem. Mater. 2019, **31**, 21, 9040.