## **Electronic Supplementary Material (ESI)**

## Monitoring a CuO gas sensor at work: an advanced *in situ* X-ray absorption spectroscopy study<sup>†</sup>

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**Figure S1.** *In situ* measurements of urchin-like CuO nanostructures at 200°C under cyclic exposure to synthetic dry air as the baseline and to synthetic dry air (95%) plus  $H_2$  (5%) as the target gas. a) Time-resolved XANES spectra at the Cu K-edge; b) electrical resistance over time; c) copper fraction species over time and d) first derivative of the Cu K-edge XANES spectra at different times.

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**Figure S2.** *In situ* measurements of urchin-like CuO nanostructures at 300°C under cyclic exposure to synthetic dry air as the baseline and to synthetic dry air (95%) plus  $H_2$  (5%) as the target gas. a) Time-resolved XANES spectra at the Cu K-edge; b) electrical resistance over time; c) copper fraction species over time and d) first derivative of the Cu K-edge XANES spectra at different times.

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**Figure S3.** *In situ* measurements of urchin-like CuO nanostructures at 200°C under cyclic exposure to pure dry nitrogen as the baseline and to pure dry nitrogen (95%) plus  $H_2$  (5%) as the target gas. a) Time-resolved XANES spectra at the Cu K-edge; b) electrical resistance over time; c) copper fraction species over time and d) first derivative of Cu the K-edge XANES spectra at different times.

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**Figure S4.** *In situ* measurements of urchin-like CuO nanostructures at 300°C under cyclic exposure to pure dry nitrogen as the baseline and to pure dry nitrogen (95%) plus  $H_2$  (5%) as the target gas. a) Time-resolved XANES spectra at the Cu K-edge; b) electrical resistance over time; c) copper fraction species over time and d) first derivative of Cu the K-edge XANES spectra at different times.



**Figure S5.** EDX spectra of urchin-like CuO nanostructures (a) after reducing process with hydrogen gas and (b) after oxidation process with oxygen gas at 400°C