

## ***In situ* diffraction of highly dispersed supported platinum nanoparticles**

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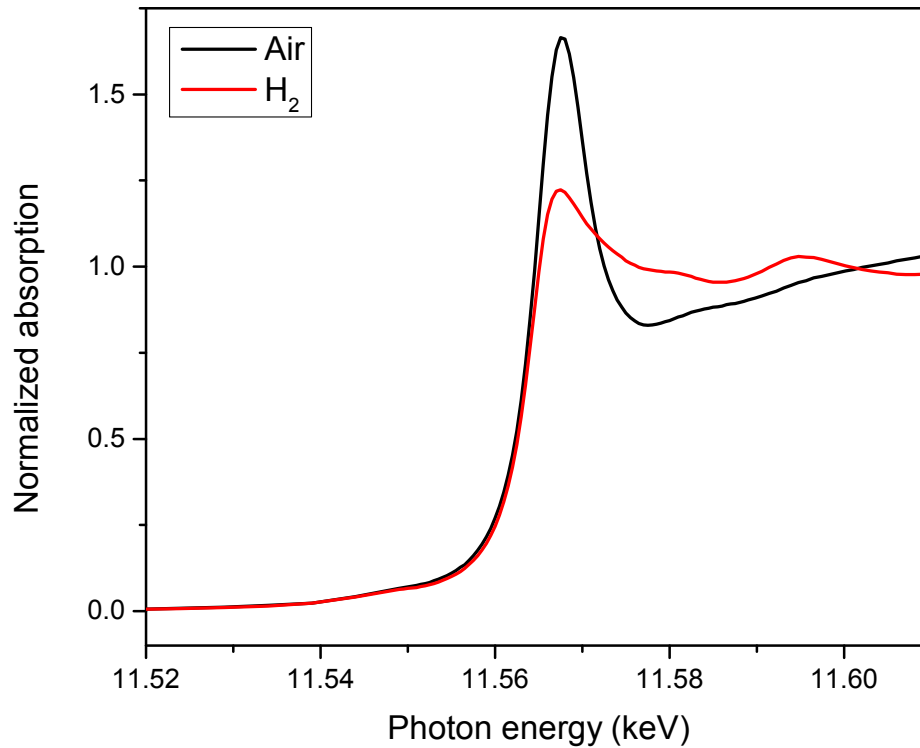
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### **Electronic Supplementary Information**

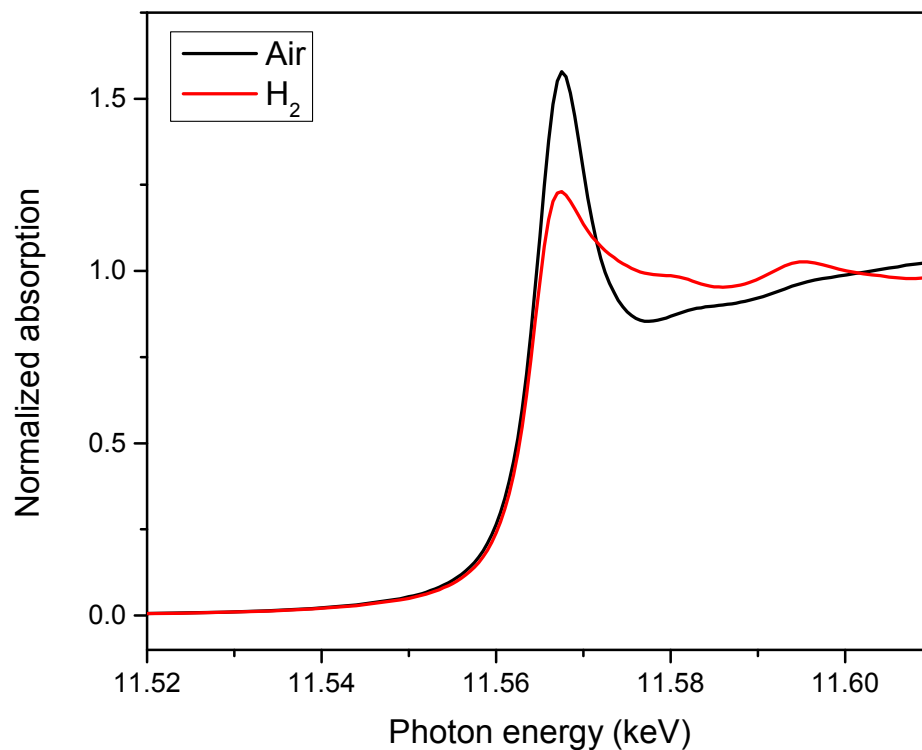
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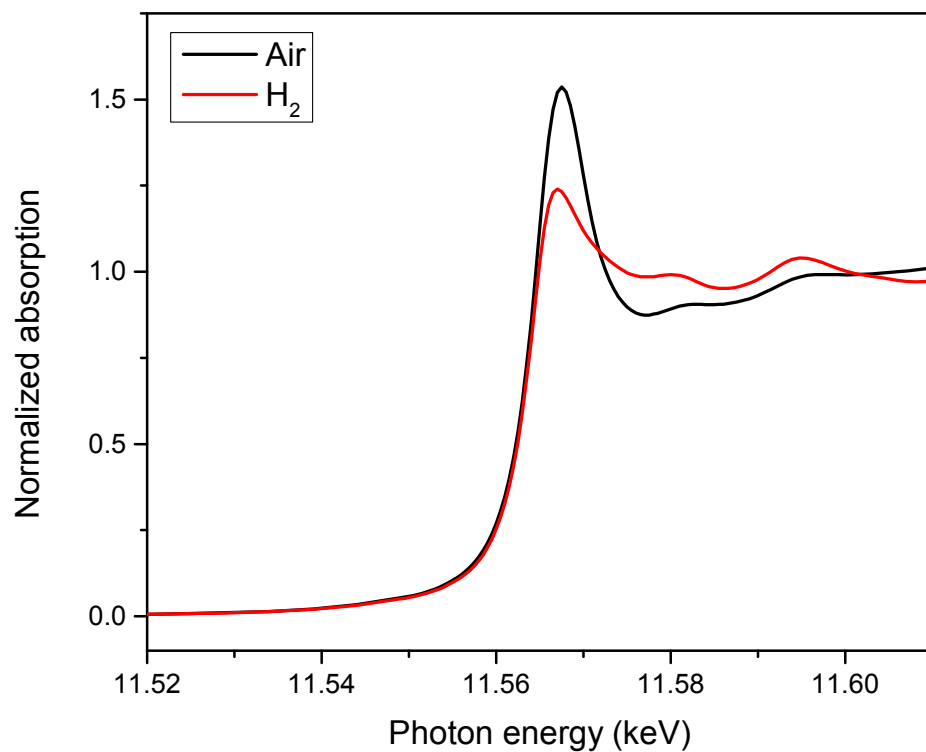
## XANES



**Supplementary figure S1.** XANES Pt L<sub>3</sub>-edge spectra from 11.52 keV to 11.61 keV of pre-reduced 2cPt-Al<sub>2</sub>O<sub>3</sub> in air (black) and after reduction in H<sub>2</sub> without exposure to air (red).

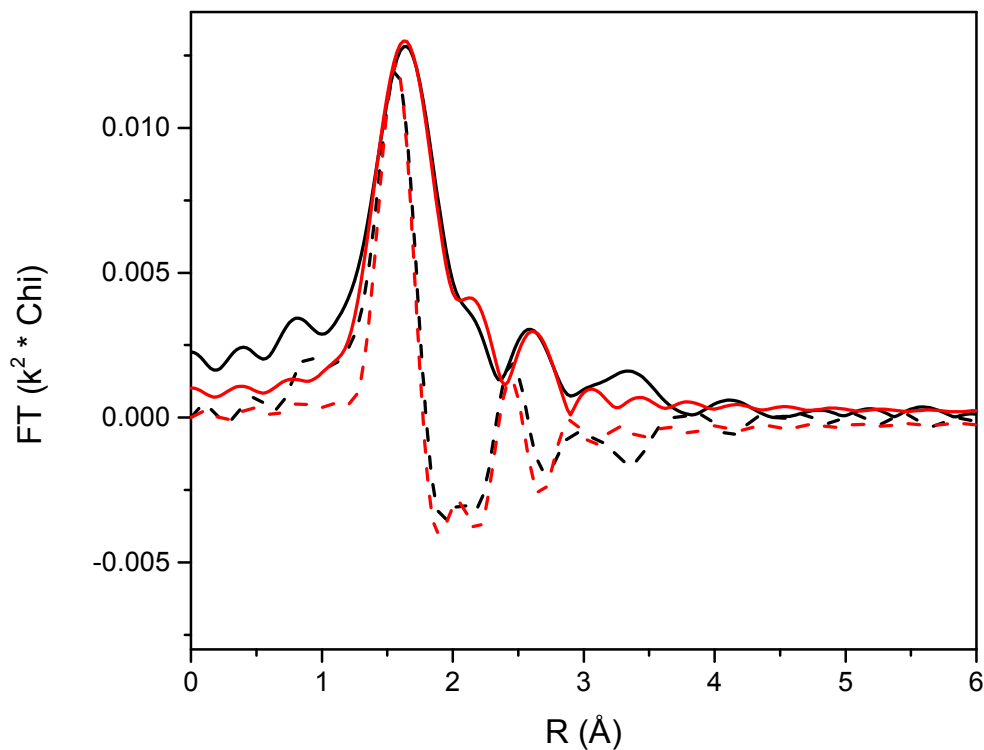


**Supplementary figure S2.** XANES Pt L<sub>3</sub>-edge spectra from 11.52 keV to 11.61 keV of pre-reduced 2cPt-CNT in air (black) and after reduction in H<sub>2</sub> without exposure to air (red).

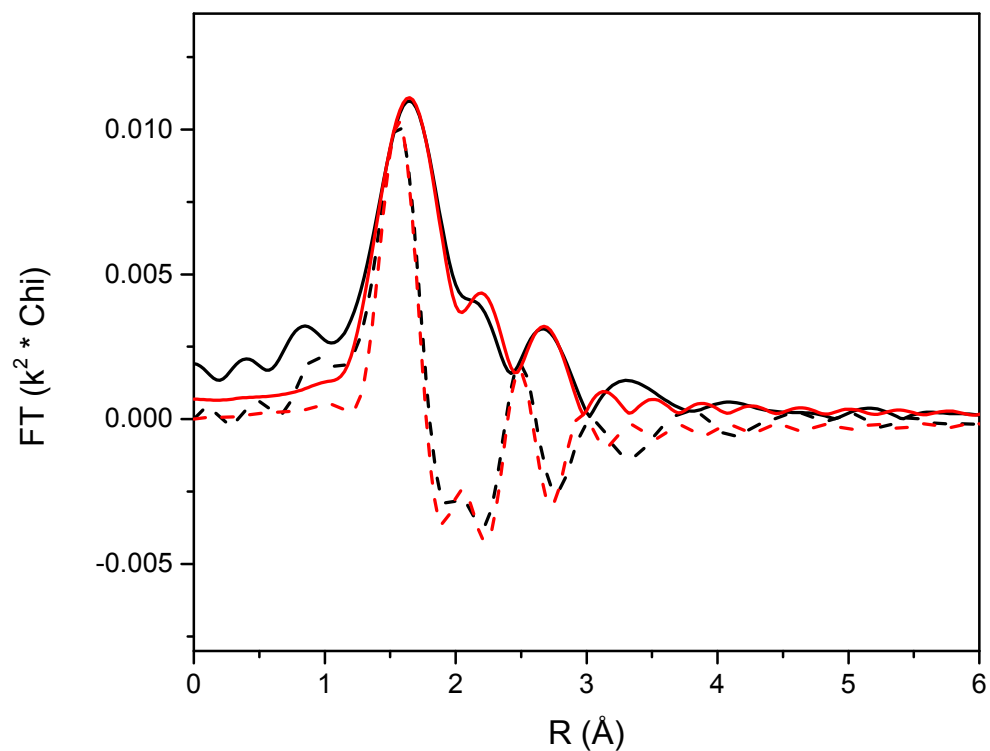


**Supplementary figure S3.** XANES Pt L<sub>3</sub>-edge spectra from 11.52 keV to 11.61 keV of pre-reduced 5cPt-Al<sub>2</sub>O<sub>3</sub> in air (black) and after reduction in H<sub>2</sub> without exposure to air (red).

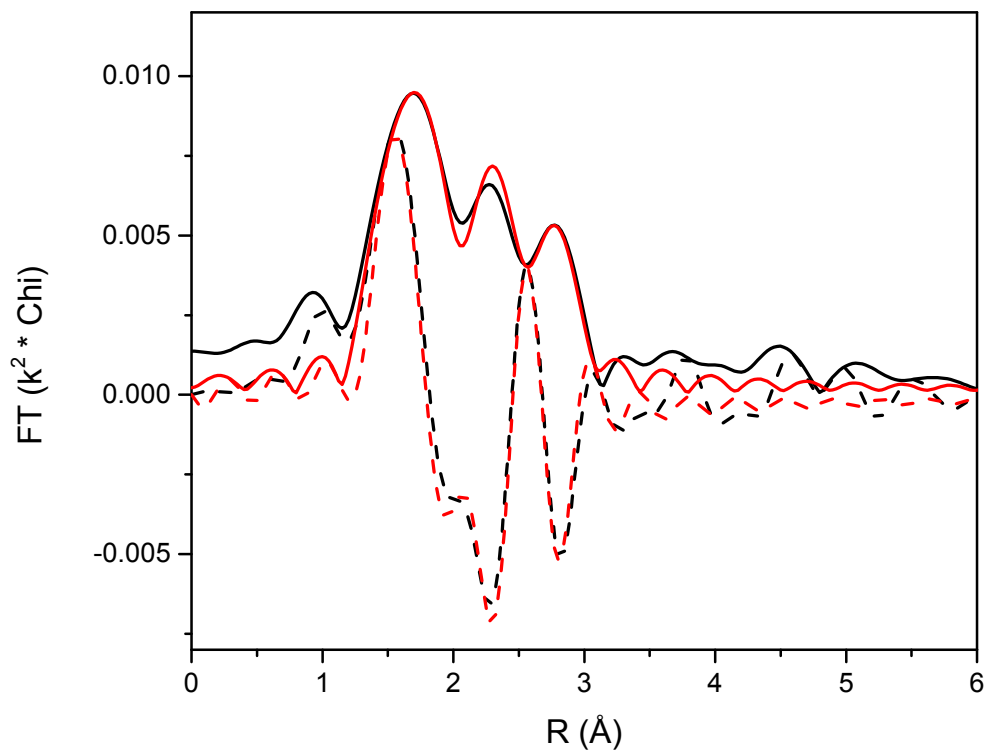
## EXAFS



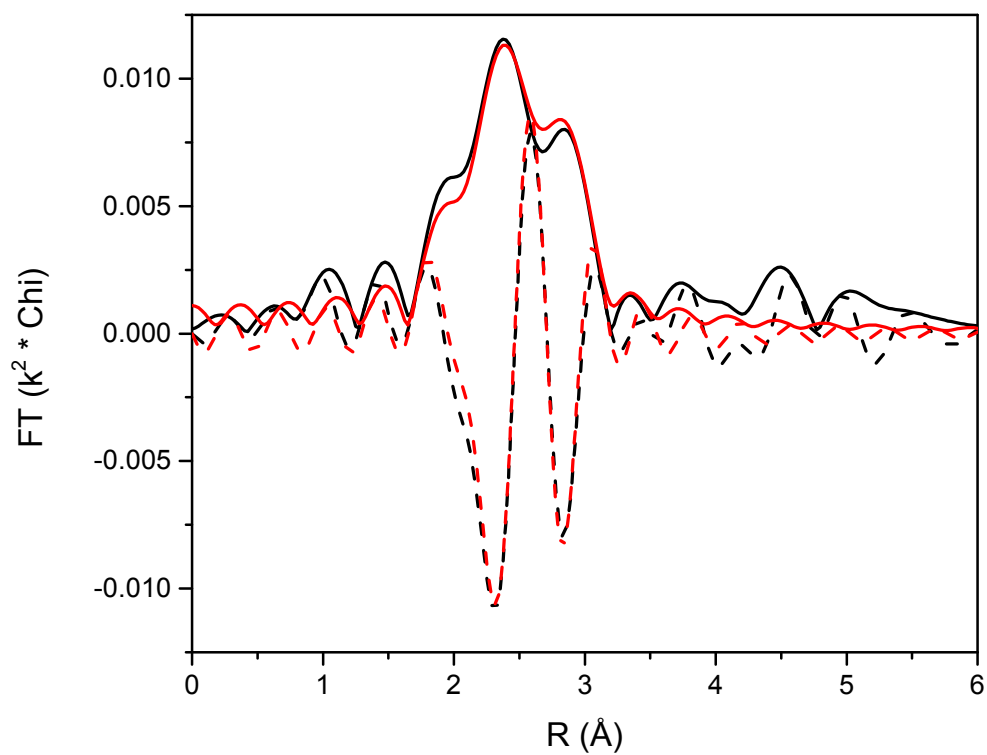
**Supplementary figure S4.** Fitting results of the R-space EXAFS spectrum of pre-reduced 2cPt-Al<sub>2</sub>O<sub>3</sub> in air. The solid black line represents the FT magnitude, the dashed black line the imaginary part of the FT while the red solid and dashed lines are the fits of the magnitude and the imaginary part respectively. ( $3.01 \text{ \AA}^{-1} < k < 11.42 \text{ \AA}^{-1}$ ,  $1.28 \text{ \AA} < R < 3.10 \text{ \AA}$ ).



**Supplementary figure S5.** Fitting results of the R-space EXAFS spectrum of pre-reduced 2cPt-CNT in air. The solid black line represents the FT magnitude, the dashed black line the imaginary part of the FT while the red solid and dashed lines are the fits of the magnitude and the imaginary part respectively. ( $3.01 \text{ \AA}^{-1} < k < 11.42 \text{ \AA}^{-1}$ ,  $1.28 \text{ \AA} < R < 3.10 \text{ \AA}$ ).

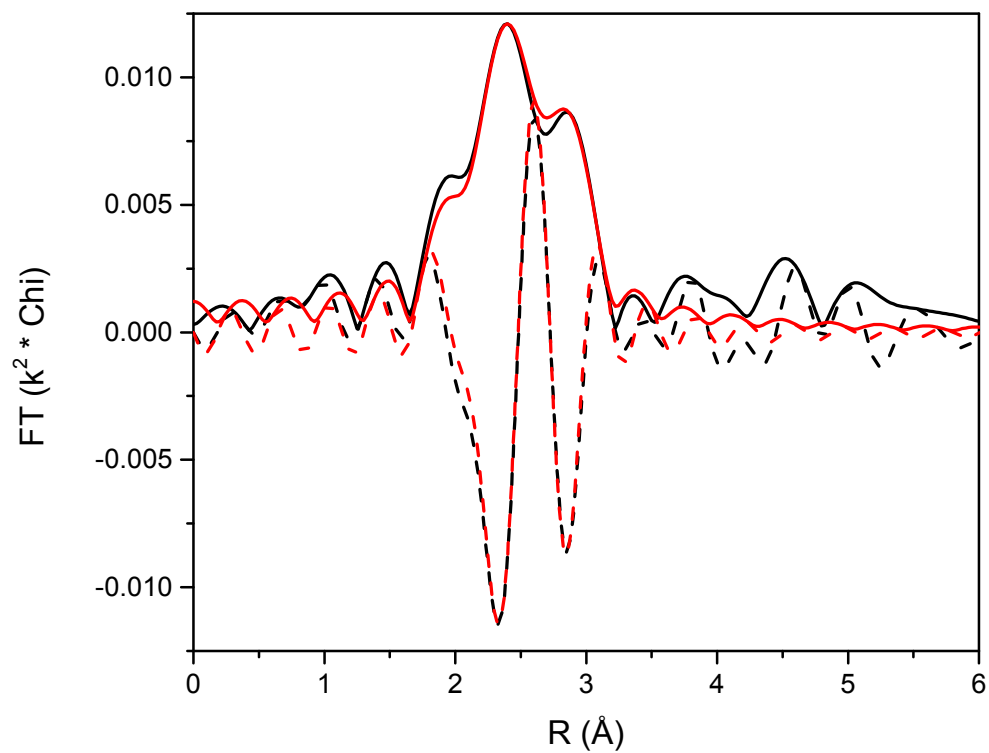


**Supplementary figure S6.** Fitting results of the R-space EXAFS spectrum of pre-reduced 5cPt-Al<sub>2</sub>O<sub>3</sub> in air. The solid black line represents the FT magnitude, the dashed black line the imaginary part of the FT while the red solid and dashed lines are the fits of the magnitude and the imaginary part respectively. ( $3.01 \text{ \AA}^{-1} < k < 11.42 \text{ \AA}^{-1}$ ,  $1.28 \text{ \AA} < R < 3.10 \text{ \AA}$ ).

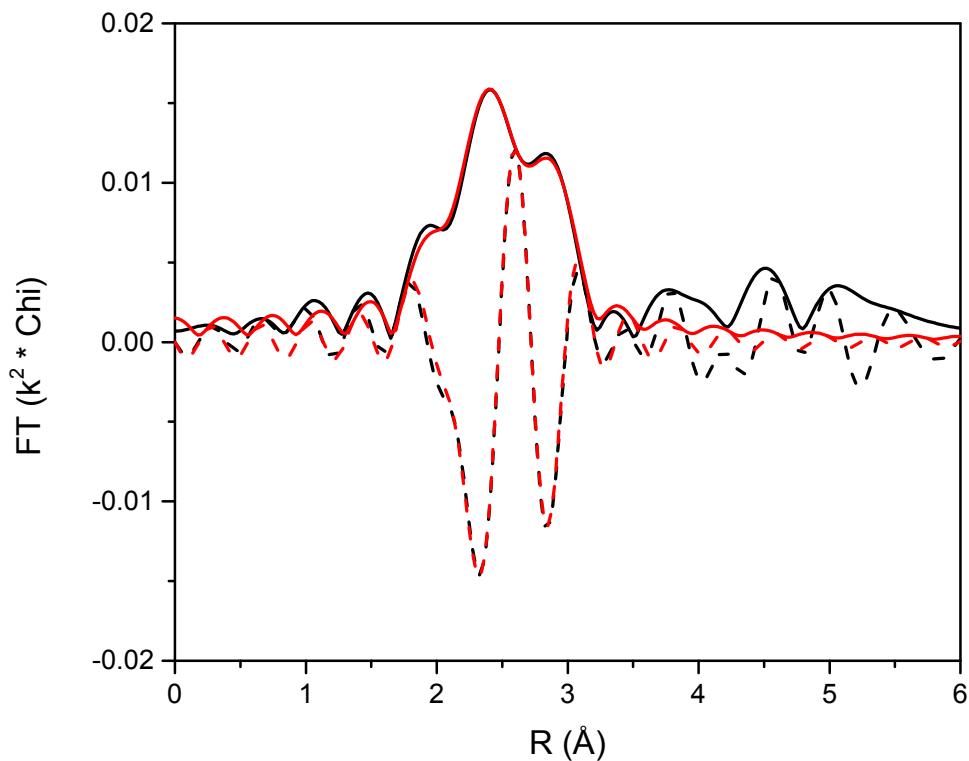


**Supplementary figure S7.** Fitting results of the R-space EXAFS spectrum of 2cPt-Al<sub>2</sub>O<sub>3</sub> in H<sub>2</sub>. The solid black line represents the FT magnitude, the dashed black line the imaginary part of the FT while the red solid and dashed lines are the fits of the magnitude and the imaginary part respectively. ( $3.01 \text{ \AA}^{-1} < k < 11.42 \text{ \AA}^{-1}$ ,  $1.77 \text{ \AA} < R < 3.10 \text{ \AA}$ ).



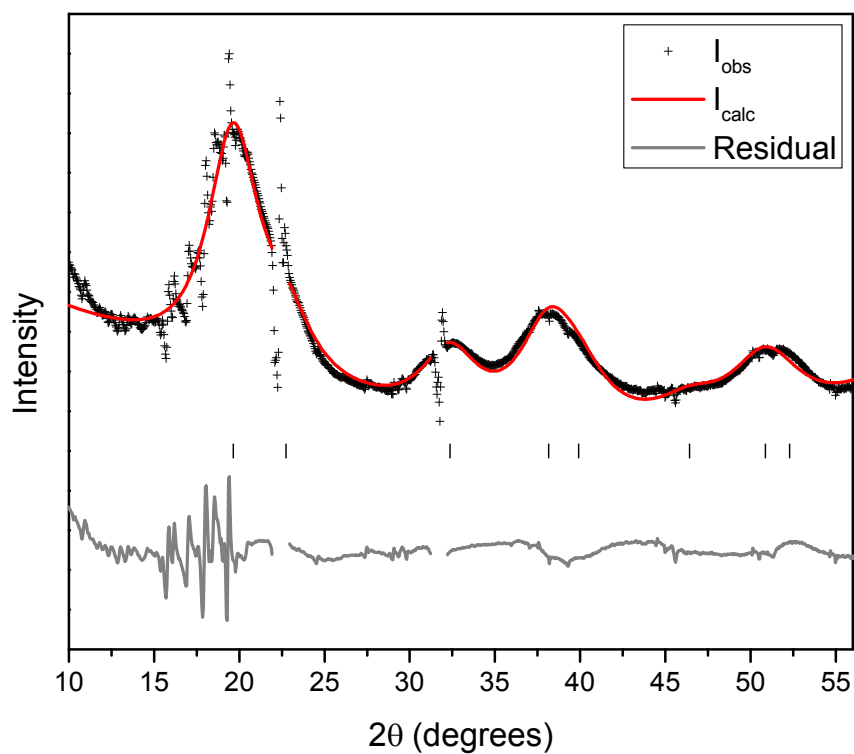


**Supplementary figure S8.** Fitting results of the R-space EXAFS spectrum of 2cPt-CNT in H<sub>2</sub>. The solid black line represents the FT magnitude, the dashed black line the imaginary part of the FT while the red solid and dashed lines are the fits of the magnitude and the imaginary part respectively. ( $3.01 \text{ \AA}^{-1} < k < 11.42 \text{ \AA}^{-1}$ ,  $1.77 \text{ \AA} < R < 3.10 \text{ \AA}$ ).

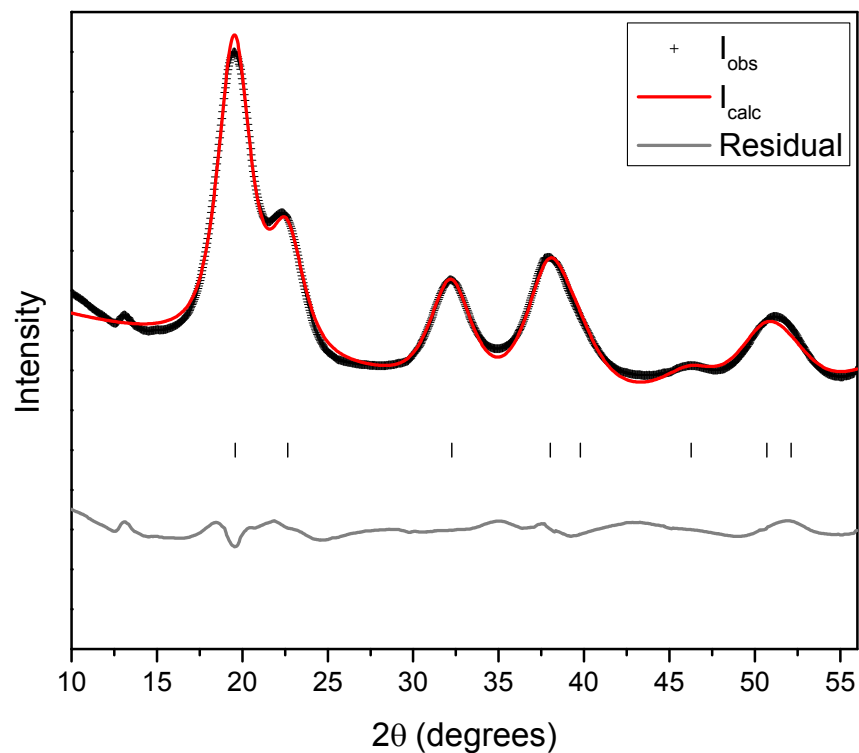


**Supplementary figure S9.** Fitting results of the R-space EXAFS spectrum of 5cPt-Al<sub>2</sub>O<sub>3</sub> in H<sub>2</sub>. The solid black line represents the FT magnitude, the dashed black line the imaginary part of the FT while the red solid and dashed lines are the fits of the magnitude and the imaginary part respectively. ( $3.01 \text{ \AA}^{-1} < k < 11.42 \text{ \AA}^{-1}$ ,  $1.77 \text{ \AA} < R < 3.10 \text{ \AA}$ ).

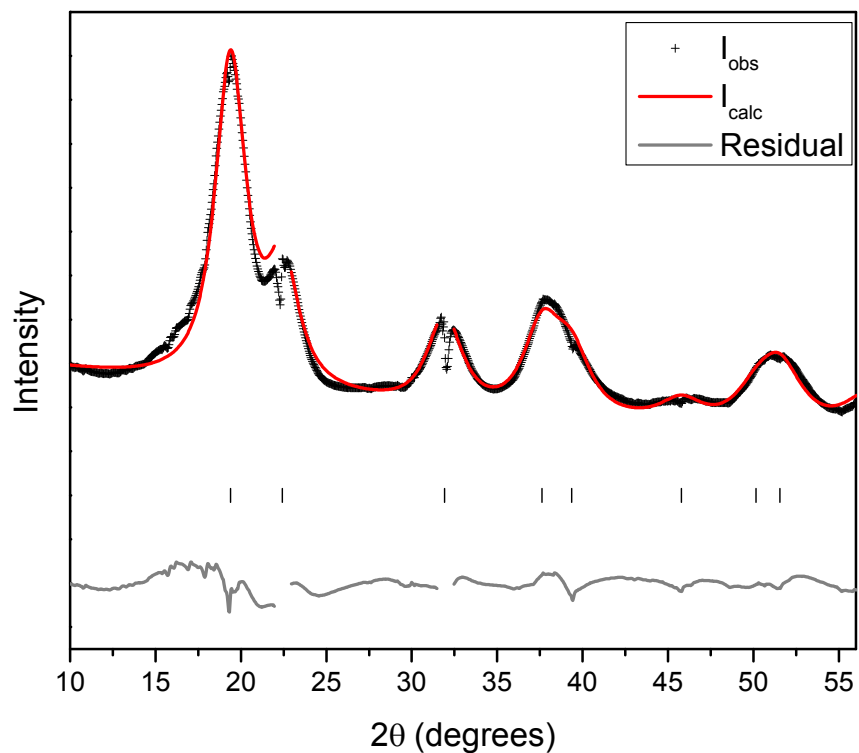
## XRD



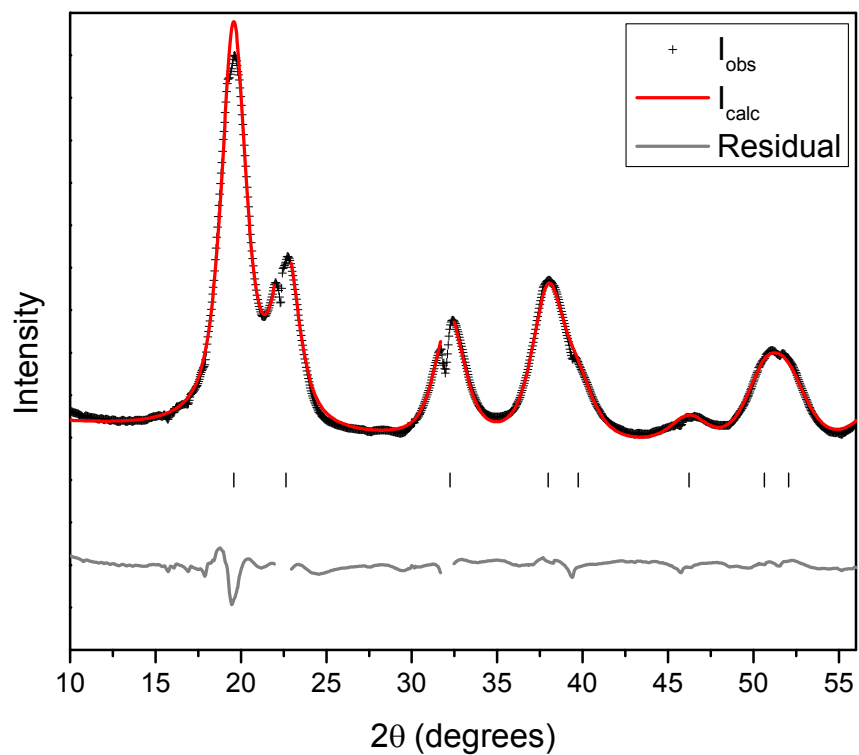
**Supplementary figure S10.** LeBail refinement of *in situ* synchrotron XRD data of 2cPt- $\text{Al}_2\text{O}_3$  in  $\text{H}_2$  at room temperature after subtraction of the contribution due to the  $\text{Al}_2\text{O}_3$  support. Black crosses represent observed data ( $I_{obs}$ ) and the red solid line is the calculated pattern ( $I_{calc}$ ); tick marks below the diffraction patterns are the positions of the reflections. The solid grey line is the residual ( $I_{calc}-I_{obs}$ ). Gaps in  $I_{calc}$  and residual are regions which were excluded from the refinement due to significant artifacts from the support subtraction.



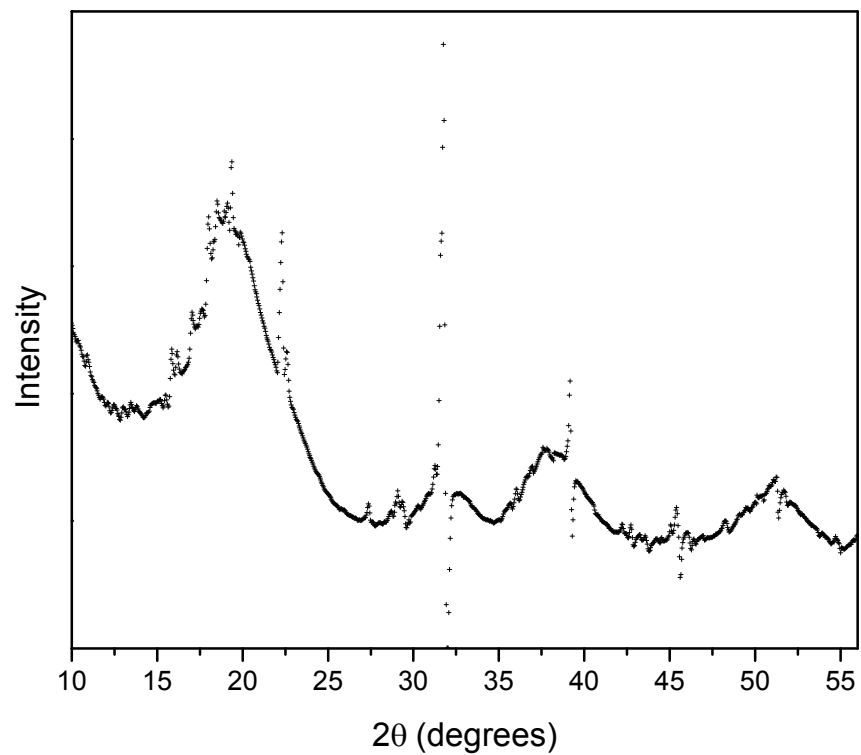
**Supplementary figure S11.** LeBail refinement of *in situ* synchrotron XRD data of 2cPt-CNT in H<sub>2</sub> at room temperature after subtraction of the contribution due to the carbon nanotubes support. Black crosses represent observed data ( $I_{\text{obs}}$ ) and the red solid line is the calculated pattern ( $I_{\text{calc}}$ ); tick marks below the diffraction patterns are the positions of the reflections. The solid grey line is the residual ( $I_{\text{calc}} - I_{\text{obs}}$ ). Gaps in  $I_{\text{calc}}$  and residual are regions which were excluded from the refinement due to significant artifacts from the support subtraction.



**Supplementary figure S12.** LeBail refinement of synchrotron XRD data of 5cPt- $\text{Al}_2\text{O}_3$  in air at room temperature after subtraction of the contribution due to the  $\text{Al}_2\text{O}_3$  support. Black crosses represent observed data ( $I_{\text{obs}}$ ) and the red solid line is the calculated pattern ( $I_{\text{calc}}$ ); tick marks below the diffraction patterns are the positions of the reflections. The solid grey line is the residual ( $I_{\text{calc}} - I_{\text{obs}}$ ). Gaps in  $I_{\text{calc}}$  and residual are regions which were excluded from the refinement due to significant artifacts from the support subtraction.



**Supplementary figure S13.** LeBail refinement of *in situ* synchrotron XRD data of 5cPt-Al<sub>2</sub>O<sub>3</sub> in H<sub>2</sub> at room temperature after subtraction of the contribution due to the Al<sub>2</sub>O<sub>3</sub> support. Black crosses represent observed data ( $I_{\text{obs}}$ ) and the red solid line is the calculated pattern ( $I_{\text{calc}}$ ); tick marks below the diffraction patterns are the positions of the reflections. The solid grey line is the residual ( $I_{\text{calc}}-I_{\text{obs}}$ ). Gaps in  $I_{\text{calc}}$  and residual are regions which were excluded from the refinement due to significant artifacts from the support subtraction.



**Supplementary figure S14.** *In situ* synchrotron XRD data of 2cPt-Al<sub>2</sub>O<sub>3</sub> after 2 min in H<sub>2</sub> at room temperature after subtraction of the contribution due to the Al<sub>2</sub>O<sub>3</sub> support.