

Electronic Supporting Information (ESI) for:

Oxygen-assisted reduction of Au species on Au/SiO₂ catalyst in room temperature CO oxidation

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1. Experimental

1.1 Catalyst preparation

The synthesis of the Au precursor Au(en)₂Cl₃ (en=ethylenediamine) is described previously¹. To prepare the Au/SiO₂ sample, 0.27 g Au(en)₂Cl₃ was dissolved in 150 ml H₂O, the pH of the solution adjusted to 9.8 by 5.0 wt.% NaOH solution, and 3.0 g Cab-O-Sil fumed SiO₂ (S_{BET} = 175 m²/g). The pH value of the solution dropped drastically after adding SiO₂, and was re-adjusted to approximately 9.8 by NaOH solution. The suspension was stirred at 60 °C for 2 h, then filtered and washed with H₂O to remove Cl⁻. The product was dried in vacuum at 70 °C for 5 h and then a reduction in 4% H₂/Ar at 120°C for 2 h to reduce the Au before the storage of the sample. ICP measurement showed the actual Au content is 4.0 wt% and confirmed undetectable Cl⁻ ion concentration. Before use the catalyst was calcined to 500 C to remove the ethylenediamine ligands

1.2 IR study

In a typical IR study, 12 mg Au/SiO₂ sample powder was calcined in 2% O₂/He at 500 °C (ramping rate of 10 °C/min at flow rate of 22 ml/min) for 2.5 h in a DRIFTS cell (HC-900, Pike Technologies). After cooling down to room temperature (23 °C) in O₂/He, a stream of 2% CO/2%Ar/He was added into the 2%O₂/He flow. The total flow rate was kept at 22 ml/min with CO : O₂ = 1 : 4 to achieve a space velocity of 110, 000 cm³/(h g_{cat}). IR spectra were collected during the reaction on a Thermo Nicolet Nexus 670 spectrometer while the exiting stream was analyzed by a quadruple mass spectrometer (QMS) (OmniStar GSD-301 O2, Pfeiffer Vacuum). In the case of flowing CO alone over the sample, the calcined sample was first purged with He at room temperature for 20 min before contacting the CO flow (CO/Ar/He : He = 1 : 4). After flowing for 20 h, He was substituted by O₂/He. IR spectra and QMS were followed the process all the time. QMS CO conversion (%) is calculated as following:

$$\text{Con\%} = 100 - 100 * P_{\text{CO}} / (P_{\text{CO}} + k * P_{\text{CO}_2})$$

where P_{CO}, P_{CO₂} are the QMS partial pressure of CO and CO₂, respectively; k is QMS response factor ration between CO and CO₂, calculated from a QMS calibration curve of a know mixture of CO and CO₂ with different CO/CO₂ ratios.

1.3 TEM measurement

Z-contrast STEM (Hitachi HD2000 STEM) measurements were made on the calcined Au/SiO₂ sample before and after CO oxidation at room temperature.

References:

1. H. G. Zhu, Z. Ma, J. C. Clark, Z. W. Pan, S. H. Overbury and S. Dai, *Applied Catalysis a-General*, 2007, **326**, 89-99.

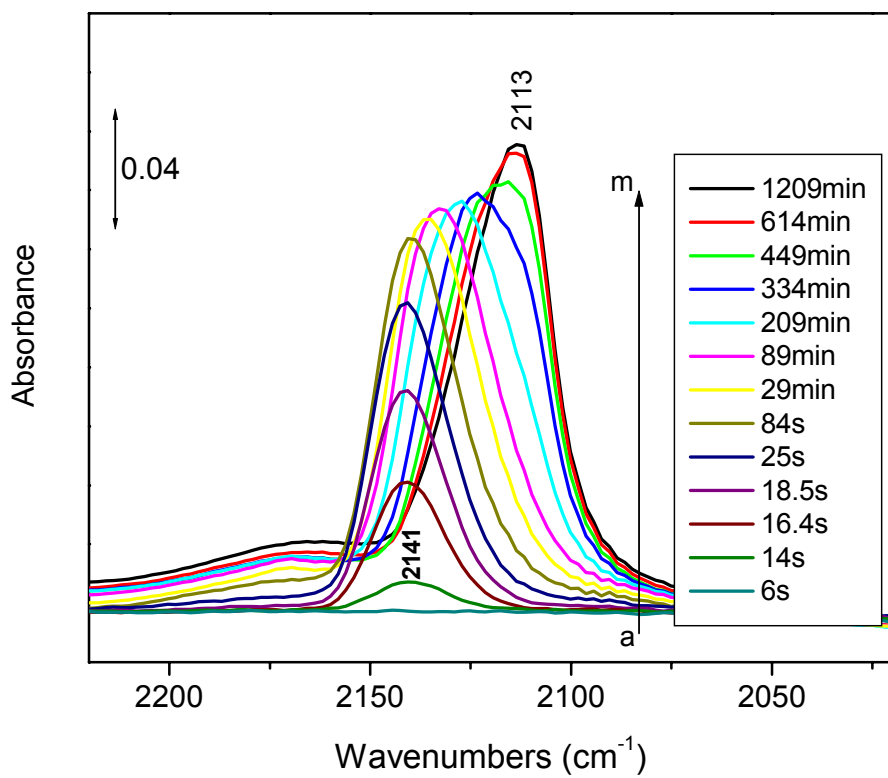
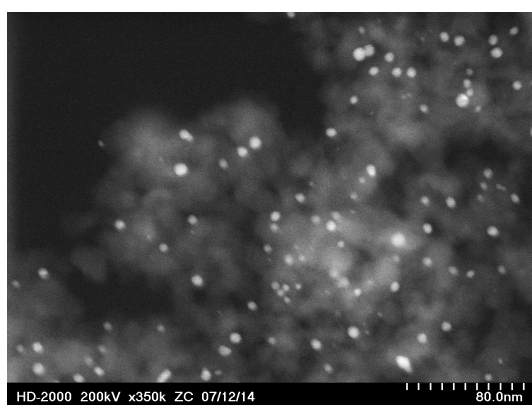
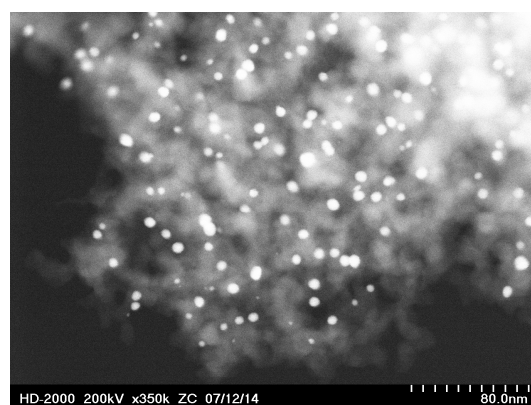
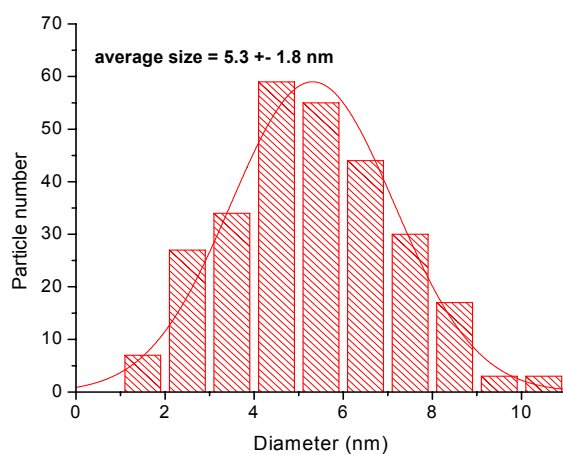


Figure S1. IR spectra during CO oxidation on calcined Au/SiO₂ at room temperature.



Before reaction



After reaction

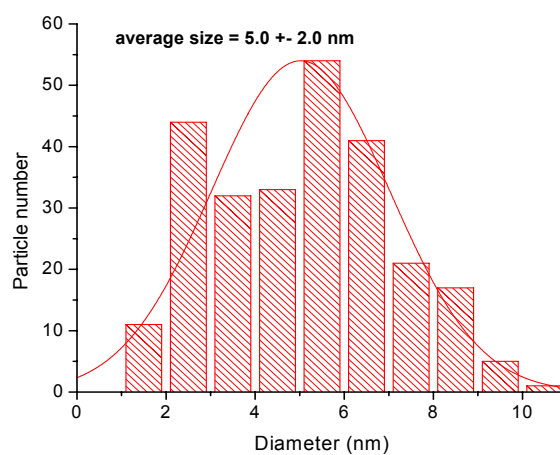


Figure S2. TEM graphs of 500°C -calcined Au/SiO₂ before and after CO oxidation (for about 20 hours) at room temperature.

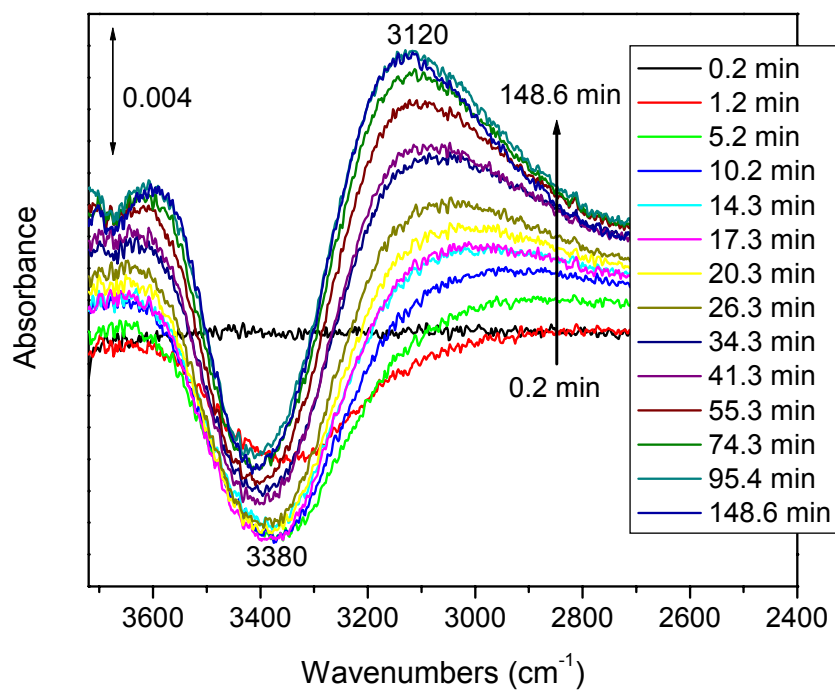


Figure S3. Difference IR spectra between before and after addition of O₂/He into CO flow on Au/SiO₂ after CO flows for 20 h.