GATAN

Experiment Brief

Model 628 *In-Situ* TEM Heating Holder

Title

Atomic scale observation of oxygen delivery during catalyzed oxidation of carbon nanotubes

Gatan instrument used

The model 628 is a furnace-type holder designed to allow the direct observation of microstructural phase changes, catalysis, nucleation, growth, and dissolution processes in 3 mm TEM samples at elevated temperatures.

Background

Heterogeneous catalysis plays an important role in many clean energy technology applications, including soot emissions reduction. In heterogeneous catalysis, reactants are transformed into products on the surface of a solid catalyst material. Current development of catalyst materials often focuses on nanoparticles (NP), since these have a large surface-to-volume ratio. Ag has been the subject of significant study for oxidation of soot and other carbonaceous materials. This work attempts to explore the atomic structure of Ag nanoparticle active sites for carbon oxidation using a model carbonaceous material: multi-walled carbon nanotubes (MW-CNT).

Materials and Methods

A model 628 heating holder from Gatan was used within an aberration-corrected environmental TEM (AC-TEM) to achieve an elevated temperature in an O_2 environment. Video recording was avoided, to keep the electron beam exposure as low as practically possible. Single frame images were acquired using an UltraScan® camera with the sample at 250 °C, but the oxygen removed to avoid ionization damage. A series of these images is shown in Figure 1.

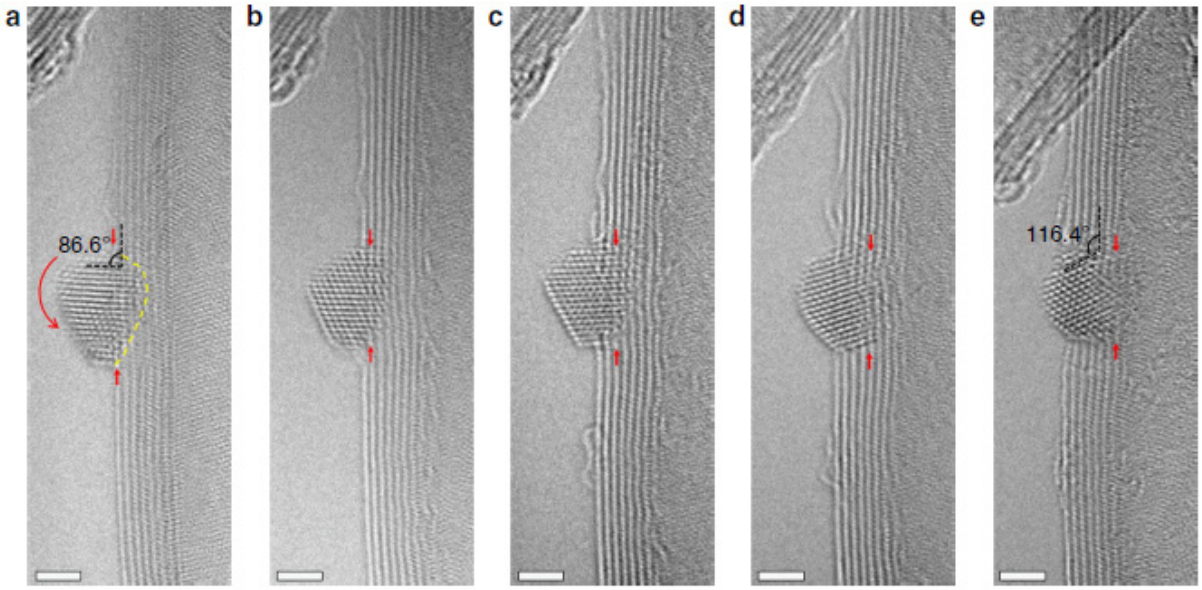

Figure 1. Series of AC-ETEM images during nanotube oxidation. The oxidation reaction was conducted at 250 °C in 2 mbar O_2 images were acquired after: (a) 0 s, (b) 300 s, (c) 600 s, (d) 900 s, and (e) 1,800 s. The two dashed black lines in a) mark the angle between the Ag NP/MW-CNT system, which was used to monitor the rotation of the Ag NP. The curved red arrow denotes the rotation direction, and the yellow dashed line marks the contact interface between the Ag NP and the MW-CNT. Scale bars are 2 nm. Small red arrows indicate the depth to which the oxidation has reached. The turnover frequency of the Ag NP sites on this single particle could be directly determined from these images.

Yue, Y., Yuchi, D., Guan, P., Xu, J., Guo, L., Liu, J., 2016. Atomic scale observation of oxygen delivery during silver-oxygen nanoparticle catalyzed oxidation of carbon nanotubes. Nature Communications 7, 12251. doi:10.1038/ncomms12251

Summary

The careful *in-situ* observation of Ag nanoparticles catalyzing the oxidation of carbon nanotubes was made possible through the use of a model 628 heating holder in an aberration-corrected ETEM. The high-resolution images of the catalyst particle at various points in the tube oxidation enabled the quantification of the turnover frequency for this single nanoparticle. First-principles simulations guided by these observations suggested a Mars-van Krevelen type mechanism, in which oxygen is dissociated on the Ag surface and diffuses through the Ag NP to reach the MW-CNT surface.

Credit(s)

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Gatan, Inc. is the world's leading manufacturer of instrumentation and software used to enhance and extend electron microscopes—from specimen preparation and manipulation to imaging and analysis.

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