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Characterization of Heterogeneous Catalysts in Different Environmental Transmission Electron Microscopes at 80kV to 1MV Accelerating Voltage

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Platinum nanocatalysts on titanium dioxide (Pt/TiO_2) exhibits remarkably high activity towards the decomposition reaction of organic molecules such as acetaldehyde and carboxylic acid [1]. Recently the size of Pt clusters has been reduced to less than 1 nm. In contrast, the size of TiO_2 support particles has been kept at sub-micron sizes to ensure stability at high temperature. The surface has also been modified to form a heterogeneous nanostructure. Although these complex nanocatalysts can be visualized by transmission electron microscopy, switching between several accelerating voltages improves the reproducibility and reliability of the results.

Here we report a progressive electron microscopy study of such heterogeneous catalysts using accelerating voltages of 80 kV to 1 MV. Switching voltages is also essential for estimating the beam effect when performing environmental transmission electron microscopy (ETEM). We present EM images of Pt/TiO_2 photocatalysts by advanced ETEM taken over this range of accelerating voltages.

Platinum nanoclusters are synthesized on titanium dioxide (TiO_2) surface by the photochemical deposition. The sample is characterized using a spherical aberration corrected differentially pumped environmental TEM (Titan ETEM, FEI Company) at 80-300kV [2], a modified double spherical aberration corrected environmental TEM/STEM (JEM-2200FS, The University of York) at 200kV [3], and Ultra-High Voltage environmental TEM (JEM-1000K RS, Nagoya University) [4] at 1MV.

Figure 1(a) is a schematic diagram showing the heterogeneous catalyst with wide range in size. Electron tomogram visualizes partly the complicated structure of the present Pt/TiO_2 (Figure 2). In the 80 keV TEM image, selective lattice imaging of the Pt nanoparticle was achieved as shown in Figure 3. In addition, greater contrast was obtained in comparison with a conventional 200 keV image. On the other hand, a 300 keV accelerating voltage provided clearer lattice imaging of the TiO_2 supporting particles. In the 1 MeV TEM image, surface steps and defect structures of TiO_2 were successfully visualized (Figure 4).

References

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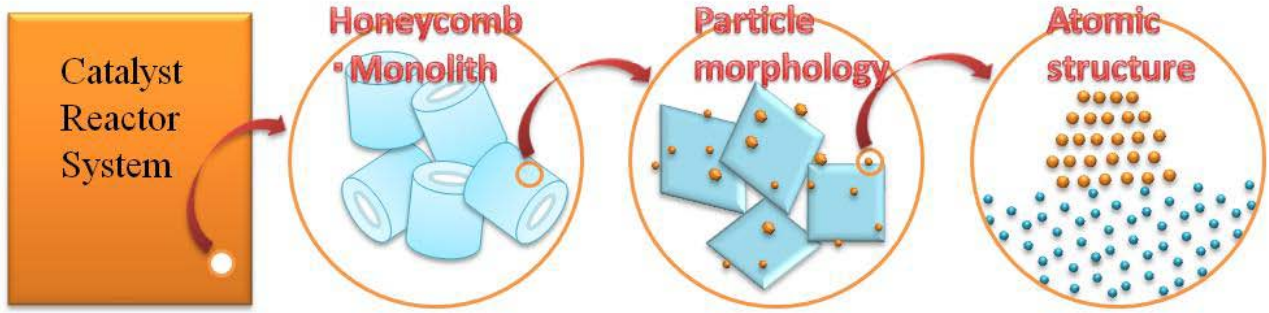

FIG. 1. Wide-scale analysis for heterogeneous catalysis.

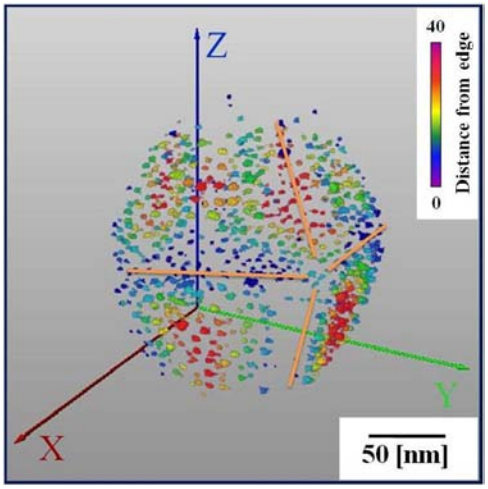

FIG. 2. Reconstructed 3D structure of the Pt/TiO_2. The green, red and blue arrows show the incident axis of the electron beam (Z axis), the rotation axis of tomography (Y axis) and another X axis, respectively. The scale bar is an estimate only. The voltex image was also produced (amira®, Visage Imaging Co.).

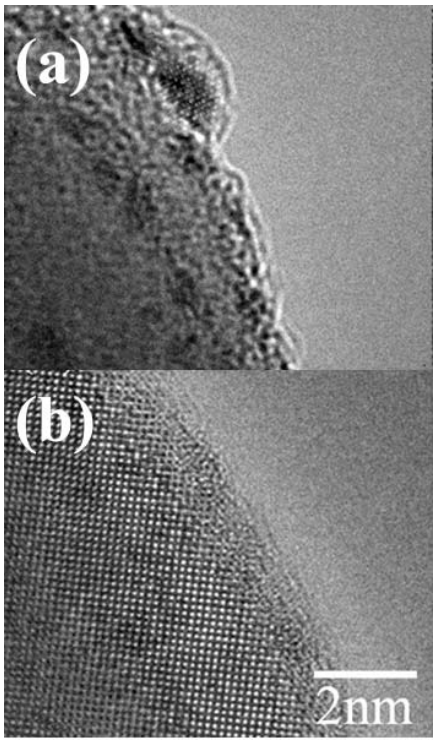

FIG. 3. AC-TEM image of Pt/TiO_2 obtained with 80kVand 300kV utilizing FEI Titan ETEM.

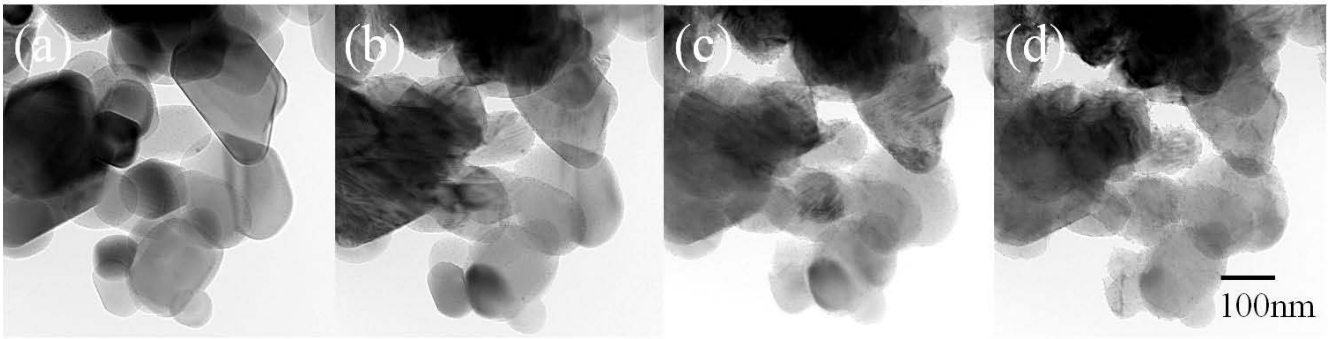

FIG. 4. In-situ observation of reduction process of Pt/TiO_2 by Ultra-High Voltage EM (JEM-1000K).

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