Supporting Information

In Situ Study Wet Chemical Etching of ZnO Nanowires with Different Diameter and Polar Surfaces by LCTEM

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Figure S1. (a-b) Scanning electron microscopy (SEM) images of ZnO nanowires. (c) High resolution transmission electron microscopy (HRTEM) image of ZnO nanowire. Inset: Fast Fourier transformation (FFT) pattern of the HRTEM image. The black circle outlines the (0000) transmission point.



Figure S2. TEM images of a typical ZnO nanowire on SiN_x window in the vacuum under electron beam irradiation with a constant dose rate of ~ 2000 e⁻/Å²·s. The scale bar is 100 nm for all the images. (a) TEM image of the nanowire before being irradiated by the electron beam with highdose rate. (b) TEM image of the nanowire after being irradiated by the high-dose rate electron beam for 120 s. (c) TEM image of the nanowire after being irradiated by the high-dose rate electron beam for 240 s.



Figure S3. (a-f) Selected time sequential TEM images showing the etching process of a ZnO nanowire with original diameter being 40 nm. The scale bar is 200 nm for all the images. Electron dose rate: 16.7 e⁻/Å²·s. The labeled time is the etching time. (g) The plots of the ZnO nanowire diameter pointed by red arrow in (a) as a function of the etching time.



Figure S4. (a-b) The enlarged time sequential TEM images of the two ends of the nanowire shown in Figure 2a during the etching process. The scale bar is 100 nm for all the images. The etching time is labeled in the images.



Figure S5. Selected time sequential TEM images from Movie S5 showing the formation and evolution of hillocks marked by the blue segments on O-terminated planes at the end of ZnO nanowire. The etching time is labeled in the images.



Figure S6. Selected time sequential TEM images from Movie S6 showing the evolution of hillock at the side of ZnO nanowire. The scale bar is 200 nm for all the images. The etching time is labeled in the images.



Figure S7. Selected time sequential TEM images from Movie S7 showing the evolution of hillock at the side of ZnO nanowire. The scale bar is 200 nm for all the images. The etching time is labeled in the images.



Figure S8. *Ex situ* characterizations of the nanoparticles after etching process. (a) TEM image of a nanowire shows nanoparticles on the side surface of the nanowire. (b) TEM image of one end of the nanowire. (c) Scanning transmission electron microscopy (STEM) image of the region outlined by the red box in Figure S8b. (d) Au elemental distribution mapping obtained by energy-dispersive X-ray spectroscopy (EDS) analysis. (e) TEM image of the end of the ZnO nanowire in the center in Figure 6a. (f) STEM image of the nanowire in (e). (g, h) EDS elemental distribution mapping of elements Zn, O.



Figure S9. Selected time sequential TEM images from Movie S8 show the shedding of sidewall cones at the end of ZnO nanowire. The scale bar is 500 nm for all the images. The etching time is labeled in the images.