Fabrication of Uniform Anatase TiO$_2$ Particles Exposed by \{001\} Facets

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Experimental information of controlling samples

TiF$_4$ aqueous solution was prepared by the method reported previously.$^1$ Hydrochloric acid (HCl, 1.5 M) was used to adjust the pH of deionized water (1.0 L) to 2.1. Titanium tetrafluoride (TiF$_4$, Aldrich Chemical) was then dissolved in this solution under vigorous stirring to give a concentration of 0.04 M. In a typical experiment, deionized water was used to dilute above TiF$_4$ aqueous solution (0.04 M) to a concentration of 2.5 mM. Then 60 mL of adjusted TiF$_4$ aqueous solution was added into a Teflon-lined stainless steel autoclave. The autoclave was kept at 170 °C for 6 - 14 h in an electric oven. After reaction, the products were separated by centrifugation, washed with deionized water for 5 times and then dried in vacuum at 60 °C for 7 h. Table 1 shows the detailed experimental parameters to synthesize TiO$_2$ particles without using disodium ethylene diamine tetraacetate (EDTA).

<table>
<thead>
<tr>
<th>Sample name</th>
<th>Concentration of TiF$_4$ (mM)</th>
<th>Temperature (°C)</th>
<th>Time (h)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S1</td>
<td>2.5</td>
<td>170</td>
<td>6</td>
</tr>
<tr>
<td>S2</td>
<td>2.5</td>
<td>170</td>
<td>14</td>
</tr>
</tbody>
</table>

Materials Characterization

The morphology and structure of the samples were characterized by high-resolution
transmission electron microscopy and selected area electron diffraction (HRTEM/SAED, Philips Tecnai T30F FEG Cryo AEM), scanning electron microscopy (SEM, JEOL JSM6400F), X-ray spectroscopy (XRD, Bruker D8 Advanced Diffractometer, Cu KR radiation, 40 kV). Surface binding elements were analyzed with X-ray photoelectron spectroscopy (XPS, Kratos Axis Ultra DLD). All binding energies were referenced to the C1s peak (284.8 eV) arising from surface hydrocarbons (or possible adventitious hydrocarbon). Prior peak deconvolution, X-ray satellites, and inelastic background (Shirley-type) were subtracted for all spectra. Samples were centrifuged and washed with deionized water twice and then redispersed in water and dropped on a conductive SEM sample holder or a carbon-coated copper grid with irregular holes for TEM analysis. Samples for XPS were prepared by drying the sedimented particles 24 h at 30 °C then slightly grinding and drying another 4 h at 60 °C so that the samples were fully dried.

Reference


Fig. S1 XRD patterns of samples S1 (above) and S2 (below).
Fig. S2 SEM/TEM/HRTEM images of sample S1.
Fig. S3 SEM/TEM images of sample S2.