

Electronic Supplementary Information for

Supercritical Synthesis of Layered Elongated Hexagonal Titanium Phosphate Nanoplates

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Experiment Section

Ti(OC₄H₉)₄ (Tetrabutyl titanate) was supplied by Beijing Jinlong Chemicals Company. Phosphoric acid (≥85%) and dehydrated alcohol were purchased from Beijing Chemicals Company. All chemicals are analysis grade and used as received without further purification.

In a typical experiment, Tetrabutyl titanate (0.2 – 1.0 g) was first added into a cell with 8.3 g anhydrous ethanol to get very stable tetrabutyl titanate/ethanol solution. Then phosphoric acid (~5.2 g) was added under stirring to get a clear solution. The mixture solution was transferred into a

stainless steel autoclave which has a volume of 20 ml, and bear the maximum working pressure and temperature are 16 MPa and 280 °C respectively. The autoclave was kept at a certain temperature (200 °C, 250 °C and 270 °C, respectively) for 5 hours or 10 hours, then allowed to cool to room temperature naturally. The product was washed for several times with ethanol to remove residual phosphoric acid and then dried at 80 °C for 10 hours. The synthesis route was showed as follows:

Powder X-ray diffraction (XRD) patterns were recorded on a Philips PW1820 diffractometer with Cu K α radiation. Scanning electron microscopy (SEM) was carried out with a Philips XL-20 at 15 keV. HRTEM images were obtained with JEOL JEM-2100F at 200 kV. Thermogravimetric analyses were performed using a DTG60 instrument under air with a heating rate of 10 °C/min in 195–900 °C temperature range. Fourier transform infrared (FT-IR) spectroscopy was performed with a Perkin-Elmer Spectrum 2000 spectrometer. KBr pellet technique was used for the framework vibration characterization. X-ray photoelectron spectroscopy data were obtained with an ESCALab220i-XL electron spectrometer from VG Scientific using 300 W AlK α radiation. The base pressure was about 3×10^{-9} mbar. The binding energies were referenced to the C1s line at 284.8 eV from adventitious carbon.

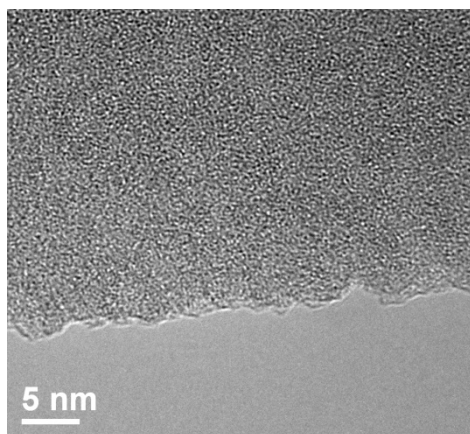


Figure S1. HRTEM image of elongated hexagonal TiP nanoplate.

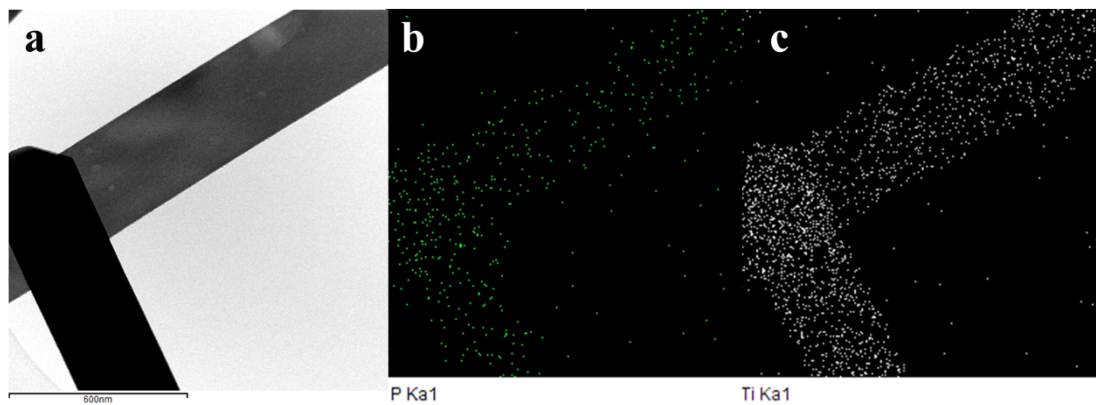


Figure S2. STEM image of TiP (a) and elemental mapping of P and Ti (b and c).

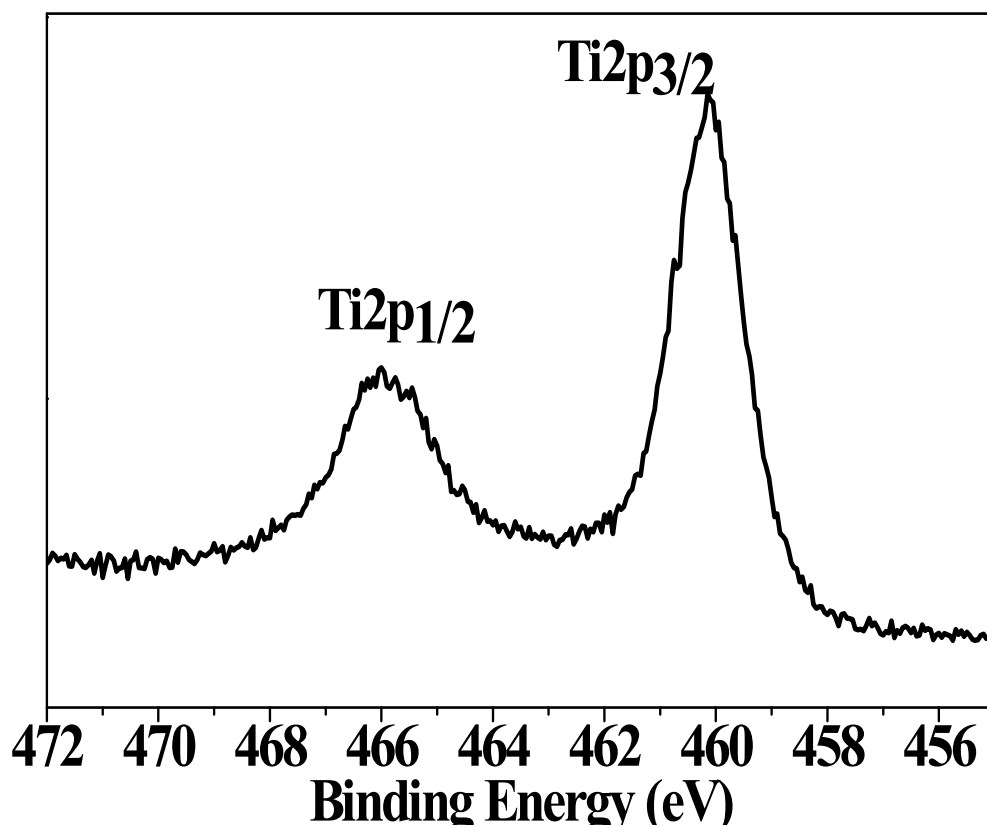


Figure S3. XPS spectrum of Ti2P of α -TiP obtained at 250 °C for 5 hours.

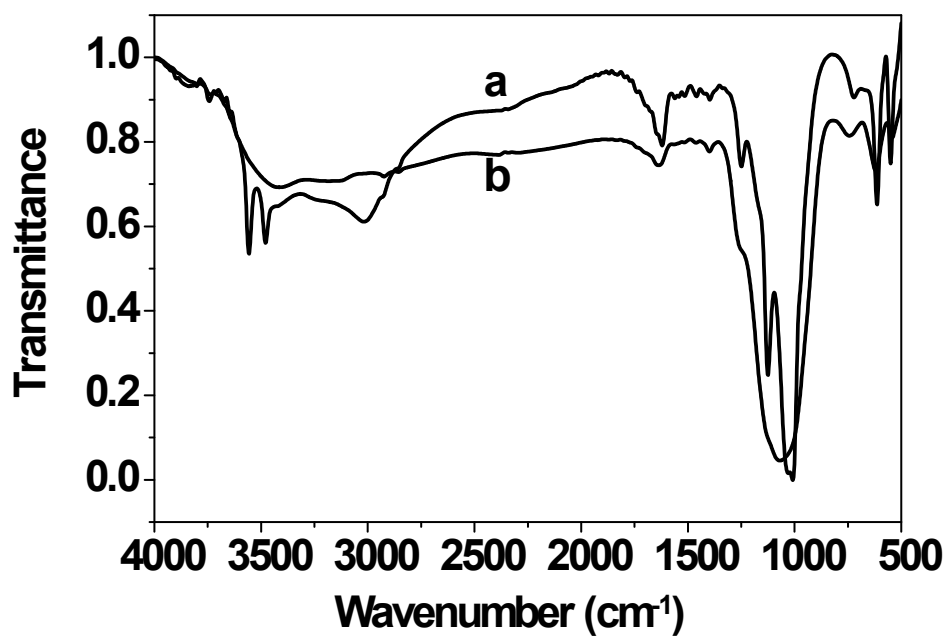


Figure S4. FTIR spectra of TiPs obtained at 250 °C for 5 hours without calcination (a) and with calcination at 500 °C (b).

