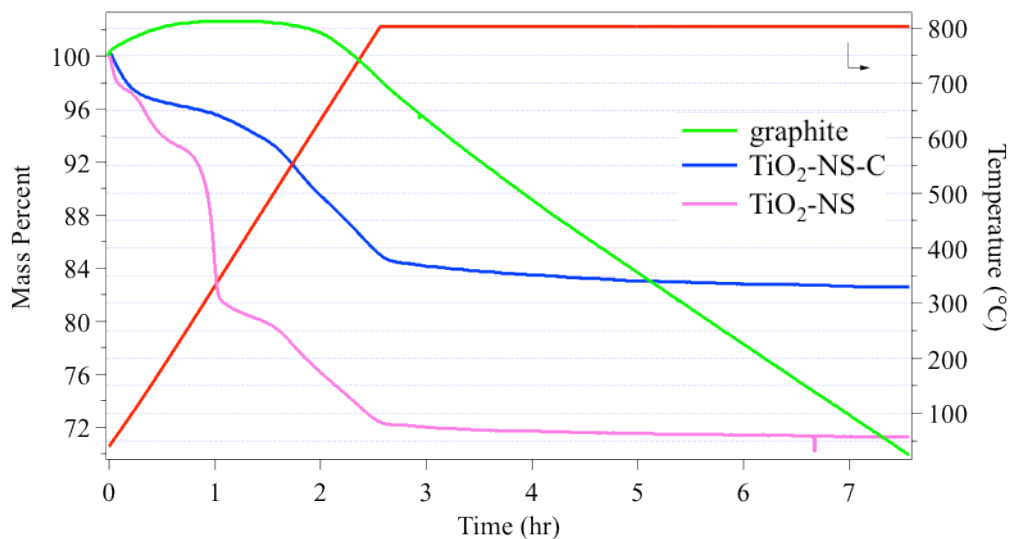
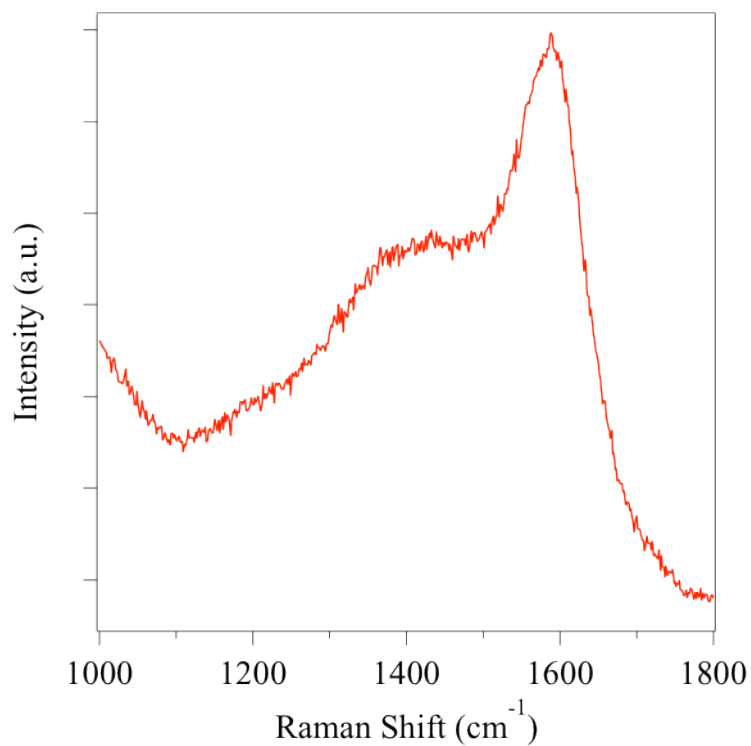


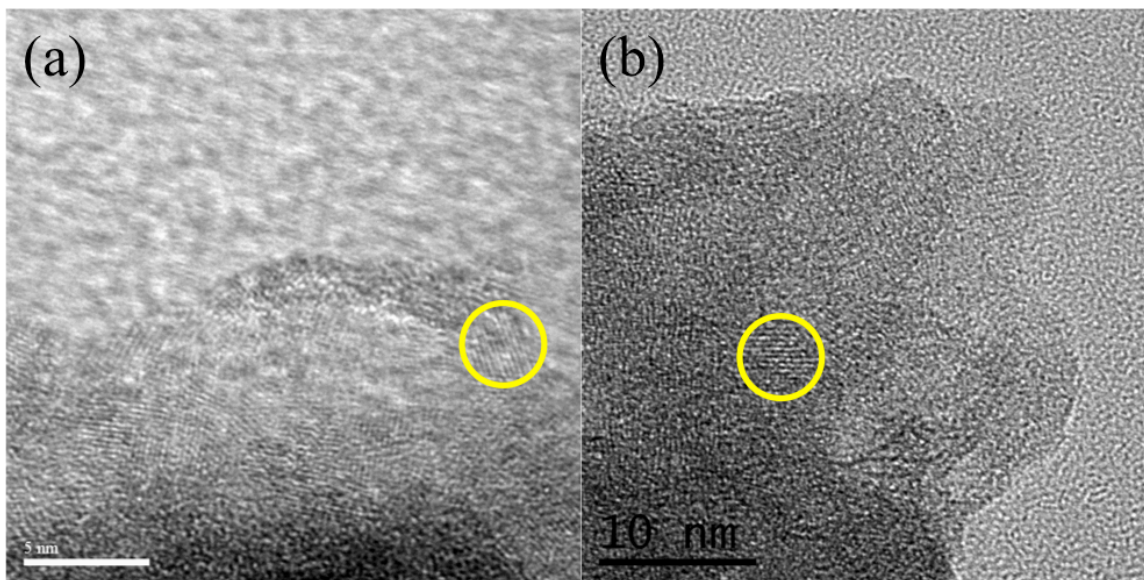
Supporting information for “Facile formation of Pt and PtPd nanoparticles on reactive carbon-TiO₂ nanosheet substrates.”



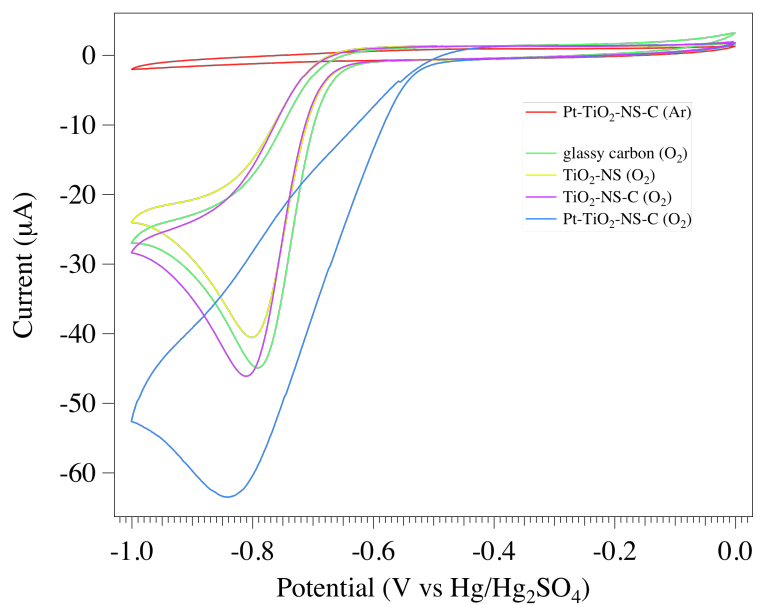
S.I. 1. TG Analysis (TGA) of TiO₂-NS and TiO₂-NS-C performed in air atmosphere at a ramp rate of 5 °C/min. The TGA profile of pure graphite is included as reference. TiO₂-NS shows considerable mass loss between 200 and 350 °C likely due to decomposition of surface ethylene glycol adsorbates followed by further mass loss starting at 525 °C that is due to oxidation of graphite-like surface carbon species. In the case of TiO₂-NS-C, minimal mass loss is observed up to 400 °C due to ethylene glycol already being decomposed as surface graphite species from the reforming gas treatment at 300 °C. The remaining mass loss observed for TiO₂-NS-C at temperatures above 500 °C track well with the graphite reference. These results suggest that ethylene glycol first catalytically decomposes on the TiO₂-NS surface to form TiO₂-NS-C followed by further oxidation of surface graphite-like species at temperatures above 400 °C.



S.I. 2. Raman spectra of TiO₂-NS-C. The bands centered at 1380 and 1580 cm⁻¹ are assigned to the D and G bands commonly observed for graphite. The spectrum was collected using a Renishaw InVia Raman microscope equipped with a 3 mW 514 Ar⁺ laser focused to a 1 μm spot size.



S.I. 3. TEM of TiO₂-NS (a) and TiO₂-NS-C (b). Both systems show the ultra thin sheet morphology as well as domains of crystallinity circled in yellow.



S.I. 4. Cyclic voltammetry of Pt-TiO₂-NS-C on glassy carbon in Ar and O₂ saturated 0.1 M KOH solutions. See characterization section for further details.

Characterization: Dilute solutions of the materials were drop cast onto carbon coated Cu transmission electron microscopy (TEM) grids and allowed to dry at room temperature. Scanning electrochemical microscopy (SEM) was performed on a Hitachi S-5500 operated at 30kV and high resolution TEM (HR-TEM) was performed on a Jeol 2010F operated at 200 kV. X-ray diffraction (XRD) data were collected in transmission mode on a Rigaku R-Axis Spider diffractometer with an image plate detector using a graphite monochromator with Cu K α radiation ($\lambda = 1.5418\text{\AA}$). The powder sample was mounted on a Hampton Research CryoLoop. Thermogravimetric analysis was collected on a Mettler-Toledo TGA/DSC-1 under air atmosphere. Raman spectra were acquired using a Renishaw InVia microscope equipped with a 514 nm Ar⁺ laser operating at 3 mW.

Cyclic voltammetry (CV) measurements were performed using a CHI 400D potentiostat operated between 0 and -1.0 V at a scan rate of 10 mV/s with 0.1 M KOH serving as the electrolyte. Ar or O₂ was bubbled through the electrolyte for 20 minutes prior to CV and only the 5th cycle is shown. 5 μL of an ink containing Pt-TiO₂-NS-C, water and Nafion was deposited on a polished glassy carbon (GC) electrode to serve as the working electrode. Au wire and Hg/Hg₂SO₄ served as the counter and reference electrodes, respectively.

Synthesis of TiO₂-NS: See reference from main text.

Synthesis of TiO₂-NS-C: Approximately 100 mg of TiO₂-NS was loaded into a ceramic boat that was loaded into a quartz tube inside of a furnace. Reforming gas (5% H₂ / 95% Ar) was set to flow at 1000 sccm and the temperature was ramped to 300 °C at a rate of 10 °C/min and held at 300 °C for 2 hours.

Synthesis of M-TiO₂-NS-C: 2 mL of absolute ethanol is added to 15 mg of TiO₂-NS-C in a 25 mL round bottom flask and sonicated for 10 minutes at room temperature to create a well mixed suspension. Appropriate amounts of K₂PtCl₄ and/or K₂PdCl₄ are dissolved into a minimum of water such that 5% (wt%) metal loading is achieved for the product M-TiO₂-NS-C (where M = Pt, Pd or PtPd). The product was washed several times in water or ethanol followed by centrifugation and overnight drying in a 60 °C oven.