

Effect of NH₃ gas treatment temperature on the morphology and oxygen reduction electrocatalytic performance of TiO_{1-x}N_x nanosheets

Jueun Yim

Graduation Thesis for Chemistry & Nano Science, Feb. 2019

Abstract

Due to the high electrochemical activities, high chemical stabilities and economic merits, the transition metal oxide (TMO) nanosheets have become one of the best candidates for electrocatalyst. However, poor electrical conductivity is a serious limitation for their application as electrocatalyst. Thus, the 2D holey nanosheets of titanium oxynitride (TiO_{1-x}N_x) were successfully synthesized by the heat-treatment of freeze-dried thin titanate nanosheets under NH₃ gas flow. The calcination at 700°C, 800°C, and 900°C induced the phase transition from layered lepidocrocite-type titanate precursor to anatase TiO₂ phase and then rocksalt-type TiO_{1-x}N_x phase. Interestingly, the formation of surface holes was observed due to the elimination of TMO domains initiated by the loss of lattice oxygen. The lateral size of several hundred nanometers to several micrometers of nanosheets were found on average, with thickness of several tens of nanometers. The surface holes were 5-60 nanometers and the elevation of heating temperature led to the enlargement of the holes. The electrocatalytic activity of oxygen reduction reaction (ORR) were measured in 0.1M KOH and the onset potentials of titanate nanosheets calcinated at 700°C, 800°C, and 900°C (at -0.1 mA cm⁻²) were ~0.74, 0.75 and 0.75V vs RHE. The decrease of overpotential upon heat-treatment can be ascribed to increased electrical conductivity and ion diffusivity through lattice holes. Besides, this work provides an efficient synthetic route to holey metal oxynitride nanosheets with improved functionalities.

Keywords: Transition metal oxides, Nanosheets, Titanate, titanium nitride, titanium oxynitride
