Type of presentation: Oral

MS-1-O-3257 Atomic Level In-situ Characterization of Metal/TiO_2 Photocatalysts under Light Irradiation in Water Vapor

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Photocatalysts have potential applications for solar fuel generation either through water splitting or CO_2 reduction. It is now recognized that atomic level in situ observations are critical for understanding the structure-reactivity in photocatalysts in the presence of reactant and product species and during in-situ light illumination. TiO_2 is a promising photocatalyst used for self-cleaning, pollutants degradation and water splitting etc. Metal particle co-catalysts such as Pt are coupled to the semiconductor to provide chemically active sites and attract excited electrons preventing charge recombination. Herein we use TiO_2 as a model material to develop in situ photocatalytic experimental methodology and explore structure changes of metal/semiconductor photocatalysts. We employ a modified ETEM with a broadband light source to study the behavior of metal particles on TiO_2 semiconductor surfaces under photoreaction conditions.

Well defined anatase nanoparticles were prepared following a hydrothermal method. Metal co-catalysts such as Pt, Ag and Cu were loaded onto the anatase nanobars using methods such as dry impregnation, photo-deposition and sputtering. An FEI Tecnai F20 ETEM was modified to allow samples to be illuminated with light with intensity up to 10 suns [1]. In situ analysis was performed tracking structure changes in photocatalytic vapor phase water splitting reactions. Ex-situ experiments were performed to compare or confirm in-situ observations under exposure to a 450W xenon lamp with a mirror reflecting 360nm to 460nm light. TEM images for ex-situ experiments were taken from an FEI aberration corrected Titan TEM.

The initial anatase particles shown in Figure 1a appear crystalline on the surface and the surface is smooth and atomically abrupt. Figure 1b shows a crystal after 40 hrs exposure to water and light without prior exposure to the electron beam. When the titania is exposed to light and water vapor, the initially crystalline surface converts to an amorphous phase one to two monolayers thick [2]. 5% wt Pt particles were loaded onto anatase nanoparticles and well dispersed through proper heat treatment. Figure 2a also shows initial Pt on TiO_2 materials. After exposure to a xenon lamp in liquid water for 6hrs, Pt particles show significant sintering as shown in Figure 2b. Pt/TiO_2 sample shows significant surface disordering (Figure 2b). In-situ experiments were performed to study the evolution of the Pt sintering, TiO_2 surface roughening and the Pt/TiO_2 interface changes. Other metal co-catalysts will also be discussed in the presentation.

References:

[1]. Miller, B.K.; Crozier, P.A. Microscopy and Microanalysis 2013, 19, 461-469
[2]. Zhang, L.; Miller, B. and Crozier P. A. Nano Lett. 2013 13 (2), 679–684

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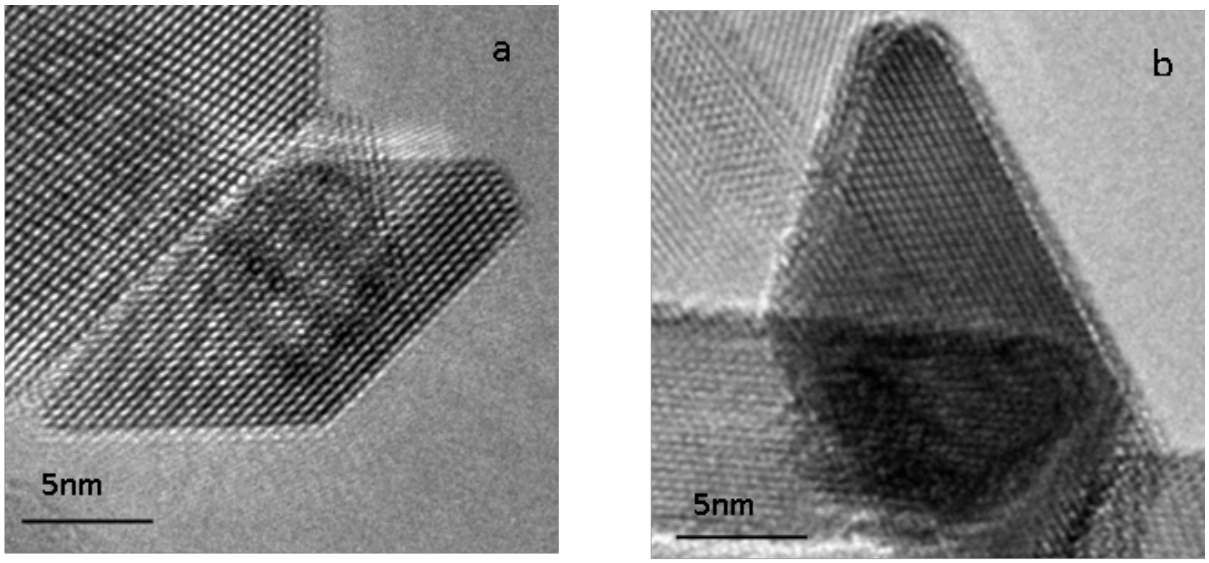

Fig. 1: a) Initial anatase crystal at 150°C without exposure to water or light. b) After 40 hrs in 1 Torr H_2O, 12 hrs light exposure.

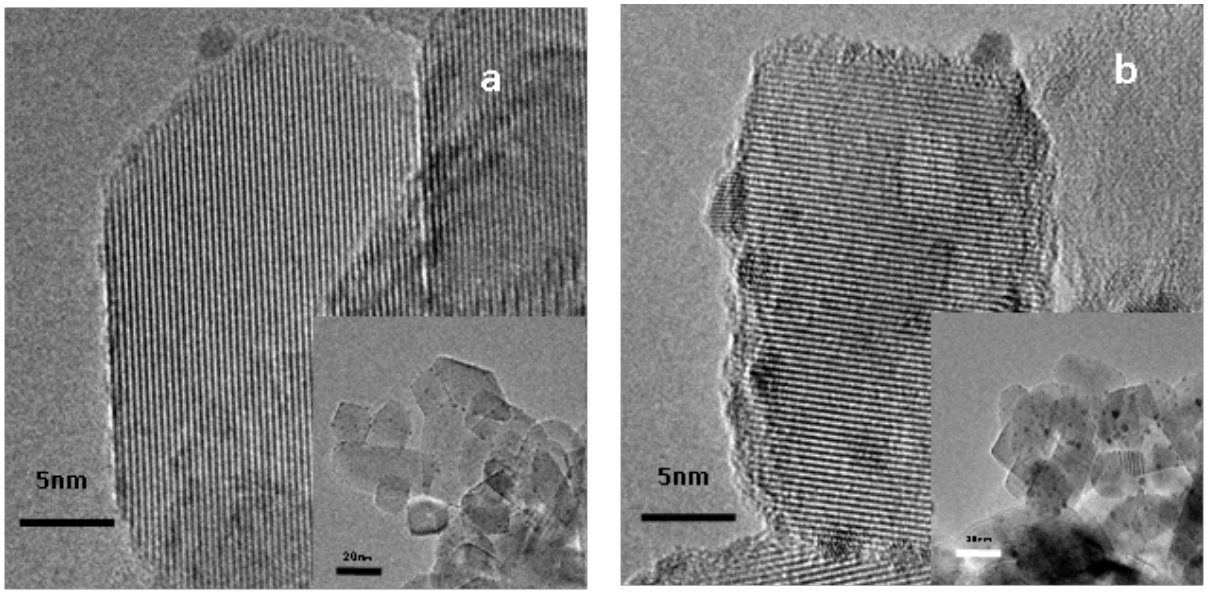

Fig. 2: a) Initial 5%wt Pt on anatase particles, b)after ex-situ 6hrs exposure to 360nm-460nm light in liquid water; (with insertions low magnification images).