

Electron Microscopy Characterization of Gold Nanoparticles Supported on an Ordered TiO₂ Nanowires Array.

Ernesto Neri-Cruz^{1*}, David Levi Quiroz-Aguilera², Ricardo Ortega-Diaz², Vicente Garibay-Febles³, Hector A. Calderon¹.

1. Instituto Politécnico Nacional, Mexico City, Zacatenco, Mexico.
 2. Escuela Nacional de Ciencias Biológicas, Instituto Politécnico Nacional, Mexico City, Santo Tomás, Mexico.
 3. Instituto Mexicano del Petróleo, Mexico City, Gustavo A Madero, Mexico.
- * Corresponding author: ernesto.neri@live.com.mx

The sustainable energy production via Artificial Photosynthesis has received much attention in recent years. The solar energy is absorbed by a semiconductor material and used to activate the involved catalytic reactions to form a fuel. This involves generation of Hydrogen from water and chemical reduction of carbon dioxide. Heterostructures are created by growing an ordered array of nanowires and depositing catalytic particles on them. Titanium dioxide semiconductor has been investigated as a photocatalytic material to integrate such devices capable to capture sunlight and transform water and carbon dioxide into a fuel. The herein investigated heterostructures TiO₂ nanowires and Au nanoparticles are prepared via chemical methods, they have a high surface ratio due to the nanowires morphology with plasmonic gold nanoparticles.

The heterostructure has been synthesized by the following experimental technique. As a first step, a porous anodic alumina template is fabricated [1]. Then, TiO₂ is electrochemically deposited inside the pores of the template to produce a nanowires array [2]. Nanoparticles are then attached on TiO₂ nanowires by following a process of precipitation with urea as described by Tsubota et al. [3]. Such an array of nanowires and nanoparticles are finally heated to 723K for a time of 2h to develop the final chemical compositions. In this work, electron microscopy is used to characterize these heterostructures.

Figure 1a shows a highly ordered aspect array of nanopores in the synthesized alumina template. This will become a mask to deposit the TiO₂ nanowires. The high order is confirmed by means of a Fourier Transform of the SEM image. The mask has an average pore diameter (D_p) of 46 ± 3nm determined by the histogram of pore size (Fig 1b). The Figure 1c shows the highly ordered and vertical array of nanowires that has been synthesized by means of electrodeposition. The total length of the nanowires array is about 7µm. The length is adjusted by the deposition time. Figure 2a shows a typical transmission electron microscopy (TEM) image of the Au/TiO₂ heterostructures. The average size of the gold nanoparticles has been determined as 4 nm as shown in Fig. 2b.

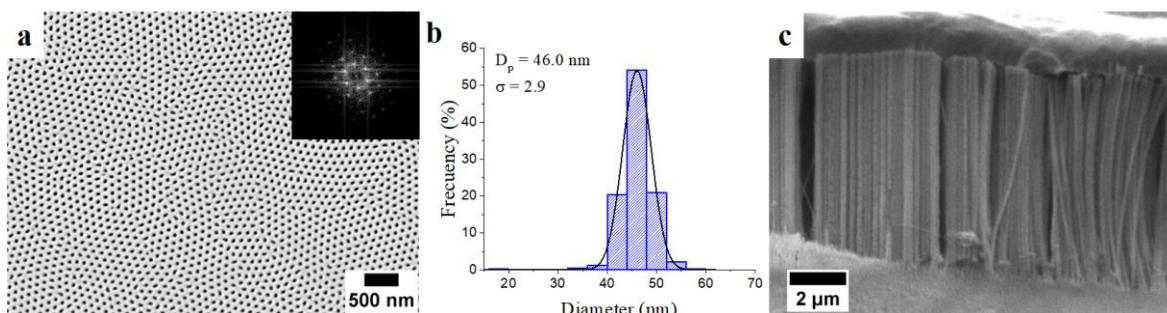


Figure 1. SEM images of (a) alumina mask, (b) diameter pore size analysis, (c) TiO₂ nanowires array.

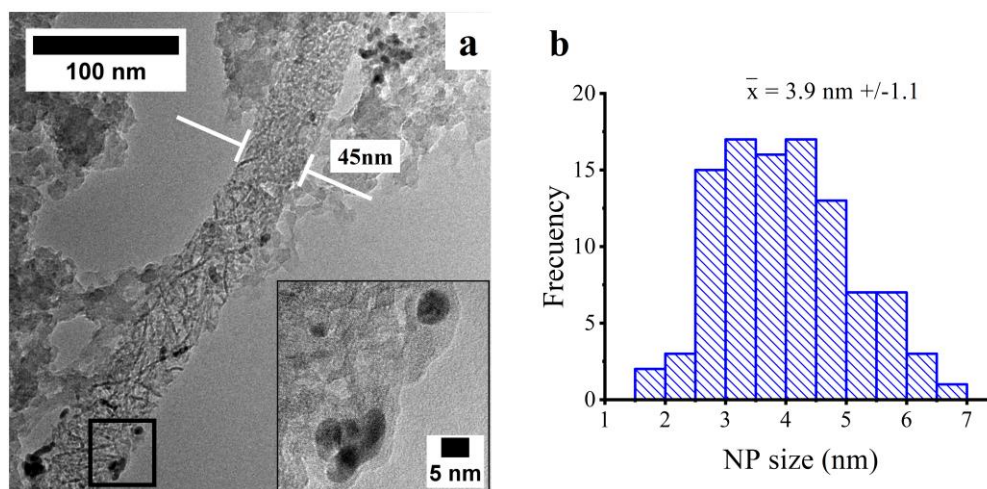


Figure 2. TEM images of (a) gold nanoparticles attached to TiO₂ nanowires, (b) nanoparticle size analysis.

- [1] H. Masuda and K. Fukuda, *Science* **268** (1995), p. 1466–1468. DOI: 10.1126/science.268.5216.1466
 [2] S. Tsubota et al., *Prep. Catal.* VI **91** (1995), p. 227–235. DOI: 10.1016/s0167-2991(06)81759-3
 [3] L. Kavan et al., *J. Am. Chem. Soc.* **118** (1996), p. 6716–6723. DOI: 10.1021/ja954172l
 [4] The authors acknowledge funding from the CONACYT and IPN (COFAA-SIP). Thanks for the technical support of the Instituto Mexicano del Petróleo and Micra Nanotecnología S.A. de C.V.