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Metallic Pt Clusters on TiO_2 Nanotubes: An Electron Microscopy Study

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High-angle annular-dark-field scanning transmission electron microscopy (HAADF-STEM), STEM-energy dispersive X-ray spectroscopy and energy filtered transmission electron microscopy are relatively new analytical techniques of electron microscopy that expands its application in studies of new materials, mainly materials of nanometric size as nanoparticles and one-dimensional nanostructures[1]. The brighter contrast in a HAADF image is strongly correlated to the atomic mass which is very useful in the catalytic materials where generally the support is compose of lighter elements while the active phase are heavier elements as noble metals. In other hand, high-resolution transmission electron microscopy (HR-TEM) image provide very reliable information about the crystallography of the support and the active phase in a chosen crystallographic orientation[2]. Together provide invaluable information about of the chemical composition and structural characteristics of the sample.

Currently, 1-D nanostructures as rods, wires, belts, ribbons and tubes have become in a focus of intensive research by their fascinating applications in mesoscopic physics, fabrication of nanodevices and as catalytic support. For catalysts, the metallic dispersion on 1-D nanostructures promise gives higher catalytic activity. Currently, the new catalyst generation is involving nanostructured support as one-dimensional nanostructures where can be utilized their surface properties in order to deposit more active phases and improve significantly the catalytic properties. In this work, we will present results of 1-D nanostructures of titanium oxide obtained from anatase nanoparticles. This material was used as catalytic support in order to disperse different load of platinum (1-10 wt %). The one-dimensional nanostructures were prepared by alkali hydrothermal method. Metallic nanoparticles were deposited on support catalytic by vapor-phase impregnation-decomposition method. Textural properties and chemical and structural characterization were investigated by Nitrogen Physisorption (BET method), X-ray diffraction and transmission electron microscopy.

XRD results evidenced a change in the structure of the anatase phase. The XRD pattern showed reflections in the position 2-theta = 9.03°, 24.89° and 48.53° which has been indexed as an orthorhombic structure of TiO_2 [3]. Transmission electron microscopy revealed that the anatase nanoparticles were transformed to nanotube morphology. The nanotubular features of the sample are shown in Fig. 1. The nanotubes have outer diameter ca. 7 and 10 nm, inner diameter around 4.5 to 5.5 nm and several hundreds of nm in length. The walls are being composed by two or three staked layers with an interlayer space of 0.74 nm. The specific surface area of the dried TiO_2 nanotubes was 350 m^2/g. These nanotubes were used as support in order to deposit different metallic load of Pt. XRD study over the supports impregnated not revealed the presence of Pt phase indicating a high dispersion of Pt on TiO_2 nanotube surface. This result was confirmed by a transmission electron microscopy study performing under the HAADF-STEM mode. Brighter dots very well dispersed on nanotube of gray contrast was evidenced, see HAADF image of Fig. 2. Statistical analysis

performed by hundreds of small nanoparticles showed that they have a size around of 1.5 nm in all samples. Big Pt particles segregated was not observed. HRTEM analysis revealed that these nanoparticles were grown as small crystallite with faceted morphology presenting hexagonal shape. The nanoparticle can be described as a truncated cuboctahedron with {1 1 1} faces and {2 0 0} surfaces because it has a hexagonal shape in its 2-dimensional projection that present four {111} surfaces and two {200} surfaces.

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[3]M.A. Cortes-Jacome et al, Catal. Today 126 (2007) 248.

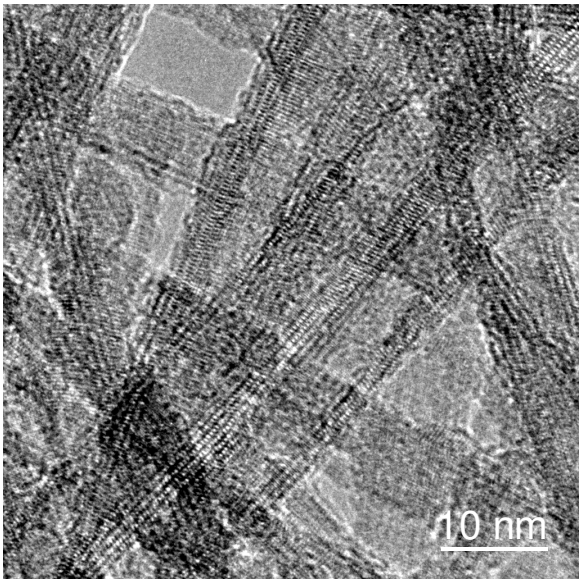

Fig. 1. TEM image showing the nanotubular morphology of the TiO_2 after the synthesis.

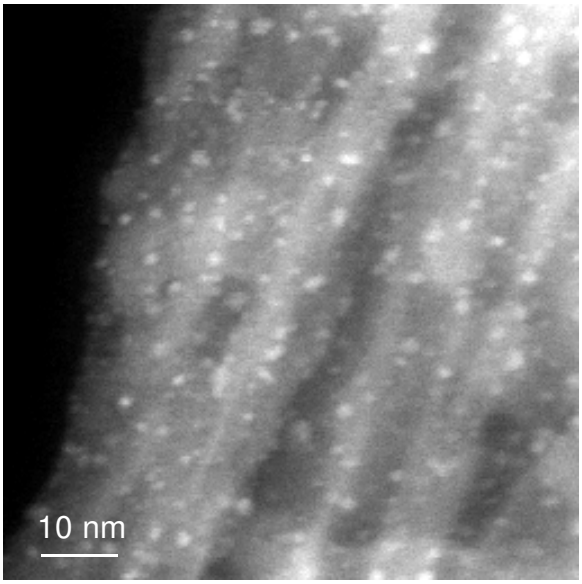

Fig. 2. HAADF image showing brighter dots on titania nanotube. The brighter dots were produced by Pt atom clusters. Nanoparticle size around of 1.5 nm can be observed.

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