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Sol-gel Nanostructured Titania: A Phase Transformation Study from beta-TiO_2-Anatase-Rutile

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Abstract – Nanostructured titania powders have been prepared by sol-gel method sintered at different thermal treatments (250 to 700 ºC), during different periods of times. Nanostructured ceramic powders contained beta-TiO_2, anatase, rutile and/or the co-existence of all of them, as shown in Fig.1. A phase transition to the anatase phase and to the rutile phase of TiO_2 occurred gradually from 250 to 500 ºC and from 500 to 700 ºC respectively. The average crystallite size increased from 10 nm to 100 nm.

Nanostructured titanium dioxide (TiO_2, or titania) and titanium dioxide-based ceramic have been widely investigated for its promising applications in different fields: as electrical and optoelectrical material, as gas sensor, as catalyst and photocatalyst, etc. One of the aspects for effectively using nanostructured titania for these applications is to understand the science behind tailor-making nanostructures with respect to crystal structure phase, crystal size and porosity [1]. Particularly, photocatalysis is a very promising method to face most of the problems connected with the increasing environmental pollution. The photocatalytic activity of TiO_2 has been found to depend on its structural form and is commonly higher in the anatase form (E_g=3.2 eV) compared to that of rutile (E_g=3.0 eV), which has been associated to its Fermi level. Although the anatase to rutile phase transformation has been extensively studied [1-4], there are few reports concerning monoclinic beta-TiO_2 phase formation, which is a low temperature phase, and its influence on the photocatalytic activity.

In this work, nanostructured titania powders have been prepared by sol-gel method sintered at different thermal treatments (250 to 700 ºC), during different periods of times. Titanium isopropoxide, isopropanol, metanol and acetic acid were used as precursors in order to prepare high purity ceramic powders. The influence of thermal heating treatments on the kinetics for nanocrystalline monoclinic beta-TiO_2-to-rutile phase transformation has been investigated. The structure, morphology and composition have been investigated by Fourier transform infrared (FTIR), X-ray diffraction (XRD), Scanning and Transmission electron microscopy (SEM and TEM, respectively). Rietveld refinement has been made with the aim to study the composition of different TiO_2 phases and their crystal sizes. The Brunauer, Emmett and Teller (BET) adsorption-desorption of nitrogen gas for specific surface area determination at the temperature of liquid nitrogen was performed on TiO_2 samples.

The results suggested the preparation of nanostructured ceramic powders contained beta-TiO_2, anatase, rutile and/or the co-existence of all of them, as shown in Fig.1. For low calcination temperatures, the powders mainly consisted of beta-TiO_2 phase, while rutile phase was only present at temperatures higher than 700 ºC (Fig.2). In this case, the crystallite size increases from 5 nm to 100 nm between 350 ºC and 700 ºC. A phase transition to the anatase phase and to the rutile phase of TiO_2 occurred gradually from 250 to 500 ºC and from 500 to 700 ºC respectively. The average crystallite size increased from 10 nm to 100 nm.

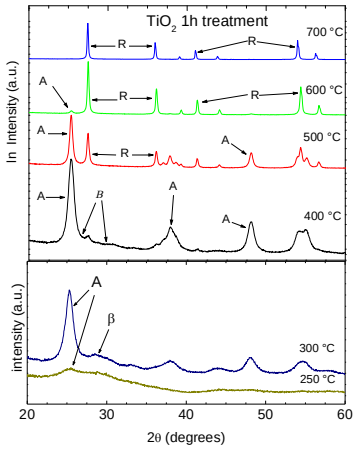

Figure 1: a) XRD patterns of nanostructured TiO_2 powders sintered at different thermal treatments.

Figure 2: TEM image of TiO_2 powders treated at 700 °C during 240 min, showing crystals of rutile.

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