11th International Conference on Advanced Materials
Rio de Janeiro Brazil
September 20 - 25
ICAM2009

Phase Transition Induced on Ag/TiO_2 Thin Films by Transmitted Electron Beam

M. M. Viana[(1)], N. D. S. Mohallem[(1,2)], D. R. Miquita[(2)] and K. Balzuweit[(2,3)]

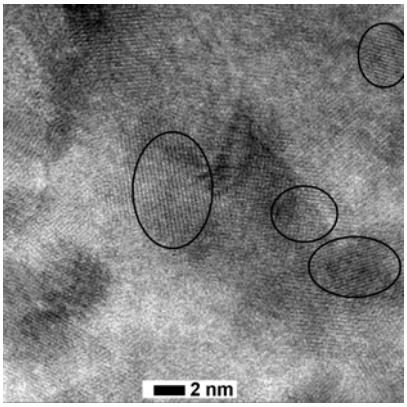
(1) Laboratório de Materiais Nanoestruturados, DQ, UFMG, Belo Horizonte, Brazil
(2) Centro de Microscopia, UFMG, Belo Horizonte, Brazil
(3) Departamento de Física, UFMG, Belo Horizonte, Brazil
nelcy@ufmg.br

Abstract – Ag/TiO_2 thin films have been deposited by sol-gel process in glass substrates. The films were characterized by X-ray diffraction (XRD), electron diffraction and high-resolution transmission electron microscopy (HRTEM). Only anatase phase was identified by XRD but electron diffraction shows the presence of anatase and rutile phases. Changes in diffraction pattern were visually observed during the HRTEM measurement, showing the sensitivity of these samples to the electron beam.

Ag/TiO_2 thin film is an important technological material researched as a photocatalyst [1] and an antimicrobial [2] agent. These properties are related to anatase phase-TiO_2 which exhibits a relative high energy band gap (~3.2 eV) reached only using UV irradiation. Noble metal particles, such as Ag, have been added in the matrix to improve the TiO_2 properties. Nanocomposite thin films have been deposited on glass, steel and other surfaces by a wide range of techniques, especially by sol–gel and chemical vapor deposition [2]. HRTEM is a very powerful tool for microstructural observation of thin films and it can provide dynamic information of phase transitions on the solid surface, which cannot be obtained directly by other techniques.

The thin films were prepared by sol-gel process from alcoholic solution containing tetraisopropyl orthotitanate and silver nitrate dissolved in a mixture of isopropyl alcohol. The atomic ratio Ag:Ti was equal to 1:6. The starting solution was submitted to UVC irradiation (254 nm) treatment in air for 8 hours to produce Ag by reduction of Ag^+. Ag/TiO_2 thin films were dip-coated onto clean glass substrates with withdrawal speed of 1.5 $cm.sec^{-1}$, dried in air for 30 min and heated for 1 hour at 100 and 400 °C. The evolution of the crystalline structure was observed by low angle X-ray diffractometry (incidence angle of 5^o). HRTEM images and electron diffraction patterns were obtained using a 200 KV transmission electron microscope.

Diffraction peaks for the anatase phase and face centered cubic (fcc) silver have been identified in the film calcined at 400 °C. X-ray diffraction analysis of Ag/TiO_2 thin films calcined at 100 °C shows a high amorphous character. However, the HRTEM image (Figure 1) shows very small oriented grains in the samples (Ag/TiO_2) calcined at 100 °C. The corresponding electron diffraction pattern is illustrated in Figure 2, showing a set of diffraction spots that correspond to interplanar distances of 3.51 Å and 3.25 Å related to (1 0 1) anatase plane and to (1 1 0) crystalline rutile plane, respectively. This result suggests that a dynamic phase transition occurred in the thin films during electron exposure.



(a)

Figure 1: HRTEM image of Ag/TiO_2 thin film calcined at 100 °C.

(b)

Figure 2: Diffraction pattern of Ag/TiO_2 thin film calcined at 100 °C.

References

[1] C-C. Chang, J-Y. Chen, T-L. Hsu, C-K. Lin, and C-C. Chan, Thin Solid Films 516 (2008) 1743–1747.
[2] K. Page, R. G. Palgrave, I. P. Parkin, M. Wilson, S. L. P. Savin, and A. V. Chadwick,J. Mat. Chem. 17 (2007) 95–104.