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Synthesis and Characterization of TiO_2 Films Obtained by Sol-Gel Method

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Abstract — Borosilicate coated titanium dioxide thin films were produced by the sol-gel method. The films were calcinated at 450°C for 20 minutes for crystalization. X-ray diffration analyses show that before the heat treatment the film was amorphous and after that it crystallized in anatase phase presenting mean grain size of 34 nm and RMS roughness of 8 nm. The crystallized film was used as photocatalist in the degradation of methyl orange dye presenting an efficiency of 25% after 2h under UV light.

Keywords* — *sol-gel, TiO_2, photocatalysis.

I. INTRODUCTION

Titanium dioxide is a semiconductor that exhibits chemical stability, absence of toxicity, corrosion resistance, water insolubility, photostability and band gap energy compatible with sunlight, in addition to the relatively low cost[1]. The physical and chemical properties of TiO_2 make it an excellent material for several applications, such as photocatalysis, photovoltaic cells, gas sensors and electrochemical applications[2]. TiO_2 can exist in three main crystalline structures: anatase and rutile, tetragonal, and brookite, orthorhombic. The photocatalytic process consists in irradiating the semiconductor with UV light. This energy is absorbed by the catalyst and excites the electrons of the valence band to the conduction band. In this electronic transition a pair (e -)/hole (h +) is generated, originating oxidizing and reducing sites on the surface of the semicondutor. The major areas of application of photocatalysis are air deodorization, self-cleaning and anti-fogging surfaces, auto-sterilizing materials[3], and water treatment, where it can be used for the degradation of organic pollutants in water, due to its capacity to generate hydroxyl radicals in aqueous medium[4-8]. The properties of nanostructured TiO_2 films are function of the crystalline structure, grain size, morphology and are highly dependent on the synthesis method used. The scope of this research was to synthesize TiO_2 films by using the sol-gel process, and to characterize its morphological, structural and photocalalytic properties.

II. MATERIAL AND METHODS

The sols were prepared from a solution of titanium isopropoxide IV ($Ti[OCH(CH_3)_2]_4$) 97% (PA) in isopropanol (PA) at pH 2.5. The solution was kept for 1 hour under vigorous stirring at 50°C. Borosilicate substrates of size 26x76x1mm, used as substrate, were immersed in the sol, in a dip coating process. The films were dried in a oven for 30 minutes at 100°C. The samples were then calcinated in a muffle furnace for 20 min at 450°C. The characterization of the films was carried out by using Scanning Electron Microscopy (SEM) in a Jeol microscope model JSM6701F to measure the thickness of the film. Atomic force microscopy (AFM) was used for the topographic evaluation of the films and for measure the grain size and surface roughness in a Bruker SPM equipment, model Nanoscope IIIA. The X-ray diffraction (XRD) technique was used to evaluate the crystallinity and phase identification of TiO_2 films by using a Multiflex Rigaku equipment with a monochromatic CuKα source (λ = 1.54148 nm) in the range of 10º to 80º, step of 0.02º, and incidente angle of 2.5 degree.

The photocatalytic properties of the TiO_2 film was analyzed by spectrophotometry under UV light, using the methyl orange dye as the model pollutant. The film was placed in a reactor containing 50 mL of the dye solution and illuminated by two tubular UV lamps for 2 hours. To evaluate the degradation of the methyl orange solution the spectrophotometer Global Trade Technology was used.

III. RESULTS AND DISCUSSION

The diffraction spectra of the TiO_2 films are shown in Fig.1, and suggests that before calcination the film was amorphous, and after the heat treatment at 450° for 30 min TiO_2 in anatase phase (JCPDS 21-1272) was formed.

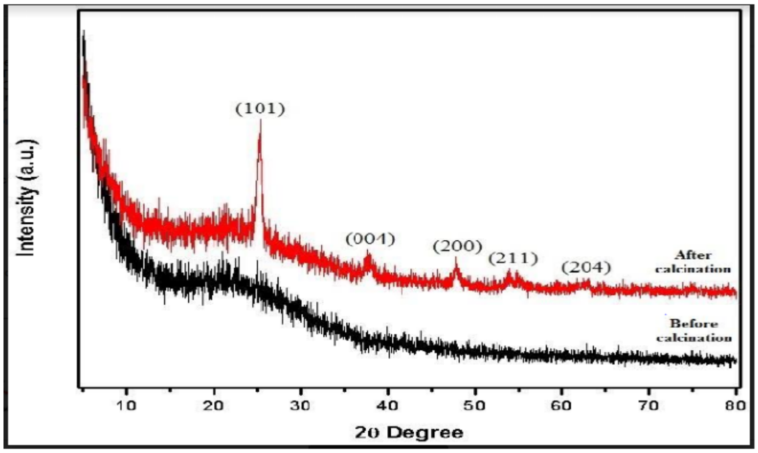

Fig. 1. XRD spectra of TiO2 thin films before and after calcination for 30 min at 450°C.

Fig. 2 shows the AFM topography of the film after calcination. The surface is homogeneously covered by TiO_2 film. Analyzes performed on this surface indicated that the mean grain size is 34 nm and the RMS roughness is 4 nm. Fig. 3 shows a SEM image of the cross section of the same sample. The thickness of the film is 171 nm.

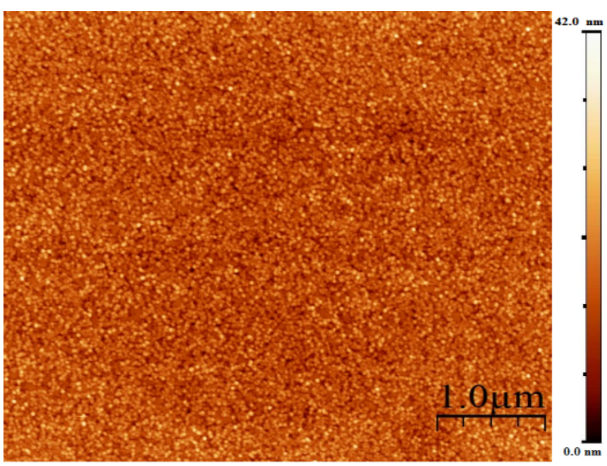

Fig. 2. AFM topography image of TiO_2 film after calcination at 450° for 30 min.

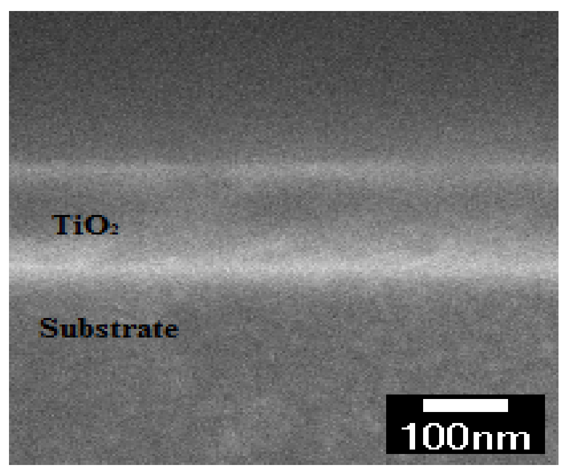

Fig. 3. Cros sectional SEM image of sample after calcination at 450° for 30 min.

Fig. 4 shows the degradation curves of the methyl orange solution as a function of the exposure time to UV light, both in the absence (photolysis) and in the presence of the photocatalyst (TiO_2 film). It was observed that the solution illuminated by UV light without the TiO_2 showed no degradation, whereas in the presence of TiO_2 the degradation reached 25%. Other studies are underway to improve the photocatalytic properties of TiO_2 films.

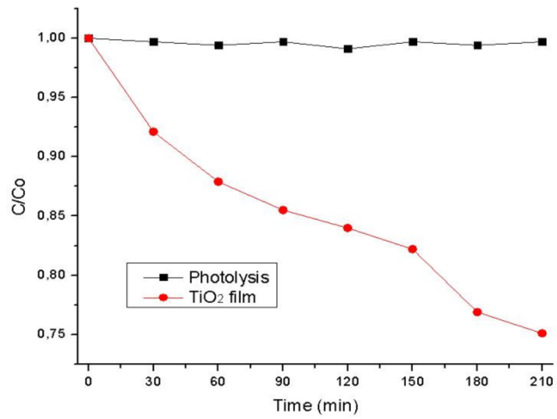

Fig. 4. Photocatalitic degradation activity of methyl orange dye on TiO_2 film under UV light.

IV. CONCLUSIONS

The results allow to conclude that the TiO_2 films obtained by sol-gel are amorphous, and that after calcination for 20 minutes at 450°C the crystallization occurs in the anatase structure. Under these conditions the film presented a mean grain size of 34 nm and a roughness of 4 nm. The photocatalytic test conducted for 2h under UV light shows that the film was capable of degradate 25% of the methyl orange dye and that more developments are needed in order to improve the photocatalytic activity of the TiO_2 film.

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