ID 162: SYNTHESIS AND CHARACTERIZATION OF N-DOPED TiO_2 THIN FILMS

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1. Introduction

Most of the metallic materials used in modern technology require certain properties of the alloy, and a different set of properties of the surface. The requirements for the alloy are toughness, tensile strength, among others, while those for the surface are resistance to oxidation, wear, erosion, etc. [1,2]. It is rare that this combination can be presented by a single material, hence the need for the use of coatings, which have the advantage of not altering the mechanical and microstructural properties of the alloys. A variety of techniques are available for deposition of thin films, such as ion implantation [3], sputtering [4], chemical vapor deposition (CVD) [5], sol gel [6], and metallorganic chemical vapor deposition (MOCVD) [7,8].

Titanium dioxide is a biologically and chemically inert material [9], nontoxic, exhibits photostability, good mechanical and corrosion resistance [7], and significant photocatalytic activity. It is used as dielectric material, anti-reflective coatings, anti-corrosive barriers [6,7], solar cells, biomaterials [10], and in optical devices [10]. The interest in Ti-N-O films has increased in recent years because the presence and control of oxygen in titanium nitride leads to the formation of gradient coatings ranging from TiO_2 to TiN, and where resistivity varies as a function of the N/O ratio [11]. Similarly, the increase of the nitrogen content incorporated into TiO_2 alters functional properties such as refractive index, electrical conductivity, hardness, and modulus of elasticity of the original oxide film because the metal-nitrogen bonds are less polar than the substituted metal-oxygen bonds [12].

This work reports the growth of TiO_2 and N-doped TiO_2 thin films at 500° C by MOCVD on stainless steel substrate. X-ray diffraction diagrams and XPS analysis of N-doped TiO_2 are presented and discussed in relation to the undoped TiO_2.

2. Experimental

Samples of AISI 316 stainless steel with 20x20x5 mm were used as substrate for the deposition of TiO_2 films and N-doped TiO_2 via MOCVD process. The samples were grounded with SiC abrasive discs up to 600 grit and polished with diamond paste until 3 μm. Substrates were ultrasonically cleaned in acetone, in ethanol, and rinsed in deionized water in abundance. They were then dried in nitrogen and immediately inserted into the reactor.

Titanium isopropoxide IV was used both as titanium and oxygen sources to obtain TiO_2. For the production of N-doped TiO_2 NH_3 was added in the reactor. Both reagents were introduced into the system simultaneously, and were mixed before reaching the reaction chamber. The growth parameters used were: nitrogen and ammonia flow 0.5 slm, titanium precursor temperature 39°C, chamber pressure 50 mbar, deposition temperature 500°C. The thickness of the films is close to 300 nm.

The samples were characterized by X-ray diffractometry (XRD) in a Rigaku equipament with incident angle of 3°, and by X-ray photoelectrons spectroscopy (XPS) in a thermoscientific equipment, model k-alpha.

3. Results and Discussions

XRD analysis, Figure 1, indicates that in the non-doped TiO_2 film anatase was formed, whereas N-doped TiO_2 sample presents the rutile phase. This fact suggests that nitrogen induces the anatase-rutile phase transformation. No peak relative to nitrogen-containing phases was observed. Similar results were obtained by F.Peng [13] and M. Sathish [14]. The XPS spectrum is shown in Figure 2. On the survey, it can be seen that the doped film is composed of Ti, O, and C as contaminant [13,15], in addition to nitrogen, which was incorporated in the ratio of 8.29 at%. N 1s linkage energy was detected at 396.9 eV resulting from the nitrogen substitution of the TiO_2 lattice. According to other researchers that agree on that N 1s peak at 396-397.5 eV is responsible for nitrogen atoms substitutionally doped into the TiO_2 lattice as characteristic peak of Ti-N-Ti linkages [14-17]. Further analyses are under way.

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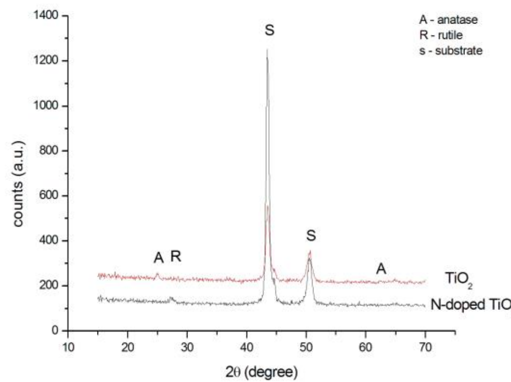

Fig. 1.. XRD diagram of samples.

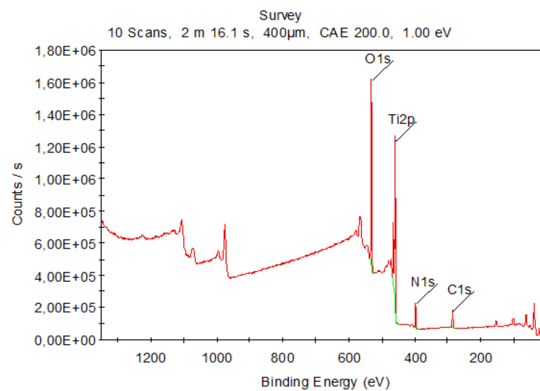

Fig.2. XPS spectrum of N-doped TiO_2 film.

TiO_2 and N-doped TiO_2 films depositions were performed in a single step by using the MOCVD technique. Titanium isopropoxide IV was used as a precursor of both titanium and oxygen, and NH_3 as a source of nitrogen. The growth was carried out at 500°C and the films presented about 300 nm of thickness. The nitrogen content incorporated into the film was 8.3 at%. Results of XRD indicate the formation of the anatase phase for the non-doped film, and rutile for the nitrogen-doped film, suggesting that the nitrogen induces the transformation of the anatase to rutile phase.

4. References

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