

ITO AND FTO COATED GLASS CHARACTERIZATION USING SEM AND AFM TECHNIQUES

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Abstract: *Transparent conducting oxides like indium tin oxide (ITO) and fluorine doped tin oxide (FTO) represent an active area of research in the field of nanomaterials. This study aims to investigate the surface profile of the ITO and FTO coated glass used for dye-sensitized solar cells (DSSCs) manufacturing process. The surface morphology of the samples was probed by scanning electron microscopy (SEM). The atomic force microscopy (AFM) technique was used in order to obtain the topography and roughness for these samples.*

Key words: *ITO, FTO, SEM, AFM, roughness.*

1. Introduction

Transparent conducting oxides (TCOs) are used as transparent electrodes in a wide variety of applications (solar cells, liquid crystal displays, light-emitting diodes) due to their properties such as optical transparency and electrical conductivity [1].

Tin doped indium oxide (ITO) and fluorine doped tin oxide (FTO) are most frequently used materials for dye-sensitized solar cells (DSSC) manufacturing process because of its unique properties such as high optical transmittance over the visible wavelength region, low resistivity and chemical stability [3].

Scanning electron microscopy (SEM) is a non-destructive materials characterization technique that uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid samples. The signals that derive from electron-sample interactions reveal information about the sample including surface morphology, chemical composition and crystalline structure [4].

Atomic Force Microscopy (AFM) technique is one of the most commonly used scanning probe microscopy. Compared to optical and electron microscopy, the atomic force microscopy offers the advantage of direct measurement of X, Y, Z dimensions. By

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using this method, the material surface can be seen in three-dimensional (3D) at nanometer scale.

The aim of the present article is to investigate surface morphology and topography of indium tin oxide and fluorine doped tin oxide as transparent conducting film used for dye-sensitized solar cells (DSSCs) manufacturing process. Topographical studies were obtained by using atomic force microscopy (AFM) technique and the surface morphology was probed by scanning electron microscopy (SEM).

2. Materials and Methods

2.1. Materials

ITO and FTO coated glass samples (Figure 1) were purchased from the Sigma Aldrich Chemical Company (Milwaukee, Wisconsin).

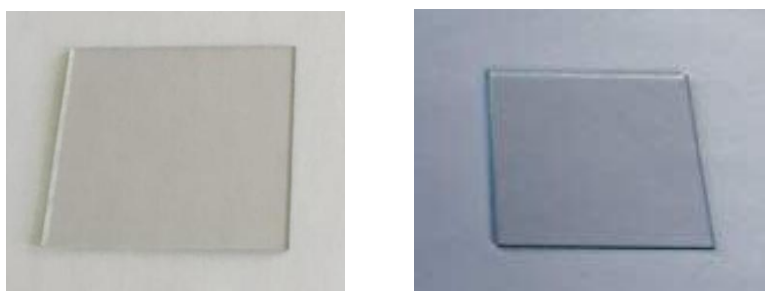


Fig. 1. FTO (left) and ITO (right) coated glass samples

ITO and FTO glasses are inorganic thin films commonly used to provide high conductivity as well as optical transparency. ITO conductive glass consist of 90% indium oxide (In_2O_3) and is doped with 10% tin (Sn) while FTO conductive glass consist of 95% tin oxide (SnO_2) and is doped with a small amount of fluorine ($\approx 5\%$) [5].

2.2. Methods

The SU-70 Scanning Electron Microscope (SEM) was used to obtain the surface morphology of the samples. The accelerating voltage (V_{acc}) of SU-70 may vary from 0.1 kV to 30 kV and the resolution is 1 nm (at $V_{\text{acc}} = 15$ kV) [2].

The NTEGRA Prima Atomic Force Microscope (AFM) was used to obtain the surface topography of the ITO and FTO. The average profile and the 3D height roughness surface parameters were obtained using the Nova PX software.

3. Results and Discussions

By using scanning electron microscopy (SEM), for lower amplification order, some spots may appear on the surface of the analyzed samples, which may be due to several

factors. Although these spots can provide information of different chemical composition or mechanical defects, there are cases in which they occur due to the surface shape.

Increasing the amplification order to 50000x can be seen the shape of the material crystals and their dimensions (Figure 2).

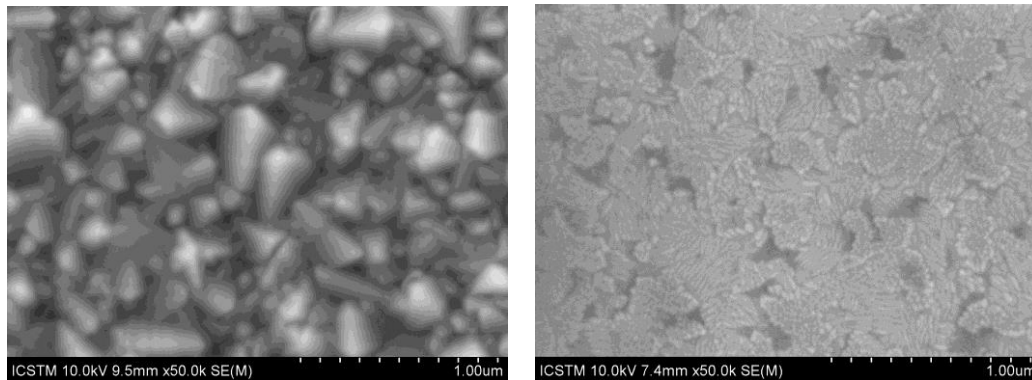


Fig. 2. Surface morphology of the FTO (left) and ITO (right) coated glass

In the case of FTO, minor formations sizes are smaller than 10 nm, while larger formations sizes reach about 300 nm. For ITO, the formations sizes are approx. 7-75 nm.

The atomic force microscopy (AFM) technique in contact mode was used to minimize the effects of friction and other lateral forces and to measure the roughness of the samples surfaces.

The height roughness parameters (S_q , S_a , S_p , S_v , S_z) (Table 1) can be evaluated from the AFM measurements, their values providing information for the investigated surface.

3D height roughness parameters description Table 1

Category	Parameter	Description	Notes
Height parameter s	S_q	Root mean square height	This parameter corresponds to the standard deviation of distance from the mean plane
	S_a	Arithmetical mean height	This is the arithmetic mean of the absolute value of the height from the mean plane of the surface
	S_p	Maximum peak height	This parameter represents the maximum value of height from the mean plane of the surface
	S_v	Maximum pit height	This is the absolute minimum value of height from the mean plane of the surface
	S_z	Maximum height	This parameter represents the distance between the highest point and the lowest point on the surface

In this context, using AFM technique the obtained surface topography of the FTO and ITO coated glasses is showed in Figures 3 and 4.

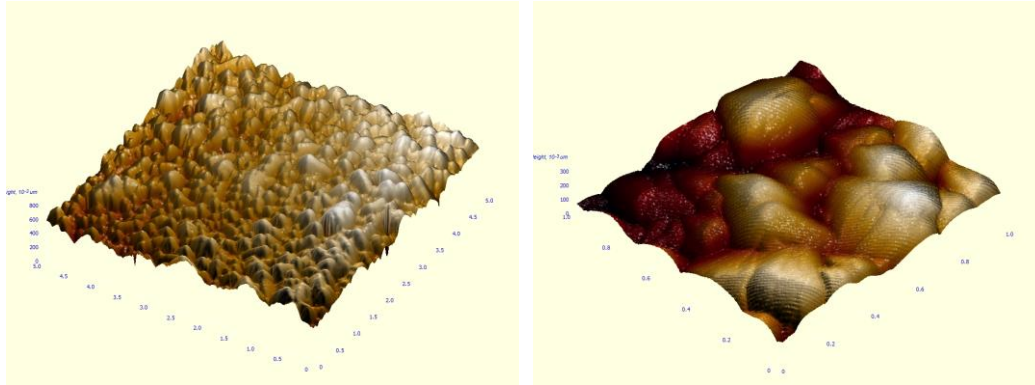


Fig. 3. Surface topography of the FTO coated glass $5 \times 5 \mu\text{m}$ (left) and $1 \times 1 \mu\text{m}$ (right)

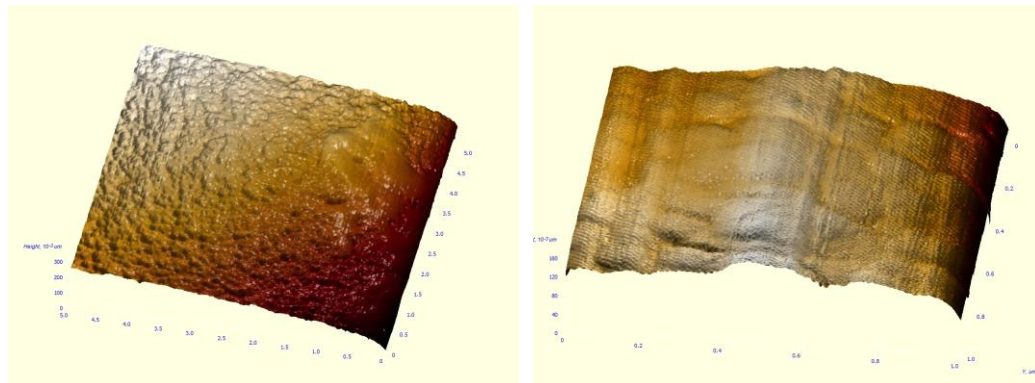


Fig. 4. Surface topography of the ITO coated glass $5 \times 5 \mu\text{m}$ (left) and $1 \times 1 \mu\text{m}$ (right)

Analyzing the samples surface topography, the homogeneity and uniformity of the deposited layer can be observed, even if at the resolution of $1 \times 1 \mu\text{m}$, for SnO_2 : F large deposits are visible on the glass surface (Figure 3). These observations are not confirmed in the case of ITO (In_2O_3 : Sn) as the spatial dimensions of Sn and In not so different.

If the atomic radius of its component elements is taken into account, the doping contribution to the ITO is much larger in dimensional terms (Sn having an atomic radius of 145 pm, much higher than F, the FTO dopant having the atomic radius 50 pm).

By using the Nova PX software was obtain an average profile for the selected area ($5 \times 5 \mu\text{m}$) of the FTO and ITO coated glasses samples. In Figures 5 and 6 on the left is the source image. On the right is the result of applying the Average profile for the aforementioned area.

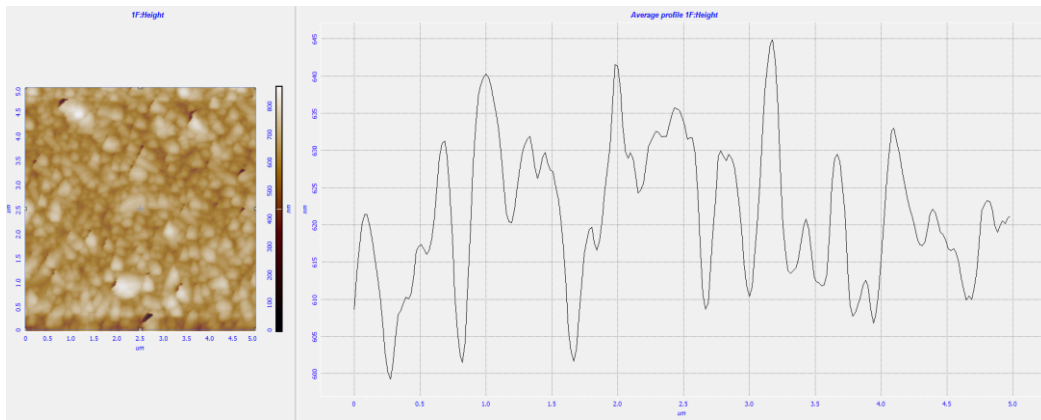


Fig. 5. Average height profile of the FTO coated glass

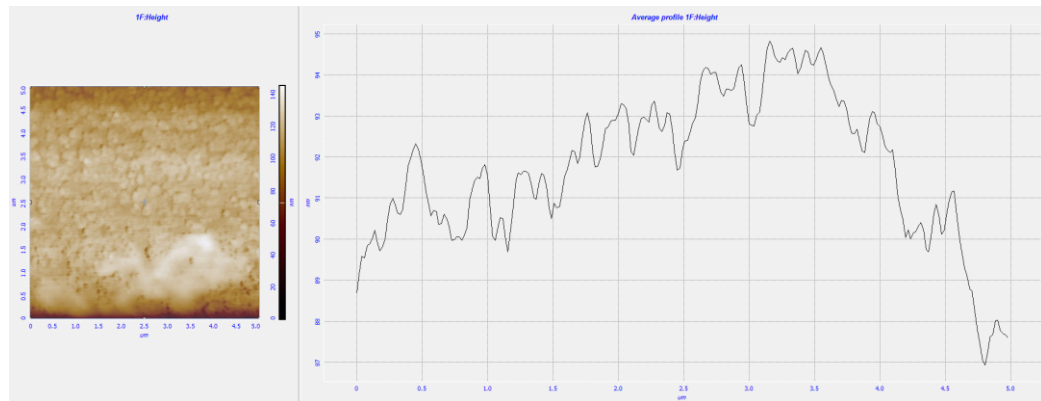


Fig. 6. Average height profile of the ITO coated glass

Analyzing the average height profile can be observing the same conclusion as in the topographical study above mentioned, being a confirmation of these results. FTO shows a uniform dimensional profile compared to ITO which is not uniform from the aforementioned considerations.

The values of the 3D height parameters, defined by the ASME B46.1 standard, were calculated using, also, Nova PX software and are presented in the Table 2.

3D height roughness parameters values Table 2

	S_q [nm]	S_a [nm]	S_p [nm]	S_v [nm]	S_z [nm]
FTO coated glass sample	65	50	245	621	866
ITO coated glass sample	15	10	35	109	144

Even if it has a superior chemical stability, the FTO suffer from a high surface roughness compared to ITO. But, FTO is preferred instead of ITO as an electrode for

DSSCs because when an annealing process is required for the thin layer to be deposited on the conductive side (for example, in DSSCs a layer of TiO_2 is deposited onto a conductive side of the TCO and should be thermally treated) the ITO electrical properties can degrade in the presence of oxygen at a relatively high temperature (i.e., about 500 °C). FTO is much more stable under such conditions.

4. Conclusions

ITO and FTO coated glasses are inorganic thin films commonly used to provide an optical transparency as well as a high conductivity.

The surface morphology of the samples was probed by scanning electron microscopy (SEM) and by using AFM technique were obtained surface topography of the FTO and ITO coated glasses.

Average height profiles shows that FTO has a uniform dimensional profile compared to ITO which is not uniform.

Surface roughness is quantified by deviations in the direction of the normal vector of a real surface from its ideal shape. If these deviations are high, the surface is harsh; if deviations are small, the surface is smooth.

This morphological and topographical surface study of the FTO and ITO coated glass will help in our future experiments.

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References

1. Klein, A., Körber, C., et al.: *Transparent Conducting Oxides for Photovoltaics: Manipulation of Fermi Level, Work Function, and Energy Band Alignment*. In: *Materials* 3 (2010), p. 4892-4914.
2. Olteanu, L., Ion, R.M., et al.: *New metallo-porphyrins for solar energy conversion*. In: *Bulletin of the Transilvania University of Braşov* (2017), Vol. 10 (59), Series I, p. 47-54.
3. Ramanathan, G., Murali, K.R.: *Dye Sensitized Solar Cells Behaviors of TCO Materials*. In: *International Journal of Advanced Scientific Technologies in Engineering and Management Sciences* (2016), Vol. 2 (10), p. 11-14.
4. Rocha-Santos, T., Duarte, A.C.: *Characterization and Analysis of Microplastics*. Amsterdam. Elsevier, 2017.
5. Yaacob, M.H., Ahmad, M.Z. et al.: *Optical response of WO_3 nanostructured thin films sputtered on different transparent substrates towards hydrogen of low concentration*. In: *Sensors and Actuators B* 177 (2013), p. 981-988.