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Thermal oxidation of tin layers and study of the effect of their annealings on their structural and electrical properties

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The main objective of this article is the control of tin dioxide preparation process on glass substrate. Layers of pure tin with thicknesses of 500 and 1000 Å are first deposited. Their enrichment with oxygen is ensured by thermal annealing for 1 and 2 h in a continuous tube furnace with temperatures varying between 300 and 500 °C.

The tin film formed by vacuum evaporation has tetragonal crystalline structure, and is composed of grains of various sizes separated by grain boundaries. After annealing in oxygen, the formed phases consist of a mixture of SnO and SnO₂ crystalline mixtures and sometimes amorphous tin oxide. The more the time or the temperature of annealing, the more the quantity of SnO₂ and SnO. For an annealing at 500 °C for 10 h the size of grains increases more than annealing for 2 h. This is confirmed by the study of their micrographs.

The electrical resistivity of these layers, measured by the 4 point method, is correlated to the size of the oxide particles: the smaller the particle size, the lower the electrical resistivity.

1. Introduction

Tin dioxide is an n-type, wide band gap semiconductor and owing to its optical, electrical and structural properties it is adapted for many applications in opto-electronic devices [1,2], solar collectors [3] and gas sensors [4–6].

Whatever the methods of preparation of tin dioxide layers, the properties of films are influenced by the surrounding medium (gas, liquidates, etc.), the reagents used (nature, concentration, purity, etc.), the substrate (nature, temperature, etc.) and the annealing of the layers. The complexity of these processes and the various modes of growth of nanocrystals from these layers have led to

several techniques of preparation: sol-gel [7,8], thermal oxidation [9,10], pyrolysis by spray [11,12] and chemical vapor deposition (CVD) [13]. The characteristics of the obtained layers depend on the used technique.

In this work the thermal oxidation technique of thin tin layer is used: tin layers of different thicknesses, first of all, were obtained by vacuum evaporation on ordinary glass and then oxidized under constant oxygen flow at various temperatures and for various times of annealing, in order to obtain tin dioxide. The final objective is the control of SnO₂ deposited layers for a possible doping, which would allow the realization of transparent conducting glasses used as electrodes in the solar cells.

2. Experimental

Vacuum evaporation permits the preparation of films of well defined thickness from 50 to more than 2000 Å

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range with a high degree of accuracy and a perfect purity. Before evaporation, it is necessary to clean the substrates and the materials to be evaporated in acids and solvents. Taking such precautions is necessary due to the fact that the physico-chemical properties of the layers obtained are strongly dependent on the conditions of preparation of the substrate and also those of deposition of these same thin layers. The used substrates are made of pieces of ordinary glass whose dimensions are $2\text{ cm} \times 1\text{ cm} \times 2\text{ mm}$. Evaporation was performed in an ALCATEL model ATP150 evaporator.

The intervening parameters in this technique are on one hand the thickness of the layer deposited and on the other hand the time and the annealing temperature under oxygen flow. Dieguez et al. [14], who carried out thermal oxidation of layers of tin, showed that a minimum time of oxidation is required in order to allow the total conversion of tin to tin dioxide (SnO_2). This time is approximately 9 h at ambient temperatures of about 500°C . Metal tin passes through the formation under air to tin oxide then there is rupture of this oxide and formation of dioxide [14]. This leads to a significant roughness, which allows the improvement of the film's electrical properties [14]. Therefore according to Dieguez et al. [14], at 600°C metallic tin changes directly to tin dioxide without forming tin monoxide. In addition Devers et al. [15] prepared tin dioxide films of about 20 \AA thickness by electrodeposition on a glass substrate. The annealing of these nanoaggregates was carried out between 270 and 300°C for 2 h. Such obtained films are not only amorphous, but also characterized by strong proportion of SnO_2 . The oxidation of our deposited films with variable thicknesses is carried out at 300 , 400 and 500°C for 1 and 2 h, according to the cases, in a continuous tube furnace of mark BL Barnstead/Thermolyne (tube furnace 21100) under oxygen flow.

3. Characterisation of the elaborated layers

X-ray diffraction analysis of these layers was made using a Philips PW1820 diffractometer, of type $2\theta-\theta$ (Bragg-Brentano geometry). The spectra of diffraction were obtained with radiation K_α of copper ($\lambda = 1.54\text{ \AA}$); the filter used is made of nickel.

The morphology and the nanostructures of these films were observed using an environmental scanning electronic microscope (FEI XL 30 FEG ESEM INSA, Lyon, France). ESEM is equipped with both secondary and backscattered electron detectors, and an EDAX Phoenix Energy Dispersive Spectrometer (EDS). The EDS system is used to identify the major elements present in a sample in concentrations greater than about 1 wt% and can detect elements as low as carbon in the periodic table.

4. Results and discussion

4.1. Macroscopic observation

We prepared tin layers of 500 and 1000 \AA thickness by vacuum evaporation. The films thus obtained are reflective. After annealing of these layers under oxygen flow at temperature varying between 300 and 500°C , they become transparent with light opacity. Macroscopic observation with the naked eye informs us about the change of structure of the layer obtained after its annealing under oxygen.

4.2. X-Rays diffraction analysis

The spectrum of Fig. 1(a) and (b) corresponds to the deposited tin films of 500 and 1000 \AA carried out by the evaporation vacuum technique on glass substrate. In this case we notice the occurrence of two peaks, which are located at the following angles: 15.5° and 32° ; these two peaks characterize the crystalline tetragonal metal tin (86-2264 (c)). The first peak is intense; this shows that the deposited grains on glass substrate are oriented. The diffraction spectra of Fig. 1(b) show well that the quantity of tin detected is more significant when the thickness of the film increases from 500 to 1000 \AA .

The thermal treatment of tin layer of thickness 500 \AA at 300°C for 1 h under oxygen flow modifies the initial structure leading to disappearance of the peaks characterizing the initial metal tin (Fig. 2). Indeed, in this case there is an enrichment of oxygen. The structure of pure tin disappears. There is occurrence of another oxide of amorphous structure. The majority of the authors report crystallinity, and the preferential orientation of the layers of tin dioxide depends a lot on the deposition method, the temperature, the duration of their annealing and also on the nature of the used substrate [16,17]. Thus, according to Refs. [18,19] the films annealed at temperatures lower than 375°C are amorphous.

We then increased the thickness of the layer of tin to 1000 \AA and the temperature of annealing to 400°C . The annealing of this layer of tin at 400°C under constant oxygen flow for 1 h leads to the formation of oxidized and crystalline phases, as indicated by the diffraction spectrum of Fig. 3(a). In fact many peaks appear. These characterize tin oxide

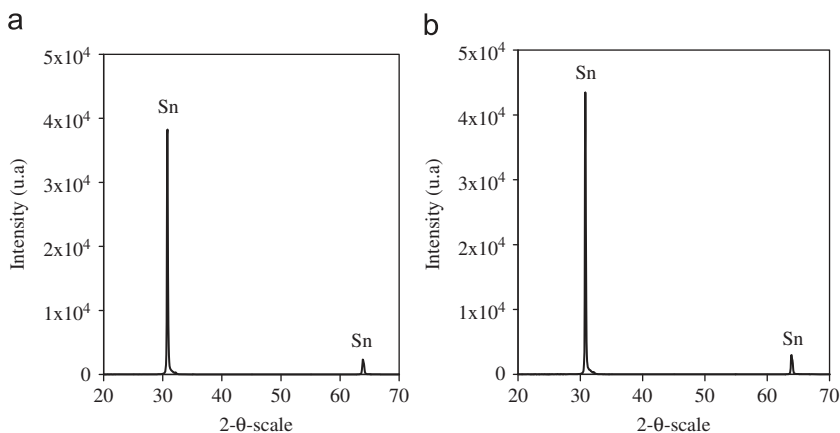


Fig. 1. X-ray diffraction spectra: tin on glass (a) 500 \AA and (b) 1000 \AA .

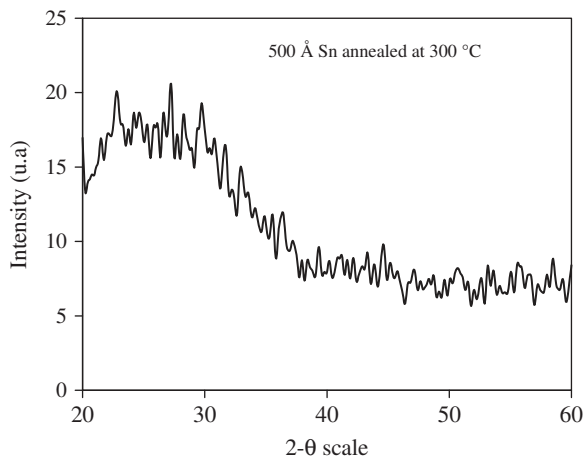


Fig. 2. X-ray diffraction spectrum of tin layer with 500 Å on glass annealed at 300 °C for 1 h under oxygen flow.

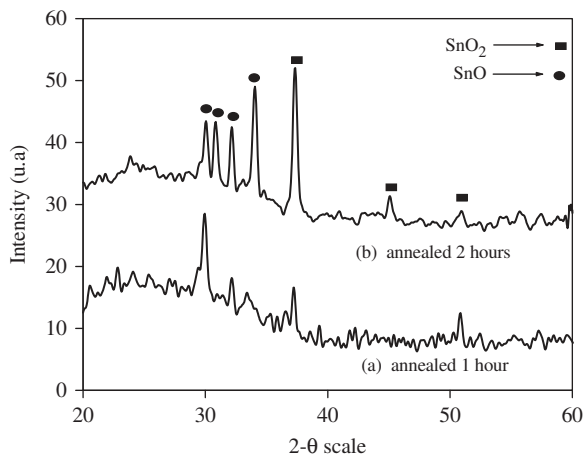


Fig. 3. X-ray diffraction spectra of tin layer of 1000 Å on glass annealed at 400 °C under oxygen flow for: (a) 1 h and (b) 2 h.

(SnO) and tin dioxide (SnO₂) both of which have a tetragonal structure according to the patterns (77-0450 (C)) and (06-0395(I)).

The duration of oxidation is very significant during the transformation of tin to tin oxide and tin dioxide. Samples annealed for 2 h at 400 °C showed peaks that characterized crystalline SnO and SnO₂ with significant intensities (Fig. 3(b)). This means that the quantity of crystals SnO and SnO₂ is more for an annealing for 2 h than for 1 h.

Concerning the case of 1000 Å thickness deposited film annealed at 500 °C the formation of SnO₂ of tetragonal structure according to pattern (77-0447) is obvious as indicated in Fig. 4(a). Note that the temperature was increased to 500 °C in order to obtain crystalline structure of SnO₂. Fig. 4(b) shows that SnO₂ (tetragonal) amount increases a lot with annealing time and SnO (tetragonal) still persists even when the annealing time reaches 10 h. Increase in the size of grains is the reason for the increase in peak intensity.

The intensity of peak located at 34° shows that there is a preferential orientation of SnO₂ in the crystallographic direction $\langle 101 \rangle$, which indicates a textured structure.

Briefly the temperature of annealing affects tin oxidation a lot to both SnO and SnO₂; for the oxidation temperature of 300 °C and an initial layer of 500 Å, the obtained film is oxidized but amorphous. At 400 °C and for a layer of 1000 Å, crystalline tin oxide and dioxide are formed. The quantity of tin dioxide increases in a significant way when

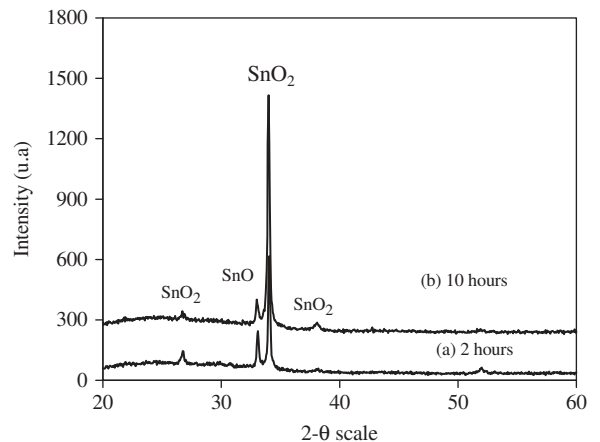


Fig. 4. X-ray diffraction spectrum of tin layer of 1000 Å annealed at 500 °C (a) 2 h and (b) 10 h.

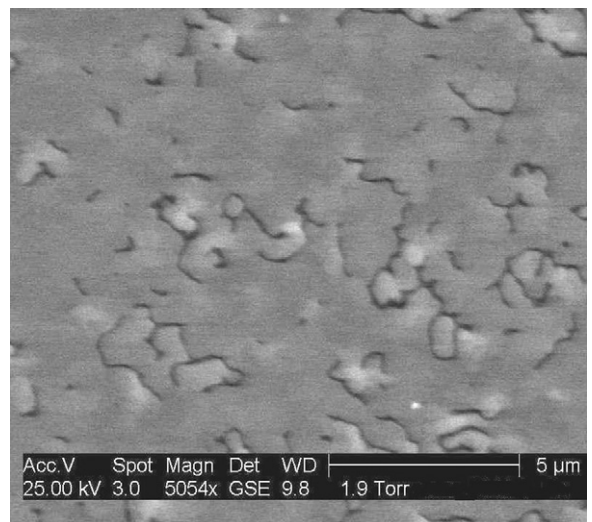


Fig. 5. SEM image of 1000 Å tin layer deposited on glass substrate.

the temperature of annealing is 500 °C for 2 and 10 h. In these cases the corresponding SnO₂ peaks are well marked, thus showing a strong proportion of this phase, but the Sn→SnO₂ transformation is not completed; indeed there remains a small proportion of SnO. Meanwhile the crystallization of tin dioxide for the sol-gel layers preparation process is complete after an annealing at 500 °C [8]. It is clear that the transformation to tin dioxide is dependent on the preparation method and the layer thickness.

4.3. Environmental scanning electronic microscopy analysis

The analysis by scanning electronic microscopy (SEM) of the layers obtained was carried out with two types of detectors: a detector of Gaseous scattering electronics (GSE) type and another of Back scattering electronics (BSE) type.

For 1000 Å tin layer deposited on glass, a crystalline microstructure formed of grains separated by grain boundaries (Fig. 5) is observed; this is confirmed by the X-ray crystallographic study, which identifies two peaks corresponding to crystallized tin of centered cubic structure. These Sn grains are made of crystals of various diameters and the diameter of these aggregates is approximately 0.5 μm (Fig. 5). After oxidation by annealing at 400 °C under oxygen flow for 2 h the morphology changes with the appearance of two quite distinct phases

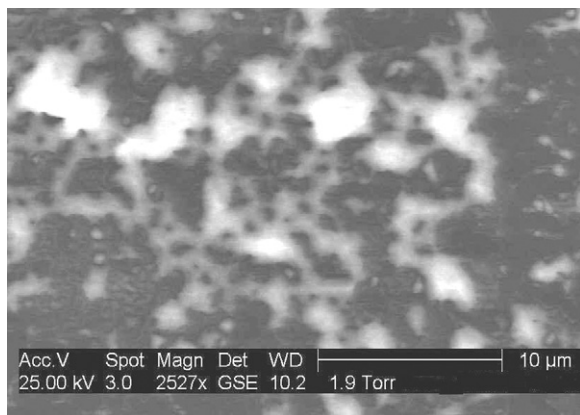


Fig. 6. SEM image of 1000 Å tin layer on glass substrate annealed at 400 °C for 2 h under oxygen flow.

(Fig. 6): the oxidized areas appear light in contrast to the substrate. This confirms the results obtained by X-ray diffraction.

The nature and the geometry of detector BSE in the microscope used in this study permit collection of two types of signals and investigation of specific information related to each one. This detector is split into two parts. When various signals are added we obtain the composition image. On the other hand when we realize, by means of associated electronics, the differences between these signals, we access topographic information.

It appears that the tin film oxidized at 400 °C for 2 h under oxygen flow consists of two different phases: oxides of tin and a fine “carpet” that corresponds to the glass substrate or to the initial metallic tin.

Fig. 7(a) and (b) showing the composition contrast and Fig. 8(a) and (b) showing the topographic contrast indicate that the light regions have a different chemical composition than the gray ones. These light regions correspond to the oxidized form of tin (SnO or SnO_2) with amorphous or crystalline structure. For an annealing at 400 °C for 2 h they are dispersed and are of a low amount (quantity).

When annealing is performed at 500 °C and under the same conditions as before, the quantity of oxide and especially that of dioxide increases; indeed this structure is in the shape of the agglomerates formed starting from the crystals. This is well illustrated on the micrographies of Fig. 9(a) and (b). The average diameters of these aggregates are about 1 μm after annealing for 2 h and increase when the annealing time is 10 h (Fig. 10(a) and (b)).

Dieguez et al. [14] mention this concept of aggregates and clusters. However the kinetics of transformation of tin to tin oxide or dioxide are different. Indeed at high temperature molten tin does not wet the glass substrate and then nanospheres are formed. Oxidation starts from the surface of these nanospheres towards their centers and tin oxide (SnO) is first observed.

The formation of SnO_2 (cassiterite) occurs only after annealing at 500 °C for 9 h. This formation of SnO_2 makes it possible to have good electrical properties [14]. However, the transformation of SnO into SnO_2 splits up the spheres and consequently the surface roughness increases [14]. In the present work, tin is deposited on a substrate at room temperature and therefore, it forms a flat layer, which is then oxidized during annealing. Small clusters nucleate directly on the surface of the substrate and grow into small islands when the atoms or molecules arrive on the surface. It is a three-dimensional growth. A typical case of this growth is that of a metal film on an insulating substrate [20]. Grains are formed and coalesce by constituting small agglomerates. This nodular structure is generally observed for compounds with a low melting point. The final microstructure is of nodular type, with more or less spherical clusters.

This stacking of nanocrystalline grains into small clusters, with an average dimension of about 1 μm is quite visible in Figs. 9 and 10.

When the tin layer thickness increases, the quantity of tin to be oxidized becomes more important and then there is simultaneous formation of several agglomerates and the layer appears more uniform on the surface. In conclusion, it is necessary to increase the thickness of the initial layer to get a more uniform surface.

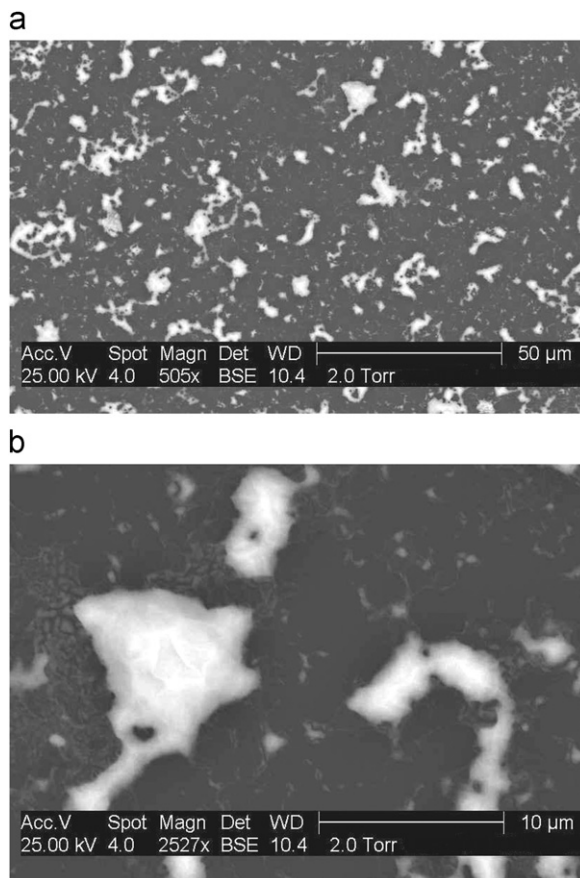


Fig. 7. Composition image with various growths showing the morphology of 1000 Å tin layer on glass annealed at 400 °C for 2 h under oxygen flow.

4.4. EDX analysis

EDX analysis of naked glass used shows that it is composed of a mixture of oxygen, sodium, magnesium, silicon (majority) and calcium and also Sn (Fig. 11(a)).

Fig. 11(b) presents the EDX spectrum of 1000 Å tin layer on glass; we noticed the appearance of a characteristic tin peak. Fig. 11(c) is related to the 1000 Å tin layer film annealed at 500 °C for 2 h under oxygen flow. This spectrum shows that the oxygen amount increases compared to the non-treated case. This confirms that there is oxygen enrichment of the tin layer after thermal annealing.

4.5. Electric properties of tin dioxide layers

Microstructure of the layers (size of the grains, surface–volume ratio, porosity, etc.) determines the electrical properties of the semiconductor oxide [7,8]. Ben Shalom et al. [21], who used the filtered vacuum arc deposition method for the manufacturing of their films, found resistivity of about $3 \times 10^{-3} \Omega \text{ cm}$. Similar values, $7 \times 7210^{-3} \Omega \text{ cm}$, have been reported by Shadia et al. [22] in the case of $\text{SnO}_2:\text{F}$ prepared by the spray-deposited method.

In the present study, the resistivity of the layers was obtained by the classical 4 point method. We measured the resistivity of the layers that have been annealed at 500 °C for 2–10 h and their values are, respectively, 0.27×10^{-3} and $5.70 \times 10^{-3} \Omega \text{ cm}$. According to the evolution and nucleation of SnO_2 the SEM micrographies show that SnO_2 grains are gathered into small aggregates, are separated by non-covered surfaces as tin is totally transformed, and the distance between two aggregates increases with annealing time (Figs. 9 and 10). This makes

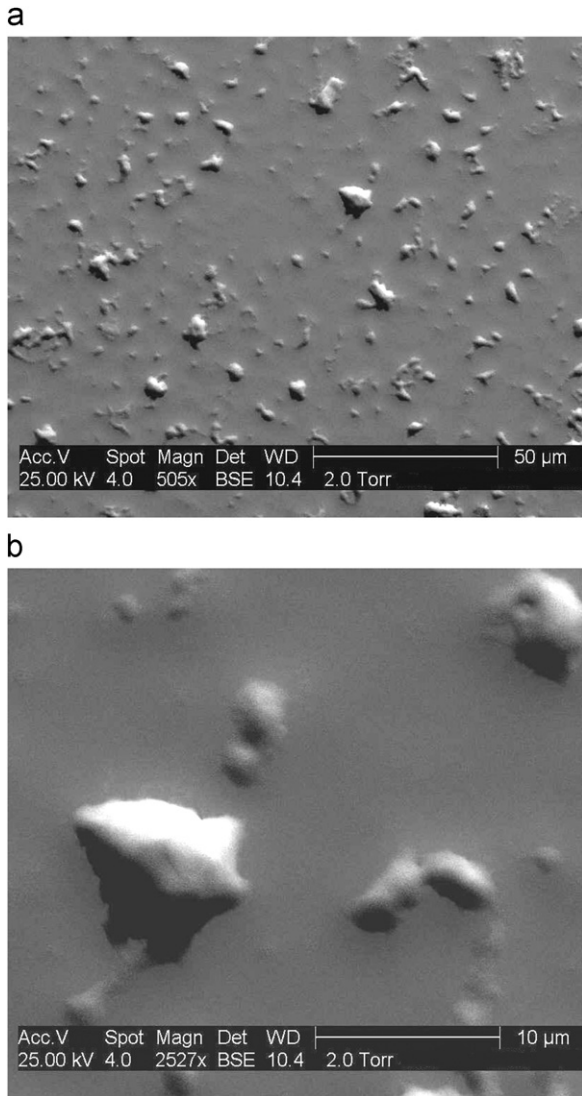


Fig. 8. Topographic image with various growths showing the morphology of 1000 Å tin layer on glass annealed at 400 °C for 2 h under oxygen flow.

the electronic conductivity very difficult from one grain to another grain localized in the vicinity of the aggregates. This is the main cause for the general resistivity enhancement. Increase in the annealing time increases both grain size and the distance between two aggregates.

The resistivity measured on the layers annealed for 10 h is about $5.70 \times 10^{-3} \Omega \text{ cm}$. SnO_2 coatings on glass can be used as electrodes in nanocrystalline photovoltaic cells if their electrical conductivity is increased by adequate doping. Their structure has a spongy morphology with voids or pores and large grains formed by agglomeration of smaller grains of SnO_2 . Indeed, in this case the specific surface of these layers is very large compared with the projected surface of these layers. This specific surface depends on several parameters: grain size, thickness of the layers, density of pores, oxygen flow, temperature of annealing, time of annealing and roughness of surface.

5. Conclusion

SnO_2 thin layers are essential in the design of nanocrystalline photovoltaic cells. The control of preparation

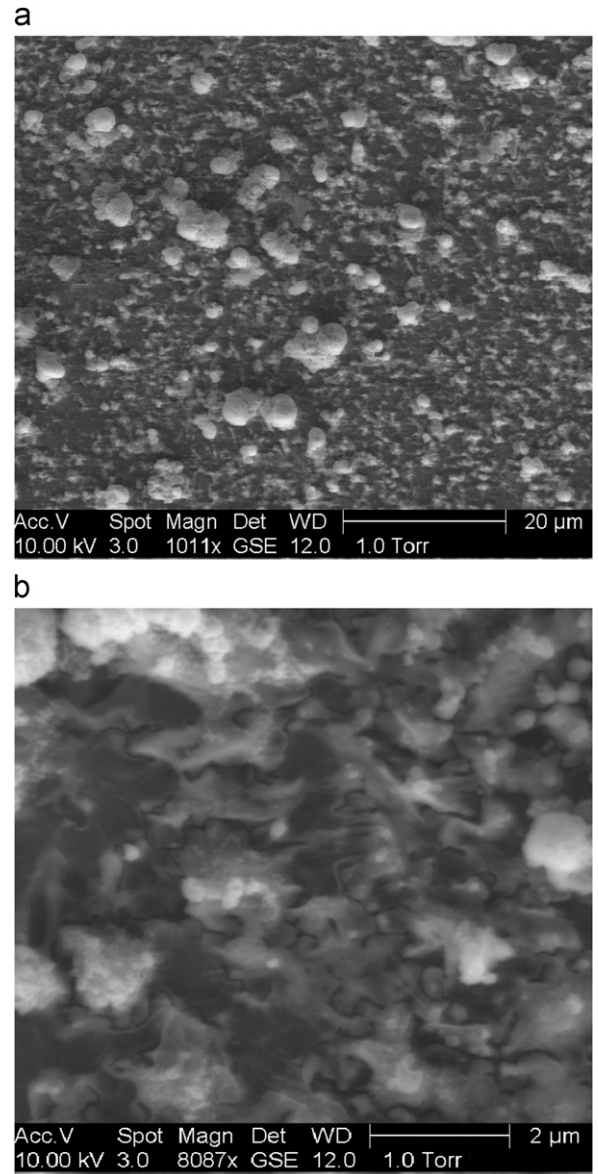


Fig. 9. Scanning electron microscopy with various growths showing the morphology of 1000 Å tin layer on glass substrate annealed at 500 °C for 2 h under oxygen flow.

of these layers is a significant step for the design and the realization of these solar cells. Several techniques of deposition of these layers are quoted in the bibliographical references. Our method consists of depositing a thin film of tin by vacuum evaporation on glass and then oxidizing it thermally. Macrographic observation with the naked eye shows that evaporated tin is reflective, thus identifying the structure of metal tin, which is confirmed by X-ray diffraction characterization. When this metal is thermally oxidized at 300, 400 or 500 °C, the observation with the naked eye shows a transparent layer with light opacity. This indicates that the structure of metal tin is modified: it is replaced by crystalline and/or amorphous tin oxide and tin dioxide according to conditions of the

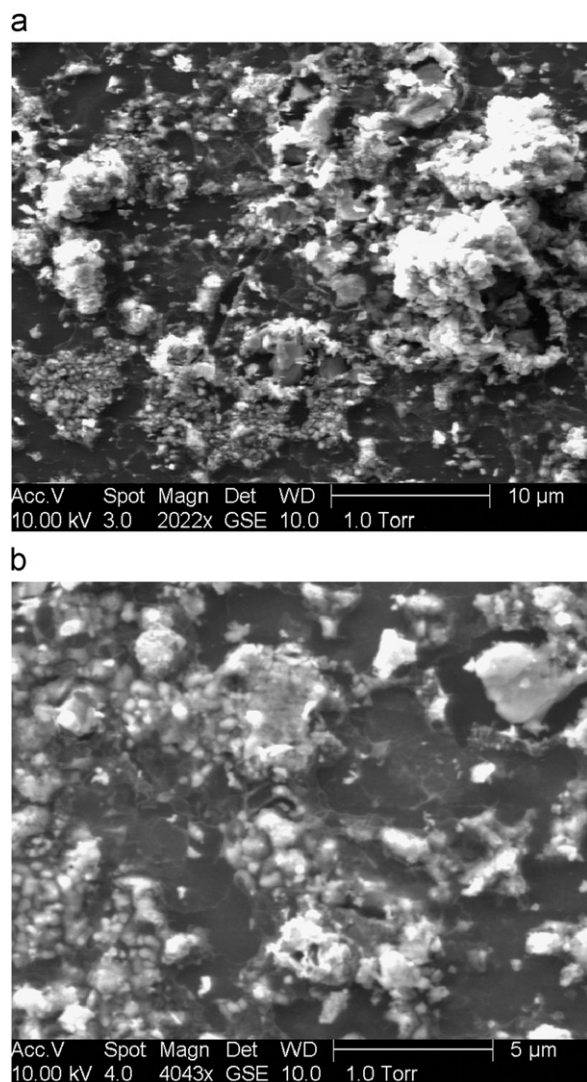


Fig. 10. Scanning electron microscopy with various growths showing the morphology of 1000 Å tin layer on glass substrate annealed at 500 °C for 10 h under oxygen flow.

elaboration. In fact, for a 500 Å tin layer with oxidation at 300 °C for 1 h, the obtained structure is amorphous. When we increase the thickness of the layer to 1000 Å and the temperature of annealing to 400 °C, tin is oxidized to tin monoxide and tin dioxide. When the time of annealing is 2 h the quantity of crystalline tin oxide and dioxide increases. Annealing a 1000 Å thick tin layer at 500 °C for 2 h induces the formation of tin oxide and nanocrystalline tin dioxide (tetragonal), as shown by X-ray diffraction. A longer annealing (10 h) leads to an increase of the tin dioxide proportion.

Scanning electron microscopy, combined with X-ray diffraction, reveals that after annealing, two different phases (SnO and SnO₂) coexist within the amorphous matrix. Both the volume fraction and the size of the oxide particles increase with annealing time. These particles are in the form of agglomerates whose size is about a micrometer.

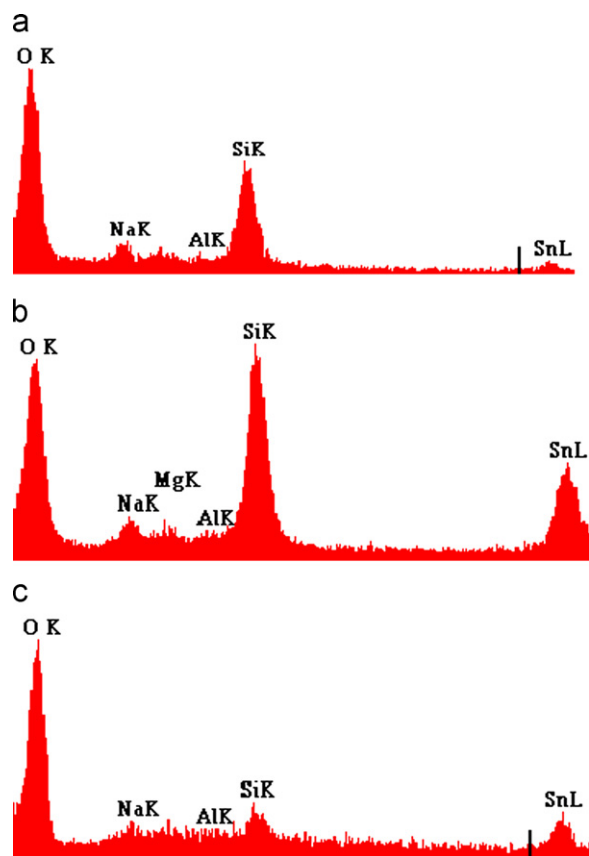


Fig. 11. EDX spectra: (a) glass, (b) 1000 Å tin layer deposited on glass and (c) the same layer annealed for 2 h at 500 °C.

The electrical resistivity of these layers, measured by the four point method, is correlated with the size of the oxide particles: the smaller the particle size, the lower the electrical resistivity.

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