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MAGNETRON SPUTTERED SnO_x FILMS ON TIN PROBED BY SLOW POSITRON IMPLANTATION SPECTROSCOPY

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SnO_x films grown on tin substrates via d.c. magnetron sputtering at different bias were studied by slow positron implantation spectroscopy. The change of substrate bias from -40 V to -140 V and its influence on the films is shown and discussed.

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1. Introduction

Tin-oxide-based films have several interesting applications in optics and ionics. The research interest to SnO_x thin films is due to their industrial applications — specific uses in liquid crystal displays, as heated windscreens for cars, as a mean of ensuring the mechanical reaction stability of glass bottles, as transparent heat reflectors in sodium or incandescent lamps and as high quality optical elements [1]. Most of these applications hinge on the ability of tin oxide to attain different properties as a result of doping [2]. Among the semiconducting oxides used for gas detection, SnO_2 is the most interesting material, particularly in the form of thin films, in miniaturized sensor devices [3]. However, the properties of the films depend strongly on the mode of preparation and could vary considerably from one technique to another [4]. Some of the most important factors affecting the microstructure of sputtered films are: the kind and the temperature of substrate, the rate of deposition of the condensing atoms, the pressure of the working gas, the substrate surface roughness, and substrate bias and bombardment of the surface with ions or electrons. The increase in oxygen pressure leads to the formation of films with different grain size and a mixture of different phases and defect structures [5]. Slow positron implantation spectroscopy (SPIS) has been used to successfully investigate a variety of thin film structures [6–8].

The purpose of the present study is to investigate the effect of negative substrate bias on the thickness and defect structure of SnO_x films, deposited on tin substrates. The deposition conditions were chosen as to vary the substrate bias but constant target input power.

2. Experimental

The SnO_x films with a thickness $d = 1$ μm were deposited on tin plates of 20 mm diameter by d.c. magnetron sputtering in argon–oxygen atmosphere at an oxygen partial pressure of 5×10^{-4} mbar. The sputtering system was with a substrate carrier arranged for application of a negative bias potential. The thickness d and the deposition rate $v_{\mathrm{d}} = 18$ Å/s were measured during deposition by using a quartz oscillator MIKI FFM. The substrate temperature was not controlled during the deposition but was always $< 60°$C. The total pressure was kept constant ($p_{\mathrm{total}} = 8 \times 10^{-3}$ mbar). The source material was 99.99% pure tin. During the deposition the current was kept constant ($I = 0.3$ A). The sputter target input power was held constant at 90 W with the bias stepped from −40 V to −140 V.

SPIS measurements were performed at UEA Norwich [9]. Sample characteristics were extracted from the $S(E)$ data by fitting with the program package VEPFIT [10].

3. Results and discussion

The obtained SPIS data are shown in Fig. 1. In order to know exactly the bulk characteristics S_{b} (0.4540) and L_{b} (306) needed for the evaluation of these data one sample was measured from its back side. Every $S(E)$ spectrum shown in Fig. 1 was fitted by a three-layer model, i.e. two "defected layers" and bulk (reaching to "infinity"). Results are collected in Table. The thickness of layer 1 (l_1) and 2 (l_2) is related to zero at the surface, i.e. the absolute thickness of layer 2 is given by subtraction of l_1 from l_2. The results from SPIS may be summarized as follows:

1. The total layer thickness sputtered to the substrate is about 400–600 nm, as given by l_2. This points to a removal of some substrate and already deposited film during sputtering as the sputtered thickness should be 1 μm according to its control by a quartz oscillator. Such effect was described to exist in Refs. [11, 12].

2. There is a surface layer of about 120 nm thickness characterised by a very high S value (about 10.5% increase compared to the bulk) and very short positron diffusion length (about 1–2 nm). This might be due to the formation of open-volume defects corresponding in size to the agglomeration of several vacancies. Another but less defected layer towards the substrate follows having about 8.8% increase in S compared to the bulk and a diffusion length of 6 nm. Its thickness is given by $l_2 - l_1$. Open-volume defects should be smaller in size compared to the surface layer.

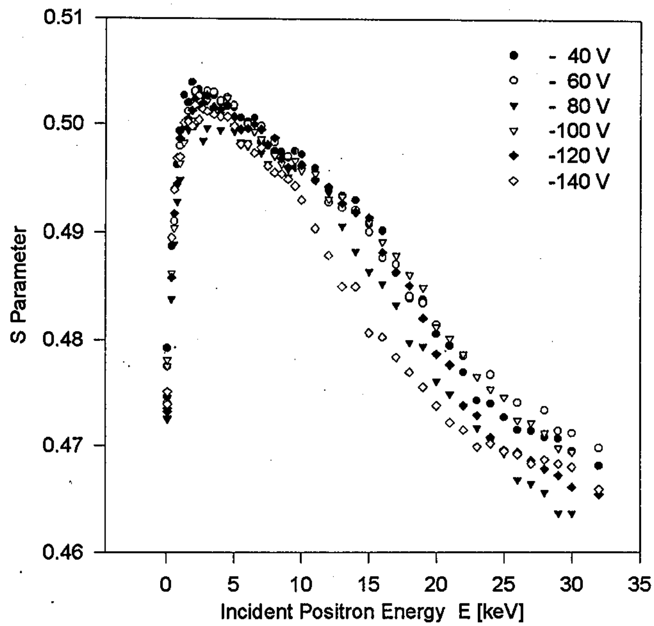

Fig. 1. S-parameter versus incident positron energy of magnetron sputtered SnO_x films on tin for different bias potentials.

TABLE

VEPFIT results (values with no given errors were fixed).

Bias [V]	S_1	S_2	S_{bulk}	l_1 [nm]	l_2 [nm]
–40	0.5030	0.4953(3)	0.4626(4)	108(5)	627(6)
–60	0.5030	0.4927(3)	0.4653(4)	145(6)	640(9)
–80	0.4999	0.4945(3)	0.4544(4)	122(11)	467(6)
–100	0.5010	0.4939(2)	0.4587(4)	127(6)	600(31)
–120	0.5020	0.4946(2)	0.4580(3)	124(6)	639(6)
–140	0.5012	0.4915(3)	0.4645	133(5)	409(26)

This interpretation is supported from the results in Ref. [13] where the presence of a positron lifetime of about 400 ps in all films prepared at identical conditions (bias, oxygen partial pressure) implied the existence of defects like vacancy clusters. However, in this work no depth resolution was possible.

3. The substrate below the deposited films appears to be still damaged compared to the bulk as measured from the back side of the sample. This damage may originate from ion bombardment because of the "compressing" effect of Ar^+ ions [14] and is attended with the process of implantation of Ar^+ ions and removing of the growing film [11, 12]. It is indicated and characterised by an increase in S of 1.9–2.2% compared to the bulk and a diffusion length of about 80 nm.

However, at a bias of –80 V and –100 V the positron parameters characterizing the substrate approach almost the values of the "true" bulk as measured from the back side of the sample. It is difficult to judge if at these voltages we really do have "ideal" sputtering conditions. However, this finding is consistent with hints from earlier studies by conventional positron lifetime spectroscopy [13] which indicate a decrease in the concentration of vacancy clusters around –100 V bias.

4. Conclusion

It could be shown that SnO_x films sputtered on tin substrate at different conditions consist of two layers having different defect structure on top of a damaged substrate. Furthermore, the film thickness is found to be substantially smaller than indicated from a separate measurement by a quartz oscillator. The optimum deposition conditions as indicated by an almost undamaged substrate are observed at –80 V and –100 V bias.

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