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ScienceDirect
Thin Solid Films 515 (2007) 2921 – 2925
www.elsevier.com/locate/tsf

Transparent conducting indium oxide thin films grown by low-temperature metal organic chemical vapor deposition

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Received 4 January 2006; received in revised form 1 August 2006; accepted 29 August 2006
Available online 2 October 2006

Abstract

We have grown indium oxide thin films on silicon substrates at low temperature by metal organic chemical vapor deposition. Polycrystalline film growth could only be obtained at temperatures below 400 °C. Above 400 °C, metallic indium deposition dominated. We have investigated the effect of substrate temperature and reactor pressure on the film growth and structural properties in the range of 250–350 °C and $5*10^3$–$4*10^4$ Pa. The film grown at 300 °C exhibited a resistivity of about 3.6×10^{-3} Ω cm and a maximal optical transmittance of more than 95% in the visible range. The film showed an optical band gap of about 3.6 eV.
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Keywords: Metal organic chemical vapor deposition; Indium oxide; X-ray diffraction; Grain size

1. Introduction

Indium oxide is a very important material for microelectronic applications. It is a transparent semiconducting oxide material, which is n-type with a wide band gap (~3.6 eV). Indium oxide (InO_x) is widely used as transparent conductive electrode in electronic devices of various types, i.e., in liquid crystal displays [1], solar cells [2], in tunnel junctions as barrier layers [3] and in gas sensors [4–6].

Various methods for the preparation of indium oxide thin films like evaporation [7,8], sputtering [9–12], spray pyrolysis [13–17], atomic layer deposition [18], thermal oxidation [19], ion assisted deposition [20], pulsed laser deposition [21–23], ultrasonic spray chemical vapor deposition (CVD) [24] and sol–gel [25] have been described already. High-quality films are expected to be grown by metal organic CVD (MOCVD) [26], which was rarely reported in the literature. Kim et al. [26] grew indium oxide films with MOCVD using triethylindium as metal precursor and O_2 as oxygen precursor at temperatures in the range of 200–300 °C. Maruyama and Kitamura [27] used plasma enhanced MOCVD to grow indium oxide films. They obtained amorphous indium oxide films at substrate temperatures in the range of 130–200 °C and got polycrystalline indium oxide between 200 and 360 °C.

Accordingly, in this paper, we report on a simple method for the deposition of low-temperature InO_x on Si substrates using a reaction of a trimethylindium (TMIn) and H_2O vapor. We have varied the substrate temperature in order to obtain single phase InO_x thin films. The temperature and reactor pressure have an effect on the grain sizes of the deposited InO_x films. Finally, the electrical and optical properties of the InO_x films were determined.

2. Experimental details

The InO_x films were deposited in a horizontal MOCVD reactor (AIX200) on Si substrates with (100) orientation. TMIn and H_2O have been used, respectively, as indium and oxygen sources. N_2 was used as carrier gas, which passed through the TMIn and H_2O bubbler. The substrate was cleaned with HF-based mixture ($NH_4F/HF/H_2O$, BOE = 7:1) and dried before

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doi:10.1016/j.tsf.2006.08.030

loading into the system. The substrate temperature and the reactor pressure were varied in the range of 250–700 °C and $5*10^3$–$4*10^4$ Pa. The flow rate of TMIn and H_2O were adjusted to 6 μmol/min and 431 μmol/min, respectively. The flow rate of TMIn is comparable to the one used by Maruyama and Kitamura [27] (growth rate of about 780 nm/h corresponding to the flow rate of TMIn of about 68 μmol/min). We obtained a maximal growth rate of about 170 nm/h.

To study the structural properties of the thin InO_x films (50–170 nm), an X-ray diffractometer (XRD, Siemens D5000, $CuK_{\alpha 1}$-$\lambda = 0.15405$ nm) and scanning electron microscopy (SEM, Hitachi S-2700) with an operating voltage of 12 kV were used. Because of the small thickness of deposited films, we used small angle diffraction in XRD measurements to suppress substrate signals. The film thickness was determined by a multiangle single-wavelength ellipsometer (Sentech CER ellipsometer). The electrical properties of InO_x thin films were examined by Hall measurements (accent HL5500). The optical properties were characterized by an optical spectrophotometer (UV-VIS-NIR Cary 5000 spectrophotometer) and a self-build photothermal deflection spectrometer (PDS).

3. Results and discussion

3.1. Effect of the substrate temperature and reactor pressure on growth rate and grain size

In Fig. 1, the XRD pattern of deposits on Si substrates at substrate temperatures in the range of 250–700 °C was shown. The reactor pressure was held constant at $2*10^4$ Pa. The spectra from the deposits achieved at 500 °C and 700 °C exhibit main peaks at 32.97°, corresponding to the (101) diffraction peak of body-centered tetragonal metal indium [28]. The diffraction pattern from the sample deposited at 400 °C shows not only peaks from indium but also a peak at 30.58° corresponding to the (222) main diffraction peak of body-centered cubic indium oxide [28]. The diffraction spectra in Bragg Brentano geometry for the samples deposited below 400 °C (250–375 °C) indicate that they are pure indium oxide films with a preferred (222) orientation. Thus, only below 400 °C pure indium oxide can be grown. This behavior is the result of the pyrolysis of TMIn. Jacko and Price [29] concluded that the mechanism of the pyrolysis of TMIn is as follows:

$$In(CH_3)_3 \rightarrow In(CH_3)_2 + CH_3, R1,$$

$$In(CH_3)_2 \rightarrow In(CH_3) + CH_3, R2,$$

$$In(CH_3) \rightarrow In + (CH_3), R3,$$

$$nIn(CH_3) \rightarrow [In(CH_3)]_n, R4,$$

$$2(CH_3) \rightarrow C_2H_6, Rr,$$

$$(CH_3) + C_6H_5{-}CH_3 \rightarrow C_6H_5{-}CH_2 + CH_4, Ra.$$

They found that below 400 °C reaction 2 (R2) occurs rapidly after reaction 1 (R1), but that reaction 4 (R4), the formation of an $In(CH_3)$ polymer, occurs in preference to breaking of the third In–C bond. Above 400 °C, reaction 3 (R3) takes place. Consequently, metallic indium can be deposited. Accordingly, metallic indium exists equal and above 400 °C in the process.

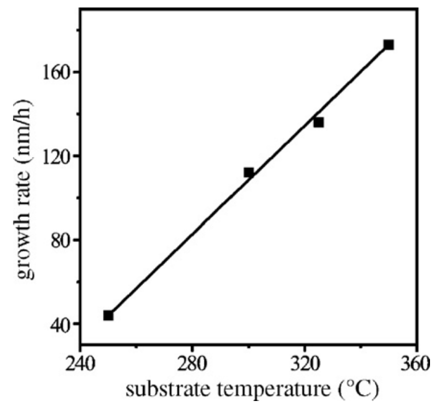

Fig. 2. Dependence of growth rate on the substrate temperatures at a constant reactor pressure of $2*10^4$ Pa.

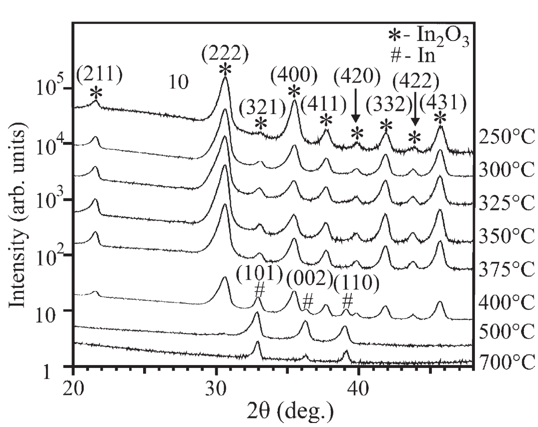

Fig. 1. XRD patterns of InO_x on Si substrate grown at different substrate temperatures and a constant reactor pressure of $2*10^4$ Pa (* for In_2O_3 and # for In).

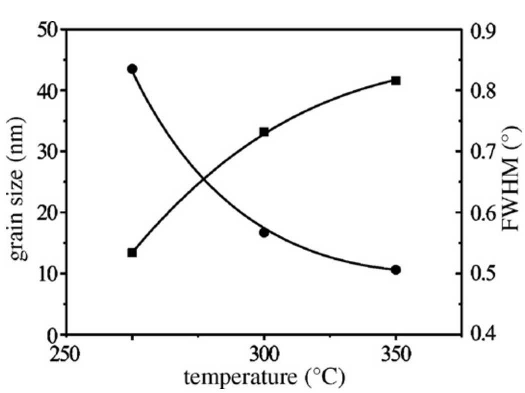

Fig. 3. FWHM from (222) diffraction peak in XRD and grain size from SEM at different substrate temperatures and a constant reactor pressure of $2*10^4$ Pa.

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