การเคลือบฟิล์มบางอินเดียมทินออกไซด์นำไฟฟ้าบนกระจก ด้วยวิธีการควบคุมกำลังของดีซีแมกนีตรอนสปัตเตอริง

Deposition of ITO Conducting Thin Film on Glass By Controlling DC Magnetron Sputtering Power

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บทคัดย่อ

ฟิล์มบางอินเดียมทินออกไซด์ (ITO) เคลือบบนกระจำด้วยวิธีการควบคุมกำลังไฟฟ้าของดีซีแมกนีตรอนสปัตเตอริง โดยความหนาของชั้น ITO ที่เคลือบบนกระจกสามารถวัดได้ด้วยเทคนิคอินเตอร์เฟอร์โรความต้านทานแบบแผ่นของฟิล์มได้ทำการวัดด้วยเทคนิคการวัดแบบสี่จุด จากนั้นฟิล์มบางถูกนำไปวิเคราะห์หาโครงสร้างผลึกและพลังงานยึดเหนี่ยวของโมเลกุล ด้วยเทคนิคการเลี้ยวเบนของรังสีเอกส์และเทคนิคการเรืองแสงของรังสีเอกส์ตามลำดับ พบว่าฟิล์มบางมีความหนาอยู่ในช่วง 204 ถึง 793 นาโนเมตร เมื่อทำการเปลี่ยนแปลงกำลังของดีซีแมกนีตรอนสปัตเตอริงจาก 50 ถึง 150 วัตต์ อีกทั้งความต้านทานแบบแผ่นของฟิล์มบางมีค่าระหว่าง 264 ถึง 499 โอห์มต่อตารางเซนติเมตร พร้อมกันนี้ ค่าพลังงานเหนี่ยวนำและโครงสร้างผลึกที่วิเคราะห์ได้ชี้ชัดว่าฟิล์มบางที่เตรียมขึ้นมานั้นเป็นฟิล์มบางของอินเดียมทินออกไซด์

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Abstract

Indium-Tin-Oxide (ITO) thin films were deposited on glass substrates using DC magnetron reactive sputtering at different bias sputtering power deposition conditions. The ITO film thickness was measured by interferometry technique. The sheet resistances of the films were measured by the four-point probe method. The films were characterized by x-ray diffractrometer (XRD) and synchrotron x-ray photoemission spectroscopy (XPS) for their crystal structure and binding energies, respectively. It was found that the film thickness changed from 204 to 793 nm when the sputtering power was changed from 50 to 150 W. The resistances of the films were from 264 to 499 Ω/cm^2. The obtained binding energies and crystal structure indices confirmed ITO film.

Keywords : Indium Tin Oxide, ITO, Thin film, magnetron sputtering

Introduction

Tin-doped indium oxide (ITO) thin films have been extensively studied for transparent conducting substrates that are widely used as electrodes in high conductivity and transparency in the visible region solar cells, organic light emitting diodes (OLED), touch screen panels and flat panel displays (FPDs). ITO films can be fabricated by various techniques, such as chemical vapor deposition (CVD) (Maki-2003), thermal evaporation deposition (Belo-2008), the sol-gel method (Cho, 2011; Yang, 2009), electron beam evaporation (Senthilkumar, 2010; Fallah, 2007; Coutal, 1996), and magnetron sputtering (Kim-2010, Guillén, 2006; Teixeira, 2002). Indium oxide (In_2O_3) based material has been doped with tin (Sn) to improve the electrical conductivity. Sputtering is the most widely used for large-scale deposition since it is a simple deposition technique.

Maki et al. (Maki-2003) fabricated ITO thin films by aerosol CVD. The electrical resistivity was related to the carrier concentration and its mobility. The optical band gap was 3.90 eV, for all prepared film conditions , compred with 3.75 eV for In_2O_3 lattice.

Cho et al. (Cho-2011) prepared ITO thin film by a sol-gel spin coating process fired, and then annealed in the temperature range of 450 - 600^{o}C. The film that was annealed at 500^{o}C and subsequently treated with 0.1 N HCl solutions for 40 s showed a sheet resistance of 225 Ω/square. It was confirmed that the decrease in the sheet resistance is related to the removal of residual carbon, as identified in the XPS spectrum.

Kim et al. (Kim, 2010) deposited ITO on glass substrates at various oxygen flow rates using a planar magnetron sputtering system with facing targets. In this system, the strong internal magnets inside the target holders confined the plasma between the targets. The electrical resistivity and optical percentage transmission properties of the ITO films produced at the optimal oxygen flow rate were approximately 4×10^{-4} Ω cm and >80%, respectively.

Teixeira et al. (Teixeira, 2002) deposited amorphous ITO thin films on glass substrates using DC magnetron reactive sputtering at different bias voltages and substrate temperatures. Some improvements on the film properties, microstructure and other characteristics with the deposition conditions were observed. Amorphous and polycrystalline films can be obtained at different sputtering deposition parameters.

In this work, we report on the preparation of thin films by DC magnetron sputtering method. The films thickness, sheet resistance, crystal structure, and binding energy of the ITO thin film were also reported.

Experimental

ITO films were deposited on glass substrate by DC magnetron sputtering using ITO target with a purity of 99.99 % (90 wt% In_2O_3 and 10 wt% Sn_2O). The glass substrate temperature was room temperature. The chamber was evacuated to a base pressure of 4×10^{-5} torr. The pressure during deposition was 6×10^{-4} torr or less. The 20 sccm argon gas flow rate was used as sputtering gas in the chamber. The distance between target and substrate was 90 mm. The DC power was varied from 50, 100 and 150 W and a deposition time was 20 min. The as-deposited films were post-annealed in air at temperature of 450°C for 2 h.

The film thickness and sheet resistance of ITO films were measured using interferometer and four-point probe technique, respectively. The crystallographic structures and binding energy of ITO films were analyzed by X-ray diffraction and synchrotron XPS at the Siam Photon Laboratory, SLRI, Thailand, respectively.

Results and discussion

In Table 1, an overview of the thickness and sheet resistance of the depositedfilms ITO at different sputtering powers is shown. With deposition powerchanging from 50 to 150 W, the thickness increases from 204 to 793 nm. The lowest value of the sheet resistance is 264 ohm/cm^2 for sputtering power of 150 W. Other values of sheet resistance in ITO film include 351 Ω/cm^2 for sputtering power of 100 W and 499 Ω/cm^2 for sputtering power of 50 W. Obviously, a decrease in the sheet resistance was observed after increasing the power of sputtering at the same condition.

Table 1 Sputtering power, thickness, and sheet resistance of deposited ITO films

Sputtering power (W)	Thickness (nm)	Sheet resistance (Ω/cm^2)
50	204	499
100	560	351
150	793	264

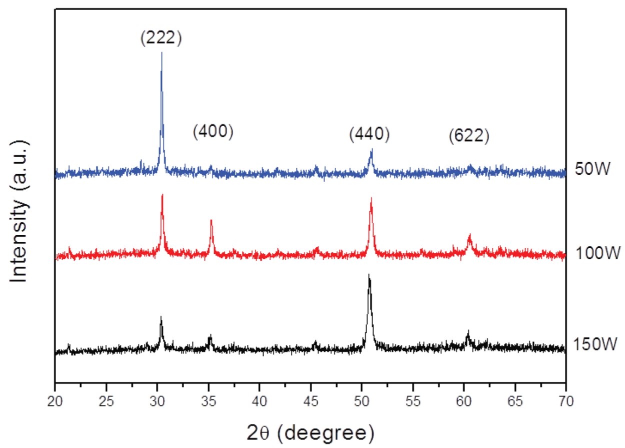

Figure 1 XRD pattern of ITO films with power of sputtering

Figure 1 shows the XRD pattern of ITO deposited on glass substrate for different powers of sputtering. For 50 W sputtering power shows diffraction peak along the (222) and (440). The (222) peak has the preferred orientation (maximum intensity). For 100 and 150 W sputtering power conditions show additional diffraction peaks indicating the formation of crystallite in (400) and (622) orientations with a preferred orientation along (440) direction. The orientations are in good agreement with previous reported values (Kumar, 2011; Sathiaraj, 2008).

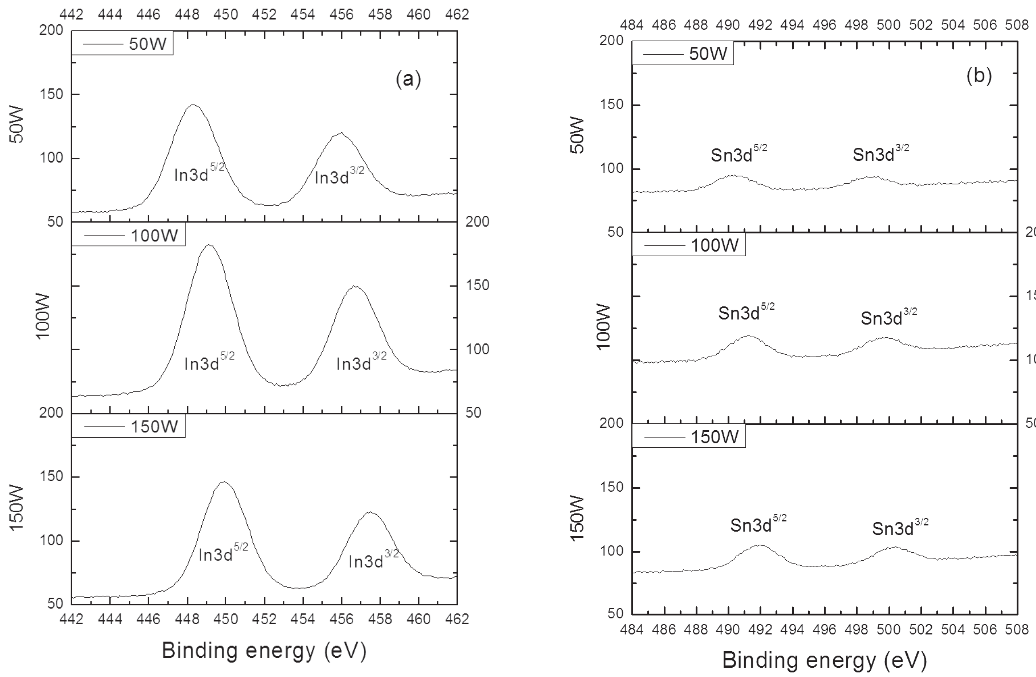

Figure 2 XPS spectra of ITO (a) In3d and (b) Sn3d

Figure 2 shows the XPS spectra of ITO deposited on glass substrate for different powers of sputtering. The XPS spectra for peaks of In 3d and Sn 3d doublet are shown in Figure 3(a) and (b), respectively. These peaks show the evidence of only one binding state for In and Sn since there is no sign of broadening or splitting of the peaks. The binding energy of In $3d^{5/2}$ at 448.1 eV can be attributed to the In^{3+} bonding state from In_2O_3. The binding energy of Sn $3d^{5/2}$ at 489.7 eV corresponds to the Sn^{4+} bonding state from SnO_2 usually found in the ITO lattice (Cho, 2011; Teixeira, 2002).

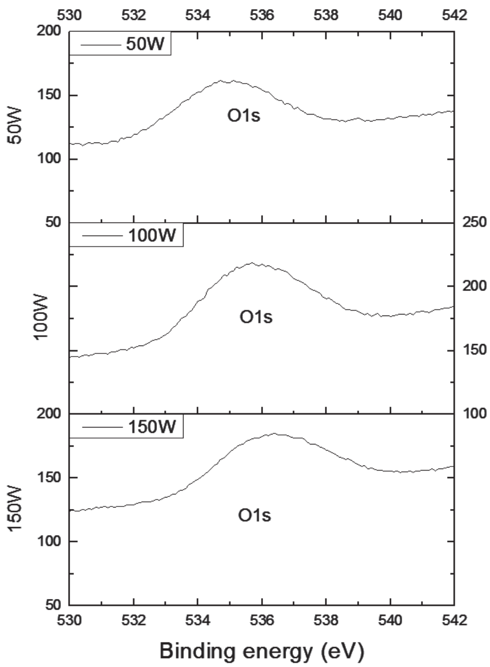

Figure 3 XPS spectra of ITO O1s

Figure 3 indicates that oxygen is bonded to the indium and tin and that ITO is formed. The binding energy of O1s at 535.9 eV can be attributed to oxygen bonded as In_2O_3 and SnO_2. The O 1s peak is no sign of broadening or splitting of the peaks can be assigned to lattice oxygen in a crystalline ITO phase (Chuang, 2010).

Conclusion

ITO conducting thin films was successfully deposited on glass substrates by DC magnetron sputtering. The characterization of ITO film by interferometry technique revealed thickness of around 204 to 793 nm and it depended on sputtering power. The sheet resistances of the films vary between 264 and 499 Ω/cm^2 measured by the four-point probe method. In_2O_3 phases observed from peak (222), (400), (440) and (622) of XRD pattern agreed with sputtering process. XPS from synchrotron showed peaks of binding energy of In3d and Sn3d can be attributed to In_2O_3 and SnO_2, respectively. The obtained binding energies and crystal structure indices confirm ITO crystal structure. It can be concluded that the ITO film by DC magnetron sputtering with the sputtering power of 100 W gives the optimum conduction and thickness to be used as conducting substrate for solar cells.

References

C. Coutal et al. (1996). Fabrication and characterization of ITO thin films deposited by excimer laser evaporation. Thin Solid Films. 288. 248-253.

C. Guillén, J. Herrero. (2006). Influence of oxygen in the deposition and annealing atmosphere on the characteristics of ITO thin films prepared by sputtering at room temperature. Vacuum. 80. 615-620.

G.S. Belo, B.J.P. da Silva, E.A. de Vasconcelos, W.M. de Azevedo, and E.F. da Silva Jr. (2008). A simplified reactive thermal evaporation method for indium tin oxide electrodes. Applied Surface Science. 255. 755-757.

H. Cho, and Y.H. Yun. (2011). Characterization of indium tin oxide (ITO) thin films prepared by a sol-gel spin coating process. Ceramics International 37. 615-619.

H. R. Fallah, M. Ghasemi, A. Hassanzadeh, and H. Steki. (2007). The effect of annealing on structural, electrical and optical properties of nanostructured ITO films prepared by e-beam evaporation. Materials Research Bulletin. 42. 487-496.

J. Kumar, N. R. Chandra Raju, and A. Subrahmanyam. (2011). Thickness dependent physical and photocatalytic properties of ITO thin films prepared by reactive DC magnetron sputtering. Applied Surface Science. 257. 3075-3080.

K. Maki, N. Komiyaa, and A. Suzuki. (2003). Fabrication of thin films of ITO by aerosol CVD. Thin Solid Films. 445. 224-228.

L.L. Yang, X.D. He, F. He, Y.B. Li, S. Zhang, T. An, and W.T. Zheng. (2009). Surface characterization of Ag-ITO cermet films prepared by a new sol-gel method. Thin Solid Films. 517. 4979-4983.

M. Chuang. (2010). ITO Films Prepared by Long-throw Magnetron Sputtering without Oxygen Partial Pressure. Journal of Materials Science & Technology. 26(7). 577-583.

T.S. Sathiaraj. (2008). Effect of annealing on the structural, optical and electrical properties of ITO films by RF sputtering under low vacuum level. Microelectronics Journal. 39. 1444-1451.

V. Senthilkumar, P. Vickraman, M. Jayachandran, and C. Sanjeeviraja. (2010). Structural and optical properties of indium tin oxide (ITO) thin films with different compositions prepared by electron beam evaporation. Vacuum. 84. 864-869.

V. Teixeira, H.N. Cuia,1, L.J. Mengb, E. Fortunatoc, and R. Martins. (2002). Amorphous ITO thin films prepared by DC sputtering for electrochromic. applications Thin Solid Films 420-421. 70-75.