Antistatic and antireflection coating using indium tin oxide prepared by magnetron reactive sputtering

Haixing Chen (陈海星)*, Yuyong Huang (黄翌勇), Xuanqian Xu (徐玄前), and Shengyuan Bai (白胜元)

Optics Division, Oerlikon (Shanghai) Co., Ltd., Shanghai 200131, China

**E-mail: haixing.chen@gmail.com*

Received December 3, 2009

Antistatic and antireflection (ASAR) coating using indium tin oxide (ITO) by magnetron reactive sputtering (MRS) technique is presented. The relationship between sheet resistance and optical transmittance of ITO prepared by MRS is investigated, and the optimum ITO parameters by MRS are studied through the variation of oxygen flow, temperature, argon flow, and sputtering power. The optical constant of ITO is modeled by combining a single Lorentz oscillator and a Drude free-electron component in the range of 300–1500 nm, which fits well with the experimental data. ASAR coating is designed using ITO based on the optimized parameters, and is implemented by MRS. Experimental results show that ASAR coating by MRS displays high transmittance of up to 99.2%, and low sheet resistance of less than 1.1 kΩ.

OCIS codes: 310.0310, 160.0160, 220.0220.

doi: 10.3788/COL201008S1.0201.

Static electricity is harmful in many applications such as ophthalmic lenses, medical devices, panel displays, and others as it has the tendency to collect particles and dust. Therefore, it is necessary to fabricate components using an antistatic and antireflective (ASAR) coating solution in order to avoid static accumulation while keeping the transmittance as high as possible[1,2]. Static electricity can be prevented by using either ion conduction or electron conduction. The latter is nearly free from environmental conditions and provides a stable preventive performance. Conductive oxides such as indium tin oxide (ITO) are used for the electron conduction method. In this method, an electric current is generated by the motion of electrons or halls caused by defects in a crystal structure. In this letter, we have achieved the ASAR coating with multi-layers of ITO, Nb_2O_5, and SiO_2, which meet the requirement for high transmittance and low resistivity.

ITO, a well-known material with good optical property and low resistivity[3], is the most commonly used transparent conductive coating material for panel displays, electro-magnetic interference (EMI) coatings, touch-screens, and others. The purpose of our study is to achieve ITO film with both high transmittance and high conductivity. The key factor is that the ITO film should be prepared with low absorption and low resistivity. Conductive ITO film has been widely researched and fabricated by various processes such as electron beam evaporation[4], ion assisted deposition, direct current (DC) magnetron sputtering, mid-frequency sputtering[5], and others, all of which are aimed at obtaining stable film properties.

The ITO film was prepared by reactive DC magnetron sputtering based on Balzers MSP$^{\rm TM}$ platform. Weight ratio of the ITO target was 90:10 for In_2O_3 and SnO_2. Niobium (99.999%) and silicon (99.999%) targets were used as materials for dielectric layers. The base pressure was typically 1.1×10^{-4} mbar, obtained by the use of a turbo pump backed by roughing pump. DC magnetron sputtering was performed in the reactive mode using an argon/oxygen mixture. Argon and oxygen had purities of 99.999% and were introduced by gas flow controllers. Plasma emission monitoring was used to control the reactive sputtering with a planar magnetron.

Since ITO films can have a wide range of properties depending on deposition method and parameters, it is relatively difficult to determine the optical constant of a given ITO film in comparison to other materials. ITO films are sensitive to environmental conditions; therefore, they can be used as gas sensors. To obtain a good fit to the optical constant of ITO film and simplify the calculation, the model, which is a combination of a single Lorentz oscillator and a Drude free-electron component, was employed[6]:

$$\varepsilon_1(\omega)=\varepsilon_\infty-\frac{\omega_p^2}{\omega^2+\gamma^2}+\omega_p^2\frac{\omega_1^2-\omega^2}{\left(\omega_1^2-\omega^2\right)^2+\beta^2\omega^2},\quad(1)$$

$$\varepsilon_2(\omega)=\frac{\gamma}{\omega}\frac{\omega_p^2}{\omega^2+\gamma^2}+\omega_p^2\frac{\beta\omega}{\left(\omega_1^2-\omega^2\right)^2+\beta^2\omega^2},\quad(2)$$

where γ and β are the damping factors,

$$\omega_p=\sqrt{\frac{N_v e^2}{\varepsilon_0 m_c^*}},\quad(3)$$

$$\omega_1^2=\omega_0^2-\frac{N_v e^2}{3\varepsilon_0 m_c^*},\quad(4)$$

where N_v is the carrier concentration, e the elementary charge, ε_0 the vacuum permittivity, and m_c^* the effective electron mass in conduction band. Based on the above equations, the transmittance spectrum can be calculated by the characteristic matrix used in thin film optics. The basic matrix can be described as[7]

$$\begin{bmatrix} B \\ C \end{bmatrix}=\prod_{j=1}^{k}\begin{bmatrix}\cos\delta_j & \frac{\rm i}{\eta_j}\sin\delta_j \\ {\rm i}\eta_j\sin\delta_j & \cos\delta_j\end{bmatrix}\begin{bmatrix}1\\ \eta_{k+1}\end{bmatrix},\quad(5)$$

where $\delta_j=\frac{2\pi}{\lambda}d_j$ is the phase thickness of the jth layer, d_j is the physical thickness of the jth layer, and η_j is the admittance of the material in the jth layer, $Y=B/C$

equals the admittance of the total thin film stack. One could then determine the transmission spectrum of the device as

$$T = \frac{4\eta_0 Re\left(\eta_m\right)}{\left(\eta_0 B + C\right)\left(\eta_0 B + C\right)^*}, \quad (6)$$

where η_0 and η_m are the admittance and exit of incident indium, respectively. The asterisk equals the complex conjugate. According to the Kramers-Kronig relation, the optical constant can be defined as

$$\varepsilon_1\left(\omega\right) = n^2 - k^2, \quad (7)$$

$$\varepsilon_2\left(\omega\right) = 2nk. \quad (8)$$

Substituting Eqs. (7) and (8) into Eq. (6), we can calculate the simulated transmittance. By the least square method, one can simply fit the experimental spectrum with the simulated transmittance based on the model used in Eqs. (1) and (2).

The measurement and the simulation of the ITO transmittance are plotted in Fig. 1. The dashed curve is the measured transmittance and the solid curve is the fitting data by the above dispersion model in the 300–1500 nm wavelength range. The two curves are in exact agreement, which proves that the dispersion model comprising the Lorentz oscillator and Drude free-electron component can be used to describe the ITO optical property very well from ultraviolet (UV) to near infrared (NIR) range. In this study, the simulated thickness for ITO film is 80.2 nm and the sheet resistance is 30 Ω/□ , measured by the four-point method. The fitted optical constant (refractive index n and extinction coefficient k) is shown in Fig. 2.

To obtain the optimized parameters for ITO film by reactive magnetron sputtering, we performed the experiments and defined the power, oxygen flow, argon flow, and temperature as the variables, keeping the sputtering time at 100 s for the test. As shown in Table 1, the sputtering power is changed from 3 to 4 kw. Oxygen flows were set at 2, 4, and 8 sccm. Argon flows were set at 30, 50, and 70 sccm. Temperature was varied from 250 °C to 350 °C.

The transmittance was measured by Lambda 950 from PerkinElmer in the 300–1500 nm range. The sheet resistance was measured by the four-point method. The simulated thickness was accomplished by the model described above. Thus, the conductivity of ITO film can be derived from

$$R_{\mathrm{s}} = \rho_0/d, \quad (9)$$

where R_{s} is the sheet resistance, d is the thickness of the film, and ρ_0 is the resistivity of the ITO film.

The final results are listed in Table 2, and the fitting results for n and k are shown in Fig. 3. Considering the low absorption and resistivity in the visible range, which is the case for our application, the parameters of

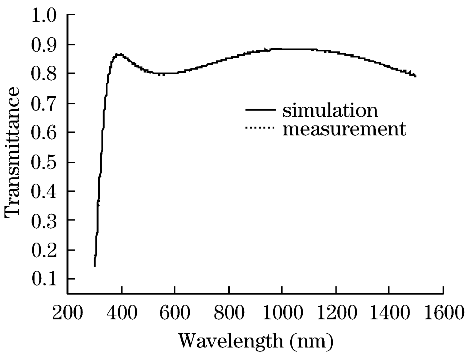

Fig. 1. Transmittance comparison of the simulation and the measurement for ITO film.

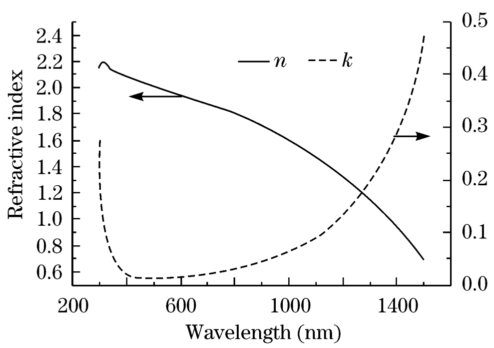

Fig. 2. Simulated optical constant of ITO film.

Table 1. Design of Experiments for Optimized Parameters of Sputtering ITO Film

No.	Power (kw)	Oxygen (sccm)	Argon (sccm)	Temperature (°C)
1	3	2	30	350
2	3	4	50	300
3	3	8	70	250
4	4	2	50	250
5	4	4	70	350
6	4	8	30	300

Table 2. Resistivity and Extinction Coefficient of ITO Film

No.	Sheet Resistance (Ω/□)	Thickness (nm)	Resistivity ($\times 10^{-4}$ Ω·cm)	Extinction Coefficient at 550 nm ($\times 10^{-2}$)
1	142	12.8	1.82	1.70
2	163	10	1.63	1.57
3	320	7.8	2.50	2.12
4	200	16.8	3.36	1.75
5	106	12.7	1.35	1.47
6	390	10	3.90	1.61

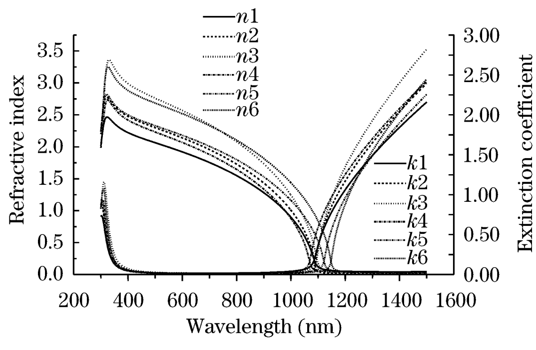

Fig. 3. Comparison on the simulated optical constant of ITO film from six test runs.

Table 3. Experimental Data for Sheet Resistance and Transmittance of ITO Film

No.	Thickness (nm)	Sheet Resistance (Ω/□)	Transmittance in the 420–670 nm Range (%)
1	11.8	132	88.40
2	25.5	70	81.91
3	86.9	30	81.08
4	122.6	17	87.40
5	182.4	12.5	84.73

test run No. 5 is the optimum, which has low extinction coefficient of 1.47×10^{-2} at 550 nm and the low resistivity of 1.35×10^{-4} Ω·cm compared with other parameters.

It is known that the carrier concentration of ITO film becomes higher as the thickness increases, which results in good conductivity. Meanwhile, absorption becomes higher and the optical transmittance decreases. Therefore, the tradeoff between low resistivity and high transmittance always has to be considered.

To investigate the relationship between sheet resistance and optical transmittance of ITO film, five test runs were performed with different film thicknesses. The results are shown in Table 3. By varying the sputtering time, different thicknesses of ITO film can be achieved. The average transmittance was measured with average of 420–670 nm. The sheet resistance was measured by the four-point method. The thickness listed in Table 3 is the fitting result.

According to the test results, sheet resistance decreases as the thickness of ITO film increases; however, average transmittance in the 420–670 nm range does not rise continuously when thickness varies from 122.6 to 86.9 nm. This is caused by the spectrum ripples in the calculated range, which affects the final average value. However, as the thickness of ITO film decreases to 11.8 nm, a high average transmittance of 88.4% can be achieved in the 420–670 nm range.

Based on the above basic works for the ITO film, we can start the design for ASAR coating. The normal requirement for such a coating is to achieve low absorption and low reflectivity in the visible range. As mentioned above, in addition to ITO, the other two materials used were Nb_2O_5 and SiO_2. In our study, all the layers were deposited using the reactive magnetron sputtering. The four-layer thin film stack was designed as substrate|Nb_2O_5|SiO_2|Nb_2O_5|ITO. Generally, to make a good antireflection (AR) coating, SiO_2 is added to improve the optical performance as the outermost layer; therefore, one SiO_2 layer was added on the whole stack to decrease reflectivity. The substrate used was Corning Eagle XG glass.

Figure 4 shows the actual spectra by the measurement of reflectance and transmittance. The sample was

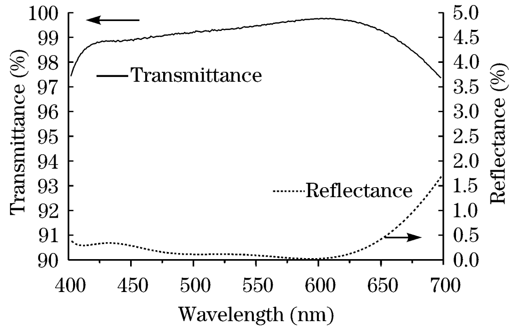

Fig. 4. Measured transmittance and reflectance of ASAR coating from 400 to 700 nm.

coated on both sides with ASAR by reactive magnetron sputtering. The sheet resistance measured by four-point method is approximately 1000 Ω/□, which meets the requirement for the static prevention on the surface of component itself. From the test result, the average reflectance can achieve 0.19% and the final transmittance after double-sided ASAR coating can reach 99.2% in the 420–670 nm visible range.

In conclusion, ASAR coating with ITO film is developed by reactive magnetron sputtering in Balzers MSP^{TM} platform. The process parameters for ITO film are investigated for the purpose of achieving low absorption and low resistivity in the visible range. The optical constant of ITO film is modeled by combining a single Lorentz oscillator and a Drude free-electron component in the range of 300–1500 nm, which fits very well with the experimental data. The ASAR coating is designed using ITO based on the optimized parameters and implemented by reactive magnetron sputtering. The experimental data shows that the ASAR coating with ITO can achieve the requirement for both high transmittance and low resistivity.

References

1. H. P. Lobl, M. Huppertz, and D. Mergel, Surf. Coatings Tech. **82,** 90 (1996).
2. N. G. Marechal and R. S. Blacker, "Anti-static, anti-reflection coating" Patent US 6852406 (2005).
3. J. F. Smith, A. J. Aronson, D. Chen, and W. H. Class, Thin Solid Films **72,** 469 (1980).
4. J. George and C. S . Menon, Surf. Coatings Technol. **132,** 45 (2000).
5. C. May and J. Strümpfel, Thin Solid Films **351,** 48 (1999).
6. M. Losurdo, M. Giangregorio, P. Capezzuto, G. Bruno, R. De Rosa, F. Roca, C. Summonte, J. Plá, and R. Rizzoli, J. Vac. Sci. Technol. A **20,** 37 (2002).
7. H. A. Macleod, Thin-film optical filters (Adam Hilger Ltd., Bristol, 1986).