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Effects of gas environment on electronic and optical properties of amorphous indium zinc tin oxide thin films

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The electronic and optical properties of indium zinc tin oxide (IZTO) thin films grown under different gas environments were investigated by means of x-ray photoelectron spectroscopy and reflection electron energy loss spectroscopy (REELS). REELS spectra revealed that IZTO thin films under argon mixed with oxygen had band gaps of 3.07 eV before annealing and 3.46 eV after annealing at 350 °C in air. Meanwhile, the band gap for IZTO thin film grown under oxygen mixed with water and annealed at 350 °C in air was 3.26 eV. Band gaps obtained from REELS spectra are consistent with the optical band gaps obtained using UV-spectrometry. The REELS spectra were quantitatively analyzed based on comparison of the effective cross section for inelastic electron scattering in the REELS experiment to determine the dielectric function and transmittance of the IZTO thin films. It was found that amorphous IZTO films grown under argon mixed with oxygen followed by annealing at 350 °C exhibit higher optical transmittance in the visible-light region, higher carrier mobility, and a high on–off current ratio. © 2013 American Vacuum Society.

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I. INTRODUCTION

ZnO-based materials have applications as transparent conducting oxides (TCO) that serve as active channel materials in thin film transistors (TFTs). This is due to their higher carrier mobility and transparency in the visible light region compared to the conventional TFTs, which are based on hydrogenated amorphous silicon (a-Si:H) having a carrier mobility of less than 1 cm² V⁻¹ s⁻¹. High performance has been reported for ZnO-based materials used as active layers in TFTs with field-effect carrier mobilities higher than 10 cm² V⁻¹ s⁻¹, using amorphous oxide semiconductors such as indium zinc oxide, zinc tin oxide, gallium zinc oxide, indium gallium zinc oxide (IGZO), and hafnium indium zinc oxide. IGZO is an especially promising channel material due to its large field-effect mobility (>10 cm² V⁻¹ s⁻¹), moderate device stability, high uniformity, and excellent electrical properties. In order to obtain high-quality TFTs, many attempts have been made to replace Ga³⁺ with Sn⁴⁺, as it generates carrier concentration more effectively, has higher electron negativity and more valence electrons than Ga³⁺. Nevertheless, Kim et al. reported that the carrier mobilities of indium zinc tin oxide (IZTO) thin films are 10 times higher than IGZO, and IZTO has a current on/off ratio of >10⁵ for films annealed at a relatively low temperatures (400 °C). A number of studies have found that amorphous IZTO has excellent optical transmission in the visible light region and it could have good electrical conductivity by controlling the chemical bonding composition of IZTO thin films.

A recent study reported that the properties of IZTO thin films could be improved by changing the environment during film deposition. Ko et al. reported that the electrical properties were enhanced by optimizing the oxygen partial pressure. They showed that the IZTO films, made by pulsed laser DC magnetron sputtering at 2% oxygen partial pressure, yielded an electrical resistivity of 5.1 × 10⁻⁴ Ω cm. In other study, Kim et al. found that IZTO films prepared by facing target sputtering system in oxygen with a gas flow rate of 0.4 sccm (standard cubic centimeters per minute) had a low resistivity value of 4.99 × 10⁻⁴ Ω cm and they confirmed that high performance IZTO TFTs could be realized at room temperature (RT). Argon gas is chemically inactive, and argon mixed with oxygen environment is often used to improve the electrical properties of ZnO based TCO materials. Furthermore, studies on the effect of water adsorption onto the surface of IGZO TFTs show that water may act as an acceptor-like trap site and lead to degradation of IGZO transistors. However, so far there are no detailed reports on the effect of gas environments (such as water or argon mixed with oxygen) on the electronic and optical properties of IZTO thin films.

Reflection electron energy loss spectroscopy (REELS) is very useful for examining the electronic structure of ultrathin gate oxide films, dielectric materials, polymers, and transparent conducting materials because the low-energy-loss region reflects the valence and conduction band structures of...
samples. REELS spectra consists of elastic and inelastic parts, but we consider the inelastic part only because the energy loss of electrons can be used to calculate the dielectric properties for thin films. The excitation of outer-shell electrons may produce energy losses of less than 100 eV, which provides a straightforward way to determine the dielectric function \( \varepsilon(k, \omega) \) because it determines the response of the material to a moving electron. The quantitative analysis of REELS spectra gives us direct information on the optical parameters (derived from the dielectric function). In this work, we investigated the effect of gas environment used during preparation on the band gaps and transmission coefficients of IZTO thin films by utilizing REELS and X-ray photoelectron spectroscopy (XPS) methods.

II. EXPERIMENT METHODS
IZTO thin films were deposited on glass substrates at room temperature by RF magnetron sputtering with a RF power of 200 W. The target composition ratio of In:Zn:Sn in an IZTO thin film was 15:51:34. The physical thickness of each IZTO thin film was 35 nm, which was measured using the cross sectional transmission electron microscopy. Three samples were prepared using different conditions. Sample 1 (denoted as IZTO-I) was deposited in argon mixed with oxygen (Ar:O\(_2\) = 20:80) environment at RT without annealing. Sample 2 (denoted as IZTO-II) was grown in the same environment as sample 1, and then annealed at 350 °C for an hour in ambient air. Sample 3 (denoted as IZTO-III) was deposited by wet annealing in water mixed with oxygen (H\(_2\)O + O\(_2\)) environment with a volumetric flow rate of 2 slm (standard liters per minute), and annealed at 350 °C for an hour. In order to make IZTO thin-film transistors, the gate insulators were made using silicon nitrides, which were deposited by plasma-enhanced chemical deposition at 350 °C. Molybdenum was used for the source and drain electrodes. The studied device has the dimension of W/L = 90 μm/8 μm. All electrical properties were measured using a Keithley 4200 semiconductor parameter analyzer. The voltages applied between the gate and the source (V\(_{GS}\)) and between the drain and the source were –20 V and 10 V, respectively. XPS and REELS spectra were obtained by using a VG ESCALAB 210 equipment. XPS measurements were performed using a Mg K\(_\alpha\) x-ray source and the energy analyzer pass energy of 20 eV. The binding energies were referenced to the C 1s peak of hydrocarbon contamination at 284.5 eV. The REELS spectra were measured with primary electron energies of 1.0, 1.5, and 2.0 keV and with a constant analyzer pass energy of 20 eV. The inelastic mean free path values for IZTO thin films for 1.5, and 2 keV energy electrons were 1.6, 2.2, and 2.8 nm, respectively, which were estimated through a quantitative analysis of the REELS data. The incident and take-off angles of electrons from the surface normal were 55° and 0°, respectively. The full width at half maximum of the elastic peak was 0.8 eV. The transmittance spectra of the IZTO thin films were measured by GENESYS 6 model from Thermo Electron Corporation in the wavelength range of 300–1000 nm at RT at increments of 0.1 nm.

III. QUANTITATIVE ANALYSIS OF REELS SPECTRA
A. Inelastic scattering cross section and energy loss function for electrons
We quantified the REELS spectra by using the Tougaard–Yubero QUEELS-\(\varepsilon(k, \omega)\)-REELS software package. Here, we calculated the optical parameters of the IZTO thin films in different gas environments. The model adopted in Tougaard–Yubero QUEELS-\(\varepsilon(k, \omega)\)-REELS software package removes multiple scattering electrons from the REELS spectra and determines an effective single-scattering cross section for inelastic electron scattering, \(K_{\text{exp}}(\hbar\omega)\), times the corresponding inelastic mean free path \(\lambda\), in the form of \(\lambda K_{\text{exp}}(\hbar\omega)\). The main purpose of this model is to interpret the effective inelastic cross section in terms of the dielectric function, \(\varepsilon(k, \omega)\). The dielectric function is the response of the material to a moving electron, from which we calculate the energy loss function (ELF) \(\text{Im}(-1/\varepsilon)\). Drude–Lindhard type oscillators were used to parameterize the ELF of IZTO films as follows:

\[
\text{Im}\left\{ -\frac{1}{\varepsilon(k, \omega)} \right\} = \theta(\hbar\omega - E_g) \\
\times \sum_{i=1}^{n} A_i \gamma_i \hbar\omega_0 \left( \frac{\hbar^2 c^2 \gamma_i^2}{\hbar^2 \omega_0^2 + \hbar^2 \omega^2} \right)^2 + \chi^2 \hbar^2 \omega^2,
\]

in which the dispersion relation is given by

\[
\hbar\omega_{0}\hbar\omega = \hbar\omega_0 + \chi \frac{\hbar^2 k^2}{2m}.
\]

Here \(A_i\), \(\gamma_i\), \(\hbar\omega_0\), and \(\chi\) are the oscillator strength, the damping coefficient, the excitation energy, and the momentum dispersion coefficient of the \(i\)th oscillator, respectively. The \(\hbar k\) is the momentum transferred from the REELS electron to the solid. The step function \(\theta(\hbar\omega - E_g)\) is included to simulate a possible energy band gap, \(E_g\), estimated from the onset of the energy loss in the REELS spectrum. The details of the simulated oscillators in this model were described in our previous study. The oscillator strengths are adjusted so as to \(\varepsilon(k, \omega)\) fulfilling the well-established Kramer–Kronig sum rule:

\[
\frac{2}{\pi} \int_0^\infty \frac{\text{Im}\left\{ \frac{1}{\varepsilon(k, \omega)} \right\}}{\varepsilon(k, \omega)} \, d(\hbar\omega) = 1 - \frac{1}{n^2}.
\]

Note that \(n\) is the refraction index in the static limit. The refraction index of IZTO thin films is 2.0. Here, the refractive index accuracy does not significantly affect the calculation result.

B. Optical properties from REELS spectra
The optical parameters of IZTO thin films grown in different gas environments are described by the complex dielectric function when the electron travels through the medium. Using Eq. (1), we can perform the Kramer–Kronig transformation of \(\text{Im}\{1/\varepsilon\}\) to obtain the real part \(\text{Re}\{1/\varepsilon\}\) of the
the reciprocal of the complex dielectric function. The real and imaginary parts of the dielectric function can be expressed by the following formula:

\[
\varepsilon_1 = \frac{\text{Re}\{1/\varepsilon\}}{\left(\text{Re}\{1/\varepsilon\}\right)^2 + \left(\text{Im}\{1/\varepsilon\}\right)^2},
\]

\[\varepsilon_2 = \frac{\text{Im}\{1/\varepsilon\}}{\left(\text{Re}\{1/\varepsilon\}\right)^2 + \left(\text{Im}\{1/\varepsilon\}\right)^2}.\]

The refractive index \(n\) and extinction coefficient \(k\) are determined from the dielectric function by using the relations,

\[n = \sqrt{\frac{1}{2} \left( \sqrt{\varepsilon_1^2 + \varepsilon_2^2 + \varepsilon_1} \right)} \quad \text{and} \]

\[k = \sqrt{\frac{1}{2} \left( \sqrt{\varepsilon_1^2 + \varepsilon_2^2 - \varepsilon_1} \right)}.\]

We determined the calculated transmission coefficient \(T\) from the relation \(R + T + \mu = 1\), where \(R\) is the reflection coefficient given by the relation,

\[R = \frac{(n-1)^2 + k^2}{(n+1)^2 + k^2},\]

and \(\mu\) is the absorption coefficient related to the extinction coefficient \(k\) as follows:

\[\mu = 0.82 \hbar \omega k.\]

Here, \(\hbar \omega\) is the energy loss.

**IV. RESULTS AND DISCUSSION**

The chemical states of the components of IZTO thin films on a glass substrate were investigated by means of XPS for the samples prepared in different gas environments. Figure 1(a) shows that the binding energy of the In 3d electrons of 444.4 eV in all IZTO thin films corresponds to an In-O bond. Figure 1(b) shows that the binding energy of Zn 2p3/2 electrons of 1021.9 eV in all IZTO thin films corresponds to a Zn-O bond. Figure 1(c) shows that the binding energy of Sn 3d5/2 electrons of 486.1 eV in all IZTO thin films corresponds to a Sn-O bond.

The REELS spectra of IZTO thin films on glass substrates were shown in Fig. 2. As shown in Fig. 2(a), the plasmon loss in each IZTO thin film generates a broad peak in the energy loss spectrum away from the elastic peak at 0 eV, and it appears below the electron-hole interband transition. The

\[\text{O}_2^+ \rightarrow \frac{1}{2}\text{O}_2 + V^+ + 2e^- ,\]

where \(\text{O}_2\) is lost from the oxide sublattice (\(\text{O}^2^-\)) to create a double oxygen vacancy (\(V^+\)) and two electrons. It is shown that the intensity of the higher binding energy component of the O 1s peak in IZTO-II is greater than that in the case of IZTO-I. Therefore, the electron density of an annealed IZTO film is expected to be much higher than that of an unannealed IZTO film. Another peak from O 1s appeared at 532.3 eV in the IZTO-III film, but it did not appear in the presence of other gas environments. Szördényi et al. suggested that this peak could be attributed to the presence of a hydroxide bond (O-H) caused by adsorbed water on the surface of a thin film. Hence, water still remains on the surface of a IZTO-III thin film even after annealing at 350 °C. The composition ratios of In:Zn:Sn are 17:51:32, 16:53:31, 18:54:28 for IZTO-I, IZTO-II, and IZTO-III, respectively, estimated by the quantitative analysis of the XPS spectra. The composition ratios of the IZTO thin films are nearly the same as the target composition ratios. We carried out x-ray measurements on all IZTO thin films to investigate the diffraction peaks of the films (which is not shown here), and our results imply that all samples including those annealed at 350 °C are amorphous.

The REELS spectra of IZTO thin films on glass substrates are shown in Fig. 2. As shown in Fig. 2(a), the plasmon loss in each IZTO thin film generates a broad peak in the energy loss spectrum away from the elastic peak at 0 eV, and it appears below the electron-hole interband transition. The

\[\text{O}_2^+ \rightarrow \frac{1}{2}\text{O}_2 + V^+ + 2e^- ,\]
bulk plasmon peaks of the IZTO-II thin film are located around 21.3 eV and shifted to lower energy by 0.8 and 2.0 eV for IZTO-I and IZTO-III thin films, respectively. We made use of REELS spectra to estimate the band gaps of the IZTO thin films (details of the method are described in our previous work). The onset values of the loss peak correspond to the band gap. The measured band gap of the IZTO-I thin film was 3.07 eV within an uncertainty of 0.1 eV. The band gap was increased to 3.46 and 3.26 eV for IZTO-II and IZTO-III thin films, respectively. The band gaps measured by using REELS were compared with the optical band gaps obtained using a UV-spectrometer. The optical band gaps were calculated on the basis of the optical spectral absorption, and they can be determined by extrapolating the best fit line between \((\alpha h \nu)^2\) and \(h \nu\), where \(\alpha\), \(h\), and \(\nu\) are the absorption coefficient, the Planck’s constant, and the frequency of incident photons, respectively. As shown in Fig. 2(b), the obtained optical band gaps of IZTO thin films are 3.09, 3.45, and 3.28 eV for IZTO-I, IZTO-II, and IZTO-III films, respectively. The values of optical band gaps are consistent with those determined by the REELS spectra analyses. The results from IZTO-II and IZTO-III indicate that the band gap values of IZTO thin films change with preparation with differing gas environments. Notice also that the band gap values of IZTO-I and IZTO-II grown in the same gas environment are different before and after annealing. The shift of the band gap can be explained by the Burstein–Moss Effect, which caused a shift of the band gap due to much higher electron density and the increase in the filling states of the conduction band in the IZTO films. Therefore, the Fermi level in the conduction band increases and leads to an energy band shift effect.

The I-V characteristic curves of IZTO TFTs for IZTO-II and IZTO-III are shown in Fig. 3. It shows that the on–off current ratio of IZTO-II is higher than 10^8. The off current is less than 10^{-12} A, and the turn-on voltage is approximately -2.1 V. The threshold voltage \(V\text{th} \text{(a)}\) in the linear region and the carrier mobility of IZTO-II are 1.8 V and 21.39 cm^2 V^{-1} s^{-1}, respectively. In contrast, an on–off current ratio greater than 10^10 and an off current of less than 10^{-13} were observed for IZTO-III, and the turn-on voltage was approximately -3.1 V as can be seen in Fig. 3(b). The carrier mobility and the threshold voltage of IZTO-III were 23.46 cm^2 V^{-1} s^{-1} and 1.2 V, respectively. The very small difference in the carrier mobility between IZTO-II and IZTO-III films implies that the mobility of IZTO is not greatly affected by the gas environments.

Figure 4 shows the experimental values of the \(\lambda K_{\text{exp}}\) inelastic cross section of IZTO thin films, which were obtained from the REELS spectra analysis, and they are compared with \(\lambda K_{\text{th}}\) values for model spectrum, which were calculated by utilizing the QUEELS-e(k,ω)-REELS in differing gas. The experimental inelastic cross section was fitted with

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**Fig. 2.** (Color online) (a) Reflection electron energy loss spectra with the primary energy of 1.5 keV. (b) Plot of \((\alpha h \nu)^2\) versus \(h \nu\) of IZTO thin films in different gas environments.

**Fig. 3.** (Color online) Drain current \(I_{ds}\) versus gate voltage \(V_g\) for the transfer curves of IZTO thin films in different gas environments.
the parameters of $A_i$, $\gamma_i$, $\hbar \omega_i$, and $\chi_i$ by trial-and-error procedures at 1.0, 1.5, and 2.0 keV. The results show good agreement between the cross sections obtained from the model spectrum by fitting it to the experimental data and the experimental cross sections for all IZTO films for different primary energies. The oscillators for each IZTO thin film are tabulated in Table I.

The ELFs $\text{Im}[1/e]$ and surface energy loss functions (SELFs) $\text{Im}[-1/(1+e)]$ for IZTO prepared in different gas environments are plotted in Fig. 5. The ELF of IZTO-I has five oscillators at 5.5, 8.5, 15.0, 20.5, and 34.0 eV (Table I). The energy position of the second, third, and fifth oscillators for IZTO-I and IZTO-II are nearly the same, but the strength of IZTO-II for the fifth oscillator is larger (by about 1.2 eV) than that of IZTO-I. The first and fourth oscillators for IZTO-II are shifted to higher energy loss positions at 7.0 and 21.0 eV, respectively. The fourth and fifth oscillators for IZTO-III are in the energy loss position of 20.0 and 30.0 eV, respectively, much lower than those of IZTO-I and IZTO-II. The energy position of the first oscillator of IZTO-III is the

| TABLE I. Parameters that give the best fit to experimental cross sections for inelastic electron scattering derived at 1.0, 1.5, and 2.0 keV primary energies for the energy loss functions of IZTO thin films in different gas environments. |
|---|---|---|---|
| Gas environments | $i$ | $\hbar \omega_i$ (eV) | $A_i$ (eV$^2$) | $\gamma_i$ (eV) |
| IZTO-I-Ar + O$_2$ without annealing | 1 | 5.5 | 1.2 | 5 |
| $\left(E_g = 3.07 \text{ eV}\right)$ | 2 | 8.5 | 3.9 | 7 |
| $\left(\chi_i = 0.05\right)$ | 3 | 15.0 | 5.5 | 16 |
| | 4 | 20.5 | 356.5 | 14 |
| | 5 | 34.0 | 27.1 | 25 |
| IZTO-II-Ar + O$_2$ with annealing in air | 1 | 7.0 | 1.3 | 5 |
| $\left(E_g = 3.46 \text{ eV}\right)$ | 2 | 8.7 | 3.9 | 8 |
| $\left(\chi_i = 0.05\right)$ | 3 | 15.3 | 6.1 | 18 |
| | 4 | 21.0 | 364.5 | 14 |
| | 5 | 34.0 | 28.3 | 25 |
| IZTO-III-H$_2$O + O$_2$ with annealing | 1 | 5.5 | 1.3 | 5 |
| $\left(E_g = 3.26 \text{ eV}\right)$ | 2 | 8.5 | 3.6 | 10 |
| $\left(\chi_i = 0.05\right)$ | 3 | 14.8 | 4.9 | 19 |
| | 4 | 20.0 | 346.9 | 15 |
| | 5 | 30.0 | 44.3 | 25 |

FIG. 4. (Color online) Comparison of experimental inelastic cross section, $\lambda K_{exp}$ (solid line) obtained from REELS data with the model spectrum result, $\lambda K_{th}$ (circle) calculated by fitting it to the experiment with the simulated energy loss function for primary energies of 1.0, 1.5, and 2.0 keV.

FIG. 5. (Color online) ELFs and SELFs calculated for IZTO thin films by utilizing the parameters given in Table I.
same as that of IZTO-I. The intensity, peak position, and shape of SELFs are different from those of the ELFs for all samples grown in gas environments. The main peak of SELFs in all IZTO thin films is located at about 15 eV, which corresponds to the surface plasmon peak. Figure 6 shows the real part $\varepsilon_1$ and imaginary part $\varepsilon_2$ (corresponding to the absorption spectrum) of the dielectric functions of IZTO films. The main peaks of $\varepsilon_1$ for IZTO-I, IZTO-II, and IZTO-III thin films are at 4.7, 5.0, and 4.5 eV, respectively. Likewise, the main peaks of $\varepsilon_2$ for IZTO-I, IZTO-II, and IZTO-III thin films are at 5.5, 5.8, and 5.3 eV, respectively, as can be seen in Fig. 6.

Figure 7(a) shows the calculated transmittance values as a function of wavelength for IZTO thin films prepared in different gas environments. In the visible spectra region, they are approximately 88, 84, and 87% for IZTO-I, IZTO-II, and IZTO-III thin films, respectively. We measured the spectral transmittances of all IZTO thin films grown in differing gas environments using a UV-spectrometer with the wavelength ranging from 300 to 1000 nm to compare with the calculated transmittance. The spectral transmittances of all IZTO thin films are shown in Fig. 7(b). The IZTO-II thin film grown in argon mixed with oxygen environment followed by annealing showed an optical transmittance of over 83% in the visible light region. The optical transmittance of IZTO-III grown under water mixed with oxygen was 87%. The IZTO-I thin film grown under argon mixed with oxygen environment without annealing exhibited an optical transmittance over 89%. The measured transmission coefficients are consistent with the calculated transmission coefficients. As can be seen from the results, the difference in the transmittance for the two samples (IZTO-I and IZTO-II films) grown in the same gas is 6%. Similarly, the transmittance difference between IZTO-II and IZTO-III films grown in differing gas environments is 4%. Considering the uncertainty of 2% in the transmittance, we conclude that the annealing process and differing gas environments have a very little influence on the optical properties of IZTO films.

V. SUMMARY

The electronic and optical properties of IZTO thin films were investigated using XPS and REELS. XPS results showed that the IZTO thin films have a mixed metal and oxide phase. The REELS spectra show that the band gap values are strongly affected by the differing gas environment and the annealing processes. On the other hand, the transmission coefficients of IZTO thin films are not affected by gas environment. Films grown in gas environment can be a guide for growing IZTO thin films for the device applications that require TFTs with high field-effect mobility and good transparency. In summary, the quantitative analysis of REELS spectra enables us in straightforward way to obtain the dielectric function and transmission coefficient of the transparent thin films, and the validity of this calculation is confirmed with UV spectrometric measurements.

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