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Fabrication of Indium Gallium Zinc Oxide (IGZO) TFTs Using a Solution-Based Process

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Fabrication of Indium Gallium Zinc Oxide (IGZO) TFTs Using a Solution-Based Process

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Highly transparent (~90% in the visible region) indium gallium zinc oxide (IGZO) thin films were deposited using a spin coating process with a newly developed precursor solution. Acetonitrile was used as the solvent in the preparation of the metal halide precursor solution for the deposition of the IGZO thin films. Ethylene glycol was added to the solvent at four different volume ratios of acetonitrile to ethylene glycol to complement the chemical properties of acetonitrile in order to avoid the de-wetting phenomenon during the deposition process. The IGZO thin films were prepared at a stoichiometric molar ratio of 2:2:1 on the basis of the theoretical In₂O₃-Ga₂O₃-ZnO structure. The IGZO metal-oxide-semiconductor field-effect transistor (MOSFET), with a field-effect mobility (μ_{FE}) as high as 1.1 cm²/V s, a turn on voltage of 15.8 V, and a current on-to-off ratio greater than 10⁷, was successfully fabricated in this study. This low cost solution-based deposition process was applicable for the fabrication of transparent conducting oxide (TCO)-based devices.

Keywords Indium gallium zinc oxide (IGZO); solution based deposition; TFTs; thin film; transparent amorphous conducting oxide semiconductor

Introduction

Transparent amorphous conducting oxide semiconductors have attracted a great deal attention as the channel layer of thin film transistors (TFTs) because of their high performance in flexible TFTs that are fabricated on plastic substrates [1]. The mobilities of transparent amorphous conducting oxides are comparable to crystalline oxide semiconductors. Their energy band gaps are wide enough to exhibit good transparency in the visible spectral range. Transparent amorphous conducting oxide thin films have been studied for application in many areas, including organic

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light emitting diode (OLED) displays, large, ultra-high definition liquid crystal displays (LCDs), electrophoretic displays, electrochromic windows, electro-optical devices, gas sensors, and solar cells [2–4].

Currently, various Zn-based transparent conducting oxides, such as IGZO [1–3], ZnO [5], ZIO [6,7], GZO [4,8], SGZO [1], and IZTO [9], are important in the field of TFTs. Among these Zn-based oxides, amorphous In-Ga-Zn-O (IGZO) is a key semiconductor for transparent oxide TFTs because of its high channel mobility, high on-to-off ratio, low toxicity, excellent environmental stability and good optical transmittance [2,10]. A number of studies have recently developed IGZO thin films. Especially, Hosono *et al.* deposited an amorphous IGZO thin film onto a flexible substrate, and the field effect mobility of the IGZO thin films was greater than $10 \text{ cm}^2/\text{V} \cdot \text{s}$ [1].

Until recently, IGZO thin films were formed using pulsed laser deposition (PLD) [1,10–12], RF magnetron sputtering [2,13,14], direct current (DC) magnetron sputtering [3], inkjet printing [15] and spin coating [16–18]. The PLD, RF sputtering, and DC sputtering methods are all based on the vapor deposition process. These processes require sophisticated laser systems, expensive vacuum systems, or high temperature conditions. Additionally, environmentally toxic templates are often used for the synthesis of the inorganic materials that are used in these methods. Meanwhile, chemical solution deposition (CSD) methods, such as spin coating, have many advantages because of their low cost and low temperature processing nature. Additionally, CSD processes do not require sophisticated vacuum systems or expensive equipment [5,15]. Although the mobilities of the solution-based deposition methods are lower than the corresponding vapor phase depositions, the CSD processes are of great importance to the fabrication of amorphous TFTs.

In this study, IGZO thin films were synthesized using the spin coating method, and then effects of the solvent on the surface morphology and characteristics of the IGZO thin films were investigated. The IGZO metal-oxide-semiconductor field-effect transistors (MOSFET) were developed using the conventional TFT fabrication method, and their properties were characterized in order to verify their performance.

Experimental

For the IGZO thin film deposition method, a precursor solution was prepared by dissolving 0.03 M of indium chloride (InCl_3 , ALDRICH Inc.), 0.03 M of gallium chloride (GaCl_3 , ALDRICH Inc.), and 0.015 M of zinc chloride (ZnCl_2 , SIGMA-ALDRICH Inc.) into 30 ml of a mixture of acetonitrile (CH_3CN , SIGMA-ALDRICH Inc.) and ethylene glycol ($\text{C}_2\text{H}_6\text{O}_2$, SIGMA-ALDRICH Inc.) at room temperature. The stoichiometric molar ratio of In_2O_3 : Ga_2O_3 : ZnO for IGZO was 2:2:1 in this experiment. In order to investigate the effect of solvent on the properties of IGZO thin films, four different volume ratios (1:0, 3:1, 1:1, and 1:3) of acetonitrile to ethylene glycol were used in the solvent mixture. Both commercial microscope glasses (Fisher Scientific) and Si wafers containing thermally oxidized SiO_2 layers (1000 Å thickness) were used as the substrates, and the amorphous IGZO thin films were deposited using a spin coater (YS-100MD, Yooil engineering) at 4000 rpm for about 30 seconds. The substrates were ultrasonically cleaned with a 1 M sodium hydroxide (NaOH, Duksan) solution and then chemically cleaned with acetone, methanol, and deionized water (AMD), sequentially. The substrates were completely

dried using nitrogen gas prior to the deposition of the IGZO thin films. The as-deposited IGZO thin films were annealed for 1 hour at 600°C in air. The fabrication of the IGZO MOSFET device was completed through the thermal deposition of Al as both the source and drain electrodes using a shadow mask.

A scanning electron microscope (SEM, HITACHI S-4800) and an atomic force microscope (AFM, DI Instruments Nanoscope IIIa) were used to examine the surface morphology and the surface roughness of the prepared films, respectively. An X-ray diffraction spectrometer (XRD, Panalytical, MPD for thin film) was used to analyze the structure of the IGZO thin films. The optical properties, such as the transmittance and energy band gap, were measured using a UV-visible spectrophotometer (Ocean Optics Inc, USB 4000 optic spectrometer). The chemical composition of the IGZO thin films was analyzed using an X-ray photoelectron spectroscopy (XPS, ESCALAB 250 XPS spectrometer), and the device characterization was performed using a probe station (KEITHLEY 4200-SCS).

Results and Discussion

A. De-Wetting and Surface Morphology

Acetonitrile was used as the solvent during the preparation of the metal halide precursor solution for the deposition of the IGZO thin films because acetonitrile was capable of forming uniform and continuous metal halide thin films over a large area [7]. However, de-wetting traces were observed on the IGZO thin films when the precursors dissolved in pure acetonitrile solvent. During thin film formation, the de-wetting phenomenon is mainly influenced by the existence of defects on the solid surface, the de-wetting speed on the smooth surface, the contact angle of the solution droplet, and the surface energy related to the chemical and physical characteristics of the solution [19]. The de-wetting spots that were left on the deposited films were most likely caused by the de-wetting speed on the substrate because acetonitrile has a high volatility. Therefore, ethylene glycol was added to the solvent to complement the chemical properties of acetonitrile so that the de-wetting phenomenon could be avoided during the deposition process. Ethylene glycol has desirable physical properties, including a desirable viscosity and density. This study investigated the influence of ethylene glycol on the occurrence of de-wetting spots, and the optimum ethylene glycol content in the solvent mixture was also examined. In this work, ethylene glycol was mixed with acetonitrile at four different volume ratios from 0 to 3. Figure 1 shows the SEM images of the IGZO thin films that were deposited onto SiO₂/Si substrates at four different ethylene glycol concentrations. The content of ethylene glycol exerted a great influence on the morphology of the synthesized IGZO thin film. Figure 1(a) presents the SEM image of the IGZO thin film that was deposited using a pure acetonitrile solvent, and in this image, de-wetting spots were distinctly formed on the film surface. The IGZO films that were prepared with different volume ratios of acetonitrile and ethylene glycol are shown in Figures 1(b)~(d). The volume ratios of (b), (c), and (d) were 3:1, 1:1, and 1:3, respectively. In these figures, the de-wetting spots faded away as ethylene glycol was added to the solvent. The addition of ethylene glycol was believed to increase the viscosity and, consequently, positively influence the de-wetting speed of liquid film on the solid surface. In Figure 1(b), the film that was deposited at a ratio of 3:1 exhibited a uniform, smooth surface morphology without any de-wetting spots. Non uniform patterns were observed in Figures 1(c)

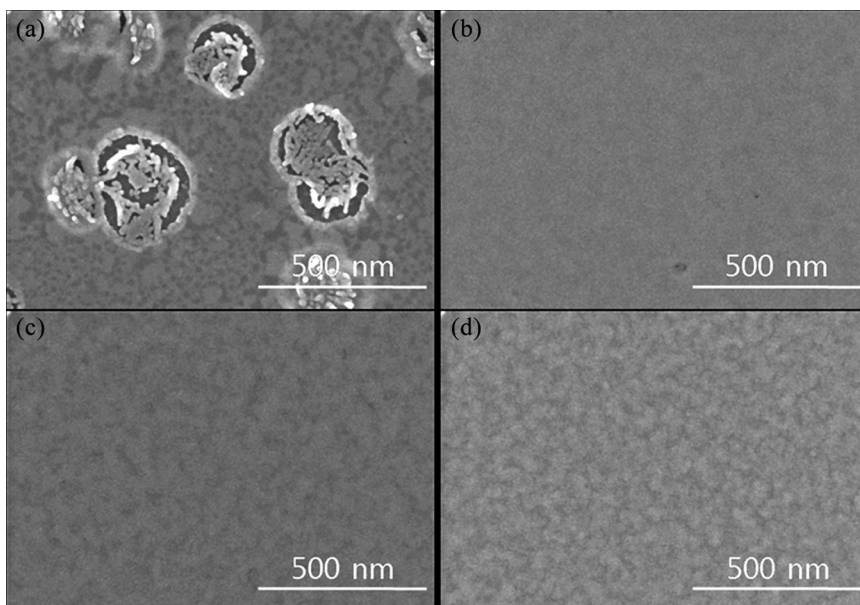


Figure 1. Scanning electron microscopy (SEM) images of the IGZO thin films that were deposited onto the SiO_2/Si wafers with different volume ratios of acetonitrile to ethylene glycol in the solvent mixtures: (a) 1:0, (b) 3:1, (c) 1:1, and (d) 1:3.

and (d) when more ethylene glycol was added to the mixture. The increased viscosity that was caused by the higher ethylene glycol content in the solution lowered the de-wetting speed and, subsequently, formed the patterns on the films. AFM was used to examine the surface roughness of the prepared IGZO films. Figure 2 shows the AFM images of the IGZO thin films that were deposited onto the SiO_2/Si substrates. The volume ratios of acetonitrile to ethylene glycol were 3:1, 1:1, and 1:3 in Figures 2(a)~(c), respectively. The surface roughness was 0.336, 0.487, and 0.537 nm in Figures 2(a)~(c), respectively. These results corresponded well to the surface morphologies that were determined using SEM. The SEM and AFM results

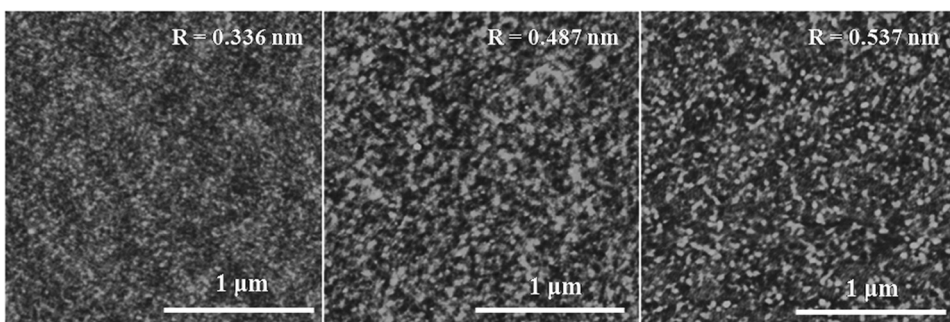


Figure 2. AFM images of the IGZO thin films that were deposited onto the SiO_2/Si wafer with different volume ratios of acetonitrile to ethylene glycol in the solvent: (a) 3:1, (b) 1:1, and (c) 3:1.

confirmed that the roughness of film increased as the volume ratio of acetonitrile to ethylene glycol increased in this experiment.

B. Optical Characteristics

The optical transmittance and optical band gap of the amorphous IGZO thin film were measured in the UV-to-near IR using a UV-visible spectrophotometer and are shown in Figure 1(b). The amorphous IGZO thin film exhibited a good surface morphology and roughness without any de-wetting traces and patterns. The optical band gap of the IGZO films was obtained by extrapolating the slope of the square root of $\alpha h\nu^2$ versus $h\nu$, which was plotted on the basis of the absorbance of UV-visible measurements. In Figure 3(a), an optical band gap of ~ 3.5 eV was

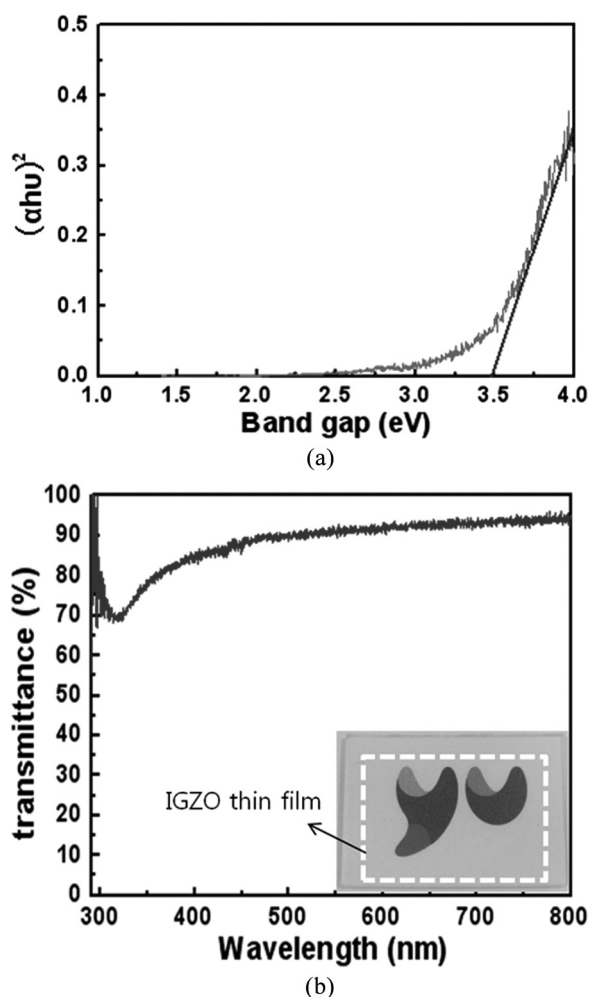


Figure 3. UV-visible absorption measurements for the spin coated IGZO thin film: (a) Optical transmission spectra for the 3:1 volume ratio of acetonitrile to ethylene glycol in the solvent mixtures (b) Optical band gap estimation.

observed for the IGZO thin film with a volume ratio of 3:1. The optical transmission spectrum of the IGZO film is shown in Figure 3(b). The inset image shows an optical image and the transmission of the real IGZO TFT. Transmittance of the film exceeded 90% in the visible region. A desirable optical band gap for the transparent IGZO TFT device is in the range of 3.1~3.3 eV [11], which is associated with the electronic band structure of In-Ga-Zn-O. Therefore, the amorphous IGZO thin film prepared in this study was a good candidate for the fabrication of transparent TFTs on flexible substrates.

C. Structural Characteristics

The IGZO thin film was characterized using an X-ray diffraction spectrometer (XRD) in order to determine its structural features. However, the formation of IGZO was difficult to determine through XRD analysis because no peaks were observed in the spectra. The XRD data indicated that the IGZO film was amorphous. The X-ray photoelectron spectroscopy (XPS) measurements were performed to determine structural and binding information of the IGZO thin film in Figure 1(b). The XPS data was measured in the spectral range of 430–1200 eV in Figure 4. In Figure 4(a) the observed binding energy peaks located at 444.3 and 452.05 eV corresponded to the electronic states of In $3d_{5/2}$ and In $3d_{3/2}$, respectively. The reference peaks for metallic In and pure In_2O_3 are found in the ranges of 443.6~444.0 eV and 444.3~444.8 eV, respectively [20,21]. In this study the observed binding energy of In

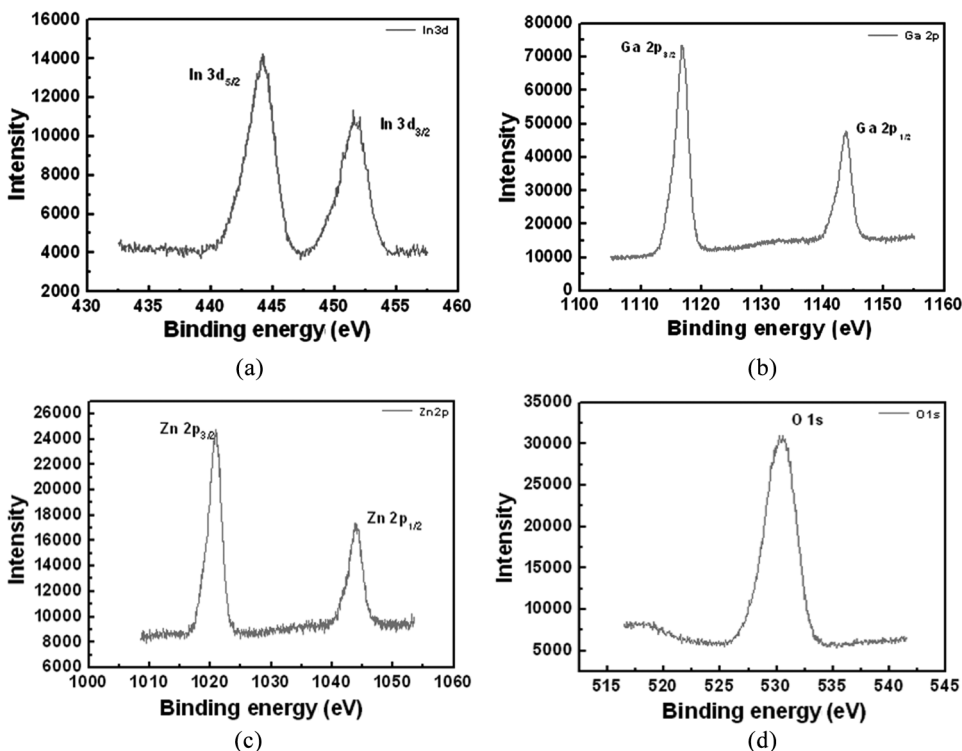


Figure 4. XPS spectra of the IGZO thin film that was annealed at 600°C.

$3d_{5/2}$ was consistent with the value of In_2O_3 from the literature. In Figure 4(b), the peaks at 1117.1 and 1144.15 eV corresponded to the electronic state of Ga $2p_{3/2}$ and Ga $2p_{1/2}$, respectively. The reference binding energy of metallic Ga was 1117.8 eV, and the binding energy of Ga_2O_3 was 1119.6 eV, which was about 2 eV higher than metallic Ga. The peak for Ga $2p_{3/2}$ was detected at a lower binding energy location than the peak for pure Ga_2O_3 in the reference [22]. The doped Ga was not completely converted to Ga_2O_3 and, consequently, the coexistence of the unreacted Ga with Ga_2O_3 in the film contributed to the shift in the Ga $2p_{3/2}$ peak to a lower binding energy. In Figure 4(c) the peaks located at 1022.2 and 1044.2 eV corresponded to the electronic states of Zn $2p_{3/2}$ and Zn $2p_{1/2}$, respectively. From the reference binding energy in the literature, the component with a binding energy of about 1022.2 eV corresponded to ZnO that was produced by the majority of Zn^{2+} within the oxygen deficient ZnO_{1-x} matrix [20,23]. In Figure 4(d) the peak detected at 531 eV in Figure 4(d) corresponded to the electronic state of O 1s. In general, the O 1s peaks appears in a broad range from 530 ~ 536 eV. Previous reports showed that the binding energy peaks centered at around 530.15 and 531.25 eV are attributed to the O^{2-} ions at the intrinsic sites and the O^{2-} ions in the oxygen deficient regions, respectively [20,23]. The XPS analysis showed that the prepared precursors were sufficiently converted to indium gallium zinc oxide on the thermally oxidized SiO_2/Si substrates.

D. Electrical Characteristics

A metal-oxide-semiconductor field-effect transistor (MOSFET) was fabricated using the IGZO thin film as the active channel layer. The IGZO thin film was deposited through spin coating onto an oxidized silicon substrate using a solution of InCl_3 , GaCl_3 and ZnCl_2 (2:2:1 molar ratio) in a mixture of acetonitrile and ethylene glycol (3:1 volume ratio). Figure 5(a) shows the cross-sectional schematic structure of the amorphous IGZO-MOSFET device that was fabricated in this work. In the MOSFET fabrication, a heavily boron (p+) doped silicon substrate served as the gate in the inverted-gate structure ($\text{Al}/\text{IGZO}/\text{SiO}_2/\text{Si}/\text{Au}$). The performance and device characteristics were investigated using a KEITHLEY 4200-SCS.

The IGZO MOSFET was tested by measuring the drain-to-source current (I_{DS}) as the drain-to-source voltage (V_{DS}) was swept from 0 to 40 V as the gate-to-source voltage (V_{GS}) was increased from -5 to 40 V in 10 V increments. Figure 5(b) is a plot of the drain-to-source current versus the drain-to-source voltage ($I_{DS} - V_{DS}$) output characteristics for the IGZO MOSFET with a channel width-to-length ratio of 10 (gate W/L dimensions of $1020\mu\text{m}/102\mu\text{m}$). The transistor exhibited good gate-modulated behavior with drain-current saturation. The MOSFET was characterized and its device parameters, including the field-effect mobility, the on-to-off ratio of I_{DS} , S.S. (subthreshold swing) and the turn-on voltage (V_{on}), were determined. In Figure 5(b), the transfer characteristics of the IGZO MOSFET were investigated in the saturation regime of the $I_{DS} - V_{GS}$ curves. A series of measurements were carried out for a V_{GS} from -5 to 40 V and a V_{DS} from 0.1 V to 20 V. The drain-to-source current was plotted on a logarithmic scale as a function of the gate-to-source voltage in the inset of Figure 5(c). The on/off ratio ($I_{on/off}$) of I_{DS} was greater than 10^7 , and the turn on voltage was 15.3 V. Figure 5 (c) presents the on-to-off ratio of I_{DS} for the transfer characteristics determination at $V_{DS} = 20$ V. The turn on voltage was used instead of the threshold voltage (V_{th}) in order to determine the minimum voltage that was required to induce the current in the active layer.

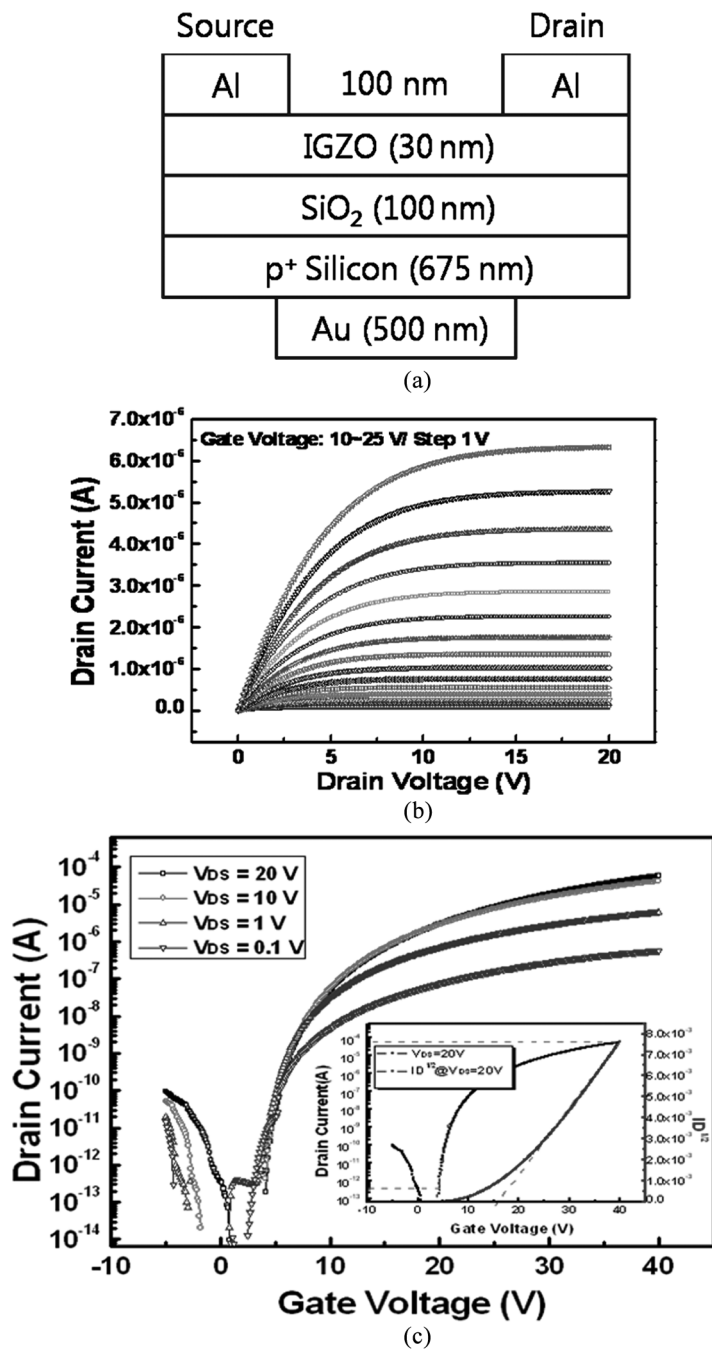


Figure 5. Characterization of the electrical properties for the a-IGZO-MOSFET: (a) a schematic cross-sectional view of the a-IGZO-MOSFET structure, (b) The drain current-drain voltage ($I_{DS} - V_{DS}$) output characteristics, (c) The drain current-gate voltage [$\text{Log}(I_{DS}) - V_{GS}$] transfer characteristics at $V_{DS} = 20 \text{ V}$.

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The value of the threshold voltage, which was extrapolated from the linear portion at $V_{DS} = 20$ V from the $I_{DS}^{1/2} - V_G$ plot, is sometimes arbitrary as discussed in previous literature [24]. The field effect mobility in the channel region determined the maximum on-current of the device at saturation. The maximum current at saturation was independent of V_{DS} and was expressed using Eq. (1).

$$I_{D,sat} = \mu C_{ox} \frac{W}{L} (V_G - V_T)^2 \quad (1)$$

In Eq. (1), μ is the field effect mobility, C_{ox} is the dielectric capacitance, W/L is the aspect ratio of the device, V_{GS} is the gate voltage, and V_{th} is the threshold voltage [6]. In the inset of Figure 5(c), the field mobility was obtained from the slope of the linear portion of a plot of the square root of I_{DS} on the right axis versus V_{GS} . Additionally, the extrapolation of the linear fit intersected the V_{GS} axis at the threshold voltage, V_{th} . The field-effect mobility (μ_{FE}) for this device was as high as $1.1 \text{ cm}^2/\text{V sec}$, and the device exhibited a good current on-to-off ($I_{on/off}$) ratio that was greater than 10^7 . The S.S. value of the device was 0.61 V/dec , and the turn on voltage was 15.8 V . These values were similar to the values that were previously reported for IGZO transistors that were deposited using solution-based processes [15–18,25].

Conclusions

The IGZO thin film was deposited through solution-based spin coating on a thermally oxidized SiO_2/Si substrate using a precursor solution containing InCl_3 , GaCl_3 , and ZnCl_2 (2:2:1 molar ratio) at various volume ratios of acetonitrile to ethylene glycol in the solvent solution. Ethylene glycol was added to the solvent to complement the chemical properties of acetonitrile in order to avoid the de-wetting phenomenon during the deposition process. In this study, the volume ratios of acetonitrile to ethylene glycol were 1:0, 3:1, 1:1, and 1:3. SEM and AFM confirmed that the best surface morphology and surface roughness results were obtained for the thin films at a solvent volume ratio of 3:1. In the visible region, the optical band gap and transmittance of the IGZO thin film were $\sim 3.5 \text{ eV}$ and $>90\%$, respectively. The structural characteristics of the IGZO thin film were confirmed using XPS analysis of the binding energies for the In-O, Ga-O, and Zn-O chemical bonds in the IGZO compound system. A metal-oxide-semiconductor field-effect transistor (MOSFET) was successfully fabricated as an active channel layer using the IGZO thin film. The field-effect mobility, turn on voltage, and current on-to-off ratio of the MOSFET were $\sim 1.1 \text{ cm}^2/\text{V s}$, 15.8 V , and $>10^7$, respectively, which were all higher than the previously reported values for IGZO transistors that were fabricated using solution-based deposition methods.

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