

Research Article

High-Performance Solution-Processed Amorphous InGaZnO Thin Film Transistors with a Metal–Organic Decomposition Method

Yingtao Xie ^{1,2}, Dongping Wang² and Hon Hang Fong ^{2,3}

¹Department of Electronic Engineering, Chongqing University of Posts and Telecommunications, 2 Chongwen Road, Chongqing 400065, China

²Department of Electronic Engineering, National Engineering Lab for TFT-LCD Key Materials and Technologies, Shanghai Jiao Tong University, 800 Dongchuan Road, Shanghai 200240, China

³Department of Materials Science and Engineering, Cornell University, Ithaca, NY 14853, USA

Correspondence should be addressed to Hon Hang Fong; hhfong@sjtu.edu.cn

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A facile solution process was introduced for the preparation of IGZO thin films via a metal–organic decomposition (MOD) method. The IGZO ink was synthesized by mixing the solutions of gallium acetylacetonate [$\text{Ga}(\text{C}_5\text{H}_7\text{O}_2)_3$], zinc acetylacetonate hydrate [$\text{Zn}(\text{C}_5\text{H}_7\text{O}_2)_2 \cdot x\text{H}_2\text{O}$] dissolved in ethanol, and indium acetylacetonate [$\text{In}(\text{C}_5\text{H}_7\text{O}_2)_3$] dissolved in tetrahydrofuran (THF). The deposited films by spin-coating were annealed at moderate process temperature ($\leq 500^\circ\text{C}$). The relationship between device performance and postannealing temperature was studied. The result demonstrated that mobility of IGZO TFT increased as the annealing temperature increased. Based on the analysis of O 1s statement, the annealing temperature can influence the number of oxygen vacancy to further affect the carrier concentration. In addition, the IGZO TFT devices with various Ga molar ratios were compared to demonstrate the influence of the Ga addition. The result demonstrated that the saturated mobilities (μ_e) decreased and V_{TH} shifted to positive voltage as the Ga molar ratio was increased. It is likely that Ga can offer stronger chemical bonds between metal and oxygen that reduced the concentration of free carriers and thus help reducing V_{TH} . As a result, the optimized performance of IGZO TFT with the mobility of $3.4 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ showed the MOD process was a promising approach.

1. Introduction

Due to high charge carrier mobility and high optical transparency, InGaZnO (IGZO) have attracted considerable interest from the electronic industry for application for thin film transistors (TFT) display backplanes [1–8]. Recently, the IGZO films are deposited typically by magnetron sputtering deposition, or/and chemical vapor deposition (CVD), or/and atomic layer deposition (ALD) processes. Although these processes can obtain the uniform films, it must require vacuum environment to add to cost of TFT display backplanes. Fortunately, solution process method is developed to deposit the films via spin-coating or/and printing to obtain the low-cost fabrication. More recently, many reports in the literature

have demonstrated TFT using IGZO thin film as the channel layer, which are prepared by solution deposition processes such as spin-coating or printing with sol-gel precursor solutions [9–16]. There are the two different synthetic sol-gel approaches [17]. The first approach is conventional processes, which consist of $\text{Mn}^+ + \text{X}^- + \text{stabilizer} + \text{ROH}$ or H_2O ($\text{X}^- = \text{Cl}^-, \text{CH}_3\text{COO}^-, \text{NO}_3^-,$ and so on). Its disadvantage is that oxide formation via conventional precursors based on metal hydroxide and/or alkoxide conversion is endothermic, requiring significant external energy input to form metal–O-metal lattices. Furthermore, conventional processes and precursors typically require high temperatures for oxidizing organic impurities to achieve phase-pure products. The second approach is combustion process, which consists of Mn^+

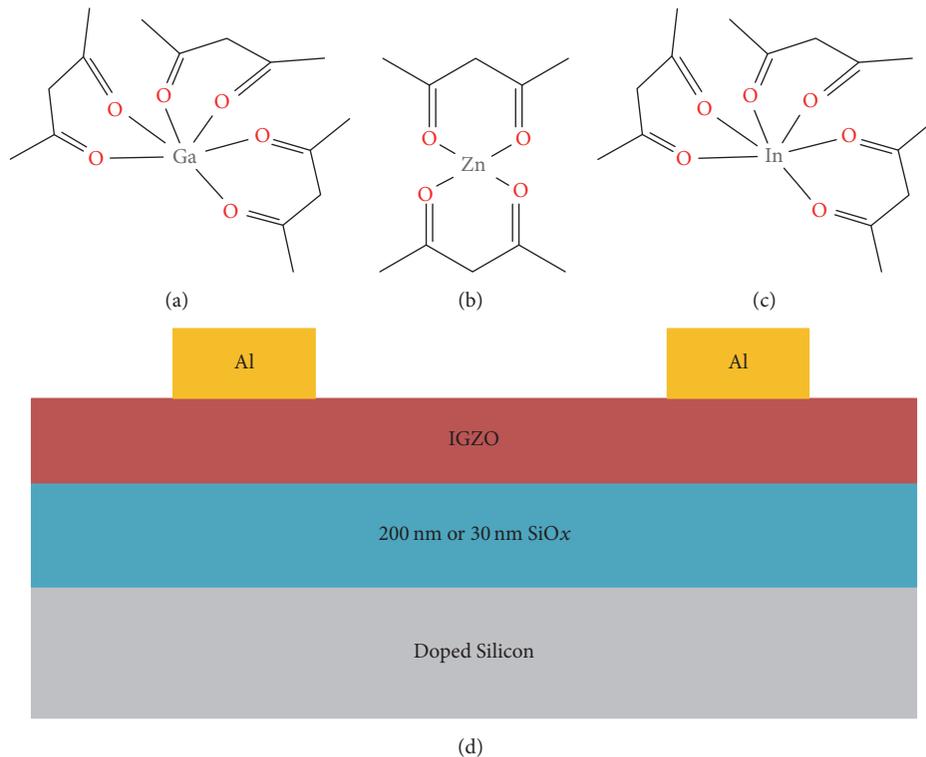


FIGURE 1: Chemical structures of (a) gallium acetylacetonate; (b) zinc acetylacetonate; (c) indium acetylacetonate; (d) the structure of bottom-gate top contact IGZO TFT device.

+ oxidizer + fuel (Oxidizer = NO_3^- , fuel = urea, glycine, citric acid). Combustion reactions with balanced redox chemistry are exothermic, not requiring external energy input once ignited. Although they overcome shortcoming of the first approach, these fuels' molecular combustion precursors anyway are formed in solution. It means that it is difficult to control the structure and morphology of the final product via postannealing. In this way Cu(II) acetylacetonates were used for obtaining controlled polymerization of the complex molecules [18].

In this work, we present a facile solution process for the preparation of IGZO thin films via a metal-organic decomposition (MOD) method, which are executed by all metal acetylacetonates precursors and postannealing treatment. The solution of metal acetylacetonates ($\text{In}(\text{acac})_3$, $\text{Zn}(\text{acac})_2$ and $\text{Ga}(\text{acac})_3$) is adopted as the precursor. The IGZO films are deposited by spin-coating and then annealed at moderate process temperature ($\leq 500^\circ\text{C}$) in air. We study the effect of postannealing time and temperature on performance of IGZO TFT devices. Results show that the field effect mobility is up to $3.4 \text{ cm}^2 \text{ V}^{-1} \text{ s}^{-1}$ under the optimized process condition. In addition, the IGZO TFT devices with various Ga molar ratios are compared to demonstrate the influence of the Ga addition. The result demonstrates that the saturated mobilities (μ_e) decrease and V_{TH} shifts to positive voltage as the Ga molar ratio is increased. The prepared IGZO thin films are confirmed to possess promising electric properties suitable for the TFT usage.

2. Experimental

2.1. IGZO Solution Preparing. The precursor solutions of IGZO were prepared by the following procedure. Firstly, metal precursors powders of gallium acetylacetonate [$\text{Ga}(\text{C}_5\text{H}_7\text{O}_2)_3$] ($\text{Ga}(\text{acac})_3$, 99% pure) and zinc acetylacetonate hydrate [$\text{Zn}(\text{C}_5\text{H}_7\text{O}_2)_2 \cdot x\text{H}_2\text{O}$] ($\text{Zn}(\text{acac})_2 \cdot x\text{H}_2\text{O}$, 99% pure) were dissolved in ethanol, and the powders of indium acetylacetonate [$\text{In}(\text{C}_5\text{H}_7\text{O}_2)_3$] ($\text{In}(\text{acac})_3$, 99% pure) were dissolved in tetrahydrofuran (THF) with molar concentrations of 0.05 M. The chemical structures of gallium acetylacetonate, zinc acetylacetonate, and indium acetylacetonate were shown in Figures 1(a)–1(c), respectively. Secondly, the three metal precursor solutions were mixed at different volume ratios to render the varied molar ratios of In, Ga, and Zn needed for this study. The precursor solutions were stirred for 24 hours at room temperature in N_2 atmosphere and then preheated at 65°C before using.

2.2. Oxide Thin Film Transistor Fabrication. Heavily doped Si(100) wafers were used as gate electrodes with a 30 nm or 200 nm thermally grown silicon dioxide layer as the gate dielectric. Figure 1(d) showed that the structure of the common gate top contact IGZO TFT. Substrates were cleaned by sonication in semiconductor grade acetone and isopropanol for 15 minutes in each solvent and then given a 2-minute 20 W O_2 plasma treatment. The prepared solutions were spin-coated onto the cleaned Si/SiO₂ substrates at a

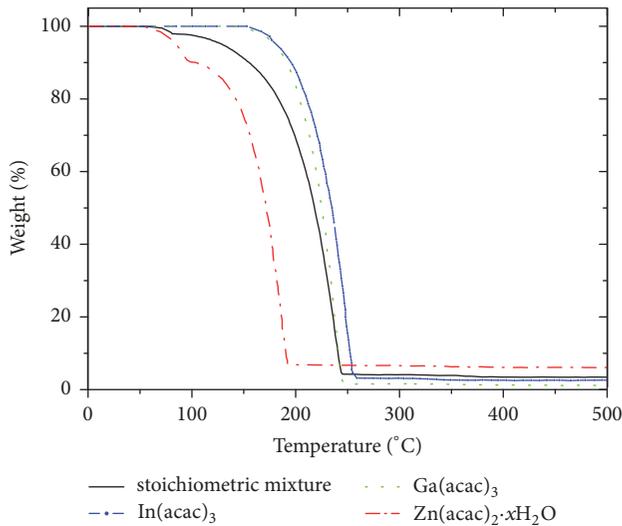


FIGURE 2: TGA results of starting materials used in this MOD process. The analyzed powder samples include In, Ga, Zn acetylacetonates, and the In : Ga : Zn = 1 : 1 : 1 stoichiometric mixture.

spinning rate of 5000 rpm. And then the IGZO film were annealed directly on a hot plate in air at temperatures in the range 340°C~500°C for 1 hour to decompose the organic components. At last, 60 nm Al was thermally evaporated to form the S/D contacts through a metal shadow mask which defined the transistor physical dimension ($L = 50\sim 150\ \mu\text{m}$, $W = 1000\ \mu\text{m}$). Electrical characteristics of all transistors were measured by Keithley 4200 SCS in air.

2.3. IGZO Film Characterization. Thermogravimetric analyses (TGA) were performed to investigate the thermal behavior of the stoichiometric mixture of starting materials. The typical thickness of IGZO films was measured around 30 nm by KLA-Tencor XP-200 step profiler. The surface morphology of the films was observed with an atomic force microscope (AFM) using tapping mode. The composition of as-pyrolyzed films was analysed by X-ray photoelectron spectroscopy (XPS, monochromatic Al K α ; 1486.6 eV) at selected take-off angles (TOA).

3. Results and Discussion

3.1. Effect of Thermal Process to IGZO Film. Thermal behavior of the starting materials was investigated by Thermogravimetric analyses (TGA). All three starting materials and the mixture with stoichiometry of a-IGZO (In : Ga : Zn = 1 : 1 : 1) were heated to 600°C at a rate of 10°C/min in standard air atmosphere. As shown in Figure 2, significant weight losses of In(acac)₃ and Ga(acac)₃ were observed starting around 200°C, while the weight of Zn(acac)₂·xH₂O started to sharply decrease at around 150°C after the removing of hydrated water at around 100°C. The significant weight losses of In : Ga : Zn (1 : 1 : 1) stoichiometric mixture at the range from 100°C to 250°C indicated the formation of metal-oxide bond and decomposition of the acetylacetonate ligands should occur with the rising of temperature. At over 250°C, no change in

weight loss was observed, which suggested that all organic components could be eliminated from the IGZO precursor film. From these results, the annealing temperature was set at 300°C.

To study the effect of thermal process to surface morphology, the as-deposited films were annealed in air via two types of heating processes. The first method was to anneal the films from room temperature at a slow heating rate of 5°C/min. The second method was to anneal the precursor films directly on a hotplate with the set temperatures to make a rapid temperature rising. Both processes were to heat the precursor films for 1 hour at setting temperatures. The crystal phases present in the as-annealed thin films were examined by XRD. After annealing in air for 1 hour at temperatures within our operation window (340°C to 500°C), all of the obtained IGZO thin films were confirmed to be amorphous.

The surface morphology of IGZO thin films annealed at 500°C via the slow and rapid heating was examined by AFM. As shown in Figure 3, flat and smooth surface could be obtained with the slower heating rate, the RMS roughness of the smoother film (over a scan area of $1\ \mu\text{m} \times 1\ \mu\text{m}$) was only 0.48 nm. The film heated directly on the hot plate show was essentially flat (RMS ~ 0.9 nm) but with poles around 10 nm heights distributed over the surface of the films. The formation of these poles could be due to the sudden sublimation of starting materials before decomposition. Thus in this study, the IGZO thin films prepared for TFT device fabrication were heated by a slow heating process.

3.2. Effect of Annealing-Time and Annealing Temperature on Performance of IGZO TFT. To study the effect of annealing-time on TFT performances, the as-deposited IGZO films were annealed at 450°C in air with 0.5 hour, 1 hour, 2 hours, and 4 hours, respectively. The representative transfer characteristics of IGZO TFTs with different annealing-time in air were shown in Figure 4(a). Table 1 showed that the corresponding electric performances were extracted and summarized from Figure 4(a). The IGZO film annealed for 0.5 hour showed a typical transistor characters and exhibited a field effect mobility of $0.25\ \text{cm}^2\text{V}^{-1}\text{s}^{-1}$ with a V_{TH} of -25 V. It demonstrated that the decomposed precursors could be fully converted into IGZO under a shorter annealing-time. Under prolonging annealing-time to 1 hour, the IGZO TFT performance increased to $0.63\ \text{cm}^2\text{V}^{-1}\text{s}^{-1}$ with a V_{TH} of 6 V. And then further prolonging annealing-time to 2 hours and 4 hours, the mobilities further increased to $\geq 1\ \text{cm}^2\text{V}^{-1}\text{s}^{-1}$, but the V_{TH} seriously shifted to negative voltages and the off-current increased to 1~2 orders. The increase in I_{off} and the positive shift in V_{th} for prolonging annealing-time might be attributed to the increased conductivity of the oxide semiconductor film [19]. Based on the performances of IGZO TFT with various annealing-times, we chose the IGZO film annealed for 1 hour as the optimized process condition.

Figure 4(b) showed representative transfer characteristics for TFTs based on IGZO (In : Ga : Zn = 1 : 1 : 1) films with different annealing temperatures in the range 340°C~500°C for 1 hour in ambient environment. From Figure 4(b), we could see that the deduced onset voltage ($V_{\text{on}} @ I_{\text{DS}} = 10^{-9}$

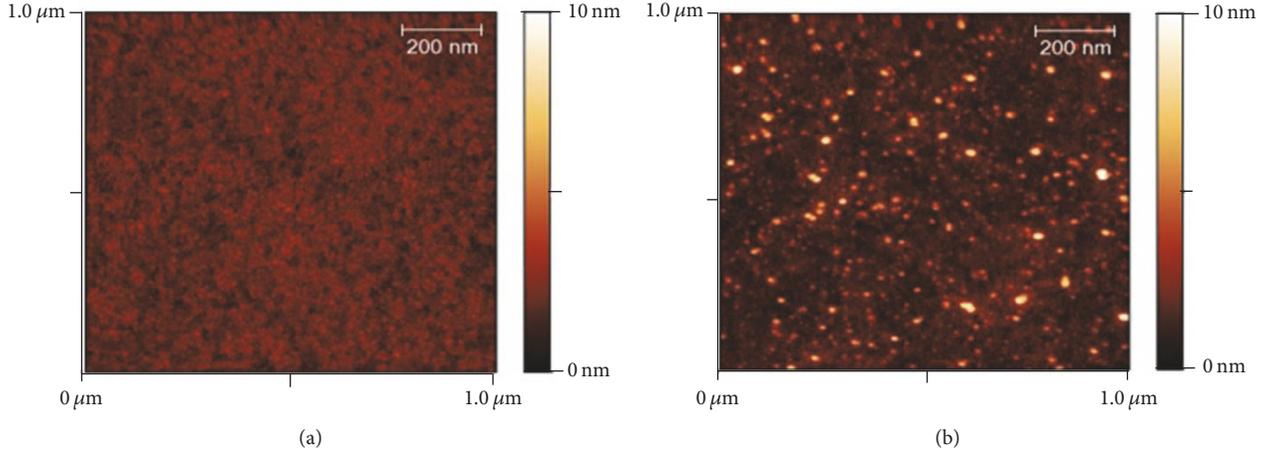


FIGURE 3: AFM images for IGZO films annealed at 500°C via (a) the slow heating and (b) rapid heating.

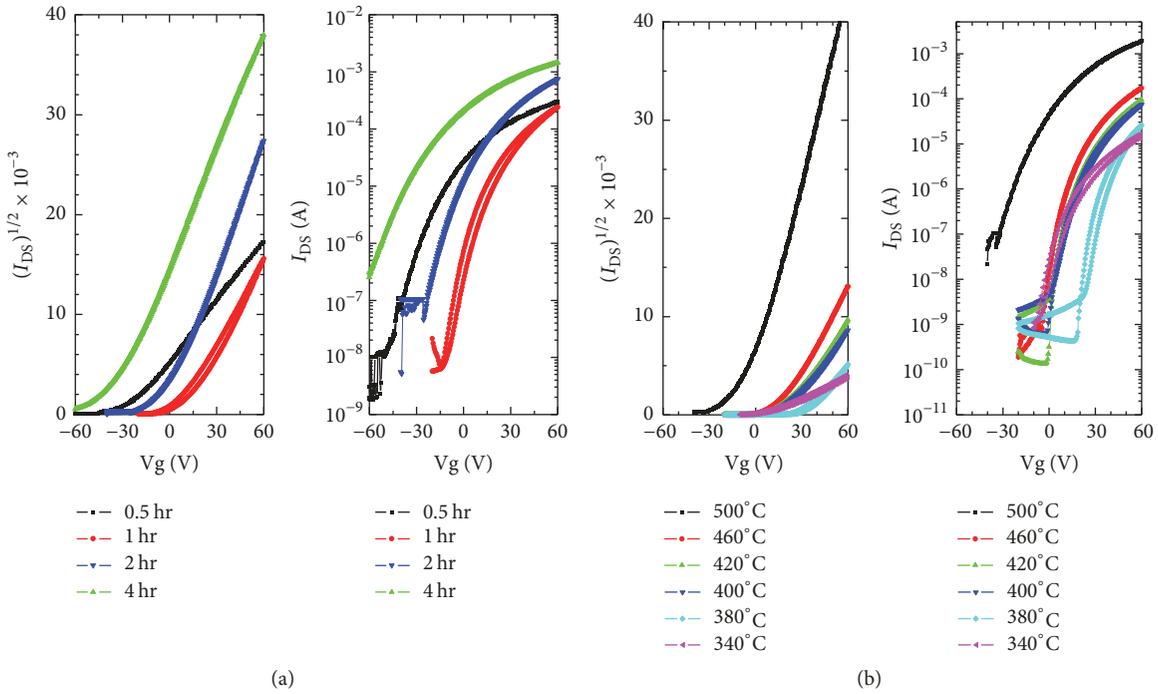


FIGURE 4: Drain current (I_{DS} and $\sqrt{I_{DS}}$) versus gate voltage (V_g) transfer curves of TFTs with IGZO active layers prepared with different process conditions (a) 0.5 hr, 1 hr, 2 hr, and 4 hr annealing at 450°C; (b) 340°C~500°C annealing for 1 hr.

TABLE 1: The TFT device performance of the IGZO films prepared with different annealing-time.

Annealing time (hour)	Mobility ($\text{cm}^2\text{V}^{-1}\text{s}^{-1}$)	V_{TH} (V)	I_{off} (A)
0.5	0.25	-25	10^{-9}
1	0.63	6	10^{-9}
2	1.3	0	10^{-8}
4	1	-32	10^{-7}

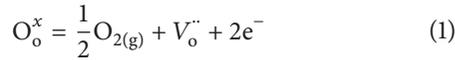
A) appeared to be positively shifted to ~ 20 V at 380°C. Under higher temperature annealing (400°C~460°C), V_{on} shifted to zero. At 500°C annealing temperature, V_{on} (@ $I_{DS} = 10^{-7}$ A)

further shifted negatively to -30 V. It could be inferred that the 1:1:1 IGZO film operated at enhancement mode with lower annealing temperatures and converted to depletion mode with higher annealing temperatures. According to Figure 4(b), the corresponding saturation mobility and the threshold voltage were extracted and summarized in Table 2. The IGZO film annealed at low temperature (340°C, 1 hour) showed typical transistor characters and exhibits a low field effect mobility of $0.06 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ with a V_{TH} of 20 V. It was likely that the decomposed precursors were partially converted into IGZO under low temperature annealing. Upon further increasing of the annealing temperature (380°C~460°C), the mobility gradually increased from 0.32 to

TABLE 2: The TFT device performance of the IGZO films prepared with different annealing temperatures.

Annealing temperature (°C)	Mobility ($\text{cm}^2\text{V}^{-1}\text{s}^{-1}$)	V_{TH} (V)
340	0.06	20
380	0.32	28
400	0.39	17
420	0.43	15
460	0.56	10
500	3.4	0

$0.56 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ while the corresponding V_{TH} decrease down from 28 V at 380°C to 10 V at 460°C. This V_{TH} reduction and mobility increase suggested that the IGZO films approach to the fully oxidized condition at around 380°C. When the annealing temperature was further up to 500°C, the mobility sharply rose to $3.4 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$. The enhancement in carrier mobility for high annealing temperatures could be attributed to an enhancement in oxygen vacancies of IGZO film. According to (1), the oxygen vacancy formation process is closely related to the generation of charge carriers.



where O_2 was lost from the oxide sublattice (O_o^x) to create a doubly charged oxygen vacancy ($\text{V}_\text{o}^{\cdot\cdot}$) and two free electrons. In addition, the mobility of amorphous IGZO films depended on the carrier concentration, since the carrier transport was governed by percolation conduction over trap states and was enhanced at high carrier concentrations by filling the trap states [20, 21].

Based on XPS spectrum, We fitted the oxygen peak (O 1s) to three different oxygen peaks, centred around 530, 531, and 532 eV. The first peak labelled as O_I (M-O) centred at 530 eV was attributed to O_2^- ions surrounded by metal atoms in the IGZO film, such as In, Ga, and Zn. The second peak labelled as O_II (oxygen vacancy) centred at 531 eV had been attributed to oxygen atoms in the vicinity of an oxygen vacancy. The third peak labelled as O_III (O-H) centred at 532 eV possibly originated from hydroxide bonds, which possibly arose from some intermediate metalorganic compounds (M-OH) typically present in a solution-processed film. For the data shown in Figure 5, it should be noted that the O_III (O-H) did not completely disappear up to 340°C. With the annealing temperature increasing to 380°C, the O_III (O-H) could completely diminish. This result demonstrated that the 340°C annealed film still contained a significant amount of residual organics, which were fully removed at a temperature as high as 380°C. In addition, it is worth mentioning that O_I (M-O, 530 eV) and O_II (oxygen vacancy, 531 eV) percentage ratio drastically rises as the annealing temperature increases above 380°C, which might mean that some local ordering had occurred around the temperature range. With the increase of annealing temperatures to 500°C, $[\text{O}_\text{II}]/[\text{O}_\text{I} + \text{O}_\text{II} + \text{O}_\text{III}]$ gradually increases from 55% to 65%. It was believed that the enhancement of the mobility upon higher temperature annealing was attributed to the increase of the number of oxygen vacancies (O_II). In addition, the too much number

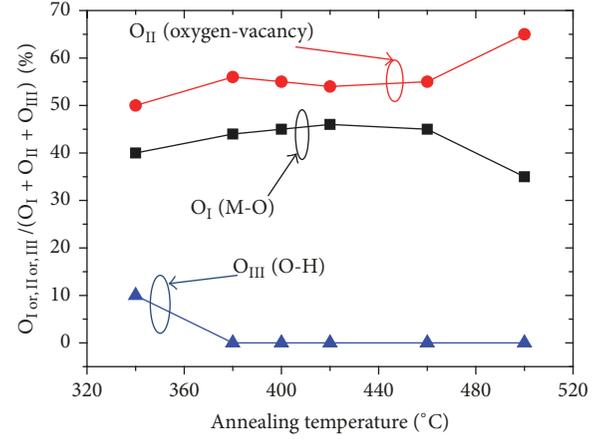


FIGURE 5: Relative percentage ratios of different oxygen species changing with the annealing temperatures based on XPS spectrum.

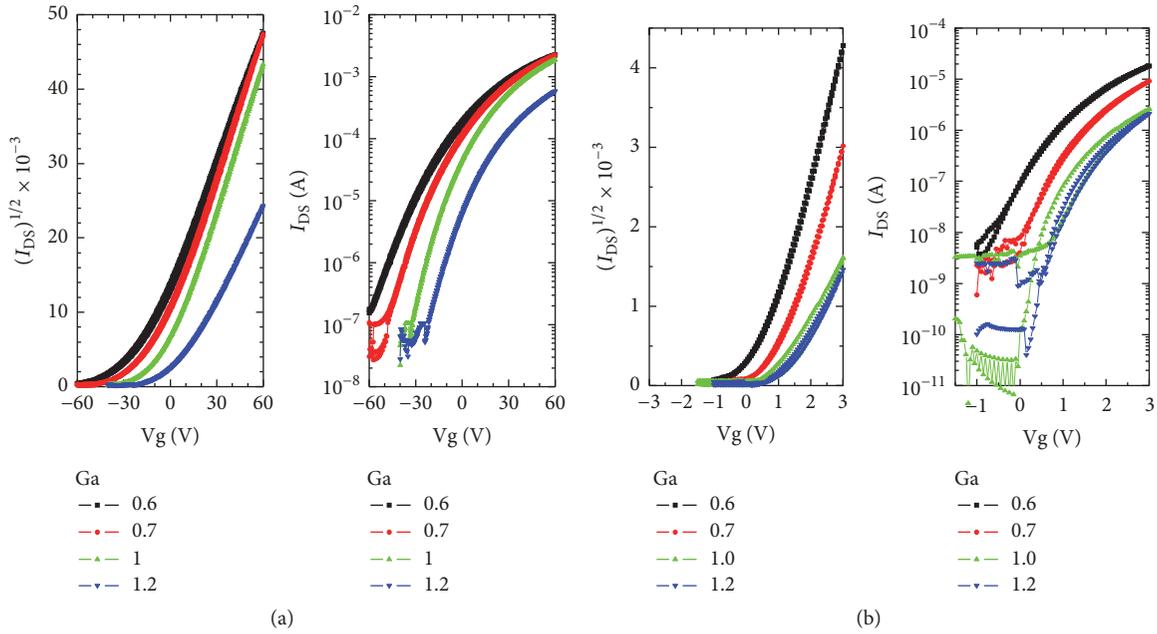
of oxygen vacancies (O_II) was to make the IGZO TFT hard to be turned off.

3.3. Effect of Ga Doping Molar Ratios on Performance of IGZO TFT. Figure 6 showed the transfer characteristics of IGZO TFT devices with various Ga molar ratios under different process conditions, and the related electric properties were listed in Table 3. And more importantly, the transfer curves of the IGZO films with various Ga molar ratios offered a powerful demonstration of the influence of the Ga addition. The Ga molar ratio of 0.6 in precursor made the IGZO TFT hard to be turned off even the applied gate voltage increased to -60 V, which was an indication of conductor-like behavior. Increasing the Ga molar ratio to 0.7 had generated a transfer curve that showed a distinct transition from off-state to on state as the off-current level declined significantly. As the Ga content increased further, the threshold voltage (V_{TH}) swung toward positive and the current level difference between on and off-states became greater, clearly displaying transistor characteristics. These systematic variations including the V_{TH} shift and the off-current reduction were possibly caused by the addition of Ga, because increasing the Ga content was expected to suppress carrier generation through oxygen vacancy formation and hence help achieve a lower off-current.

From Table 2, we can see that the field effect mobilities (μ_e) of IGZO ($1:x:1$, $x = 0.6, 0.7, 1, 1.2$) TFTs in the saturation region were extracted as 3, 3.6, 3.4, and $1.4 \text{ cm}^2/\text{V}\cdot\text{s}$ with 200 nm SiO_x , respectively. In order to obtain the low-voltage TFT devices (V_g is $-3 \text{ V} \sim 3 \text{ V}$), 30 nm SiO_x was used as the gate dielectrics under 450°C annealing temperature. The saturated mobilities decreased as Ga molar ratio increased, which exhibited the similar trend to that of case 1. In addition, V_{TH} of IGZO TFT shifted to positive voltage as the Ga molar ratio was increased. In a word, the variation of μ_e and V_{TH} with Ga molar ratio exhibited the similar trend under different conditions. This was almost certainly due to the increase in the number of free electrons associated with the decreasing Ga content. It is well known that Ga could offer stronger chemical bonds between metal and oxygen

TABLE 3: The TFT device performance of the IGZO films prepared with different Ga molar ratios in precursor solutions.

Process condition	Ga(x) molar ratio (In : Ga : Zn = 1 : x : 1)	Mobility ($\text{cm}^2\text{V}^{-1}\text{s}^{-1}$)	V_{TH} (V)	I_{off} (A)
Case 1 (200 nm, 500°C annealing)	0.6	3	-20	N/A
	0.7	3.6	-10	10^{-8}
	1.0	3.4	0	10^{-8}
	1.2	1.4	5	10^{-8}
Case 2 (30 nm, 450°C annealing)	0.6	3.1	0.6	NA
	0.7	2.4	0.9	10^{-9}
	1.0	0.63	1	10^{-11}
	1.2	0.64	1.2	10^{-11}

FIGURE 6: Drain current (I_{DS} and $\sqrt{I_{\text{DS}}}$) versus gate voltage (V_g) transfer curves of TFTs with IGZO active layers prepared with different Ga molar ratios in precursor solutions (a) annealed at 500°C for 1 hour, the thickness of SiO_2 is 200 nm; (b) annealed at 450°C for 1 hour, the thickness of SiO_2 is 30 nm.

that reduces the concentration of free carriers and thus help reducing the V_{TH} .

4. Conclusion

In summary, it had been demonstrated that MOD processed IGZO transistors had the basic characteristics of thin film transistors. The performances of MOD processed IGZO transistors were determined by the annealing temperatures. Based on the analysis of O 1s statement, the annealing temperature can influence the number of oxygen vacancy to further affect the carrier concentration. In addition, the IGZO TFT devices with various Ga molar ratios were compared to demonstrate the influence of the Ga addition. As a result, the performance of IGZO TFT with the mobility of $3.4 \text{ cm}^2\text{V}^{-1}\text{s}^{-1}$ showed the MOD process was a promising approach. The spin-coating preparation of IGZO film was a rather simple process for transistor fabrication and therefore provided the possibility

of low-cost manufacture of transparent TFTs with good performance.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

Acknowledgments

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