Aqueous Solution Derived Amorphous Indium Doped Gallium Oxide Thin-Film Transistors

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ABSTRACT In this study, we report high-performance amorphous Ga$_2$O$_3$ metal-oxide (AMO) thin film transistor (TFT) using an low-temperature solution-process coupling with In alloy engineering. In doping can lower the activation temperature of gallium oxide and increase the oxygen vacancy concentration to further activate the device. The optical bandgap of IGO film can be changed from 5.3 to 4.25 eV with the In doping concentration ($C_{\text{In}}$) increasing from 0 % to 50 %. All TFTs with IGO channels exhibit n-type transistor characteristics and the evolution of their key electrical parameters with the In-dopant is well elucidated by the structural and morphological characterization. With the increase of $C_{\text{In}}$, the performance of the device becomes better. Finally, a saturation field-effect mobility of 3.63 cm$^2$V$^{-1}$s$^{-1}$, a current on/off ratio of $10^6$, and a threshold voltage of 2.5 V are achieved by the In$_{0.5}$Ga$_{0.5}$O ($C_{\text{In}}=50\%$) based device. The In$_{0.5}$Ga$_{0.5}$O TFT also demonstrates good bias stress stability. Under the action of 20 V and $-20$ V gate bias for 3000 s, the $\Delta V_{TH}$ is $+2.27$ V and $−1.95$ V, respectively.

INDEX TERMS Thin film transistor (TFT), solution process, gallium oxide (Ga$_2$O$_3$), in doping.

I. INTRODUCTION

The requirement to grow oxide semiconductor films by low cost (easy-to-operate, large-area fabrication, non-vacuum) techniques has made solution process attractive in the past few years [1]–[6]. In the process of solution preparation, metal oxide semiconductor is more suitable for preparing transparent thin film transistor (TFT) circuits [7]–[8]. For all metal oxide materials, the empty metal cation s-states constitute the conduction band minimum. When the spatial overlap of metal s-states is bigger than the interaction distance of metal ions, the electron mobility of amorphous phase can be equivalent to or slightly lower than that of the corresponding single crystal. The main quantum number (n) mainly determines the spatial expansion of metal s-states, so the transition metal elements with (n−1)d$^{10}$ns$^0$ (n ≥ 5) peripheral electronic arrangement, are ideal oxide semiconductor candidates [9], like In element which satisfies this requirement. Further research has found that mixing metal cations with different sizes can effectively enhance thin film properties in multiple compounds, such as binary, ternary, or even quaternary components. So far, many researchers have studied that the AOS thin films fabricated by solution process could be employed to obtain high quality TFTs [3], [10]–[17]. For instance, electron mobilities larger than 2 cm$^2$V$^{-1}$s$^{-1}$ are obtained by the amorphous ZnO, IZO and Ga$_2$O$_3$ transistors [18]–[19]. However, the acquisition of outstanding performance depends on the high temperature, since these oxide films are usually annealed at a temperature larger than 450 °C.

Among these AOSs, Ga$_2$O$_3$ is a large band gap semiconductor with extremely high electric field strength which is higher than GaN and SiC [20]–[24], and hence, it exhibits great potential for various optoelectronic applications. Recently, compared to single crystal and bulk materials, the
Ga$_2$O$_3$ thin film has been increasingly of greater interest to researchers because of its low cost, easy growth, excellent repeatability, and flexibility. Various approaches have been employed to prepare β-Ga$_2$O$_3$ thin films, such as radio-frequency magnetron sputtering [25]–[26], molecular beam epitaxy, etc. However, solution processed β-Ga$_2$O$_3$ has less been reported due to the very high transition temperatures above 700 °C. Previously, Thomas et al. reported the ultrasonic spray pyrolysis method to grow β-Ga$_2$O$_3$ thin films, and achieved good performance TFTs by using Ca as the electrode [27].

Chemical doping is an effective approach to lower down the transition temperature. For example, Tobin et al. used Ga doping to lower down the ZnO transition temperature. Moreover, various doping methods have been used to control the film electrical properties [28]–[29]. In doping could introduce a large number of oxygen vacancies ($V_0$) and increase the carrier concentration of the thin film due to the weaker In-O bonding compared to Zn-O and Al-O bindings. Meanwhile, it has little effect on the lattice structure of Ga$_2$O$_3$. Hence, In is an appropriate doping source in the insulating Ga$_2$O$_3$ thin film to improve device performance. In this study, we propose aqueous solution-grown In doped Ga$_2$O$_3$ ($C_{In} \leq 50\,\text{mol}\%$) and elaborate the role of In doping in enabling low-cost, low-temperature, low-pollution and high-quality AOS thin films preparation. The electrical characteristics of TFTs based on Ga$_2$O$_3$ thin films with different In doping concentrations are compared in detail to clarify the influence of chemical composition and annealing temperature on the performance parameters of AOS-TFTs.

**II. EXPERIMENT**

The gallium nitrate [Ga(NO$_3$)$_3$.xH$_2$O] powder and indium nitrate [In(NO$_3$)$_3$.xH$_2$O] powder were dissolved in deionized (DI) water (10 mL) to obtain the corresponding precursor solutions. The molar ratios of In/(In+Ga) are 0 %, 5 %, 10 %, 25 % and 50 % respectively. To obtain the IGO films the precursor solution was spin coated at 3000 rpm for 30 s. These P$^{++}$-Si/SiO$_2$/IGO samples were then annealed at 450 °C for 1 h in ambient air. Finally, a 100 nm thick patterned Al (as source/drain electrodes) was thermally evaporated on the IGO films ($W/L = 1000/100 \, \mu \text{m}$). An Agilent 1500 semiconductor parameter analyzer was employed to obtain the thin film transistor characteristics in the dark at room temperature.

The surface roughness of the IGO films has been tested by Atomic force microscopy (AFM). The X-ray diffraction (XRD) was used to study the structural characteristics of the IGO films. Photoluminescence (PL) spectroscopy was measured with 325 nm He-Cd laser as excitation source. In order to explore the bandgap of IGO, a UV–visible spectrophotometer (UV-Vis) measurement was carried out. X-ray photoelectron spectroscopy (XPS) was also carried out to study the film composition.

The models of IGO were investigated by the VASP code using the Perdew–Burke–Ernzerhof (PBE) of Generalized gradient approximation (GGA) [30]–[32]. The $1 \times 3 \times 2$ supercell models of β-Ga$_2$O$_3$ were employed to construct IGO with different concentration oxygen-vacancies. A plane-wave basis with energy cutoff of 400 eV and an energy convergence criterion of $10^{-5}$ eV were used to perform the geometry optimization. Meanwhile the $d$ states of Ga were treated as the valence states. Just the gamma point was set for the geometry optimization calculations due to the large size of supercell.

The alloying formation enthalpies ($\Delta H$) and vacancy formation energies ($E_f$) were calculated by the following formula [33]–[34]:

$$\Delta H[(In_xGa_{1-x})_2O_3] = E[(In_xGa_{1-x})_2O_3] - xE[In_2O_3] - (1 - x)E[Ga_2O_3]$$

$$E_f = E_w - E_{w/o} - n_O\mu_O$$

where $E[(In_xGa_{1-x})_2O_3]$, $E[In_2O_3]$, and $E[Ga_2O_3]$ were the total energies of (In$_x$Ga$_{1-x}$)$_2$O$_3$, In$_2$O$_3$, and Ga$_2$O$_3$ in the same supercell. The $E_w$ and $E_{w/o}$ were respective the total energies of the IGO models with and without oxygen-vacancies. $n_O$ and $\mu_O$ were respective the number and chemical potential of oxygen atom.

**III. RESULTS AND DISCUSSIONS**

All films with a thickness of 5-10 nm are spin-coated from 0.1 M precursor solutions and then annealed at a selective temperature to ensure high-quality films. Figure 1 (a)–(e) exhibit the AFM images of IGO films deposited on the sapphire substrates. The root mean square (RMS) roughnesses of $C_{In}$-0%, $C_{In}$-5%, $C_{In}$-10%, $C_{In}$-25% and $C_{In}$-50% thin films are 0.161 nm, 0.183 nm, 0.178 nm, 0.169 nm and 0.226 nm, respectively. All films show sub-nanometer surface morphology. The extremely smooth IGO films can lead to a good quality of the interface area between the drain/source electrodes and the semiconductor layer, and then reduces the scattering effect. To further reveal the structure characters of these fabricated IGO films, the XRD curves are illustrated in Figure 1(f). None peaks are observed in...
The bandgap reduces from 5.30 eV (C In-0%) to 4.25 eV (C In-50%). This is because the deposition and annealing temperatures are far lower than the crystallization temperature of the fabricated films, suggesting that all fabricated films are amorphous.

The transmittance spectra for different C In thin films on sapphire substrates is shown in Figure 2(a). The extracted direct bandgap ($E_g$) is plotted as a function of C In, shown in Figure 2(b). The inset panel gives the bandgap of IGO and IGO+V$_O$ calculated by the DFT. The formation enthalpies of IGO and IGO+V$_O$ and O-vacancy formation energy in IGO is displayed in Figure 2(c). (d) Photoluminescence intensity profiles of the IGO thin films with different C In.

Figure 2. (a) The transmittance spectra for different C In thin films on sapphire substrates. (b) The extracted direct bandgap ($E_g$) is plotted as a function of C In. The line is drawn to show the variation trend. The inset panel gives the bandgap of IGO and IGO+V$_O$ calculated by the DFT. (c) The formation enthalpies of IGO and IGO+V$_O$ and O-vacancy formation energy in IGO. (d) Photoluminescence intensity profiles of the IGO thin films with different C In.

The Figure 1(f), regardless of the concentration of In component, suggests that all fabricated films are amorphous. That is because the deposition and annealing temperatures are far lower than the crystallization temperature of $\beta$-Ga$_2$O$_3$ ($>700^\circ$C).

Figure 2(a) displays the transmittance spectra of the IGO films. It is seen that, all the films possess high transmittance over 97% in the visible region and a sharp absorption edges in the deep ultraviolet region. The transmittance is reduced and the absorption edges blue-shifts with the increasing In concentration because the lower bandgap of In$_2$O$_3$ can reduce the bandgap of IGO alloys. The detailed bandgaps of IGO alloys are extrapolated through the (ahv) plots using Tauc model [2], and listed in Figure 2(b). The bandgap reduces from 5.30 eV (C$_{In}$-0% Ga$_2$O$_3$) to 4.25 eV (C$_{In}$-50%), demonstrating an effective variation of the bandgap of Ga$_2$O$_3$ by In-alloying. Note that, the bandgap of Ga$_2$O$_3$ film is larger than the band gap (4.6-4.9 eV) of $\beta$-Ga$_2$O$_3$ single crystal due to Burstein-Moss effect [35]. Meanwhile, the oxygen-vacancy may exist in the fabricated films, which can shift up the conduction band and then enlarges the bandgap, as displayed in Figure S1, in the supplementary material. The inset of Figure 2(b) lists the calculated band gap of IGO and IGO with an oxygen-vacancy (IGO+V$_O$). Obviously, the latter is higher than the former. To verify the existence of oxygen-vacancy, the formation enthalpies of IGO and IGO+V$_O$ are calculated and displayed in Figure 2(c). The formation enthalpies of IGO+V$_O$ are lower than those of IGO, suggesting the larger stability for the IGO+V$_O$ than the IGO. Meanwhile, the formation energy of V$_O$ in IGO is relative low, suggesting the spontaneous formation of V$_O$. Moreover, the alloying formation enthalpy and oxygen-vacancy formation energy decrease with the increasing In concentration. It means the enlarging oxygen-vacancy concentration when more and more In component is introduced into the Ga$_2$O$_3$ film. Figure 2(d) gives the photoluminescence (PL) of IGO to confirm such character. Regardless of the In concentration, there are three PL peaks around the 403 nm, 537 nm, and 710 nm, respectively. The peak centered at 403 nm due to the recombination of a trapped electron in a donor with a trapped hole in an acceptor, where the origins of the donor and acceptor are the V$_O$ and the self-trapped hole and Ga-O vacancy pair, respectively. The peak centered at 537 nm is caused by the self-trapped hole and Ga-O vacancy pair, and the peak centered at 719 nm is attributed to the V$_{O}$s, as exhibited in Figure S2. In addition, the peak intensity around 719 nm continuously increases with the increasing C$_{In}$, suggesting the enlarged V$_O$ concentration induced by In dopant.

To further unveil the film characters, the Ga 3d and O 1s of different In concentrations are analyzed via XPS spectra. Figure 3(a)-(e) shows the O 1s spectra of films with different In concentrations (C$_{In}$). In particularly, the peaks of the O 1s are located at $\sim$530.8 eV and $\sim$531.9 eV. For all IGO films, the lower binding energy centered at $\sim$530.8 eV should be attributed to metal oxygen lattices (M-O). While the higher binding energy ($\sim$531.9 eV) is related to the residual metal hydroxide species coupling with oxygen vacancies (V$_O$+M-OH). Figure 3(f) exhibits that M-O have the relationship with V$_O$+M-OH. It can be inferred that all IGO films possess large amounts of oxygen vacancies (V$_O$) and metal hydroxide (M-OH). This is mainly due to the low annealing temperature caused incomplete conversion. Meanwhile, the V$_O$ concentration increased when increasing the C$_{In}$, as can be seen from Figure 3(f), since In-O has a higher formation energy compared to Ga-O. This is consistent with the above DFT analysis and PL results. In fact, the carrier mobility of metal oxide films is related to oxygen vacancies and metal oxygen lattices. The metal oxygen lattices can contribute to trap sites reduction, which can induce high concentration of free electron. Meanwhile, at high carrier concentration, the free electrons generated...
In order to explore the feasibility of IGO films as channel layers, the structure of P+/Si/SiO2/IGO was successfully manufactured (Figure 4(a)). And all resulted films are annealed at 450 °C for 1h. The transfer characteristic curve of the IGO thin film transistors is presented in Figure 4(b). The output characteristics of the optimized IGO TFT based on different chemical components are shown in Figure 4(c)-(f).

Table 1 presents the electronic parameters of different IGO TFTs with CIn of (c) 50 %, (d) 25 %, (e) 10 % and (f) 5 %. (g) Positive gate bias and (h) Negative gate bias test result for IGO (CIn = 50 %) TFT.

### Table 1. Electrical parameters of IGO TFTs with different CIn (molar ratios) with a thermal annealing temperature of 450 °C.

| CIn | IOn/off (cm²V⁻¹s⁻¹) | Vth (V) | SS (V DECADE⁻¹) | N2 (cmz⁻²V⁻²) |
|-----|----------------------|---------|-----------------|--------------|
| 0 % | -                    | -       | -               | -            |
| 5 % | 1.7×10⁻⁴             | 20      | -10⁴            | 3.24         |
| 10 %| 2.9×10⁻⁴             | 20      | -10⁴            | 2.79         |
| 25 %| 4.2×10⁻⁴             | 17.5    | -10⁴            | 1.52         |
| 50 %| 3.63                 | 2.5     | -10⁴            | 1.38         |

by oxygen vacancies can enhance carrier transport by filling trap states, which is conducive to improving the mobilities of the IGO films. The ratios of In content in different IGO films are also confirmed by XPS measurements (Table S1), in the supplementary material. The results show that the ratio of In to Ga is almost similar to the precursor used.

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| 50 %| 3.63                 | 2.5     | -10⁴            | 1.38         |

IV. CONCLUSION

In conclusion, we have developed a non-toxic, environment friendly, water-based technology to fabricate In-doped Ga2O3 TFTs. All the data indicate that In doping is beneficial to improve the electron mobility and decrease the annealing temperature of gallium oxide. Especially, the TFT based on IGO film with CIn = 50 % exhibits good electrical performance with μSat of 3.63 cm²V⁻¹s⁻¹, Vth of 2.5 V, Ion/off of 10⁶ and SS of 1.38 V decade⁻¹ at VDS of 30 V. All the measurements provide a comprehensive study on the evolution of the energy band, optical properties and electrical performance of In-doped Ga2O3 thin film materials, which will promote further research on the application of Ga2O3 TFTs.

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