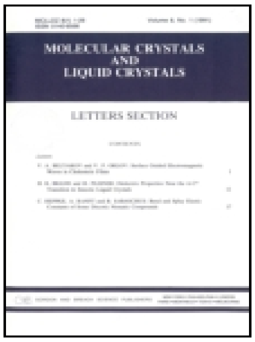

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Molecular Crystals and Liquid Crystals

ISSN: 1542-1406 (Print) 1563-5287 (Online) Journal homepage: http://www.tandfonline.com/loi/gmcl20

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To cite this article: Van Thi Thanh Ho, Long Giang Bach, Tran Thanh, Nam Giang Nguyen & Lu-Sheng Hong (2015) Effect of Gallium Source Material on the Transparent Conducting Properties of Ga:ZnO Thin Films Through Metalorganic Chemical Vapor Deposition, Molecular Crystals and Liquid Crystals, 623:1, 433-443, DOI: 10.1080/15421406.2015.1076321

To link to this article: http://dx.doi.org/10.1080/15421406.2015.1076321

Mol. Cryst. Liq. Cryst., Vol. 623: pp. 433–443, 2015
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ISSN: 1542-1406 print/1563-5287 online
DOI: 10.1080/15421406.2015.1076321

Effect of Gallium Source Material on the Transparent Conducting Properties of Ga:ZnO Thin Films Through Metalorganic Chemical Vapor Deposition

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We present the first experimental comparisons of growth of Ga-doped ZnO (GZO) thin films through metalorganic chemical vapor deposition (MOCVD) using Triethylgallium (TEG) and Trimethylgallium (TMG) as dopant sources with qualified opt-electrical properties. Interestingly, we found the GZO films growth by using TEG source exhibited the preferred orientation of (100) and (110) planes which (100) plane is the dominant orientation as well as the formation of pyramidal texture on the film surface compared to the one using TMG source that is highly (002)-orientated ZnO films and formation of planar structure. Additionally, the total C incorporation into GZO films prepared by TEG is slightly lower than that of using TMG source, indicating the formation of stable C_2H_4 *species during reaction when using TEG source can reduce the C incorporation of GZO films compared to the less stable* CH_3 *species by using TMG source. It also found that the doping efficiency by using TEG as a dopant source was much lower than that of using TMG source due to the different thermal decomposition between two sources. These results indicate clearly that the Ga dopant source is crucial to determine the morphology, structure, carbon incorporation as well as the doping efficiency of GZO films that can motivate further research in this field by using various materials.*

Keywords MOCVD; GZO; Triethylgallium; Trimethylgallium

1. Introduction

Transparent conducting oxides (TCOs) are critical components, used as transparent electrodes, in many optoelectronic devices, including thin film solar cells, light emitting

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This paper was originally submitted to *Molecular Crystals and Liquid Crystals*, Volume 617, Proceedings of the 18th International Symposium on Advanced Display Materials and Devices.

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diodes, and flexible displays [1–4]. The most utilized TCO layers are SnO_2, In_2O_3 and their mixture such as Tin-doped In_2O_3 (ITO), F-doped SnO_2 (FTO). This situation is about to change because ITO and FTO suffers of several drawbacks such as the increasing cost of indium, scarce material-whose main constituent, together with instability both of ITO and FTO under H_2 plasma conditions that are limited its wide applications [5–6]. These issues have stimulated numerous developments aiming at finding out the next generation of transparent electrode materials. Recently, ZnO-based TCO is promising and attracted much attention because of inexpensive, relatively abundant, chemically stable, easy to prepare and non-toxic. Another benefit of ZnO is that it can be synthesized using various manufacturing process. For example, the ability to use low pressure chemical vapor deposition (LPCVD) techniques is particularly attractive, resulting in better step coverage, higher deposition rates and low cost. Currently, much attention has been centered on the doped-ZnO thin films such as B-doped ZnO (BZO), Ga-doped ZnO (GZO) and Al doped ZnO (AZO) through CVD technique that can alternative the traditional ITO and FTO [7–9]. However, the LPCVD of BZO films is typically performed using B_2H_6 as the dopant source - a highly toxic material that requires very expensive handling. AZO films prepared easily to form the Al_2O_3 cluster because the high reactivity of Al with oxygen which was responsible for the degradation of electrical properties of these films [10]. Additionally, it has been reported that the resistivity stability of Ga-doped ZnO (GZO) thin films is higher than that of AZO thin films because Ga is less reactive and more resistant to oxidation than is Al [11]. In particular, the length of the Ga–O covalent bond (1.92 Å) is very close to that of the Zn–O bond (1.97 Å), based on calculations of ionic radii, meaning that only small deformations in the ZnO lattice will occur even at high levels of Ga doping [12]. So serials of breakthroughs have been achieved to synthesize GZO films through MOCVD with good opto-electrical properties. And most of studies have been focused on the effect of process parameters such as flow rate, deposition temperature, doping concentration, film thickness, and annealing treatment and using different substrate or deposition technique etc. to improve characteristics of the GZO films required for different applications [13–23]. Nevertheless, the influence of the type of source has surprisingly been received less attention, even though the type of source actually decides critically to the quality as well as the cost of thin films due to the difference in the structural, morphological, optical-electrical properties and carbon incorporation into the GZO films can be obtained by using different material sources. To this end, our group has developed a remarkably simple and versatile way based on employing an inexpensive solution of diethylzinc in *n*-hexane (ca. 17 wt.%) as a Zn source to synthesize GZO films with qualified opto-electric properties through MOCVD that could comparable to the GZO films prepared by the high purity of Zn source [24]. Most importantly, our work have also demonstrated the critical effect of source materials such as Zn source on the quality as well as the cost of GZO film growth that opened the potential approach in the fabrication of inexpensive transparent conducting oxides for use in solar cells. This motivated us to make a new comparison between the two gallium source materials on the transparent conducting properties of GZO thin films through MOCVD in this study.

Here we report the first experimental comparisons of growth of GZO thin films through MOCVD using Trimethylgallium (TMG) and Triethylgallium (TEG) as dopant sources. The structural, morphological, residual carbon and opt-electrical properties of GZO films as well as the effect of post annealing treatment of GZO films growth by two sources were investigated and compared in detail in this work.

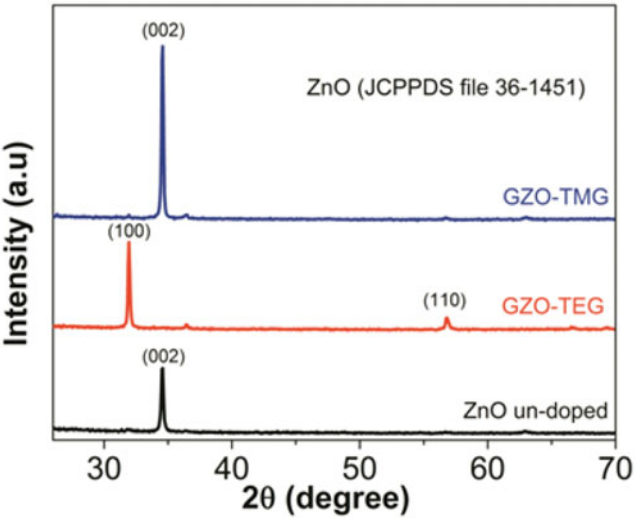

Figure 1. XRD patterns of un-doped ZnO, GZO prepared using TMG and TEG as dopant sources. Deposition temperature = 400°C, and film thickness ~ 800 nm.

2. Experimental Section

The GZO thin films were deposited, using a homemade cold-wall–type LPCVD reactor system, onto Corning EAGLE 2000 glasses, which had been cleaned through sonication in acetone for 5 min, cleaning in ethanol for 5 min, rinsing with DI water, and then blowing dry under N_2. Diethylzinc (DEZn, 5N, Nanmat), Trimethylgallium (TMG, 5N, US Epichem), Triethylgallium (TEG, 5N, Nanmat) and ultra-pure H_2O were used as the sources of Zn, Ga and O atoms, respectively; N_2 (5N) was employed as the carrier gas. Film deposition was performed using a two-step process. First, an undoped ZnO buffer layer (thickness: ca. 20–30 nm) was grown at low temperature (150°C) under a gas flow ratio ([H_2O]/[DEZn]) of 5.1. Next, GZO films were grown by further adding TMG or TEG into the reactor at a substrate temperature of 400°C; the growth time was adjusted to give a whole film thickness of 800 nm. The doping gas flow ratio of TMG or TEG with respect to DEZn was varied to obtain GZO films with various degrees of Ga doping. The total pressure during film deposition was kept constant (~0.47 torr).

The GZO films were characterized using X-ray diffraction (XRD, Bruker D2 PHASER), scanning electron microscopy (SEM, JEOL JSM-6500F), X-ray photoelectron spectroscopy (XPS, Thermo VG Theta Probe), and UV–visible spectrophotometry (JASCO V-670) over the wavelength range from 300 to 1200 nm. Prior to XPS measurement, the sample surface was carefully cleaned through Ar sputtering for 4 min to ensure removal of all contaminants physically adsorbed to the surface. The electrical properties of the films were determined using a Hall effect measurement system (Van der Pauw Ecopia HMS-3000) and a standard four-point probe (KeithLink Technology).

3. Results and Discussion

The XRD pattern of the as-deposited films growth using TMG and TEG as dopant sources are shown in Fig. 1. The un-doped ZnO film was also synthesized and measured XRD

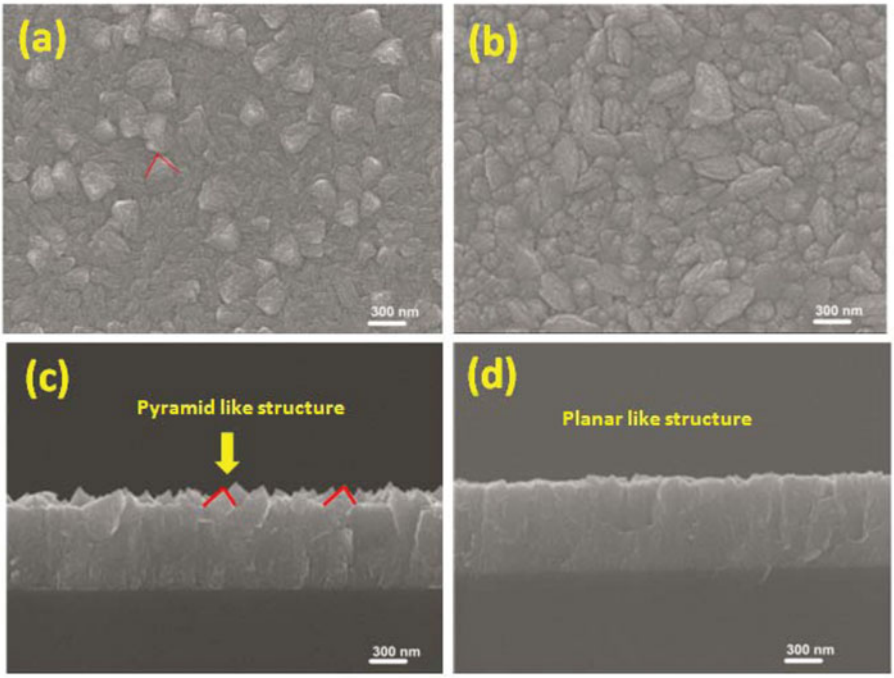

Figure 2. SEM images of the plan view and cross-sectional of the deposited GZO films using (a)-(c) TEG, (b)-(d) TMG as dopant sources. Deposition temperature = 400°C, and film thickness ~ 800 nm.

for comparisons. On the Fig. 1, it can be seen that the ZnO films exhibited a preferred orientation along the (002) plane for both un-doped samples and for GZO films prepared using TMG as a dopant source. On the other hand, by using TEG as a dopant source the preferred orientation of ZnO films were (100) and (110) planes which (100) plane is the dominant orientation. These findings agree well with other published results [25]. The difference in preferred orientation of ZnO thin films growth could be understood due to the effect of different dopant sources on the structure of GZO films. Since TEG was employed of as a dopant source it could be changed the growth kinetics and pathway for precursor decomposition because of different thermal decomposition between TEG and TMG sources [26] with resulting in crystallites growing in the preferred (100) and (110) planes not (002) plane as in the case of using TMG source. In addition, it is worthy of note that there is only once difference in Ga dopant sources between two deposition techniques but it can change the orientation of ZnO films, so we suspected that the forming of stable C_2H_4 species during the CVD reaction in the case using TEG source [27] can be affected to the change preferred orientation of ZnO films. The detail mechanism for the change in preferred orientation by using TEG is very interesting and still under investigates.

In order to better understand the different structure of GZO films when using different dopant sources, we conducted the SEM measurement to further verify the formation of thin films morphologies by using TEG and TMG dopant sources. Figure 2 shows plan-view and cross-view of deposited GZO films using TEG and TMG as dopant sources. Interestingly, the GZO film surface was grown by using TMG as a dopant source (Fig. 2(b)) exhibited a smoother than GZO films using TEG as a dopant source (Fig. 2(a)). Furthermore, the

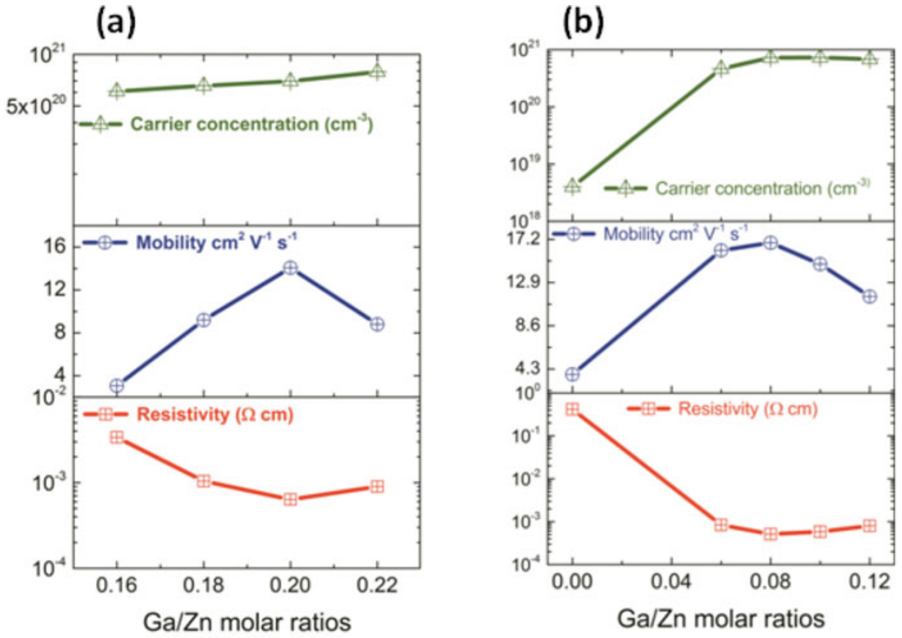

Figure 3. Effect of molar ratio of [Ga]/[Zn] doping to GZO electrical properties (a) TMG; (b) TEG. Deposition temperature = 400°C, and film thickness ~ 800 nm.

introduction of TEG as dopant source to the system leads to appear clearly of small pyramid structure on the surface (Fig. 2(a)) that does not appear for films using TMG source, suggesting there have a transformation of the films structure from a (002) plane to (100) and (110) planes when using TEG source; this is in good accord with the XRD pattern (Fig. 1). Noticeable, in crystalline hexagonal system the growing of the pyramidal shape structure is realized by means of sequentially packaging of (110) planes along the vertical direction and by means of (0 0 2) and (100) planes along the horizontal direction for growing of the planar structure [28]. Evidently, by calculating the intensity ratio of (100) and (110) planes (denoted $I_{(100)}/I_{(110)}$) from XRD data for GZO films prepared by TEG source (Fig. 1), the intensity ratio of $I_{(100)}/I_{(110)}$) to be ~6, which is corresponding to the estimation calculation for area ratio of (100) and (110) planes (denoted $S_{(100)}/S_{(110)}$) was about ~5 that estimated from SEM data (Fig. 2a). These data indicate that the preferred orientation of GZO films with (110) planes can enhance the formation the pyramid like structure on GZO film surface. On the other hand, the morphology and the orientation of the subsequent GZO thin films significantly affect by using different dopant Ga sources such as TEG and TMG source in this case. This finding is confirmed that the Ga dopant source is crucial to determine the morphology and orientation transformation of GZO films.

The electrical properties of GZO films prepared by TEG and TMG sources were determined by Hall measurement at room temperature on Fig. 3. It is interesting to note that the carrier (electron) concentration in the deposited films by using TMG increased upon increasing the TMG gas flow, reaching a saturated level at 7.2×10^{20} cm^{-3} when the molar of [TMG]/[DEZn] reached 0.08 (Fig. 3(b)) while the carrier concentration in the deposited films by using TEG source, reaching a saturated level at 6.97×10^{20} cm^{-3} when the molar ratio of [TEG]/[DEZn] reached 0.20 (Fig. 3(a)). The significant difference in dopant ratio (at saturated level) between using TMG and TEG sources could be explained

Table 1. Summary of the composition of as-deposited GZO films prepared using TMG and TEG as dopant sources

Element	TMG as a dopant sources (at.%)	TEG as a dopant sources (at.%)
Zn	47.02	48.11
O	42.17	41.79
Ga	4.97	5.06
C	5.84	5.04

Note: The molar [Ga]/[Zn] ratio use for TMG and TEG dopants were 0.08 and 0.20, respectively.

due to decomposition temperature of TEG is much lower than that of TMG source [26,29], resulting the TEG source more easily facilitated decomposition at above the surface of films during high temperature reaction (400°C) before it reacts with H_2O and comes to the film surfaces compared with TMG source. Therefore, this result suggests that the doping efficiency by using TEG as a dopant source was much lower than that of using TMG as a dopant source. However, except the different doping efficiency between two sources, the film deposited by TEG source also exhibit a low resistivity of 6.35×10^{-4} Ω cm and high mobility of 14.07 $cm^2\ V^{-1}\ s^{-1}$ at saturated level of concentration (6.97×10^{20} cm^{-3}) that is similar with the film deposited by TMG source (with mobility of 15.0 $cm^2\ V^{-1}\ s^{-1}$ and resistivity of 5.13×10^{-4} Ω cm at saturated level of concentration 7.2×10^{20} cm^{-3}), indicating the high quality GZO films could be obtained by using both TEG and TMG sources.

The effect of different Ga dopant sources on the composition of GZO thin film growth as well as the concentration of C incorporation into the films were determined by XPS analysis. The composition of GZO films prepared by TEG and TMG sources were shown on Table 1.

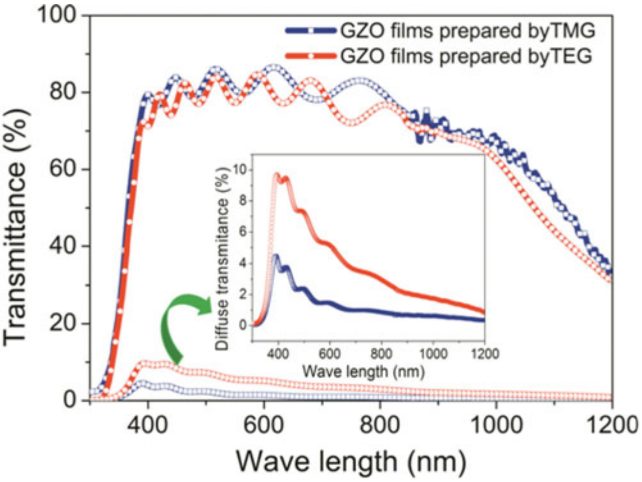

Figure 4. Optical properties of GZO film when using different type of Ga doping sources. The molar [Ga]/[Zn] ratio use for TMG and TEG dopants were 0.08 and 0.20, respectively. The deposition temperature was 400°C and thickness ~ 800 nm.

Table 2. Summary the electrical properties of GZO films annealed at various temperature. The molar [Ga]/[Zn] ratio use for TMG and TEG dopants were 0.08 and 0.20, respectively

	Using TMG as a dopant			Using TEG as a dopant		
Anneal temperature (°C)	Resistivity x 10^{-4}(Ωcm)	Carrier concentration (cm^{-3})	Mobility ($cm^2V^{-1}s^{-1}$)	Resistivity × 10^{-4}(Ωcm)	Carrier concentration (cm^{-3})	Mobility ($cm^2V^{-1}s^{-1}$)
As-deposited (400)	5.13	7.20×10^{20}	16.86	6.35	6.97×10^{20}	14.07
450	5.25	6.15×10^{20}	19.31	6.03	6.22×10^{20}	16.67
475	5.20	6.05×10^{20}	20.10	6.11	5.95×10^{20}	17.20
500	5.23	5.50×10^{20}	21.50	5.90	5.60×10^{20}	18.80
525	5.10	4.79×10^{20}	25.60	6.09	5.01×10^{20}	20.50

Table 3. Summary of the carbon incorporation and carrier concentration variations in the GZO films prepared using pure DEZn before and after thermal annealing

Properties and composition GZO films	As-deposited	After annealing at 450°C	After annealing of at 525 °C
Total C incorporation[a]	5.04%	4.57%	4.57% (−0.47%)[b]
C-C/C-H[a]	4.53%	4.08%	4.01% (−0.52%)[b]
COOR[a]	0.38%	0.25%	0.14% (−0.24%)[b]
O-C-O[a] ((C_{Zn} + $2O_i$)″)	0.13%	0.24%	0.41% (+0.28%)[b]
Holes created after annealing (cm^{-3})[c]	—	~0.93×10^{20}	~ 2.36×10^{20}
Carrier (electron) concentration (cm^{-3})[d]	6.97×10^{20}	6.22×10^{20}	5.01×10^{20}
Estimated electron concentration after hole compensation (cm^{-3})[e]	—	0.75×10^{20}	1.96×10^{20}

[a] the atomic ratio determined by XPS measurement.

[b] the change of atomic ratio before and after annealing.

[c] the holes created by formation of ($C_{Zn} + 2O_i$)″ complex based on the change amount of O-C-O, assuming two holes are released from one active ($C_{Zn} + 2O_i$)″ defect associate.

[d] the experimental data determined by Hall measurement.

[e] the estimated electron concentration after hole compensation.

The results from Table 1 revealed that percentages of Ga doping in the GZO films prepared by TMG or TEG are similar, even though the molar ratio of [Ga/Zn] by using TEG as a dopant source was about ~0.2, that is more than twice times higher than that prepared by TMG (about ~0.08). This result confirmed again the low doping efficiency of TEG compared with TMG source as discussed previously in Fig. 3. Nevertheless, the total C incorporation into GZO films prepared by TEG was 5.04% is slightly lower than that of using TMG as a dopant (5.84%). These results indicate clearly that the formation of stable C_2H_4 species during reaction when using TEG source can reduce the C incorporation of GZO films compared to the formation of less stable CH_3 species by using TMG sources [27,29]

Figure 4 reveals that the optical properties of the GZO films with different dopant sources. The transmittance of GZO films prepared using TMG is similar with those prepared using TEG, suggesting that the transmittance properties of GZO films are not depend on the type of Ga dopant sources. All GZO films exhibit relatively high total transmittance (>80%) in visible region. However, the diffuse transmittance of GZO films prepared using TEG sourcer at wavelength 400 nm is (10%) twice times higher than those prepared by TMG source (5%). The enhancement in the diffuse transmittance of GZO films prepared by TEG source was original from the formation of pyramidal texture of films prepared by TEG as discussed previously from SEM data (Fig. 2). Furthermore, the electrical properties of GZO films could be improved by additional annealing in N_2 atmosphere in a short time (10 min). Table 2 summaries the electrical properties of GZO films prepared by TEG and TMG sources were determined by Hall measurement at various annealing temperature.

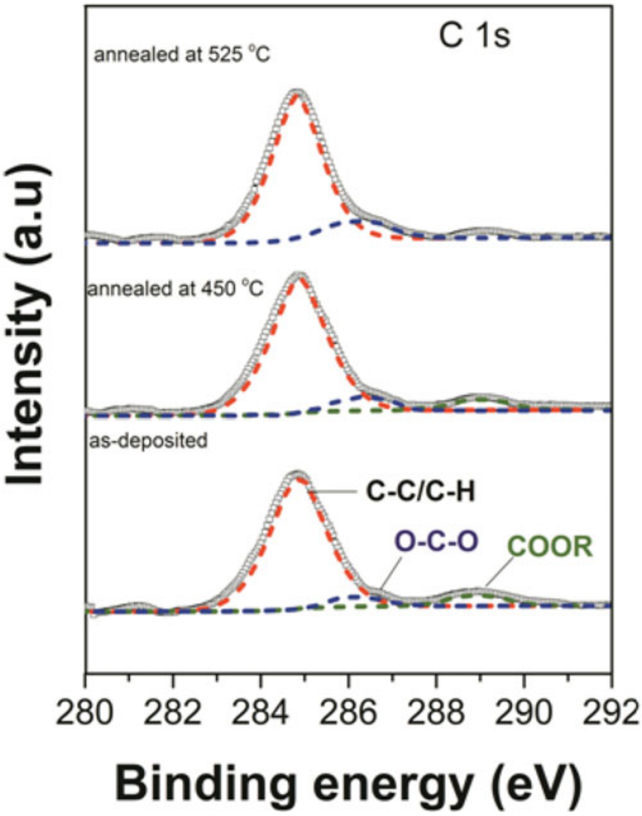

Figure 5. Narrow scan of C 1*s* peak of the GZO films prepared by using TEG as a dopant with [TEG]/[DEZn] = 0.20, deposition temperature = 400°C, and film thickness ~ 800 nm.

It is observed that the carrier mobility in the GZO film for both sources increased upon increasing the annealing temperature from 400 to 525°C; the corresponding carrier concentration decreased from 7.20×10^{20} cm^{-3} to 4.79×10^{20} cm^{-3} for GZO films prepared by TMG source and from 6.97×10^{20} cm^{-3} to 4.90×10^{20} cm^{-3} for GZO films prepared by TEG source. We ascribe this decrease in carrier concentration in the films to variations in the chemical bonding of the incorporated carbon atoms that further confirm by the XPS analysis data (Fig. 5) and Table 3. This phenomena was explained due to the transformation of C-C/C-H groups into O–C–O bonding states to form $(C_{Zn}+2O_i)''$ complexes during annealing under N_2, thereby decreasing the carrier concentration. The diminishing of COOR groups may be due to their decomposition to form CO_2 at the high annealing temperatures, thus causing decrease in the total carbon incorporation amount. The detail mechanism for these phenomena of GZO films prepared by TMG source was described in our previous research [24].

The effects of carbon incorporation during annealing treatment on the film properties prepared by TEG here (Fig. 5, Table 3) are similar to those of the films prepared using TMG source. These results can be understood due to there aren't significant difference in the amount of the total C incorporation in the as-deposited GZO film prepared using TMG source (5.84%) with films prepared using TEG source (5.04%) as well as the amount of Ga doped in the GZO films for both source almost the same (~5%). Additionally, we used pure DEZn as the source of Zn for both case using TMG and TEG sources thus there was not any additional contaminant that contributed to the total incorporation carbon or transformation stage during reaction or post- annealing treatment. These findings indicate that the behavior of C incorporation of as-deposited GZO films and during the annealing treatment almost depended on the Zn source – the main constituent of GZO films- do not depend on Ga dopant sources.

4. Conclusions

In summary, we have grown GZO films by using TEG and TMG as dopant sources through MOCVD in this work. The film deposited by TEG source also exhibit a low resistivity of $6.35 \times 10^{-4}\ \Omega$ cm and high mobility of 14.07 $cm^2\ V^{-1}\ s^{-1}$ at the molar of [TMG]/[DEZn] = 0.2, that is similar with the film deposited by TMG source with mobility of 15.0 $cm^2\ V^{-1}\ s^{-1}$ and resistivity of $5.13 \times 10^{-4}\ \Omega$ cm at [TMG]/[DEZn] = 0.08, indicating the high quality GZO films could be obtained by using both TEG and TMG sources. The post-annealing treatment in N_2 in 10 min was carried out to improve the opt-electrical properties of films for both Ga dopant sources. Due to the different thermal decomposition of TEG source and TMG sources, the GZO films growth by using TEG source exhibited the preferred orientation of (100) and (110) planes compared to the one using TMG source with highly (002)-orientated ZnO films. The total C incorporation into GZO films prepared by TEG is lower than that of using TMG source. It also found that the doping efficiency by using TEG as a dopant source was much lower than that of using TMG source because of the different thermal decomposition between two sources that strongly affected to its doping efficiency. These results indicate that the Ga dopant source significantly affected to the morphology, structure, carbon incorporation that can motivate further research in this field by using various material sources.

Acknowledgment

This study was supported in part by the National Science Council of Taiwan (grant no. NSC 100-2811-E-011-025).

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