Mg acceptor doping in MOCVD (010) β-Ga_2O_3

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In this letter, in-situ Mg doping in β-Ga_2O_3 was demonstrated via metalorganic chemical vapor deposition (MOCVD) epitaxy. The electrical insulating property of the Mg acceptors in β-Ga_2O_3 was found to be intrinsically activated in the as-grown Mg-doped β-Ga_2O_3 thin films. Growth conditions for MOCVD β-Ga_2O_3 were further explored and optimized at a lower growth temperature regime, leading to a better confinement of the Mg-doping profile. Detailed analysis of Mg diffusion characteristics revealed a diffusion barrier energy $E_{barrier} \sim 0.9$ eV for Mg in MOCVD β-Ga_2O_3, which is likely related to an interstitial-assisted process. Surface morphologies and electron transport were characterized on samples grown with different growth temperature and Mg doping level. The MOCVD growth method demonstrated its feasibility to grow semi-insulating Mg-doped β-Ga_2O_3 epilayers with controllable Mg incorporation while maintaining good material quality and smooth surface morphology. From capacitance-voltage charge profiling, it is verified that the Mg-doped buffer layer grown at substrate-epilayer interface could effectively compensate the charge accumulation at the interface. The in-situ acceptor doping of Mg in MOCVD β-Ga_2O_3 will provide versatility for designing β-Ga_2O_3 power devices.

Keywords: Ultrawide bandgap, β-Ga_2O_3 thin films, acceptor dopant, metalorganic chemical vapor deposition

β-Ga_2O_3 has unique advantages for high power electronics as well as high power RF applications owing to its ultrawide bandgap (UWBG, 4.8 eV) and consequent high breakdown field (8 MV/cm). [1] The commercially available high quality and scallable native substrate is another key benefit for developing β-Ga_2O_3 based devices. Taking the advantages from the high quality native substrates, high quality epitaxial film growths have been demonstrated by various growth methods [2-9] on different crystal orientations. [5, 6] Despite its UWBG, β-Ga_2O_3 possesses reliable n-type doping properties with a wide range between 10^{16} to 10^{20} cm^{-3}, [2, 6, 10, 11] and good metal contact performances. [12-14] Although p-type doping seems impossible in β-Ga_2O_3, various device designs could still utilize its high-breakdown field strength and achieve normally-off operation. For example, vertical fin-structured field-effect transistors (FET) with the normally-off operation achieved breakdown voltage (BV) > 2 kV. [15] Schottky barrier diode (SBD) with a similar vertical fin structure, can withstand up to 2.89 kV of breakdown voltage (BV) with Baliga's figure-of-merit (BFOM) of 0.80 GW/cm^2 ($BV^2/R_{on,sp}$). [16] In addition, inspired by a current aperture vertical electron transistor (CAVET) in GaN power electronics, recently a vertical β-Ga_2O_3 metal-oxide-semiconductor field-effect transistor (MOSFET) was also demonstrated for normally-off operation. [17-19] One key aspect in these device designs is to introduce charge compensation or current blocking layer (CBL) in the desired device region. Without effective p-type β-Ga_2O_3, an alternative route is to use semi-insulating layer to engineer the electric field in devices. Thus far, there are limited reports on the epitaxy of semi-insulating β-Ga_2O_3, [20-23] and more studies are indispensible.

Charge compensation by in-situ epitaxy has several advantages over the ex-situ ion-implantion process. Epitaxial insulating thin film can be precisely engineered with flexibility during the growth, without the concern of potential damage from ion-implantation or the subsequent thermal annealing process. [24, 25] In β-Ga_2O_3 epitaxy, it is commonly observed the existence of interface charges at the substrate/epilayer growth interface. This phenomenon has been studied in metalorganic chemical vapor deposition (MOCVD) β-Ga_2O_3, in which a spike of Si peak was detected at the growth interface, possibily generated from the substrate polishing process. [2, 3, 20, 26] The interface charges not only induced complications for material characterization but also detrimentally affected device performance such as causing buffer leakage current in lateral power devices. [20] The suppression of this interface leakage current can be practically significant in device performance.

Among various acceptors in β-Ga_2O_3, Mg represents one of the most promising candidates with relatively shallow acceptor level and the lowest formation energy as compared to other cation-site acceptors from DFT calculation. [27] Mg-doped β-Ga_2O_3 bulk crystals have been demonstrated with semi-insulating properties. [28] Mg ion-implantation was also studied in vertical diode structures, [18] in which the high temperature annealing process caused a significant diffusion of Mg profile. On the other hand, the most recent DFT studies on acceptor diffusion suggested that Mg is unlikely to diffuse via Ga vacancy (V_{Ga}), but via an interstitial-assisted process. [29] However, understanding of Mg incorporation and diffusion in in-situ β-Ga_2O_3 epitaxy is still lacking. In MOCVD epitaxy, bis(cyclopentadienyl) magnesium (Cp_2Mg) has been widely used as the Mg precursor for p-type dopants in GaN. [30, 31] With similar MOCVD growth conditions, it is likely that Cp_2Mg can serve as a suitable precursor for Mg-doping of β-Ga_2O_3. With the previous demonstrations of high quality n-type β-Ga_2O_3 via MOCVD [2, 26], the development of in-situ MOCVD of Mg-doping can potentially pave different ways for device designs and device fabrication.

In this study, Mg in-situ doping in MOCVD β-Ga_2O_3 was conducted based on previously established growth conditions for Si-doped MOCVD β-Ga_2O_3, [2] with trimethylgallium (TEGa) and O_2 as Ga, O precursors and Ar as the carrier gas. Mg doping was introduced by using Cp_2Mg as precursor. Chamber pressure was set at 60 Torr in this study. The MOCVD growth temperature for β-Ga_2O_3 was expanded to the range from 650 °C to 900 °C. The growth was conducted on commercial Fe-doped (010) β-Ga_2O_3 substrates. Substrate surface was cleaned with acetone, isopropanol, and de-ionized water prior the growth. Quantitative secondary ion mass spectroscopy (SIMS) was utilized to quantitatively probe the impurity profile of Mg and other impurity elements. Atomic force microscopy (AFM) (Bruker AXS Dimension Icon) was used to characterize the surface morphologies of the as-grown samples. Capacitance-voltage (C-V) measurements were used to probe the charge carrier compensation effect in a lateral Schottky diode structure with Mg-doped buffer layer.

Mg doping concentration as a function of the precursor molar flow rate was studied by quantitative SIMS on a multi-layer stack, as illustrated in Fig. 1(a). The sample was grown at 880 °C, with the Ga molar flow rate set at 31.06 μmol/min, and varied Mg/Ga molar flow rate ratio of 2.1×10^{-4}, 4.2×10^{-4}, and 8.4×10^{-4}. Fig. 1(b) shows the SIMS depth profile of selected emements including Mg, H, and C. The background C concentration shows a level at the detection limit of 5×10^{16} cm^{-3}. The Mg concentration in each sub-layer increases monotonically as the Mg flow rate increases. H impurity concentration exhibits an obvious companion with Mg doping concentration, which indicates a Mg-H complex formation. This is commonly observed in MOCVD grown Mg doped GaN. [30-33] In previous studies, Mg-doped bulk β-Ga_2O_3 with H passivation exhibited infrared (IR) spectra which indicated an O-H bond configuration with possible Mg-O-H complexes [34]. Shown in Fig. 1(b), as the Mg doping reaches the concentration of $>10^{19}$ cm^{-3}, the H concentration shows a trend of saturation. Between each Mg-doped β-Ga_2O_3 sub-layer, the undoped layers are intentionally designed to analyze the Mg diffusion profile. The symmetrical distribution of the Mg profile on both sides indicate that the broadening of the Mg is likely from diffusion.

Following the SIMS characterization as a function of the Mg flow rate, the effects of growth temperature on Mg incorporation and diffusion were analyzed with another designed growth stack, as shown in Fig. 2(a). Three sub-layers of Mg-doped β-Ga_2O_3 were grown with growth temperature of 900, 800, and 700°C with a constant Mg/Ga molar flow rate ratio of 4.2×10^{-4}. From the SIMS profile shown in Fig. 2(b), the Mg incorporation has a minimum dependence on the growth temperature within the investigated temperature regime of 700-900 °C. Instead, the Mg diffusion has a strong dependence on the growth temperature. The temperature-dependent property of the Mg spreading strongly suggests a diffusion mechanism of the Mg impurities in β-Ga_2O_3. Also, from the doped layers, a gradual increase of Mg concentration over time was observed, possibly due to the ramp up of Cp_2Mg precursor delivery to the growth system. Further analysis of Mg diffusion will be discussed in the next section. It is noteworthy that the saturation of H concentration is independent on the growth temperature with the relatively high Mg concentration of $>10^{19}$ cm^{-3}. Also note that the solubility limit of Mg in β-Ga_2O_3 requires further growth studies combined with advanced materials characterization and first principle calculations. Additionally, a trace amount of Fe impurity was detected in the Mg-doped sub-layers, which indicates the Fe incorporation is related to the Mg source flow. This could be related to the coupling between

Cp_2Mg source and the growth system, as stainless steel parts are commonly used in the MOCVD source delivery lines. [35]

To analyze the Mg diffusion at different temperatures of 700 °C, 800 °C, 900 °C, Mg SIMS depth profiles at the end of each Mg doped stack are separately shown in Fig. 3(a)-(c). Fick's law on the diffusion of chemical species requires that: [36]

$$J = -D\frac{\partial c}{\partial x}, \tag{1}$$

$$\frac{\partial c}{\partial t} = D\frac{\partial^2 c}{\partial x^2} \tag{2}$$

where D is the diffusion coefficient of the chemical species, c is the chemical concentration, and J represents the diffusion flux. Also, the change of chemical concentration with respect of time can be expressed in Eq. 2. In this case, the initial Mg doping profile was assumed to be a step function and the total diffusion time was counted by the remainder of the growth duration after the Mg dopant flux was stopped at each temperature. The Mg concentration profiles were fitted by numerically solving Eq. 2 and the diffusion coefficient of 4.82×10^{-16} cm^2/s, 1.04×10^{-15} cm^2/s, and 3.07×10^{-15} cm^2/s was extracted at 700 °C, 800 °C, and 900 °C. The temperature dependence of the impurity diffusion coefficient follows the form of transition state theory: [37]

$$D(T) = D_0 e^{-E_{Barrier}/kT} \tag{3}$$

where $E_{Barrier}$ is the diffusion barrier energy.

The Arrhenius plot on diffusion coefficient versus reciprocal temperature was plotted, as shown in Fig. 3(d). It revealed a diffusion barrier energy $E_{Barrier}$ of ~ 0.9 eV, which is close to the value predicted from first-principle calculations on Mg impurity diffusion in β-Ga_2O_3. [29]

The DFT calculations also suggested that the Mg impurity (Mg_{Ga}) is less likely to diffuse via a Ga vacancy, whereas a Mg_{Ga} and Ga interstitial are more energetically preferred to swap and form Mg interstitials. [29] This interstitial-assisted diffusion process is much less energetically demanding, with small barrier heights of 0.56 eV with the Mg_i to Ga(I) site and 0.75 eV with the Mg_i to Ga(II) site. [29] Therefore, the Mg diffusion in MOCVD β-Ga_2O_3 can be related to these processes. However, in MOCVD of Mg-doped β-Ga_2O_3, H impurity and its related complexes could be another non-negligible factor. Since the H impurity concentration follows closely with Mg concentration (below $\sim 10^{19}$ cm^{-3}). The diffusion processes involving Mg-H complexes cannot be excluded. [34] And the effect of possible complex configurations on diffusion is yet to be theoretically calculated and experimentally studied. Further investigations are still required to identify the diffusion mechanism, as well as the Mg site configuration in MOCVD Mg-doped β-Ga_2O_3.

The Mg SIMS profiling from different growth temperature indicated that a lower growth temperature can suppress the Mg diffusion significantly. The corresponding material characterization on β-Ga_2O_3 thin films grown at lower temperatures is necessary. As shown in Table 1, Si-doped β-Ga_2O_3 thin films were grown under different growth temperatures ranging from 800 to 650 °C with consistent Si target doping at $\sim 1\times10^{17}$ cm^{-3}. Room-temperature Hall measurement reveals comparable Hall mobility values of $\sim$150 $cm^2/V\cdot s$ for samples grown at 700, 750, and 800 °C. For the growth temperature of 650 °C, the mobility was significantly impacted due to possible defects formation and reduced crystalline quality at low growth temperatures. From the SIMS profile in Fig. 2(b), the C impurity level also showed an increasing trend as growth temperature reduces. And the average C concentration was summarized in Table 1. However, the effect of C incorporation on electrical properties is yet to be studied, as DFT calculation indicated a shallow donor state of C in oxide semiconductors. [38] The transport characterization indicates that high-quality homoepitaxy β-Ga_2O_3 could still be achieved at around 700 °C. The effect of growth temperature on surface morphology was also characterized by AFM, as shown in Fig. 4 (a)-(c). The Si-doped homoepitaxial samples with growth temperatures of 700, 750, and 800 °C were characterized, which shows thhat surface RMS roughness increased against the decreased growth temperature. The surface diffusion of adatoms would be more limited at lower growth temperatures and thus led to a greater surface roughness. In Fig. 4 (d), and (e), the impact of Mg incorporation on surface morphology was also studied with samples grown at 700 °C but two different Mg target concentrations. With a moderate Mg doping concentration of ~ 10^{19} cm^{-3}, the RMS value only showed a slight increase as compared to the high-quality Si-doped β-Ga_2O_3 grown at 700 °C. However, as Mg concentration further exceeds above 10^{20} cm^{-3}, a significant deterioration of the surface morphology was observed due to the strong surface segregation from the high source flow rate of Mg. The RMS values and their corresponding growth conditions are listed in Table 2.

To verify the charge compensation effect from Mg doping, a homoepitaxial structure with a Mg-doped buffer layer at the growth interface and a Si-doped channel layer on top was designed, as shown in Fig. 5(a). An UID spacer with 450 nm was grown to separate the potential diffusion of Mg impurity. The Mg doping concentration was designed with ~ 5×10^{18} cm^{-3}, which is higher than the peak Si concentration at the substrate growth interface. [2, 26] The n+ Si-doped channel was doped with a total sheet charge concentration around 3.5×10^{12} cm^{-2}. Ohmic contacts by Ti/Au and Schottky contacts by Ni/Au were consequently deposited on the as-grown samples for C-V measurements. For comparison, a reference sample without the Mg-doped buffer layer was prepared, as illustrated in Fig. 5(b). In Fig. 5(c), the C-V profile shows clear pinch off characteristics in the case of Mg doped buffer sample, while the sample without Mg doped buffer does not pinch off. The extracted charge concentration profile (Fig. 5(d)) also confirmed the depletion of interface charge with the Mg-doped buffer layer, as compared to the sharp interface charge profile shown in the sample without the buffer layer. This clearly demonstrates the effectiveness of Mg-doping for charge compensation, even if H-related complexes might be of neutral charge state. [37] Previous studies of H passivation on Mg-doped β-Ga_2O_3 also suggested that Mg can still effectively provide charge compensation with complexes formed.

In summary, electrically insulating Mg-doped homoepitaxial (010) β-Ga_2O_3 films were demonstrated in as-grown MOCVD epitaxial thin films. The growth conditions for Mg-doped MOCVD β-Ga_2O_3 were established with controllable doping between 10^{18} cm^{-3} to 10^{20} cm^{-3} and a wide growth temperature regime, ranging between 700 °C and 900 °C. While the surface morphology had only minimal impact in films with moderate Mg doping. The companion concentration of H and Mg impurities indicated the formation of Mg-H related complexes in Mg-doped MOCVD β-Ga_2O_3. However, the charge compensation was still effective with the possible existence of complexes across the controllable doping range. The analysis of Mg diffusion profiles revealed a diffusion barrier energy $E_{Barrier} \sim 0.9$ eV. Additionally, the demonstration of low temperature growth of β-Ga_2O_3 at 700 °C with in-situ Mg-doping can significantly suppress the Mg diffusion while maintain high epitaxial quality. The extended growth window to lower temperature regime can be advantageous for in-situ acceptor doping in β-Ga_2O_3. The demonstration of in-situ Mg-doping in MOCVD β-Ga_2O_3 can provide different routes for high-performance device design and device fabrication.

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Data Availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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Table Caption

Table 1. Summary of the MOCVD Si-doped β-Ga_2O_3 samples with different growth temperatures. Samples were all grown with a targeted thickness of 600 nm. The VI/III ratio was kept constant at 1150, and the growth chamber pressure was constant 60 Torr.

Table 2. RMS values representing surface roughness on as-grown samples with different growth temperatures and different target Mg concentration.

Figure Caption

Figure 1. Structures and SIMS impurity profiles of Mg-doped homoepitaxy samples with multiple layers grown at different Mg flow with (a) schmatics on the sample layer stack (b) SIMS profile on the sample. The growth temperature is constant at 880 °C.

Figure 2. Structures and SIMS impurity profiles of Mg-doped homoepitaxy samples with different layers grown at different temperature with (a) schmatics on the sample layer stack (b) SIMS profile on the sample.

Figure 3. Mg diffusion profile (numerically solved) as compared to the actual Mg impurity concentration in the film, with scoped view at different temperature (a) 700 °C, (b) 800 °C, (c) 900 °C, and (d) Arrhenius plot of diffusion coefficient versus 1/T.

Figure 4. Surface AFM images on the surface of all as-grown samples under different temperature, (a) 700 °C, (b) 750 °C, (c) 800 °C as well as with different Mg doping concentration, (d) $\sim 5\times10^{19}$ cm^{-3}, and (e) $> 2\times10^{20}$ cm^{-3}

Figure 5. Structures on homoepitaxy samples, (a) without Mg-doped buffer layer, and (b) with Mg-doped buffer layer. (c) Capacitance-voltage characteristics on the two corresponding structures and (d) extracted charge concentration profile versus depletion width from C-V.

Table 1

Sample	Growth Temperature (°C)	Growth rate (μm/h)	RT Bulk Carrier Concentration (cm^{-3})	RT Hall Mobility ($cm^2/V\cdot s$)	Carbon Concentration (from SIMS)
#1	800	0.74	1.2×10^{17}	150	1×10^{17} cm^{-3}
#2	750	0.72	1.1×10^{17}	152	--
#3	700	0.70	1.7×10^{17}	144	$\sim7\times10^{17}$ cm^{-3}
#4	650	0.68	1.9×10^{17}	60	--

Table 2

Growth Temperature (°C)	Mg-doping (cm^{-3})	Surface roughness RMS (nm)
800	No Mg	3.2
750	No Mg	4.1
700	No Mg	4.7
700	5×10^{19}	5.0
700	$\sim2\times10^{20}$	13.9