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Effects of Drying Temperature and Molar Concentration on Structural, Optical, and Electrical Properties of β -Ga₂O₃ Thin Films Fabricated by Sol–Gel Method

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Abstract: In this study, β -Ga₂O₃ films were fabricated on a quartz substrate by the sol–gel method using different drying temperatures and solutions of different molar concentrations, and their structural, optical, and electrical properties were evaluated. The as-fabricated films exhibited a monoclinic β -Ga₂O₃ crystal structure, whose crystallinity and crystallite size increased with increasing molar concentration of the solutions used and increasing drying temperature. Scanning electron microscopy of the as-prepared samples revealed dense surface morphologies and that the thickness of the films also depended on the deposition conditions. The average transmittance of all the samples was above 8% in visible light, and the calculated optical bandgap energy was 4.9 eV. The resistivity measured using a 4-point probe system was $3.7 \times 10^3 \Omega \text{ cm}$.

Keywords: β -Ga₂O₃; sol–gel; film; solution process; semiconductor

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1. Introduction

Wide-bandgap semiconductors exhibit excellent properties such as high breakdown field, high temperature stability, and high thermal conductivity. Due to these properties, such semiconductors have been investigated for application in various devices that require these properties (e.g., power semiconductors such as Schottky barrier diodes, thin-film transistors, ultraviolet (UV) photodetectors, and gas sensing devices) [1–5].

Silicon carbide (SiC), gallium nitride (GaN), and gallium oxide (Ga₂O₃) are well-known wide-bandgap semiconductor materials [6]. Among them, Ga₂O₃ is a promising candidate material for application in next-generation power devices operated at high voltages and temperatures. Ga₂O₃ has five crystal structures: rhombohedral (α), monoclinic (β), defective spinel (γ), cubic (δ), and orthorhombic (ϵ). Notably, β -Ga₂O₃ ($a = 1.2 \text{ nm}$, $b = 0.3 \text{ nm}$, $c = 0.6 \text{ nm}$, $\beta = 104^\circ$) has a wide bandgap ($E_g = 4.8\text{--}4.9 \text{ eV}$) and a high breakdown field ($E_{br} = 6\text{--}8 \text{ MV/cm}$) [7,8], along with the most stable properties at high temperatures compared to the other crystal structures. The Johnson's figure of merit (JFOM), which is a performance index for maximizing frequency, of β -Ga₂O₃ is 40 times higher than that of Si [9]. In addition, the Baliga's FOM (BFOM), which is a performance index for minimizing conduction loss, of β -Ga₂O₃ is 3444 times higher than that of Si [10]. Therefore, β -Ga₂O₃ films have been investigated for applications in power semiconductors.

Various deposition methods have previously been proposed for the deposition of high-quality β -Ga₂O₃ films including pulsed laser deposition, chemical vapor deposition, metal organic chemical vapor deposition, and radio-frequency sputtering [11–14]; however, they are energy-intensive and require expensive equipment [15]. On the other hand, the solution process for film fabrication has many advantages including low environmental load and cost [15–17]. However, the structural properties of β -Ga₂O₃ are not stable, and the solution process requires an annealing process at temperatures higher than 900 °C; therefore, a solution process for fabricating β -Ga₂O₃ films is required [18].

In this study, β -Ga₂O₃ films were prepared using a sol-gel method, which has advantages of low-temperature processing, uniform coating thickness, and low manufacturing cost [19]. In this process, solutions containing Ga ions at different molar concentrations were spin-coated on a quartz substrate, followed by treatment at different drying temperatures. The molar concentration of the solution and the drying temperature are important factors governing film formation and crystal structure. The thickness, structural, optical, and electrical properties of the film were compared according to each change in the drying temperature and molar concentration of the solution.

2. Experimental Procedure

The quartz substrate (1 cm × 1 cm, thickness: 0.5 mm, iNexus, Inc., Seongnam, Republic of Korea) was ultrasonically cleaned for 10 min with ethyl alcohol (C₂H₅OH, 94%, extra pure, DUKSAN pure chemicals, Ansan, Republic of Korea) and acetone (CH₃COCH₃, 99.5%, extra pure, SAMCHUN pure chemicals, Pyeongtaek, Republic of Korea), followed by rinsing with deionized water. The solution process has poor adhesion between the substrate and film compared to vapor deposition, which may cause peeling off [20]. UV irradiation has been suggested to have more hydroxyl groups attached to the surface, indicating a hydrophilic surface [21]. To improve the adhesion between the film and substrate, the substrate was subjected to UV-C irradiation (G15T8, wavelength: 253.7 nm, 1.4 mW/cm², SANKYO DENKI, Hiratsuka, Japan) for different durations to modify the hydrophilic surface before deposition [22]. Among the substrates irradiated for different irradiation times, those irradiated with UV-C for 24 h exhibited the lowest contact angle of 24.7°. The details of the contact angle data are presented in Figure 1.

Figure 1. Contact angles of the quartz substrate irradiated with UV-C for different irradiation times. (a) Without UV irradiation, (b) 3 h, (c) 6 h, (d) 12 h, (e) 24 h.

The reagents used in this study for the fabrication of β -Ga₂O₃ films were gallium nitrate hydrate (Ga(NO₃)₃·xH₂O, 99.9% pure, supplied by Alfa Aesar, Seoul, Republic of Korea) and polyoxyethylene lauryl ether (non-ionic, Brij-4, Sigma-Aldrich, Seoul, Republic of Korea). Different amounts of water-soluble gallium nitrate hydrate were dissolved in deionized water to form solutions with molar concentrations ranging from 0.5 to 1.0 M. These solutions were added to 0.1 vol.% Brij-4 (used as a surfactant) for the fabrication of β -Ga₂O₃ films [23]. Then, the solution was stirred at 60 °C for 90 min. The gallium solutions with different molar concentrations were drop-casted onto the UV-C-treated quartz substrates, and then the coated films were dried at different temperatures ranging from 60 to 100 °C. This procedure was repeated 10 times. Then, the dried films were annealed in a muffle furnace (DMF-3T, Lab House, Pocheon, Republic of Korea) at 700 °C for 30 min in ambient air. Detailed conditions for film deposition and sample names are listed in Table 1.

Table 1. Conditions for film deposition.

Sample	A	B	C	D	E	F	G	H	I
Drying temperature (°C)		60			80			100	
Precursor concentration (M)	0.5	0.7	1.0	0.5	0.7	1.0	0.5	0.7	1.0

The hydrophilic surface was evaluated by using a contact angle meter (DSA100, Kruss, Seoul, Republic of Korea) and the intensity of UV-C irradiation was measured using a UV-C light meter (UVC-254SD, Lutron, Seongnam, Republic of Korea). The crystallographic properties of the fabricated films were evaluated using X-ray diffraction analysis (XRD, SmartLab, Rigaku, Tokyo, Japan). Scanning electron microscopy (SEM, S-4700, Hitachi, Tokyo, Japan) was used to observe the surface morphologies and cross-sections of the films. Atomic force microscopy (AFM, Park NX10, Park Systems, Suwon, Republic of Korea) was used to evaluate the surface morphologies and roughness of the films. The optical and electrical properties of the fabricated films were evaluated using a UV-Vis spectrometer (Lambda 750 UV-Vis-NIR, Perkin Elmer, Seoul, Republic of Korea) and a 4-point probe system (CMT-SR1000, Changmin Tech, Seongnam, Republic of Korea), respectively.

3. Result and Discussion

3.1. Structural Properties

Figure 2 shows the XRD patterns of β -Ga₂O₃ films fabricated using different drying temperatures and gallium solutions of different molar concentrations. These patterns confirmed that β -Ga₂O₃ films were successfully formed without any impurities by the sol-gel process. In addition, with increasing molar concentration of the gallium solution, the peak intensity increased, implying improved crystallinity of the β -Ga₂O₃ films.

Figure 2. The XRD patterns of the β -Ga₂O₃ films prepared at different drying temperatures: (a) 60 °C, (b) 80 °C, and (c) 100 °C. (d) Crystallite size of the β -Ga₂O₃ thin films fabricated at drying temperatures of 80 and 100 °C.

Figure 2a shows weak peaks at $2\theta = 30.1^\circ$, 31.7° , and 64.7° , which corresponded to the (400), (202), and ($\bar{1}12$) planes, respectively, of monoclinic β -Ga₂O₃, according to the reference data of ICDD card (41-1103). Figure 2a also shows that for a drying temperature of 60 °C, broad peaks were obtained and crystallite size could not be calculated [24]. However, sharper peaks of greater intensity were obtained at higher drying temperatures (Figure 2b,c). Additional peaks appeared at $2\theta \approx 35.2^\circ$ and 38.5° , corresponding to the (111) and ($\bar{3}11$) planes of β -Ga₂O₃, respectively, indicating that crystallization of β -Ga₂O₃ was facilitated by increasing both the molar concentration and the drying temperature. The Scherrer formula was used to calculate the crystallite sizes at the (202) plane, which is the preferential growth plane, as follows:

$$D = \frac{k \lambda}{\beta \cos \theta} \quad (1)$$

Here, k is the Scherrer constant ($=0.9$); D is the crystallite size; λ is the X-ray wavelength ($=0.15406$ nm); θ is the Bragg angle; and β is the full width at half maximum (FWHM) [25]. The crystallite sizes obtained using the above equation increased with increasing molar concentration of the gallium solution (Figure 2d). The increase in molar concentration leads to an increase in Ga concentration on the substrate, which results in faster nucleation and greater condensation of Ga atoms, leading to the growth of larger crystallites [26]. The crystallite size of the samples also increased with increasing drying temperature. This phenomenon may be related to the film thickness [27]. In addition, the micro-strain (ϵ) induced in the thin films due to lattice misfit was calculated using Equation (2).

$$\epsilon = \frac{\beta}{4 \tan \theta} \quad (2)$$

The variations in ϵ are shown in Figure 3. The ϵ value decreased with increasing molar concentration and drying temperature. Compared with Figure 2d, we can conclude that the crystallite size of the thin film decreased with increasing microstrain, which supports the fact that reduction in the quantity of imperfections promotes the formation of larger crystals. Reportedly, a decrease in the microstrain of the film indicates improved crystallinity of the film, along with a reduction in lattice imperfections [28]. Therefore, increasing both the molar concentration and the drying temperature is favorable for the crystallization of monoclinic β -Ga₂O₃.

Figure 3. Strain of the β -Ga₂O₃ films fabricated using solutions of different molar concentrations at drying temperatures of 80 and 100 °C.

The surface morphologies of the β -Ga₂O₃ films fabricated using different drying temperatures and solutions of different molar concentrations are shown in Figure 4. The top-view SEM images show that all the samples exhibited a dense surface morphology regardless of the experimental conditions. In addition, it was confirmed through the SEM images that the surface density increased and became more compact when the molar concentration of the solution was increased at the same drying temperature and also when the drying temperature was increased at the same molar concentration. It has been reported in previous studies [29,30], and it can be seen that a compact thin film can be obtained by increasing the molar concentration and drying temperature. However, the thickness of the samples changed upon varying the experimental conditions, and Table 2 summarizes the thicknesses of all samples fabricated under different conditions. As shown in Table 2, for samples prepared at the same drying temperature, thickness of the β -Ga₂O₃ films increased with increasing molar concentration. Furthermore, for samples prepared at the same molar concentration, the thickness of the β -Ga₂O₃ films increased with increasing drying temperature. These results have been reported in previous papers [31,32], and are closely related to the crystallite size calculated using the Scherrer formula.

Figure 4. SEM images of the as-deposited β -Ga₂O₃ thin films at different molar concentrations and drying temperatures.

Table 2. Thickness values of the samples prepared under different conditions.

Sample	A	B	C	D	E	F	G	H	I
Thickness (nm)	52	65	70	120	130	176	238	254	412

The root-mean-square (RMS) roughness and surface morphologies of the samples were further investigated using AFM. The three-dimensional (3D) AFM images of the β -Ga₂O₃ thin films fabricated under different experimental conditions (Figure 5) showed that all samples possessed flat surface morphologies, and sample A had a low RMS roughness value of 0.2 nm. For samples A, B, and C (drying temperature = 60 °C), the RMS roughness increased with increasing molar concentration. Furthermore, for samples A, D, and G (molar concentration = 0.5 M), the RMS roughness of the thin film increased with increasing drying temperature. These results suggest that using a high molar concentration and a high drying temperature increased the thickness of the β -Ga₂O₃ films, leading to large crystallite sizes and large surface roughness values [33].

Figure 5. 3D AFM images of the as-deposited β -Ga₂O₃ thin films at different molar concentrations and drying temperatures. (a) 0.5 M, 60 °C, (b) 0.7 M, 60 °C, (c) 1.0 M, 60 °C, (d) 0.5 M, 80 °C, and (e) 0.5 M, 100 °C.

3.2. Optical Properties

The UV–Vis spectra shown in Figure 6 revealed that in the wavelength range from 190 to 850 nm, the optical transmittance of the samples in the visible region decreased with increasing molar concentration from 0.5 to 1.0 M, regardless of the drying temperature. Furthermore, in the spectra of samples A, D, and G prepared at a molar concentration of 0.5 M, the transmittance in the visible region decreased with increasing drying temperature, which is attributed to an increase in the thickness of the β -Ga₂O₃ films with increasing drying temperature [34]. Optical transmittance is very sensitive to changes in the surface scattering caused by surface roughness and film thickness [35]. It was found that the absorption edge shifted slightly to longer wavelengths with increasing film thickness, which was due to the change in thickness with increasing crystallite size. Hence, optical scattering may increase due to the increase in surface roughness and film thickness, which may reduce the optical transmittance.

Figure 6d shows the change in the optical bandgap energy of β -Ga₂O₃ film calculated by the Tauc plot (Equation (3)).

$$\alpha h\nu = \beta(h\nu - E_g)^{1/2} \quad (3)$$

Here, $h\nu$ is the photon energy; α is the absorption coefficient; and β is a fixed constant. The Tauc plot shows that the optical bandgap energy is estimated by extrapolating the linear section to the axis energy. It was found that the optical bandgap energy decreased from 4.9 to 4.1 eV with increasing molar concentration and drying temperature. Among the as-prepared β -Ga₂O₃ films, sample A deposited at 0.5 M and 60 °C showed the highest

optical bandgap energy (4.9 eV), which is consistent with previously reported values for β -Ga₂O₃ [36–38]. However, the optical bandgap energies of the samples decreased with increasing molar concentration as well as drying temperature. Reportedly, the optical bandgap energy depends on the thickness of the films [39]. This is in good agreement with the reports on films prepared by direct current magnetron sputtering [40], radio frequency sputtering [41], spin pyrolysis [42], and ion beam sputtering [43].

Figure 6. Optical properties of the as-deposited samples prepared at different molar concentrations and drying temperatures. Transmittance (%) values of different samples at drying temperatures of (a) 60 °C, (b) 80 °C, and (c) 100 °C. (d) Optical bandgap energies of samples prepared at different drying temperatures using different molar concentrations.

As above-mentioned, it was confirmed that the crystallite size and thickness of the β -Ga₂O₃ films increased as the molar concentration and drying temperature increased, which might be one of the reasons for the decreased optical bandgap energy [44].

3.3. Electrical Properties

Figure 7 shows that the electrical resistivity of the as-deposited samples, measured by a 4-point probe system, increased with increasing molar concentration and drying temperature. The fabricated films had high resistivities in the range from 3.7×10^3 to $5.1 \times 10^4 \Omega \text{ cm}$, regardless of molar concentration and drying temperature, and film thickness. However, a high resistivity implies a low electrical conductivity, according to the equation $\rho = 1/\sigma$, which is due to the large optical bandgap energy. Therefore, this high resistivity hinders practical applications that require conduction response [45].

Figure 7. Electrical resistivities of all samples.

Based on these results, we plan to improve the electrical properties (i.e., lower the electrical resistivity) by raising the annealing temperature or by doping with Sn, Ti, etc.

4. Conclusions

The β -Ga₂O₃ films were successfully fabricated on a quartz substrate by spin-coating, and the effects of drying temperature and molar concentration of the starting solution on the film properties were investigated. With increasing molar concentration and drying temperature, crystallinity of the β -Ga₂O₃ thin film increased gradually, with a crystallite size of ~21.1 nm. All the films showed a dense surface morphology, and their roughness and thickness both increased with increasing molar concentration and drying temperature. Sample A showed a high transmittance of over 80% to visible light, and the corresponding optical bandgap energy calculated from the Tauc plot was 4.9 eV. The transmittance and optical bandgap energy decreased due to the increase in thickness according to the experimental conditions. The electrical resistivity measurement results showed that the films exhibited a high resistivity ranging from 3.7×10^3 to 5.1×10^4 Ω cm. It is difficult to use it as an n-type semiconductor at present due to its high resistivity. However, due to its wide band-gap energy and other features, it is used as a depletion layer in semiconductor devices, and is suitable for power electronics applications such as pin diodes, Schottky barrier diodes and thin-film transistors. In future studies, we plan to improve the electrical properties of these films (i.e., lower their resistivities by using doping or annealing methods).

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