

Preparation and lithium doping of gallium oxynitride by ammonia nitridation via a citrate precursor route

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Abstract

Gallium oxynitride, isostructural to hexagonal gallium nitride (h-GaN), was obtained by ammonia nitridation of a precursor prepared from the addition of citric acid to an aqueous solution of gallium nitrate. Gallium oxynitride produced at 750 °C had a small amount of gallium vacancies, and was formulated as $(\text{Ga}_{0.89}\square_{0.11})(\text{N}_{0.66}\text{O}_{0.34})$ where the symbol \square stands for gallium vacancy. Both the gallium vacancies and oxygen substituted for nitrogen were randomly distributed within the structure. The amount of vacancies decreased with nitridation temperatures in the range of 750–850 °C. Approximately, 10 at% Li^+ was doped into the gallium oxynitride, using a similar preparation with the additional presence of lithium nitrate, resulted in the random substitution of Ga^{3+} in an atomic ratio of $\text{Li}/\text{Ga} < 1$ at 750 °C. Oxygen was codoped with lithium and substituted nitrogen in the wurtzite-type crystal lattice. These substitutions reduced the electrical conductivity in the gallium oxynitride semiconductor. A new oxynitride, $\text{Li}_2\text{Ga}_3\text{NO}_4$, was also obtained with Li_2CN_2 impurity using similar preparations from a mixture of $\text{Li}/\text{Ga} \geq 1$. The crystal structure was isostructural with h-GaN, and was refined as $P6_3mc$ with $a = 0.31674(1)$ nm, and $c = 0.50854(2)$ nm. The Ga and Li occupancies at the $2b$ site were refined to be 0.6085 and 0.3915, respectively, assuming that the other $2b$ site was randomly occupied with 1/5O and 4/5N. When the new compound was washed for over 1 min for the removal of Li_2CN_2 impurities, it was decomposed to a mixture of $\alpha\text{-GaOOH}$ and $\alpha\text{-LiGaO}_2$. The as-prepared product with $\text{Li}/\text{Ga} = 1$ showed the highest intensity in yellow luminescence among the products under excitation at 254 nm.

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1. Introduction

Semiconductor materials with a wide band gap have many applications. Gallium nitrides are one attractive type of gallium-based semiconductor, and have been widely used in optoelectronic devices, such as blue light emitting diodes (LEDs) and laser diodes [1]. $\beta\text{-Ga}_2\text{O}_3$ with a direct band gap of 4.7 eV has also been a candidate as an LED material for ultraviolet (UV) applications [2]. Various other Ga_2O_3 phases have gained interest for gas sensor applications [3]. Gallium oxynitride is an intermediate between the nitride and the oxide. Its polymorph with a ccp anion

arrangement was reported as an ammonia reaction product with gallium double oxides such as GaFeO_3 , NiGa_2O_4 and so on [4]. A large response for the gas sensing of ethanol and water was recently reported for such gallium oxynitrides [5]. A gallium oxynitride, $\text{Ga}_3\text{O}_3\text{N}$, with a spinel structure was theoretically predicted [6], and recently synthesized using high pressure techniques [7]. Although the direct band gap for $\text{Ga}_3\text{O}_3\text{N}$ was calculated to be 4 eV, its photoluminescent signal began below 2.5 eV and extended to 1.5 eV. The former gallium oxynitride with a wurtzite-type structure was also reported on a MOCVD film deposited on a GaAs substrate [8]. Its powdered product was also briefly reported in a preparation of a manganese doped product from the ammonia nitridation of an oxide precursor through the citrate route [9]. There

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are possibilities to partially substitute the gallium with many kinds of other elements in this preparation method to add new properties for the semiconductor.

Manganese-doped gallium nitride is theoretically expected to behave as a diluted magnetic semiconductor at room temperature [10]. The theoretical prediction stimulated many experimental researches especially on thin film. Researches on bulk materials had been required because the results on thin film were very scattered and misleading [9]. Finally, paramagnetic crystals of GaN doped with $\text{Mn}^{2+} < 0.35 \text{ at\%}$ were grown by a Na flux method [11]. The doping limit itself was very narrow to GaN in high purity. A large amount of manganese could be doped to the wurtzite-structure gallium oxynitride; 10 at% at 750 °C and 5 at% at 850 °C [9], and the products were paramagnetic at room temperature. The preparation through citrate route led to the wide solid solution range on the gallium oxynitride.

Many kinds of element might be doped to the gallium oxynitride to hybridize new properties for the semiconductor. Lithium doping may affect its gas sensing behavior and electrical property as reported on a similar doping on zinc oxide [12]. The addition of lithium has been reported to promote nitrogen dissolution in a Ga–Na melt for the growth of GaN single crystals [13]. A liquid-phase epitaxy of GaN has been reported from a Ga–Li melt [14]. The preparation of GaN crystals by the reaction of Ga_2O_3 with Li_3N has also been reported [15]. These preparations suggest that lithium is not incorporated into the GaN crystal lattice. There have been several reports of compounds containing Li, Ga and N together. For example, the double nitride Li_3GaN_2 , with an anti-fluorite superstructure, has been reported [16]. The possible presence of Li_4GaNO_2 during the thermal reaction of Ga_2O_3 with Li_3N and of LiGaO_2 with Li_3N had been also discussed [17].

In the present study, lithium was doped into a wurtzite structure gallium oxynitride by the ammonia nitridation of oxide precursors obtained through the citrate route. A new oxynitride, $\text{Li}_2\text{Ga}_3\text{NO}_4$, was obtained in a similar preparation method in the presence of an excess amount of lithium.

2. Experimental

Gallium nitrate hydrate $\text{GaNO}_3 \cdot n\text{H}_2\text{O}$ ($n = 7\text{--}9$) of 3 N purity and citric acid with higher than 98% purity were purchased from Wako Pure Chemicals. Both $\text{GaNO}_3 \cdot n\text{H}_2\text{O}$ (1.0 g) and citric acid (0.75 g) were dissolved in 50 mL of distilled water. The mixed aqueous solution was heated on a hot plate with stirring. The gel obtained was fired at 350 °C for 60 min to yield a brown colored amorphous precursor. After grinding, the gel was nitrided at 750–850 °C in a mullite boat using a 50 mL/min flow of ammonia, yielding a light yellow colored product. For lithium doping, various amounts of lithium nitrate ($\text{Li/Ga} < 1$) were added to an aqueous solution with equimolar amounts of citric acid to the total cations. The products were light yellow colored. In preparations with an

atomic ratio of $\text{Li/Ga} \geq 1$, the pelletized precursor was nitrided on another pellet without lithium to prevent the direct reaction with the mullite boat. The white colored products formed were contaminated with an impurity of Li_2CN_2 , which was removed by washing with water. The powdered product (0.12 g) was dispersed by stirring with ca. 40 mL of distilled water for less than a minute. The dispersion was dried at 110 °C for several minutes after centrifugation at 6000 rpm.

Powder X-ray diffraction (XRD) was measured using a diffractometer (Panalytical, X'pert-MPD) with monochromatized $\text{CuK}\alpha$ radiation. A laser Raman spectrometer (Jasco, TRS-401) was used to study the structural features of the product. A green laser (Showa Optronics, 532-100S) was used to generate an excitation beam at $\lambda = 532 \text{ nm}$. Both gallium and lithium content were determined using an inductively coupled plasma-atomic emission spectrometer (ICP-AES, Shimadzu, ICPS-1000 IV) after appropriate dilution of ca. 15 mg samples dissolved in 1 mL H_3PO_4 at 100 °C. The oxygen and nitrogen content were measured estimating the amount of N_2 and CO_2 released after thermal decomposition in carbon crucible in high temperature, using Horiba elemental analyzer (EMGA, EF-600). CHN recorder (Yanaco, MT-5, -6) was also applied to check the residual amount of carbon by firing the samples in oxygen atmosphere.

Electrical properties; inductance, capacitance and resistance, were measured with pellets of the samples using an LCR meter (Yokogawa Hewlett Packard, 4247) at 1 kHz at -50 to 100 °C. The pellets (ca. 5 mm diameter, 2 mm thick) were sintered in an ammonia flow at 750 °C for 10 h after cold isostatic pressing at 100 MPa. Both sides of the pellet were coated with silver paste as electrodes. Photoluminescence of powder samples was measured using a fluorescence spectrometer (Jasco, FP-6200) with a Xe lamp in the wavelength range between 400 and 720 nm. A UV visible spectrometer (Hitachi V-550) was used to measure the diffuse reflectance spectra.

3. Results and discussion

3.1. Lithium doping to gallium oxynitride in low lithium content

The nitrided products without Li were wurtzite-type gallium oxynitrides. The crystallinity of the oxynitrides was improved with increase in the nitridation temperature [9]. The lattice parameters, calculated using well-resolved diffraction lines (102, 110 and 103), were $a = 0.3189 \text{ nm}$, $c = 0.5181 \text{ nm}$, and were independent of the nitridation temperature. The a -value was the same as h-GaN, but the c -value was slightly smaller than that for h-GaN ($a = 0.3189 \text{ nm}$, $c = 0.5186 \text{ nm}$) [18], due to a small amount of gallium defects in $(\text{Ga}_{0.89}\square_{0.11})(\text{N}_{0.66}\text{O}_{0.34})$ as reported in our previous manuscript [9]. Hereafter the symbol \square denotes the gallium vacancy. Slightly expanded lattice parameters, $a = 0.323 \text{ nm}$, $c = 0.528 \text{ nm}$ had been

reported for an isostructural thin film deposited on a GaAs substrate [8].

All Raman peaks were broad and shifted slightly toward lower wavenumber in comparison to those for h-GaN, as shown in Fig. 1. This is probably due the partial substitution of nitrogen with oxygen. The shape of the spectra supports that the present products are isostructural with h-GaN and have a space group of $P6_3mc$, because of

their mutual similarities. The peak shifts from those observed for h-GaN can be understood by the reduced bonding strength between gallium and anions with the partial substitution of N^{3-} with O^{2-} . The wavenumbers were moved closer to those of h-GaN with increasing nitridation temperature. $E_1(\text{TO})$ and $A_1(\text{LO})$ modes appeared at 550 and 720 cm^{-1} , respectively, on the product at 850 °C. There were no peaks observed for Ga_2O_3 impurity in the present spectra [21]. Similar broadbands centered at 560 and 733 cm^{-1} have been reported for a MOCVD thin film of gallium oxynitride [8]. These

Table 1

Chemical composition of gallium oxynitrides without Li and with 5 at% Li prepared at 750–850 °C

Preparation condition	N/ wt%	O/ wt%	Li/ wt%	Ga/ wt%	Total/ wt%
Li 0%, 750 °C	11.4	6.77	0	77.9	96.1
Li 0%, 800 °C	12.8	5.47	0	79.6	97.9
Li 0%, 850 °C	13.7	3.78	0	80.7	98.2
Li 5%, 750 °C	7.95	12.9	0.41	72.9	94.2
Li 5%, 800 °C	10.3	8.93	0.45	76.4	96.1
Li 5%, 850 °C	12.6	5.83	0.35	77.5	96.3

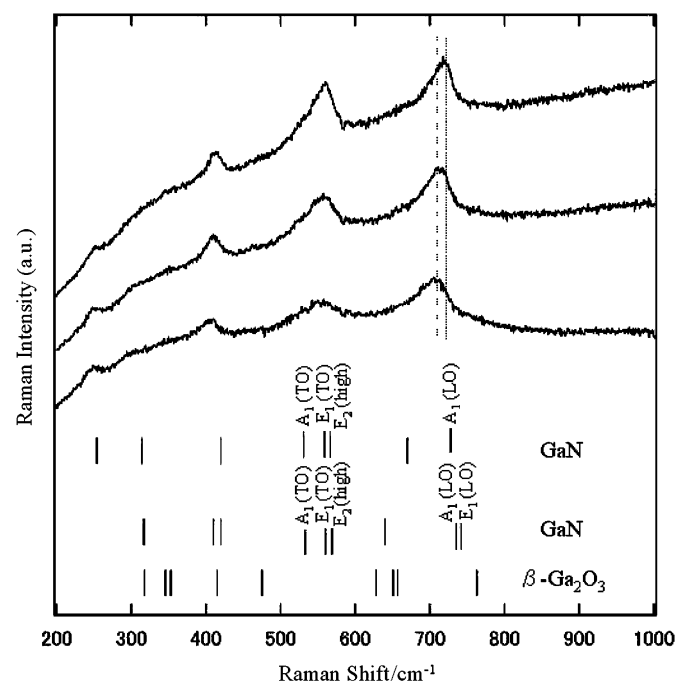


Fig. 1. Raman spectra of gallium oxynitride nitrided at (a) 750 °C, (b) 800 °C and (c) 850 °C. Peak positions for h-GaN and $\beta\text{-Ga}_2\text{O}_3$ in Refs. [19–21] are shown below the spectra.

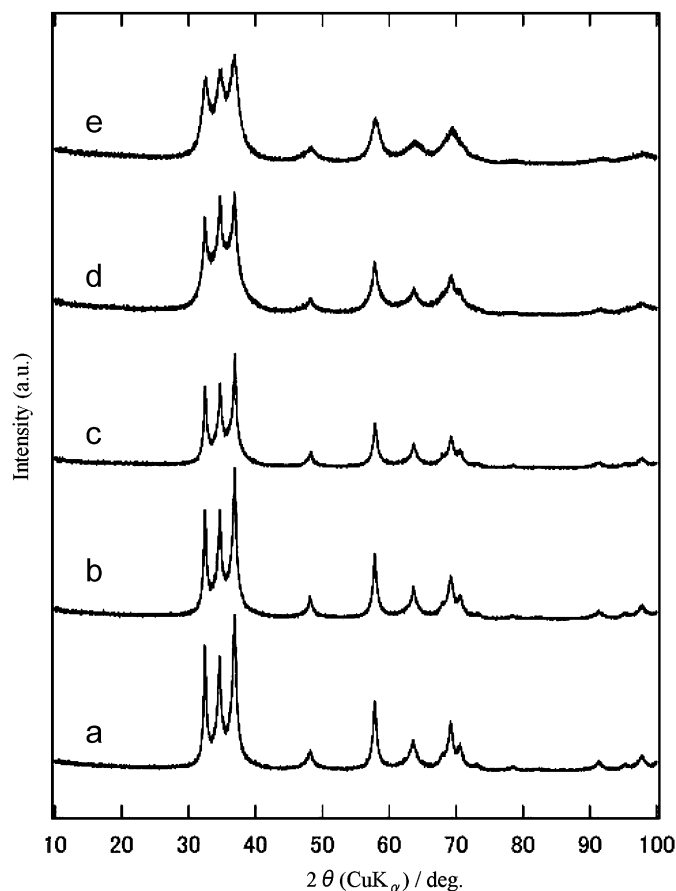


Fig. 3. X-ray diffraction patterns for gallium oxynitrides doped with various amount (x) of lithium prepared at 750 °C in low lithium content region with $x = 0$ (a), 0.05 (b), 0.08 (c), 0.10, (d) and 0.20 (e).

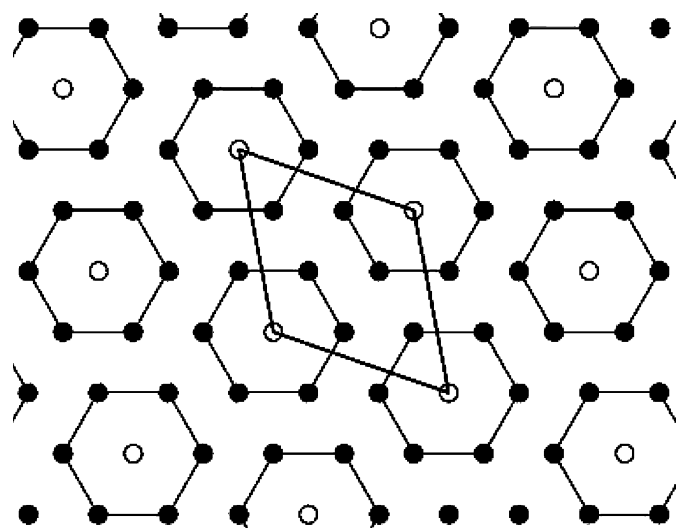


Fig. 2. Schematic gallium vacancy distribution within a Ga-plane in wurtzite-type gallium oxynitride. Closed and open circles represent gallium and its vacancy sites, respectively. This is the most probable case among various kinds of statistical vacancy distribution where Ga/vacancy = 6/1 in atomic ratio.

wavenumbers are slightly larger than the present values although the crystal lattice is a little expanded as described in the previous section. Chemical analysis revealed a composition of Ga: 77.9 wt%, N: 11.4 wt% and O: 6.77 wt% present in the nitrided product at 750 °C. The gallium vacancy was randomly distributed in the $(\text{Ga}_{0.89}\square_{0.11})(\text{N}_{0.66}\text{O}_{0.34})$ oxynitride with wurtzite-type lattice because no superlattice was observed from the XRD pattern. The vacancies are statistically distributed as far apart as possible from each other in the gallium plane, as shown by the example in Fig. 2. The nitrogen content increased and the oxygen amount decreased with increase in the nitridation temperature, as summarized in Table 1.

The nitrided products with 5 at% Li showed similar XRD patterns to that of the wurtzite-type gallium oxynitride without Li, as shown in Fig. 3(a) and (b). The diffraction lines became sharper with an increase in the nitridation temperature. The a -lattice parameter was again almost constant, but the c -value was slightly changed with the change in nitridation temperature; $a = 0.3188$ nm, $c = 0.5172$ nm at 750 °C, $a = 0.3189$ nm, $c = 0.5176$ nm at

800 °C, and $a = 0.3188$ nm, $c = 0.5177$ nm at 850 °C. The nitrogen content increased and oxygen amount decreased with increase in the nitridation temperature. However, nitrogen decreased and oxygen increased with Li-doping, as shown in Table 1. As lithium is doped, it appears that oxygen substitutes for nitrogen in the lattice. The chemical composition can be formulated as $(\text{Li}_{0.04}\text{Ga}_{0.79}\square_{0.17})(\text{N}_{0.41}\text{O}_{0.59})$ for the nitrided product at 750 °C. Semiconducting electrical resistivity increased from 3×10^5 to $7 \times 10^5 \Omega\text{cm}$ at room temperature with lithium doping, probably because the donor electrons in the gallium oxynitride were trapped by the holes formed from the substitution of gallium with lithium.

Gallium oxynitrides were prepared by doping various amounts of lithium at 750 °C. The crystallinity of the samples deteriorated with increase in the amount of lithium, as shown in Fig. 3. However, all the diffraction peaks were indexed to the wurtzite-type structure. Both a - and c -lattice parameters decreased with doping of a smaller amount (x) of lithium, as depicted in Fig. 4. The gradients of the lattice parameters changed at

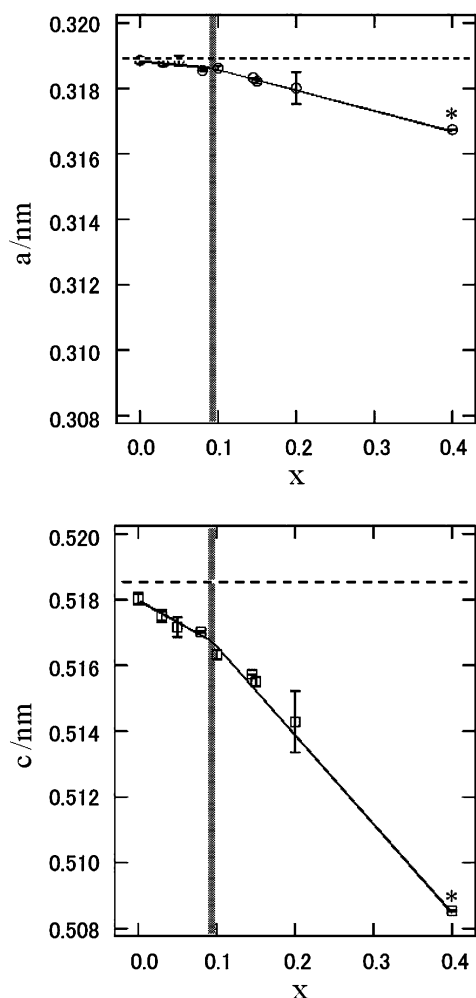


Fig. 4. Hexagonal lattice parameters a and c of gallium oxynitride against doped lithium amount x . Dotted lines and asterisks represent values for h-GaN in Ref. [18] and $\text{Li}_2\text{Ga}_3\text{NO}_4$ described below in the text, respectively.

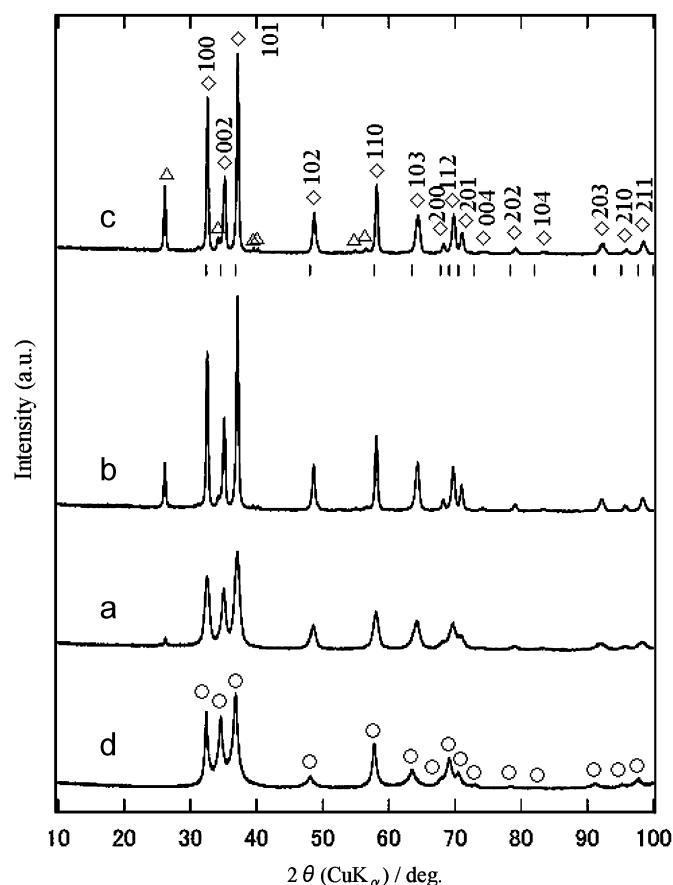


Fig. 5. X-ray diffraction patterns for gallium oxynitrides prepared at 750 °C with various amount of lithium in high lithium content: (a) Li/Ga = 1, (b) Li/Ga = 2, (c) Li/Ga = 3, and (d) without lithium as a reference ($x = 0$). Square \square , circle \circ and triangle \triangle represent diffraction lines for new compound, gallium oxynitride and Li_2CN_2 , respectively. Diffraction peak positions for h-GaN [18] are marked below (c).

approximately $x = 0.1$, probably because the new isostructural compound mentioned in the next section may have already started to appear.

3.2. Preparation of a new compound with high lithium content

Similar oxynitride preparations, via the citrate route, were performed for high lithium content with an atomic ratio of $\text{Li}/\text{Ga} = 1\text{--}3$. The products formed at 750°C were mixtures of a new compound with Li_2CN_2 , as represented in Fig. 5. The crystallinity of the new compound was improved with an increase in the amount of lithium. An excess amount of lithium might act as a flux as well as

contribute as a constituent. The product mixture was washed with water to remove Li_2CN_2 impurity. No change was observed on XRD of the new compound after 1 min washing. The new compound gradually decomposed to a mixture of $\alpha\text{-GaOOH}$ and $\alpha\text{-Li}_2\text{GaO}_2$ in further washing as shown in Fig. 6. The new compound had a similar diffraction pattern to that of h-GaN [18], but with a slight shift in the peak positions. The lattice parameters were calculated by a least squares refinement as $a = 0.3167(1)\text{ nm}$ and $c = 0.5072(3)\text{ nm}$, using the 102, 110 and 103 diffraction lines.

The chemical composition was analyzed for the product prepared with $\text{Li}/\text{Ga} = 3$ after removing the Li_2CN_2 impurity in washing for 1 min, and was Li: 3.8 wt%, Ga: 65.3 wt%, N: 4.87 wt%, O: 20.4 wt%. The analysis was performed in two separate batches for Li, Ga and O, N, because the sample amount was very limited for one batch. The composition was formulated as $\text{Li}_2\text{Ga}_3\text{NO}_4$. The crystal structure was refined as wurtzite type using a Rietveld refinement as represented in Fig. 7. Program Rietan was used for the refinement [24]. The nitrogen and oxygen atoms were randomly distributed in a molar ratio of 1:4 at the $2b$ site of $P6_3mc$. After the refinement, the occupancies of Ga and Li in the other $2b$ site were found to be 60.85% and 39.15%, respectively, as summarized in

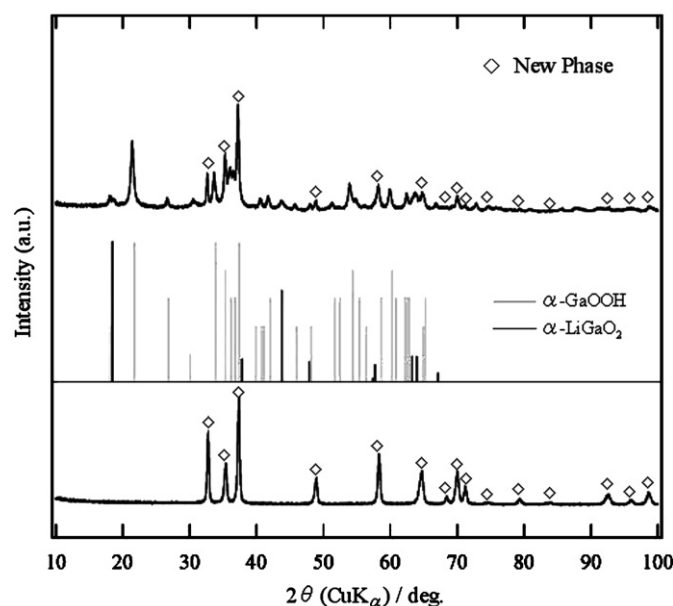


Fig. 6. X-ray diffraction patterns of the new compound obtained with $\text{Li}/\text{Ga} = 3$ (bottom) and its hydrolyzed product for 20 min (top). Diffraction data are in Refs. [22,23].

Table 2

Fractional atomic coordinates and isotropic displacement parameter for $\text{Li}_2\text{Ga}_3\text{NO}_4$

Atom	Position	x	y	z	Occupancy	$B/10^{-2}\text{ nm}^2$
Ga	$2b$	$1/3$	$2/3$	0	0.6085(5)	0.76(1)
Li	$2b$	$1/3$	$2/3$	0	0.3915(5)	0.76(1)
N	$2b$	$1/3$	$2/3$	0.3802(4)	0.2	1.30(4)
O	$2b$	$1/3$	$2/3$	0.3802(4)	0.8	1.30(4)

Space group $P6_3mc$, $a = 0.31674(1)\text{ nm}$, $c = 0.50854(2)\text{ nm}$. $R_{\text{wp}} = 6.33\%$, $R_e = 4.95\%$, $S = 1.28$.

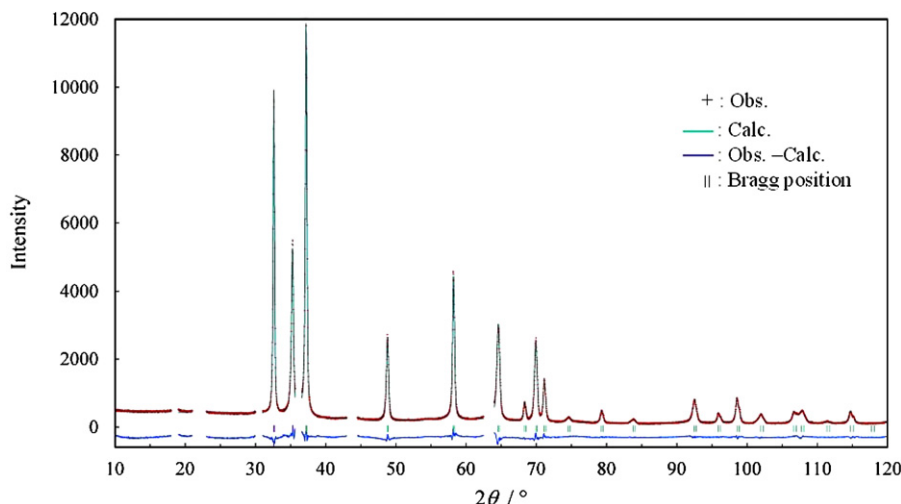


Fig. 7. Rietveld refinement of X-ray diffraction pattern for $\text{Li}_2\text{Ga}_3\text{NO}_4$.

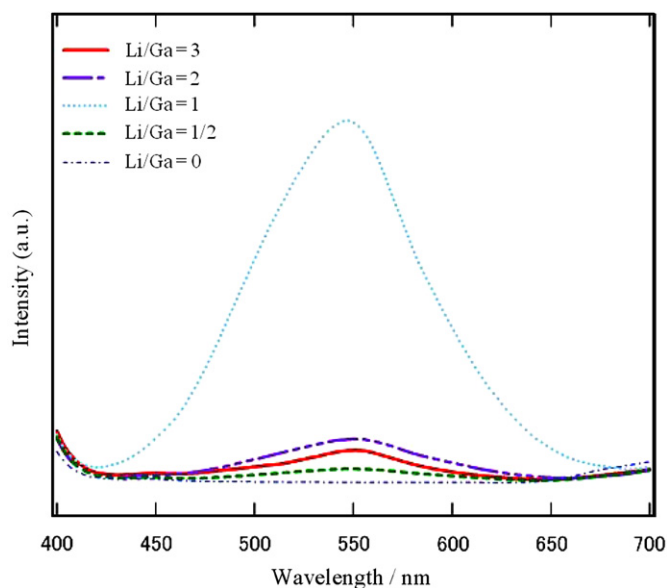


Fig. 8. Photoluminescence spectra of gallium oxynitrides prepared at 750 °C with copresence of various amount of lithium. Excitation wavelength was 254 nm.

Table 2. The refined lattice parameters were $a = 0.31674(1)$ nm, $c = 0.50854(2)$ nm. Both a and c lattice parameters slightly shrank with the partial substitution of Ga^{3+} with the smaller Li^+ from $a = 0.3189$ nm and $c = 0.5186$ nm for h-GaN [18].

The nitrided $\text{Li/Ga} = 3$ product had absorption edges at 355 and 305 nm before and after washing with water, although Li_2CN_2 gave an absorption edge at 240 nm. Yellow photoluminescence was observed at an excitation wavelength of 254 nm for the products before washing with water. The highest intensity was observed for $\text{Li/Ga} = 1$, as shown in Fig. 8. Neither Li_2CN_2 nor the present nitride products with low lithium content displayed this luminescence. Origin of the yellow luminescence is $\text{Li}_2\text{Ga}_3\text{NO}_4$. A similar yellow luminescence has been reported for MOCVD-GaN films [25,26]. The broad transition was centered at 2.2–2.3 eV, and has been assumed to be generated by gallium vacancies or by the complex formed by gallium with the oxygen substituted nitrogen [27].

4. Summary

In summary, gallium oxynitride was doped at low concentration up to approximately 10 at% into wurtzite-type gallium oxynitride with gallium vacancies associated to a partial substitution of nitrogen with oxygen. The amount of vacancies was increased by gallium substitution with lithium, because oxygen atoms were codoped by the substitution of nitrogen. The electrical resistivity was increased with lithium doping. A new oxynitride $\text{Li}_2\text{Ga}_3\text{NO}_4$ was obtained by ammonia nitridation of the precursor for the region with high lithium concentration, $\text{Li/Ga} > 1$. The new oxynitride is isostructural to h-GaN and displayed yellow luminescence at an excitation of 254 nm.

Acknowledgments

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