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Synopsis: A fully merged multiple epitaxial lateral overgrowth (multiple-ELOG) GaN thin film with a thickness of only several hundred nanometres has been grown using a graphene mask that decomposes during the growth process. A mechanism for multiple-ELOG through self-decomposing graphene due to GaN template decomposition during homoepitaxy is proposed.

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Keywords: gallium nitride; epitaxial lateral overgrowth; graphene; metal organic chemical vapor deposition

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Multiple epitaxial lateral overgrowth of GaN thin films using a patterned graphene mask by metal organic chemical vapor deposition

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Single-crystal gallium nitride (GaN) thin films were grown using a graphene mask via multiple epitaxial lateral overgrowth (multiple-ELOG). During the growth process, the graphene mask self-decomposed to enable the emergence of a GaN film with a thickness of several hundred nanometres. This is in contrast to GaN selective area growth using an SiO₂ mask, which leads to the well known hexagonal-pyramid shape under the same growth conditions. The multiple-ELOG GaN had a single-crystalline wurtzite structure corresponding to the crystallinity of the GaN template, which was confirmed with electron backscatter diffraction measurements. An X-ray diffraction rocking curve of the asymmetric 102 reflection showed that the FWHM for the multiple-ELOG GaN decreased to 405 from 540'' for the underlying GaN template. From these results, the self-decomposition of the graphene mask during ELOG was experimentally proven to be affected by the GaN decomposition rather than the high-temperature/H₂ growth conditions.

1. Introduction

Gallium nitride (GaN) has attracted steady interest as a promising semiconductor material for light-emitting diodes (LEDs) (Ponce & Bour, 1997), especially micro-LEDs (Day *et al.*, 2011; Kang *et al.*, 2018; Lee *et al.*, 2018; Sekiguchi *et al.*, 2019; Gou *et al.*, 2019), because of its excellent electrical and optical properties. Unsurprisingly, high-quality GaN is required for the high efficiency of these devices. GaN, which rarely exists in nature, is generally grown on a lattice-mismatched substrate such as sapphire. High-density threading dislocations (TDs) caused by large differences in the lattice constants and thermal expansion act as a non-radiative recombination center (Sugahara *et al.*, 1998) and a channel for electrical leakage (Li *et al.*, 2004). The latter is considered to be a major factor deteriorating the characteristics of a device, along with heat dissipation due to the low thermal conductivity of sapphire (Hornig *et al.*, 2008). As one of the most promising technique for lowering the number of TDs, epitaxial lateral overgrowth (ELOG) has been performed to block their formation by using a dielectric mask such as SiO₂ or SiN_x (Zheleva *et al.*, 1997; Mukai *et al.*, 1998; Nakamura *et al.*, 1998; Gibart, 2004; Kappers *et al.*, 2007).

In this study, graphene was used as an effective mask for the ELOG of GaN instead of a typical dielectric mask. Graphene

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has already been widely studied for GaN-based LEDs owing to its excellent properties: transferable/flexible application with mechanical properties induced from its '2D' structure (Chung *et al.*, 2010, 2016); excellent heat dissipation with high thermal conductivity (Han *et al.*, 2013; Lee *et al.*, 2012); and **ability to form** electrodes with high intrinsic mobility and transparency (Jo *et al.*, 2010, Xu & Liu, 2016). For the epitaxial growth of GaN on graphene, many early studies have been carried out with additional processing involving, for instance, ZnO (Chung *et al.*, 2010, 2016) and carbon nanotubes (Seo *et al.*, 2015), because of the low chemical reactivity induced by the stable in-plane sp^2 bonding of graphene. More recently, remote epitaxy – the epitaxy of crystalline material on 2D materials by interacting remotely with underlying substrates – has been reported for III–V compound semiconductors on graphene (Kim *et al.*, 2017; Kong *et al.*, 2018), which has many parallels with the 'wetting transparency' of graphene (Rafiee *et al.*, 2012). These results have aroused great interest in the possibilities of using graphene as a mask for ELOG of GaN, and it is also expected that graphene **could be useful in the manufacture of** GaN-based devices owing to its high thermal and electrical conductivity.

In this work, we conducted a study on applying multiple-ELOG on a GaN template using a graphene mask, and, as a result, a thin GaN film of around 400 nm thickness was obtained after 10 min of multiple-ELOG. In the ELOG of GaN with a typical dielectric mask, GaN films must be grown to a thickness of more than a few micrometres for a few hours to obtain a smooth surface owing to the limitation of the relative growth rates of each facet of GaN **[missing reference?]**. This suggests the potential of obtaining a much thinner GaN film via multiple-ELOG with graphene, which can result in lower cost, lower strain and higher heat dissipation. In an X-ray diffraction (XRD) analysis of the template after multiple-ELOG, the FWHM value of the asymmetric 102 reflection peak was reduced to 405 from 540'', which indicates reduced edge dislocations owing to applying the multiple-ELOG process (from the relationship between the TD densities and the FWHM values; Koleske *et al.*, 2001). The single-crystalline wurtzite structure of GaN was confirmed via electron backscatter diffraction (EBSD) mapping. Moreover, we propose a mechanism explaining multiple-ELOG using a patterned graphene mask on a GaN template.

2. Methods

2.1. Graphene transfer and mask patterning

Graphene grown on a Cu foil using low-pressure chemical vapor deposition (CVD) was transferred onto a c-plane **GaN or sapphire** template. For the transfer process, poly(methyl-methacrylate) (PMMA) was spin-cast onto one side of the graphene grown on Cu foil. An $FeCl_3$ -based Cu etchant solution was used to etch the Cu foil for 1 h using the PMMA as a handle layer. During several deionized water rinses, the PMMA-coated graphene was held on the water surface by surface tension. It was then transferred onto **either a GaN**

template or a sapphire substrate and dried at room temperature with ventilation for 1 day. The PMMA handle layer was then dissolved in acetone at 353 K for 40 min. The transferred graphene showed a high intensity ratio of the $2D/G$ peaks of about 2 in the Raman spectra, which is one of the spectral features of single-layer graphene that distinguishes it from multi-layer graphene (**Fig. S1 [please provide the supporting information file; we do not have a copy of this]**) (Liu *et al.*, 2013). Afterwards, a pattern of holes with a diameter of 2 μm and a period of 4 μm was formed by photolithography, which was transferred to the graphene using reactive ion etching with oxygen plasma. Finally, the photoresist mask was removed using acetone.

An SiO_2 mask was also prepared for the purpose of comparison. A 10 nm-thick SiO_2 layer was deposited by plasma-enhanced CVD. The subsequent mask patterning was the same as described above for the graphene mask.

2.2. GaN epitaxial lateral overgrowth

The ELOG of GaN was performed **either on a GaN or a sapphire** template with hole-patterned CVD graphene (**pGR**) in a close-coupled showerhead metal–organic chemical vapor deposition (MOCVD) reactor. **GaN was not grown on the GaN template and sapphire substrate with pGR in same run.** We used ammonia and trimethylgallium, respectively, as the precursors for the N and Ga sources to grow GaN. The growth proceeded in only one step: at 1323 K at 100 Torr (13.3322 kPa) in H_2 ambient for 1, 4, 10 and 60 min. This differs from the process for conventional epitaxial lateral overgrowth for flat films, which has several steps. **For the growth on the sapphire substrate with pGR, we added the growth of a buffer layer at 823 K for 100 s under 100 Torr for nucleation on the lattice-mismatched sapphire substrate. [This seems to contradict the previous statement that only one step is used; please clarify.]** However, these conditions did not affect the graphene or the results, **even when equally applied to the GaN template. [Please clarify what this means; was the buffer layer also grown on the GaN template? If so why does the previous sentence say this was just done for sapphire?]**

3. Results and discussion

3.1. Enhanced multiple epitaxial lateral overgrowth with graphene

Fig. 1 shows the ELOG sequence using a graphene mask created by the following steps: preparation of MOCVD-grown GaN/sapphire substrate [Fig. 1(a)], PMMA-assisted transfer of the CVD-grown graphene to the GaN/sapphire substrate [Fig. 1(b)], fabrication of the hole-patterned graphene mask by photolithography [Fig. 1(c)], multiple-ELOG of the single-crystalline GaN films with a self-decomposing graphene mask [Figs. 1(d)–1(f)]. The mechanism of the multiple-ELOG as shown in Fig. 1(e) is explained in detail in Section 3.2. From atomic force microscopy, the measured r.m.s. roughness value of the transferred graphene was 0.507 nm, which shows that the transferred graphene was relatively flat when considering

the r.m.s. roughness value of the GaN template of 0.354 nm and the local winkle [wrinkling? undulation?] of graphene (**Fig. S2 [please provide]**).

Fig. 2 shows scanning electron microscopy (SEM) images of GaN films grown by selective area growth (SAG) using the masks made from the two different materials. The SAG was conducted under the same conditions (1323 K in H₂ ambient) from 1 to 10 min using both patterned graphene [Figs. 2(a), 2(c) and 2(e)] and SiO₂ masks [Figs. 2(b), 2(d) and 2(f)]. In the initial stages at 1 min, the GaN SAG using pGR resulted in an irregular circular shape [which can be clearly observed in the inset of Fig. 2(a)], unlike the hexagonal structure seen in conventional SAG. In contrast with the 2 μ m-diameter hole patterns, the GaN SAG using pGR produced 3.5 μ m-diameter disc patterns covering the mask in the horizontal direction starting from the mask windows, whereas the conventional SiO₂ mask produced a pattern of micro-sized hexagons of dimensions similar to the hole pattern. In the case of GaN

SAG using pGR, a completely coalescent smooth GaN layer was grown in less than 4 min, whereas the result of SAG using an SiO₂ mask appeared to be hexagonal-pyramidal GaN deposits, which is as expected. The GaN SAG for 10 min using pGR [Fig. 2(e)] resulted in an extremely thin GaN layer of 380 nm in thickness. The minimum thickness of a conventional ELOG GaN film is governed by the growth kinetics, which results in a relative limitation of the horizontal growth rate in the case of c-plane GaN. For similar reasons, SAG with a dielectric mask of micro-sized holes resulted in the growth of several-micrometre-high pyramids. However, the growth pattern using the graphene mask showed rapid ELOG in the horizontal direction without creating the hexagonal crystalline form of wurtzite GaN in the windows.

In particular, a fully merged $\sim$ 380 nm-thick GaN film was obtained using pGR after 10 min of growth. GaN regrowth using graphene masks was mainly caused by two factors. GaN/GaN homoepitaxy predominantly began with vertical growth

Figure 1
Schematic of a method for patterning a graphene mask and multiple epitaxial lateral overgrowth of single-crystalline GaN thin films.

Figure 2
SEM images of SAG GaN with different masks for different growth times. (a), (c), (e) Graphene; (b), (d), (f) SiO₂. (a), (b) 1 min; (c), (d) 4 min; (e), (f) 10 min.

in the **c** direction through the windows by the conventional ELOG mechanism, but lateral growth occurred from the boundary where the graphene was encountered. However, when using a graphene mask, the lateral growth rate was significantly improved compared with that with the SiO₂ mask. Several literature studies have reported that GaN is decomposed in some conditions similar to MOCVD growth (Koleske *et al.*, 2001) or under graphene (Zhao *et al.*, 2015). As a result of the decomposition of the GaN template present under the graphene, the graphene self-decomposed locally, resulting in additional regrowth sites. Consequently, secondary small-scale ELOG was generated multiple times and the multiple-ELOG GaN film was obtained rapidly in comparison with the film formed via the SiO₂ mask. Further explanations and causes could be identified through further experiments comparing the GaN and sapphire substrates.

3.2. The effect of graphene on the GaN homoepitaxy

To investigate the influence of graphene on the homoepitaxy process and growth mechanism, GaN SAG was performed using a GaN template and a sapphire template. Figs. 3(a) and 3(b)

show SEM images of GaN SAG using pGR on a GaN template and sapphire substrate, respectively. Surprisingly, unlike the case of the GaN template shown previously, the sapphire substrate achieved the same result as the SAG using the SiO₂ mask. That is to say, when a sapphire substrate was used, GaN grew into hexagonal pyramids only in the windows without them being merged into a nanometre-thick film, even when a graphene mask was used. Raman measurements were performed to confirm the state of the graphene on the GaN and sapphire substrates after growth. Figs. 3(c) and 3(d) show Raman spectra [from the regions marked with a red dot in Figs. 3(a) and 3(b), respectively] before and after the growth with the pGR on the GaN template and on the sapphire substrate. For the sapphire substrate, the spectra show Raman peaks corresponding to graphene regardless of whether they were taken before or after growth, as can be seen in Fig. 3(d): *G* (1580 cm⁻¹), a primary in-plane vibrational mode, and *2D* (2690 cm⁻¹), a second-order overtone of a different in-plane vibration. On the other hand, the Raman spectra in Fig. 3(c) show evidence that the graphene is damaged after growth with the pGR on the GaN template: a weakened *2D* peak and an additional peak at ~2410 cm⁻¹. We assume that the peak at 2410 cm⁻¹ is shifted from ~2450 cm⁻¹, being a combination of a *D* phonon and an acoustic longitudinal phonon (*D'*) caused by overgrown GaN (Roscher *et al.*, 2019). The background noise levels are different for the GaN template and the sapphire substrate. Commonly, GaN has the ground noise of the upper right according to the Raman shift [please clarify what this means] and a secondary satellite peak of GaN at 1250–1500 cm⁻¹, which was observed weakly for graphene on the GaN template because of the small spot size.

It has been reported that etching of graphene is controlled by high-temperature/H₂ graphene conditions (Vlassioug *et al.*,

Figure 3
SEM images after GaN growth with a graphene mask on a (a) GaN template and (b) sapphire substrate at 1323 K at 100 Torr in H₂ ambient. Raman spectra of graphene on (c) GaN and (d) sapphire before/after GaN growth.

2011; Geng *et al.*, 2013; Wang *et al.*, 2014), which are very similar to the GaN growth conditions. However, the results in Fig. 3(c) demonstrate an interesting fact: the decomposition of the graphene in the GaN growth environment occurred mainly because of the substrate type rather than the high-temperature/ H_2 conditions.

The difference between the GaN template and the sapphire substrate was the decomposition of the substrate in the growth environment. Compared with the sapphire substrate, the GaN template might have damaged graphene bonded by van der Waals forces by its decomposition. We have recently reported that N generated during the decomposition of GaN causes the loss of graphene (Park *et al.*, 2019). Zhang *et al.* (2012) reported that copper plays an important role in catalyzing the etching reaction of graphene, **which is the reverse of the formation reaction**. The CVD of graphene is usually carried out using Cu, Ni or Pt, and a method using liquid Ga has also been reported (Ding *et al.*, 2013). In other words, since graphene decomposition is the reverse of the reaction of formation, liquid Ga can trigger graphene decomposition under certain conditions. Thereby secondary **GaN regrowth sites can be formed on graphene, which have difficulty growing due to the inherent chemical stability of GaN**. For this reason, the homoepitaxy of the GaN grown using graphene is dominated by the decomposition of the GaN template rather than H_2 . Fig. 1(e) shows the mechanism of multiple-ELOG due to the self-decomposition of graphene as time passes: (1) the beginning of SAG, (2) self-decomposition of graphene and (3) multiple-ELOG. The patterned graphene mask in this GaN homoepitaxy caused the predominant growth on the window, and a small amount of secondary ELOG occurred multiply on the self-decomposing graphene mask during the growth process, as shown in the inset of Fig. 1(e).

Multiple-ELOG can be explained as a mechanism to accelerate horizontal growth. The GaN grown on the windows reached a secondary point that was relatively close to the neighboring window and quickly grew horizontally as a stepping stone. Under equilibrium conditions, the growth of wurtzite GaN, an anisotropic crystal structure, progresses in the direction of decreasing total surface energy. For this reason, the typical GaN selective growth produced hexagonal-pyramid shapes because of the planes with the lowest surface energy. By the same token, when the grown GaN discs in the window met the adjacent secondary regrown GaN, they merged into one disc by growing horizontally instead of growing as individual hexagonal pyramids. **The merged discs have** a large surface, thereby reducing the total surface energy. This is consistent with an aspect of concave growth which is a salient feature in the coalescence. In concave growth, facets with high growth rate are predominant, and although the surface energy of facets is high, it was essentially decreased per unit volume.

The GaN grown horizontally from the window pattern in Fig. 2 and the modified surface of the graphene mask of the inset image in Fig. 3(a) match this mechanism. The growth results will vary with the degree of graphene decomposition or pattern. If the decomposition of graphene is severe, the

masked region is reduced; multiple-ELOG is possible but the area to prevent dislocation is reduced, which means that the crystal quality cannot be improved and the **separation [of the film and substrate?]** is difficult. Since the self-decomposition of the graphene is highly influenced by the decomposition of the GaN template, the growth conditions to control the GaN decomposition (e.g. high ammonia atmosphere and ammonia ratio are important to mitigate this) may allow GaN ELOG with no extra mask patterning process, and **it [the resulting film?]** may be easily separated later via the lift-off process.

3.3. The crystallinity of the ELOG GaN with graphene

EBS mapping was performed to confirm the crystal orientation of the 2 μ m-thick multiple-ELOG GaN grown for 1 h using pGR with a hole diameter of 2 μ m. The resulting GaN had a (0001) orientation, as shown in red in the inverse pole figure (IPF) color triangle [Fig. 4(a)]. The EBSD pole figure showed a clear {0001} spot and a definite sixfold symmetry in {10 $\bar{1}2$ } [Fig. 4(b)], indicating that the GaN had a single-crystalline wurtzite structure. To evaluate its crystallinity, XRD rocking curves (ω scans) of the 002 symmetric and 102 asymmetric reflections were measured [Fig. 5(a)]. The FWHMs of the multiple-ELOG GaN and bare GaN templates for the 002 reflections [Fig. 5(a)] were 221 and 227'', respectively, i.e. comparable. In the case of the 102 reflection [Fig. 5(b)], the FWHMs were 405 and 540'', respectively; the measurements for the 002 reflections are related to pure screw and mixed screw-edge dislocation, while those for the 102 reflections are related to pure edge dislocation. The ELOG GaN using pGR showed a significant decrease in the 102 reflection compared with the bare GaN template, thus indicating a significant reduction in pure edge dislocation by the ELOG process. These results are similar to those found in

Figure 4
(a) EBSD map and (b) EBSD pole figures of ELOG GaN.

Figure 5
XRD ω rocking curves of (a) the symmetric 002 and (b) the asymmetric 102 reflection of ELOG GaN.

ELOG studies using nanometre-sized masks (Lee *et al.*, 2012; Seo *et al.*, 2015).

4. Conclusion

In this study, we have discussed a mechanism for ELOG of GaN films under **single-crystalline growth** conditions by using a 2D monolayer pGR mask in relation to graphene decomposition. Multiple-ELOG occurred as a result of secondary regrowth sites of the self-decomposing graphene masks during the regrowth of GaN, thereby accelerating horizontal growth and resulting in a monocrystalline several-hundred-micrometre-thick GaN film with improved crystalline quality. The decomposition of the GaN template rather than the high temperature/ H_2 atmosphere played an important role in the graphene mask decomposition during the GaN homoepitaxial growth.

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