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In situ synchrotron X-ray diffraction studies of nitride semiconductor materials

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Abstract

For investigating crystal growth mechanisms in nitride semiconductor materials, we have recently developed a nitride molecular beam epitaxy (MBE) system that is directly coupled to the X-ray diffractometer at the synchrotron radiation facility, SPring-8/BL11XU. This system enables us to perform *in situ* synchrotron X-ray diffraction, which has many advantages compared to conventional *in situ* reflection high-energy electron diffraction (RHEED). One of them is to evaluate strain states of nitride crystals in detail. Using this system, the strain evolution of the GaN grown on 6H-SiC(0001) substrates was measured and the correlation between the growth mode (Ga-stable or N-stable) and the strain state was investigated. As the results, we found that the strain state of the GaN initial growth strongly depends on growth modes.

1. Introduction

Gallium nitride (GaN) and its alloys are largely exploited for blue and UV-range optoelectronic devices and in high speed, high power electronic applications. Despite these technological advances, many challenges in crystal growth still remain. One of them is growth mode and strain state of the GaN films grown by molecular beam epitaxy (MBE). It has been known that the growth mode of the GaN films grown by MBE strongly depends on substrate temperature and Ga/N ratio, and understood as N-stable or Ga-stable conditions [1]. Reflection high-energy electron diffraction (RHEED) is widely used to identify the growth mode during the GaN growth. Under N-stable conditions, films generally have spotty RHEED patterns and rough surface, while Ga-stable conditions generally produce films having streaky RHEED pattern with smooth two-dimensional surface morphologies. Like this, the RHEED can look a difference of the growth mode in relation to surface morphologies. However, it is difficult to obtain information about the strain state of the GaN films. We consider that the strain state at the initial stage of the GaN growth is quite important because it should directly influence the crystal quality of the GaN and eventually its device performance. In this study, therefore, *in situ* synchrotron X-ray diffraction (*in situ* XRD), which is a unique *in situ* measurement for studying growth dynamics of crystals, is carried out during the GaN growth. Using this, the strain evolution of the GaN is measured and the correlation between the growth mode and the strain state is investigated.

2. MBE/XRD system

In situ XRD has ever been reported in nitrides grown by a metal organic chemical vapor deposition [2]. However, there are no studies using the MBE so far. We recently installed a nitride MBE system that is directly coupled to the X-ray diffractometer at the synchrotron radiation facility, SPring-8/BL11XU.

3. Experimental

The GaN films were grown on Si-face 6H-SiC(0001) using plasma-assisted MBE. Following a removal of native oxides of SiC substrates by heating at a high temperature, the GaN was directly grown on SiC(0001). We varied the growth temperature (700-800^{o}C) and the Ga/N ratio to grow the GaN under N-stable or Ga-stable conditions.

The X-ray energy used was 20keV. The diffracted X-ray signals were detected by a two dimensional detector (PILATUS 100K). While the GaN film was being grown, the two dimensional reciprocal space maps (RSMs) around the 0006 diffraction peaks of SiC were measured by adjusting the sample orientation and the detector position. Time resolution of the measurement was 104 s, which corresponded to the GaN film growth of 2.2 nm in thickness. In addition to the X-ray measurements, RHEED patterns were also recorded during the growth of the GaN film.

4. Results

Figure 1 shows typical RHEED patterns of GaN films grown under Ga-stable and N-stable conditions in two different directions ([112-0] and [1-100]) of the incident electron beam. Under the Ga-stable condition, we can identify the smooth GaN surface due to the streak pattern. In contrast, the N-stable condition gives the rough GaN surface morphology resulting from the spotty RHEED pattern. It is also confirmed in this study that the RHEED is sensitive to the surface morphologies, and is useful to identify the growth modes.

In order to evaluate the strain state of the GaN films, typical RSMs during Ga- and N-stable GaN growths on the SiC(0001) substrates are shown in Figs. 2 and 3, respectively. In the case of the Ga-stable condition, thickness fringes of the GaN can be seen. The position of the GaN main peaks was located at the Bragg point for fully relaxed GaN, and did not shift with increasing thicknesses. Therefore, in this growth mode, the fully relaxed GaN with the smooth surface grows from the initial growth stage.

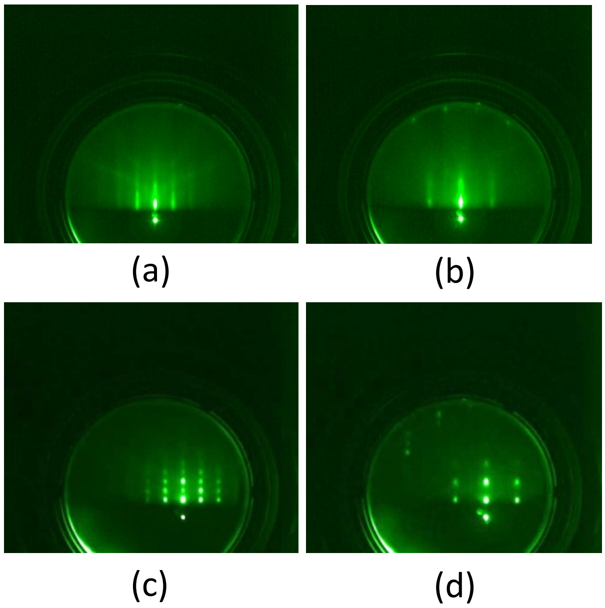

Fig. 1 RHEED patterns in GaN under Ga-stable condition, (a) [112-0] and (b) [1-100] directions, and under N-stable condition, (c) [112-0] and (d) [1-100] directions.

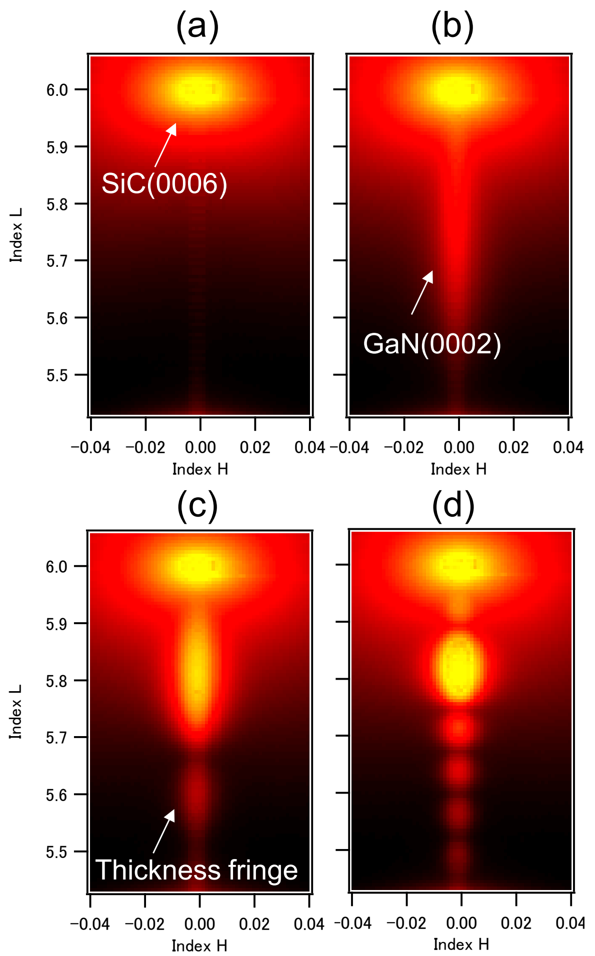

Fig. 2 Typical *in situ* RSMs during gallium-stable GaN growth on the SiC(0001) substrate. Thicknesses of GaN are (a) 0 (before growth), (b) 4, (c) 12, and (d) 20, respectively.

In the case of the N-stable condition (Fig. 3), thickness fringes of the GaN cannot be seen, but streaks showing formation of surface facets appeared. Moreover, the GaN main peaks were gradually shifted to the positions where the GaN is fully relaxed. This suggests that the strained GaN islands are growing at the initial stage. By using *in situ* XRD, we found that the strain state of the GaN initial growth strongly depends on growth modes.

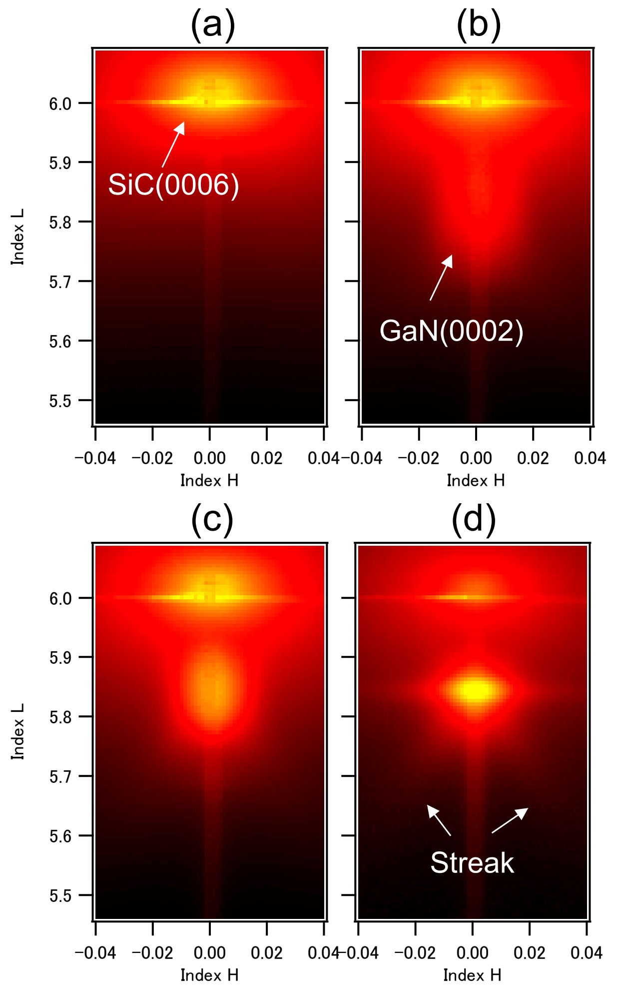

Fig. 3 Typical *in situ* RSMs during nitrogen-stable GaN growth on the SiC(0001) substrate. Thicknesses of GaN are (a) 0 (before growth), (b) 12, (c) 20 and (d) 94 nm, respectively.

5. Conclusions

Using nitride-MBE/XRD system at the synchrotron radiation facility, SPring-8/BL11XU, the strain evolution of the GaN grown on 6H-SiC(0001) substrates was measured and the correlation between the growth mode (Ga-stable or N-stable) and the strain state was investigated. As the results, we found that the strain state of the GaN initial growth strongly depends on growth modes.

References

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[2] M.-I. Richard et al., Appl. Phys. Lett. **96**, 051911 (2010).