Transactions of the Materials Research Society of Japan 33[3] 547-550 (2008)

Buried Heterostructure of Nitride Semiconductors Revealed by Laboratory Level X-ray CTR Scattering

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We set up a laboratory level X-ray CTR measurement system using a multilayer focusing mirror and a channel-cut asymmetric Ge double-crystal for a beam squeezing and an imaging plate as a 2D detector. Even though the intensity of the laboratory X-ray is by 700 times lower than that of the synchrotron X-ray, good enough signal/background ratios were achieved by the laboratory X-ray system for a reasonable measurement time of 100 minutes. This measurement system was applied to GaN/GaInN/GaN heterostructures on sapphires, which were difficult to obtain a good signal/background ratio using synchrotron X-ray CTR measurements system, and reasonable CTR data were obtained. In distributions in GaN/GaInN/GaN very different from those designed were obtained.

Key words: X-ray CTR, laboratory level X-ray, buried interface, GaInN

1. INTRODUCTION

Though there are several experiments on *in-situ* X-ray characterization of semiconductor growth, most of them are investigation on surface structures of the growth front [1-3]. Instead, we have been conducting *ex-situ* X-ray characterization of semiconductor heterostructures, from the view points of growth process dependence of the heterostructures, with X-ray CTR (crystal truncation rod) scattering measurements using synchrotron X-ray [4-18]. It is because the *in-situ* characterization of the growth front does not give us the information of the heterostructure itself after all through the growth processes. It implies that the heterostucture changes during the growth process due to the growth temperature itself, the change of the supply gases, and the continuous growth of an over-layer [14-18].

The X-ray CTR techniques have successfully been used for III-V compound semiconductors and their heterostructures [4-18], and also for GaN and related materials, including the investigation on the role of the low-temperature-deposited AlN buffer layer on sapphire and its best thickness [19-24].

In our previous report, we described application of the X-ray CTR scattering measurements to the GaN/GaInN/GaN structure and discussed not only the quality of the layers but also the layer structures quantitatively [25]. In the report, we used the synchrotron radiation as the X-ray source and the 2-dimensional CCD camera as a detector, *i.e.*, the same setup as we have been used for conventional III-V semiconductors.

However, when the GaN/GaInN/GaN samples were measured by the X-ray CTR scattering, the X-rays scattered by the relatively thick GaN buffer layer with a high density of dislocations obstructed observation of clear CTR data. To avoid this problem, we used the GaN/GaInN/GaN structures on the GaN substrates. In this case the X-ray CTR data were good enough to analyze the heterostructure, especially the In distributions. Segregation of In from GaInN to the GaN cap layer was observed [25]. In the paper, we suggested that it is necessary to use the asymmetric diffraction condition (very small incident angle to avoid X-rays to penetrate deep into the low quality GaN) if the GaN/GaInN/GaN structure was grown on the sapphire substrate.

2. LABORATORY LEVEL X-RAY CTR MEASUREMENT SYSTEM

In the mean time we have been developing a laboratory level X-ray CTR scattering measurement system as shown in Fig. 1, for the last one year. The X-ray intensity is by about 700 times higher from the synchrotron radiation than the laboratory level X-ray

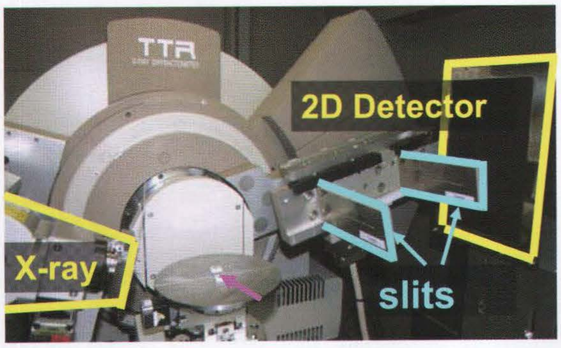

Fig. 1: Laboratory level X-ray system for CTR scattering measurements.

(rotating target Cu $K_{\alpha 1}$) even with a multilayer focusing mirror and a channel-cut asymmetric Ge double-crystal for a beam squeezing. 700 times of difference in intensity was discouraging and it seemed that this system was useless for CTR scattering measurements.

However, a combination of slits was very effective to lower the background and it greatly reduced the time for the measurements by slightly narrowing the range of index l. 100 minutes of measurement time in the range of $1.8<l<2.2$ gave us the same quality of CTR signals (and the analysis results) as that of the results by 2 minutes of measurement time in the range $1.7<l<2.3$ using the synchrotron X-ray. The measured CTR data (•) are compared in Fig. 2 (a) and (b). In the results of analysis there were no meaningful differences. Of course, when the l range is narrower, the measurement time by the synchrotron X-ray is also shorter. However, difference of the signal quality for the laboratory X-ray and synchrotron X-ray was turned out to be large in the case of GaN-based heterostructures on sapphire substrates.

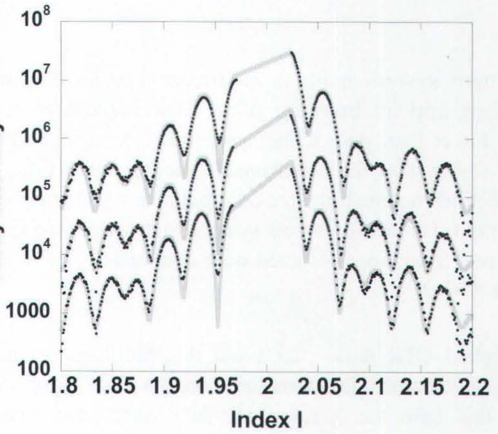

Fig. 2 (a) CTR data (•) from InP/GaInAs/InP (with different purging times at source gas change) using laboratory X-ray system in the range $1.8<l<2.2$. The gray lines are best-fit curves.

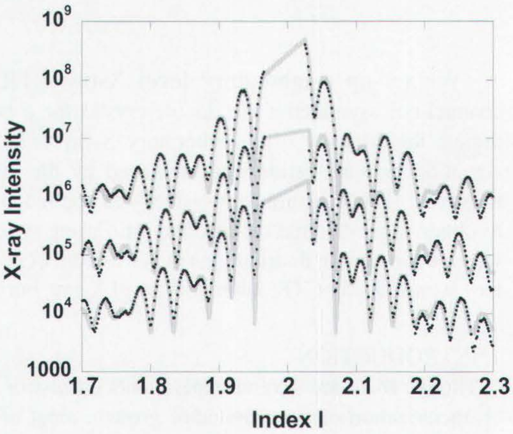

Fig. 2 (b) CTR data (•) from InP/GaInAs/InP (with different purging times at source gas change) using synchrotron X-ray in the range $1.7<l<2.3$. The gray lines are best-fit curves.

3. GaN/GaInN/GaN ON SAPPHIRE

As reported previously, the CTR signal/background ratio was so low and it was difficult to obtain good CTR data from the samples grown on the sapphire substrates, using synchrotron X-ray for the CTR measurement [25]. They are shown in Fig. 3. However, by the laboratory X-ray the signal/background ratio was much improved probably due to the slits between the sample and the detector (Imaging Plate).

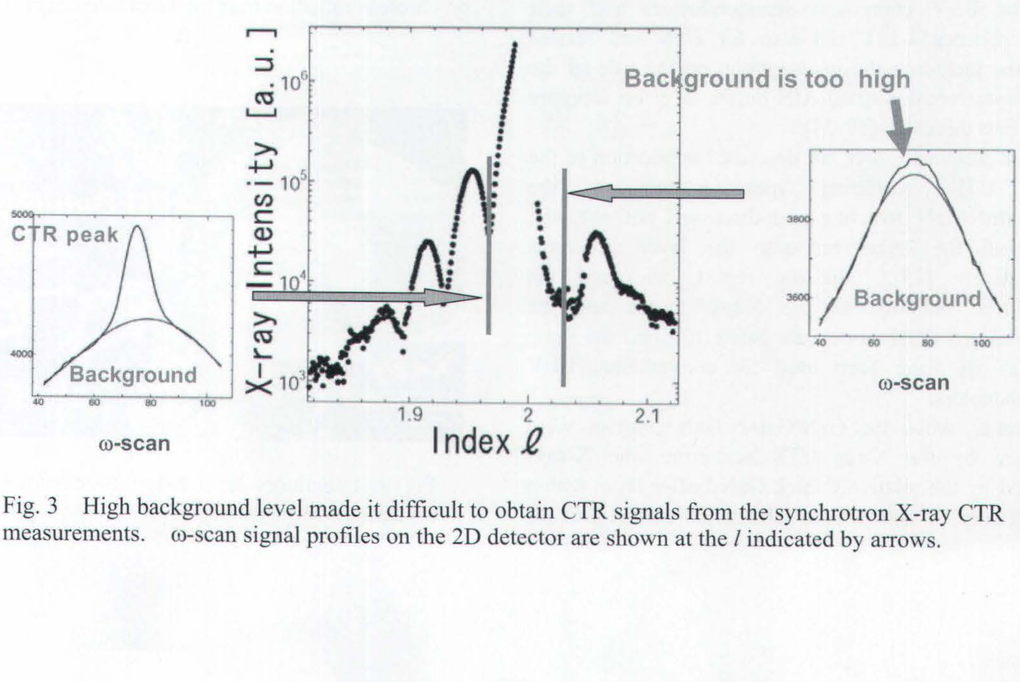

Fig. 3 High background level made it difficult to obtain CTR signals from the synchrotron X-ray CTR measurements. ω-scan signal profiles on the 2D detector are shown at the l indicated by arrows.

The best-fit curves are shown in Fig. 4 for two samples with designed In compositions in GaInN of 0.09 and 0.12. The obtained In distribution profiles are shown in Fig. 5, left and right. The cap layer thickness of GaN was designed to be 10 nm. The obtained value of 12 nm for the cap layer is close to the designed one. The GaInN layer thickness was 2.6 nm and it is half of the designed one. The distribution of In to the cap layer was considerable and the total amount of In in GaN/$Ga_{0.91}In_{0.09}N$/GaN was 1.19ML and that in GaN/$Ga_{0.88}In_{0.12}N$/GaN was 1.27.

The In profiles shown in Fig. 5 indicate quite large composition gradings both at bottom interfaces and upper interfaces. This is one of the big differences from the InP/GaInAs/InP that we used as a standard heterostructures where the bottom interface was always flat and only the upper interface was influenced by the growth conditions. In the figure the increase of the In composition toward growth direction is observed in the GaInN layer, suggesting the segregation of In atoms during the growth. The In compositions in GaInN are almost half of the designed values. We obtained similar difference in the GaInN layers grown on GaN substrates [25].

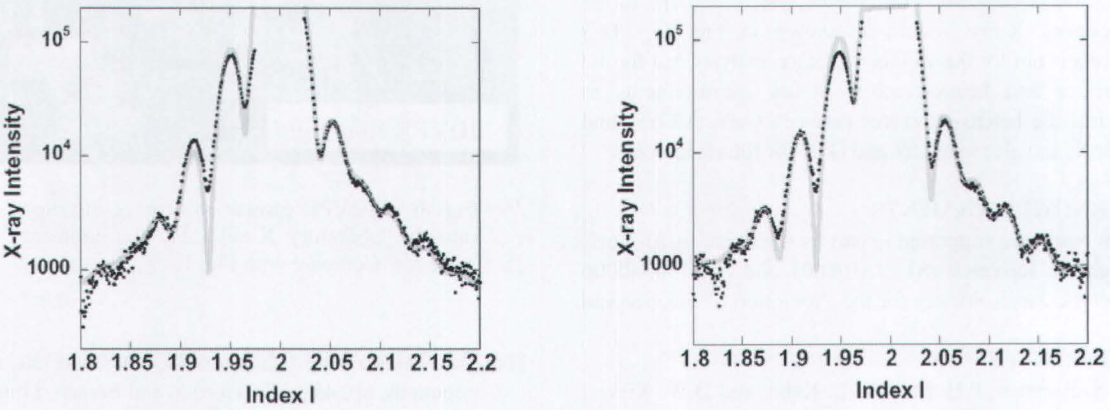

Fig. 4 Best-fit curves to the CTR data (●) from GaN/$Ga_{0.91}In_{0.09}N$/GaN (left) and GaN/$Ga_{0.88}In_{0.12}N$/GaN (right) on sapphire substrates measured by laboratory X-ray system. In compositions 0.09 and 0.12 are designed values.

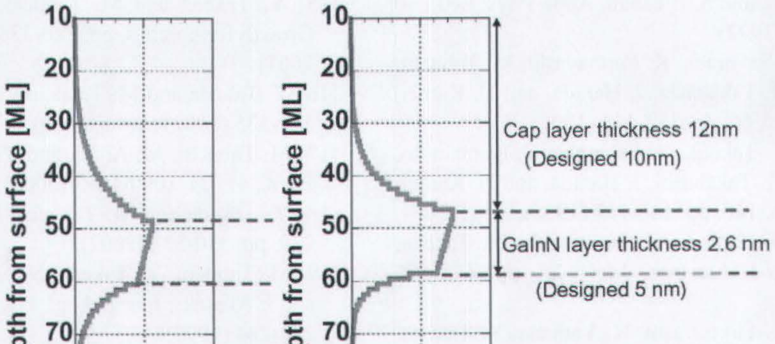

Fig. 5 Obtained In distribution profiles for GaN/$Ga_{0.91}In_{0.09}N$/GaN (left) and GaN/$Ga_{0.88}In_{0.12}N$/GaN (right) on sapphire substrates. In compositions 0.09 and 0.12 are designed values. Extension of In to the upper GaN is probably due to In atom segregation, atoms exchange, and interface roughness. Extension of In to the bottom GaN is probably due to atoms exchange and/or interface roughness.

4. SUMMARY

In summary, we set up a laboratory level X-ray CTR measurement system and applied it to GaN/GaInN/GaN heterostructures on sapphires, which was difficult to obtain a good signal/background ratio using synchrotron X-ray CTR measurements system. In distributions very different from those designed (expected) were obtained.

Even though the intensity of the synchrotron X-ray is by 700 times higher than that of the laboratory X-ray, better signal/background ratios were achieved by the laboratory X-ray system for a reasonable measurement time of 100 minutes. The reason for the better results by the laboratory X-ray CTR measurement system is simply because of a better optics design for X-rays. On the other hand, the synchrotron X-ray CTR measurement system is designed for protein structure measurements and those carefully designed for the CTR measurements is not constructed. From those experiences, the best designed beam line will be installed at the "Central Japan Synchrotron Radiation Facility" that is one of the projects going on in Nagoya area.

The development of the laboratory X-ray CTR measurements system is still on the half way. We are installing a GaN MOVPE reactor system that fits to the laboratory X-ray system as shown in Fig. 6. This system is not for the surface structure analysis, but for the interface and heterostructure *in-situ* measurements to control the hetero-interfaces between GaN, AlGaN, and GaInN, and also insulator and GaN for future devices.

ACKNOWLEDGEMENTS
This work was supported in part by the Grant-in-Aid for Scientific Research (S) #18106001 and (B) #19360006 from the Japan Society for the Promotion of Science and in part by the JST Project "Collaborative Development of Innovative Seeds–Potentiality Verification Stage–.

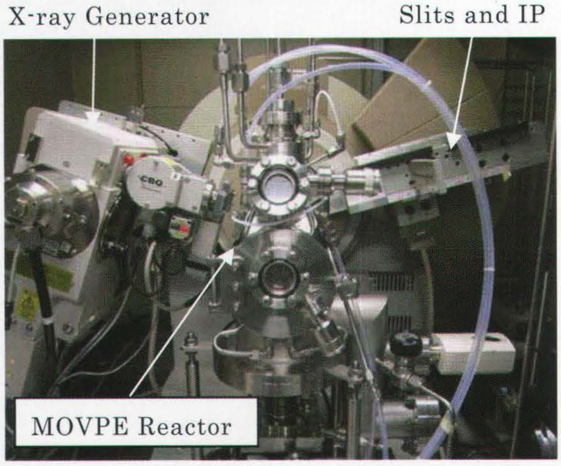

Fig. 6 MOVPE growth system is installed into the laboratory X-ray CTR measurement system. Compare with Fig. 1.

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(Recieved December 10, 2007 ; Accepted February 29, 2008)