Transactions of the Materials Research Society of Japan 33[3] 591-594 (2008)

X-ray CTR scattering measurements using conventional X-ray source to study semiconductor hetero-interfaces

Y. Maeda, T. Mizuno, A. Mori, M. Tabuchi*, Y. Takeda
Graduate School of Engineering, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan
Fax: 052-789-3239, e-mail: maeda@mercury.numse.nagoya-u.ac.jp
* Venture Business Laboratory, Nagoya University, Furo-cho, Chikusa-ku, Nagoya 464-8603, Japan

An X-ray crystal truncation rod (CTR) scattering measurement system was set up using X-ray diffractometer that has a conventional X-ray source, i.e., rotating anode X-ray tube. Optical parts such as a collimating-multilayer mirror and an asymmetric Ge(220) monochromator to obtain condensed Cu-Kα_1 beam and a set of slits to avoid scattered X-rays were installed. An imaging plate was used as a detector. It took 100 minutes for the X-ray CTR scattering measurement using the present system. Analysis of the X-ray CTR scattering spectra which were obtained using the present system showed that almost the same interface structures as those obtained using the SR were represented for the same samples. It suggests that the present measurement system with the conventional X-ray source is useful for the investigation of semiconductor heterostructures.
Key words: X-ray CTR scattering, semiconductor heterostructures, synchrotron radiation, conventional X-ray source

1. INTRODUCTION

When X-ray diffraction and scattering of a finite crystal, which is truncated at surfaces, are measured, diffraction spots extending normal to the surface in reciprocal space are obtained, which is called X-ray CTR scattering. The X-ray CTR scattering spectrum is sensitively modified as the structure near the surface is changed [1–3]. Therefore, the X-ray CTR scattering measurement has been used to investigate the structure and the roughness of the surfaces [4,5] and interfaces [6–8].

We had demonstrated that the X-ray CTR scattering measurement is a very powerful technique to analyze the interface structures in hetero-epitaxially grown III-V compound semiconductors [9–13]. In these works, it was shown that the X-ray CTR scattering measurement can reveal layer thicknesses, composition profiles, surface roughness, and lattice distortions at an atomic scale. However, it is generally considered that an X-ray source as strong as synchrotron radiation (SR) is necessary to measure the weak CTR scattering well for a precise analysis.

In this work, a new X-ray CTR scattering measurement system based on an X-ray diffractometer that has a conventional X-ray source was investigated. Interface structures of InP/GaInAs/InP and GaN/GaInN/GaN samples were analyzed from the spectra obtained by using the new system, and the results were compared with those measured using the SR.

2. NEW X-RAY CTR SCATTERING MEASUREMENT SYSTEM WITH CONVENTIONAL X-RAY SOURCE

The new measurement system was constructed based on an X-ray diffractometer, RIGAKU RINT-TTRIII. Figure 1 shows the outline of the present system. The diffractometer has an 18 kW rotating anode X-ray tube with Cu target. Optical parts such as a collimating-multilayer mirror and an asymmetric Ge(220) monochromator were installed to obtain condensed Cu-Kα_1 beam. The X-ray intensity was 2 x 10^8 cps at the sample position when the X-ray tube was driven at 15 kW(50 kV, 300 mA) and a 0.2 mm x 5 mm entrance slit was installed.

An imaging plate (IP) was used as a detector. The IP is effective to curtail the measurement time since a reciprocal space map can be obtained by only single scan on the IP. The measured X-ray CTR scattering spectra are analyzed assuming the kinetic theory. Therefore, the component of non-kinetically scattered X-ray intensity should be removed from the measured scattering X-ray intensity. In order to remove the non-kinetic component, it is important to

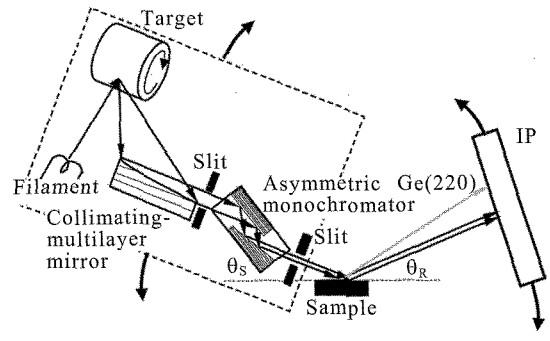

Fig. 1. The outline of the new X-ray CTR scattering measurement system.

obtain the two-dimensional reciprocal-space map around a Bragg point along the CTR. Since non-kinetic component is usually observed as a smoothly changing background on the map, and the X-ray CTR scattering, which is observed like as sharp peak on a broad background, is easily distinguished from the background.

The diffractometer was designed to rotate the X-ray source and the detector independently while the sample stage is fixed. In this work, the angle between incident X-ray beam and the sample surface was represented as θ_S and the angle between the sample surface and the line normal to the IP surface was represented as θ_R.

3. X-RAY CTR SCATTERING MEASUREMENTS USING THE NEW SYSTEM

3.1 X-ray CTR scattering measurements in ω-scan mode

We had conducted the X-ray CTR scattering measurement using the SR in ω-scan mode, i.e., only the sample was rotated while the incident X-ray and detector were fixed. Using the new system, at first, the X-ray CTR scattering measurement was conducted by changing the θ_S and θ_R keeping the relationship that $\Delta\theta_S = -\Delta\theta_R$. The motion is equivalent to the ω-scan mode. Figure 2 shows the measured X-ray CTR spectra of an InP(34 ML)/GaInAs(15 ML)/InP structure sample using the new system and the SR. The spectrum B was measured at BL6A of Photon Factory in High Energy Accelerator Research Organization at Tsukuba. θ_S was changed from -2.0 to -0.15° and +0.15 to +2.0° around the 002 Bragg point of InP. The measurement time were 1000 min using the new system, and 2 min using the SR. The measurement time, 1000 min, was determined since the X-ray intensity at BL6A was by 500 times as high as that of the new system. Comparing the spectra measured using the new system and the SR, the noise was high in the CTR spectrum measured using the new system. To obtain a clear spectrum that can be analyzed, it was necessary to find the origin of the noise and to solve the problem.

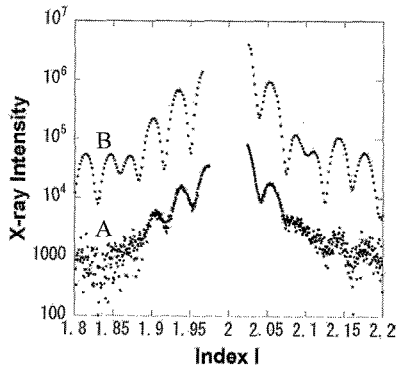

Fig. 2. The spectra A and B are the X-ray CTR spectra measured using the new system and the SR, respectively. Index l means the index in k-space.

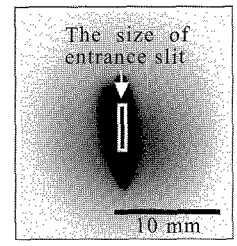

Fig. 3. The distribution of the incident X-ray beam at the sample position and the size of entrance slit.

3.2 Improvement of the measurement

Divergence of the incident X-ray beam is larger in the new system than that in the SR. The differences arised from the reason that the brilliance of the conventional X-ray source was low (divergence is large) in the new system. Figure 3 shows the distribution of the incident X-ray beam at the sample position when the θ_S was set at 15.215°, which is the 002 Bragg angle of InP. As shown in Fig. 3, the incident X-ray beam distributed in the area much larger than the size of entrance slit. It indicates that there was a significant divergence of the incident X-ray beam in the new system.

The divergence of the incident X-ray beam causes X-ray diffraction evenwhen the θ_S is not a Bragg angle since a part of the divergent incident X-ray beam can satisfy Bragg condition. In the reciprocal space, the phenomenon is represented as the intensity distribution around a Bragg point as shown in Fig. 4 (a). Therefore, when the measurement was conducted in the ω-scan mode, strong back-ground is observed since the distribution around a Bragg point are projected on the IP as the back-ground overlapping the CTR scattering as shown in Fig. 4 (b).

To suppress the back-ground caused by the divergence of the incident X-ray beam, we introduced a set of receiving slits that rotate synchronously with θ_S between the sample and the IP as shown in Fig. 5. On the other hand, the IP was fixed during the measurement. Using similar system that has a moving slit, it was reported that X-ray CTR scattering measurement was conducted[14]. The size of

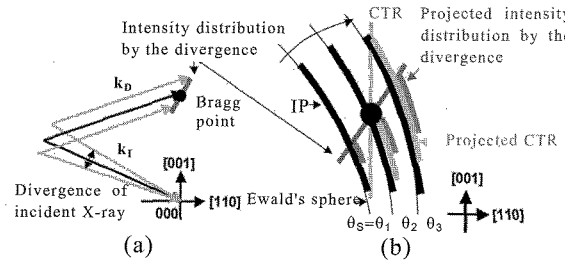

Fig. 4. (a) The intensity distribution around a Bragg point caused by the divergence of the incident X-ray beam. (b) The intensity distribution by the divergence is projected on IP overlapping the CTR scattering.

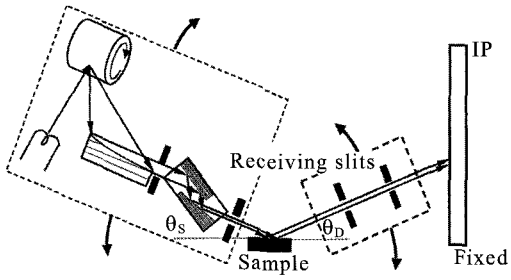

Fig. 5. The outline of the new measurement system that is designed to suppress the background.

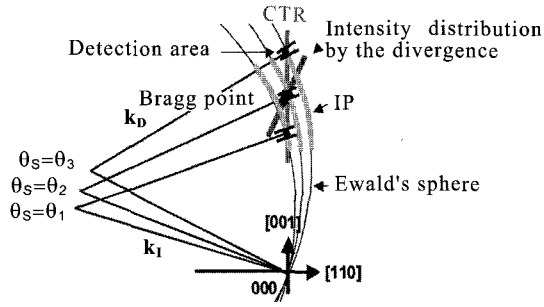

Fig. 6. The CTR spectrum measured using the receiving slits and θ-2θ scan mode. The detection area moves only on the CTR scattering since the range of detection along the surface of Ewald's sphere is narrowed by the receiving slits to avoid the projection of the background.

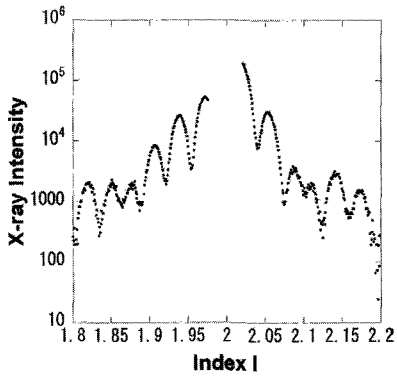

Fig. 7. The CTR spectrum measured using the receiving slits in θ-2θ scan mode.

the opening area of the slits was 1.0 mm x 90 mm. In this work, the angle between the sample surface and the opening area of the slits was represented as θ_D.

In the measurement using the receiving slits, θ_S and θ_D were changed keeping the relationship that $\Delta\theta_S = \Delta\theta_D$. In other words, it is a kind of a θ-2θ scan mode. Using the receiving slits in the θ-2θ mode, the projection of the background on IP overlapping the CTR scattering can be reduced as shown in Fig. 6.

Figure 7 shows the CTR spectrum measured using the receiving slits and the θ-2θ scan mode. The scanning time was 100 min. Comparing the CTR spectrum with that in Fig. 2, it is obvious that the noise was much suppressed and the spectrum was improved by using the receiving slits in the θ-2θ mode although the scanning times was only 100 min.

4. CAPABILITY OF THE NEW MEASUREMENT SYSTEM

4.1 Investigation of interface structures between InP and GaInAs

The capability of the new system was investigated by comparing the interface structures obtained from the analysis of the CTR spectra measured using the new system and the SR.

Figure 8 shows the layer structure of the samples and the source-gas-flow sequence to prepare the samples. The samples were grown on InP(001) substrates by a low-pressure MOVPE using triethylgallium (TEGa), trimethylindium (TMIn), tertiarybutylarsine (TBAs), and tertiarybutylphosphine (TBP) as source materials. The reactor pressure was fixed at 76 Torr. The growth temperature was set at 590°C. TBP-flushing time after the growth of the GaInAs layers was changed from 3 to 30 s. The process to analyze the spectra was described elsewhere [12,13].

Figure 9 shows the relationship between the flushing time and the integrated amounts of Ga and As in InP cap layers, which was obtained from the analysis of the X-ray CTR scattering spectra. Comparing the results obtained using the new system and the SR, almost the same results were observed for the same samples. It indicates

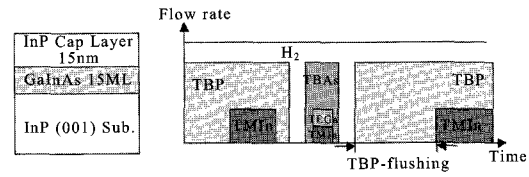

Fig. 8. The layer structure of the samples and the source-gas-flow sequence to prepare the sample. The TBP-flushing times after the growth of the GaInAs layers were changed for several samples.

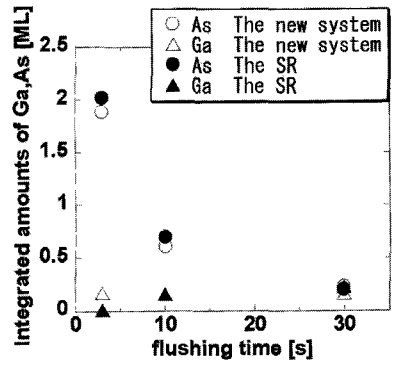

Fig. 9. Comparison of the relationships between the flushing time and the integrated amounts of Ga and As in InP cap layers obtained using the new system and the SR.

that the new measurement system is reliable for the investigation of semiconductor heterostructures.

4.2 Investigation of GaN/GaInN/GaN structures

Figure 10 shows the CTR spectra obtained from a structure of GaN(10 nm)/GaInN(5 nm) /GaN on a sapphire substrate. Comparing the CTR spectra measured using the new system and the SR, it was observed that the noise in the spectrum measured using the new system was less than that measured using the SR. When the X-ray CTR scattering spectrum was measured using the SR, the background was higher caused by the diffuse scattering from the dislocations in the sample. On the other hand, the signal/background ratio was improved by using the new measurement system due to the receiving slits. It suggests that the new measurement system is useful for the measurement of the samples of which crystalline quality is rather low, such as, group-III nitride semiconductors on sapphire substrates.

5. CONCLUSIONS

A new X-ray CTR scattering measurement system based on an X-ray diffractometer that has a conventional X-ray source was investigated. The X-ray CTR spectrum measured using the rocking curve measurement had the strong noise due to the divergence of angles of the incident X-ray beam. To suppress the noise, we introduced a set of receiving slits that rotate synchronously with θ_S. Using the receiving slits, the noise was much suppressed and spectrum was clearly improved.

Interface structures of InP/GaInAs/InP samples were analyzed from the data obtained by using the new system and the results were compared with those measured using the SR. In consequence, both of the analysis gave almost the same results for the same samples. It suggests that the new measurement system is reliable for the investigation of semiconductor heterostructures.

When the measurement was conducted by using the SR, the CTR spectra obtained from a structure of GaN/GaInN/GaN on sapphire substrate had the noise since the dislocations in the sample cause the diffuse scattering. However, the noise in the spectra measured using the new system was less than that measured using the SR due to improvement of the signal/background ration by the receiving slits. It suggests that the new measurement system is useful for the measurement of the samples of which crystalline quality is rather low.

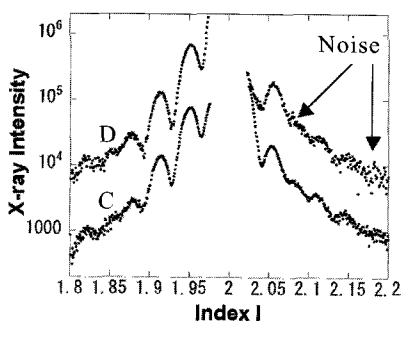

Fig. 10. The X-ray CTR scattering spectra obtained from the structure of GaN(10 nm) /GaInN(5 nm)/GaN on sapphire substrate. C is measured by using the new system. D is measured by using the SR.

ACKOWLEDGEMENTS

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. This work was performed as a part of the projects 2005G155, 2006G247 and 2007G557 accepted by the Photon Factory Program Advisory Committee.

REFERENCES

[1] I.K. Robinson, Phys. Rev., B 33 (1986) 3830-3836.
[2] I.K. Robinson and D.J. Tweet, Rep. Prog. Phys., 55 (1992) 599-651.
[3] T. Shimura and J. Harada, J. Appl. Cryst., 26 (1993) 151-158.
[4] D.W. Kisker, G.B. Stephenson, P.H. Fuoss, F.J. Lamelas, S. Brennan, and P. Imperatori, J. Cryst. Growth, 124 (1992) 1-4.
[5] F.J. Lamelas, P.H. Fuoss, D.W. Kisker, G.B. Stephenson, P. Imperatori, and S. Brennan, Phys. Rev., B 49 (1994) 1957-1965.
[6] I.K. Robinson, R.T. Tung, and R. Feidenhans'l, Phys. Rev., B 38 (1988) 3632-3635.
[7] E.D. Specht, G.E. Ice, C.J. Peters, C.J. Sparks, N. Lucas, X.-M. Zhu, R. Moret, and H. Morkoc¸, Phys. Rev., B 43 (1991) 12425-12430.
[8] H. Hashizume, M. Sugiyama, T. Niwa, O. Sakata, and P.L. Cowan, Rev. Sci. Instrum., 63 (1992) 1142-1146.
[9] Y. Takeda, Y. Sakuraba, K. Fujibayashi, M. Tabuchi, T. Kumamoto, I. Takahashi, J. Harada, and H. Kamei, Appl. Phys. Lett., 66 (1995) 332-334.
[10] M. Tabuchi, K. Hagiwara, A. Kobashi, N. Yamada, K. Fujibayashi, and Y. Takeda, Inst. Phys. Conf. Ser., 145 (1996) 227-232.
[11] M. Tabuchi, N. Yamada, K. Fujibayashi, T. Kumamoto, Y. Takeda, I. Takahashi, and J. Harada, J. Electron. Mater., 25 (1996) 671-675.
[12] M. Tabuchi, H. Kyouzu, M. Takemi, and Y. Takeda, Appl. Surf. Sci., 216 (2003) 526-531.
[13] M. Tabuchi, S. Hisadome, H. Yamada, R. Oga, and Y. Takeda, 2004 International Conference on Indium Phosphide and Related Materials (IPRM2004), WB3-2, Kagoshima, Japan, 30 May-4 June, 2004.
[14] S. Shibata, S. Doi, and I. Takahashi, Surface Science, 493 (2001) 42-48.

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