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Proposal for New X-Ray CTR Scattering Measurement System to Analyze Thick and Complicated Layer Structures

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X-ray crystal truncation rod (CTR) scattering measurement is a very powerful technique to investigate the interface structures in heteroepitaxially grown semiconductor layers. A two-dimensional detector, like Imaging Plate or charge-coupled device, is important to measure an X-ray CTR scattering spectrum in a short time. However, the resolution of the detector is not so high. The lower resolution of the detector limits the measurement of thick layers. A new X-ray CTR scattering measurement system with a 2θ-slit and a one-dimensional detector is proposed to realize a higher resolution without a serious increase of the measurement time. Using the new system, a layer up to 150nm thick is expected to be analyzed by the X-ray CTR scattering measurement.
Key words: X-ray CTR scattering, interface, heterostructure, new measurement system

1. INTRODUCTION

When X-ray diffraction of a crystal with a flat surface is measured in detail, a line-like distribution of the X-ray intensity is observed around a Bragg point, which is called a crystal truncation rod (CTR) scattering. Abrupt truncation of the periodicity of a crystal at a surface causes the line-like (rod-like) distribution of the X-ray intensity. Since structure of overlayers grown on the surface modulates the X-ray intensity in the line-like distribution, plenty of information about the structure of the overlayers can be deduced from the X-ray CTR scattering spectra [1-3]. Therefore, the X-ray CTR scattering measurement was used to investigate the structure and the roughness of the surface[4,5] and interface [6-8].

We have successfully applied the X-ray CTR scattering measurement to study the epitaxially grown semiconductor interfaces and have demonstrated that it is a very powerful technique to reveal the atomic scale interface structures[9-15].

The X-ray CTR scattering measurements have been utilized for thin and simple layer structures which represent only a part of real complex device structures.

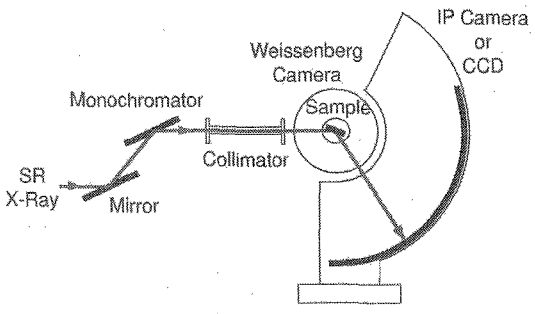

Fig. 1: X-ray CTR scattering measurement system with IP or CCD as detector.

In some case, the simplified model structures are effective. However, sometimes it is essentially important to analyze thick and complicated layer structures without the simplification.

In this paper, we discuss why only the thin and simple layer structures have been analyzed by the X-ray CTR scattering measurement and make proposal for a new X-ray CTR sacttering measurement system to analyze thick and complicated layer structures.

2. X-RAY CTR SCATTERING MEASUREMENT

We conducted the X-ray CTR scattering measurement using synchrotron radiation (SR) as an X-ray source. The scattering intensity was recorded using an Imaging Plate (IP) or a two-dimensional charge-coupled device (CCD) without slits and analyzer crystals between the sample and the detector (Fig. 1). The sample crystal was rotated typically in the range of +/- 5 degree around a Bragg angle since the X-ray CTR spectrum was observed widely around a Bragg peak. Figure 2 shows the measured X-ray scattering on the IP. The clear horizontal line-like distribution of the scattered X-ray

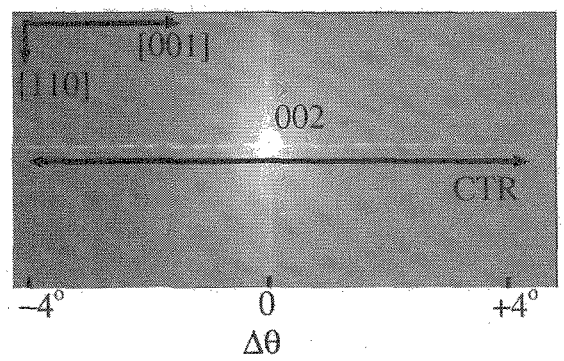

Fig. 2: Measured X-ray scattering using IP around the 002 Bragg point.

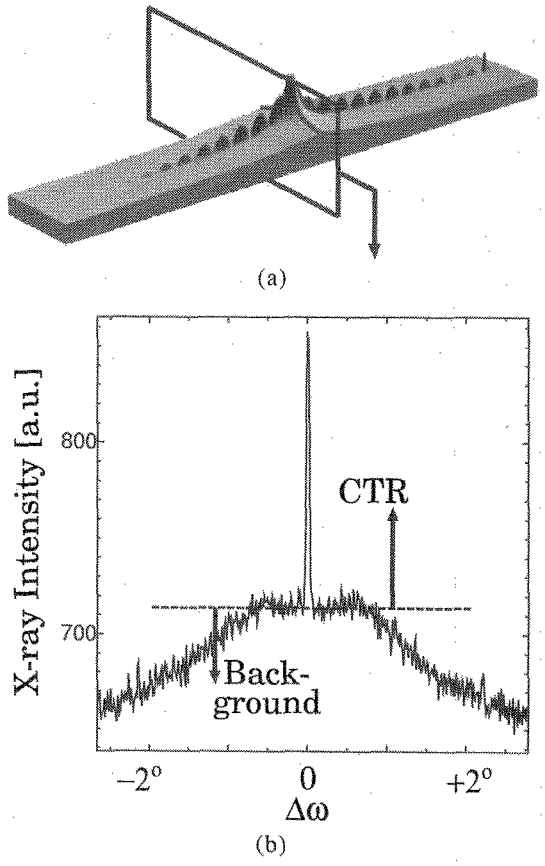

Fig. 3: (a) Three-dimensionally shown X-ray scattering intensity around a Bragg peak, and (b) a cross section of the distribution. In the cross sectional spectrum, the intensity of the X-ray CTR scattering can be separated from the background diffuse scattering.

intensity around the 002 Bragg peak was the X-ray CTR scattering. In the image, periodic modulation of the CTR intensity is observed, which was caused by epitaxially grown layers on top of a substrate.

Figure 3-(a) shows the measured scattering intensity on the IP, three-dimensionally. As shown in Fig. 3-(a), around the Bragg peak, it was difficult to neglect the diffuse scattering overlapping on the CTR. Therefore, when the CTR scattering was analyzed, the spectrum must be separated from the background diffuse scattering. The separation was achieved by evaluating the background intensity from the spectrum in the normal direction to the CTR, as shown in Fig. 3-(b). This was the reason why the distribution of the X-ray intensity must be measured two-dimensionally to investigate the CTR.

3. EXAMPLES OF THE CTR ANALYSIS

Figure. 4 shows a GaAs/InAs/GaAs sample structure[14]. The InAs layer of 1ML (molecular layer) was grown at 480°C. The X-ray CTR scattering measurement was conducted to show the distribution of In atoms in the samples before and after annealing. Figure 5 shows the distributions of the In atoms in the samples obtained. The results successfully showed that the In atoms were surely confined in 1ML before the annealing, and the In atoms drastically diffused into the GaAs layers by the annealing. This is one of the good

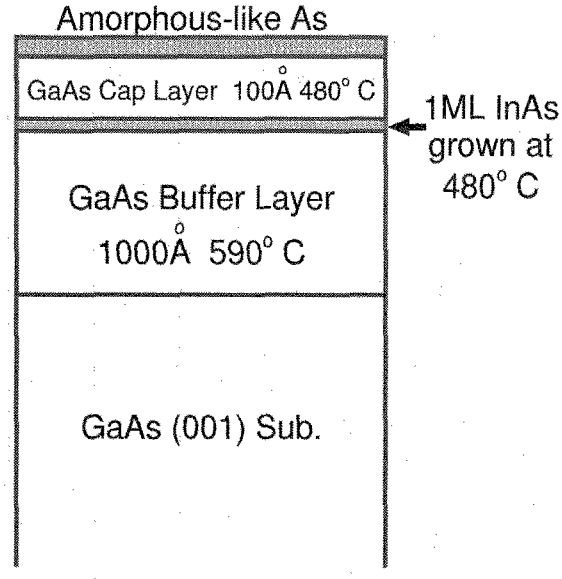

Fig. 4: GaAs/InAs/GaAs sample structure for the X-ray CTR scattering measurement. The surface was covered by amorphous-like As to prevent the surface oxidization.

examples which show that the X-ray CTR scattering measurement is a very powerful tool to investigate layers as thin as few MLs and interfaces in atomic scale.

Figure 6 shows another sample structure[15]. In this case, the distributions of In and P atoms into the GaAs layer depending on the growth temperature were analyzed by the X-ray CTR scattering measurement. Figure 7 shows the distributions of the In atoms in the GaAs/GaInP/GaAs structure samples obtained from the analysis. As shown in Fig. 7, the In atoms distribute into the GaAs cap layer widely when the layer was grown at 580°C. The results explained the reason why unintentional photoluminescence peaks were observed from the GaAs/GaInP/GaAs structure samples grown at 580°C

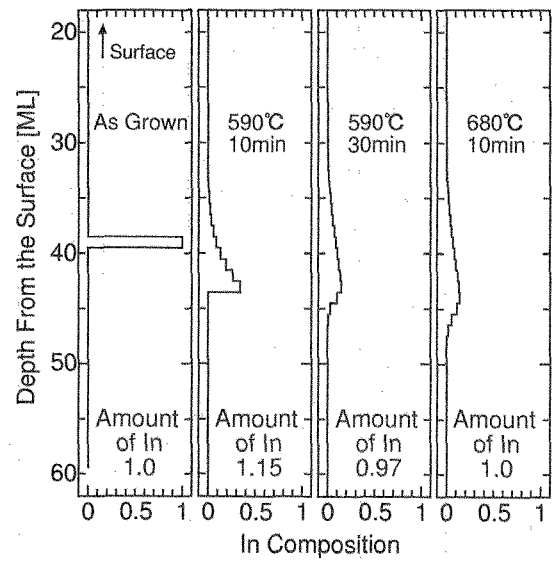

Fig. 5: Distributions of the In atoms in GaAs/InAs/GaAs structures before and after annaling obtained by the analysis of the X-ray CTR scattering spectra.

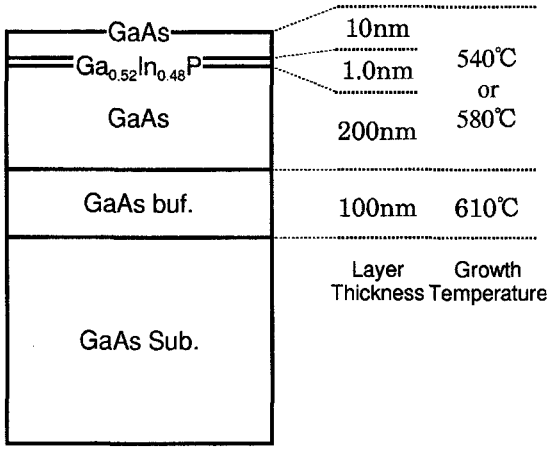

Fig. 6: GaAs/GaInP/GaAs sample structure measured to analyze the distributions of In and P depending on the growth temperatures.

On the other hand, the peak In compositions in the samples were about 0.3 when the GaInP layers were grown at 580°C. The result should not be understood that the composition of the GaInP layer cannot be 0.48 when the growth temperature is high. The result only suggested that 3ML was too thin to reach the final In composition as designed when the growth temperature was high. Therefore, we should analyze the samples with thick GaInP layers. However, it was difficult for us to measure an X-ray CTR scattering spectrum of the sample with thick layers.

4. PROPOSAL FOR NEW X-RAY CTR SCATTERING MEASUREMENT SYSTEM

The limitation of the measurement was mainly caused by lower resolution of the measurement system shown in Fig. 1. In the measurement system, an IP or a CCD

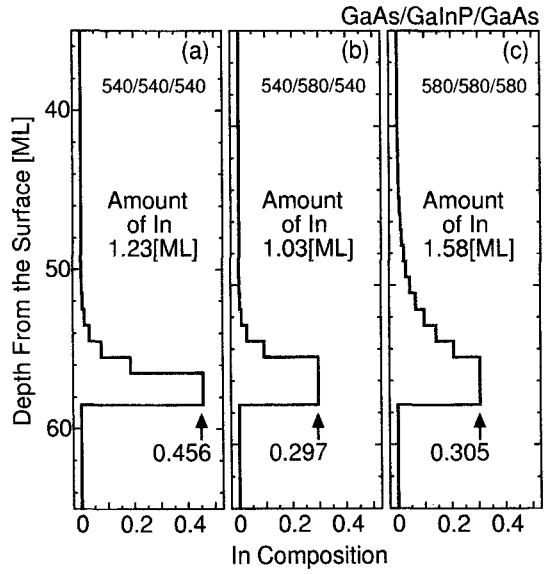

Fig. 7: Distributions of In atoms in the GaAs/GaInP/GaAs structure samles obtained from the analysis of the X-ray CTR scattering spectra. All the layers were grown at 540 and 580°C for the samples-(a) and (c), respectively. For the sample-(b), only the GaInP layer was grown at 580°C.

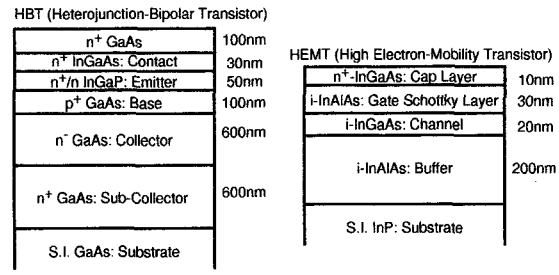

Fig. 8: Typical layer structures of recent high performance semiconductor devices: (a) HBT and (b) HEMT.

camera was used to record the scattered X-ray intensity. The pixel sizes of these devices are typically 0.1mm. The camera length is usually about 30cm in order to finish the measurement in about one hour. The resolution deduced from the parameters is about 35 arcsec (0.01°). From a spectrum measured in the resolution of 35arcsec, only the layers thinner than about 10nm (30ML) can be discussed.

Figure 8 shows examples of layer structures used for recent high performance semiconductor devices. The HBT (heterojunction-bipolar transistor) and the HEMT (high electron-mobility transistor) are very important devices since they work in the GHz region as amplifier. As shown in Fig. 8, in both of the structures, important layers (i.e., emitter, base, channel and gate layers) are usually 10-100nm thick.

To analyze such thick layers, higher resolution is required of the X-ray CTR scattering measurement. In order to realize the higher resolution without a serious increase of the measurement time, we propose a new measurement system as shown in Fig. 9. In the new system, the higher resolution along the CTR is achieved by the 2θ-slit, and two-dimensional measurement at one scan is realized using the one-dimensional detector to measure the distribution of the X-ray intensity normal to the CTR.

For example, with a 2θ-slit of 0.01mm on a 50cm long arm, the resolution reaches to 2 arcsec. With this resolution, the modulation on the CTR scattering caused by the thick layers up to about 150nm can be recorded and analyzed. It means that most of the interesting layers in the real HBT and HEMT structures can be measured using the new system.

However, when the sample consists of more than four

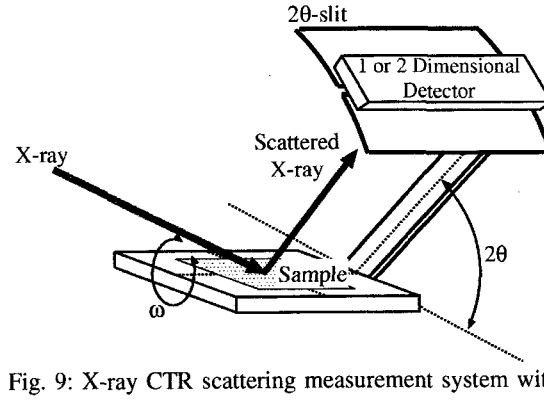

Fig. 9: X-ray CTR scattering measurement system with a 2θ-slit and a one-dimensional detector.

layers, like HEMT and HBT, the measured X-ray CTR scattering spectrum must be very complicated. In such cases, a new analysis technique is also required. One of the possibility of the new technique is to analyze only an important part of the measured spectrum. According to the layer structure, there must be some unique region in the spectrum where the change of the structure is sensitively represented. Therefore, by concentrating on the uniqe region, the analysis could be easier and in a short time.

5. CONCLUSIONS

The X-ray CTR scattering measurement showed that it was very useful technique to investigate the interface structure in heteroepitaxially grown semiconductor layers. A two-dimensional detector was important to measure an X-ray CTR scattering spectrum in a short time. However, the resolution of the detector was not so high. The resolution of the detector limited the measurement of thick layers. A new X-ray CTR scattering measurement system with a 2θ-slit and one-dimensional detector was proposed to realize a higher resolution without a serious increase of the measurement time. Using the new system, a layer up to 150nm thick is expected to be analyzed by the X-ray CTR scattering measurement.

References

[1] I.K. Robinson, *Phys. Rev. B*, **33,** 3830-3836 (1986).
[2] I.K. Robinson and D.J. Tweet, *Rep. Prog. Phys.*, **55,** 599-649 (1992).
[3] T. Shimura and J. Harada, *J. Appl. Cryst.*, **26,** 151-158 (1993).
[4] D.W. Kisker, G.B. Stephenson, P.H. Fuoss, F.J. Lamelas, S. Brennan, and P. Imperatori, *J. Cryst. Growth*, **124**, 1-9 (1992).
[5] F.J. Lamelas, P.H. Fuoss, D.W. Kisker, G.B. Stephenson, P. Imperatori, and S. Brennan, *Phys. Rev. B*, **49**, 1957-1965 (1994).
[6] I.K. Robinson, R.T. Tung, and R. Feidenhans'l, *Phys. Rev. B*, **38**, 3632-3635 (1988).
[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**, 12425-12430 (1991).
[8] H. Hashizume, M. Sugiyama, T. Niwa, O. Sakata, and P.L. Cowan, *Rev. Sci. Instrum.*, **63**, 1142-1145 (1992).
[9] Y. Takeda, Y. Sakuraba, K. Fujibayashi, M. Tabuchi, T. Kumamoto, I. Takahashi, J. Harada, and H. Kamei, *Appl. Phys. Lett.*, **66**, 332-334 (1995).
[10] M. Tabuchi, Y. Takeda, Y. Sakuraba, T. Kumamoto, K. Fujibayashi, I. Takahashi, J. Harada, and H. Kamei, *J. Cryst. Growth*, **146**, 148-152 (1995).
[11] M. Tabuchi, N. Yamada, K. Fujibayashi, T. Kumamoto, Y. Takeda, I. Takahashi, and J. Harada, *J. Electron. Mat.*, **25**, 671-675 (1996).
[12] M. Tabuchi, K. Hagiwara, A. Kobashi, N. Yamada, K. Fujibayashi, and Y. Takeda, *Inst. Phys. Conf. Ser.*, **145**, 227-232 (1995).
[13] M. Tabuchi, R. Takahashi, M. Araki, K. Hirayama, N. Futakuchi, Y. Shimogaki, Y. Nakano and Y. Takeda, *Appl. Surf. Sci.*, **159-160**, 250-255 (2000).
[14] M. Tabuchi, M. Araki and Y. Takeda, *Jpn. J. Appl. Phys.*, **41**, 1090-1093 (2002).
[15] M. Tabuchi, S. Hisadome, D. Katou, T. Yoshikane, A. Urakami, K. Inoue, A. Koizumi, Y. Fujiwara, and Y. Takeda, Inter. Conf. InP and related materials 2003, FB1.4, Santa Barbara, USA, May 12-16 (2003).

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