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Development of New X-ray CTR Scattering Measurement System Using Johansson Monochromator

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A new X-ray CTR scattering measurement system was reported that was equipped with a Johansson monocromator to focus X-rays at a sample position. Using the focused X-rays and a two-dimensional detector, X-ray CTR scattering measurement was able to be carried out without moving any component of the measurement system. The results of the X-ray CTR scattering measurement using the new system successfully demonstrated that the CTR scattering profiles were comparable to that measured at PF using synchrotron radiation even when the measurement time was 10 minutes or longer. The results suggested that the new measurement system is useful for quick and in-situ X-ray CTR scattering measurements.
Key words: X-ray CTR scattering, Johansson monochromator, focused X-ray, quick/in-situ measurement

1. INTRODUCTION

When a distribution of an X-ray intensity scattered by a crystal is measured carefully in reciprocal space, a rod-like distribution, which is normal to a surface of the crystal, could be observed around each Bragg point. The rod-like distribution of the X-ray intensity is called X-ray crystal truncation rod scattering (X-ray CTR scattering).[1-3] Since the origin of the X-ray CTR scattering is an abrupt truncation of a crystalline periodicity at a surface, and the intensity is sensitively modulated by non-periodic structures in the crystal, we could obtain much information on the surface and on the non-periodic structure from the X-ray CTR scattering profiles.[4-8] We have shown that interface structures, i.e., distributions of atoms across the interfaces, of hetero-epitaxially grown semiconductor crystals can be discussed at an atomic scale from the analysis of the measured X-ray CTR scattering profiles.[9-13]

Although, the X-ray CTR scattering measurement is so powerful to investigate an interface at an atomic scale, it has not been a popular measurement, since it is usually considered that a very brilliant X-ray source like a synchrotron is necessary for the X-ray CTR scattering measurement.

In this work, we report on the development of a new X-ray diffractometer equipped with a Johansson monochromator that is used to obtain focused X-rays at a sample position.[14,15] Using the focused X-rays, we could successfully reduce the X-ray CTR scattering measurement time even when an usual rotating-anode X-ray tube operated at 50kV and 300mA was used for the X-ray source. The system could be applicable to the in-situ X-ray diffraction and X-ray CTR scattering measurement when the measurement system is well established.

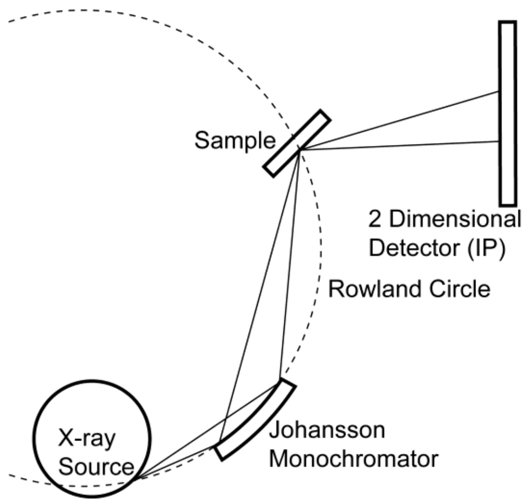

Fig. 1: Schematically drawn new measurement system equipped with a Johansson monochromator to focus X-ray at a sample position.

2. X-RAY CTR SCATTERING MEASUREMENT SYSTEM EQUIPPED WITH JOHANSSON MONOCHROMATOR

A new X-ray CTR scattering measurement system equipped with a Johansson monochromator was designed and constructed. Figure 1 shows the construction of the system schematically. The X-ray source was a line-focused rotating-anode X-ray tube operated at 50kV and 300mA (maximum operation power was 60 kV and 300 mA). The target was Cu. An asymmetrically cut Ge (111) Johansson crystal of which Rowland circle radius was 361mm was placed just after the X-ray source. Cu-Kα line was monochromatized by the Johansson crystal. The X-rays were focused at 220 mm away from the crystal and the maximum convergence angle of the X-rays

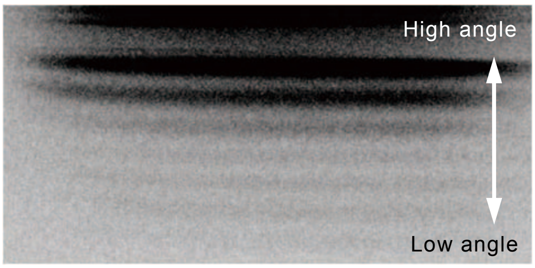

Fig. 2: Recorded X-ray scattering intensity on an Imaging Plate, using the new measurement sytem. Lower-angle-side region of InP 002 Bragg point was measured for an InP/GaInAs/InP structure sample for 10 minutes.

focused by the Johansson crystal was about 3.2 degrees, although, it was restricted to 2.85 degrees to eliminate the effect of the edge of the crystal. A goniometer was placed at the focusing point to hold a sample at a proper angle to the incident X-rays. An imaging plate (IP) was used as a detector to record the scattered X-rays two-dimensionally. It was important that there was no need to rotate X-ray source, sample, nor IP by utilizing the focused X-rays and the 2D detector. We could obtain a map of scattered X-ray intensities in a certain angle region at once without moving anything.

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

In order to discuss the potential of the new X-ray CTR scattering measurement system, an InP(30ML)/GaInAs(5ML)/InP structure crystal grown on InP substrate by OMVPE (organometallic vapor phase epitaxy) method was used. Figure 2 shows the scattered X-ray intensity distributions in the lower-angle-side region of InP 002 Bragg point for the sample recorded on the IP. The pattern was elongated in horizontal direction, since a line focused X-ray source was used. The focused image of the X-rays at the sample position was a line. The stripe pattern was observed in Fig. 2, since the CTR scattering intensity was modulated by the interference between the X-rays reflected by the InP and GaInAs layers. The Bragg peak, which should locate above the image, was not recorded intentionally, since the Bragg peak and diffuse scattering near the Bragg peak were too strong to observe the relatively weak CTR scattering profiles. As shown in Fig. 2, the X-ray CTR scattering and the modulation on the profile were clearly observed by using the proposed measurement system.

Figure 3 shows the measured X-ray CTR scattering profiles obtained from the distributions of scattered X-ray intensity as shown in Fig. 2 for the same sample. The measurement time was changed from 1 to 60 minutes and the measurement was conducted in the lower-angle-side region of the InP 002 Bragg point. The range of the index l, from 1.6 to 1.8, was corresponded to the convergence angle of the focused X-rays, 2.8 degrees. A profile measured at BL6A of Photon Factory at KEK in Tsukuba, Japan is also shown in Fig. 3, which had been already analyzed and given a good quantitative result on the interface structure at an atomic scale. Wavelength of the X-rays was set at 0.1nm and the measurement time was 15min, for the measurement at PF.

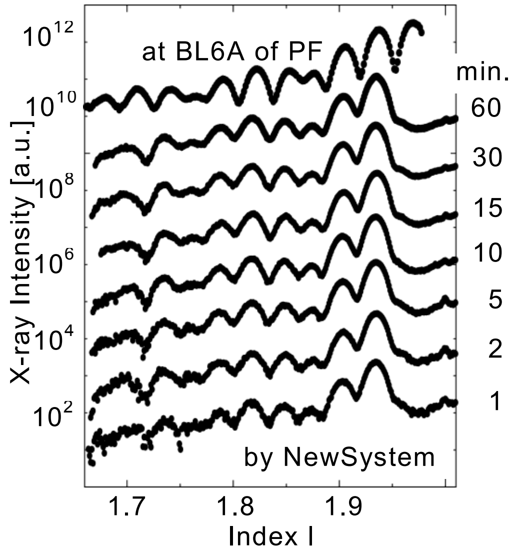

Fig. 3: X-ray CTR scattering profiles measured by the new measurement system and measured at PF. Even when the measurement time was so short as 1 minutes, the feature of the profile was well observed, though the background was relatively strong. The background level, S/N, decreased with the increase of measurement time, t, like as $S/N \propto \sqrt{1/t}$, as usual measurements using X-ray. Therefore, the background level at 60min was roughly about 1 10th of that at 1min. When the measurement time was 60 min the profile is comparable to that measured at PF.

As shown in Fig. 3, when the measurement time was 10 minutes or longer, the quality of the profiles was comparable to that measured at BL6A. We can understand that the profiles measured by using the new system showed identical features, i.e., positions of the peaks and valleys observed on the profiles and the relative difference of the heights and depths of them, to that measured at BL6A. The results indicated that the new system is available to analyze the layer structures.

Moreover, even when the measurement time was so short as 1 minutes, the quality of the profile was not so bad. It suggested that the new measurement system is potentially applicable for a quick measurement.

Further, we should point out that it is not necessary to move any components of the new measurement system including the sample, essentially. Therefore, the new measurement system should be very suitable for the in-situ measurement system combining with other experimental systems, i.e., crystal growth system, heat treatment system, optical measurement system, and so on.

4. CONCLUSIONS

A new X-ray CTR scattering measurement system was reported that was equipped with a Johansson monocromator to focus X-rays at a sample position. Using the focused X-rays and two-dimensional detector, the X-ray CTR scattering measurement could be carried out without moving any component of the measurement system. The results of the X-ray CTR scattering measurement using the new system successfully demonstrated that the CTR scattering profiles were comparable to that measured at PF using synchrotron radiation when the measurement time was 10 minutes or longer. The results suggested that the new measurement system is useful for quick and in-situ X-ray CTR scattering measurements.

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