

High-speed three-dimensional reciprocal-space mapping during MBE growth of InGaAs

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1. Introduction

Understanding strain relaxation process during III-V heteroepitaxial growth is important for reducing the dislocations in commercial electronic devices. *In situ* X-ray diffraction is a promising method for investigating both the strain relaxation and crystal quality during growth without inducing the thermal strain during quenching. Recently, we have performed *in situ* X-ray reciprocal-space mapping (RSM) during the growth of InGaAs/GaAs(001) to investigate the strain relaxation mechanisms in lattice-mismatched systems [1-3]. Evolution of strain relaxation and crystal quality was evaluated as a function of film thickness via the position and broadness of diffraction peaks in three-dimensional (3D) RSM. The time resolution was 83 s, which corresponds to an InGaAs thickness of 5 nm [2]. However, relaxation was found to happen within the film thickness increasing from 140 nm to 155 nm, which corresponded to only 4 3D-RSMs. To reveal the details of rapid strain relaxation, a faster RSM is required.

In this work, the authors have developed a high-speed 3D-RSM technique and applied it to *in situ* and real time monitoring of the MBE growth of InGaAs/GaAs(001) thin film with a time resolution of 10s.

2. Experimental procedure

The experiments were carried out at a synchrotron radiation facility, SPring-8 beamline 11XU, using an X-ray diffractometer integrated with a MBE chamber, which allows for *in situ* studies on the growth of the III-V group semiconductors. The details of this system are described elsewhere [4].

Figure 1 shows a schematic of the setup for X-ray measurements. X-ray wavelength used was 0.8270 Å. In order to obtain high time resolution RSM, diffracted X-rays were measured with a two-dimensional (2D) charge coupled device (CCD) camera placed at a distance of 697.7 mm from the sample. By using an area detector, any scan of sample or detector positions will result in a 3D data set.

For testing the feasibility of this fast technique, InGaAs/GaAs(001) was used as the sample. The substrate was cut from an epitaxial wafer of GaAs(001), mounted on

a molybdenum block, and loaded into the MBE chamber. After removal of the oxide layer and the growth of a 100-nm-thick buffer layer, 200-nm-thick In_{0.15}Ga_{0.85}As thin film was deposited at a rate of 0.2 ML/s. The growth temperature was 470°C as measured with an optical pyrometer. While the sample was continuously rotated around the surface normal axis in the vicinity of 022 Bragg point, the CCD detector took 60 images with exposure time range from 0.08s to 0.3s. Thus, 3D RSMs near the point 022 in reciprocal space was constructed. The time resolution was determined by the time required for the exposures and readout of CCD. For sample rotation, two motor speeds, 0.15°/s and 0.065°/s, were tested.

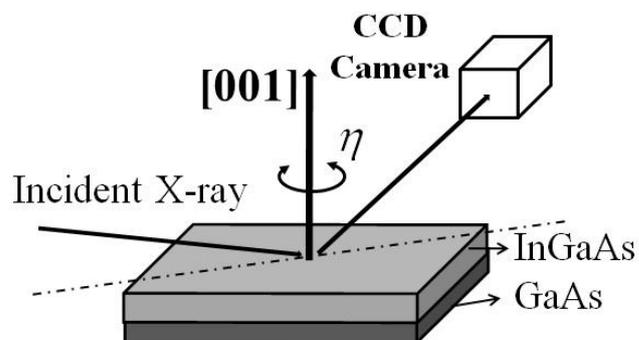


Fig. 1 Schematic drawing of the experimental setup for *in situ* x-ray diffraction of InGaAs/GaAs(001). The sample was continuously rotated around the surface normal axis, while the CCD detector took 60 images at different azimuthal angle, η , for each scan.

Furthermore, we applied this method to study the *in situ* growth of In_{0.18}Ga_{0.82}As (400 nm)/In_{0.07}Ga_{0.93}As (200 nm)/GaAs (100 nm) heterostructure onto GaAs(001) substrates. After the growth of a 100-nm-thick buffer layer, 3D-RSM measurement was carried out during the growth of first layer In_{0.07}Ga_{0.93}As and second layer In_{0.18}Ga_{0.82}As with a growth rate 0.17ML/s and 0.19ML/s, respectively. The growth temperature was 464°C.

3. Results and discussion

In order to confirm the feasibility of this fast method, we compared the peak profiles for various scan speeds and CCD exposure time (Fig. 2). The intensity profile of each scan has been corrected for sample rotation speed, exposure time, and integration area on each CCD images. Curves A and B show $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$ 022 peak profiles, which were measured by the conventional step RSMs, indicating the accurate InGaAs 022 peak position and width. For curves C to H, we first estimated a delay at the start of the motor rotation by using GaAs 022 as reference. In the inset of Fig. 2, we plot the corrected GaAs 022 Bragg diffraction profiles, which show the agreement of each scan.

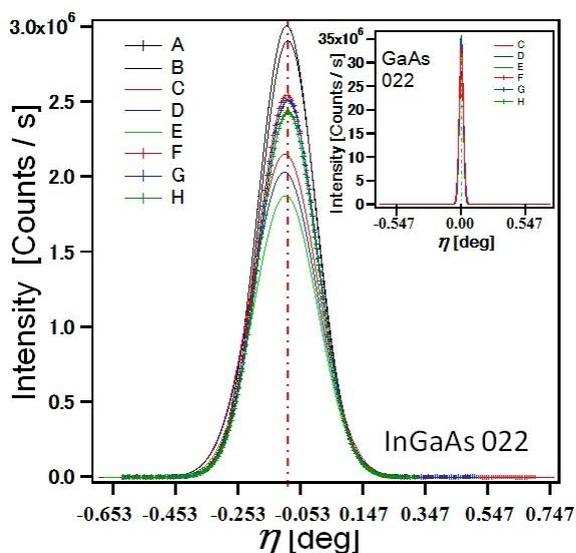


Fig. 2 Profiles of 022 Bragg diffraction of $\text{In}_{0.15}\text{Ga}_{0.85}\text{As}$ on $\text{GaAs}(001)$. Curves A and B correspond to the conventional measurements. Curves C to H show the fast RSM with different scan speed and CCD exposure time. The time resolution of solid curves is 8s, while that of marked curves is 17s

Curves C to E show the fast RSMs with a scan speed of 0.15 deg/s for CCD exposure times 0.12s, 0.10s and 0.08s, respectively. Curves F to H correspond to a scan speed 0.065 deg/s with exposure times of 0.30s, 0.25s and 0.20s, respectively. The vertical red and green dashed lines indicate the accurate of InGaAs 022 and GaAs 022 positions, respectively. The results indicate that the new fast technique provides an accuracy of 0.007 degrees in Bragg peak position and 0.0184 degrees in peak width. There are two possible error sources for peak width. One is a too short exposure time, which leads to a degraded signal statistics. Another is the fluctuation of the CCD exposure time. The accuracy of the exposure time is a few ten milliseconds. This makes it difficult to estimate the exact peak position and peak width.

Fig. 3 shows the continuous X-ray RSM during the growth of InGaAs/GaAs (100 nm) heterostructure. Three peaks indicate the 022 GaAs, 022 $\text{In}_{0.7}\text{Ga}_{0.93}\text{As}$ and 022 $\text{In}_{0.18}\text{Ga}_{0.82}\text{As}$ as marked in Fig. 3(a). Figure 3 (a)-(d) show projected images of the 3D-RSM in the $[-110]$ direction

during the growth of $\text{In}_{0.18}\text{Ga}_{0.82}\text{As}$ with thickness of 46.03 nm to 47.71 nm. The time resolution is 10s, which corresponds to 0.56 nm. The strain relaxation process was observed for both the $\text{In}_{0.18}\text{Ga}_{0.82}\text{As}$ and $\text{In}_{0.7}\text{Ga}_{0.93}\text{As}$ layers. Figures 3(a) and (b) show a drastic shift of two InGaAs peaks, indicating that a relaxation can be induced by the growth of only 2 ML $\text{In}_{0.18}\text{Ga}_{0.82}\text{As}$. In order to confirm the exact relaxation time and thickness, a higher scan speed will be necessary.

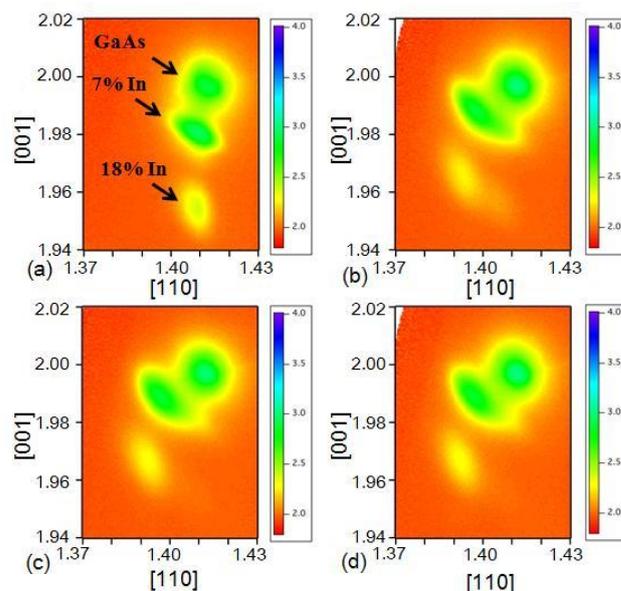


Fig.3 Real time X-ray RSM in the vicinity of 022 Bragg point during the growth of InGaAs films on $\text{GaAs}(001)$. Three peaks indicate the 022 GaAs Bragg peak, 022 $\text{In}_{0.7}\text{Ga}_{0.93}\text{As}$ peak and 022 $\text{In}_{0.18}\text{Ga}_{0.82}\text{As}$ peak, respectively.

4. Conclusion

We developed a high-speed 3D RSM technique for *in situ* monitoring of strain relaxation processes during InGaAs/GaAs(001) growth. The feasibility of this method was demonstrated by test measurements using an as-grown sample and *in situ* measurements during growth. The results show the effectiveness of this fast 3D-RSM method for the observation of rapid strain relaxation processes during heteroepitaxial growth.

References

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