High-resolution receiving optics for SmartLab X-ray diffraction system with a Hybrid Pixel Array Multi-Dimensional Detector (HyPix): HyRES220, HyRES400

1. Introduction

Diffraction-based characterization of epitaxial thin films, such as the measurement of the composition, strain, and lattice constants, requires complicated alignment processes of the X-ray optics. Rigaku's multipurpose X-ray diffractometer, "SmartLab," features an automatic alignment function, which greatly simplifies the process. However, it still requires a time-consuming re-alignment process when, for example, switching the optics between the standard and the high-resolution optical systems, which use the slits and an analyzer crystal, respectively.

Figure 1 shows an example of measurement flow for an epitaxial thin film. Generally, before the measurement of a sample with high crystallinity, such as a single crystal substrate or an epitaxial film, it is necessary to adjust the crystal orientation of the sample so that the measurement direction with a goniometer matches the crystal orientation. This procedure, called "axial adjustment," sometimes requires a modest resolution optics because the reflections can be difficult to find when the crystal orientation is tilted or the lattice constants are different from the literature values. There are also some types of measurements that do not require the high-resolution optics. Therefore, the ability to switch between the slit optical system, which has a relatively low resolution, for the axial adjustment and the high-resolution analyzer-crystal optical system for actual data collection will enhance the efficiency of the whole process.

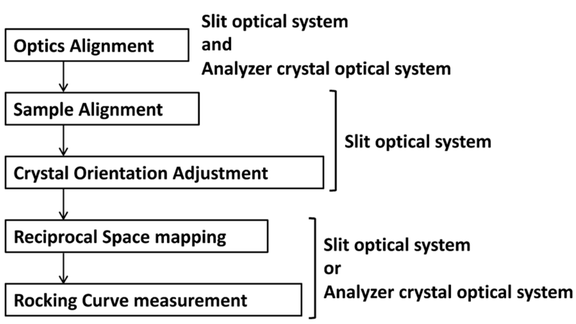

Fig. 1. An example of a measurement flow.
SmartLab Studio II adjusts both optical systems in the "optics alignment" Part Activity. Users adjust the sample position and crystal orientation by the slit optical system and measure the reciprocal space map and rocking curve by either optical system. Since the software automatically switches the optical system, adjustments and measurements can be executed without an interruption for switching the optic system.

The HyRES has been designed to simplify this switching process. This is a receiving optical device that has two optical paths: a slit optical system and a high-resolution optical system with an analyzer crystal. When combined with the HyPix series multidimensional pixel detector, the HyRES unit allows the user to seamlessly switch between the two optical systems through software control without a need of switching the optics hardware.

In this article, the principle and the features of the HyRES unit will be introduced with some analysis examples.

2. The Principle and the Features of the HyRES Unit

2.1. The internal structure of the HyRES unit

The HyRES unit has two optical paths: the slit system and the analyzer crystal optical system (Fig. 2). Those optical paths lead the diffracted beams on separate positions on the detector surface, allowing the user to measure either of these beams by changing the area of interest, or the "virtual slits," on the obtained 2-dimensional data. This means that the user can switch

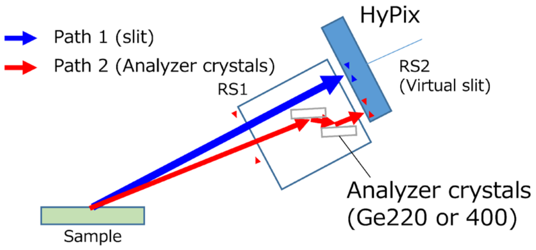

Fig. 2. The figure of the internal structure of the HyRES unit. The HyRES unit realizes a seamless switching of optical systems without the optics replacement by providing two optical paths.

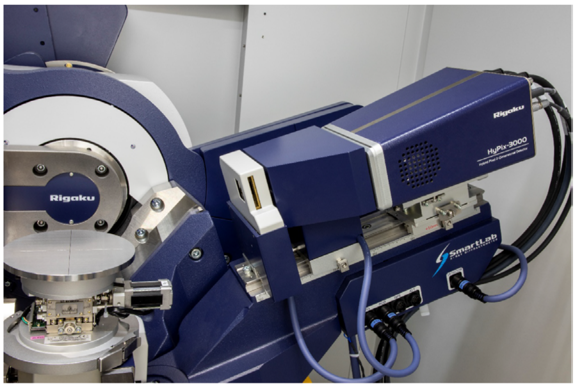

Fig. 3. The photograph of the HyRES unit.

the optics from one optical system to the other without a hardware switching. The HyRES series has two variations with different types of analyzer crystals: the HyRES 220 and HyRES 400 are equipped with a Ge220 and a Ge400 crystals, respectively, for the different ranges of the measurement angle.

2.2. Software control

SmartLab Studio II, the control software for SmartLab, can switch the optical system between the slit optical system and the analyzer crystal optical system. The software can also conduct an automatic adjustment sequence for both optical systems.

Figure 4 shows an example of the measurement-condition setting screen of SmartLab Studio II. The user can specify the type of the receiving optics for each Part Activity*. This means, by using the HyRES unit, the user can select the most efficient optical system for each process in the whole procedure. For example, he/she would want to perform the sample position adjustment and the axial adjustment with the slit optical system while he/she may want to use the high-resolution analyzer-crystal optical system for certain a rocking-curve measurements and reciprocal-space mapping. No matter how complicated the optics-switching program is, the SmartLab Studio II automatically chooses the specified optical system for each Part Activity so that the whole measurements is executed continuously and automatically.

* A Part Activity refers to a group of measurement operations performed by scanning and driving the axes for a specific purpose.

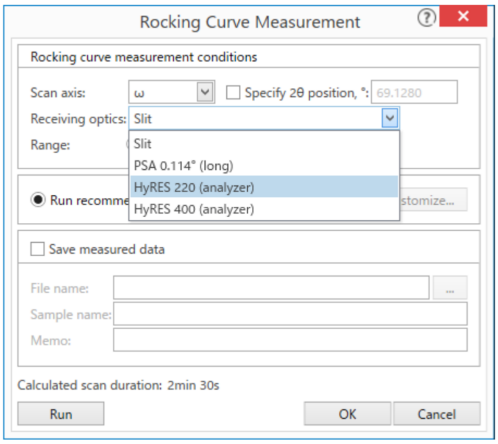

Fig. 4. The conditions dialog box of the Rocking Curve Measurement Part Activity of SmartLab Studio II.
Users can select an optical system for a measurement from the Receiving optics dropdown list in the conditions dialog box.

3. Evaluation Example of InGaN/GaN MQW Thin Film Using HyRES-220

Indium gallium nitride multiple quantum well (InGaN/GaN MQW) is an excellent material for LEDs, lasers, solar cells, etc. The properties of this material is known to be depend on the indium composition ratio and crystallinity. When used for LEDs for example, it is known that the emission wavelength and the luminous efficiency are dependent on the indium composition ratio and the crystallinity, respectively. X-ray diffraction can evaluate the indium composition ratio and the crystallinity as well as the strain using reciprocal space map and rocking curve measurements. In this chapter, we will introduce an evaluation example of InGaN/GaN MQW (Fig. 5) using the HyRES unit.

3.1. Reciprocal space mapping

Reciprocal space mapping is a method to evaluate the lattice constants, crystal orientation, and strain relaxation. This method has traditionally been conducted using a point detector, and an analyzer crystal optics has also been combined in many cases of high-resolution measurements. However, reciprocal space mapping using a point detector with analyzer crystal optics needs a long time, *e.g.* over 20 hours, in a laboratory. In recent years, it has become possible to measure the reciprocal space map in a short time by using a one-dimensional mode detector (1), and this setup is now widely used. These two types of reciprocal space mapping methods are used for different purposes depending on the required angular range, time, and resolution for the measurement.

Figure 6 shows the reciprocal space map of InGaN/GaN MQW measured with the HyRES 220 unit. Figure 6(a) shows the reciprocal space maps around the 002 reflection on the symmetric plane, and Fig. 6(b) shows the reciprocal space maps around the 105 reflection on the asymmetric plane. In both (a) and (b), the map on the left was measured using the one-dimensional mode of the detector and the slit optical system (measurement time: about 8 minutes), and the element on larger scale on the right was measured with the analyzer crystal optical system (measurement time: about 160 minutes).

The reciprocal space maps using the analyzer crystal optical system (Fig. 6, right) allows more accurate analyses because the reciprocal lattice points derived from GaN and InGaN separated clearly. On the other hand, reciprocal space maps using the one-dimensional mode of the detector and the slit optical system (Fig. 6, left) can be obtained in a shorter time. This method is useful when evaluating strain relaxation in a short time for example.

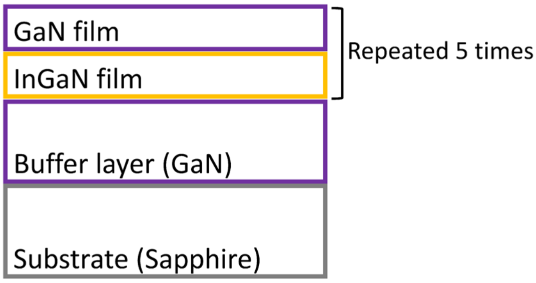

Fig. 5. Schematic diagram of the InGaN/GaN MQW thin film's structure used for the evaluation.

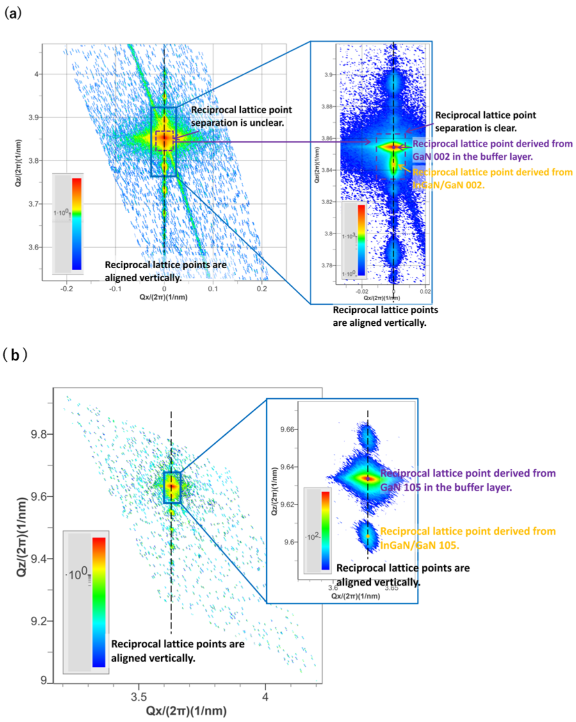

Fig. 6. Reciprocal space maps around (a) 002 reflection and (b) 105 reflection of InGaN/GaN MQW.
In both (a) and (b), the map on the left was measured using the one-dimensional mode of the detector and the slit optical system (measurement time: about 8 minutes), and the element on larger scale on the right was measured with the analyzer crystal optical system (measurement time: about 160 minutes).
The reciprocal space map of the 002 reflection suggests that the crystal orientations of InGaN aligned to the GaN. The reciprocal space mapping result of 105 reflection indicates that InGaN is fully strained to GaN and the lattice constants, *a* and *c*.

Since the *Qx* coordinates of the reciprocal lattice points derived from GaN and InGaN layers are the same in Fig. 6(a), the *c*-axes orientations of the GaN and the InGaN align to the stacking direction of the layers.

In Fig. 6(b), the *Qx* coordinates of the reciprocal lattice points derived from GaN and InGaN are equal, indicating that InGaN is completely lattice-matched to GaN. The lattice constants of GaN were calculated from the coordinates of the reciprocal lattice points as the *a*- and *c*-axes lengths to be 3.182 Å and 5.190 Å, respectively. It should be noted that the *a*-axis length of InGaN was found to be equal to that of the GaN, as expected from the conclusion that the lattices of the two layers were matched in the MQW superlattice.

3.2. High-resolution rocking curve measurement

The high-resolution rocking curve method is used for determining the film thickness and the composition ratio in thin-film samples, especially highly crystalline samples such as epitaxially grown films. In InGaN/GaN MQW, this method can evaluate the thicknesses of the individual InGaN and GaN films and the composition ratio of $In_xGa_{1-x}N$.

Figure 7 shows the rocking curve profiles of InGaN/GaN MQW measured with the optical system of the slit (blue line) and analyzer crystal (red line) in the HyRES 220 unit. The result using the analyzer crystal optics system shows Bragg peaks derived from InGaN/GaN and GaN, and the oscillation pattern derived from InGaN/GaN MQW. On the other hand, the result using the slit optical system shows a peak broadening, then the separation between Bragg peaks is unclear, and the oscillation pattern is not observed. This is because InGaN/GaN MQW has a curvature caused by a difference of coefficient of thermal expansion between GaN thin film and sapphire substrate. Therefore, the analyzer crystal optics system is suitable for the

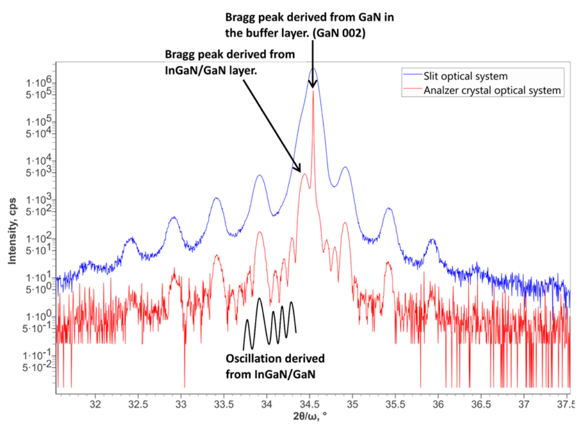

Fig. 7. The rocking curve profiles of InGaN/GaN MQW. Blue: Slit optical system, Red: Analyzer crystal optical system.
The result using the slit optical system shows the Bragg peak derived from InGaN/GaN and GaN cannot be separated, moreover, the oscillation pattern derived from the thin film cannot be observed. Therefore, the evaluation of InGaN/GaN MQW needs the measurement using an analyzer crystal optical system.

high-resolution rocking curve of InGaN/GaN MQW. The result using the analyzer crystal optical system revealed that the film thickness is InGaN: 3.37 nm, GaN: 15.21 nm, and the indium composition is 0.12.

4. Conclusion

The HyRES series provides two optical systems, the slit and the analyzer-crystal optical systems) switchable by the control software without changing optics hardware. This feature can reduce the measurement time of the whole process when the user wants to switch the system between the high-resolution optics and the faster optics, which often suit different purposes such as high-resolution data collection and other preliminary processes including the sample-position and crystal-orientation adjustments.

The feature of the HyRES was demonstrated in an evaluation of InGaN/GaN MQW thin film including the strain relaxation evaluation by reciprocal lattice mapping and film thickness and indium composition evaluation by rocking curve measurement. In this example, it turned out that the rocking-curve measurement required the analyzer-crystal optics while the either optic might suit the reciprocal-lattice mapping depending on the tradeoff between the time vs resolution. It has shown the automatic optics switching realized by the HyRES unit is useful for a series of these different types of measurements. It should also be noted that the unit is also useful for single measurement in most cases because it always involves the adjustments of the system optics and the sample orientation, which can be done faster using the slit optics.

In conclusion, the HyRES unit series enable continuous and automatic adjustments and measurements that require switching the receiving optics, while such operations used to involve the manual exchange of the optics hardware. The HyRES unit series have reduced the time and effort needed, moreover expanding its measurement application. Thus, the HyRES unit contributes to the efficiency, convenience, and productivity of all SmartLab system users.

References

(1) S. Kobayashi and K. Inaba: The Rigaku Journal, **28**(2012), 8–13.