Application Note XRD 617

Large-Area RSM with the EIGER2 R 500K

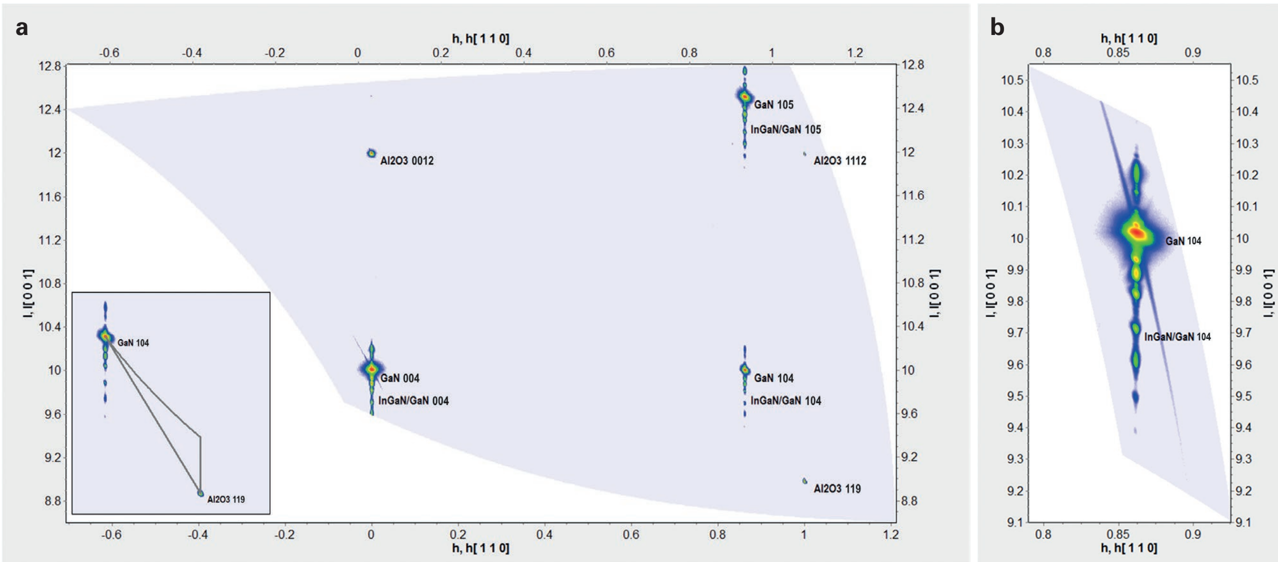
- Analysis of $In_xGa_{1-x}N$ LED, GaAs and $Si_{1-x}Ge_x$ epitaxial films

Epitaxial thin films form the basis of modern semiconductor technology with tiny nuances in manufacturing leading to dramatic changes in device performance. The ability to accurately measure subtle film properties is critical. Reciprocal space mapping (RSM) with XRD is the preferred technique for characterizing structural properties of thin films due to its ability to measure both perpendicular and lateral strain, composition, and domain effects.

Report Summary

- Decoupling relaxation, composition, tilt and mosaicity effects require capture of large areas of reciprocal space
- Detector distance setting was used to optimize reciprocal space coverage and resolution
- High dynamic-range capability ensures accurate peak-intensity determination for both film and substrate

The EIGER2 R 500K is a versatile hybrid photon counting detector with a large active area, enabling the fast collection of RSMs covering multiple substrate and film reflections with accurate peak intensities. In this report we demonstrate the benefits of this technique for two types of samples: an $In_xGa_{1-x}N$ blue LED, and single-layer GaAs and $Si_{1-x}Ge_x$ epitaxial films with different degrees of relaxation.



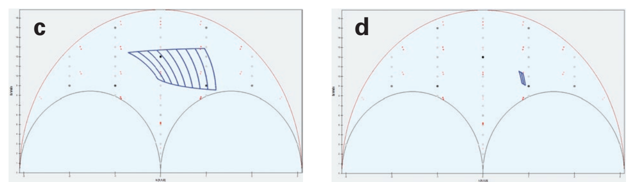


Figure 1. a) Large-area RSM of a blue LED taken at the close sample-to-detector distance (140 mm), showing multiple substrate and film reflections. Inset shows relaxation triangle indicating fully-relaxed GaN. b) Detailed RSM of GaN 104 taken at the far detector distance (470 mm). c, d) Reciprocal space coverage at the close and far detector distances, respectively.

Introduction

Epitaxial films are non-trivial to characterize due to a large number of inter-related structural effects. Strain caused by lattice mismatch can lead to peak shifts, but so does varying the alloy composition. Peak broadening may be attributed to finite crystallite size, misorientation between domains, strain relaxation with thickness, or a combination of all three. Coupled scans and rocking curves can access some of this information, but only by creating a map over reciprocal space the full picture is obtained. RSMs also make it easy to distinguish diffraction peaks from instrumental effects such as monochromator streaks, preventing erroneous interpretation of the data.

A large detector like the EIGER2 R 500K offers great flexibility for RSMs, allowing the user to capture a much wider region than would otherwise be practical. For example, figure 1 contains 2,061,030 data points. At 1 s per point this RSM would require 572 hrs to collect with a 0D detector, or 3 hrs with a typical 1D detector. With the EIGER2 the scan time is reduced to 0.5 hrs.

In the first experiment, this capability is explored to measure a complicated blue LED quantum well structure with a large mismatch between the substrate and film structure. In a second experiment, large-area RSMs of two single-layer films are collected including both on-axis and off-axis reflections allowing accurate determination of the degree of relaxation.

Experimental

Data were collected on a D8 DISCOVER diffractometer equipped with Cu radiation (40 kV/40 mA), Göbel mirror, 2-bounce Ge (004) monochromator, Centric Eulerian Cradle, and EIGER2 R 500K detector operating in 1D mode in the 2θ-optimized orientation. In this mode RSMs were generated by combining snapshot 2θ scans with scans over ω, 2θ-ω, or reciprocal space coordinates.

Optimizing Detector Distance for Coverage and Resolution

In the first experiment, a functional blue LED consisting of multiple $In_xGa_{1-x}N$/GaN quantum wells on c-plane Al_2O_3 was examined at close and far detector distances. At the close distance (140 mm) the 2θ coverage of the detector was nearly 30° (0.029° per pixel), and the RSM was generated by an h-sequence through reciprocal space (Figure 1a and 1c). Despite a fairly large lattice mismatch between Al_2O_3 and GaN, multiple substrate and film reflections are observed, making it possible to measure the relaxation of the initial GaN layer relative to the substrate (Figure 1a, inset). For improved resolution the detector was pushed back to 470 mm, and a detailed RSM of the GaN 104 peak was measured by an ω-sequence (rocking curve) through reciprocal space (Figure 1b and 1d). Here the 2θ coverage is 9° (0.009° per pixel), giving 3x the data point density allowing better separation in the device $In_XGa_{1-X}N$/GaN peaks.

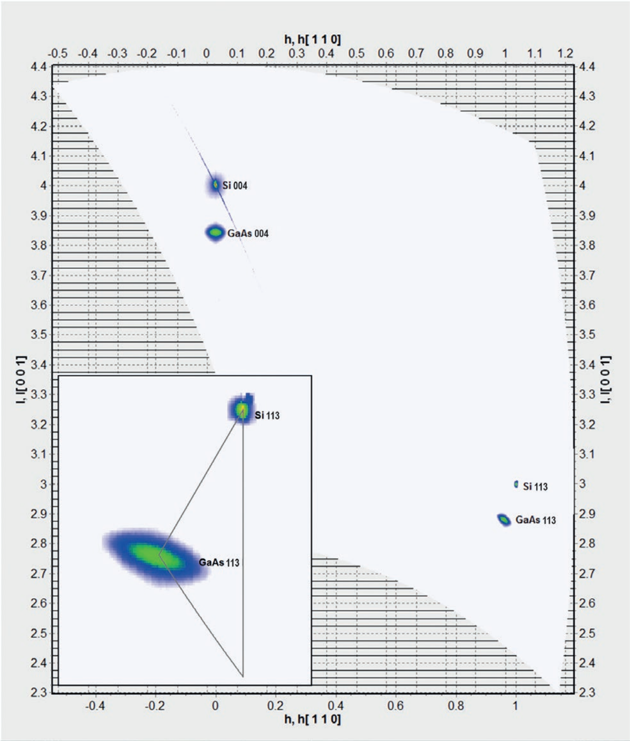


Figure 2. Large-area RSM of a relaxed GaAs film on Si. Inset shows zoomed-in image of GaAs 113 with relaxation triangle showing fully relaxed film.

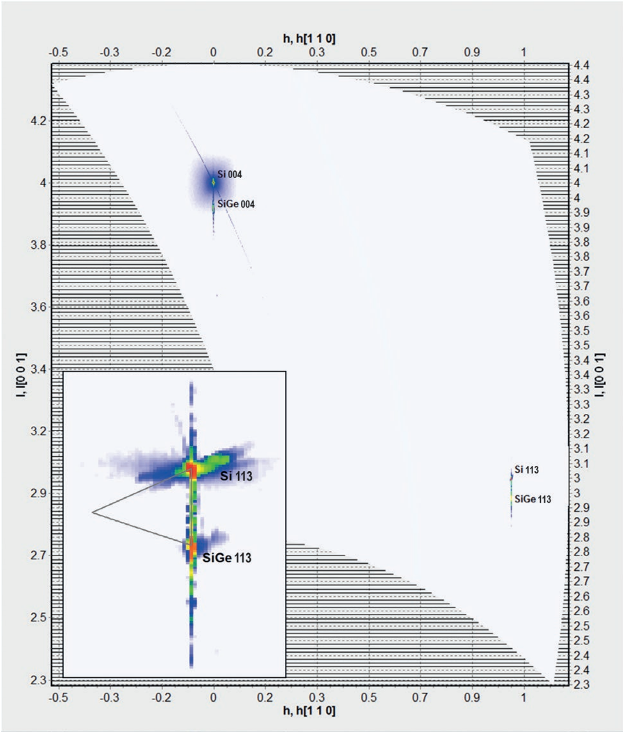


Figure 3. Large-area RSM of a strained $Si_{1-x}Ge_x$ film on Si. Inset shows zoomed-in image of $Si_{1-x}Ge_x$ 113 with relaxation triangle showing fully strained film.

Measuring Relaxation of Single-Layer Films

In a second experiment, large-area RSMs containing both on-axis and off-axis reflections of single-layer epitaxial films were collected at the close detector distance (140 mm). Having multiple reflections in the same map allows one to distinguish immediately between strain, composition, and tilt effects without introducing possible error due to variations in the sample alignment.

Figure 2 shows an RSM of a 70 nm GaAs layer on Si (001). The inset shows a relaxation triangle indicating a fully relaxed film. Vertical alignment of the 004 Si and GaAs peaks indicate the layer is crystallographically parallel to the substrate. Peak broadening is observed in a diagonal direction for GaAs 113, but a purely horizontal direction for GaAs 004, suggesting that its origin is a combination of mosaic spread and finite crystallite size.

Figure 3 shows an RSM of a 40 nm $Si_{1-x}Ge_x$ layer on Si (001). The relaxation triangle indicates a fully strained film, as the in-plane lattice constant matches that of the substrate. Furthermore, the size of the triangle corresponds to an alloy composition of x = 0.31. The sharpness of the film peak indicates well oriented domains giving rise to Pendellösung fringes. These were not observed in the GaAs film, likely due to its relaxed nature and mosaicity.

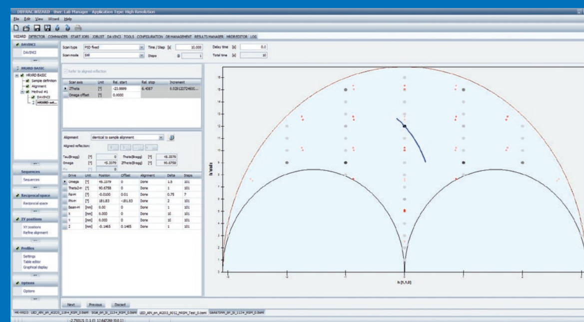
Conclusion

The coverage of the EIGER2 R 500K enables one to collect a wide region of reciprocal space in a reasonable time. This enables more comprehensive structural characterization of complicated device structures by giving access to multiple substrate and film reflections even in the case of a large lattice mismatch. Changing the sample to detector distance makes it possible to balance coverage and resolution giving the ability to resolve closely-spaced film peaks. Capturing on-axis and off-axis reflections simultaneously allows one to distinguish between effects which lead to peak shifts and/or broadening, including layer tilt, relaxation, composition and finite domain size. Additionally, capturing a large area of reciprocal space allows multiple analyses to be performed on a single data set. For example, in Figure 3 $Si_{1-x}Ge_x$ 113 is used to determine composition and strain, while thickness may be obtained more accurately from the $Si_{1-x}Ge_x$ 004 fringes due to higher specular intensity. Finally, the high dynamic range of the EIGER2 R 500K results in accurately recorded intensities enabling sections and areas to be extracted from the RSM for subsequent modeling and fitting.

DIFFRAC.SUITE Workflow for Reciprocal Space Mapping

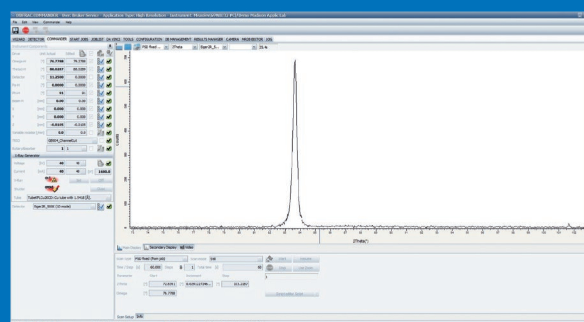
PLAN in DIFFRAC.WIZARD

- Quickly define the sample using a customizable materials database
- Preview reciprocal space coverage for different detector radii and scan types to find the perfect balance of coverage and resolution
- Visualize sequencing over angular (2θ, ω) or reciprocal space (h, k, l) coordinates to optimize data collection time



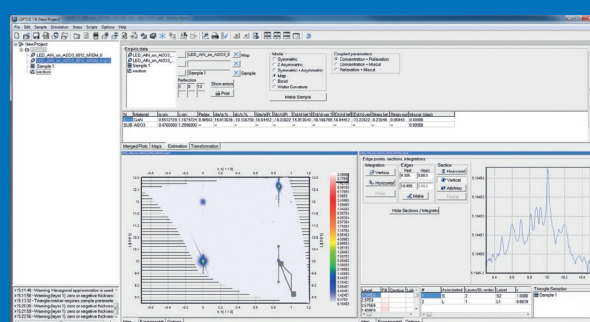
MEASURE in DIFFRAC.COMMANDER

- Direct measurement control or launch predefined experiment methods
- Automatic sample alignment with built in script editor
- Real-time data monitoring



ANALYZE in DIFFRAC.LEPTOS

- Quickly convert between angular space and reciprocal space
- Estimation tool for direct determination of film properties from the RSM
- Visualize film strain condition with relaxation triangle
- Extract sections for modeling and fitting



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