Reciprocal space mapping of epitaxial nanowires

APPLICATION NOTE

The growth dynamics, and the ultimate electronic band structure—hence the physical properties—of self-assembled epitaxial semiconductor nanowires and dots depends on the strain states of epitaxial thin films, which are functions of composition, morphology, and relaxation. High-resolution X-ray reciprocal space mapping (RSM) provides a nondestructive, yet quantitative, technique for the characterization of the strain, composition, and morphology of multi-dimensional nanowire or nanodot arrays. Rigaku's new Smartlab® automated diffractometer (Figure 1) makes reciprocal space mapping at medium, high, and very high resolution much easier and faster with minimal user interference.

Figure 1. SmartLab with X, Y stage

Measurement system description

The Rigaku SmartLab diffractometer is designed for multi-purpose applications, with a computer-guided, user-friendly optics system to achieve variable resolutions for RSM measurements for single crystals and epitaxial thin films. Rigaku's Cross Beam Optics (CBO) mirror—together with a Ge(220)x2 or Ge(220)x4 or Ge(440)x4 monochromator—conditions the incident X-ray beam into monochromatic radiation, with divergence down to a few arc seconds, as depicted in Figure 2. The automatically aligned Ge(220)x2 analyzer crystal on the detector arm completes a triple-axis configuration for high-resolution RSM measurements. The built-in Eulerian cradle and in-plane arm permits RSM measurements to be carried out in regions usually not accessible by conventional RSM methods, as shown in Figure 3.

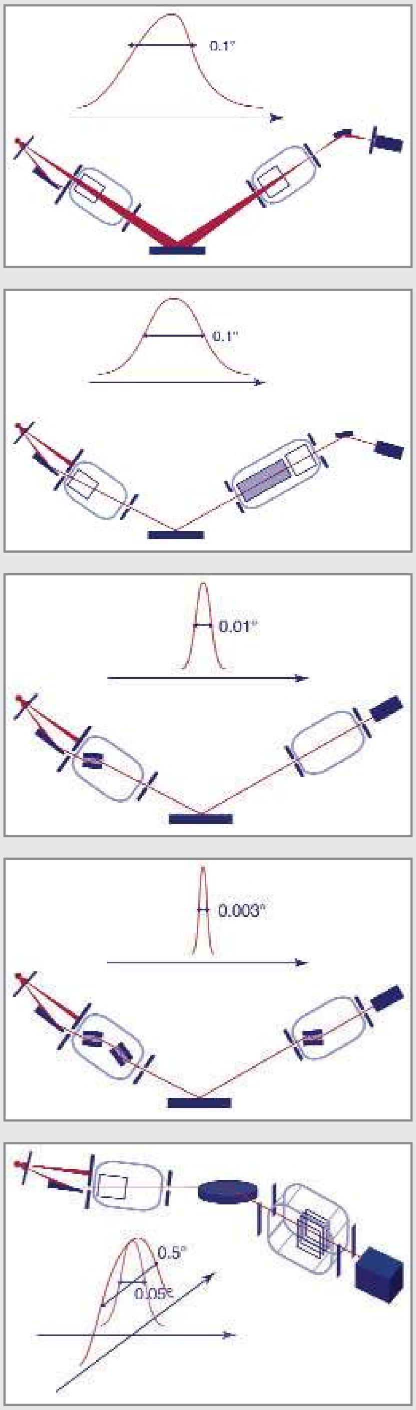

Figure 2. SmartLab configurations and corresponding resolution functions

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APPLICATION NOTE

High-resolution X-ray study of self-assembled epitaxial InAs nanowire arrays

Self-assembled semiconductor epitaxial nanowires and nanodots have been a subject of intensive study for more than a decade. A major problem that hinders investigation of these nanostructures is their poor size and spatial distributions. Since misfit strain is the driving force for the self-assembling process in an epitaxial system, it is crucial to be able to measure and control the strain state in an atomic layer level in order to improve the quality of the epitaxial nanostructures. High-resolution X-ray diffraction and reciprocal space mapping are techniques that enable quantitative characterization of strain and composition of many semiconductor epitaxial systems.

InAs on GaSb

The sample, consisting of 100-periods of 13 monolayers (ML) of InAs alternated by 13 MLs GaSb, was grown by molecular beam epitaxy (MBE). AFM and STM results indicate that an InAs nanowire array with GaSb spacer is formed in the epitaxial structure.

Data acquisition software

The SmartLab Guidance™ data acquisition software provides a package for automatic reciprocal space mapping measurement at the user's choice of resolution. The optics and sample alignments are completely automated. The diffraction space simulation function of SmartLab Guidance provides a preview of the reciprocal lattice points of both the substrate and the thin film, enabling users to move the diffractometer axes to the desired positions by a single mouse click (Figure 4).

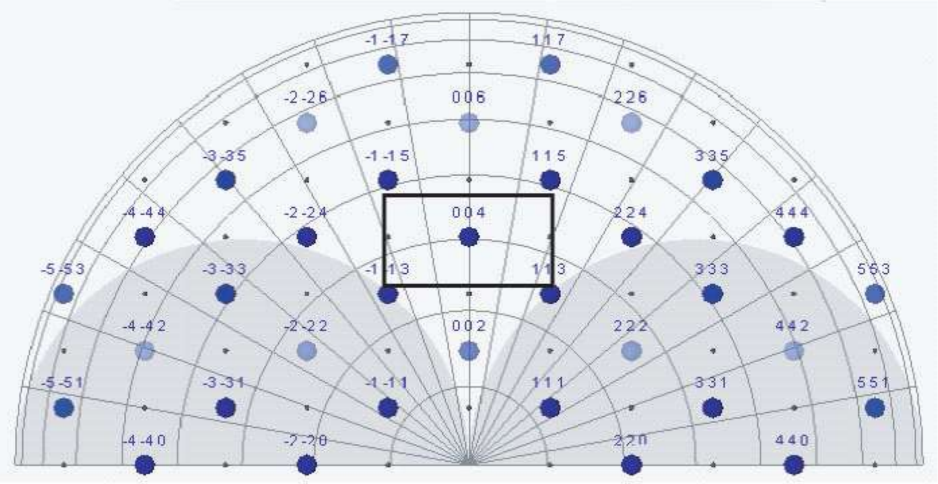
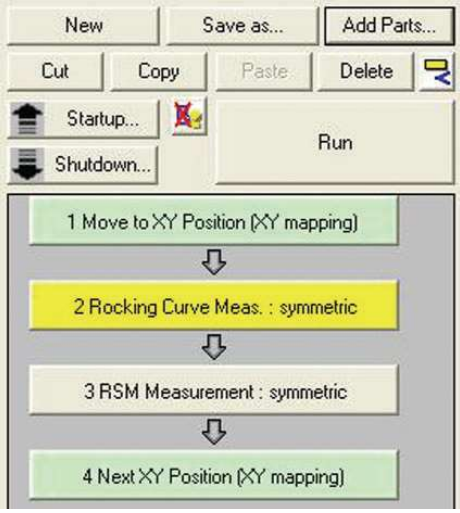


Figure 3. SmartLab allows access to the shaded reciprocal space areas forbidden for conventional RSM configurations

In addition, SmartLab Guidance's macro measurement capability allows multiple RSMs and other measurements, such as rocking curves, to be completed in a single run, as shown in Figure 4.



1 Move to XY Position (XY mapping)

2 Rocking Curve Meas. : symmetric

3 RSM Measurement : symmetric

4 Next XY Position (XY mapping)

Figure 4. SmartLab's XY mapping dialogue windows

RSM of epitaxial InAs nanowire arrays

Figure 5 shows an RSM of a two-dimensional epitaxial InAs nanowire array with GaSb spacer grown on a GaSb (001) substrate by molecular beam epitaxy (MBE). The RSM shows two-dimensional satellite peaks up to the 5th order, indicating that the nanowires are spatially well ordered and have a very narrow size distribution. From the peak separation, the nanowires were determined to have a cross section of 130 nm x16 nm.

Rigaku

Phone: 281-362-2300
Web: www.Rigaku.com E-mail: info@Rigaku.com

Model simulation

A model based on the microscopic images is proposed to simulate the experimental data, as shown in Figure 6. The nanowire array constructs a super unit cell consisting of two super "atoms" or nanowires. The diffracted X-ray intensity from such a structure can be calculated as

$$I(Q_x,Q_z) = const\cdot\left|F(Q_x,Q_z)\sum_m\sum_n\delta(Q_x \quad mG_x)\delta(Q_z \quad nG_z)\right|^2,$$

and $F(Q_x,Q_z) - [1+e^{i(Q_x\Lambda_x/2+Q_z\Lambda_z/2)}]\int\sigma(\mathbf{r})\rho_w(\mathbf{r})e^{i\mathbf{Q}\cdot[\mathbf{r}+\mathbf{u}(\mathbf{r})]}d\mathbf{r}$

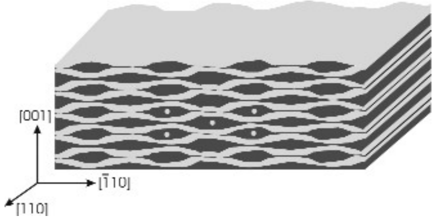


Figure 6. Model of nanowire structure used for theoretical simulations

Where, $G_i=2\pi/\Lambda_i$ (i=x,z)

(m,n) – orders of the 2D satellites

σ(r) – shape function of a single nanowire

ρ_w(r) – electron density function of a single nanowire

u(r) – the displacement field due to misfit strain

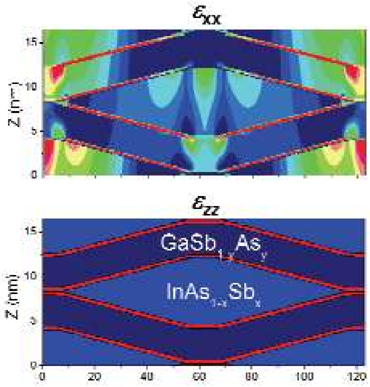


Figure 8. Calculated strain fields in a single nanowire based on continuum elastic theory

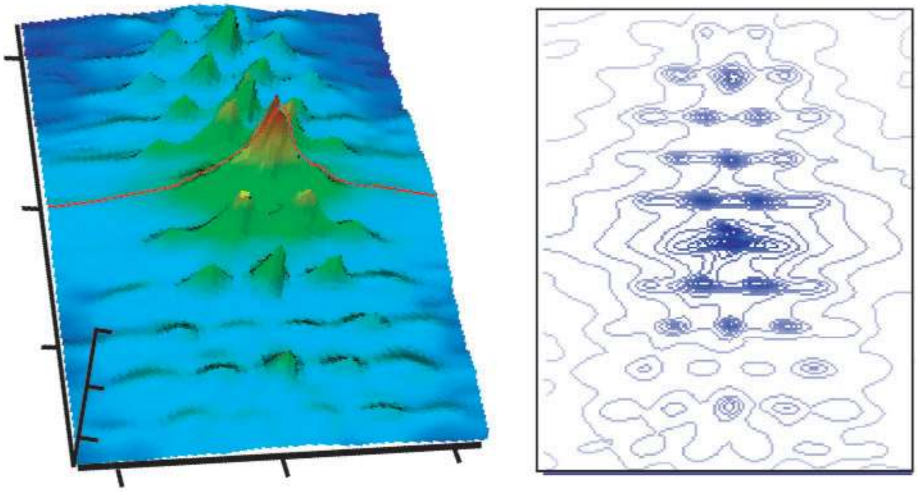


Figure 5. (004) reciprocal space map of a two-dimensional InAs nanowire array with GaSb spacer grown epitaxially on a GaSb (001) substrate. Shown on the left is a 3D contour map created by Rigaku's 3D explorer software. A 2D contour plot is shown on the right.

Results

Figure 7 shows the calculated RSM around the 004 substrate reflection. The line profiles cut along the dashed line in the RSM were compared to extract quantitative values of strain and composition of the nanowire array, (Figure 8). From the fit, we learn the following:

- The expected InAs layers have become $InAs_{0.88}Sb_{0.12}$ due to cross contamination during growth. Similarly, the GaSb layers were found to be $GaAs_{0.05}Sb_{0.95}$.
- The interfacial chemical bonds that bind the "InAs" and "GaSb" layers contains more than 95% InSb.
- InSb has a much larger lattice constant than GaSb, so that the InSb interfacial layers are subject to about 6.6% compressive strain.
- InAs, which is tensile strained upon deposition on GaSb, would counterbalance the compressive strain of the InSb interfacial layer leading to a flat superlattice structure, had it not changed to $InAs_{0.88}Sb_{0.12}$.
- The added small compressive strain of about 0.18% in the In $As_{0.88}Sb_{0.12}$ film on top of the heavily compressive strained InSb interfacial layer destabilizes the film and fine tunes the growth morphology, which is key to the formation of the observed highly ordered nanowire array.

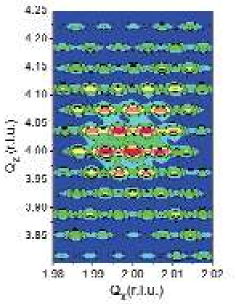


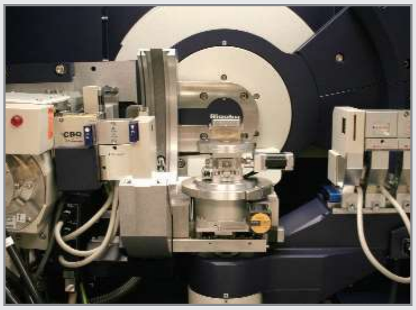
Figure 7. Calculated RSM

Acknowledgement

Donna Stokes, Kevin Bassler, and Simon Moss (University of Houston)

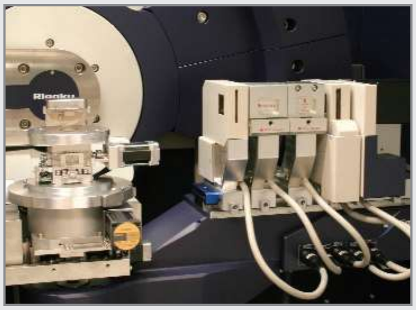
Specifications

X-ray generator:	Maximum rated output	3 kW sealed tube	9 kW rotating anode
	Rated tube voltage-current	20 - 60 kV 2 - 60 mA	20 - 45 kV 10 - 200 mA
	Stability	Within ±0.005% for 10% input power variation	
	Target	Cu (standard) (others: optional)	
	Focus size	0.4 x 12 mm line/point (standard) (others: optional)	0.4 x 8 mm line (standard)
	Radiation enclosure	Full safety shielding with failsafe open/close mechanism	
Goniometer:	Scanning mode	θs/θd coupled or θs, θd independent optical encoder controlled	
	Goniometer radius	300 mm (standard) (others: optional)	
	Minimum step	0.0001°	
	Eulerian cradle	χ: -5~95°/0.001° step ϕ: -720~720°/0.002° step Z: -4~1 mm/0.0005 mm step Optional X, Y stages: 20 mm/0.0005 mm step 100 mmϕ/0.0005 mm step 150 mmϕ/0.0005 mm step Optional Rx, Ry stage: -5~5°/0.002° step	
	Sample size	Max. 200 mmϕ x 6 mm thick (standard) (24 mm thick: optional)	
Optics:	Incidence optics	CBO, Ge 2-bounce and 4-bounce monochromators, automatic variable divergence slit	
	Receiving optics	Automatic variable scattering slit PSA, Ge 2-bounce analyzer, automatic variable receiving slit	
Detector:	Scintillation counter	Scintillator NaI, photomultiplier with preamplifier	


Goniometer system


incident beam optics


Sample stage


Diffracted beam optics