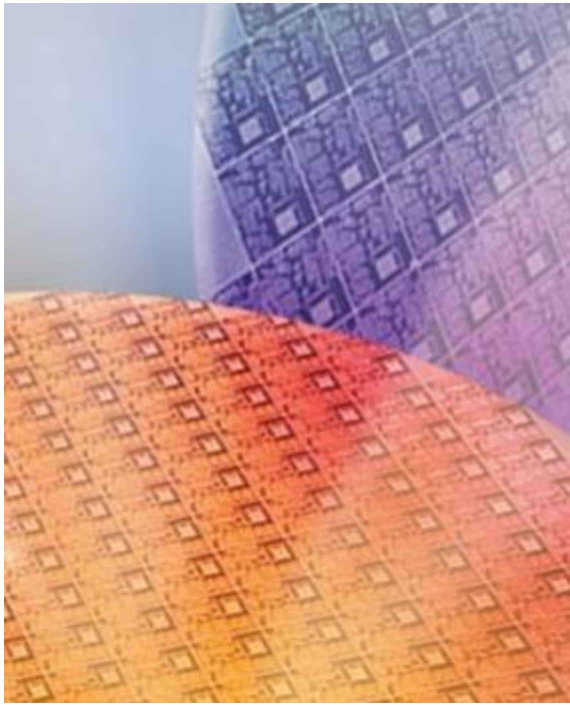
High-Resolution X-ray Diffraction of Epitaxial Thin-Films and Patterned Nanostructures

Matthew Wormington



Acknowledgements

- Colleagues
  - Paul Ryan & John Wall (JV UK)
  - Kevin Matney & Peter Gin (JV US)
  - Isaac Mazor, David Berman, Alex Krokhmal, Yuri Vinshtein & Alex Tokar (JV ILS)
- Collaborators
  - PY Hung (SEMATECH)
  - Manasa Medikonda & Alain Diebold (CNSE)
  - Jody Fronheiser & Vimal Kamineni (GlobalFoundries)
  - Benoit Lherron (IBM/ST)
  - Evguenia Karapetrova (APS)
  - Andreas Schulze, Hilde Tielens, Andriy Hikavyy, Roger Loo, Matty Caymax (IMEC)

Outline

- Introduction
  - Background
  - Principles of high-resolution X-ray diffraction (HRXRD)
  - Instrumentation
  - Diffraction geometries
- Measurements of epitaxial thin-films
- Reciprocal space and reciprocal space maps (RSMs)
  - RSMs from epitaxial thin-films
  - RSMs from patterned epitaxial nanostructures
- Synchrotron studies
- Conclusions

Jordan Valley overview

- Jordan Valley Semiconductors (JVS) develops and manufactures X-ray based in-line metrology and inspection solutions for the semiconductor industry
- Provide innovative solutions for a wide variety of materials, process and structure challenges
- Range of X-ray techniques including: XRF, XRR, (HR)XRD and XRDI
- Tools provide fully automated measurements, analysis and reporting and support semiconductor production and R&D activities worldwide

Jordan Valley overview (cont.)

- Private company
- Established 1982, HQ in Israel
- Global presence
    - > 180 employees
    - Manufacturing and demo sites (Israel & UK)
    - Local sales & support offices in strategic locations (US, Taiwan, Korea, Singapore, Europe)

Silicon semiconductor metrology tools

- **Tools:** JVX7300 series
- **Channels:** XRF, XRR and HRXRD
  Applications:
  - 7300HR: SiGe & Si:C on bulk or (FD)SOI, various ALD films, HKMG stacks, silicides…
  - 7300LSI: Ge and III/V on Si for sub-10 nm, HKMG, FinFETs, GaN-on-Si, MEMS
  - 7300F(R): Metal / magnetic films, WLP
  - 7300G: Ultra-thin films and 3D devices
- In-line tools for silicon semiconductor device manufacturers for process control of product wafers

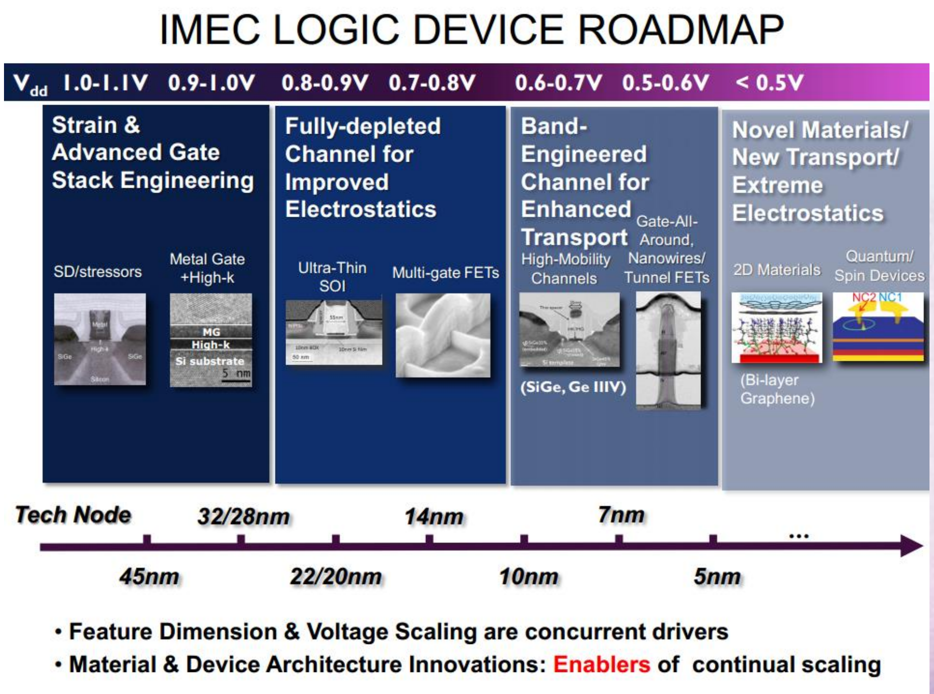
JVX7300LSI

What can HRXRD give us, who uses it and for what?

- High-resolution X-ray diffraction (HRXRD) provides a wealth of information about epitaxial materials
  - Crystal lattice misfit/strain, tilt and defectivity/quality…
  - Composition and thickness of planar films
  - Shape and lattice distortion in patterned structures
- It is first-principles (no calibration) and non-destructive characterization and metrology technique
  - Does not require material/process dependent optical constants
  - Accurate and precise with very few assumptions
- Has been used for 30+ years in the compound semiconductor industry for a wide range of materials (III-V, III-nitride, II-VI…) and devices (LEDs, lasers, CPV, detectors…)

What can HRXRD give us, who uses it and for what (cont.)?

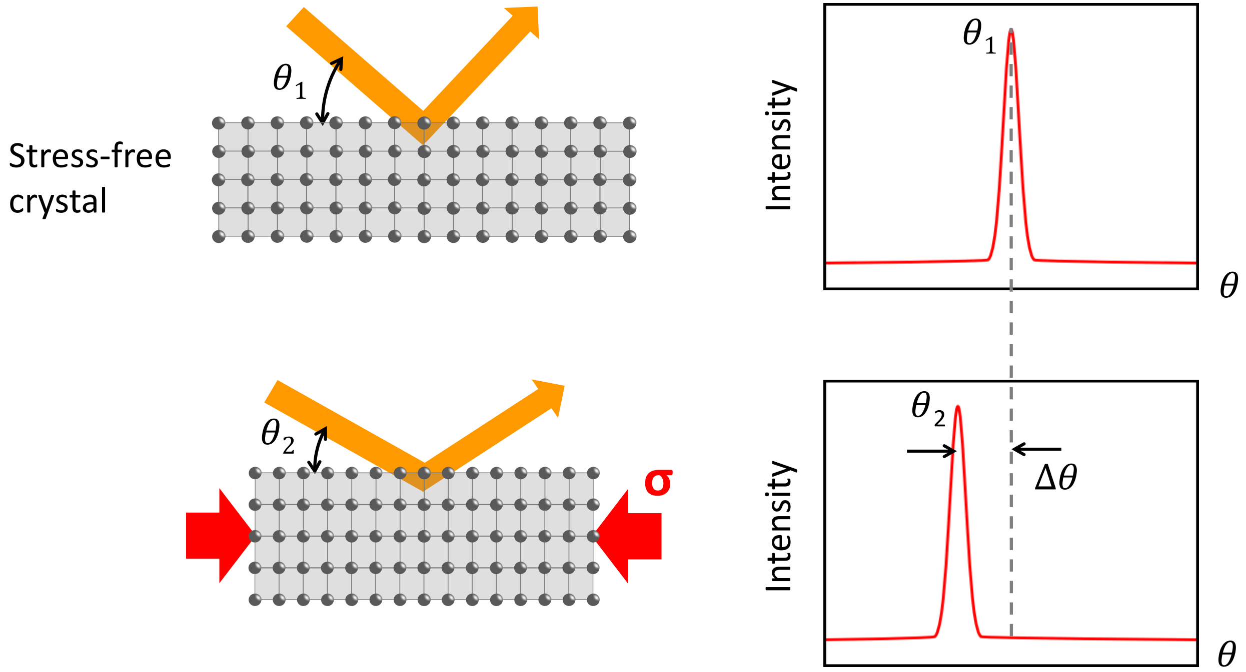
- Introduced into the Si industry with strain engineering for sub-100 nm logic devices
  - Epitaxial SiGe S/D stressors for PMOS mobility enhancement
  - Also Si:C / Si:P S/D stressors for NMOS, but less widespread
- Used for R&D, CVD chamber qual., process diagnostics / ramp and in-line metrology
- Solid metrology pads less relevant / not available
  - Transition from planar to 3D (FinFET) devices
  - Novel channel materials, e.g. SiGe, Ge and III-V for sub 10 nm nodes


IMEC LOGIC DEVICE ROADMAP

- Feature Dimension & Voltage Scaling are concurrent drivers
- Material & Device Architecture Innovations: Enablers of continual scaling

Source: A. Steegen, "Logic Scaling Beyond 10 nm", IMEC Technology Forum US (July 2013)

Principle of X-ray diffraction based stress/strain analysis



- X-ray diffraction uses the crystal lattice as a “strain gauge”
- The relation between the lattice parameter and diffraction angle is defined by Bragg’s law, $2d \sin\theta_B = n\lambda$
- Most sensitive stress/strain analysis method for semi. (ITRS 2011)

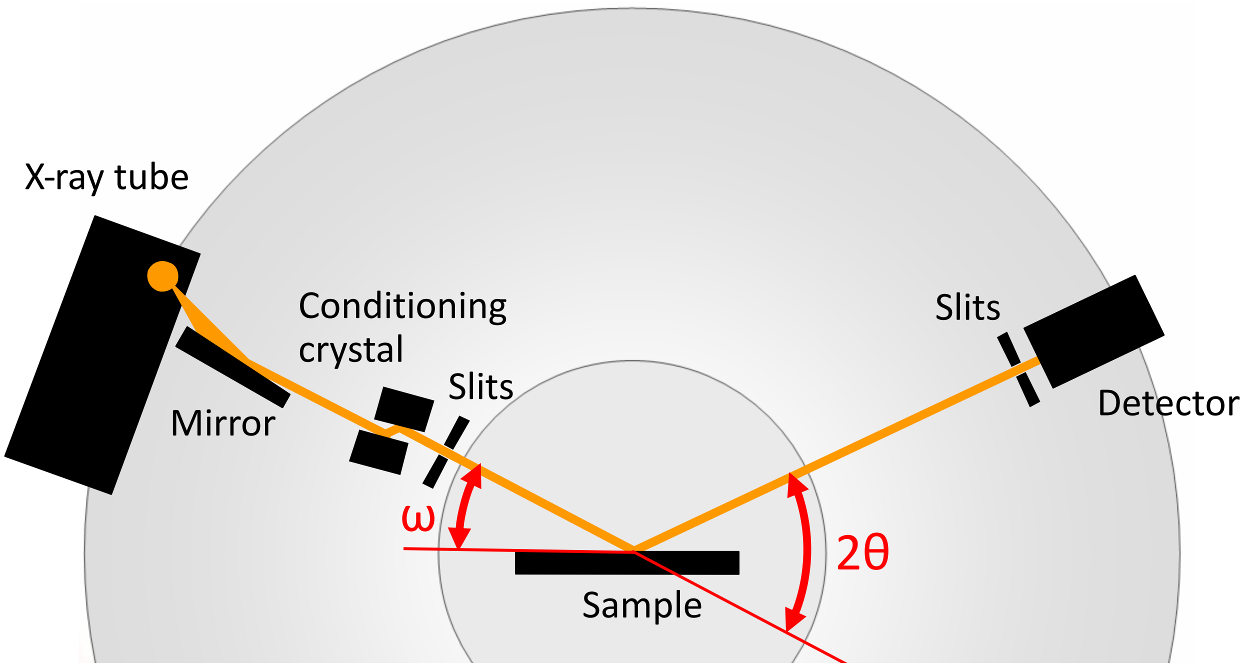
What do we mean by “high-resolution”?

- Epitaxial films and structures have a high degree of crystalline perfection
- The features (peaks and interference fringes) in the diffracted X-ray intensity from epilayer-substrate material systems are very closely spaced
  - angular range of a few degrees at most
- High-resolution is needed to resolve these features

$$\frac{\Delta d}{d} = \frac{\Delta\lambda}{\lambda} + \frac{\Delta\theta}{\tan\theta_B}$$

- High-resolution usually means highly collimated and monochromatic X-ray beams and precise goniometry

High resolution XRD setups

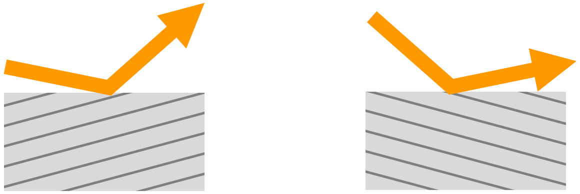


- Most common setup uses a parallel beam and point (0D) detector
  - Source and detector angles scanned using a motorized goniometer
  - Large (mm) and small (~50 um) spot configurations are available
- JV also developed an innovative small-spot, focused beam HRXRD approach for fast in-line measurements

Common Bragg diffraction geometries and anatomy of a HRXRD curve

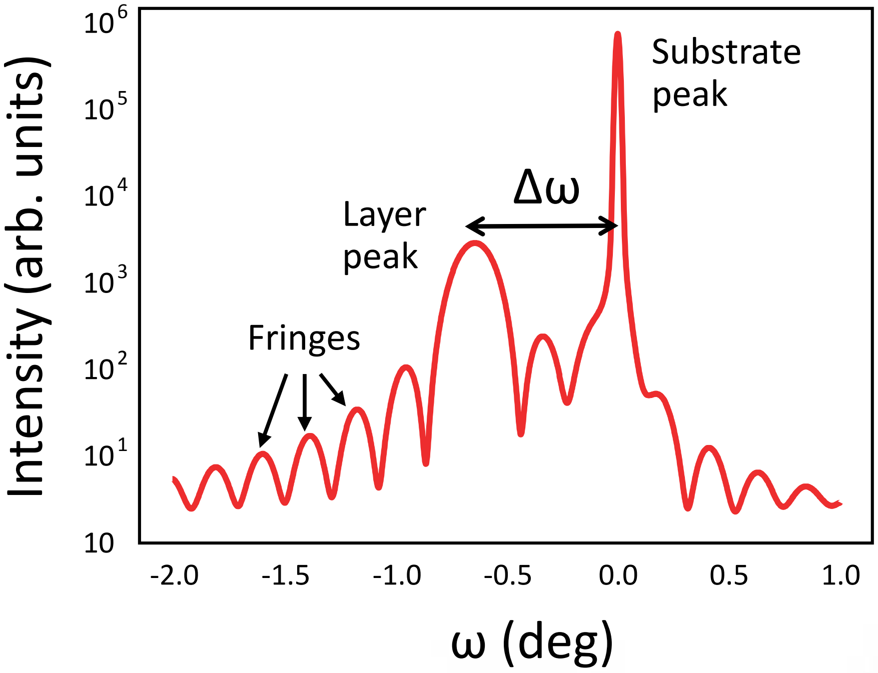
Symmetric geometry

Asymmetric geometries



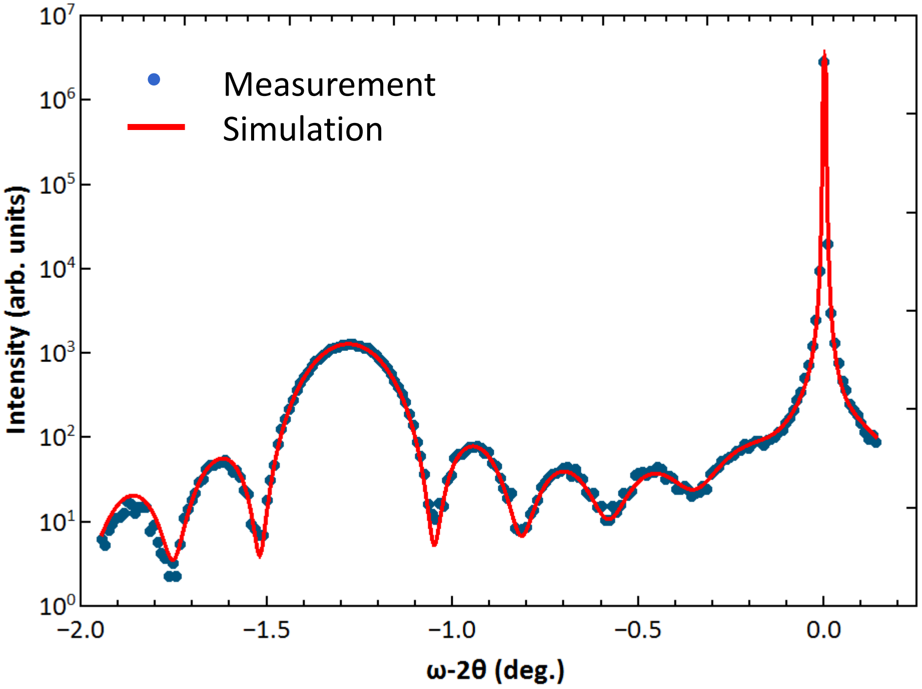
Glancing incidence

Glancing exit



- Symmetric Bragg geometry is sensitive to lattice parameter perpendicular to the surface
- Asymmetric geometries are also sensitive to the lattice parameters both parallel and perpendicular to the surface

Example: Fully strained SiGe epilayer



Symmetric 004 reflection from 22.5 nm epitaxial film of $Si_{1-x}Ge_x$ with x = 49% on a Si(001) substrate

- Composition / strain determined from measured lattice misfit
- Misfit normal to surface from layer peak position

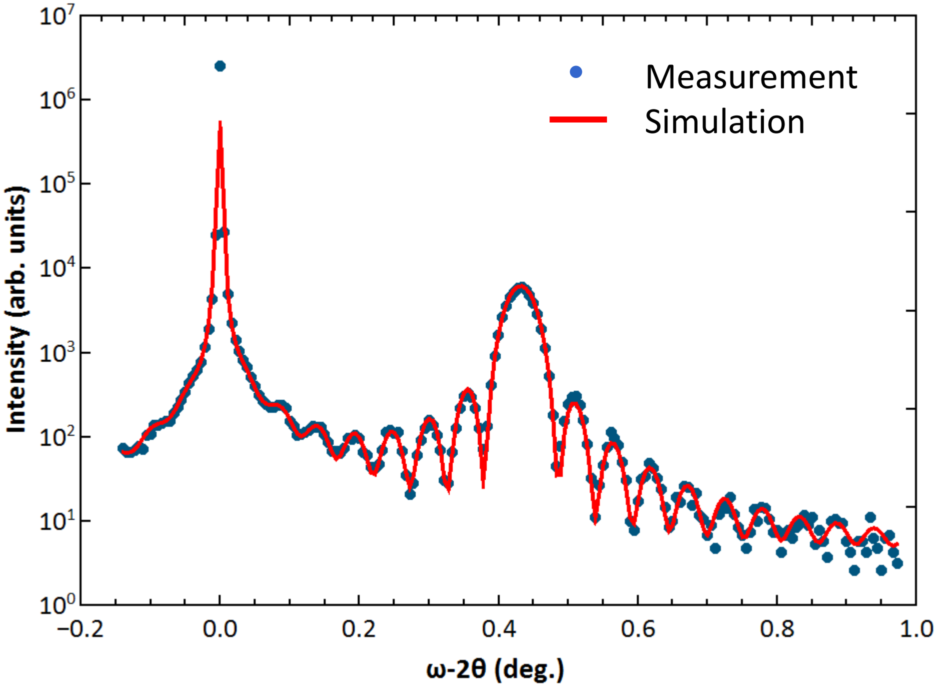
$$\Delta d/d = -\Delta\omega \cot\theta_B$$

- Thickness from interference fringe period

$$t = \lambda/(2\Delta\omega_f \cos\theta_B)$$

- No dependence on uncertain materials parameters

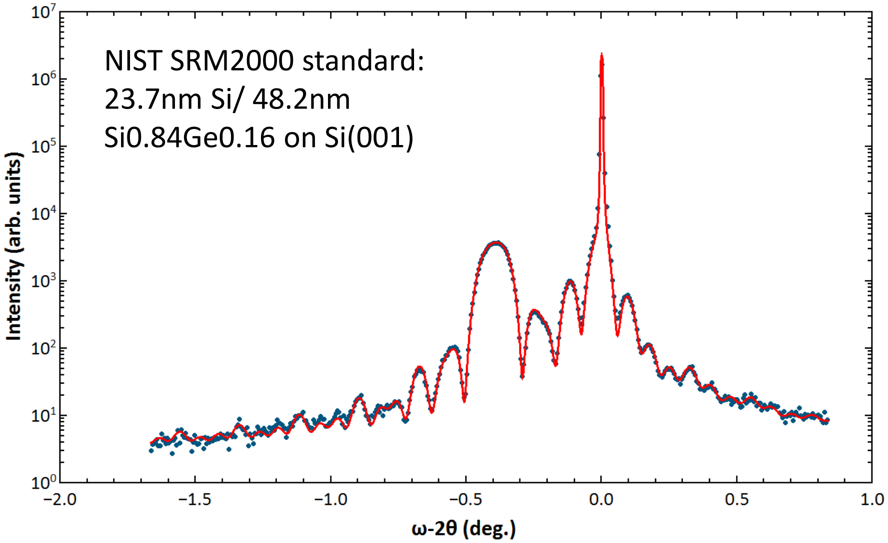
Example: Fully strained Si:C epilayer

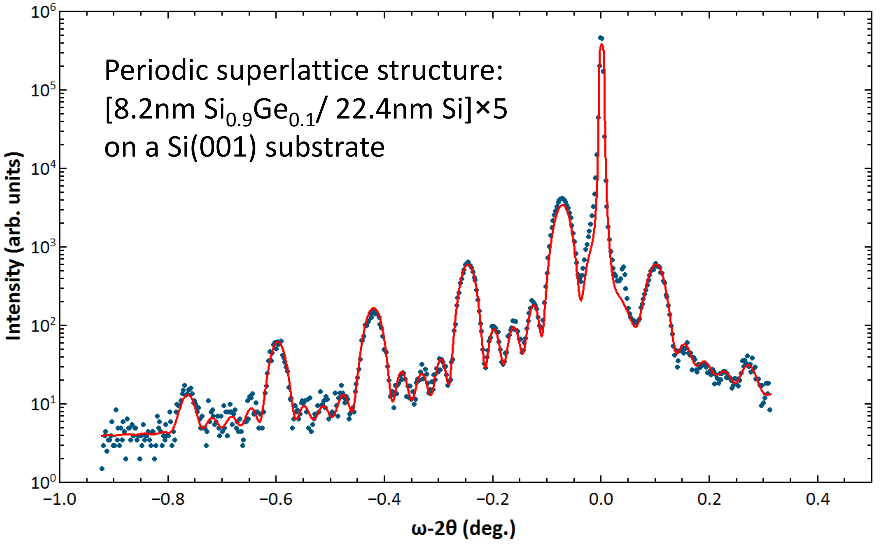


Symmetric 004 reflection from a 101.1 nm epitaxial film of $Si_{1-x}C_x$ with incorporated $x = 1.4\%$ on a Si(001) substrate

- Si:C and Si:P can be used as source/drain stressors for NMOS transistors
  - Composition and thickness metrology possible using HRXRD due to large strain
- Metrology is challenging using SE because of very low concentrations

More complex SiGe/Si examples


NIST SRM2000 standard:
23.7nm Si/ 48.2nm
Si0.84Ge0.16 on Si(001)

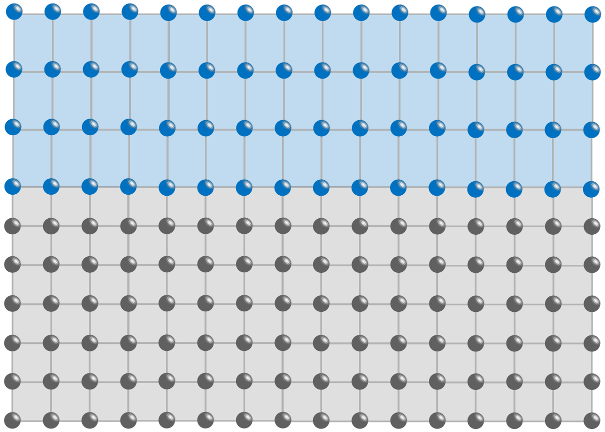

Periodic superlattice structure:
[8.2nm $Si_{0.9}Ge_{0.1}$/ 22.4nm Si]×5
on a Si(001) substrate

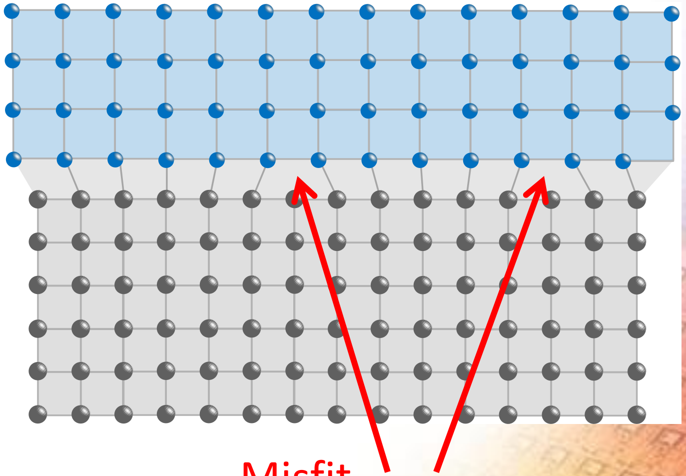
- More complex stacks give rise to more complex interference effects
- Measured data can be automatically fit to dynamical X-ray diffraction theory by refining the parameters of a structural model

Lattice deformation in epitaxial thin-films

- If the lattice mismatch to the substrate and/or thickness is small, then an epilayer can be strained so that the in-plane lattice parameter is equal to that of the substrate (fully strained)
  - Tetragonal distortion of the unit cell
- For large mismatch or thickness, it may become energetically favorable to relax
  - Creation of dislocations and/or roughening

… more on this later

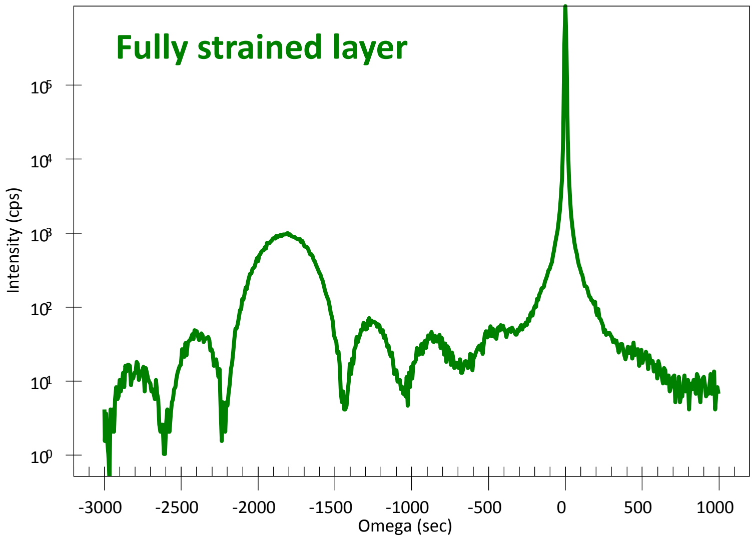


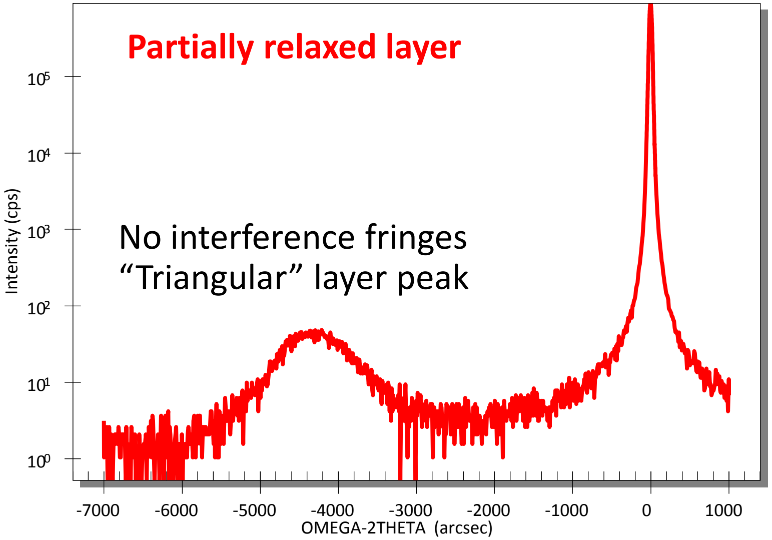


Misfit dislocations

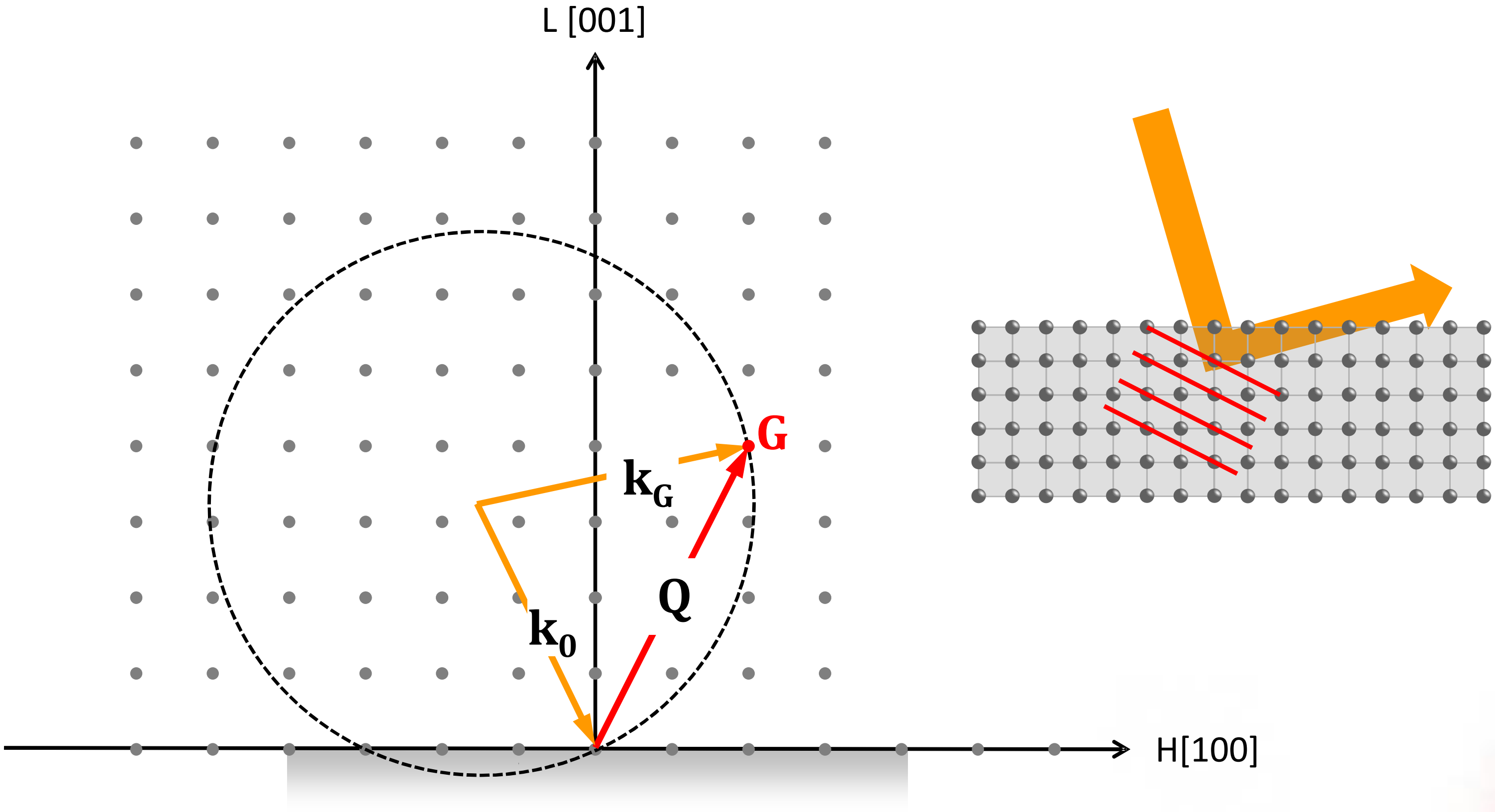
Comparison of HRXRD data from strained and relaxed SiGe epilayers

- Degradation of device performance and yield loss
  - Relaxed material has about 50% less strain than a pseudomorphic layer
  - Relaxed material will contain dislocations at the interface and in the layer - increased leakage?
- HRXRD provides a unique, automated solution for strain metrology and assessment of lattice defectivity



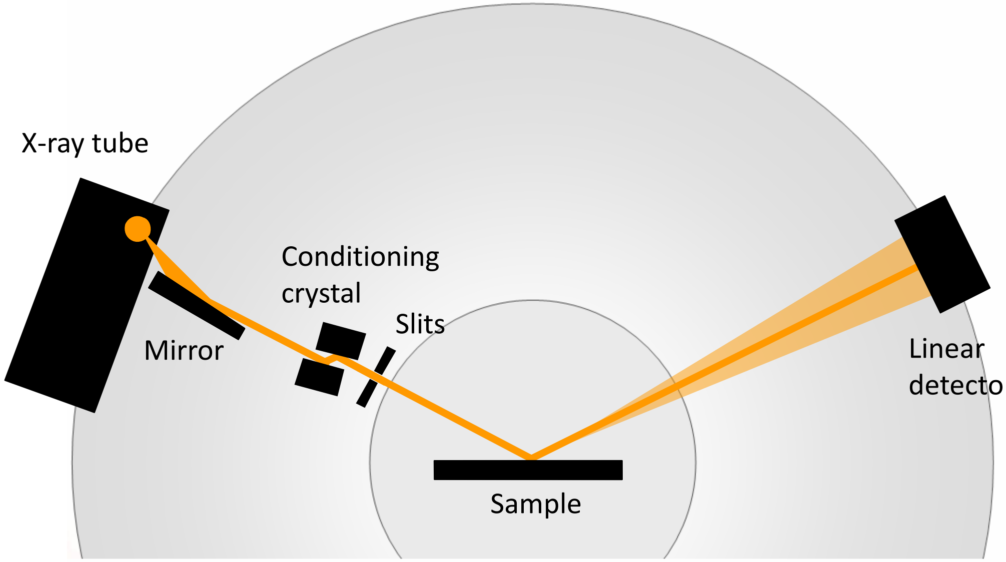


Diffraction in reciprocal space - Ewald sphere and Laue condition



- Scattering vector $\mathbf{Q} = \mathbf{k}' - \mathbf{k}$ has magnitude $|\mathbf{Q}| = 2\sin\theta \times 2\pi/\lambda$
- Reciprocal lattice vector **G** has magnitude $|\mathbf{G}| = 2\pi/d_{HKL}$
- Laue condition $\mathbf{Q} = \mathbf{G}$ is exactly equivalent to Bragg's law

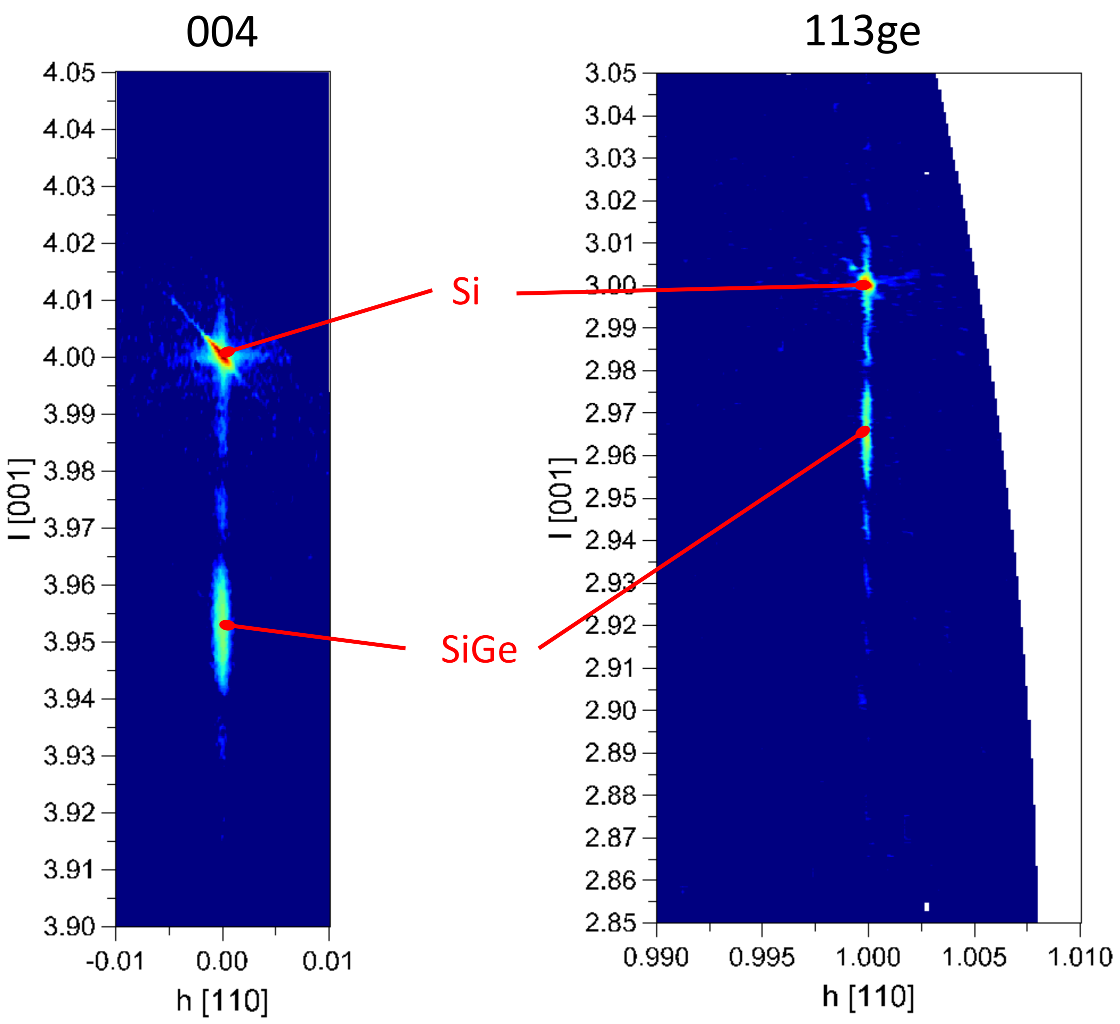
Fast reciprocal space mapping - FastRSMs



- Linear (1D) detector replaces analyzer crystal / slits and point (0D) detector and allows routine RSMs to be measured in the fab
  - Simultaneously intensity acquisition over a large range of 2θ angles
  - x10-100 faster than conventional approach (minutes not hours)
- Provides more information than available by single HRXRD curves
- Automated RSM analysis for epi. process development and control of thin-films and patterned nanostructures

RSMs from fully strained epitaxial thin-films

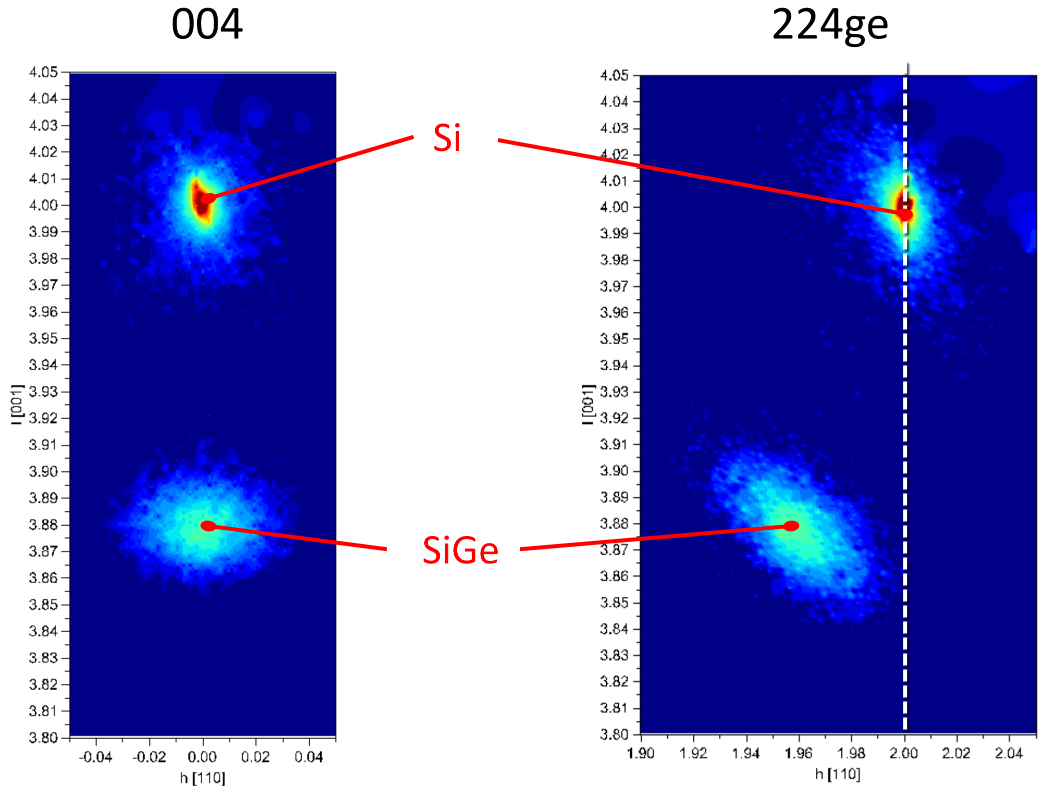
37.6 nm $Si_{0.81}Ge_{0.19}$ on Si(001)

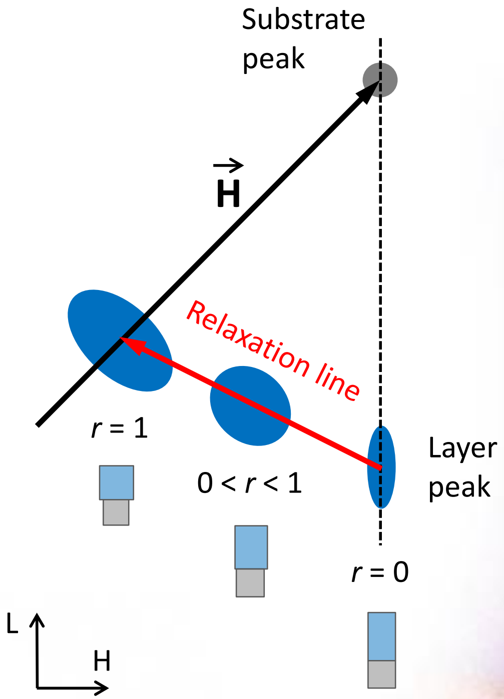


- Layer peak position gives the lattice parameters
  $$\Delta a_{\perp}/a = \Delta L/L$$
  $$\Delta a_{||}/a = \Delta H/H$$
- Composition and relaxation can be obtained
- Peak in the asymmetric RSM is located at H = 1 indicating the layer is fully strained ($a_{SiGe,||} = a_{Si}$)
- Thickness fringes are visible in the L direction
  $$t \propto 1/\Delta L_{fringes}$$

RSMs from relaxed epitaxial thin-films

~35 nm $Si_{0.33}Ge_{0.67}$ (R = 70%) on Si(001)

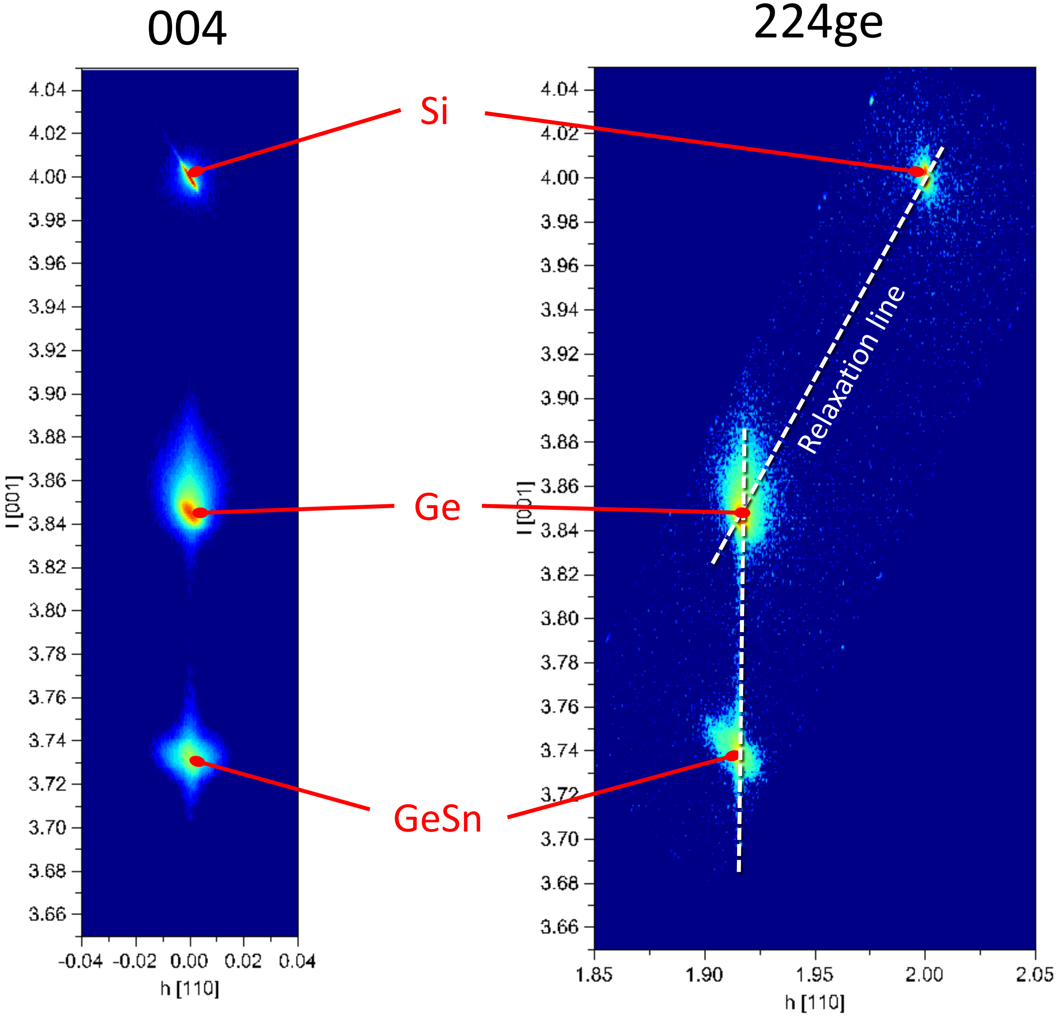




- SiGe peak is shifted away from H=2 in the asymmetric 224ge map indicating relaxation $(a_{SiGe,||} > a_{Si})$
- Peak is broadened due to dislocations, $w(H)/H \propto 1/\sqrt{\rho}$

RSMs from strained thin-films on strain relaxed buffers (SRBs)

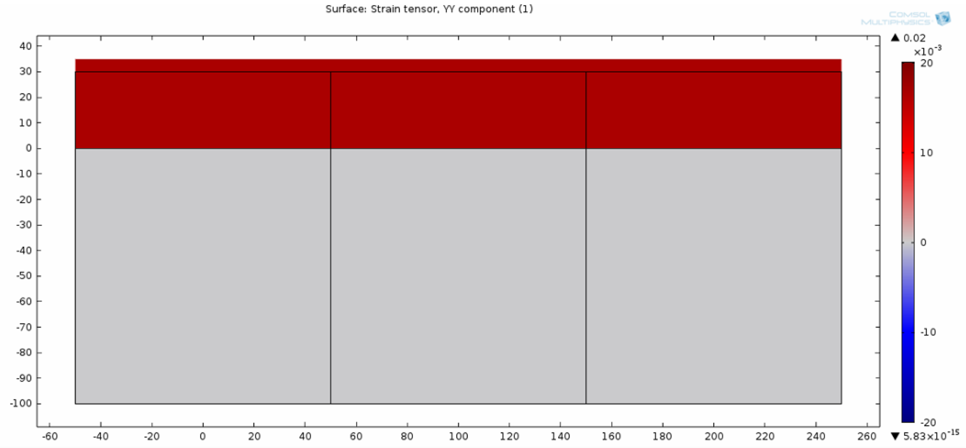
$Ge_{0.89}Sn_{0.11}$ / Ge on Si(001)

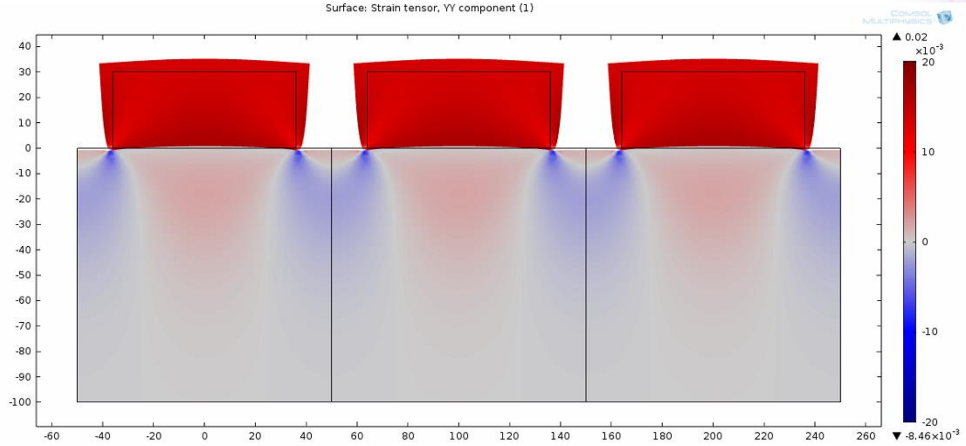


- Ge peak is shifted away from H < 2 in the asymmetric 224ge map indicating relaxation ($a_{Ge,||} > a_{Si}$)
- GeSn peak is not shifted in H wrt to Ge peak ($a_{GeSn,||} = a_{Si}$)
- Composition and relaxation of each layer can be obtained

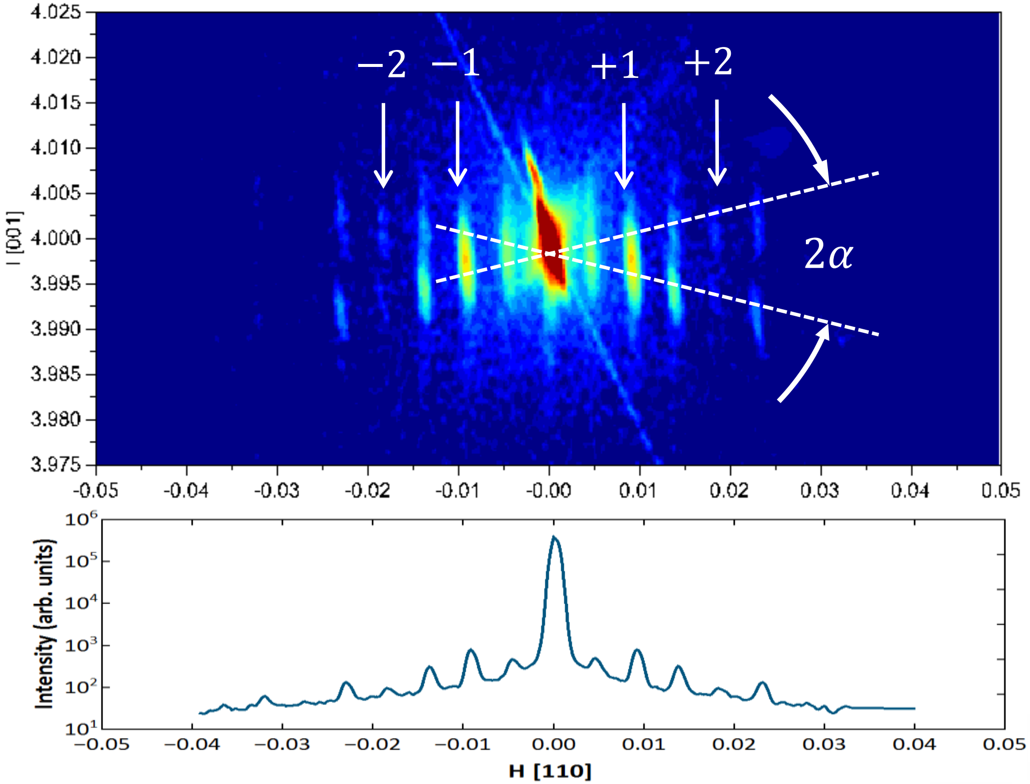
Patterned epitaxial nanostructures

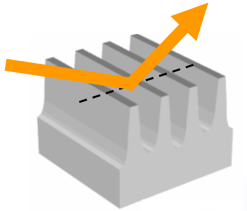
- In blanket epitaxy you have simple biaxial stress
  - Blanket pads are less relevant and / or no longer available
- In epitaxial nanostructures you have
  - Micro-loading effects in selective growth
  - Stress-state is far more complex, *i.e.* elastic relaxation of the epi and distortion of the substrate lattice





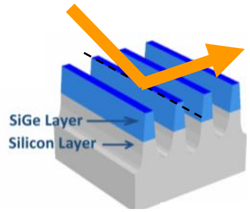
RSM from Si fins made using spacer double patterning (SDP) lithography

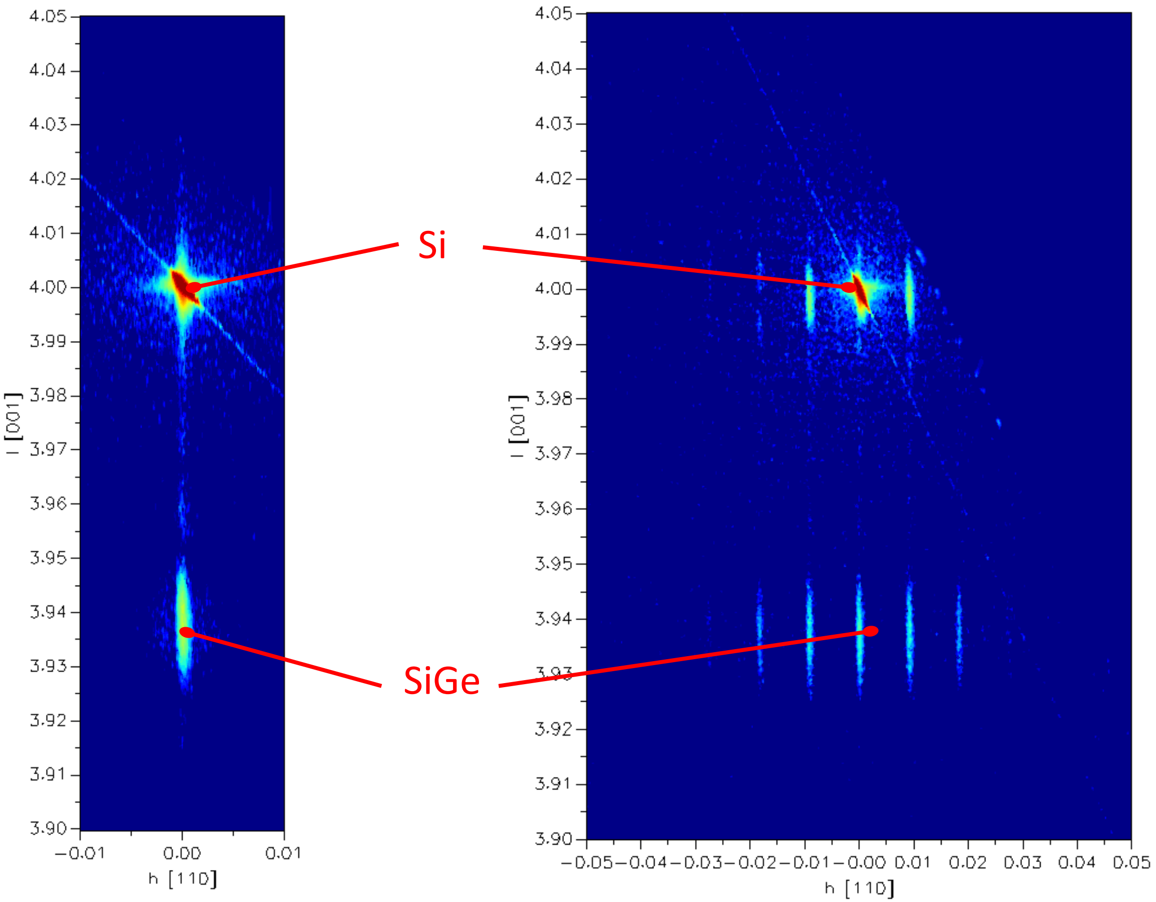


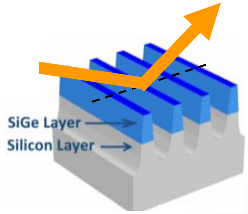


- Fins act as a diffraction grating, $P \propto 1/\Delta H_{GTR} = 42.2 \pm 0.5$ nm
- X-pattern is characteristic of trapezoidal features, $\alpha = 9 \pm 1°$
- Evidence of significant pitch walking error from SDP lithography
  - Strong half-order GTR peaks (corresponds to 2 x pitch), $\Delta P = 5 \pm 0.5$ nm

RSMs from epitaxial SiGe fins
Symmetric 004 reflection

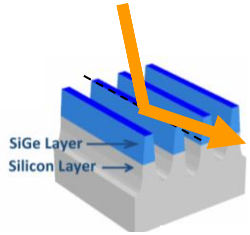


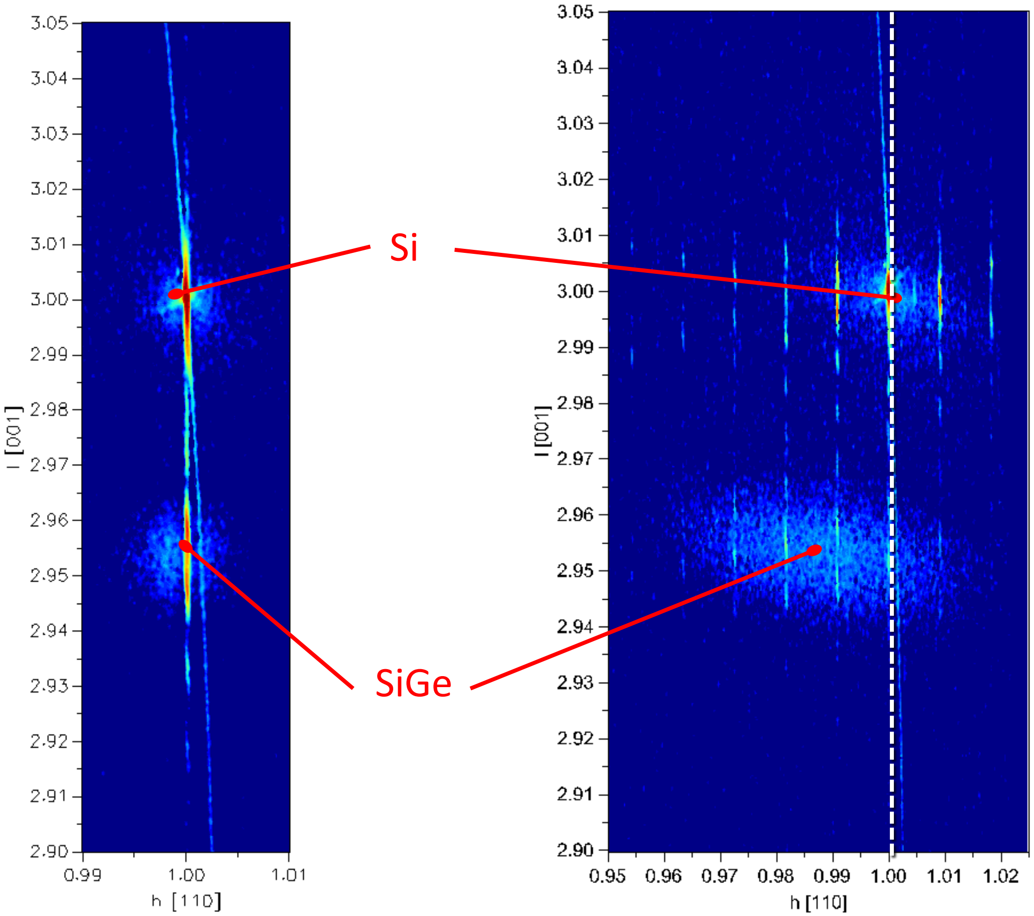


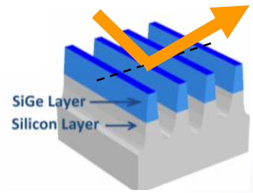


- H-spacing of the GTRs gives the pitch, $P = 42$ nm
- Components of the strain tensor can be determined from intensity envelopes by measuring asymmetric reflection at different azimuths

RSMs from epitaxial SiGe fins
Asymmetric 113ge reflection

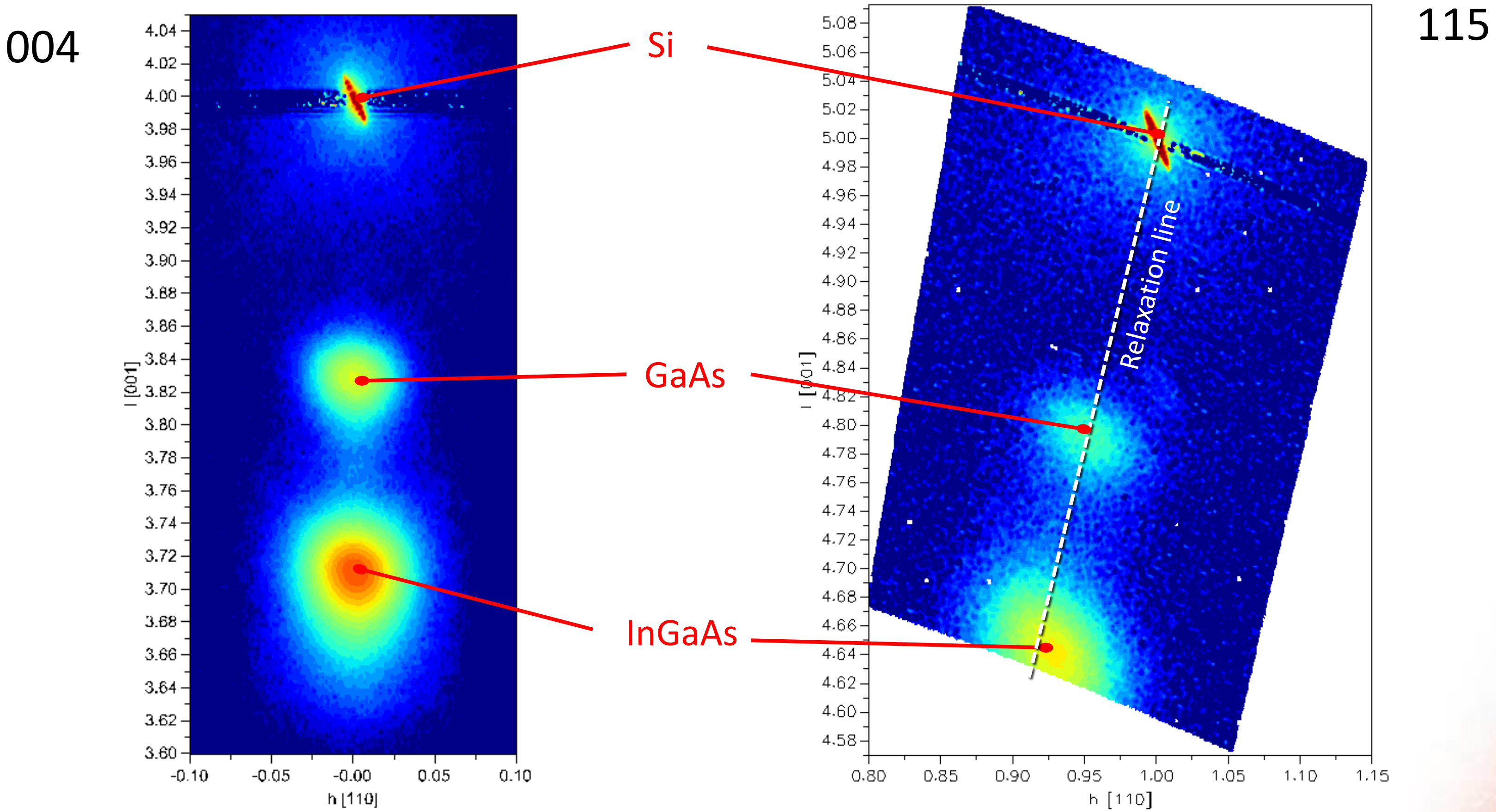






- SiGe in a uniaxial stress state, *cf.* biaxial stress state for thin-films
  - Elastic relaxation perpendicular to the line direction
- Composition and thickness determined from fitting, *x* = 25%, *t* = 39.4 nm

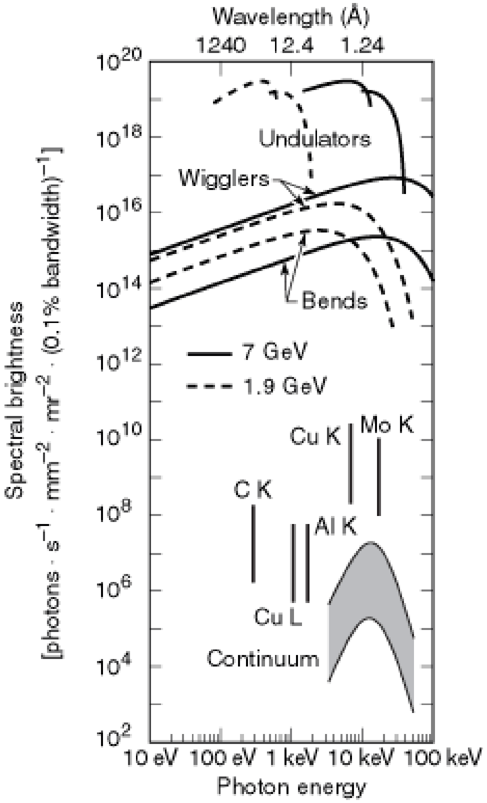
RSMs from III-Vs deposited in narrow (60 nm) trenches etched into thick SiO_2 layer on Si



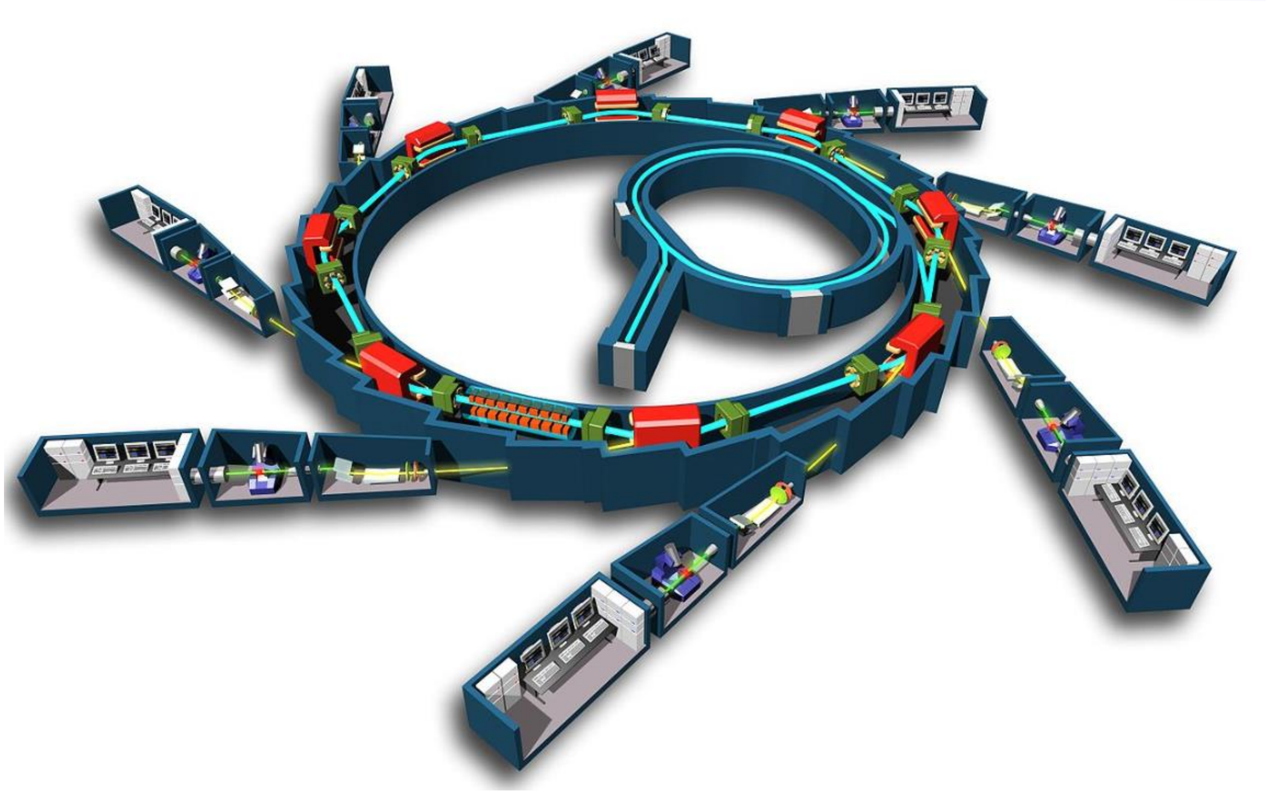
- Interest as a high mobility channel materials in sub-10 nm nodes
- GaAs and InGaAs peaks are very broad due to the high density of threading dislocations, $\rho \sim 10^{10} cm^{-2}$ despite the ART structures

Synchrotron facilities

- Orders of magnitude more brilliant than lab / fab X-ray sources
- Provide advanced measurement capabilities, but very offline

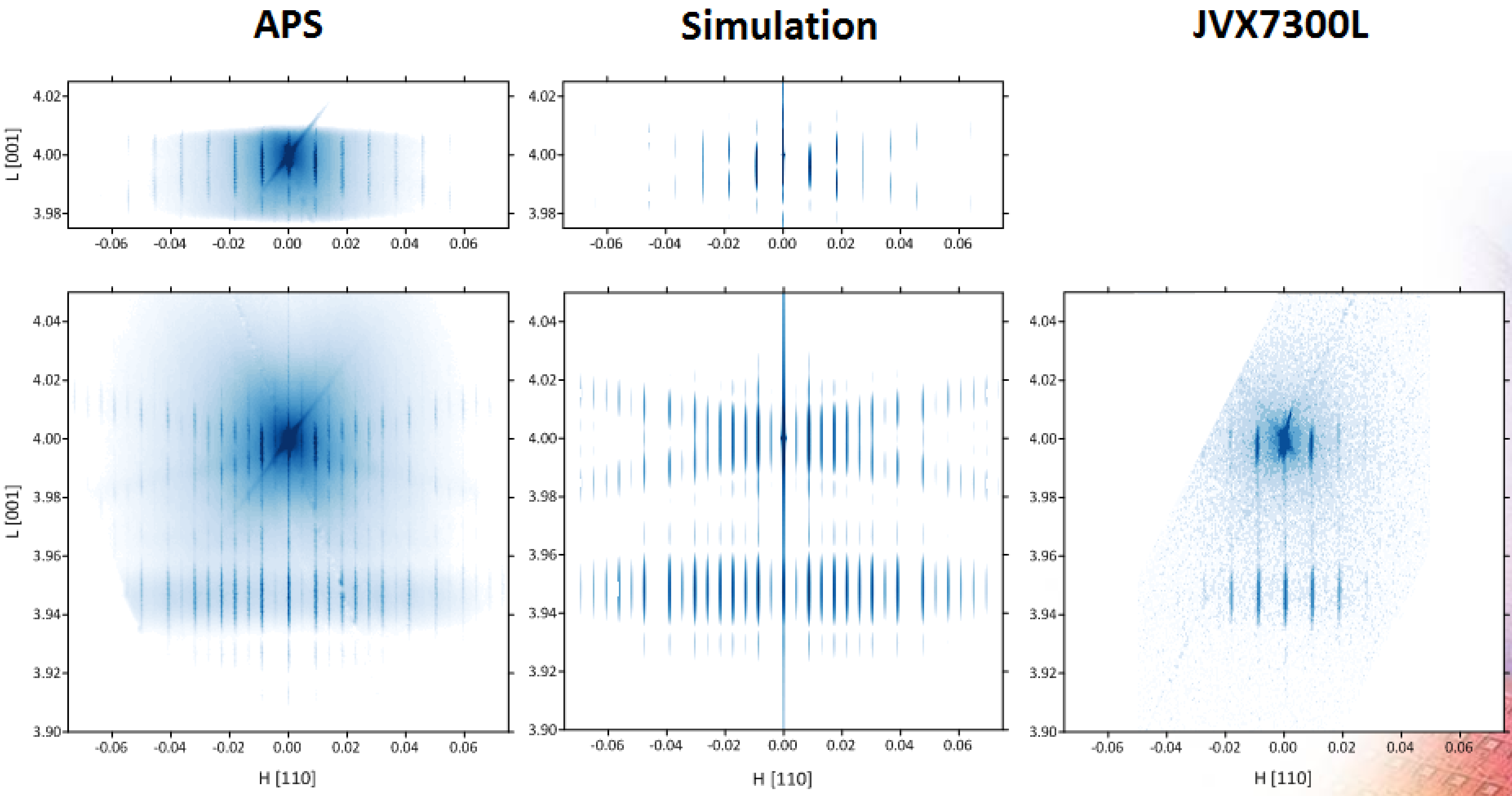


Source: X-ray Data Booklet (http://xdb.lbl.gov/)



Source: EPSIM 3D/JF Santarelli, Synchrotron Soleil, via Wikimedia Commons

Comparison of FastRSMs from SiGe fins measured at the APS and in the fab



- Increased dynamic range is valuable for model development / validation.
- Analysis of single nanostructures is also possible…

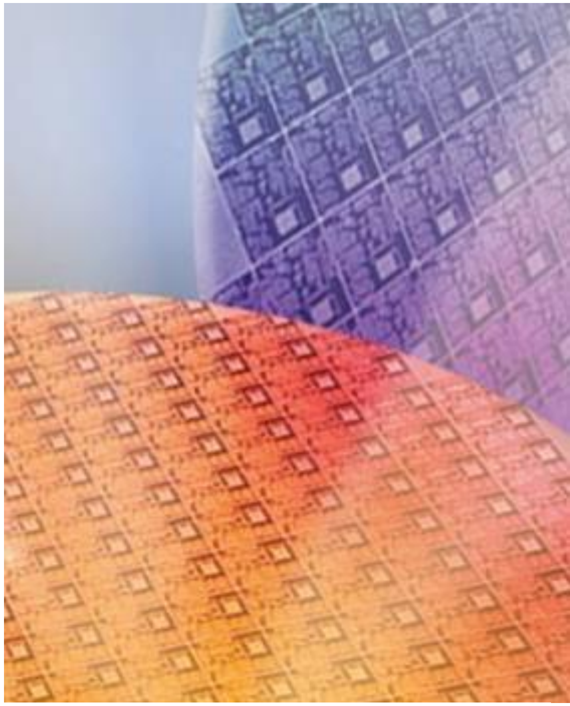
Conclusions

- High-resolution XRD delivers valuable information on epitaxial thin-films and patterned nanostructures
  - Materials include: SiGe, Si:C(P), Ge and III-Vs for current and future technology nodes
  - Parameters include: strain tensor components, composition, thickness, pitch, pitch-walk, height and SWA as well as crystalline quality
- Complements techniques such as SE / scatterometry and SEM / TEM
- The latest generation of lab / fab tools can yield good quality data in minutes not several hours
  - From patterned wafers
  - Including reciprocal space mapping using linear detectors
- In-line X-ray metrology tools, like the JVX7300 series, enable advance materials and process development and provide novel solutions for production monitoring

Thank You!

For more information contact us directly or via a local representative

www.jvsemi.com



Global HQ
Industrial Zone #6
POB 103, Ramat Gavriel
Migdal Ha'Emek 23100
Israel
T: +972 (4) 654-3666

UK Office
Belmont Business Park
Belmont
Durham. DH1 1TW
UK
T: +44 (191) 332-4700

USA Office
3913 Todd Lane
Suite 106
Austin, TX 78744
USA
T: +1 (866) 515-5200

Korea Office
3F Poongsan Bldg. 12-2
Hanil-ro Gongse-dong Giheung-gu
Yongin-si Gyeonggi-do, 446-902
South Korea
T: +82 (31) 275-3690

Taiwan office
13F.-6 (A1), No. 120,
Section 2, Gongdaowu Rd.
Hsinchu City,
Taiwan
T: +886 (3) 573-3180