X-ray Metrology by Diffraction and Reflectivity

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Overview

- High-resolution X-ray Diffraction
  - SiGe box and grade structures (X-ray vs. SIMS)
  - SiGe heterojunction bipolar transistor (HBT)
- X-ray Reflectivity
  - metallic layers
  - dielectric layers
  - gate oxide layers
- Traceability
  - Instrumentation
  - Software
- Reference Standards

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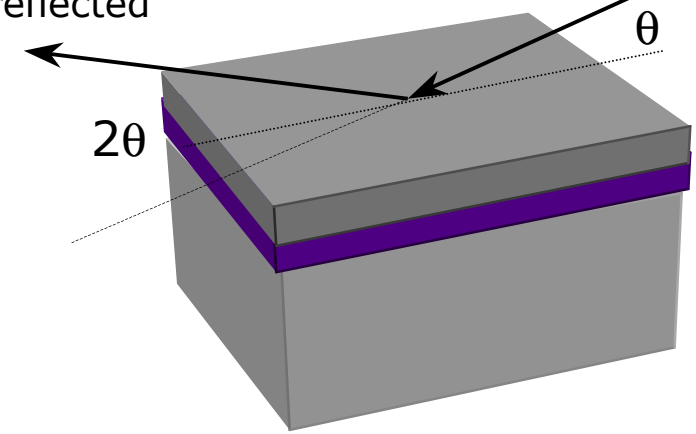
X-ray tools are well-matched to thin fim dimensions and structure

- Convenient sources in range 0.8 < l < 8.0 Å
  - Easy access to layers in range 10 - 20,000 Å
  - wavelengths nearly equal inside and out (<<0.01%)
    - *material-dependent uncertainties are very small*
- Slow but useful Z-dependence of interactions
  - "Goldilocks" values are effective in composition modeling (they are "just right")
- X-rays readily penetrate structures of interest
  - access to thin low-Z layer under thick high-Z stack
- Amplitude, not intensity, addition
  - rich interference phenomena that can be interpreted

Introduction



$2d\sin\theta = \lambda$

Measure the scattered intensity as function of angle, in $\theta/2\theta$ scan, over typically ~2°

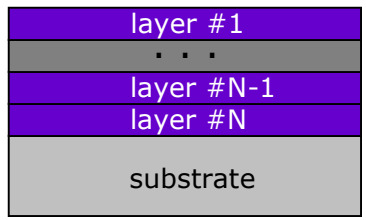
Crystalline sample for diffraction
e.g. epitaxial Si-Ge semiconductor

Crystalline *or* amorphous sample
for reflectivity
e.g. dielectric or metallic layers on Si

Data analysis

Step 1:
Measure scattered X-ray intensity,as function of incident and scattered angles

Model



Step 2:
Create model to describe sample structure
Use model to simulate diffraction from theory

Experimental data

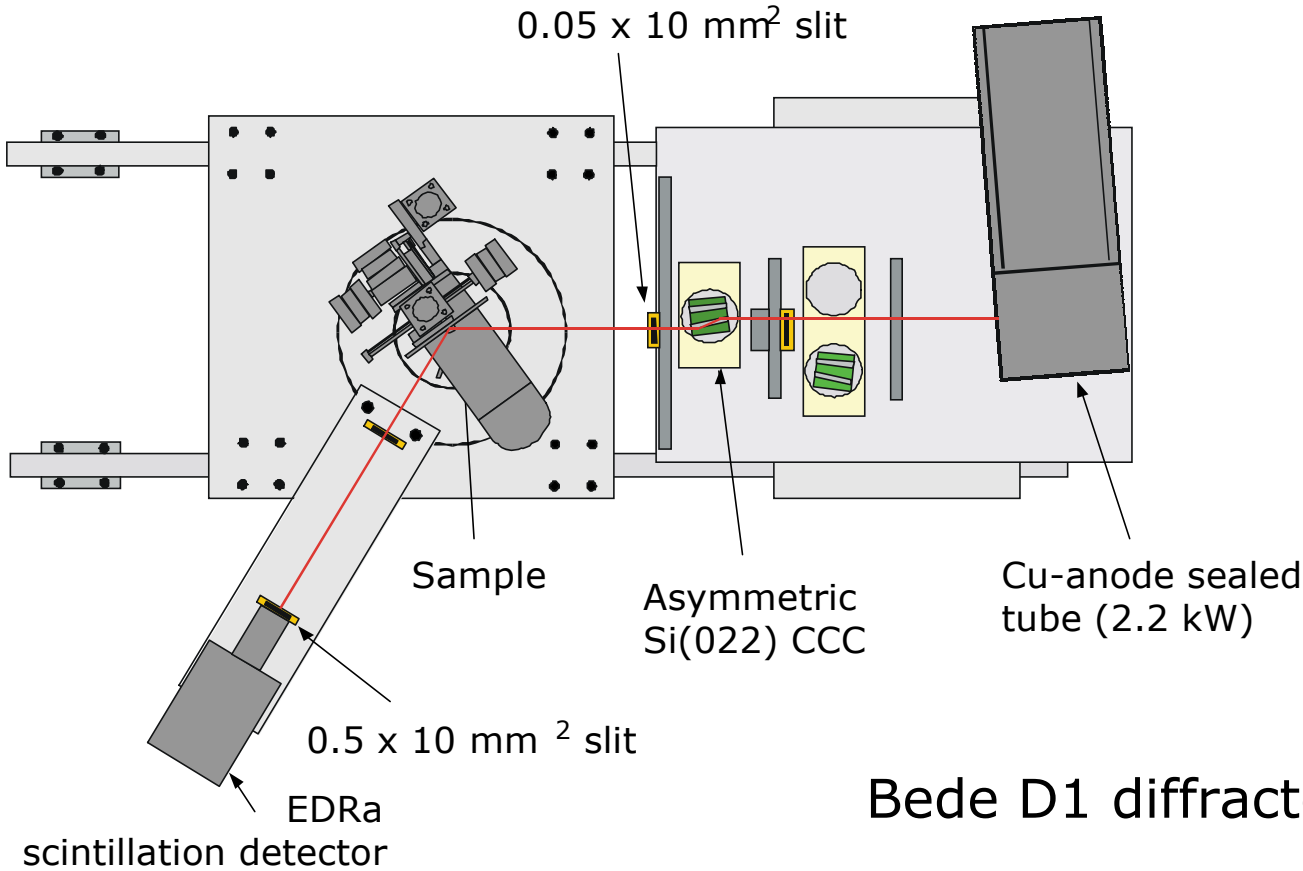
Step 3:
Compare simulated and measured data, refine model to give best agreement

Simulated data

Genetic Algorithms now largely automate this process, and make it objective

Data-fitting

Experimental

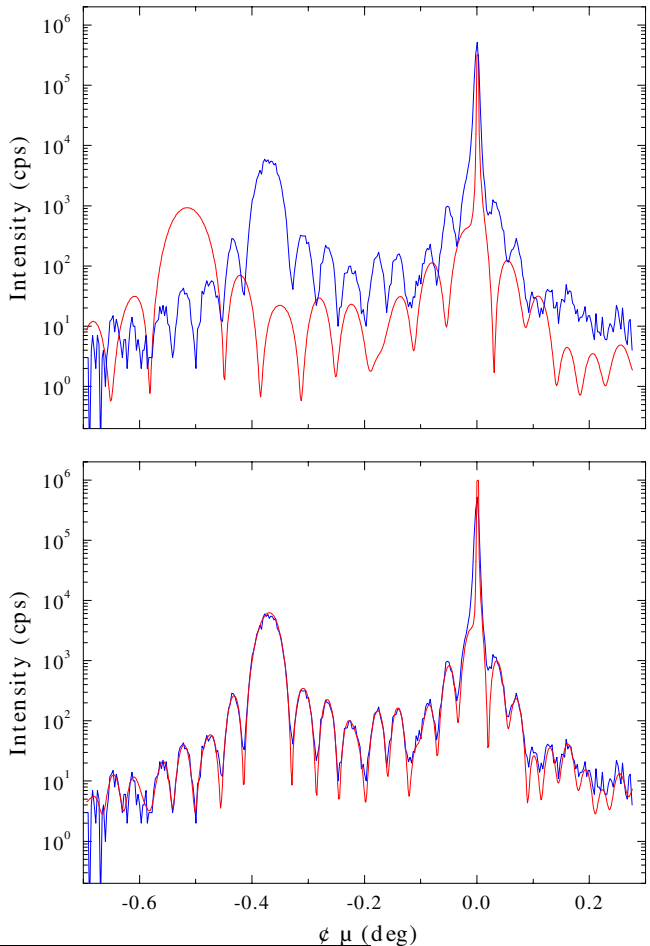


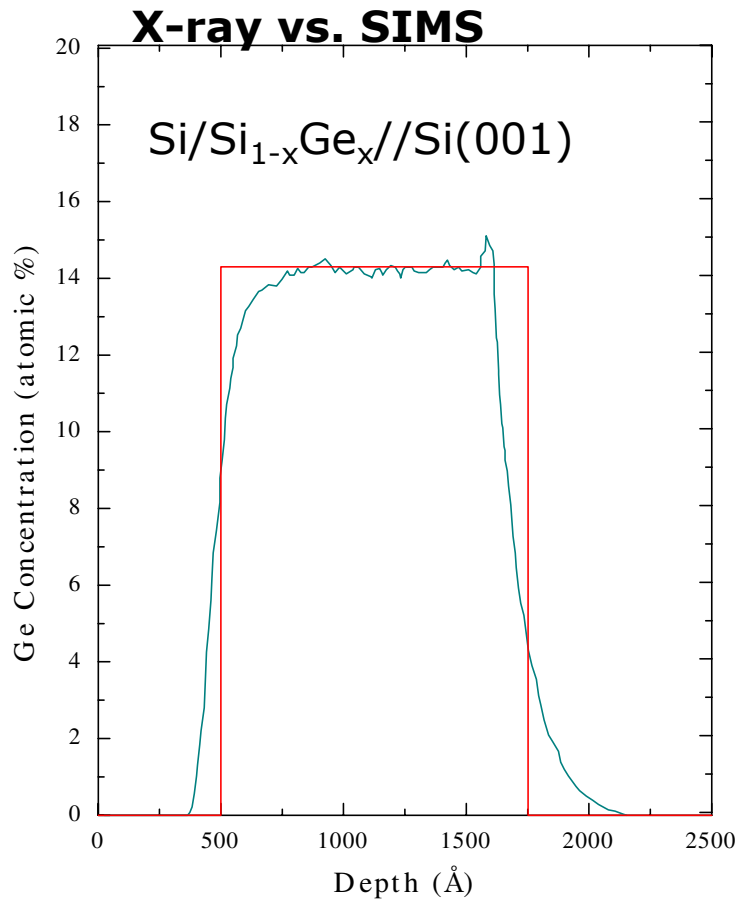
Bede D1 diffractometer

HRXRD 1: SiGe structure (box)

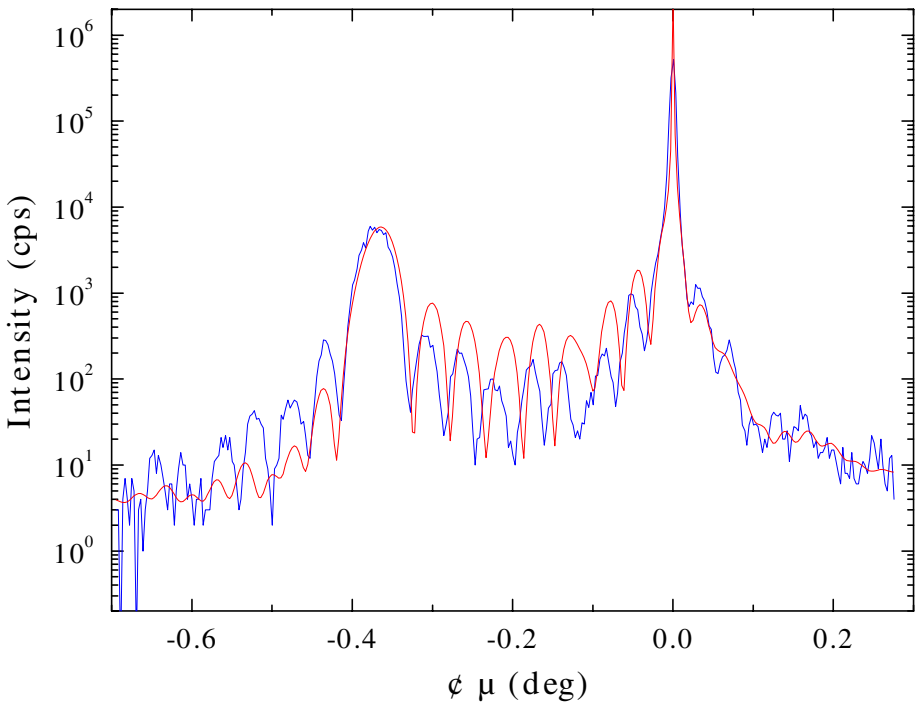
Starting model

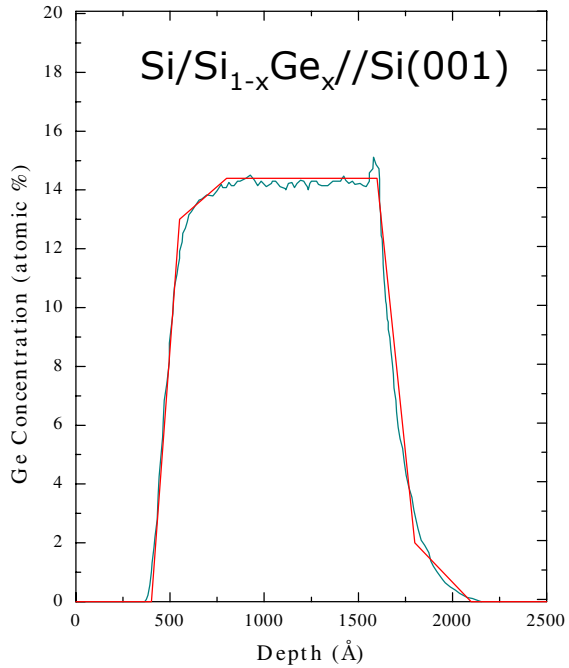
autofit





HRXRD 1: SiGe structure (box)



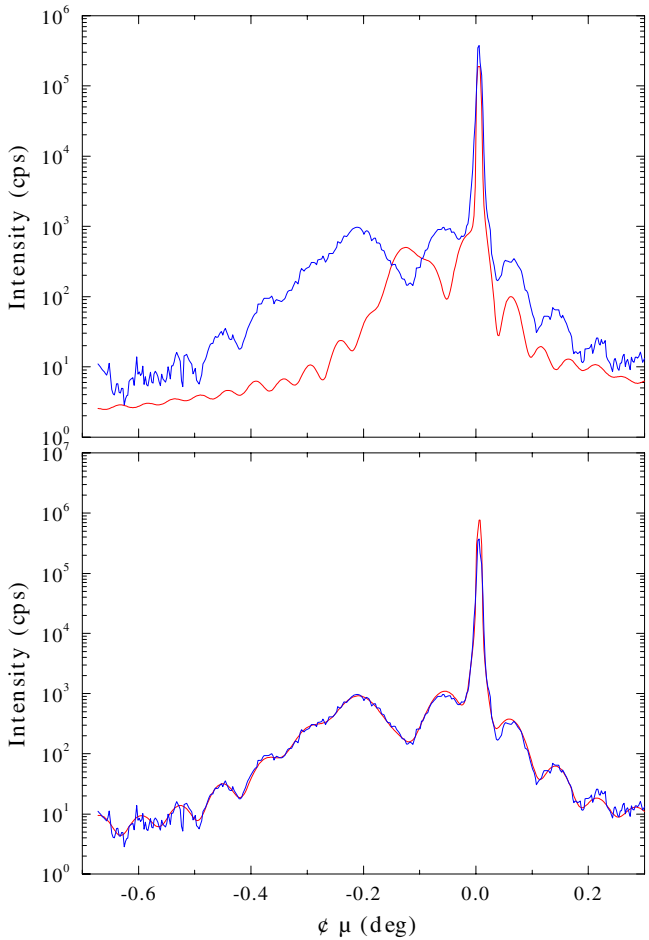


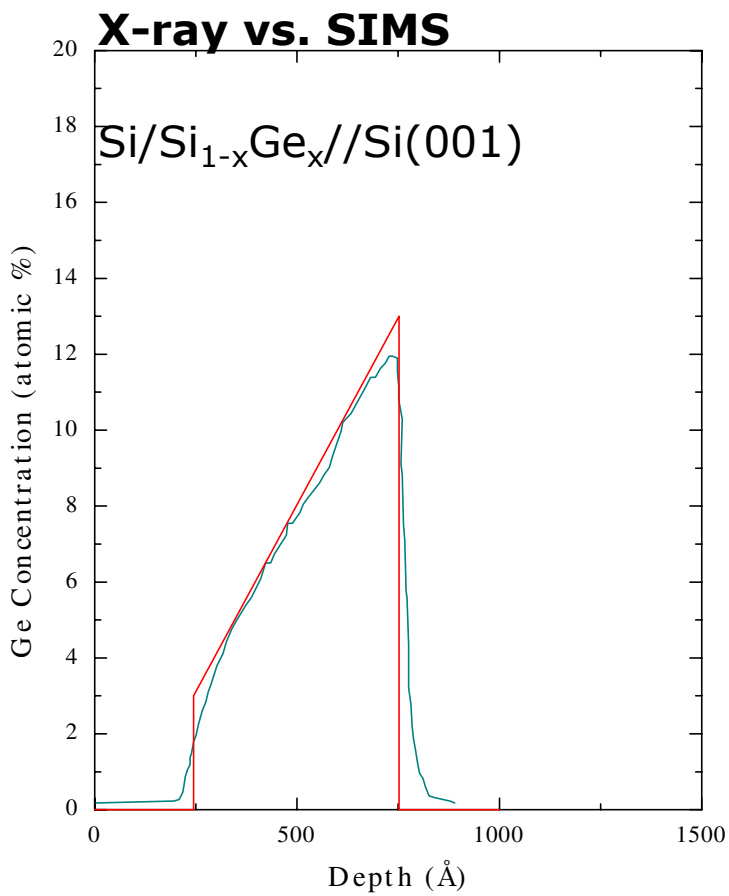
If we model the XRD from the SIMS profile it is clearly wrong. Rounded SiGe/Si interfaces and some gradients are a known SIMS artifact

HRXRD 2: SiGe structure (grade)

Starting model

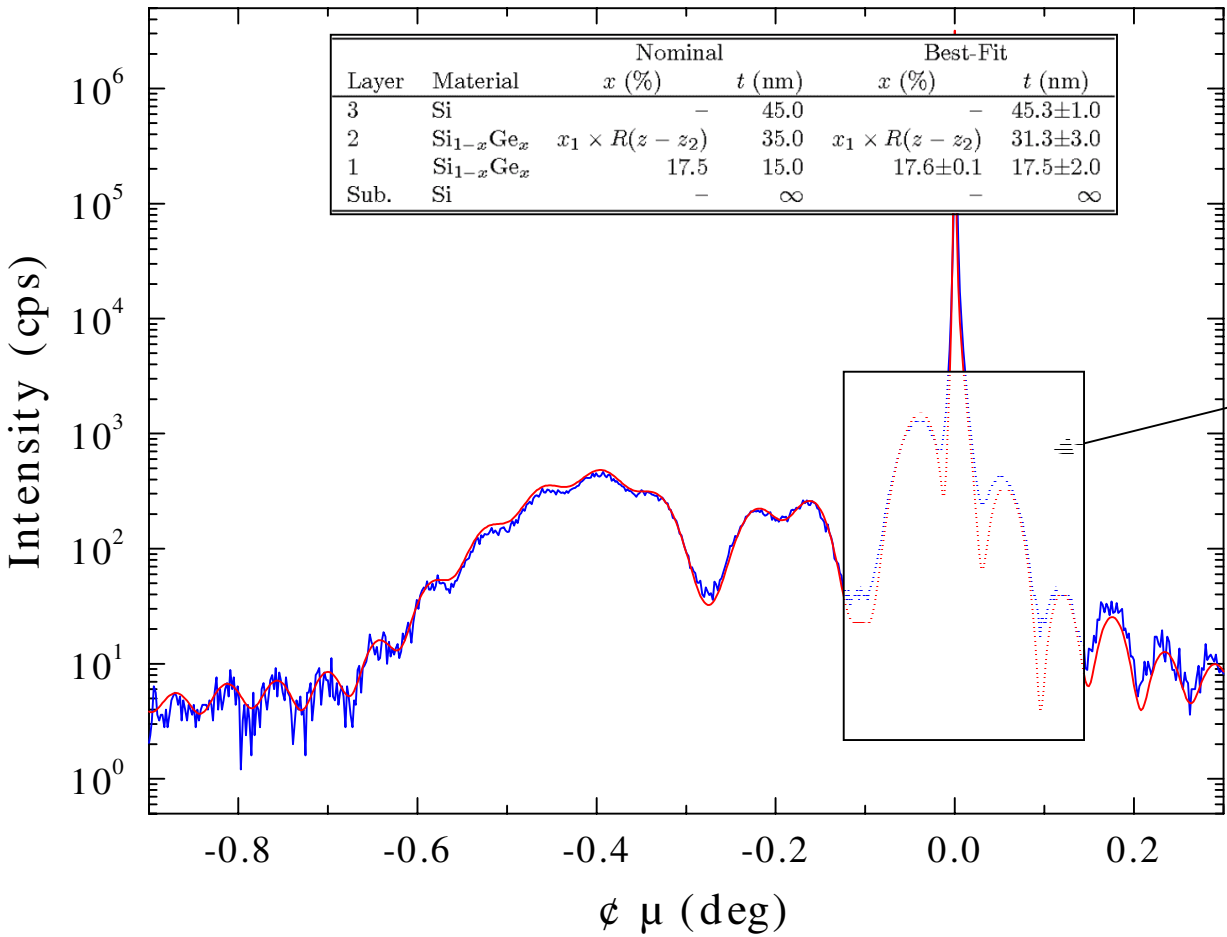
autofit





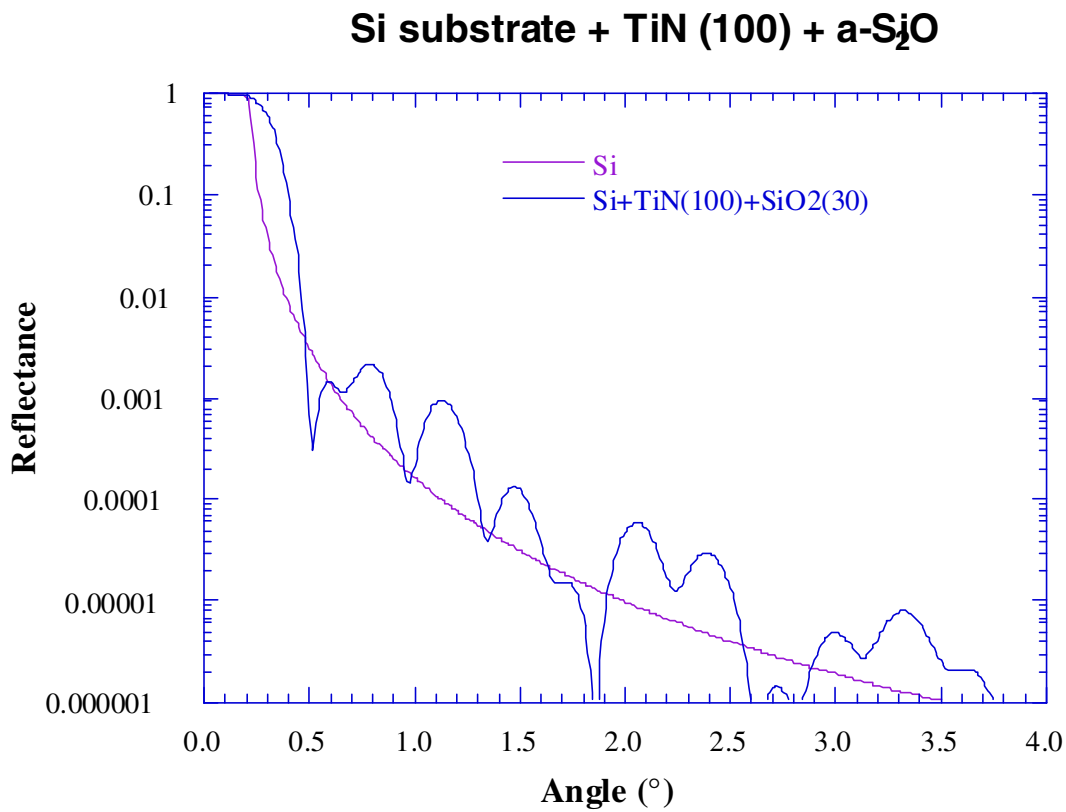
HRXRD 3: SiGe HBT

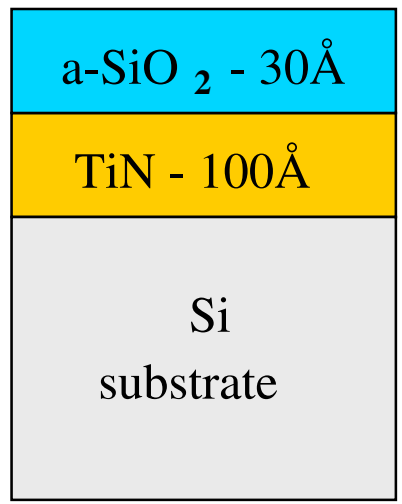
		Nominal		Best-Fit	
Layer	Material	x (%)	t (nm)	x (%)	t (nm)
3	Si	–	45.0	–	45.3±1.0
2	$Si_{1-x}Ge_x$	$x_1 \times R(z-z_2)$	35.0	$x_1 \times R(z-z_2)$	31.3±3.0
1	$Si_{1-x}Ge_x$	17.5	15.0	17.6±0.1	17.5±2.0
Sub.	Si	–	∞	–	∞



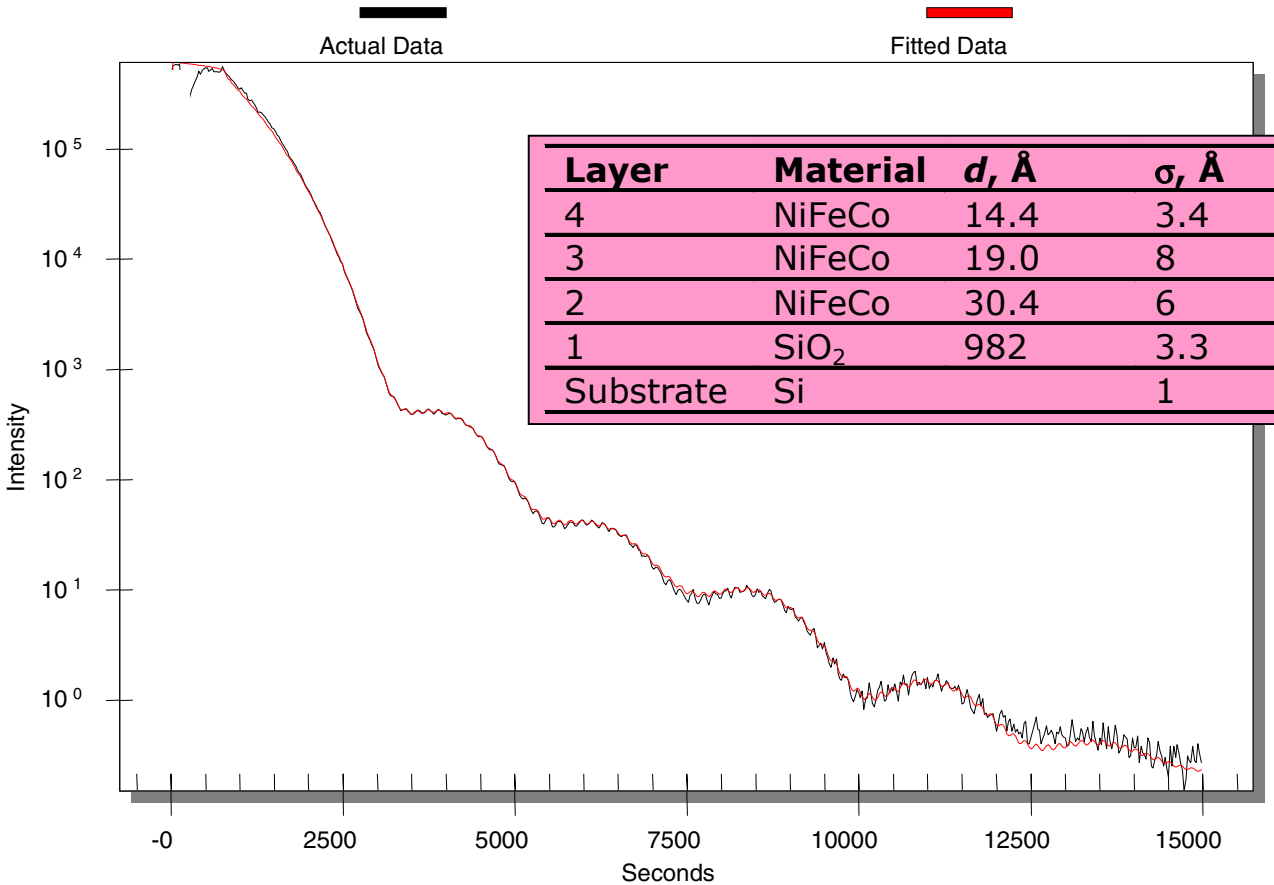
Differences in this region are diffuse scatter from defects

XRR 1: Interference from multilayers





XRR 2: metal films



Layer	Material	*d*, Å	σ, Å	ρ/ρ$_0$
4	NiFeCo	14.4	3.4	0.21
3	NiFeCo	19.0	8	0.82
2	NiFeCo	30.4	6	1.06
1	SiO_2	982	3.3	0.99
Substrate	Si		1	1

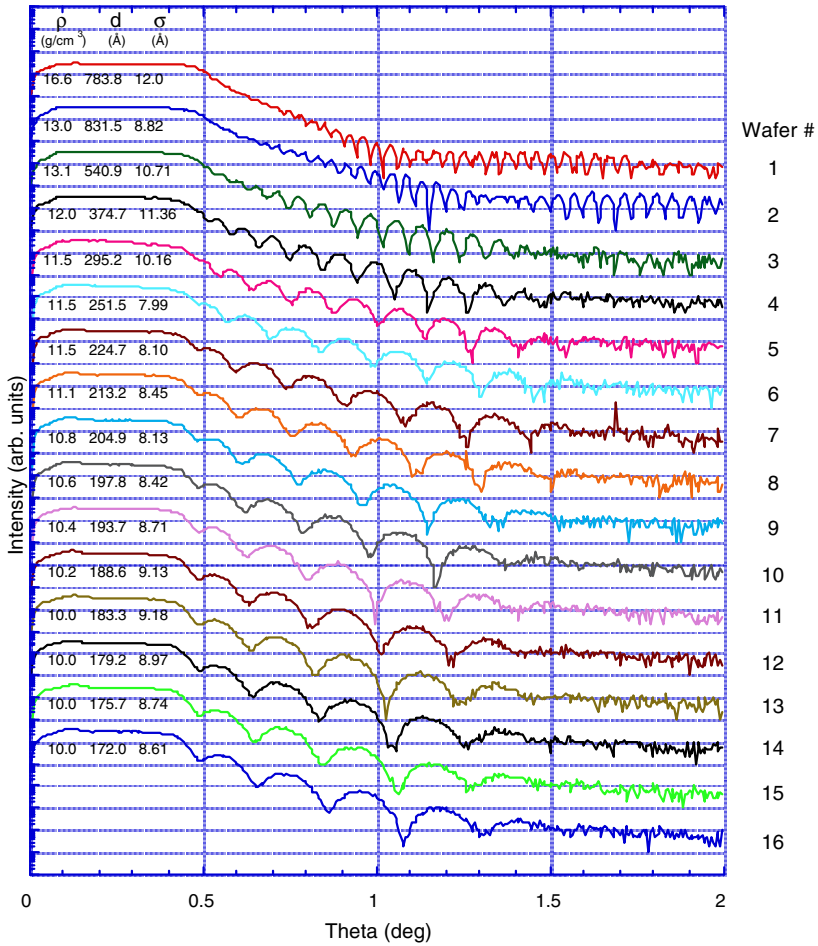
Oxide

low density

high density

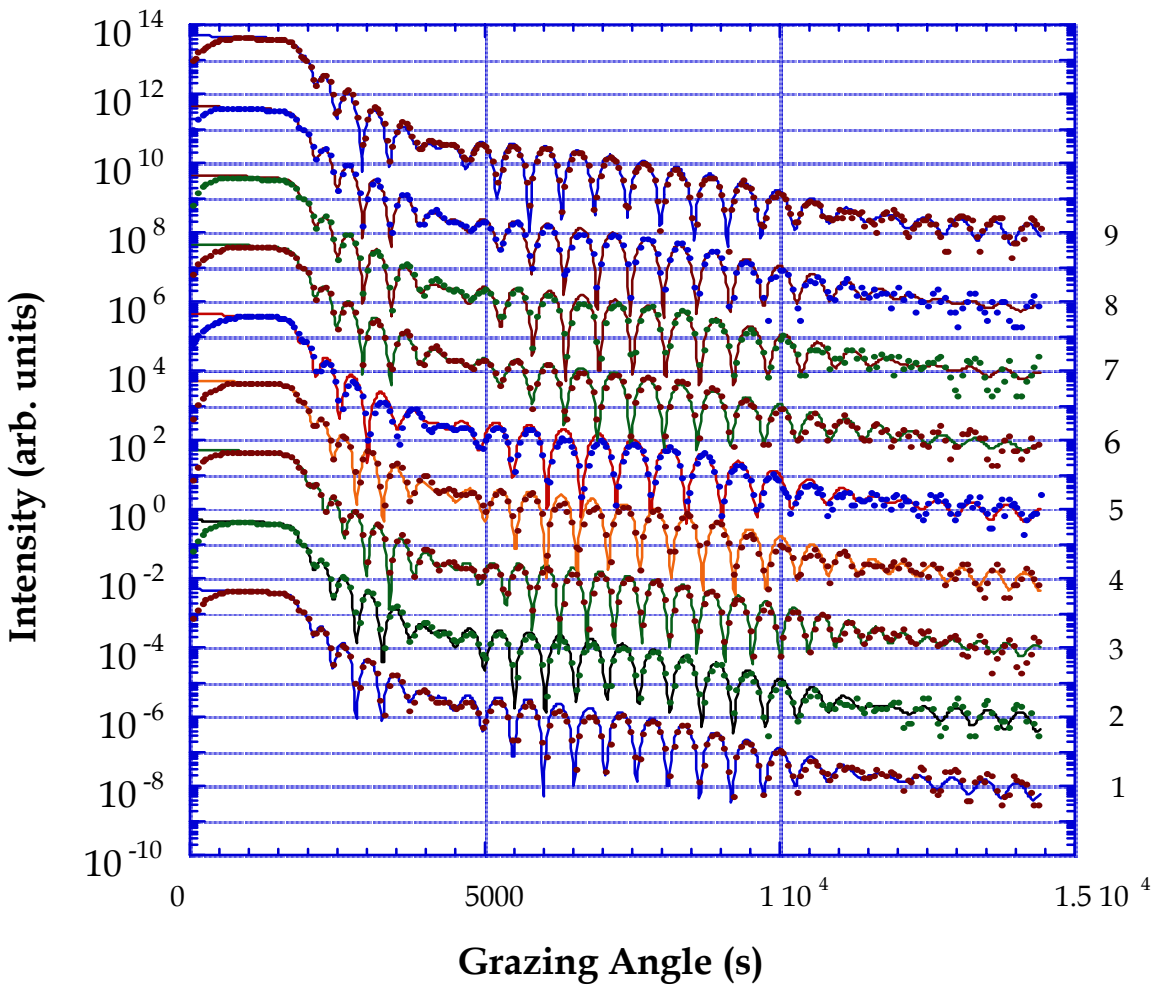
Good signal/noise is essential for correct interpretation

Tantalum Nitride (Cu diffusion barrier)



- Initial Sematech-16 (1998)
  - Expected density
    - Near metallic
  - Expected conductivity
    - Near metallic
  - Failed functional tests
    - Resistivity too high
- NIST study showed
  - Densities too low
  - Densities highly variable
  - Inhomogeneous structures
- Other structural data
  - Columnar growth
  - Leading to high resistivity

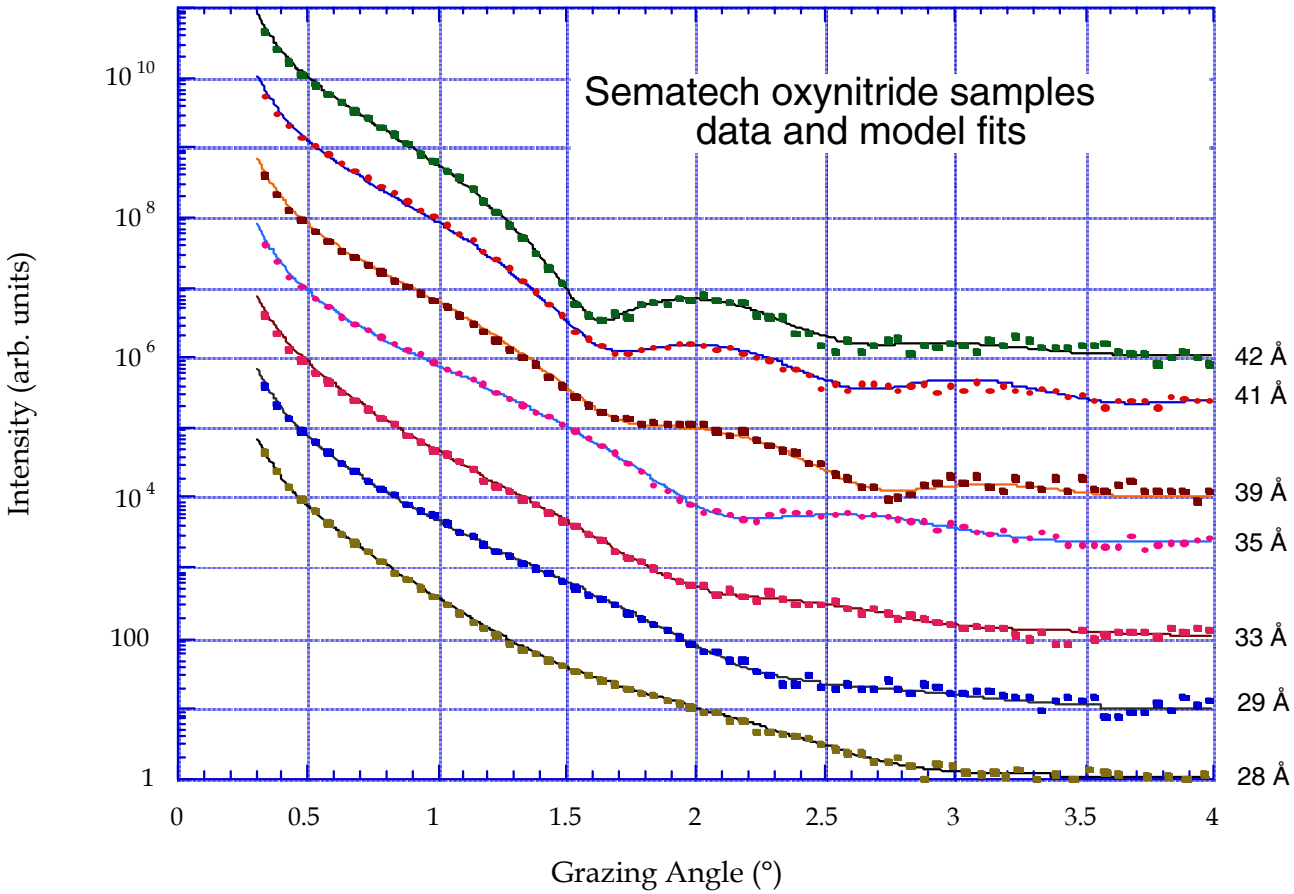
Subsequent improvement



Good uniformity

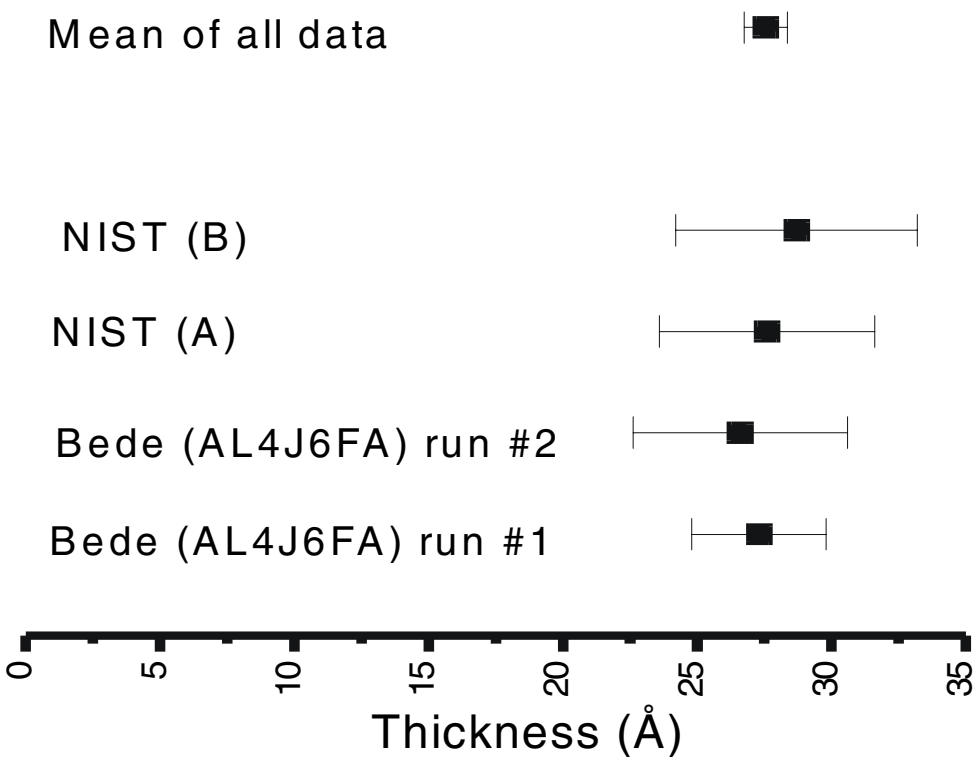
phase change part way through growth shown by beating of fringes

XRR 3: thin oxides & oxynitrides



Good matching over range

NIST round robin



Oxynitride sample

from Round Robin organised by James Ehrstein, NIST

Common specimen treatment

NIST data by J. Pedulla and R.D. Deslattes

Bede data by M. Wormington

Traceability

… link to the base unit of length in the International System of Units (the SI) by an unbroken measurement chain **that does not degrade the indicated reproducibility**.

The only stable measurement systems are those that are accurate

- Instrument calibration
- Measurement procedures
- Software certification
- Interpretation procedures

Instrument calibration

- Traceability depends on
  - wavelength
    - Cu Kα emission wavelength - already NIST-traceable
  - angle
    - self-referential to 2π
    - Calibration and angle standards available in principle
    - Could be more convenient and accessible
  - Intensity
    - Little difficulty if counting used below saturation
  - correct procedure
    - NIST specification is desirable

There is more hope of making HRXRD and XRR fully traceable than there is for most analytical techniques!

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Software traceability

- Some parameters are easily deduced from fundamentals
  - composition of isolated layer
  - thickness of isolated layer
- Others need software
  - graded composition layers
  - thickness of multiple layers
- Are all software vendors' products equivalent?
  - Relatively few studies*, but clearly **NO**!
- Rôle for NIST in certifying analytical software
  - defined limits of application

**M. Grundmann and A. Krost, "Atomic structure based simulation of X-ray scattering from mesoscopic structures", sub. Phys. Rev. Lett. 1998*

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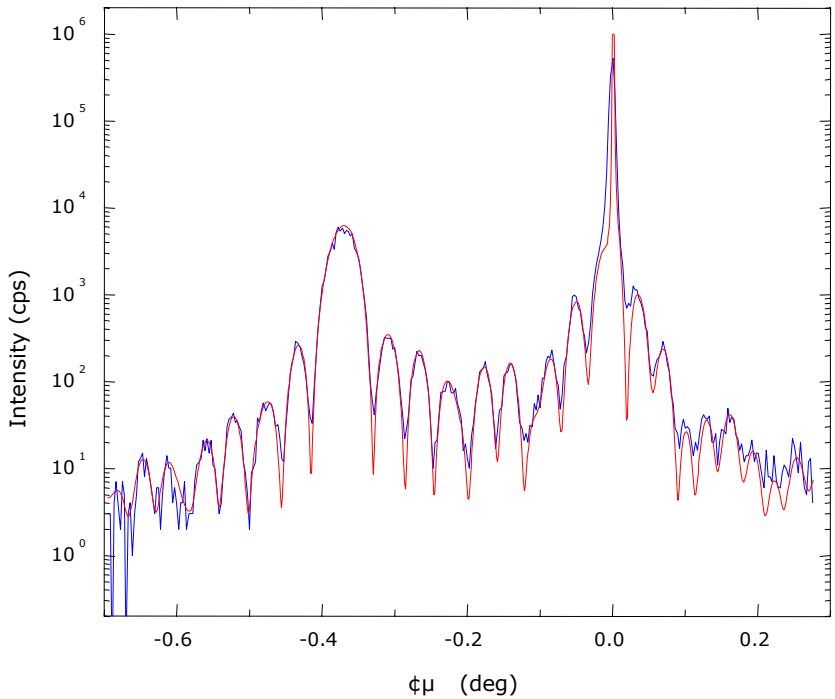
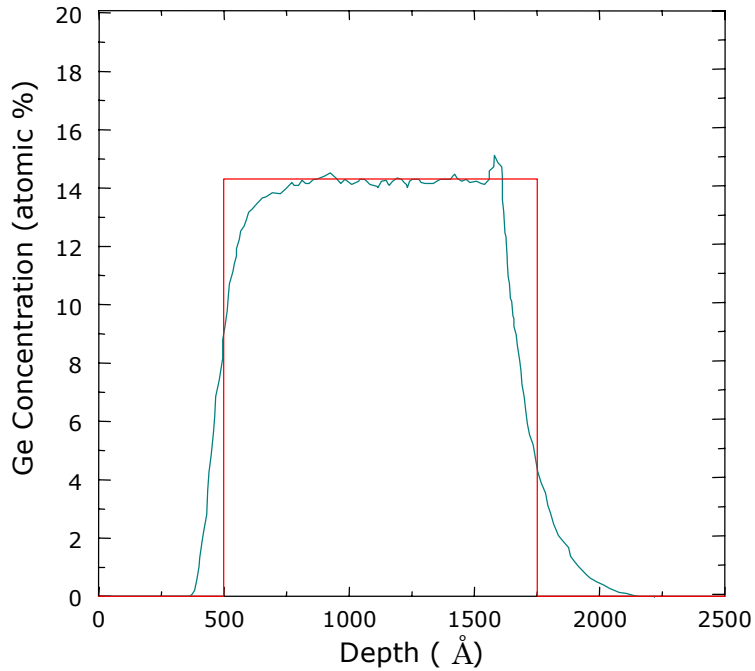
Reference standards

- Ideal standard should be
  - calibrated with traceability to NIST
  - stable over time, or have specified lifetime
  - calibrate over actual instrument ranges
  - measured in the same way that wafers are measured
  - provide quantitative assessment
- Absolute composition standard - HRXRD
- Secondary angular standard - HRXRD
- Secondary angular standard - XRR

Proposed absolute Si-Ge composition/thickness standard



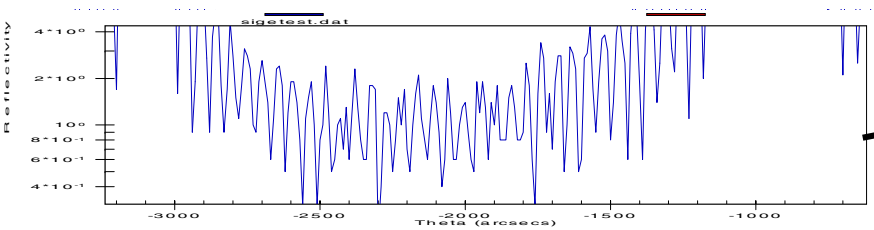


Verification of standard

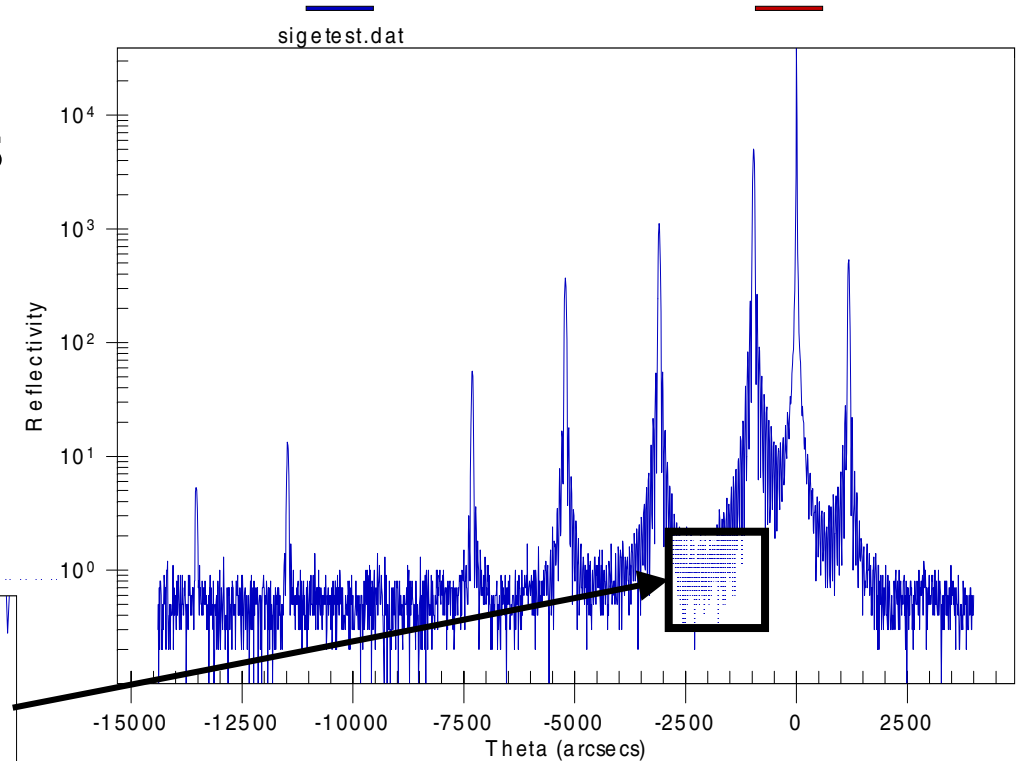
- HRXRD gives
  - Si-Ge composition from peak angles
  - thickness from fringes
- Need to know
  - wavelength (and **only** this for thickness)
  - lattice parameters and elastic constants as *f*(composition)
  - theory: Bragg law and interference equation
- Cross check: XRF
  - calibrated with pure element standards
  - gives composition*thickness product (mass)

Secondary calibration for HRXRD

- No need for absolute composition/thickness
- Calibrate coarse and fine angles
- Superlattice

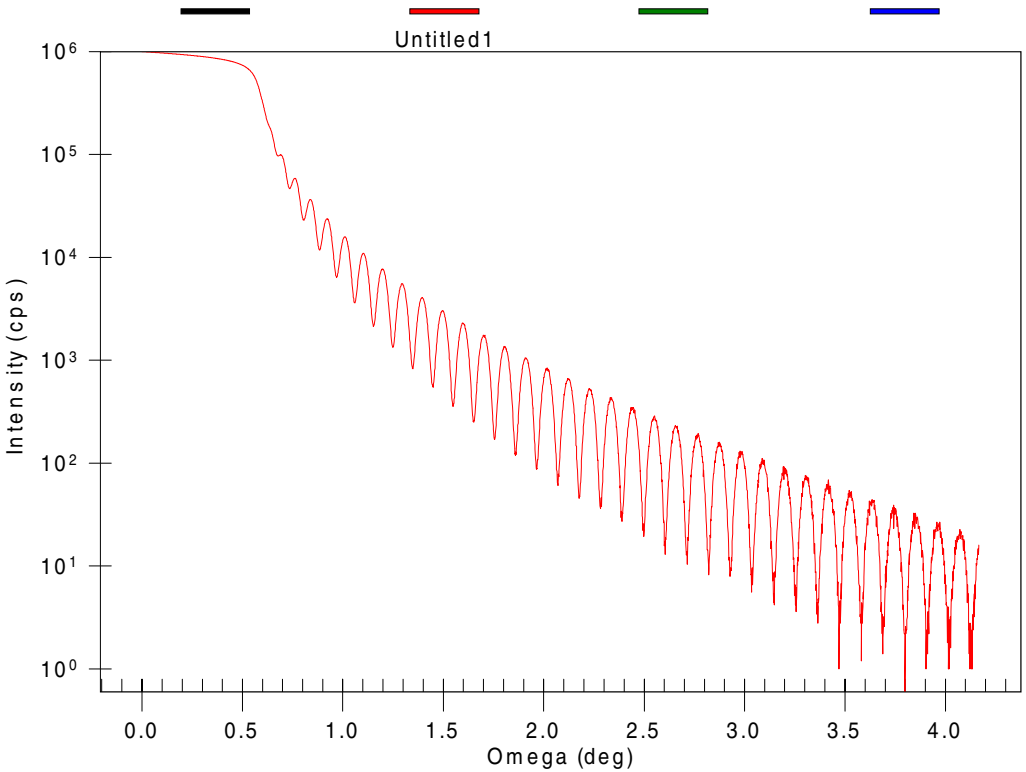


50 arcsec fringe spacing



(60 Å Si + 30 Å $Si_{0.7}Ge_{0.3}$)x 40

Secondary calibration for XRR (1)

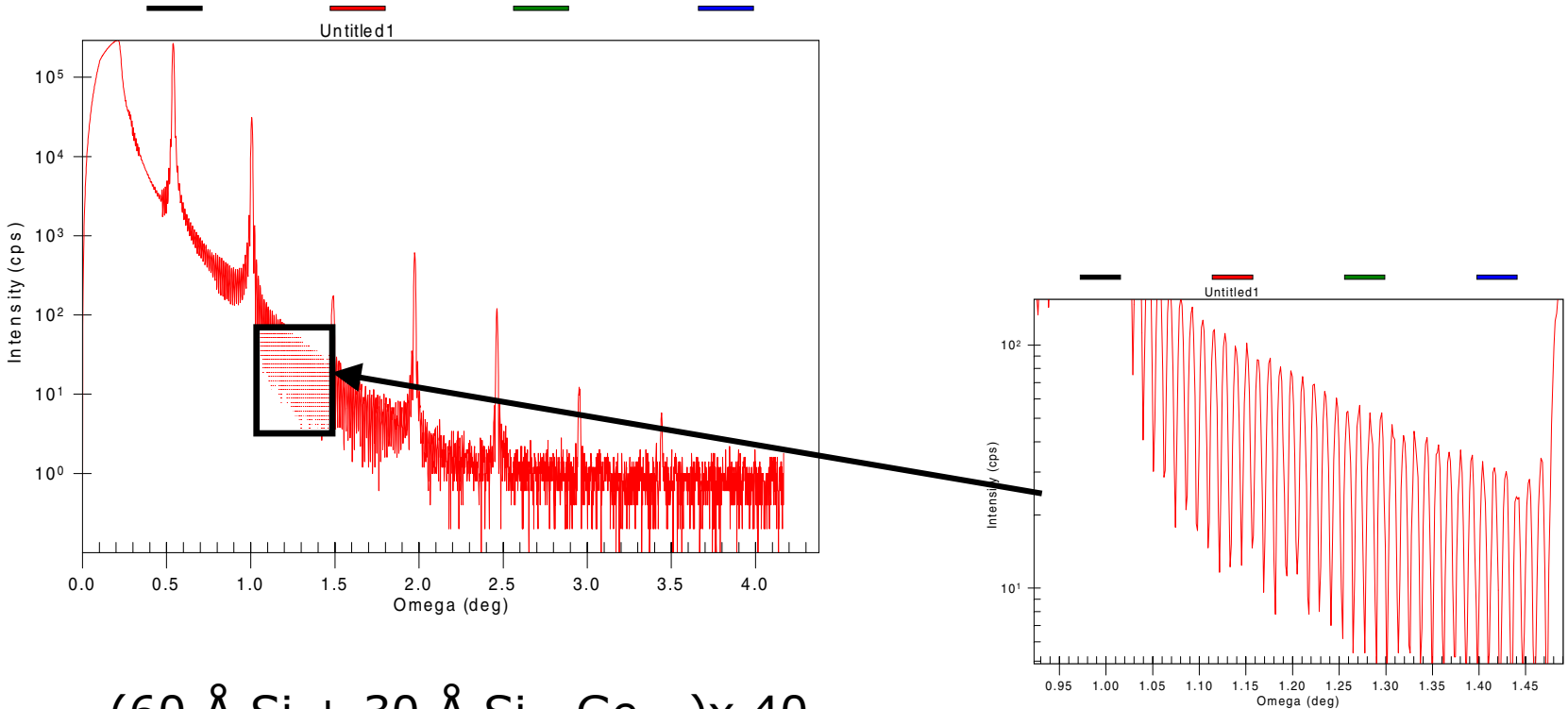


400 Å Pt on Si

Long-term stability checked at NIST over >5 years

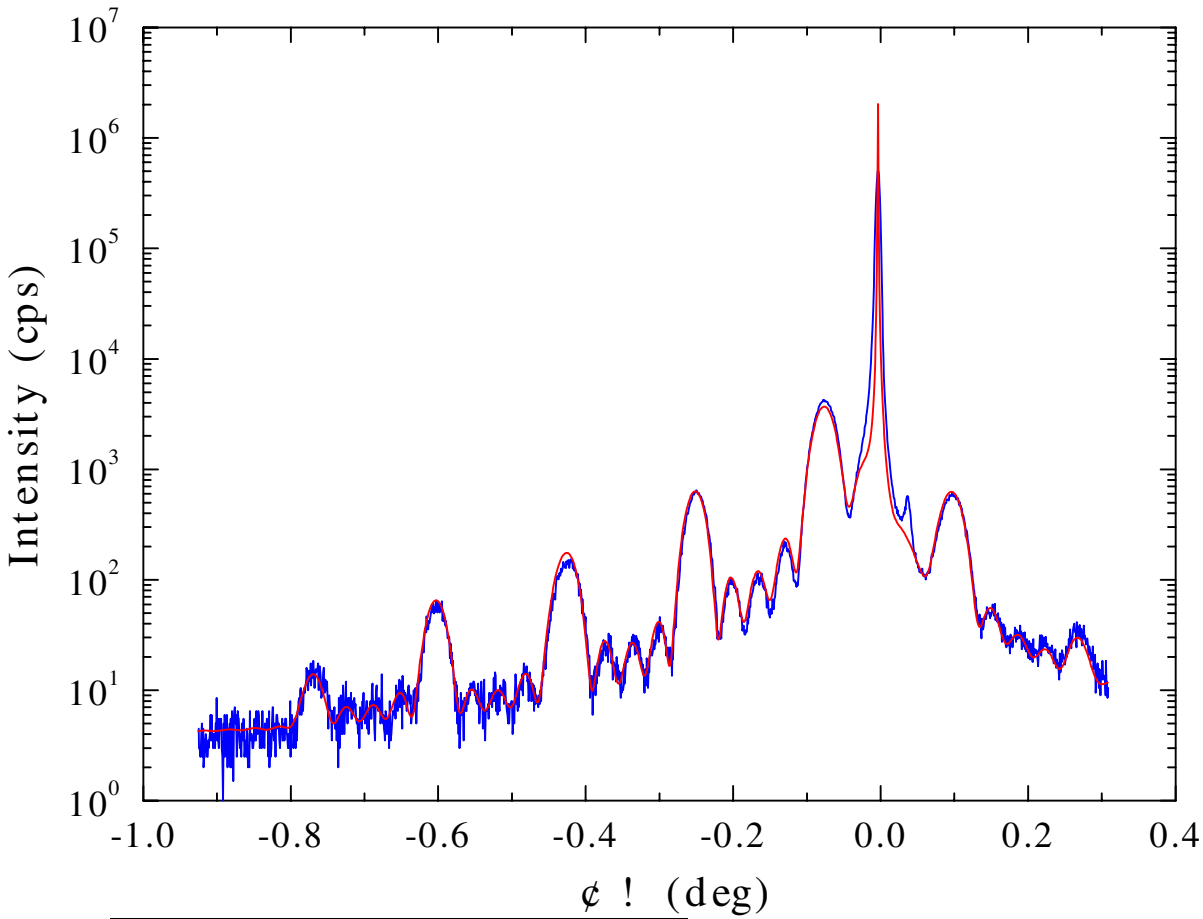
Period ~400 arcsec

Secondary calibration for XRR(2)



(60 Å Si + 30 Å $Si_{0.7}Ge_{0.3}$)x 40

Stability of Si-Ge Superlattice



5-period SL

Period:

1991: 308 ± 3 Å

(A.R. Powell)

1999: 306 ± 2 Å

(M. Wormington; see figure for data and simulation)

Different Bede instruments used in 1991 and 1999

Summary

- X-ray metrology is up and running in the semiconductor industry
- Both HRXRD and XRR are traceable techniques, which give unique information on epitaxial, polycrystalline and amorphous layers
- Traceability is excellent in principle but at present is not convenient in practice.
- NIST standards are very desirable in
  - software verification
  - procedure approval
  - secondary reference standards
- In-line fab tools are available, which do not require expert operators