2005 International Conference on Characterization and Metrology for ULSI Technology

Recent Advances in Semiconductor X-ray Metrology

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X-ray Metrology - Volume Speaks for Itself

6-Invited Talks

- The Opportunities and Challenges of Bringing New Metrology Equipment to Market
- Metrology (Including Materials Characterization) for Nanoelectronics
- Metrology Challenges for 45 nm Strained-Si Devices
- Recent Advances in Semiconductor X-ray Metrology
- X-ray Photoelectron Spectroscopy of High-k Dielectrics
- Small Angle X-ray Scattering Metrology for Sidewall Angle and Cross Section of Nanometer Scale Line Gratings

About 20 Posters

- TU-02: In-line Compositional and Thickness Metrology Using XPS for Ultra-thin Dielectric Films
- TU-03: A New NIST Database for the Simulation of Electron Spectra … : Application to Angle-Resolved XPS ….
- TU-05: The Use of Model Data to Characterise Depth Profile Generation from Angle Resolved XPS
- TU-06: Dopant Dose Metrology for Ultra-Shallow Implanted Wafers Using Electron-Induced X-ray Spectrometry….
- TU-07: Depth Resolved Composition and Chemistry of Ultra-thin Films by ARXPS
- TU-13: Simultaneous Analysis of Thickness and Composition of Ultra-thin Films and Multilayers Using XRF
- TU-14: On-product Thin Film Characterization Using XRF
- TU-28: Characterization of Atomic Layer Deposition Using XRR
- TU-29: Limits of Optical and X-ray Metrology Applied to Thin Gate Dielectrics
- WE-07: Practical Applications of XRR-XRF Metrology Tool
- WE-09: Combined XRR and Rs Measurements of Cobalt and Nickel Silicide Films
- WE-17: Calculation of Pore Size Distributions in Low-k Films
- WE-20: Optical and X-ray Metrology of Low-k Materials: Porosity Evaluation
- WE-21: X-ray Porosimetry as a Recommended Metrology to Characterize the Pore Structure of Low-k Dielectric Films
- TH-16: Quantitative Analysis by Low Energy X-ray Emission Spectroscopy (LEXES) of Metallic and Dielectric Thin Films
- TH-20: Accuracy and Repeatability of X-ray Metrology
- TH-23: In-line Monitoring of Fab Processing Using X-ray Diffraction
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What's out there?

X-ray Methods for Film Measurements

XRR
- Thickness (First Principle Method)
- Density / Porosity
- Surface and Interface Roughness
- Single or Multi-layers

XRF
- Thickness (calibrated)
- Single or Multi-layers for non-repeating materials
- Composition

XRD
- Phase
- Texture
- Strain/Stress (Hi-res. XRD)
- Thickness (rocking curves)

SAXS
- Low-k Pore Size and its Distribution

XPS
Ultra-thin films < **3nm**
- Thickness
- Composition

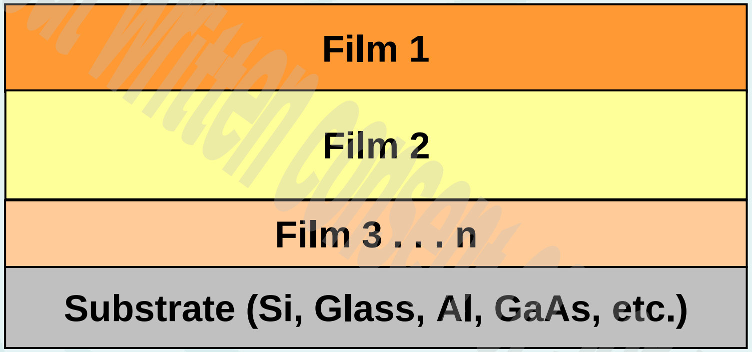
e-XRF
- Thickness (calibrated)
- Composition
- Limited capabilities due to film damage from e-beam (only low energy excitation are safe, limiting the thickness and material range)

2004 Semiconductor bookings for thin film x-ray metrology* tools >$50M and growing at over 50% year over year

* Not including TXRF (contamination) and back-end x-ray inspection tools

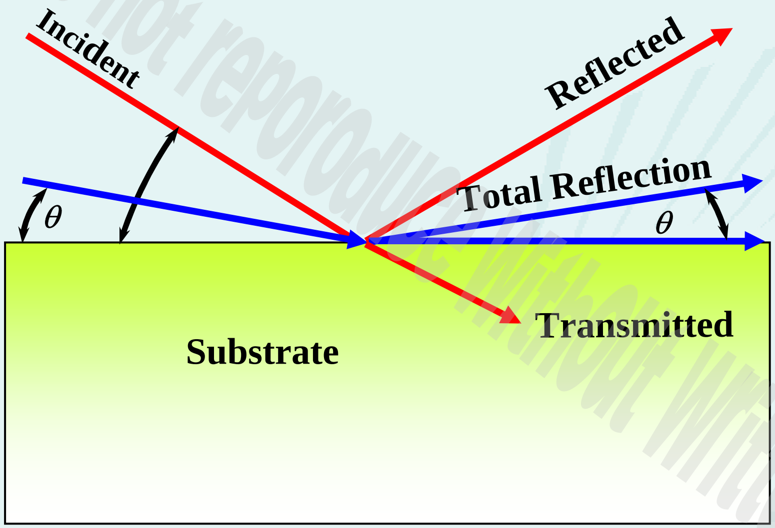
XRR – X-ray Reflectivity

- Thickness (few Å - 1μ; application dependent)
- Density (ρ) (<2% accuracy)
- Surface and Interface Roughness



Single, multi-layer or Periodic multi-layer Stacks

XRR – X-ray Reflectivity from a Substrate



$$n = 1 - \delta - i\beta$$

$$\delta \approx \frac{r_e \lambda^2}{2\pi} \sum_i N_i \cdot f_{i_1} \quad \propto \quad \rho_e$$

$$\beta \approx \frac{r_e \lambda^2}{2\pi} \sum_i N_i \cdot f_{i_2} \quad \propto \quad \mu_x$$

$$\theta_c \sim (2.\delta)^{1/2} \propto \mathbf{Z}^{1/2}$$

r_e = Classical electron radius

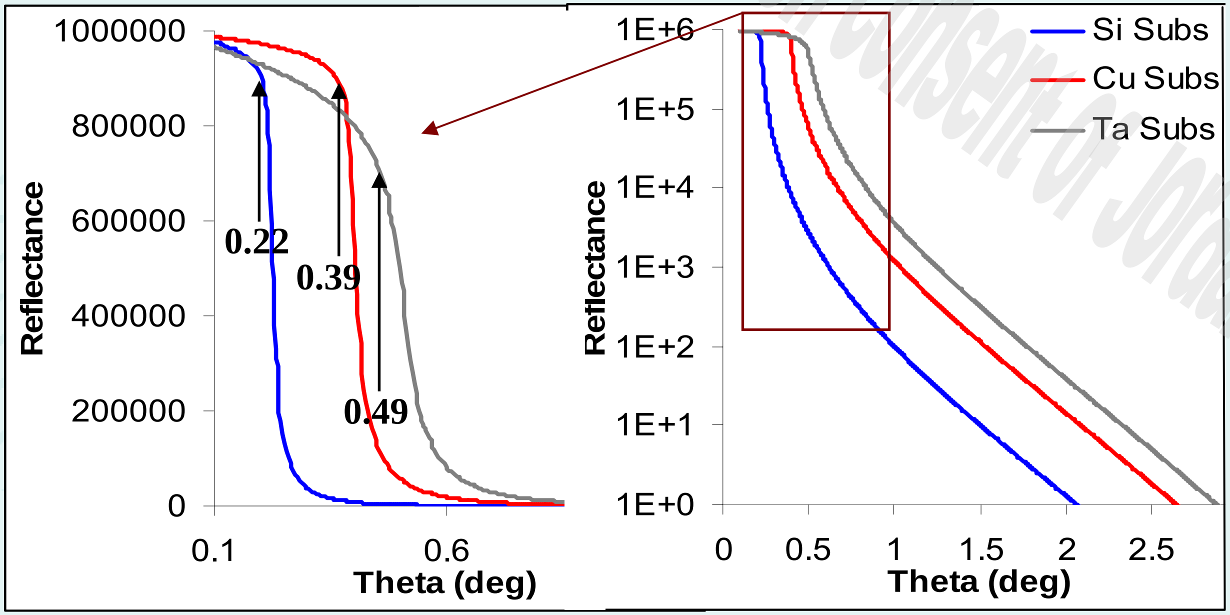
λ = X-ray wavelength ~1.5A

N_i = Atomic density

f_i = Atomic scattering factors

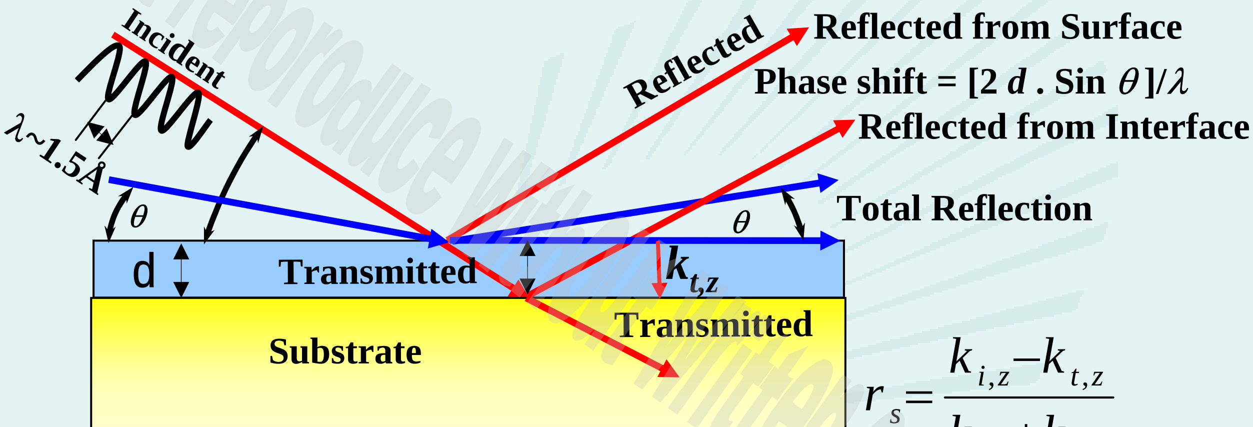
ρ_e = Electron density

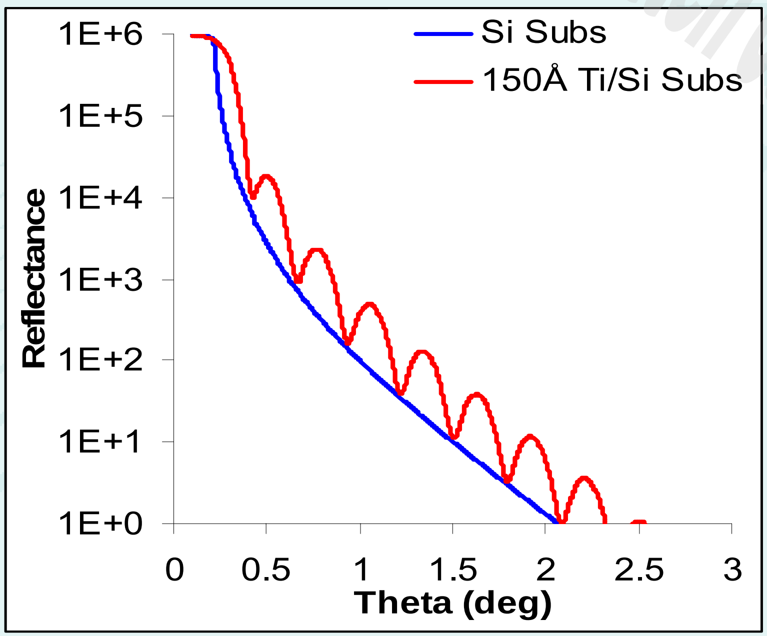
μ_x = Absorption coefficient



- The refractive index n ~ 1
- For most materials n only varies by <10^{-5}
- Below certain angles of incidence, all x-rays are reflected.
- At certain angle, θ_c, reflectivity begin to decays exponentially
- For X-rays, θ_c ranges from 0.1° to 0.6° for most materials.

XRR – X-ray Reflectivity from a Thin Film





$$r_s = \frac{k_{i,z} - k_{t,z}}{k_{i,z} + k_{t,z}}$$

$$k_{t,z} = \frac{2\pi}{\lambda}\sqrt{n^2 - \cos^2\theta}$$

$$r = r_s \cdot e^{-2k_{i,z}\cdot k_{t,z}\sigma^2}$$

$$R = \left[\frac{r_1 + r_2 e^{-2k_{i,z}\cdot k_{t,z}\sigma^2}}{1 + r_1 r_2 e^{-2k_{i,z}\cdot k_{t,z}\sigma^2}}\right]^2$$

Reflectivity (r_s) from any interface is calculated using Fresnel's equations employing continuity conditions for incident, reflected and transmitted waves.

The exponential term accounts for the reflectivity from a rough interface [Sinha et. al]

where r_1 and r_2 are the reflectivity of the two interfaces (air/film) and film/substrate).

For multi-layer, a recursive formula was developed by Parratt et. al.

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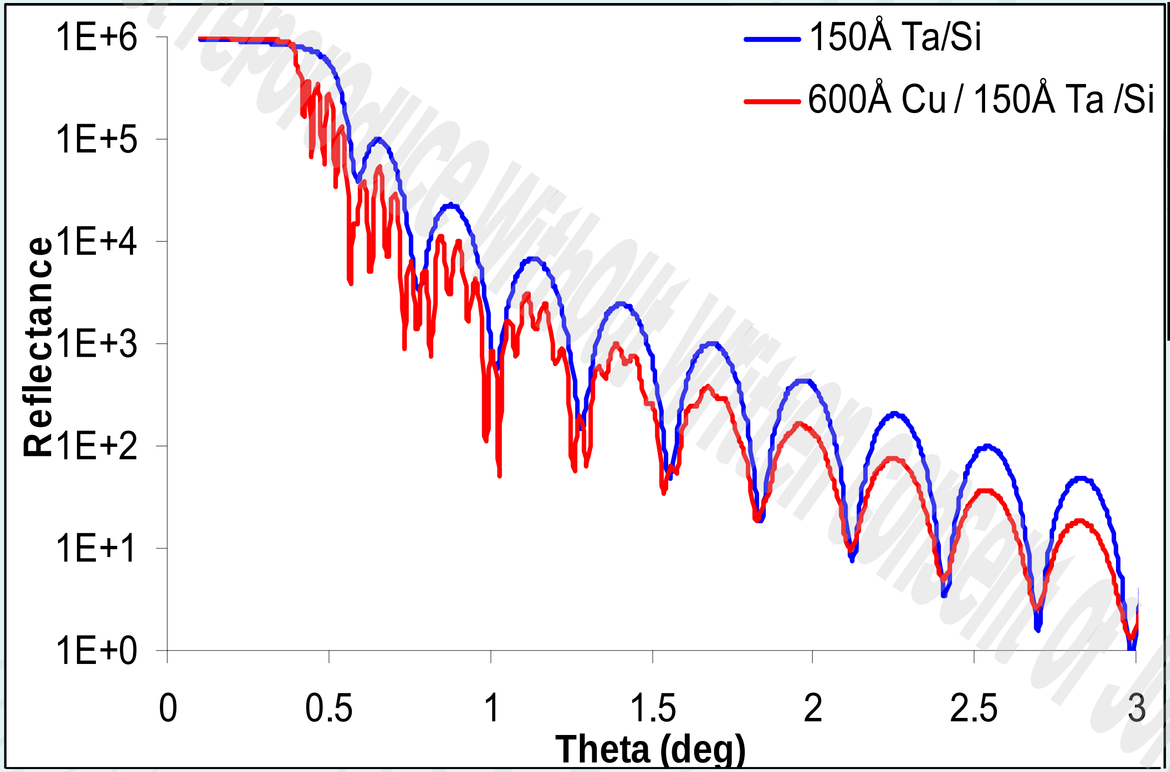
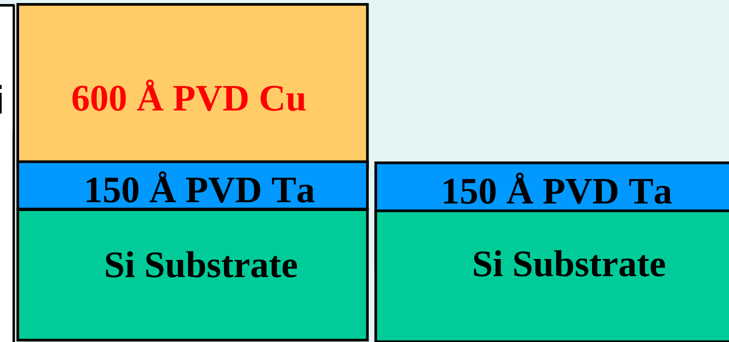
XRR – Reflectivity from Single and Bi-layer Stack





- Thicknesses of individual films in a multilayer stack are decoupled.
- The high frequencies correspond to thick films while low frequencies correspond to thin films.
- Density is obtained from the critical angle and amplitude of the fringes.
- Fringe pattern is a fingerprint of layer order with in a stack with top layers contributing at low angles and bottom layers contribution at higher angles.
- For simple stacks, the spectra is very easy to interpret.

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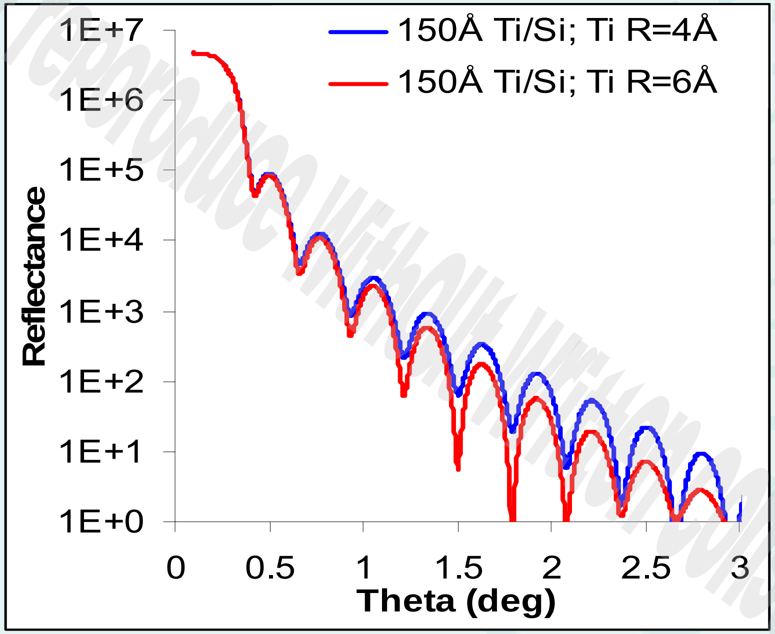
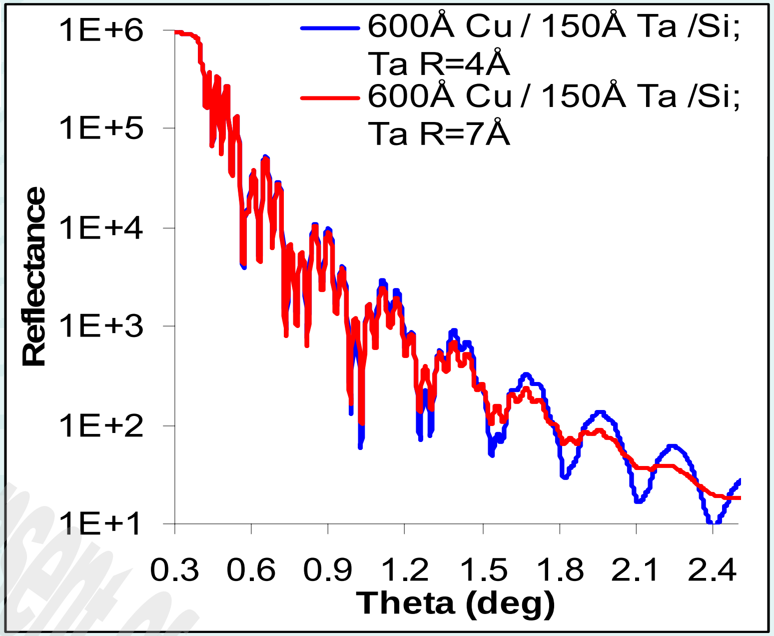
XRR – Surface and Interface Roughness





- Top surface roughness causes scatter of the incident flux => impacting the decay (exponential) of the reflectivity pattern.
- The interface roughness causes scatter of the reflected flux with in the film => dampening of fringe amplitude.
- Graded films also produce an effect similar to interface roughness.
- XRR is very sensitive to roughness <30-40A. For higher roughness, XRR signal detoriates quickly.

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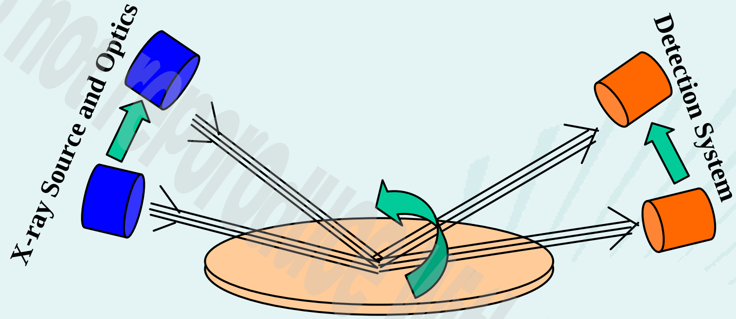
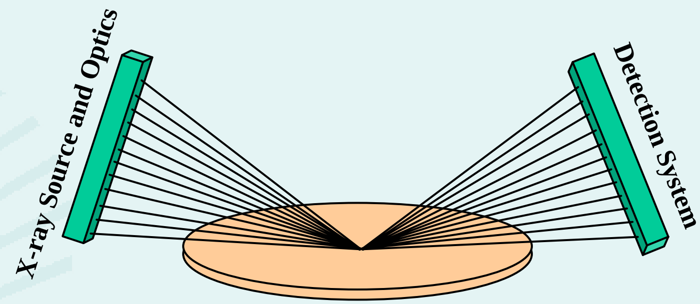
Recent Advances: Classical vs. Fast XRR



Goniometer Stage



X, Y, Z, θ Stage

Limitations of Goniometer Based Systems

- **Scans theta in small steps using Goniometer**
  - Long measurement times ~ Typical 30min-1hr.
- **Uses parallel beam optics leading to large spot**
  - Typical spot size ~ 5mmx5mm @ θ =1^0 using a 50µm x5mm slit.
  - Slits define spot size at the cost of flux.
  - No pattern wafer capability
  - Large edge exclusions.
- **Moving Goniometer, Detector, Optics, and Source**
  - Requires frequent calibrations
  - Increased maintenance
- **Uses high power x-ray sources (2kW-15kW)**
  - Limited source life
  - Film damage concerns

Fast XRR with focused beam optics

Hardware:

- **X-ray source + optics producing focused beam**
- **High dynamic range array detector (10^7) for simultaneous measurements of data at various angles.**
  - Small spot size ~60µm x 1mm @ θ =1^0
  - Allows product wafer measurements.
  - Very small edge exclusion ~ 1mm
  - Rapid data collection - Typical measurement 1-10 sec.
  - Fewer moving parts (stage only): less wear-tear, low maintenance, MTBF > 3months
  - Uses low power x-ray source. ~30-50 watts
  - Excellent source life
  - No risk of material damage

Software:

- 300mm SECS/GEM automation ready.
- Fully automated real time analysis.

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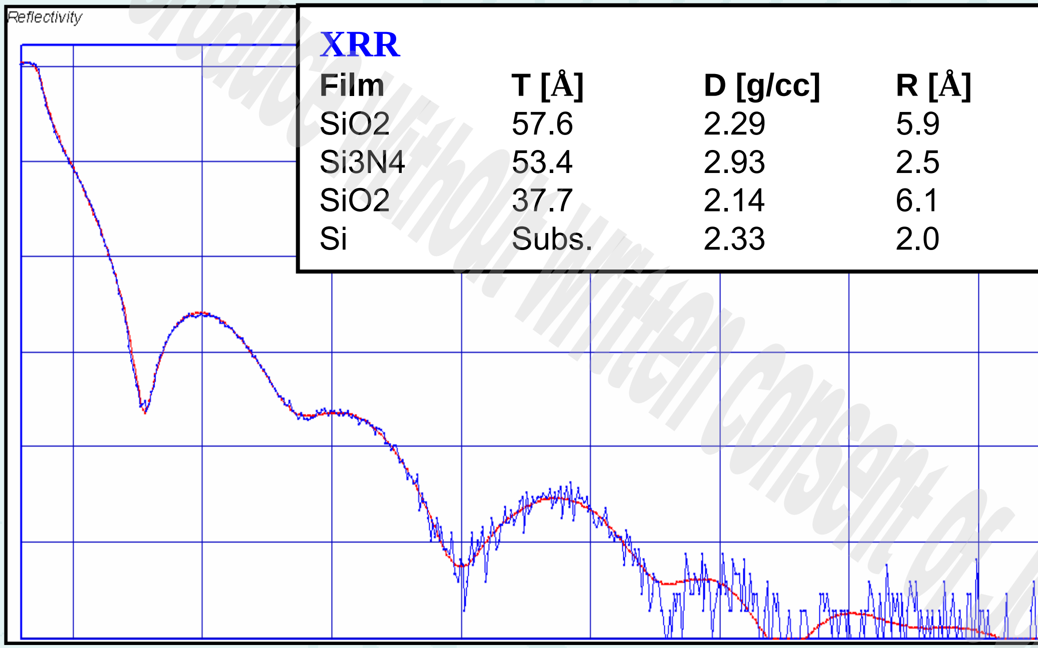
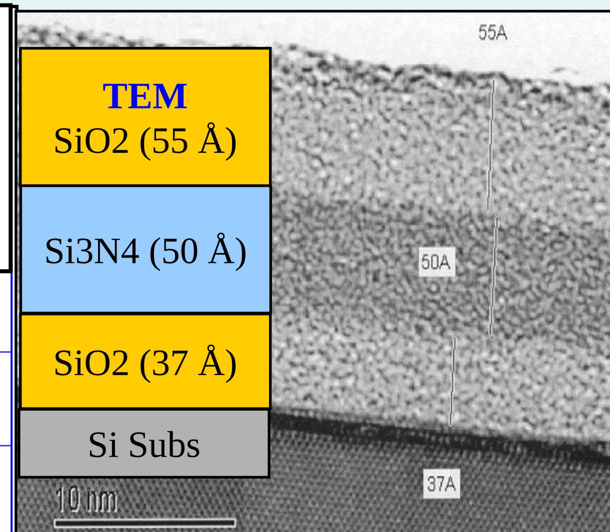
XRR Accuracy -ONO Gate Stack



XRR

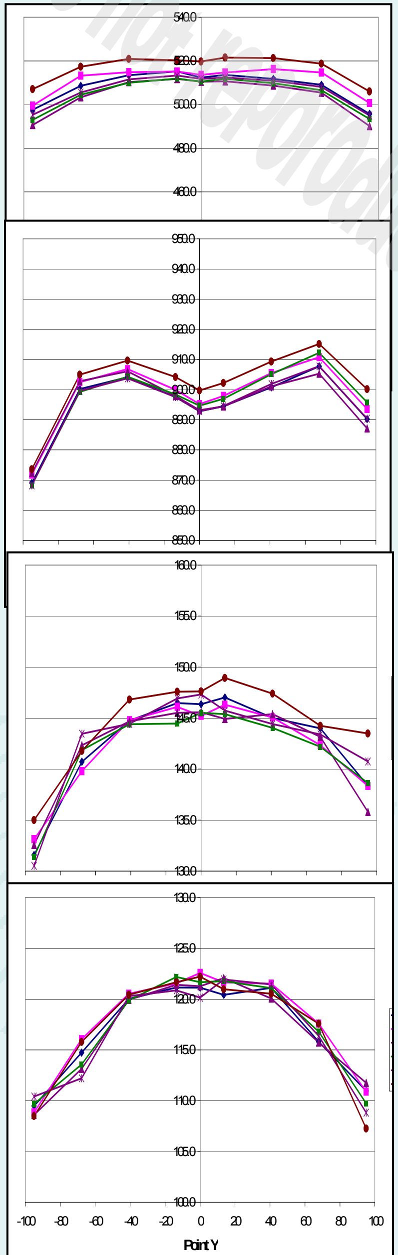
Film	T [Å]	D [g/cc]	R [Å]
SiO2	57.6	2.29	5.9
Si3N4	53.4	2.93	2.5
SiO2	37.7	2.14	6.1
Si	Subs.	2.33	2.0



XRR vs. TEM

- Angstrom level accuracy.
- Multi-layer characterization in a single measurement.
- Negligible correlation between film thicknesses.
- Optical film measurement techniques have high correlation between top and bottom oxide layers

XRR- Typical Multi-layer Analysis

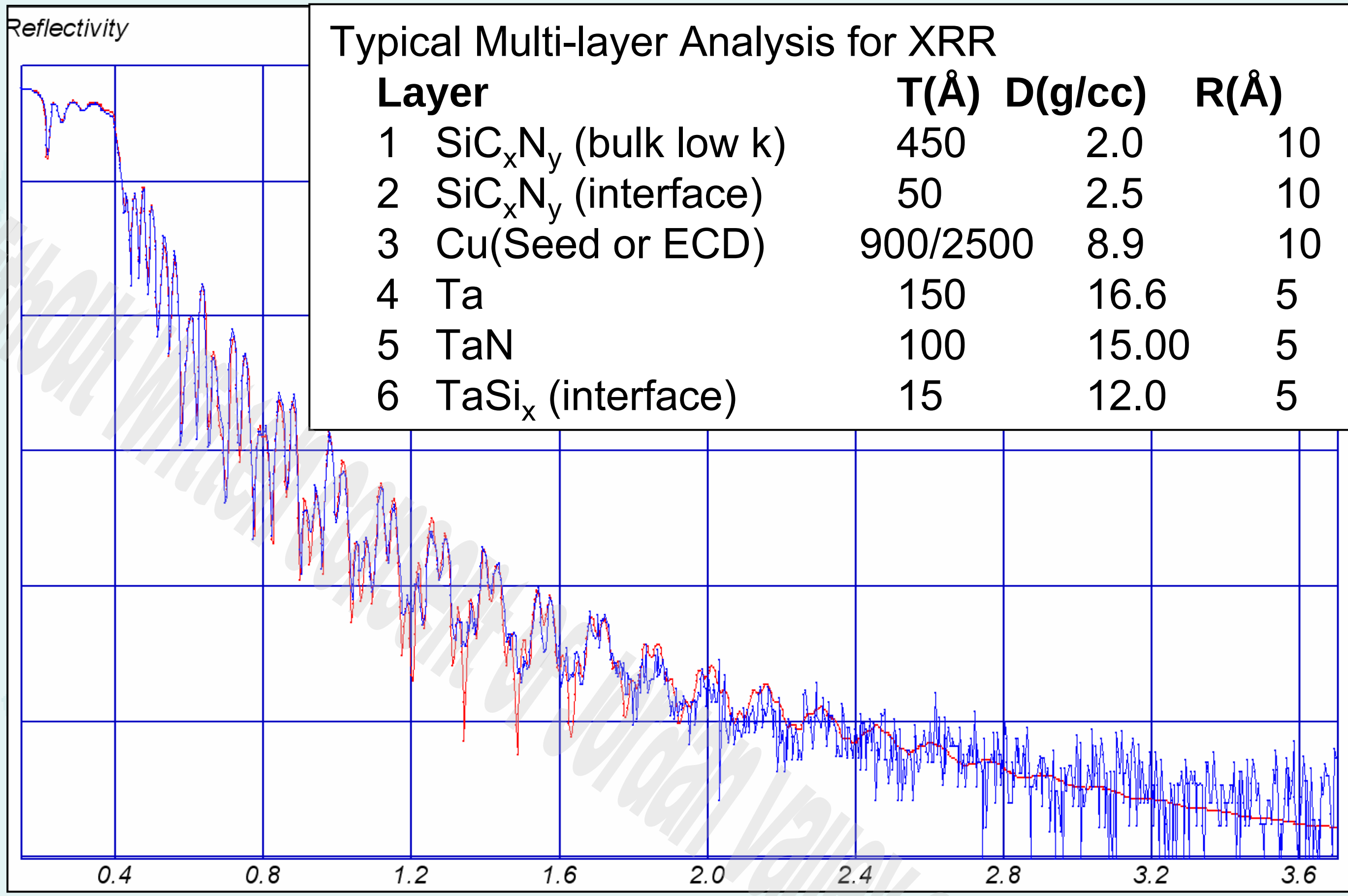


Low-K

Cu

Ta

TaN



Typical Multi-layer Analysis for XRR

	Layer	T(Å)	D(g/cc)	R(Å)
1	SiC_xN_y (bulk low k)	450	2.0	10
2	SiC_xN_y (interface)	50	2.5	10
3	Cu(Seed or ECD)	900/2500	8.9	10
4	Ta	150	16.6	5
5	TaN	100	15.00	5
6	$TaSi_x$ (interface)	15	12.0	5

A single measurement can characterize a multi-layer stack

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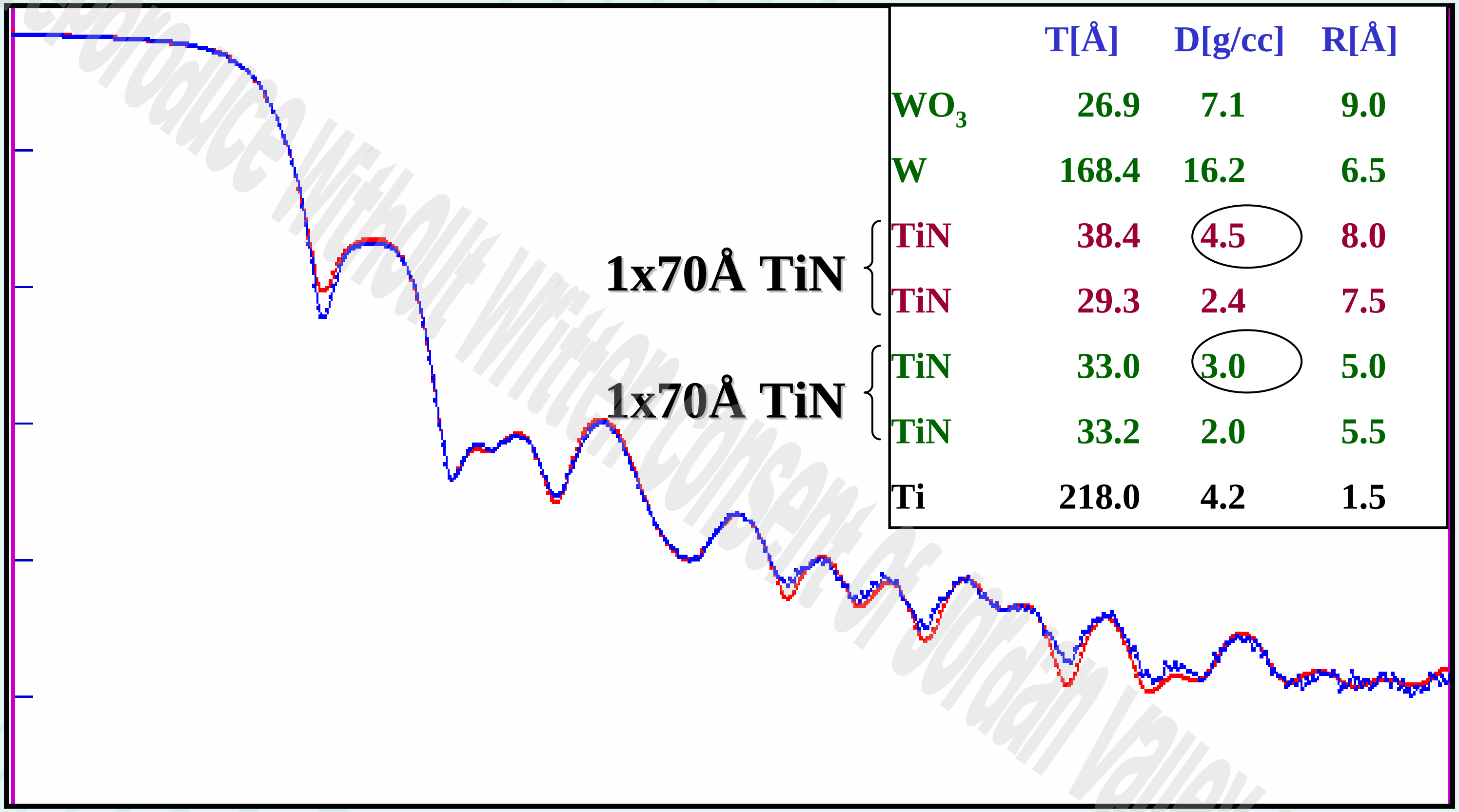
140Å W / 2x70Å TiN / 210Å Ti / 5000Å SiO_2 /Si



	T[Å]	D[g/cc]	R[Å]
WO_3	26.9	7.1	9.0
W	168.4	16.2	6.5
TiN	38.4	4.5	8.0
TiN	29.3	2.4	7.5
TiN	33.0	3.0	5.0
TiN	33.2	2.0	5.5
Ti	218.0	4.2	1.5

- Reveals the interface created by partial plasma treatment of TiN films.
- No other known non-destructive/non-contacting technique can characterize this stack simultaneously .
- 10s data is sufficient for full characterization

Typical Fab Applications of Fast XRR

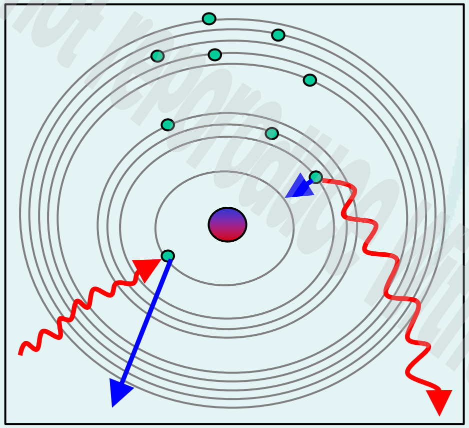
FEOL Applications

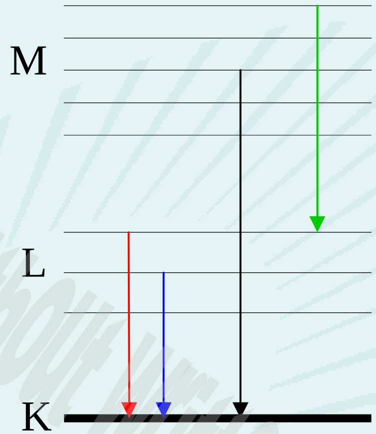
- SOI
- Advanced Gate Dielectrics -
  - SiON
  - ONO
  - High K
- SiGe, SiGe on SOI
- Metal Gate
- Silicides -
  - Co
  - Ni
- Organic ARC's

BEOL Applications

- Barrier Seed/Liner -
  - Cu Seed / Ta
  - Cu Seed / Ta /TaN
- Top Barrier and Etch Stop Layers -
  - SiCN/Cu/Ta
  - SiOC/Cu/Ta
- Low-k -
  - Low-k
  - Low-k/Cu/Ta
- Al Processing -
  - W_{nuc} on Ti/TiN
  - W
  - Ti / TiN

X-Ray Fluorescence



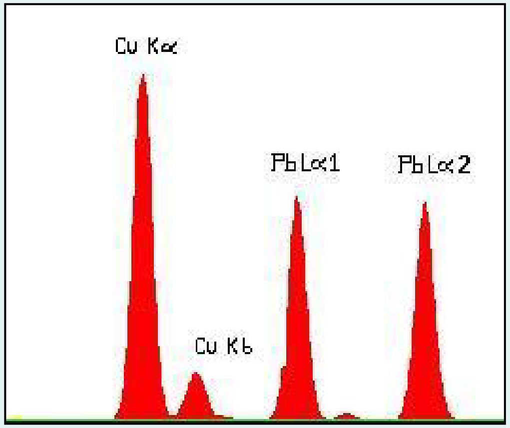


$$E_K - E_{L_3} \Rightarrow K\alpha_1$$

$$E_K - E_{L_2} \Rightarrow K\alpha_2$$

$$E_K - E_{M3} \Rightarrow K\beta_1$$

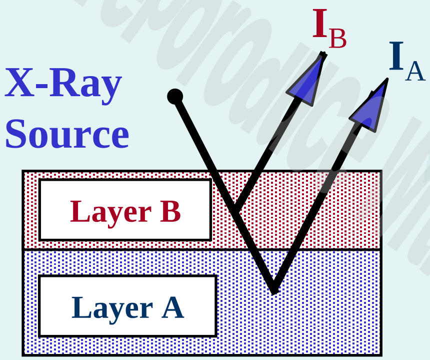
$$E_{L_3} - E_{M_5} \Rightarrow L\alpha_1$$

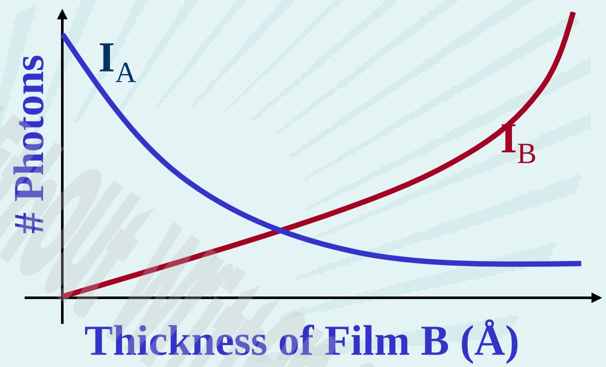


- Incident x-ray via photoelectric interaction knock out electrons from innermost atomic shells.
- Vacancy is filled by electrons from the outer shells causing emission of characteristic X-rays labelled as K, L, or M (denoting the shells they originated from)
- Additional designations of α, β, or γ, are used for transition from various atomic shells.
- Transitions from sub-shells to a shell are further designated as α1 ,α2 or β1 , β2 , etc.

- XRF is measured using either WD (Wavelength Dispersive) or ED (Energy Dispersive) methods.
- **ED-XRF** uses detectors capable of energy discrimination. This method provides higher XRF flux. Low-power x-ray sources work quite well. Typical energy resolution ~ 150-200eV.
- **WD-XRF** combines counting detectors and diffraction based crystals for energy selection. This method provides excellent energy resolution, however at cost of flux (requires high power x-ray sources). Typical resolution ~ 10-20eV.

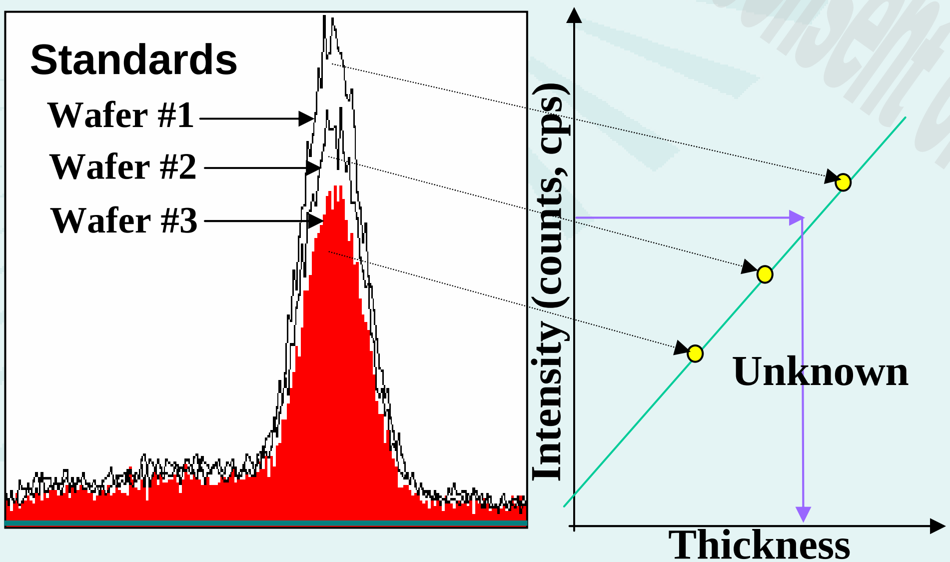
X-Ray Fluorescence





XRF yield depends on –

- XRF source energy
- Number of atoms available for excitation.
- Detection Efficiency.
- Absorption Effects.



XRF Intensity is calibrated using known standards -

- Linear (<0.5μm)
- Polynomial (0.5μm<2μm)
- Exponential (> 2 μm)
- Standard-less methods are also used

Typical Fab Applications of μ-XRF

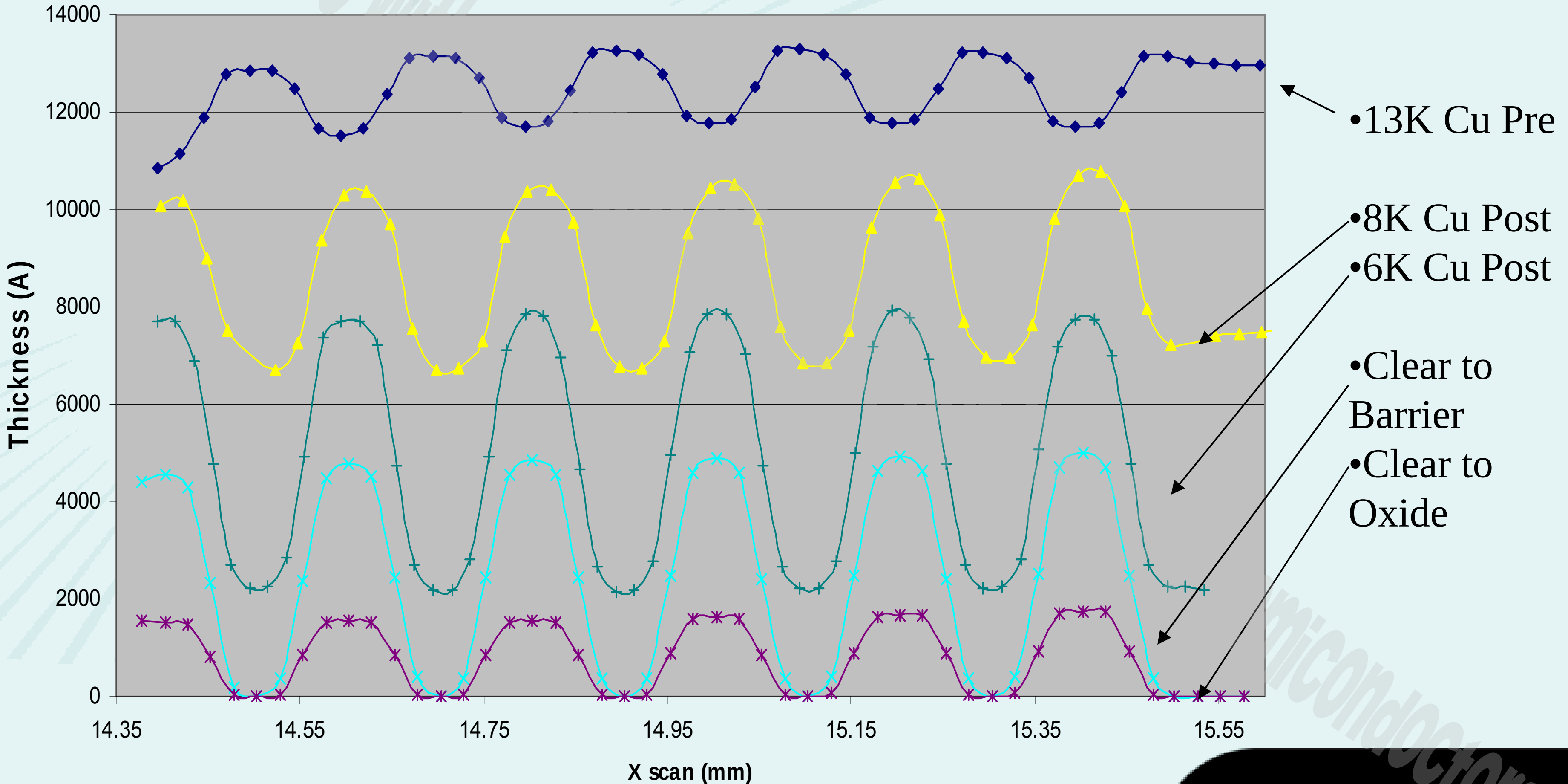
FEOL Applications

- SiGe, SiGe on SOI – Composition
- Sidewall thickness measurements
- Silicidation Process – Composition Anneal, Strip, Post Strip, etc.
  - Co
  - Ni

BEOL Applications

- Cu Processing
  - Cu Plating Thickness
  - Post CMP Thickness
  - Dishing and Erosion
- Al Processing -
  - W Plating and Polish
  - W Dishing and Erosion
  - Al Thickness

Pre and Post Polish Characterization Across Cu Trenches – Single Recipe



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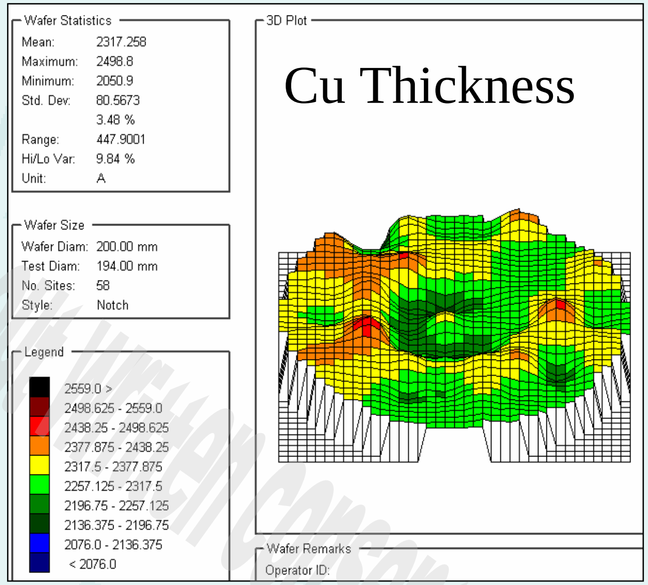
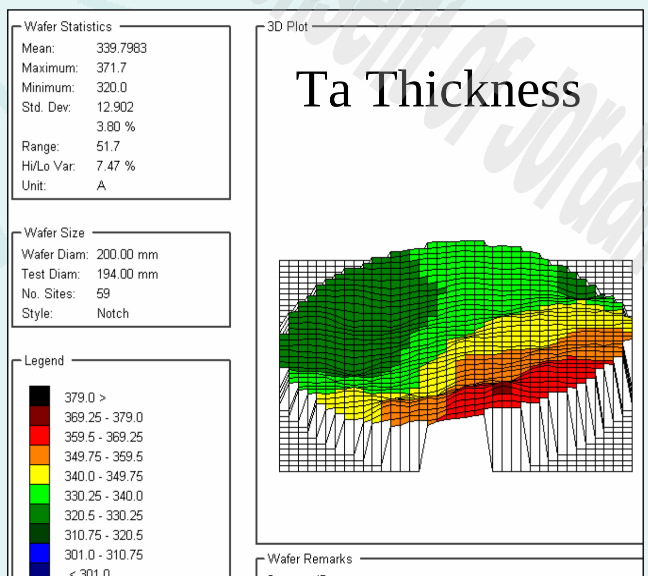
CMP Study using XRF

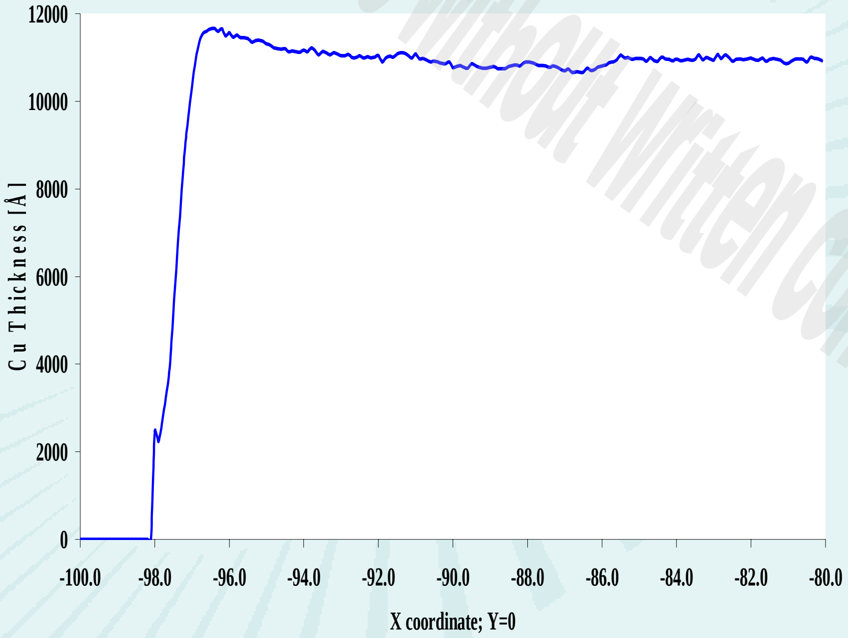


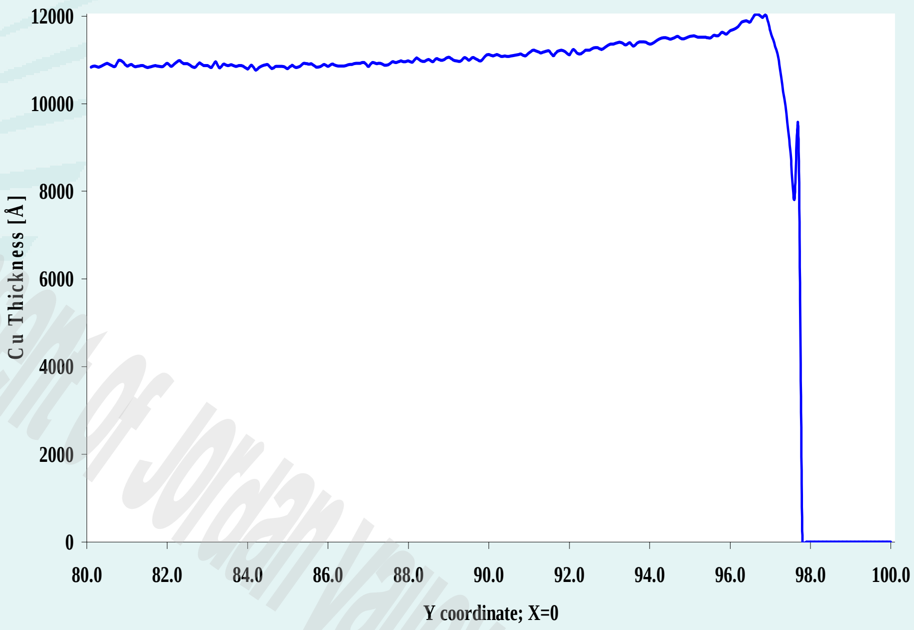


Sheet-ρ depends on combined barrier and Cu thickness

1µm Cu Characterization: XRF Data

Edge Profile: Measurement steps of 100 µm





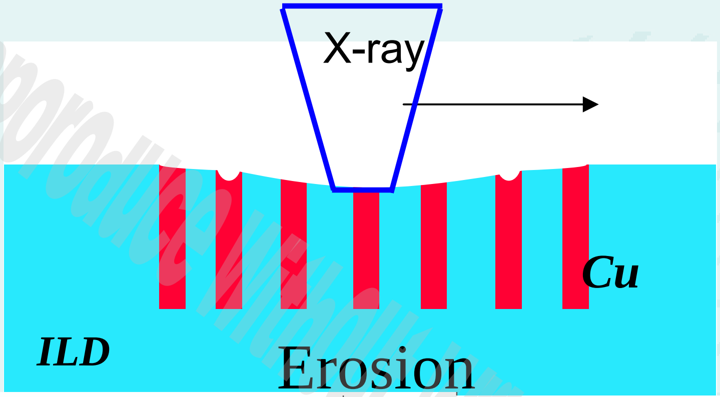
Cu film begins at X = -98.0

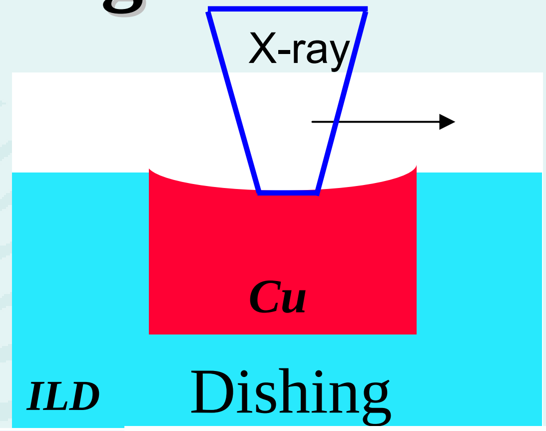
Cu film reaches maximum thickness at X = -96.4

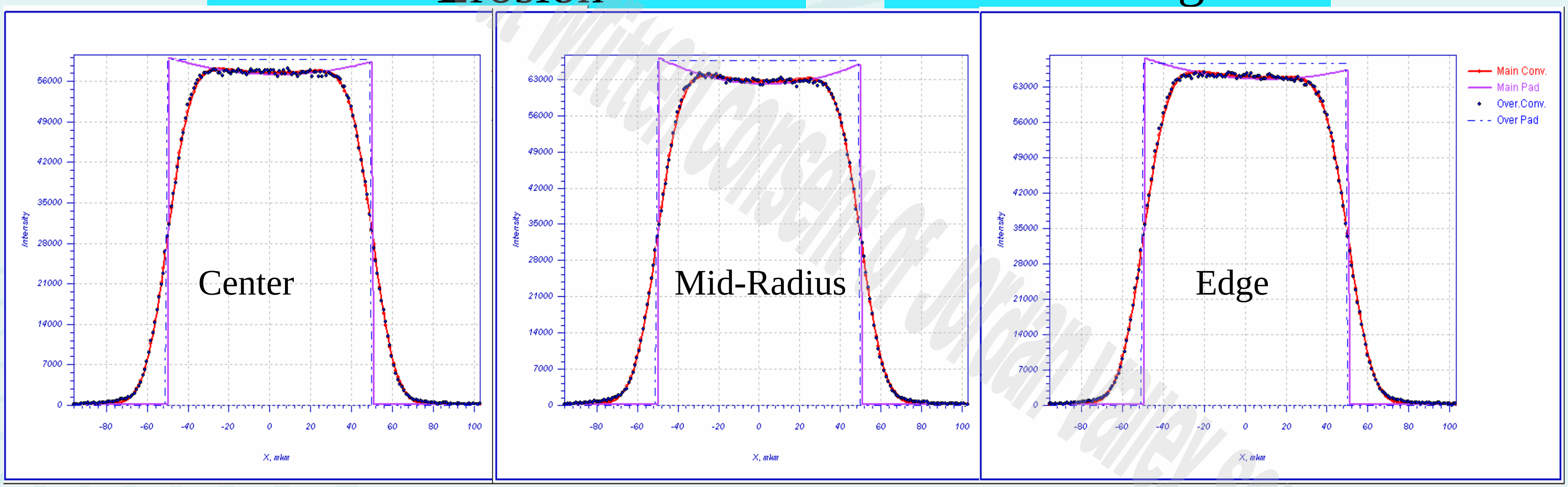
Cu film begins at X = 97.7

Cu film reaches maximum thickness at X = 96.7

Post Cu Polish Dishing and Erosion







	Center	Mid Radius	Edge
Dishing, %	4.4	6.9	4.4
Slope, %	-1.2	-1.9	-3.6

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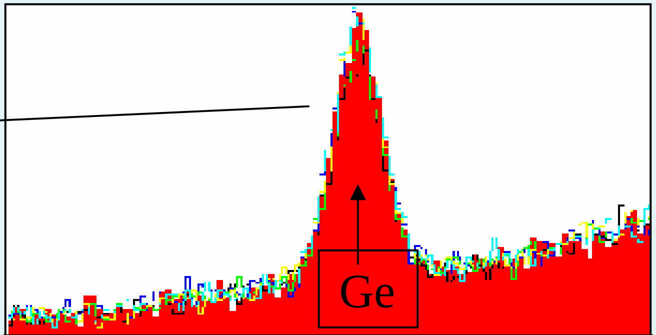
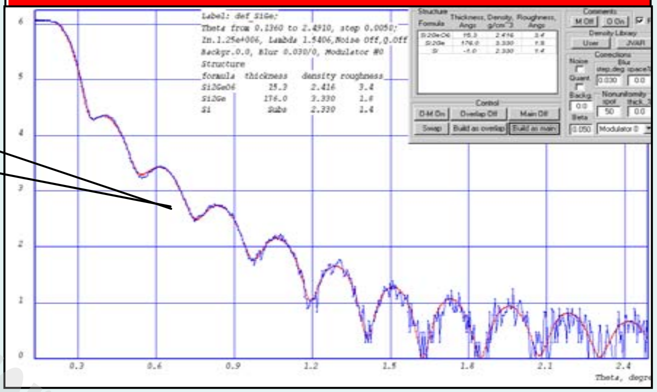
‘X’ Calculation using XRR and XRF Data

$$C_{Ge\%} = \frac{A \cdot I_{Si_{1-x}Ge_x(XRF)}}{T_{Si_{1-x}Ge_x(XRR)} \cdot \rho_{Si_{1-x}Ge_x(XRR)}}$$





$$X = \frac{C_{Ge\%} \cdot A_{Si}}{A_{Ge} + C_{Ge\%}(A_{Si} - A_{Ge})}$$

X-values for $Si_{1-x}Ge_x$ using combined XRR and XRF data

Wafer ID	Customer X - Value	JVX5200 X - Value
W1	0.301	Reference
W2	0.217	0.212
W3	0.170	0.168
	RBS	XRR/XRF

Wafer 1 was used as standard

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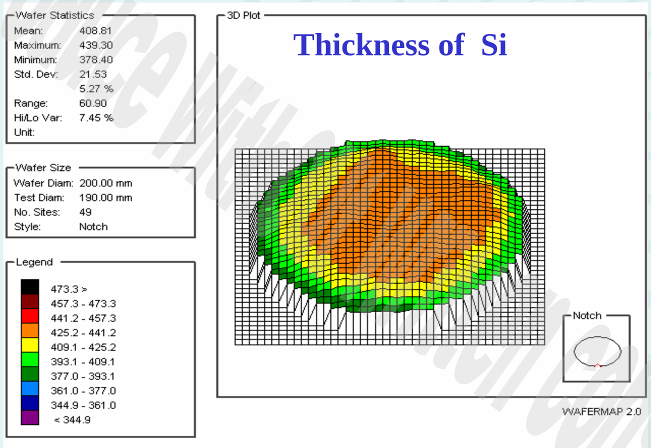
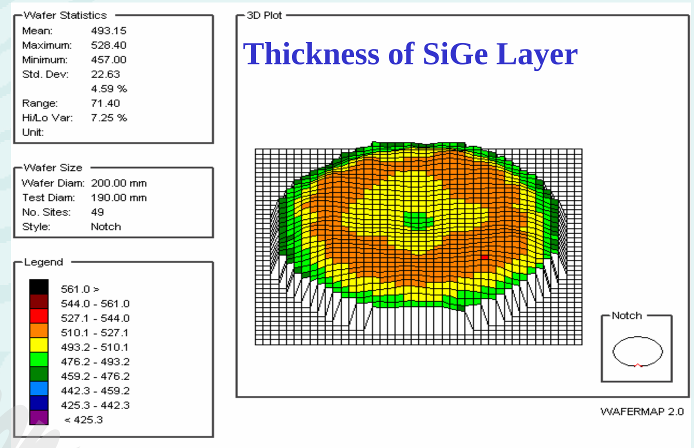
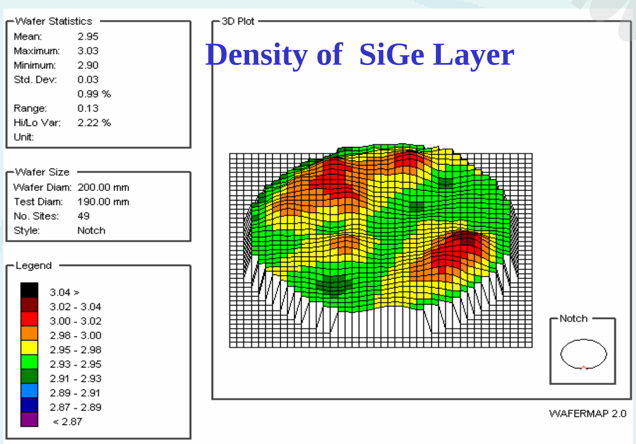
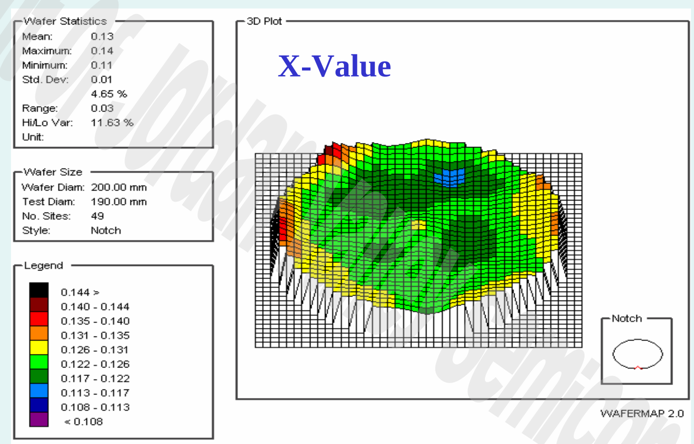
Full SiGe Process Control

500 Å Si / 500 Å $Si_{.875}Ge_{.125}$ / 20 Å Si / 500 Å SiO2 / Si









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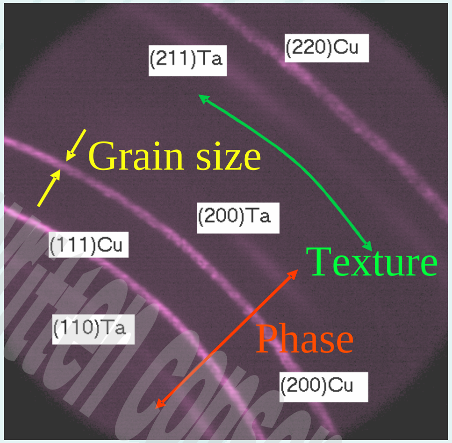
X-Ray Diffraction

Conventional XRD

- Texture (non-random or preferred orientation of crystallites)
- Phase
- Grain Size
- % Crystallinity



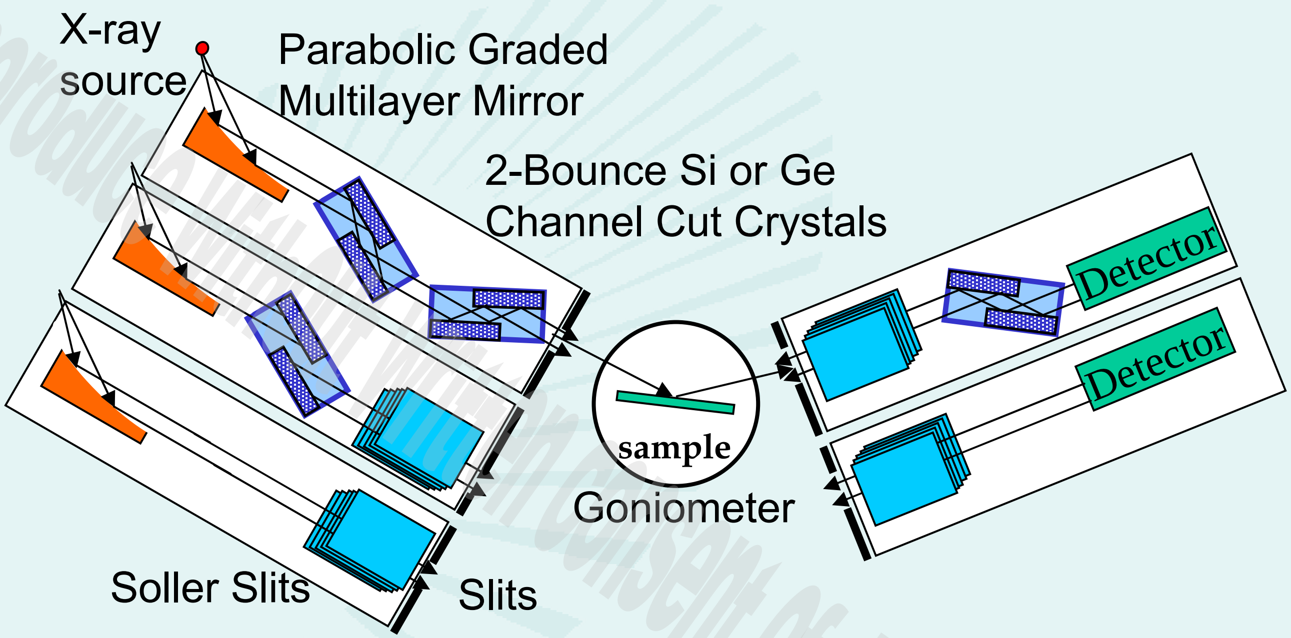
Hi-Resolution XRD

- Rocking Curves
  - Single Crystals
  - $SiGe_x$ Epitaxial films

Recent Developments

- Packaging of Conventional Diffraction Systems for in-line Fab use.
- Hi-power x-ray sources to reduce measurement times.
- Small-spot focusing optics (low-res. XRD).
- Large Area Detectors to accomplish rapid scans (low-res. XRD).
- Recipe based data collection and automated analysis.
- SECS/GEM compliant systems

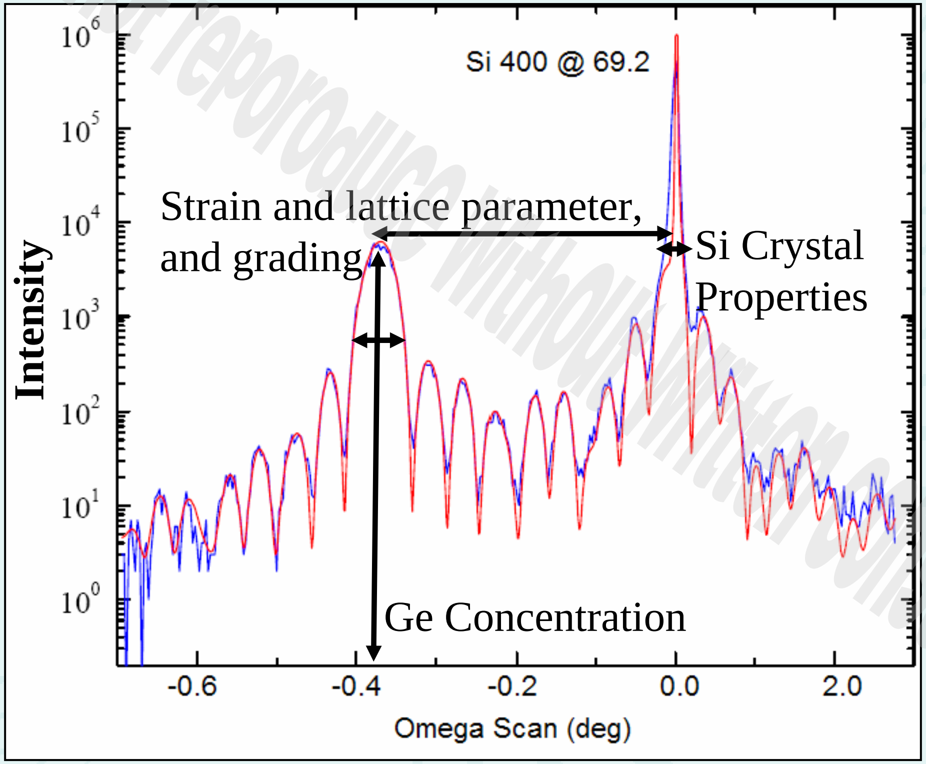
Various Flavors of Hi-Resolution XRD



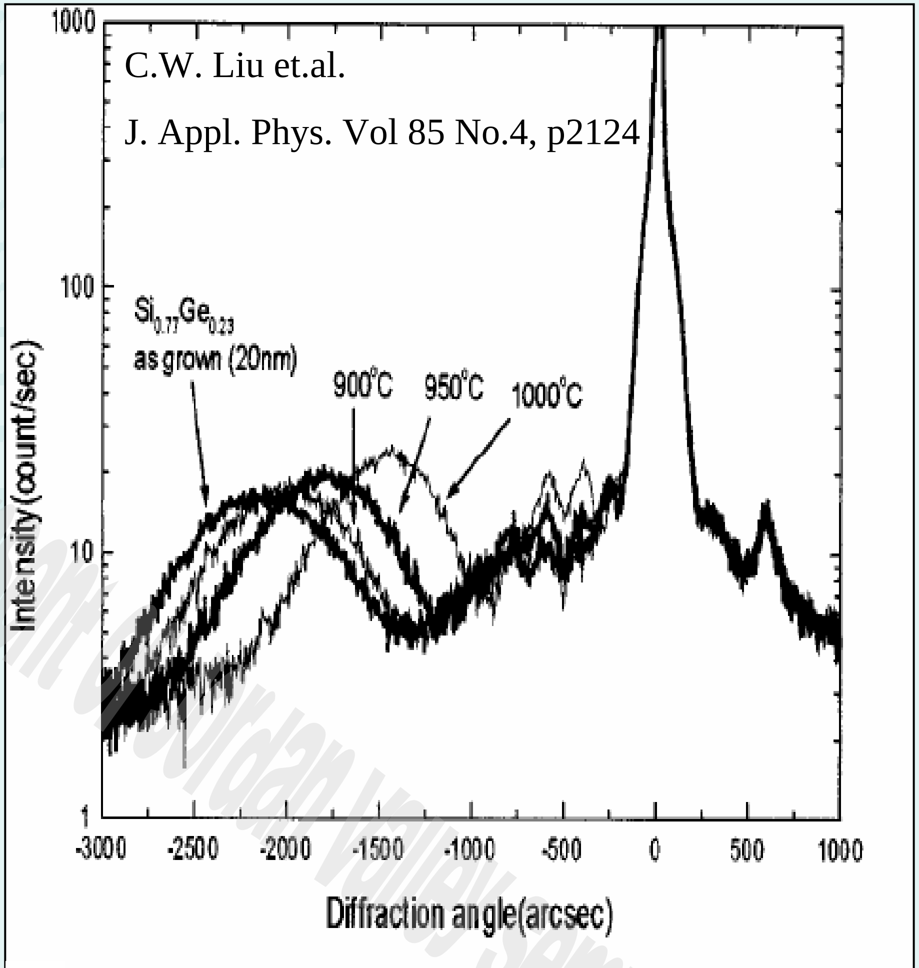
- Hi-resolution XRD requires highly parallel x-ray beam to accomplish high resolution angular scan (typical anywhere between 0.001-0.0005 degree steps)
- These systems can achieve very low divergence ~ 10-30 Arc-seconds.
- Requires precision channel cut high quality crystals.

- Applications include –
  - Study of imperfections in single crystals
  - Study of thickness, composition, and strain in Epitaxial films ($SiGe_x$)
  - Study of Super-lattices (Repeated multi-layer structure)

Hi-resolution XRD - $SiGe_x$

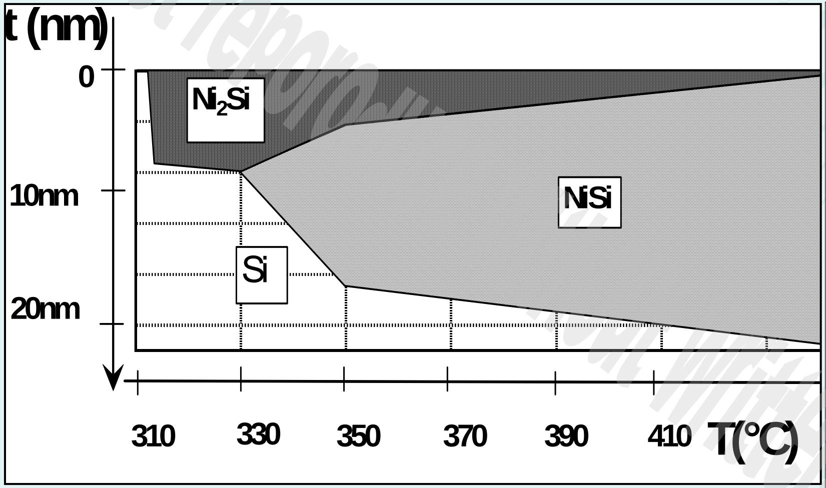


– Study of imperfections in single crystals
– Study of thickness, composition, and strain in Epitaxial films (e.g.. $SiGe_x$)
– Study of Super-lattices (Repeated multi-layer structures

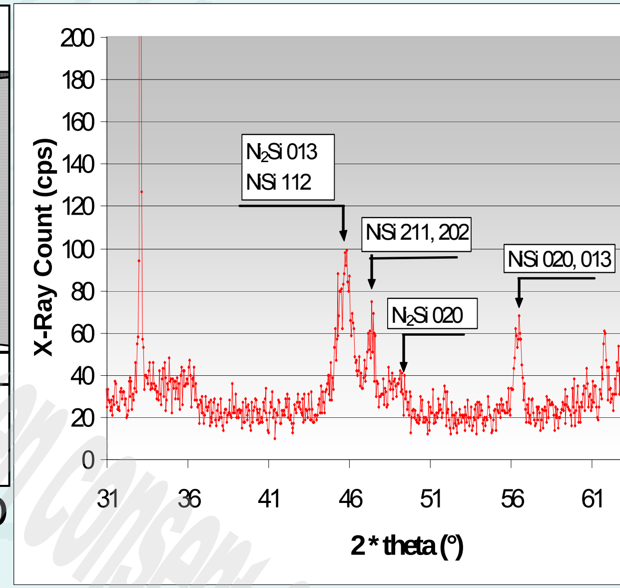


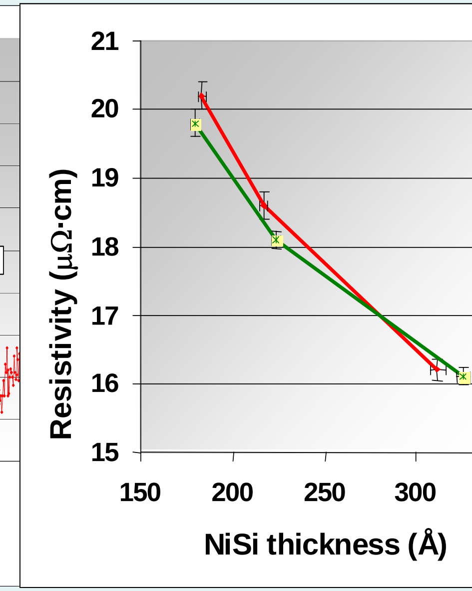
High resolution x-ray diffraction spectra of $Si/Si_{0.77}Ge_{0.23}/Si$ quan-
n wells annealed at different temperatures. The well thickness is 20 nm.

Low-resolution XRD –Phases of $NiSi_x$



XRR+XRD

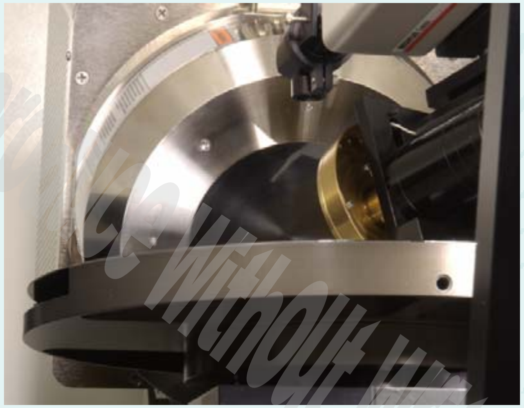


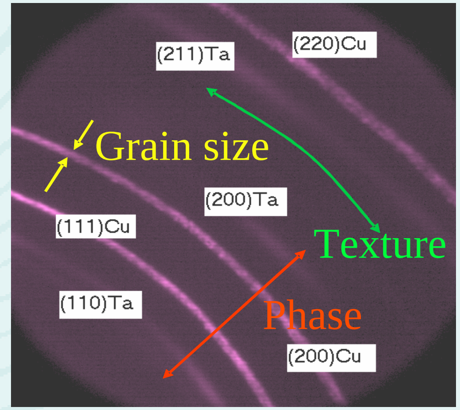


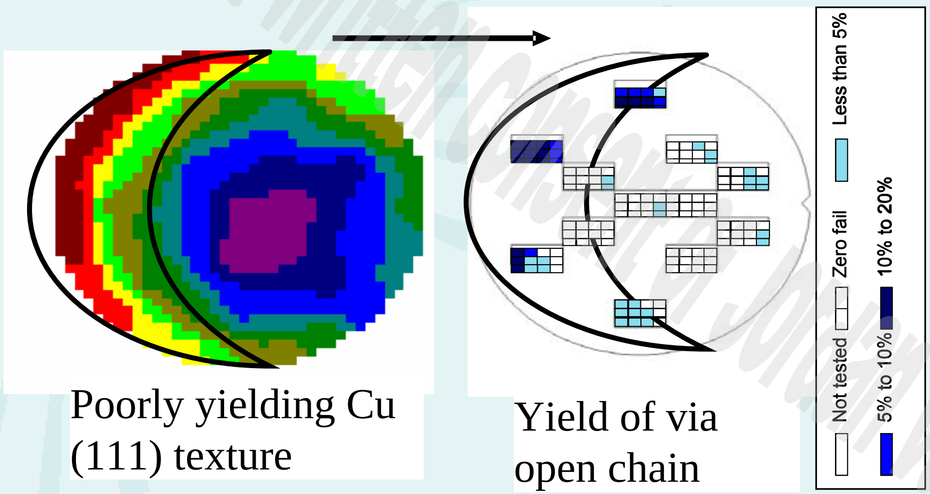
- XRD is used to study silicidation of Ni.
- Anneal temperatures directly impacts formation of a given phase.
- The resistivity of NiSi film is strongly correlates with NiSi thickness.
- Generally, Ni2Si and NiSi phases are formed during silicidation.
- The small Ni2Si (020) peak accounts for the presence of a thin Ni2Si layer,
- Most peaks correspond to the strongest reflections in the orthorhombic structures of the Ni2Si and NiSi phases.
- A typical phase diagram shown in left figure can be generated using XRD measurements.

Courtesy- ST Microelectronics

Low-resolution XRD – Cu/Ta/TaN







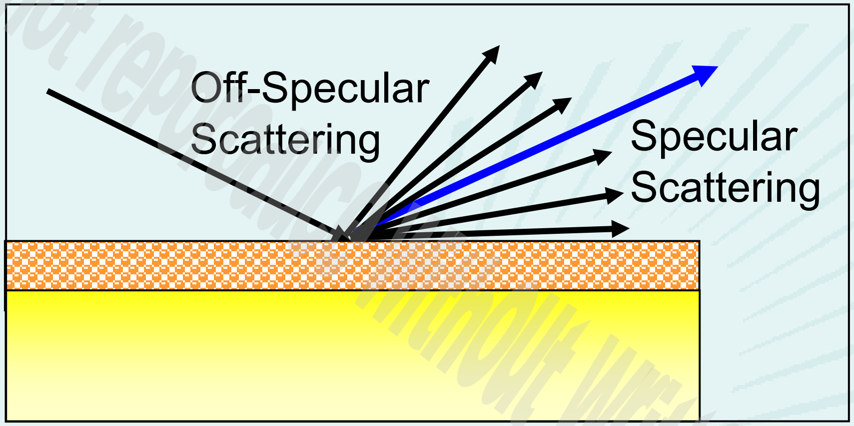
Poorly yielding Cu (111) texture

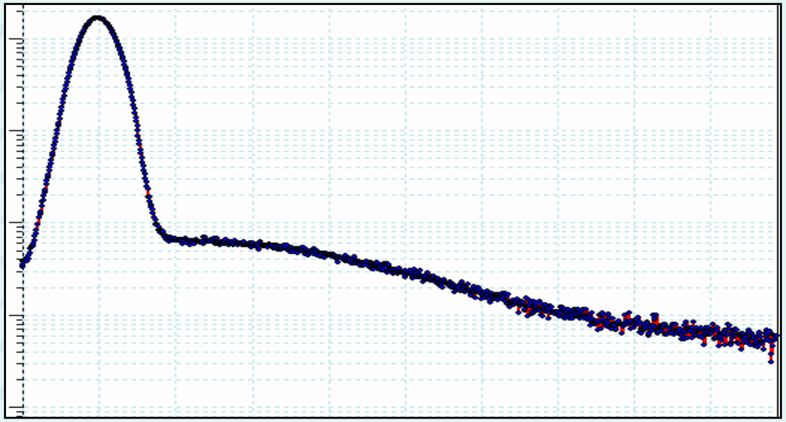
Yield of via open chain

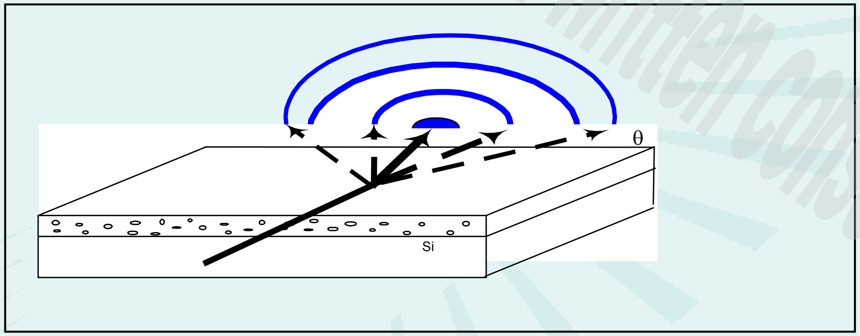
Cu Texture, i.e., non-random or preferred orientation of crystallites analysis of post-polish Cu establishes correlation of yield loss.

Courtesy- HyperNex

Small Angle X-ray Scattering (SAXS)







- SAXS is used for characterization of pore size and their distribution in a film.
- Either transmission or reflection geometry is used during SAXS data collection.
- Non-specular scattering is produced by the pores due to presence of regions of differing electron density.

- For randomly distributed pores, the scattering intensity as a function of Bragg size “d” or “r” is proportional to the number of scattering elements in the irradiated volume and its atomic scattering factor.
  – $I(q) = N_p(1/q) \cdot n_e^2(1/q)$, q= magnitude of scattering vector - $4\pi.\sin(\theta)/\lambda$ and r=1/q
- Typical scattering patterns display power-law decays in intensity which begin and end at exponential regimes that appear as knees in a log-log plot. These exponential knees reflect a preferred pore size r=1/q

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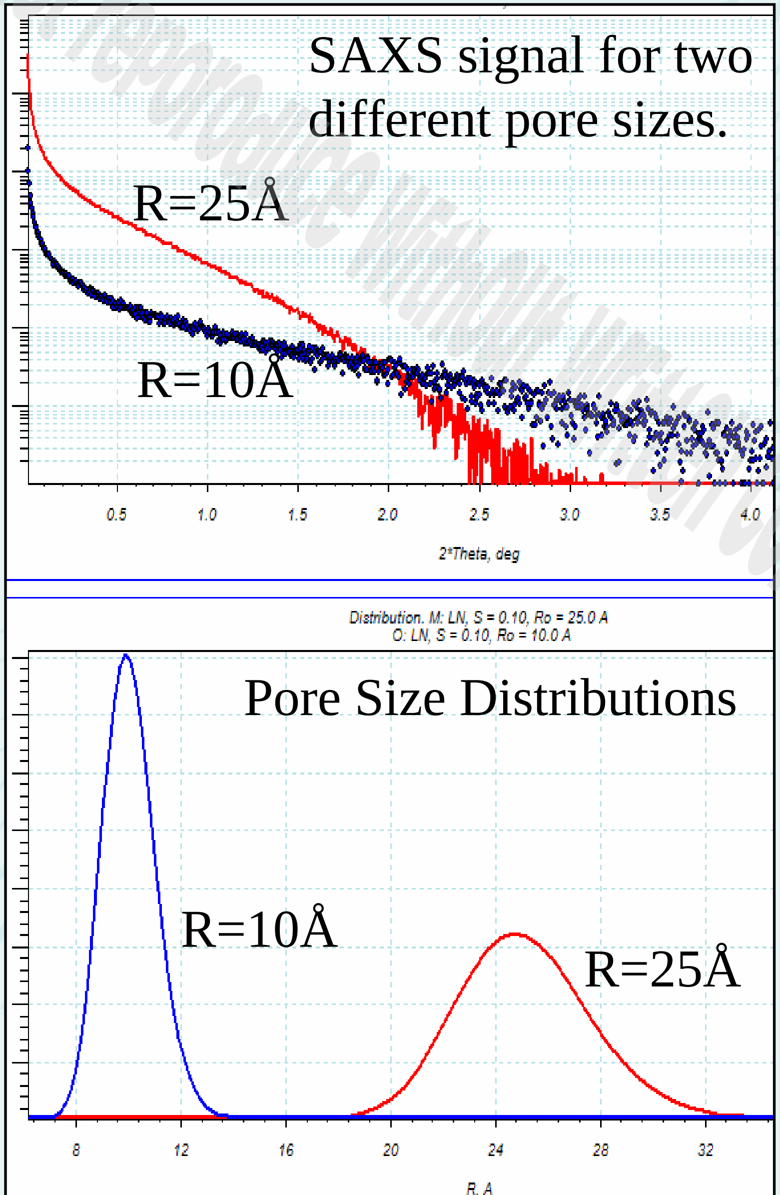
Small Angle X-ray Scattering (SAXS)



Data Analysis Methodology

- Assume a pore-size distribution function (sphere, rod, or Disk)
- Fit Data to determine mean pore-size and its distribution using a fitting simulation incorporating Guinier approximations

References

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