MATERIAL MEASUREMENT LABORATORY

Sample Alignment of X-Ray Reflectometry Using Thickness and Density From Certified Reference Materials

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Introduction to XRR Problem

Calibration of XRR measurement technique

•Why is XRR calibration important?

The semiconductor and optics industries need to precisely determine thin film character: thickness, roughness, density, composition, etc.

Nanotechnology research requires accurate thin film characterization

•What is required to calibrate XRR measurements?

Calibration artifact: *stable with time, well determined structure, rich in data features*

SI traceable measurement: *well characterized instrument response function*

Unbiased data analysis: *SI traceability between structural model and measurement*

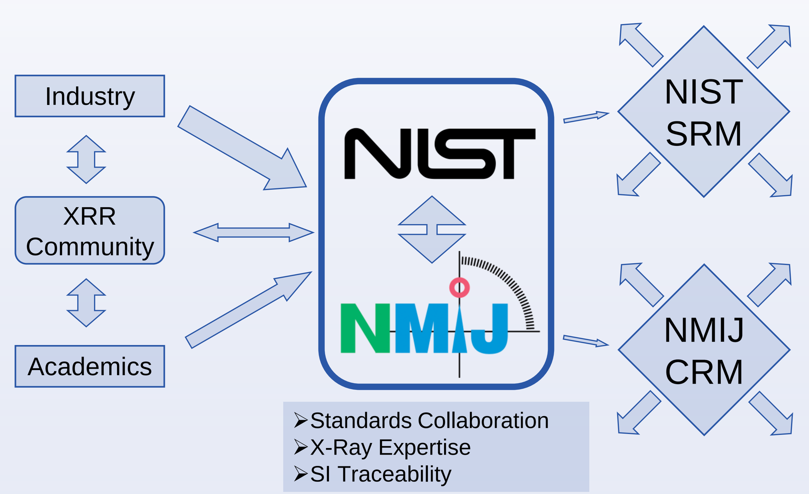
•What is the current status of an XRR calibration standard?

NMIJ & NIST collaboration on data analysis and recommended Reference Material use for calibration in the field

NIST is developing several materials in collaboration with SEMATECH and collaborators for future NIST SRMS in XRR

International Community Interaction

NIST's transfer method of calibration capability to customers: SRMs



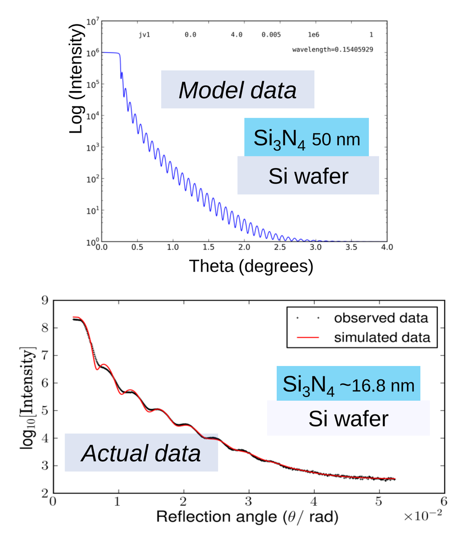
➢Standards Collaboration
➢X-Ray Expertise
➢SI Traceability

Thickness SRMs in development from NIST

	Artifact requirements	*Solutions*
Calibration Artifact		
Substrate	*Smooth, uniform density, thickness, and low cost* *Usable in laboratory, Fab tool, and synchrotrons*	***Si substrate***
Certified layer	*Uniform, well -characterized, -controlled process* *No density or thickness gradients in layers*	***Stable layer (TiN, SiN, HfO_2, ?)***
Sacrificial layer	*To minimize aging effects (e.g. oxidation) caused by exposure to environment, cap with a sacrificial layer whose properties will not be certified*	***(SiO_2 , ?) or other oxide cap***
XRR character	*Several high dynamic range oscillation fringes* *Good intensity through high angles*	***High electron density contrast***

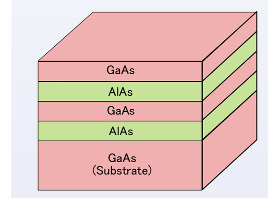
Artifact development in collaboration with SEMATECH

Proposed CVD Si_3N_4 structure



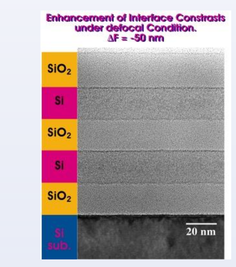
Thickness CRMs available from NMIJ

GaAs/AlAs Super lattice (NIMC CRM 5201-a)



Structure	Certified value[1] (nm)
1st Layer (GaAs)	-
2nd Layer (AlAs)	22.96 ± 0.20
3rd Layer (GaAs)	23.42± 0.33
4th Layer (AlAs)	22.98± 0.29

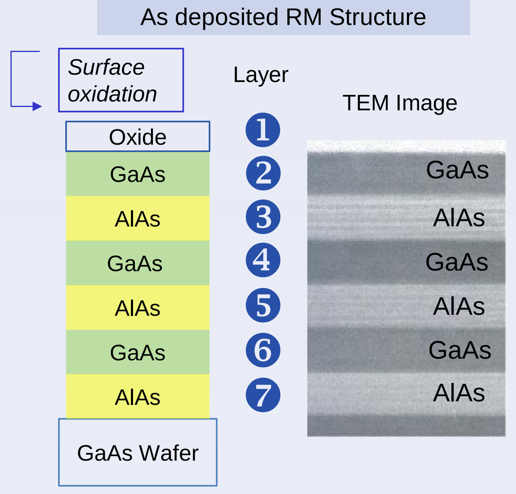
Si/SiO_2 multilayer (NMIJ CRM 5202-a)



Structure	Certified value[1] (nm)
1st Layer (SiO_2)	-
2nd Layer (Si)	20.0 ± 0.6
3rd Layer (SiO_2)	20.5 ± 0.8
4th Layer (Si)	19.9 ± 0.5
5th Layer (SiO_2)	20.5 ± 0.6

NMIJ Reference Material

As deposited RM Structure



CRM strategy

MBE epitaxial deposition of AlAs/GaAs produces high quality, stoichometric, multilayer with smooth interfaces and low inter-diffusion.

Nominal Reference values for NMIJ GaAs/AlAs RM

	Structure	Reference value (nm) (k=2) [1]	Information value (nm)
	Surface oxide	-	1.32
	1st layer (GaAs)	-	9.24
3	**2nd layer (AlAs)**	**9.65 ± 0.11**	
4	**3rd layer (GaAs)**	**9.51 ± 0.10**	
5	**4th layer (AlAs)**	**9.64 ± 0.11**	
6	**5th layer (GaAs)**	**9.51 ± 0.09**	
7	**6th layer (AlAs)**	**9.62 ± 0.11**	

NIST-NMIJ MCMC RM thickness stability study (5yrs)

	Structure (thickness in nm)	2004 NMIJ/GA	2004 NIST/MCMC (95%) k=2 [1]	2005 NIST/GA	2009 NIST/ MCMC (95%) k=2 [1]
	Al_2O_3	*1.23*	*0.89 to 1.43*	*2.779*	*2.97 to 3.66*
	GaAs	*9.05*	*8.87 to 9.60*	*8.457*	*7.88 to 8.51*
3	**AlAs**	**9.44**	9.35 to 9.64	**9.480**	9.09 to 9.69
4	**GaAs**	**9.27**	9.13 to 9.40	**9.307**	9.01 to 9.45
5	**AlAs**	**9.43**	9.33 to 9.63	**9.464**	9.24 to 9.69
6	**GaAs**	**9.26**	9.12 to 9.40	**9.303**	9.10 to 9.60
7	**AlAs**	**9.44**	*9.31 to 9.61*	**9.466**	*9.20 to 9.78*
	GaAs	-	-	-	-

Conclusions (from 2011 NIST/NMIJ study)

The buried (Layers 3-7) show stability over many years

Uncertainty analysis showed expanded uncertainties of < 0.3 nm for Layers 3-7.

NIST XRR Measurement Alignment

Goal

Develop a method to calibrate thickness and density measurement from a given XRR instrument

Step 1

Measure XRR from an "aligned" Reference Material

Align using methods provided by instrument vendor

Measure the sample several times and with several re-mountings

Certified Reference Materials are available from NMIJ and are under development at NIST

Step 2

Analyze XRR using commercially available Genetic Algorithm refinement

Very fast at providing a solution that fits the data well

Only finds a best fit, so it cannot provide detailed information about uncertainties

Available from most commercial instrument vendors and also open source

Step 3

Intentionally shift θ data from an aligned XRR measurement

θ is the sample angle from the source (typically the detector moves at 2θ)

Shifting is easily accomplished by adding a fixed value, γ, to the θ column in excel or equiv.

Shifts of +/- 0.025° are sufficient to cover alignment range (~ 7 γ shifts is sufficient)

Step 4

Compare density and thickness refinements with reference values

Goodness of Fit provides too slow of a response to determine optimal alignment

Thickness increases linearly with sample tilt for buried layers in a multilayer stack

Density decreases linearly with sample tilt for buried layers in a multilayer stack

Certified thickness or density define the aligned condition for an instrument

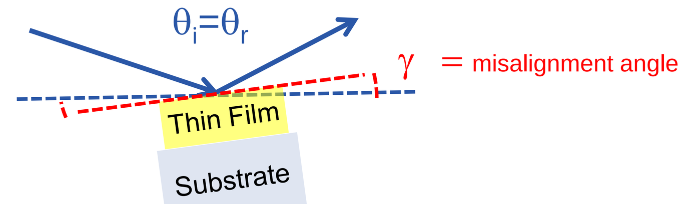
Step 5

Determine instrument repeatability

Repeat steps 3 and 4 for multiple measurements and multiple mountings

Compare results over time, and over different instrument conditions (ex. lab temperature)

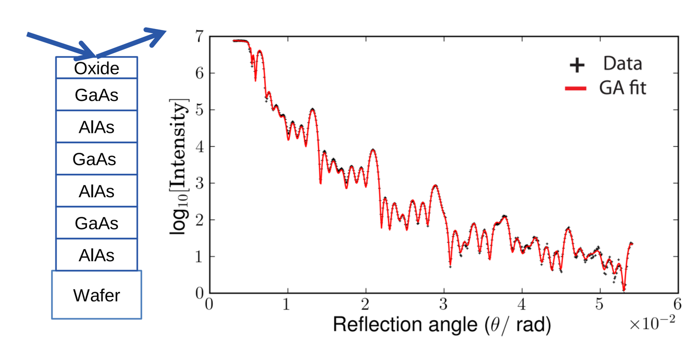
Misalignment Geometry



NIST - NMIJ GA analysis for aligned and misaligned XRR data

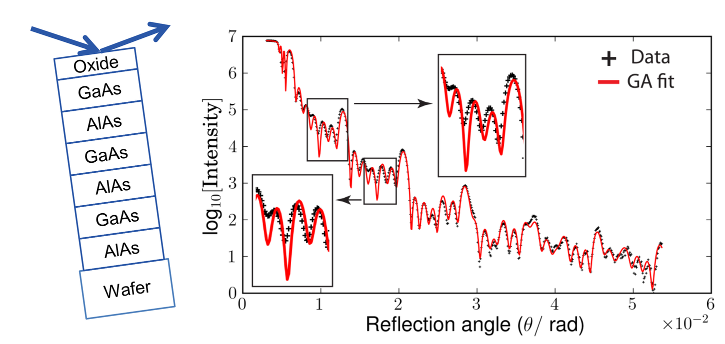
Aligned data refinement

Aligned data will catch all oscillation features provided the appropriate structural model is used



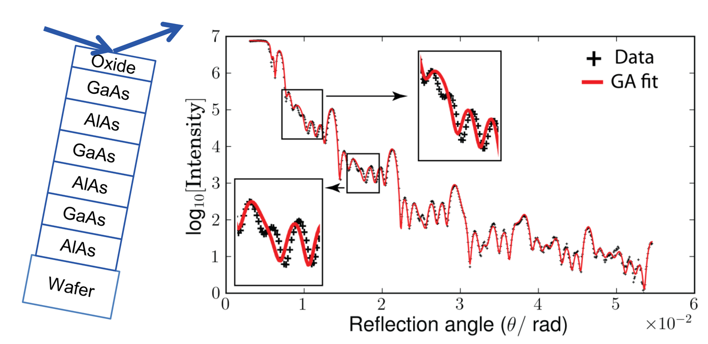
Misaligned (-0.025°) data refinement

Misaligned data will introduce distortions in roughness and density making unusual valleys



Misaligned (+0.025°) data refinement

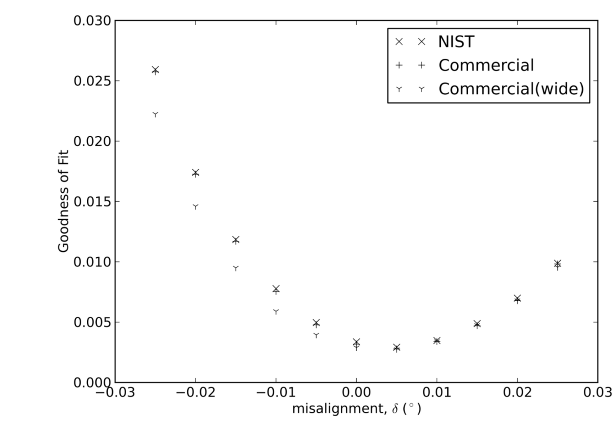
Refining misaligned data will often cause the model to skip or insert oscillations which reduces GOF



Goodness of Fit

Goodness of Fit does not provide enough information for alignment better than γ < ± 0.01°

Goodness of Fit fails to give the direction of misalignment, due to its parabolic response

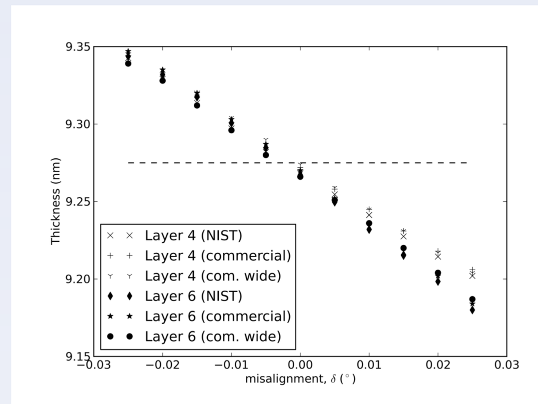


Thickness variation with misalignment

GaAs thickness misalignment calibration

High linearity for shifts in thickness from +/- 0.02° in θ

Only is linear for buried interfaces



GaAs buried layers: Oxide, GaAs, AlAs, GaAs (4), AlAs, GaAs (6), AlAs, GaAs Wafer

GaAs thickness calibration linearity

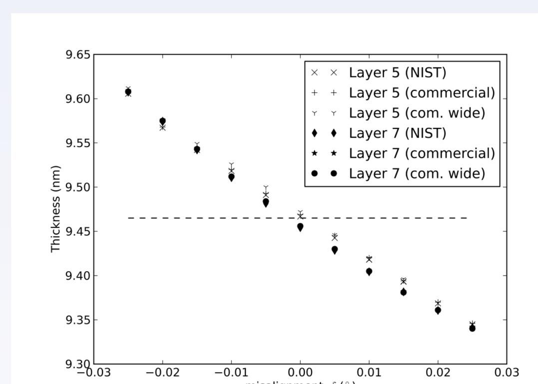
Both GaAs layers have a slope correction of ~3 nm/° (for this data set)

	Structure	Reference value (nm)	Intercept Thickness (nm)	Slope (thickness / ° misalignment)	R^2 (A measure of fit quality)
	Al_2O_3		Na		
	GaAs		Na		
	AlAs	9.44	9.476	-4.846x	.998
4	**GaAs**	**9.27**	**9.272**	**-2.845x**	**0.9956**
	AlAs	9.43	9.469	-4.990x	0.999
6	**GaAs**	**9.26**	**9.267**	**-3.3106x**	**0.9974**
	AlAs	9.44	9.461	-5.193x	0.9939
	GaAs		Na	Na	

AlAs thickness misalignment calibration

High linearity for shifts in thickness from +/- 0.02° in θ

Only is linear for buried interfaces



AlAs buried layers: Oxide, GaAs, AlAs, GaAs, AlAs (5), GaAs, AlAs (7), GaAs Wafer

AlAs thickness calibration linearity

All AlAs layers have a slope correction of ~5 nm/° (for this data set)

	Structure	Reference value (nm)	Intercept Thickness (nm)	Slope (thickness / ° misalignment)	R^2 (A measure of fit quality)
	Al_2O_3		NA		
	GaAs		NA		
	AlAs	9.44	9.476	-4.846x	.998
	GaAs	9.27	9.272	-2.845x	0.9956
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7	**AlAs**	**9.44**	**9.461**	**-5.193x**	**0.9939**
	GaAs		Na	Na	

Thickness variation conclusions

Both AlAs and GaAs buried layer thickness provide both direction of misalignment and good linearity for calibration

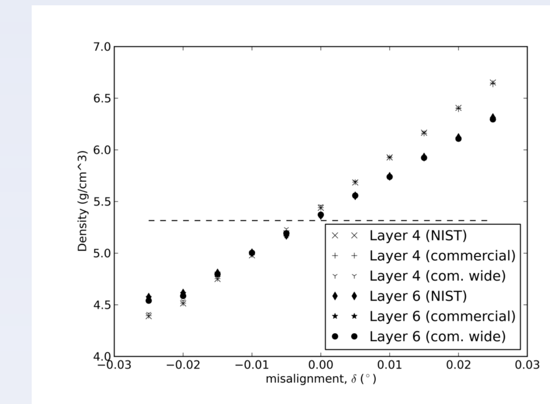
High variability in the slopes between AlAs and GaAs layers is a consequence of fringe "beating" and may adversely effect results if layers "swap" inside the model (which can sometimes happen in multilayer fitting)

Density variation with misalignment

GaAs density misalignment calibration

High linearity for shifts in density from +/- 0.015° in θ

Only works for buried interfaces



GaAs buried layers: Oxide, GaAs, AlAs, GaAs (4), AlAs, GaAs (6), AlAs, GaAs Wafer

GaAs density calibration linearity

GaAs layers have both high linearity and stability in density correction factors

	Structure	Bulk Density	Intercept Density (g/cm3)	Slope (density / ° misalignment)	R^2 (A measure of fit quality)
	Al_2O_3		Na		
	GaAs	5.316	Na		
	AlAs	3.81	3.807	31.555x	0.8905
4	**GaAs**	**5.316**	**5.455**	**47.244x**	**0.9997**
	AlAs	3.81	3.680	38.052x	0.9995
6	**GaAs**	**5.316**	**5.367**	**37.529x**	**0.9998**
	AlAs	3.81	3.633	19.014x	0.9894
	GaAs		Na	Na	Fixed

AlAs density misalignment calibration

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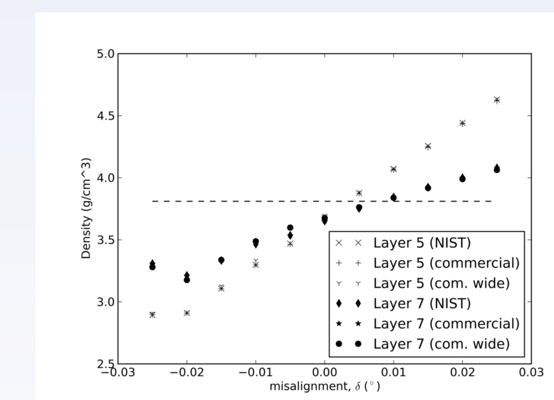

AlAs buried layers: Oxide, GaAs, AlAs, GaAs, AlAs (5), GaAs, AlAs (7), GaAs Wafer

AlAs density calibration linearity

AlAs layers have a large range of slope correction factors

	Structure	Bulk Density	Intercept Density (g/cm3)	Slope (density / ° misalignment)	R^2 (A measure of fit quality)
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	GaAs	5.316	Na		
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	GaAs		Na	Na	Fixed

Density variation conclusions

Both AlAs and GaAs buried layer density provide both direction of misalignment and good linearity for calibration

Careful selection of the layer used for density calibration is essential given the narrower range for linearity

High slope changes (~ 10 times higher than thickness) show the significant impact that slight misalignment has on density determination using XRR

Production of pre-standard reference materials courtesy of Victor Vartanian (victor.vartanian@SEMATECH.org)

MCMC & GA XRR modeling software courtesy of David L Gil (david.gil@CORUSCAVI.com)

GA XRR modeling software courtesy of Matthew Wormington (mattheww@JVSEMI.com)

[1]Standard uncertainty derived from statistical methods (Type A). Standard uncertainty a priori derived (Type B) (see Taylor NIST TN 1297)

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