The role of AIST/NMIJ Certified Reference Materials in X-ray reflectometry metrology

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Presentation Outline

NIST / NMIJ collaboration motivation

NMIJ XRR CRM stability studies

NMIJ XRR CRM for sample alignment

NMIJ XRR CRM for
metrology of XRR uncertainty analysis

NIST/AIST
Collaboration outputs

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SRM Development Difficulty

Why NIST cannot produce SRMs for all measurement needs

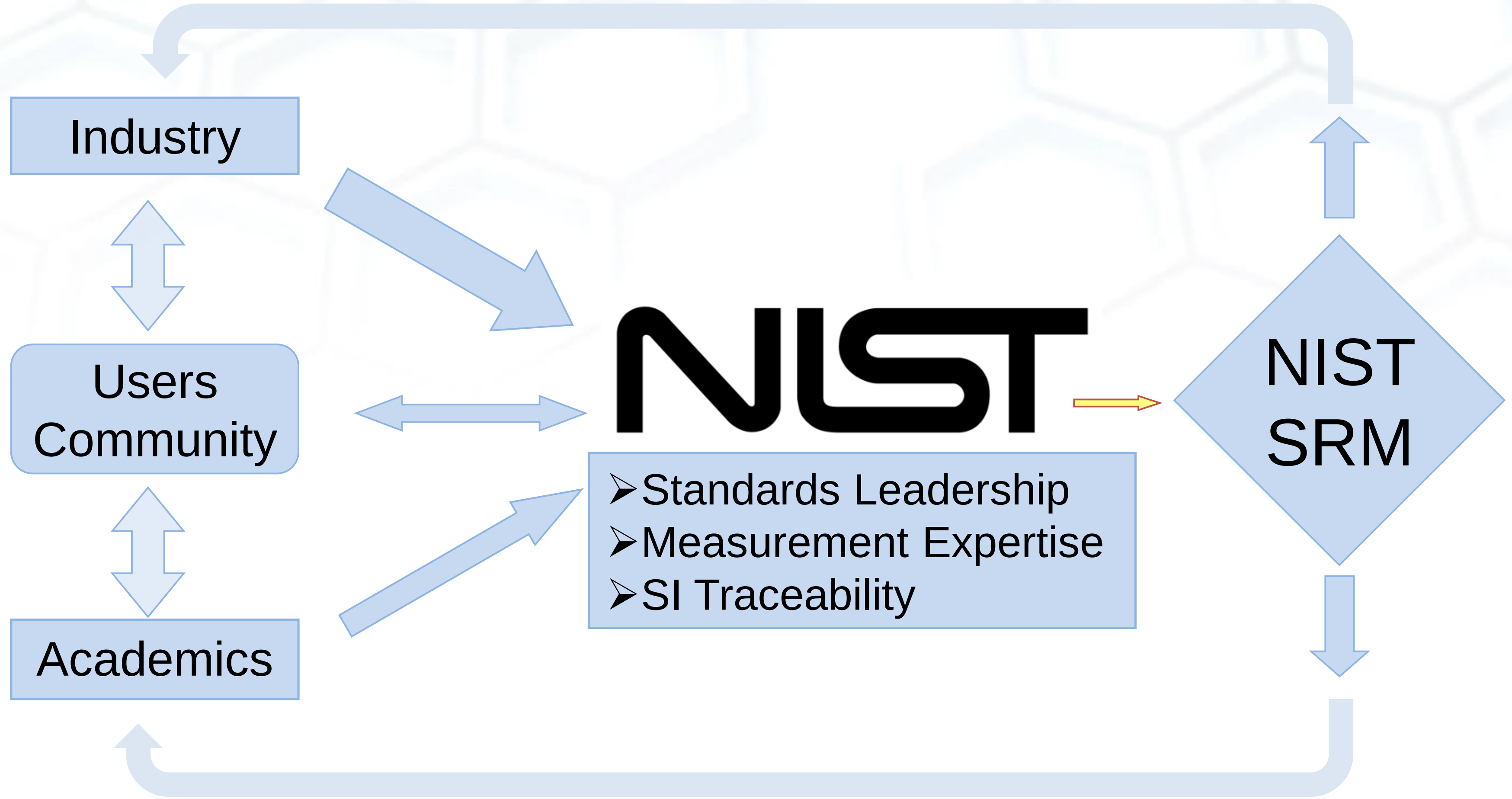
Time (Years)	Requirements for SRM Development: SI-traceable NIST Instrument	Requirements for SRM Development: Metrological First Principles Analysis Method	NIST Cost*: Labor (1 FTE is $250k)	NIST Cost*: Equipment (commercial instr. cost)
10 (+)	Necessary	Necessary	3 - 10	3-5 times
5	Necessary	Available	2 - 5	3-5 times
2 - 3	Available	Necessary	2 - 5	na
1 - 2	Available	Available	1-2	na

Assuming a stable, high uniformity material is Available for use as SRM feedstock

*Only partially recovered through SRM sales

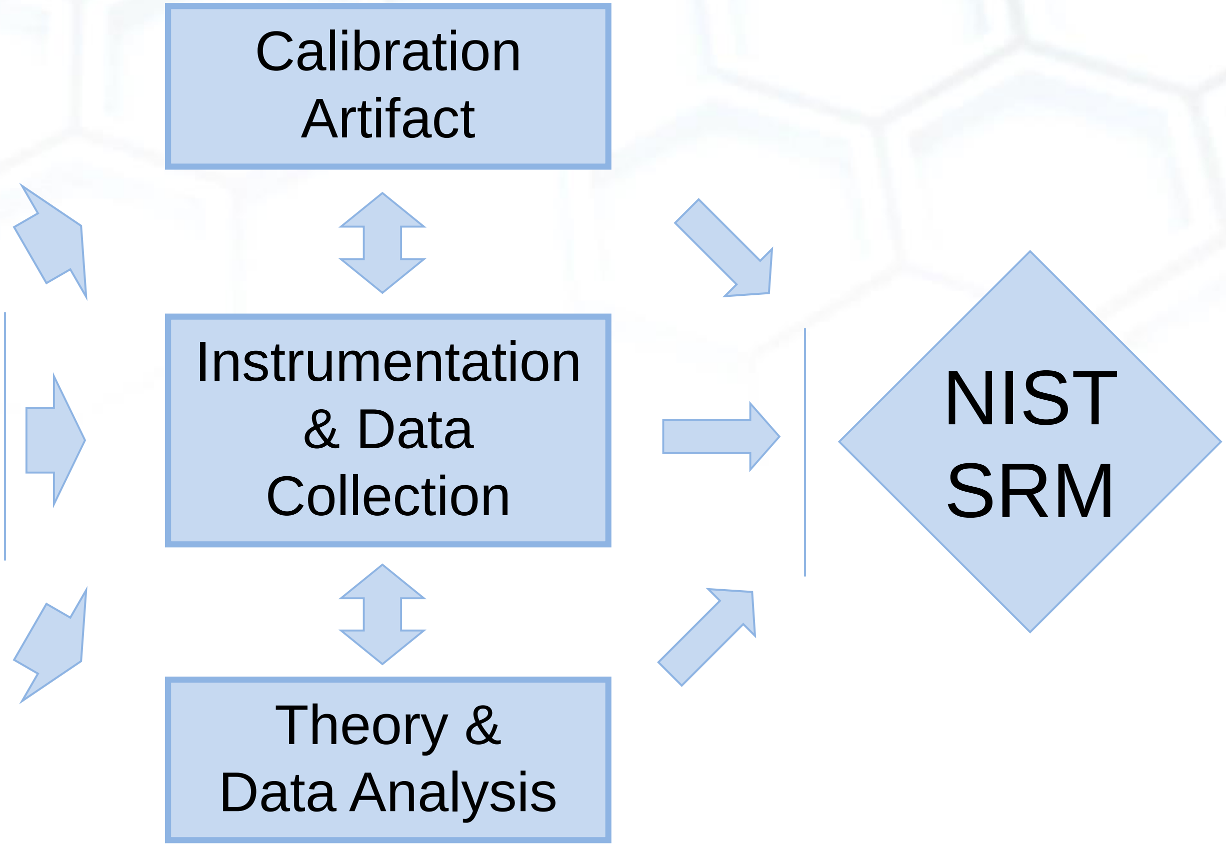
NIST - Community Interaction

NIST's transfer of calibration capability to customers by SRMs



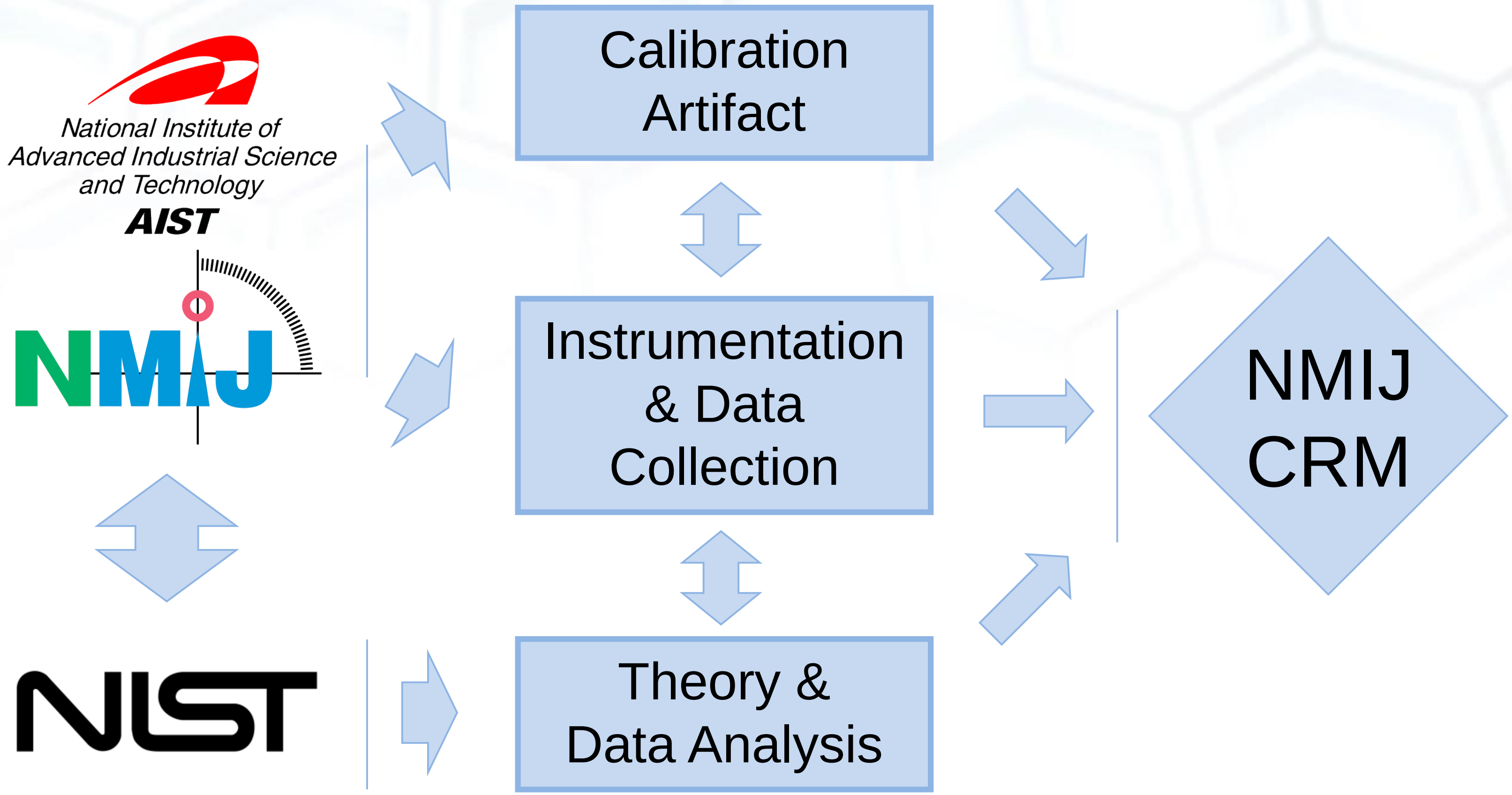
SRM Development

Elements of an SRM



NIST – NMIJ/AIST Collaboration

Providing XRR metrology to the international community



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XRR CRM structure

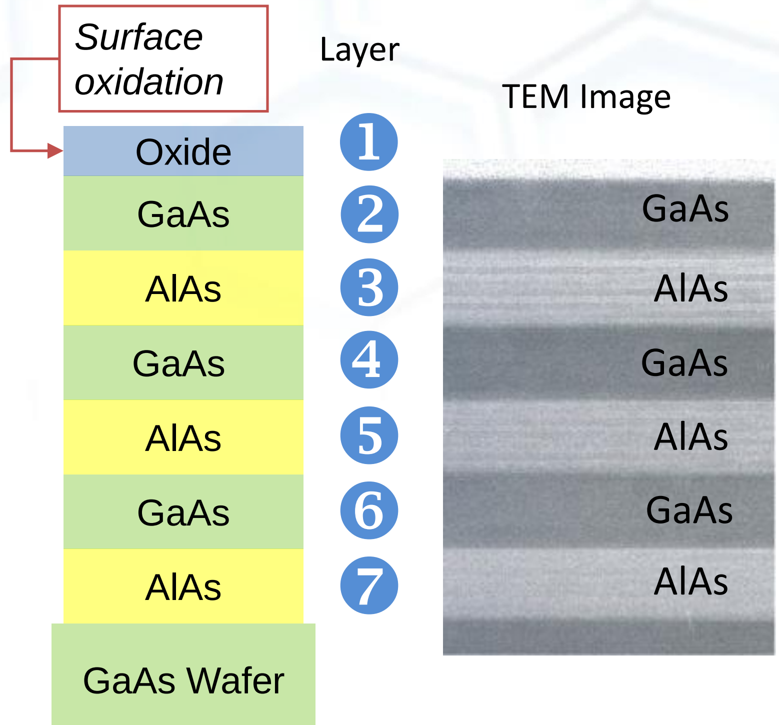
As deposited model

NMIJ

National Institute of Advanced Industrial Science and Technology AIST

NMIJ CRM

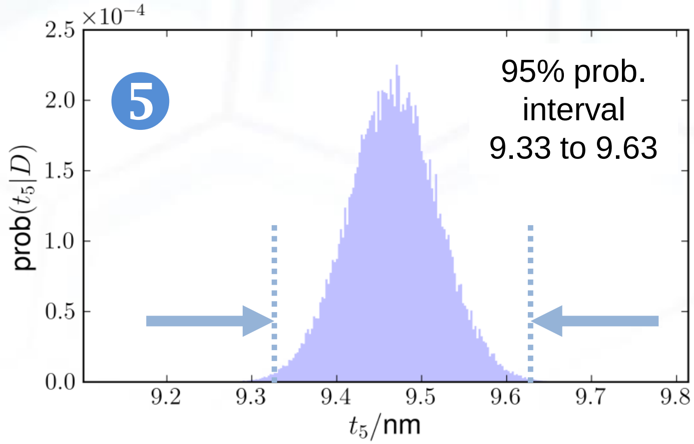
MBE epitaxial deposition of AlAs/GaAs produces high quality, stoichometric, multilayer with smooth interfaces and low inter-diffusion.

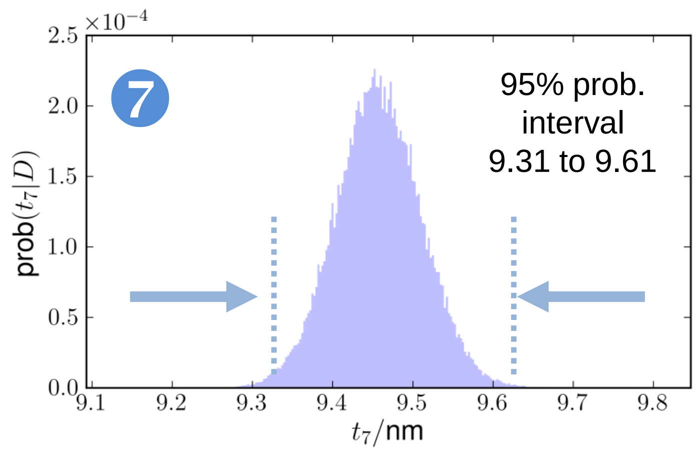


AlAs Thickness Stability

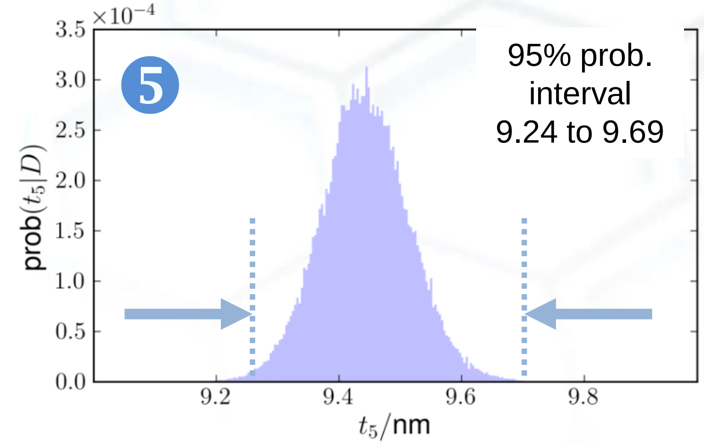
5 year comparison of thickness

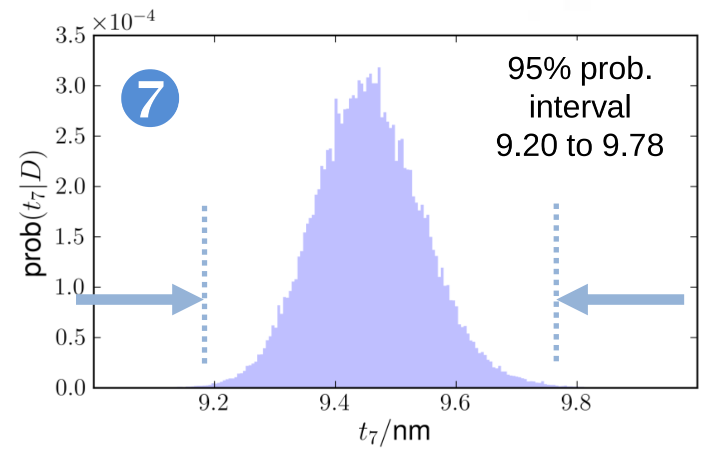
MCMC of 2004 XRR data





MCMC of 2009 XRR data





XRR CRM Thickness Stability

5 year comparison of thickness

	2004		2009	
Structure (thickness in nm)	**NMIJ/GA[5]**	**NIST/MCMC (95%) k=2 [6]**	**NIST/GA**	**NIST/ MCMC (95%) k=2 [6]**
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*
AlAs	**9.44**	**9.35 to 9.64**	**9.480**	**9.09 to 9.69**
GaAs	**9.27**	**9.13 to 9.40**	**9.307**	**9.01 to 9.45**
AlAs	**9.43**	**9.33 to 9.63**	**9.464**	**9.24 to 9.69**
GaAs	**9.26**	**9.12 to 9.40**	**9.303**	**9.10 to 9.60**
AlAs	**9.44**	***9.31 to 9.61***	**9.466**	***9.20 to 9.78***
GaAs	--	--	--	--

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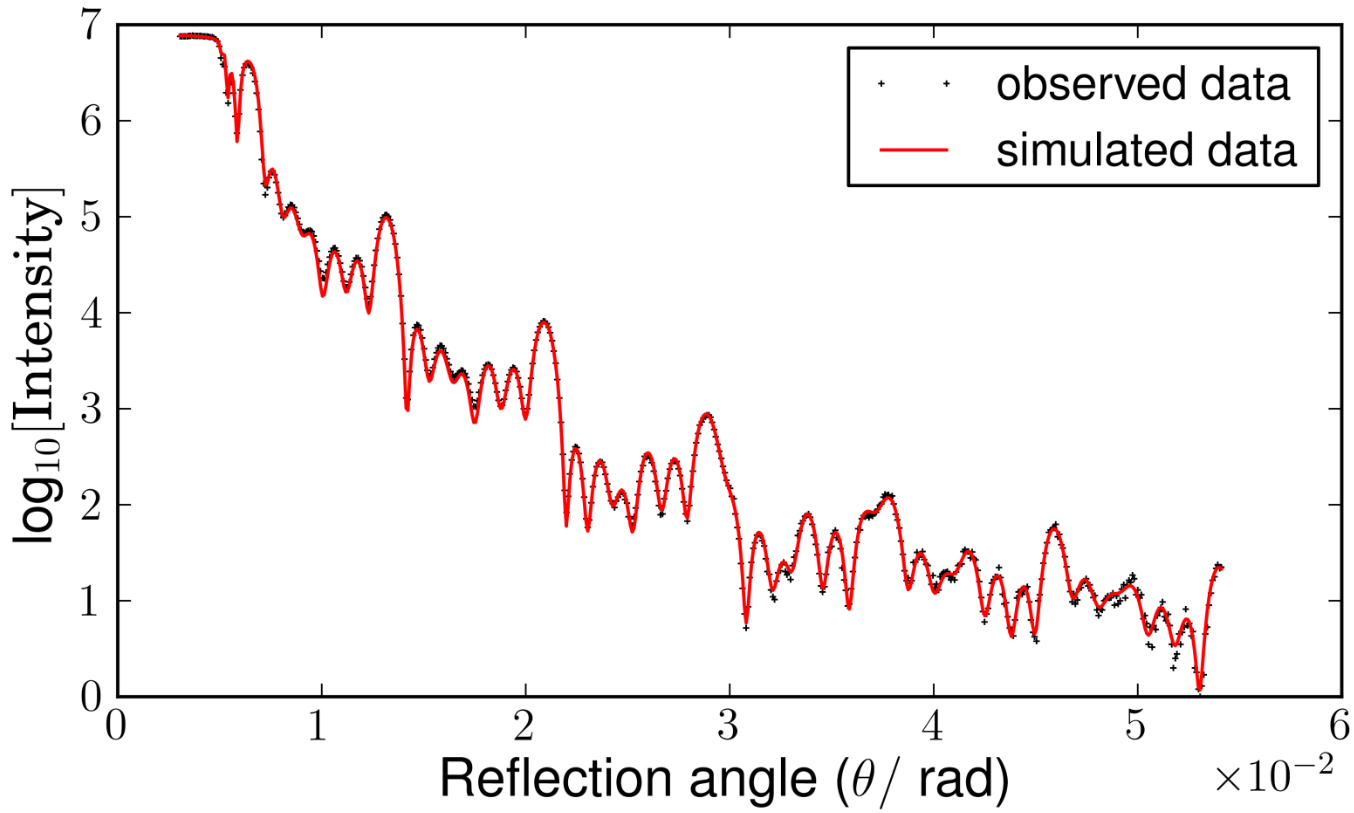
XRR Data and Fit: Aligned

2004 NMIJ data GA refinement

NMIJ Results

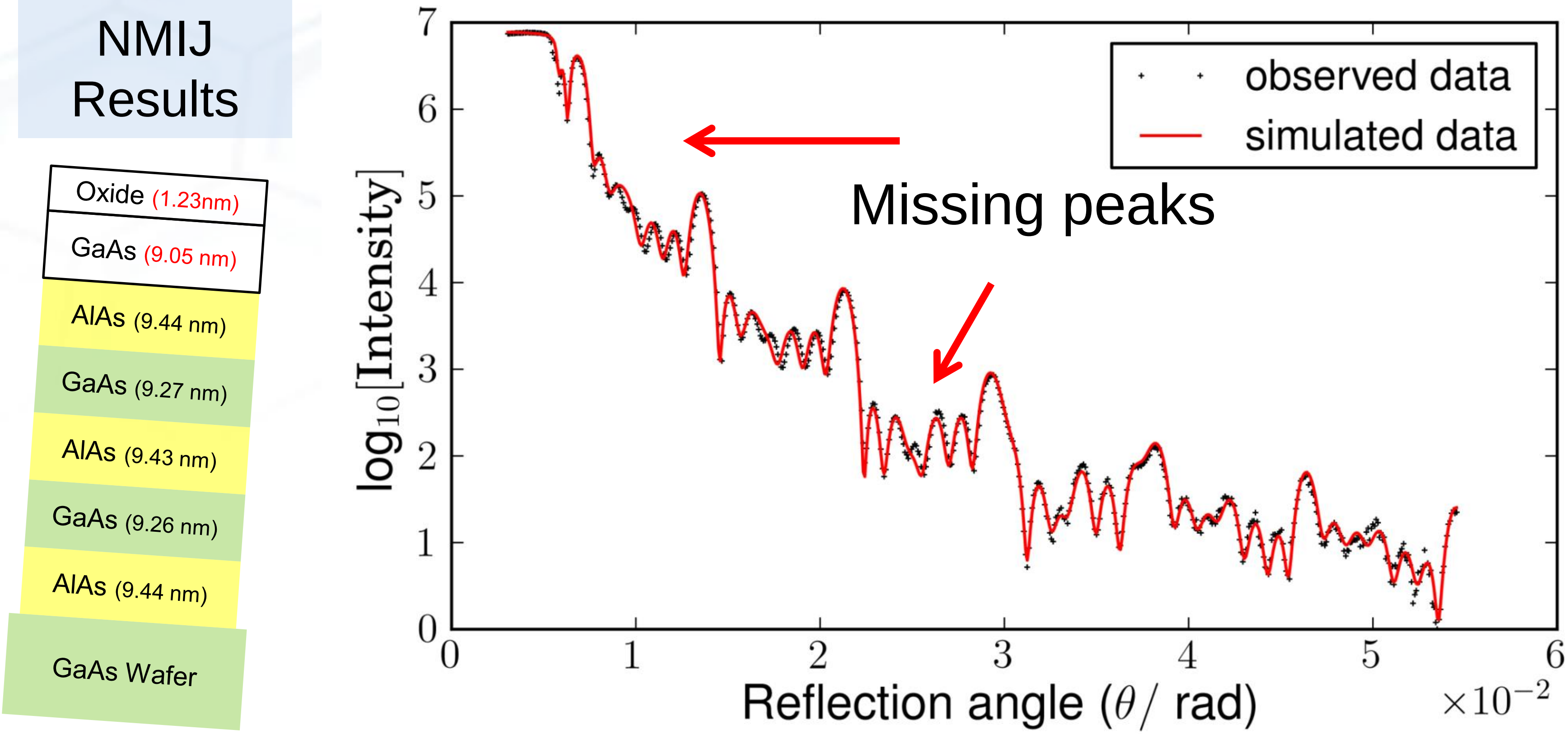
Oxide (1.23nm)
GaAs (9.05 nm)
AlAs (9.44 nm)
GaAs (9.27 nm)
AlAs (9.43 nm)
GaAs (9.26 nm)
AlAs (9.44 nm)
GaAs Wafer



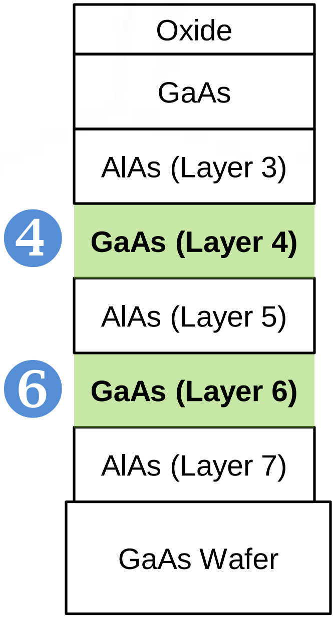
XRR Data and Fit: γ of + 0.025°

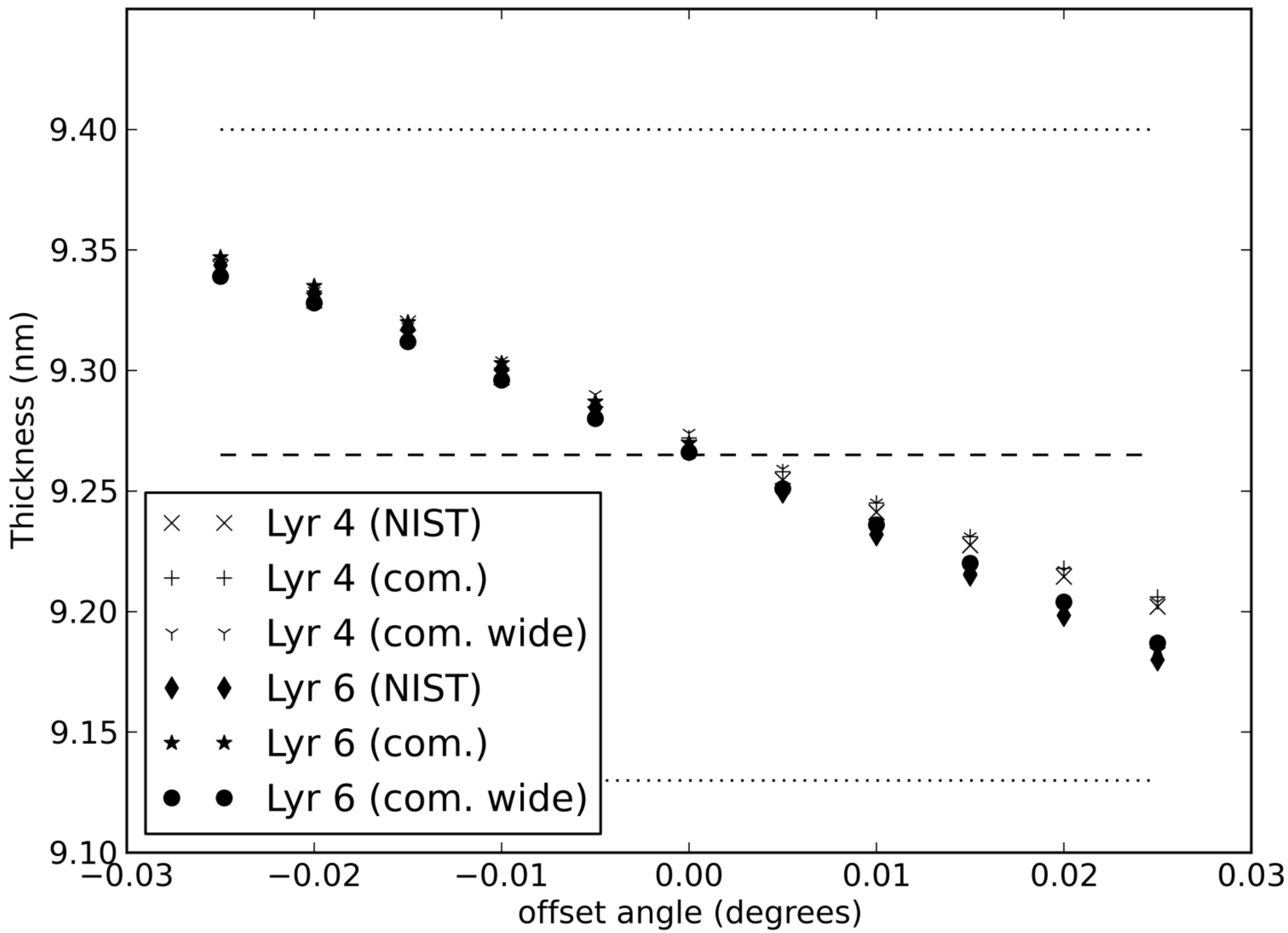
2004 NMIJ data GA refinement

NMIJ Results

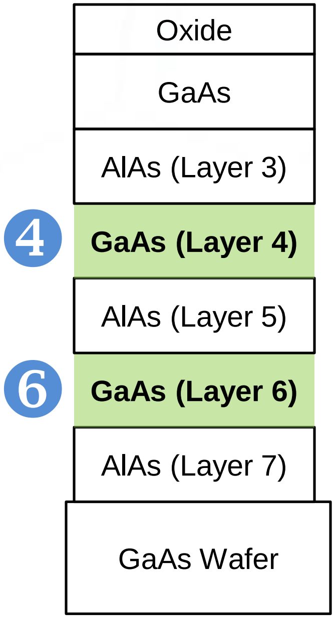


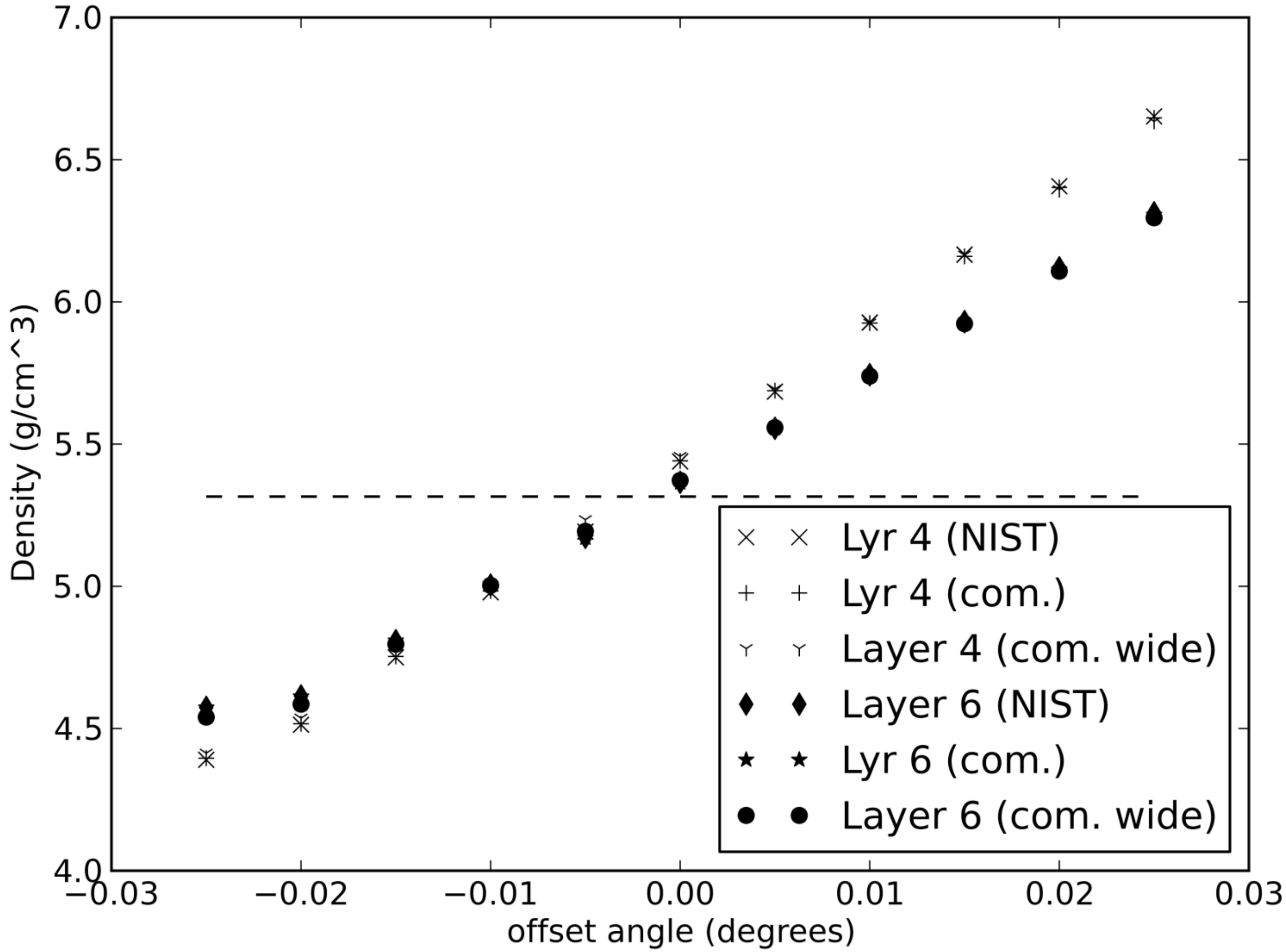
XRR Modeling: GaAs Thickness





XRR Modeling: GaAs Density





Conclusions

Goodness of Fit does not provide enough information for alignment $\gamma < \pm 0.01$ degrees

Increase in reflection angle (via misalignment) increases density and decreases thickness (as predicted)

If density and/or thickness parameters are known (certified), then either can be used for sample alignment

Density and Thickness provide alignment $\gamma < \pm 0.005$ degrees

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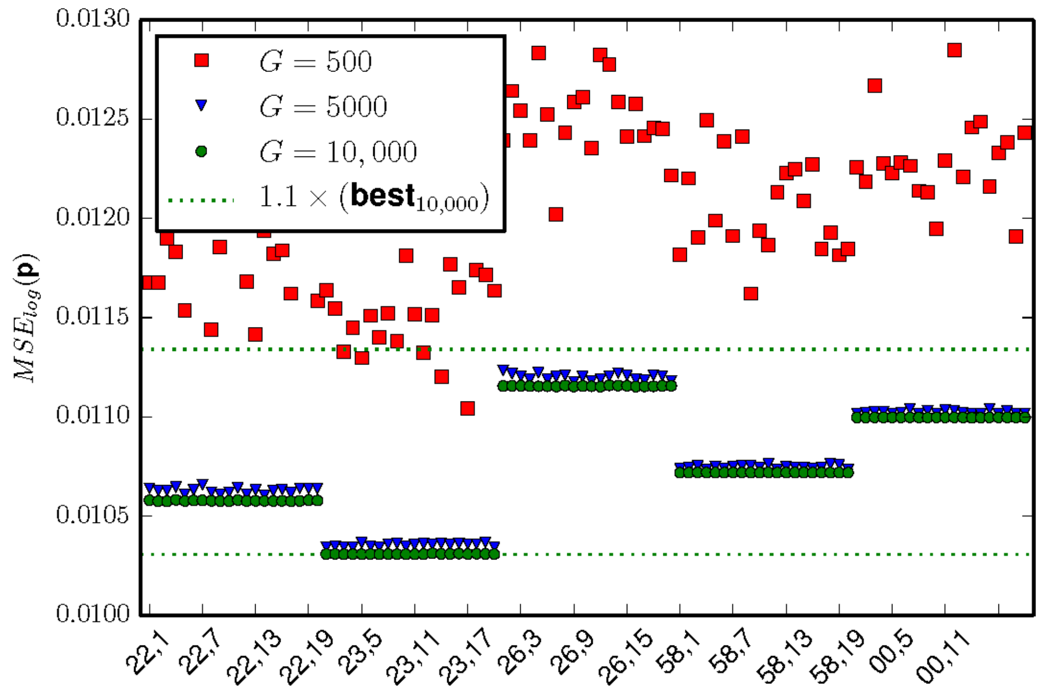
XRR uncertainty metrology using NMIJ CRM

Goodness of fit

The five different spreads of values represent best fits for 5 XRR data sets on the same sample and same alignment.

Green bars represent expanded uncertainty of parameter over the measurement set and Represents refinement variation introduced by measurement noise.

Red squares = 500 generations
Blue triangle = 5,000 generations
Green circle = 10,000 generations



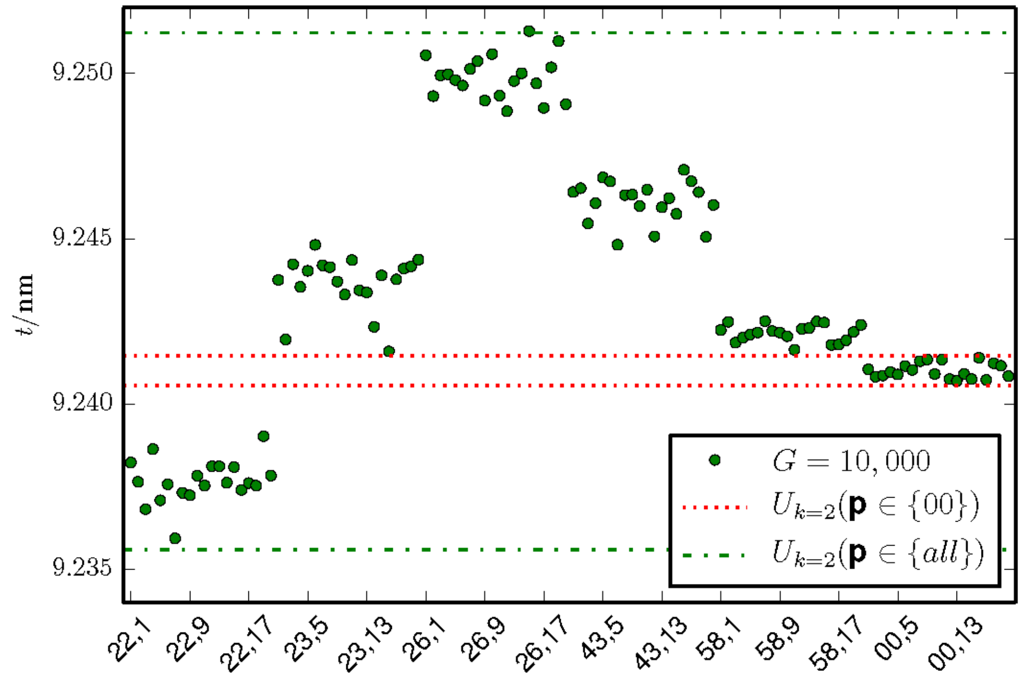
XRR uncertainty metrology using NMIJ CRM

Thickness of GaAs (layer 6)

The five different spreads of values represent best fits for 5 XRR data sets on the same sample and same alignment.

Green bars represent expanded uncertainty of parameter over the measurement set

Red bars represent expanded uncertainty of parameter for a single measurement



XRR uncertainty metrology using NMIJ CRM

Relative precision over various measurement strategies

Layer	Sealed tube				Rotating anode			
	One, 8hr set		Six, 8 hr sets		Ten, 15 min sets		Three, Six hour sets	
	30 s (00)		30 s set		1 s set		20 s set	
	<t> /nm	U (t_{00})	U(t_{30s})	U(t_{30s}) / U (t_{00})	U(t_{1s})	U(t_{1s}) / U (t_{00})	U(t_{20s})	U(t_{20s}) / U (t_{00})
Ga1	8.298	0.001	0.014	18	0.011	14	0.004	5
Al 1	9.496	0.001	0.016	21	0.024	32	0.01	14
Ga 2	9.27	0.001	0.009	15	0.009	14	0.011	17
Al 2	9.486	0.001	0.011	21	0.015	30	0.005	10
Ga 3	9.241	0.0	0.01	22	0.013	30	0.008	19
Al 3	9.462	0.001	0.012	14	0.019	23	0.004	5
<>	-	-	-	19	-	23	-	11

Conclusions

Differential Evolutions can estimate uncertainties for measurement-to-measurement bias. However, to perform this type of statistical analysis from an optimization method you must:

Measure repeated measurements
(~ 10 identical XRR measurements)

Run multiple DEs on each XRR measurement
(~ 10-20 DEs) for, m > 10 x d & G > 5,000)

Find refinement uncertainty (using narrow parameter ranges) and a 1.1x best DE selection

Estimate uncertainties using results from DEs from multiple measurements and 1.1x best set

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NIST / NMIJ Key Outputs

Posters

X-Ray Reflectometry Parameter Uncertainties for Thin Films
D.L. Gil, D. Windover, J.P. Cline, Y. Azuma, and T. Fujimoto
Presented at Frontiers of Characterization and Metrology for Nanoelectronics, Grenoble, France, 2011

Sample Alignment of X-Ray Reflectometry Using Thickness and Density From Certified Reference Materials
D. Windover, Y. Azuma, D.L. Gil, and T. Fujimoto
Presented at Frontiers of Characterization and Metrology for Nanoelectronics, Gaithersburg, MD, 2013

Papers

Determining sample alignment in x-ray reflectometry using thickness and density from GaAs/AlAs multilayer certified reference materials
D. Windover, D.L. Gil, Y. Azuma, and T. Fujimoto, Meas. Sci. Technol. 25 (2014) 105007

Reproducible x-ray reflectometry optimization: statistical analysis of differential evolution fitting of multilayer structural models
D. Windover, D.L. Gil, Y. Azuma, and T. Fujimoto, Submitted for review


thank you
danke
謝謝
ngiyabonga
teşekkür ederim
gracias
merci
obrigado
dziękuję
bedankt
спасибо
Баярлалаа
рахмат
go raibh maith agat
mochchakkeram
sukriya
kop khun krap
terima kasih
arigatô
grazie
dakujem
мерси
tapadh leat
dank je
takk
감사합니다
ありがとう
ευχαριστώ
hvala
mauruuru
enkosi
faafetai lava
blagodaram
misaotra
paldies
хвала
asante
sagolun
taiku
tanemirt
rahmet
diolch
dhanyavadagalu
shukriya
xiexie
kam sah hamnida
najis tuke
didi madloba
trugarez