Thin Film and Ultra-thin Film Metrology

GI-XRF Implementation

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Motivation – Thin film thickness & composition monitoring

Thin films, especially ultra-thin films, are ubiquitous in semiconductor devices and nano devices.

Ultra thin film examples

- **MRAM: 10 Å to several hundreds Å**
- **Liners and capping layers: $\leq$ 20 Å**
- **High-K and metal gate: 10 Å to 30 Å**

Desirable thickness and composition monitoring metrology capabilities

Thickness sensitivity – *several μm to a couple Å (mono atomic layer!)*

Good precision - *1s relative $\leq$ 1% or better*

Desirable to have composition analysis capability

Small spot size for wafer edge characterization – < 3mm

Good throughput - *Less than 10 seconds per data point desirable.*

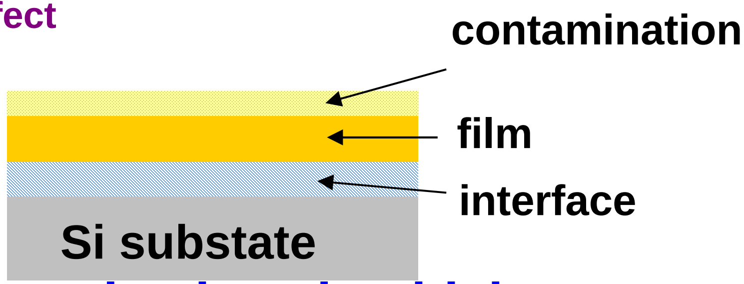
Motivation - Limitations of common metrology tools

Limitations of ellipsometry

Ultra-thin film → t, n and k cannot independently determined

Ultra-thin film → large interface and contamination effect

Thickness limitation for opaque films ≤ 500 Å



Limitations of XPS for ultra-thin film – conceived as the thickness metrology tool for ultra-thin film =>

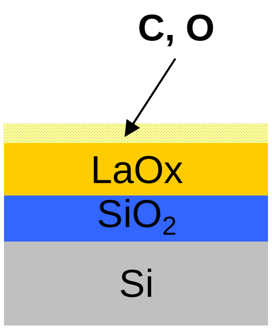
Only for thickness ≤ 100 Å

Density of the film influence thickness results

Signal exponential dependent on thickness (*$I \propto exp(- t/\lambda * cos\theta)$*)

- *Surface contamination contribution is heavily weighted*
- *difficult to quantify due to oxidation or other surface reactions*

Ion beam depth profiling alters surface composition by intermixing and preferential sputtering effect.



Limitations of ultra-sound based tool

Thickness ≥ 30 Å to 50 Å

Not for dielectrics

Pros and Cons of X-ray Fluorescence (XRF) for Thin Film Metrology

Pros:

1st order approximation, XRF intensities proportional to mass thicknesses (density * thickness) of the thin film – easy for quantification

- easy calibration of XRF intensities for thickness and composition measurement
- Matrix effects, which include primary X-ray beam attenuation, fluorescence X-ray absorption and fluorescence enhancement, are well know and easy to calculate

Immune to the effects of surface contamination, oxidation and interface reaction.

Applicable to both metals and dielectrics, transparent and opaque materials

Large thickness range 100 µm to a couple Å

Cons:

Throughput low due to weak signals

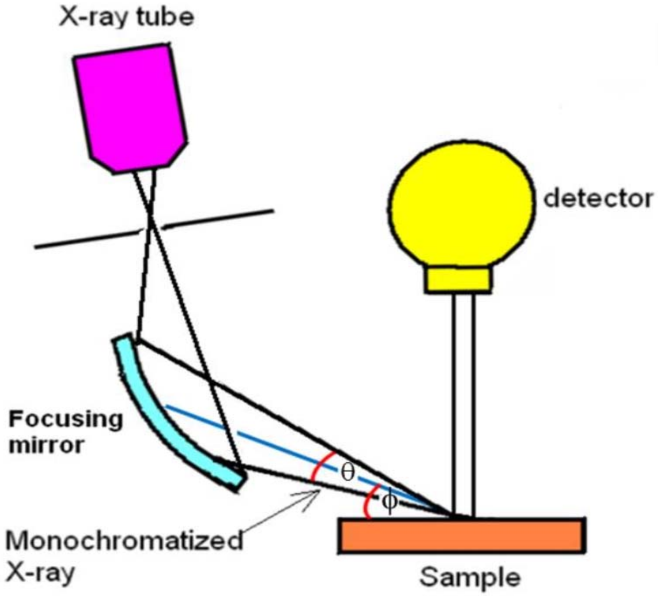
Traditional polychromatic X-ray source causes high background signals that reduce ultra-thin film measurement sensitivities.

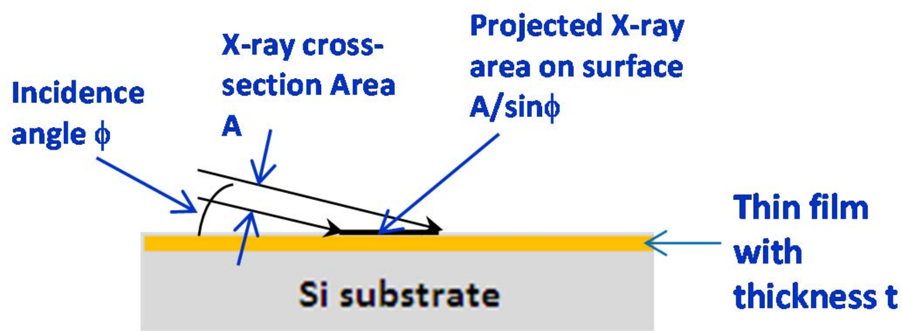
Solutions:

Grazing incidence – increase fluorescence intensity

monochromatic X-ray sources – reduce background signal

Grazing incidence XRF principles and requirements





main components in an XRF tool.

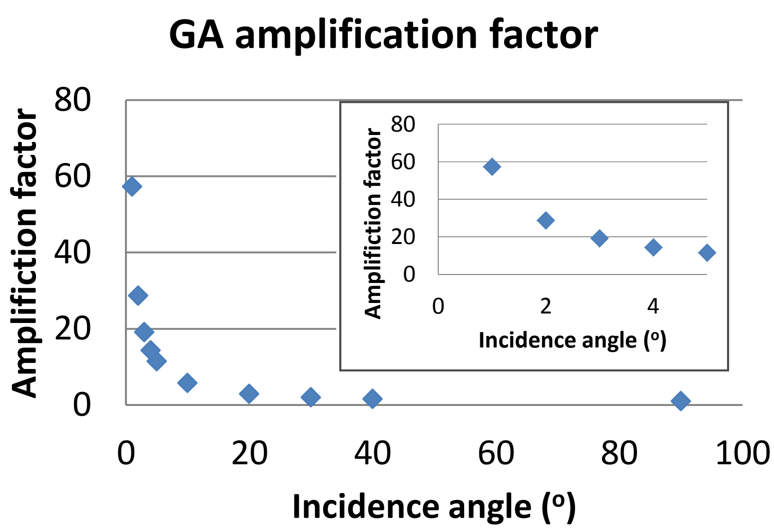
ϕ → the source X-ray grazing incidence angle.
θ → source X-ray convergent angle on sample.

Thin film → $I_i \propto I_S * C_i * t * A/\sin\phi$,
with amplification advantage **$1/\sin\phi$**.

Bulk material $I_i \propto I_S * C_i * A * L_S$,
with no amplification advantage.

I_i → fluorescence X-ray from element i
I_S → primary X-ray flux
A → primary X-ray cross-section area
Ls → attenuation length of the X-ray in the sample.

Grazing angle XRF principles and requirements



According to 1/sinϕ rule. XRF signal amplification factor as a function of the source X-ray incidence angle. The insert on the top right corner only shows data from angles less than 5°.

Table 1 - Incidence angle (IA) variation caused imprecision

Δϕ	1σ (%)						
	1°	2°	3°	4°	5°	6°	10°
1	-99.99	-49.98	-33.30	-24.96	-19.95	-16.61	-9.90
0.1	-10.00	-5.00	-3.33	-2.50	-1.99	-1.66	-0.99
0.01	-1.00	-0.50	-0.33	-0.25	-0.20	-0.17	-0.10

Local angular variation (Δϕ) of the excitation X-ray IA has to be extremely small.

For IA = 2°, Δϕ ≤ 0.01° ----> 0.5% (1σ)

Δϕ ≤ 0.01° requires

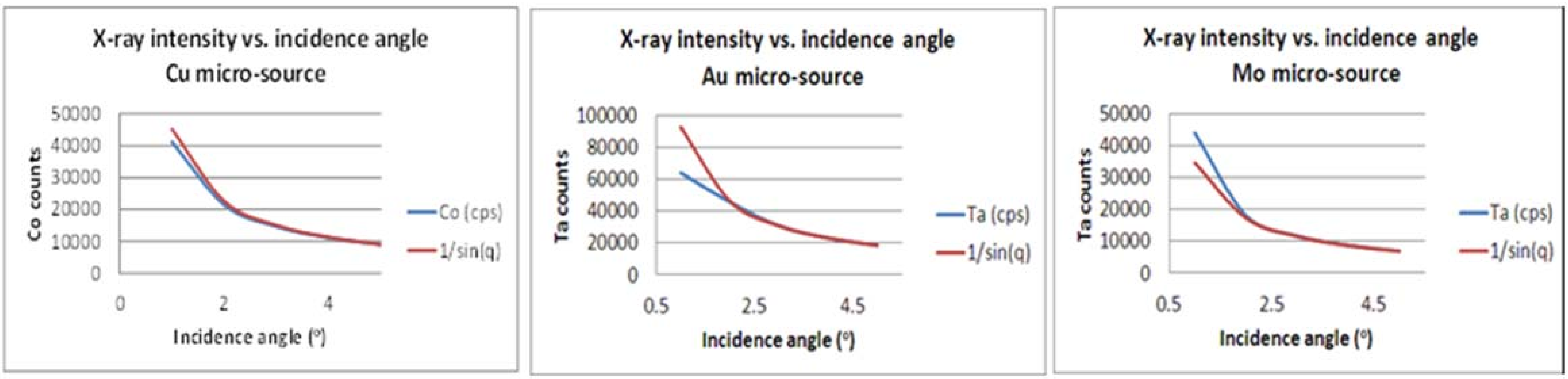
1) local height variation ≤ 0.17 um/1 mm
2) edge-to-edge height Δ ≤ 50 um for 300 mm wafer.

Implementation of GI-XRF tool

Monochromatic X-ray sources with less than 2º convergence angles

Au source for XRF	0° to 60°	**Good for Ge, Ga, Zn, Cu, Ni, Ir, W, Ta and Hf**
Cu source for XRF and XRR	0° to 40°	**Good for elements not covered by Au and Mo sources**
Mo source for XRF	0° to 20°	**Good for Y, Pb, Au, Pt, and As**

X-ray beam size 0.075 mm X 0.075/sin(ϕ)

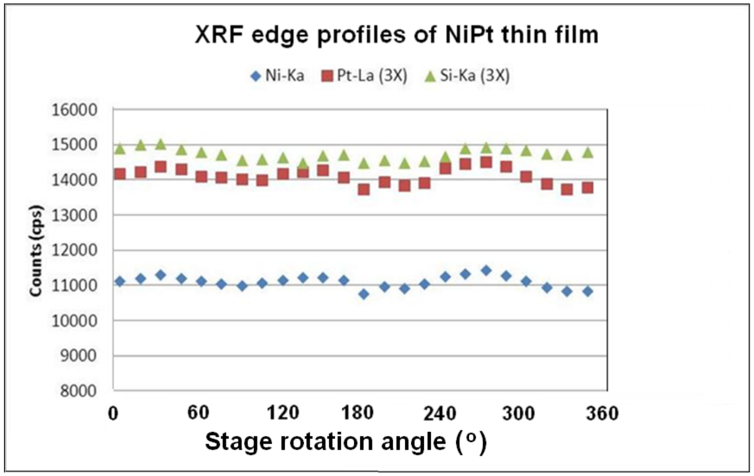


Intensities from all three X-ray sources follow 1/SIN(ϕ) down to 2º incidence angle. Therefore, the advantage of grazing incidence can be realized.

At 1º incidence angle, both Cu and Mo sources still follow 1/SIN(ϕ) closely. As for the Au source, the signal increase is less than the predication by 1/SIN(ϕ) due to more spread of the incidence angle.

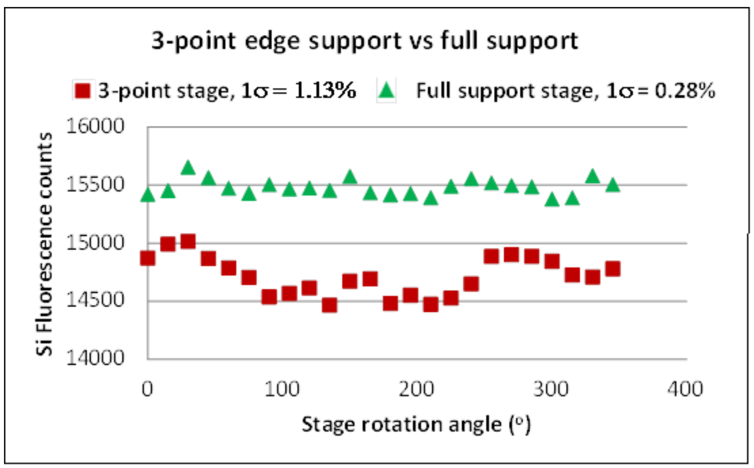
GI-XRF flat stage demonstration

GI-XRF test results of different stage design   Incidence angle = 2º



3 point edge support stage

70 µm peak to valley height variation



Full support stage

0.04 µm peak to valley height variation

A full support stage eliminates the wafer sagging problem caused by the three-point edge support stage and considerably reduces the signal fluctuation.

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Precision limitation of XRF measurement

- **X-ray intensity fluctuation obeys Poisson statistics. Precision directly related to detected X-ray counts**

N being total detected counts
n is net counts per second (cps)
b is background counts per second
T is total counting time (s)

With no background

1σ (%) = $(N)^{(1/2)}/N$,

$N = n \times T$,

with background

1σ (%) = $(N + B)^{(1/2)}/N$

$N = n \times T$, $B = b \times T$

No background

Total counts	1σ (%)
1000	3.16
10000	1.00
50000	0.45
100000	0.32
200000	0.22

Assuming counstant background counts 1000

Net counts	1σ (%)
1000	4.47
10000	1.05
50000	0.45
100000	0.39
200000	0.22

Stability of background signal affects too.

Factors affecting Intensity: 1) X-ray source power, 2) delivery optics, 3) X-ray incident angle, 4) Wavelength of X-ray source.

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Static repeatability test results

10X static repeatability test counting time (second)

Thin film (thickness)	Co (10 Å)	Ru (100 Å)	Pt (10 Å)	TiN (30 Å)	Ta (30 Å)	Cu (Cu/Ta) (200 Å)	Cu (75 Å)	Ru (8 Å)	Pt (3 Å)	Ta (2 Å)	Co (2 Å)
X-ray source	Cu	Cu	Mo	Cu	Au	Au	Au	Cu	Mo	Au	Cu
counting time	2	10	20	10	10	10	5	20	20	20	10

10X static repeatability test data (STDEV (%))

MEA #	Co (10 Å)	Ru (100 Å)	Pt (10 Å)	TiN (30 Å)	Ta (30 Å)	Cu (Cu/Ta) (200 Å)	Cu (75 Å)	Ru (8 Å)	Pt (3 Å)	Ta (2 Å)	Co (2 Å)
1	0.80	0.53	0.24	0.49	0.51	0.23	0.32	0.77	0.97	0.83	0.60
2	0.66	0.33	0.42	0.54	0.78	0.34	0.46	0.55	1.26	1.06	0.77
3	0.40	0.34	0.37	0.38	0.50	0.40	0.55	0.73	0.69	0.60	0.46
4	0.72	0.32	0.21	0.46	0.63	0.47	0.52				
5	0.52	0.31	0.21	0.63	0.71	0.35	0.64				
6	0.31	0.38	0.25	0.34	0.48	0.44	0.76				
average	0.57	0.37	0.28	0.47	0.60	0.37	0.54	0.68	0.97	0.83	0.61
STDEV	0.19	0.08	0.09	0.10	0.13	0.09	0.15	0.12	0.29	0.23	0.16
Counting statistics (1σ)	0.64	0.30	0.27	0.34	0.32	0.19	0.41	0.78	0.67	0.61	0.62
Experiment/theory	0.9	1.2	1.0	1.4	1.9	2.0	1.3	0.9	1.4	1.4	1.0

1s itself has a probability distribution:

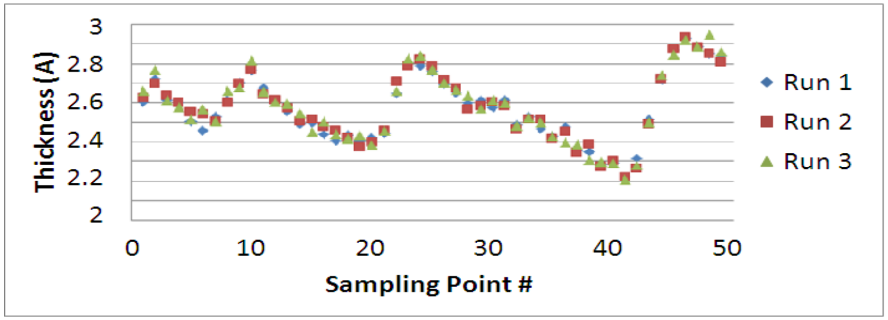
$$\sigma^2(\sigma) = \frac{1}{N}\left[N - 1 - \frac{2\,\Gamma^2\left(\frac{N}{2}\right)}{\Gamma^2\left(\frac{N-1}{2}\right)}\right]\sigma^2$$

	10X	20X
$\sigma(\sigma) =$	0.22σ	0.16σ

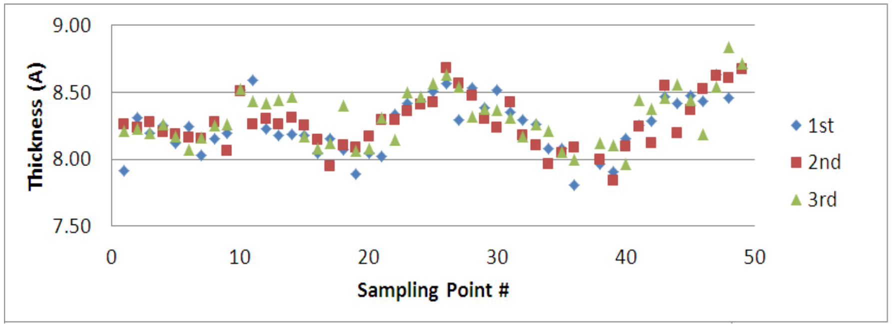
Majority of the experimental 1σ are close to theoretical 1σ.

49 point mapping dynamic load/unload tests

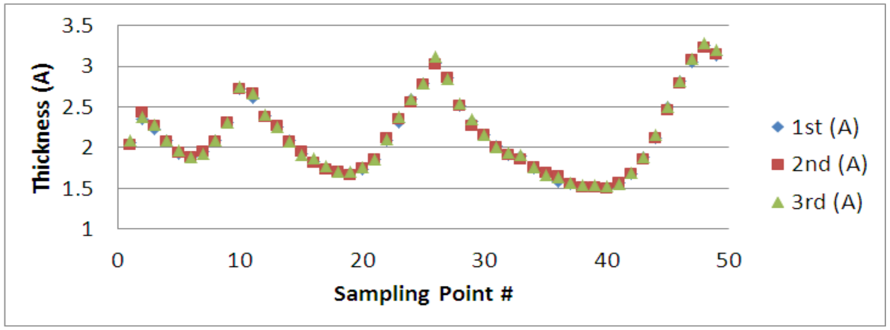
Pt 2 Å, source Mo, 2^{o} incidence angle



Ru 8 Å, source Cu, 2^{o} incidence angle



Co 2 Å source Cu, 4^{o} incidence angle



Test conditions:

1) 300 mm wafers, 2 mm edge exclusion.
2) Tests conducted on three separated days.
3) Points 1 – 25 are at center to middle radius locations. Points 26 - 49 are at the edge locations.

49 PTs Mapping dynamic load/unload data summary
Rel. STDEV (%) from 9 repeats

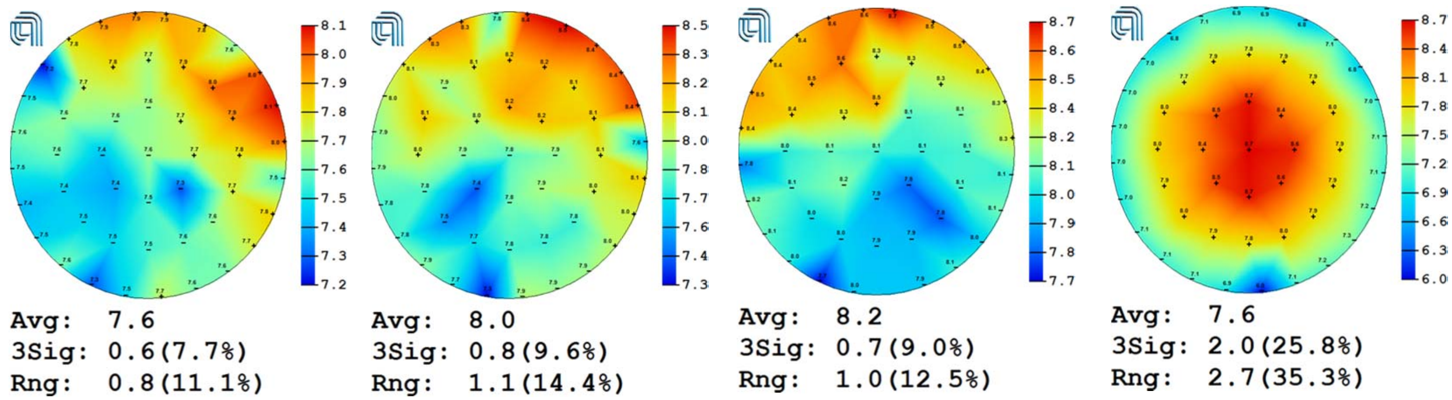
Sample	**Theoretical**	**Experimental**	**E/T**
Pt 2A	0.67	0.94	1.4
Ru 8A	0.78	1.27	1.6
Co 2A	0.62	0.90	1.5

Wafer average repeatability 1σ (%)

Pt 2 Å	0.21
Ta 2 Å	0.21
Co 2 Å	0.43
Ru 8 Å	0.35

Precision degrades slightly in the dynamic load/unload test.

49 point contour mapping of 8 Å ruthenium films



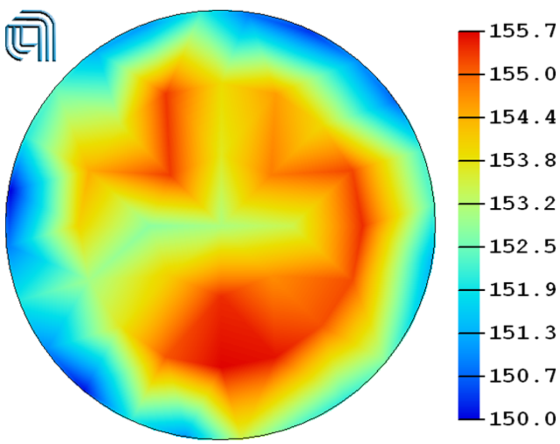
Target	Ru 7.5A	Ru 7.75A	Ru 8A	On axis Ru 8A
XRF Data	7.65	7.96	8.18	7.57
NU (3σ)	7.73%	9.59%	8.96%	25.82%

The stability of the tool allows fine tuning of thickness possible in ultra-thin film deposition.

Composition measurements

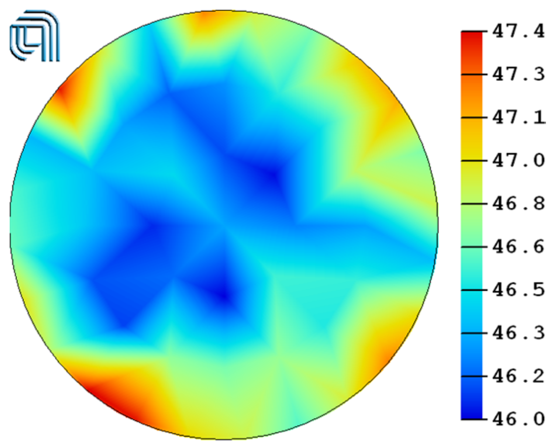
A fundamental parameter calculation was used to derive the composition of the binary alloy PtMn

Thickness (Å)



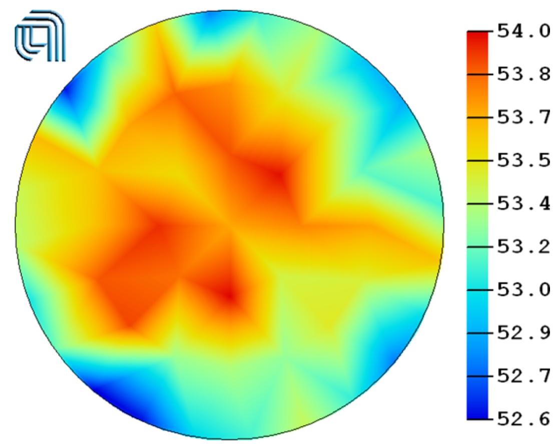
Avg: 153.04
3Sig: 5.10(3.3%)
Rng: 5.62(3.7%)

Mn (Mol%)



Avg: 46.62
3Sig: 1.15(2.5%)
Rng: 1.43(3.1%)

Pt (Mol%)



Avg: 53.38
3Sig: 1.15(2.2%)
Rng: 1.43(2.7%)

The tool is capable to analyze binary or ternary alloy/compound with suitable composition and thickness standards.

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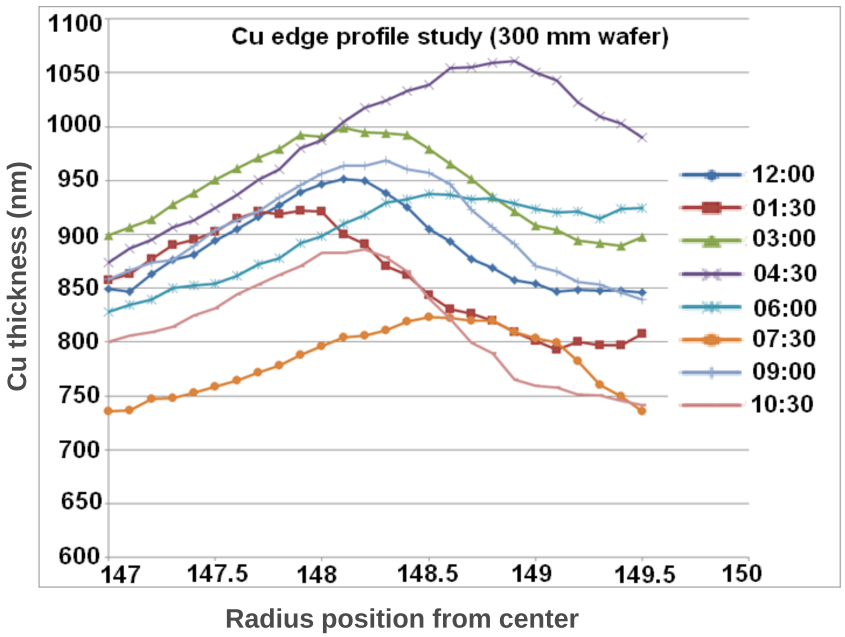
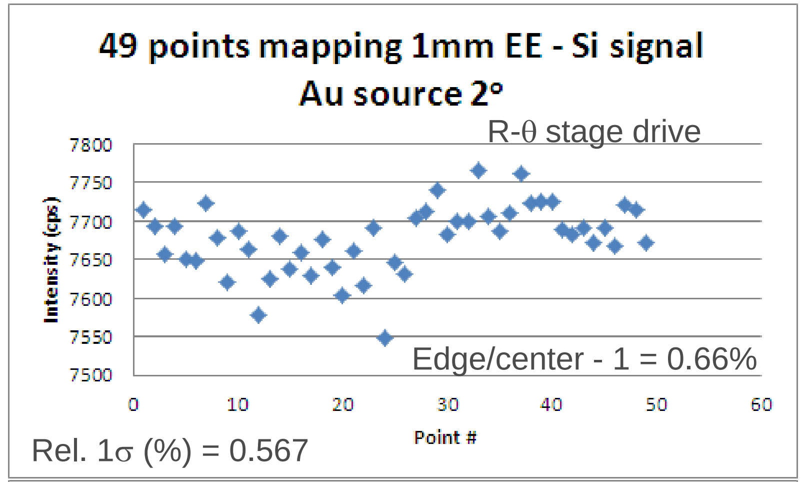
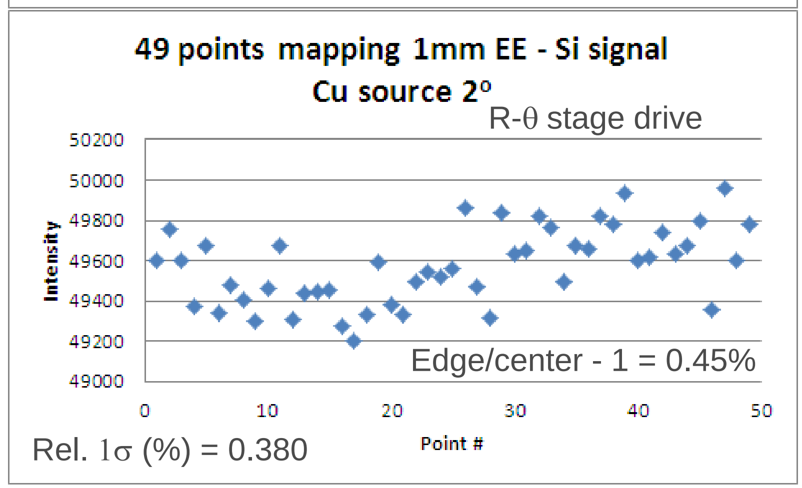
Edge exclusion study – 300 mm Si wafer study

Cu film edge profiles at different edge locations



300 mm bare Si wafer study





Silicon counts showing no significant edge signal drop off even to 1 mm edge exclusion.
The tool was used for Cu film edge profile study

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Conclusions

- GI-XRF tool is implemented that can be operated to meet the theoretically achievable 0.5% (1σ) precision with a minimum incidence angle of 2º to realize a thin film signal amplification factor of 28.7
- For thickness measurements, the tool is demonstrated to be able to achieve 1% (1σ) precision with 20 s counting time for 2 Å Pt and Co films and for 8 Å Ru film. The precision and performance for other films depends on the X-ray source and incidence angle and materials to be analyzed.
- The system operated in the ambient with EDX detector are limited to elements with atomic number larger than aluminum.
- Fundamental parameter algorithm further extend the tool capability to composition analysis of binary alloys and ternary alloys.