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Robust ARXPS data analysis

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The University of Texas at Dallas

M. Quevedo
Texas Instruments

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Content

Robust ARXPS data analysis

- Motivation
  - The structure of Hf-based high-k dielectric stacks
  - Characterization of Hf-based high-k MOS stacks (SIMS and LEIS)
- Angle Resolved XPS
  - Brief description
  - The removal of the upper layers: the need of longer attenuation lengths
  - Applying ARXPS and deconvolving the O 1s peak (HfO2 and SiO2)
- The importance of noise in XPS data peak fitting
  - Standard method (sequential fitting)
  - Robust method (simultaneous fitting)
  - Simultaneous vs Sequential fitting
- Application: Thermal stability of La in Hf-based high-k MOS stacks
  - Finding the binding energy of O 1s in HfO2 and SiO2
  - The constituting layers
  - The depth profile of La
- Conclusions

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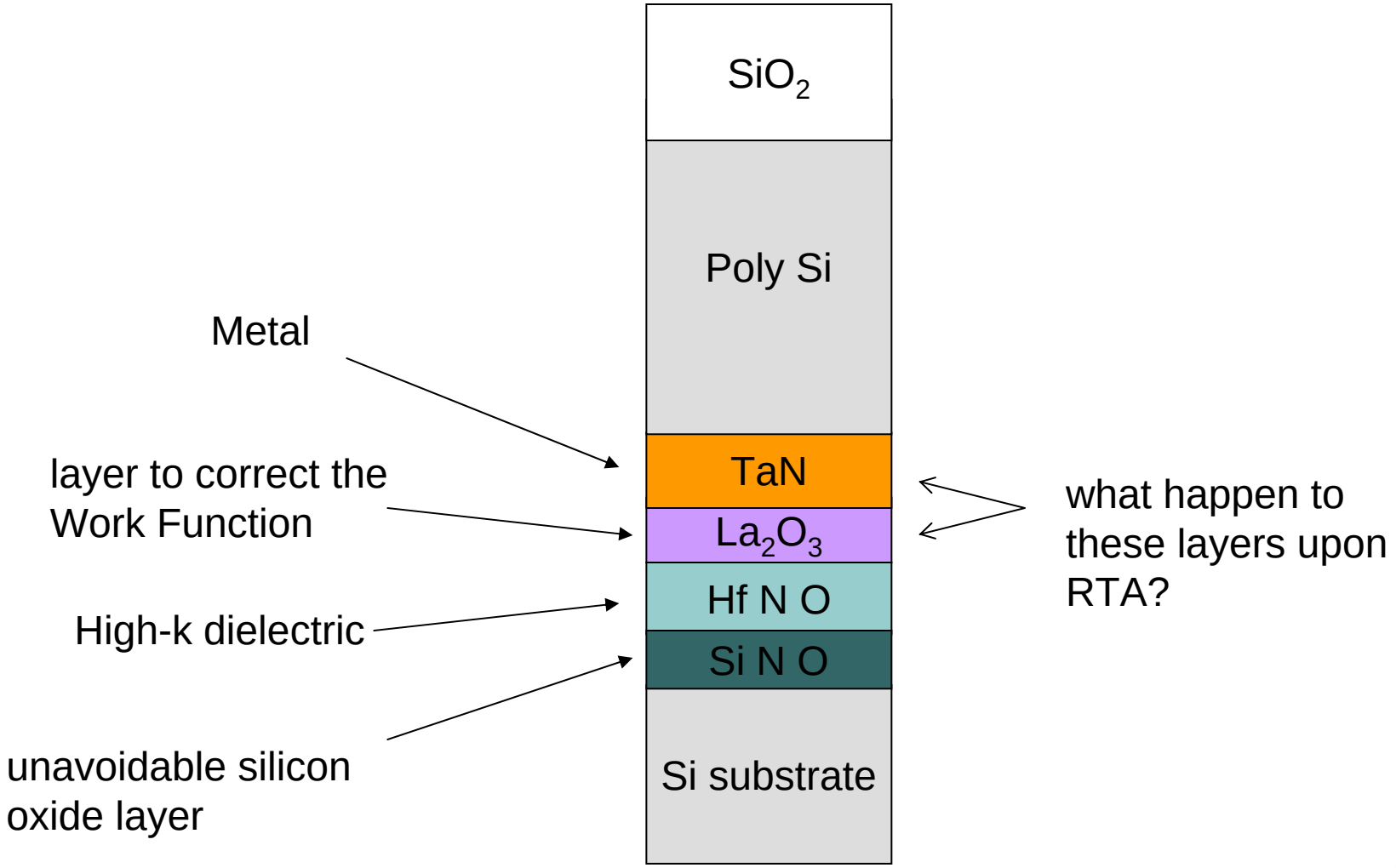
The structure of Hf-based high-k dielectric stacks

Motivation

Thermal stability of dielectric stacks

(Samples grown at Texas Instruments)



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Previous Studies of Thermal Stability

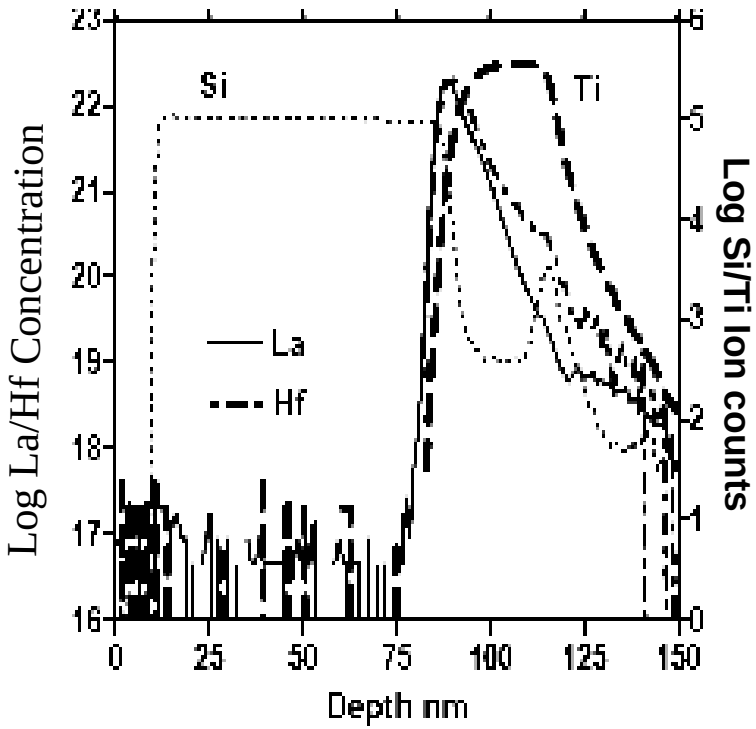
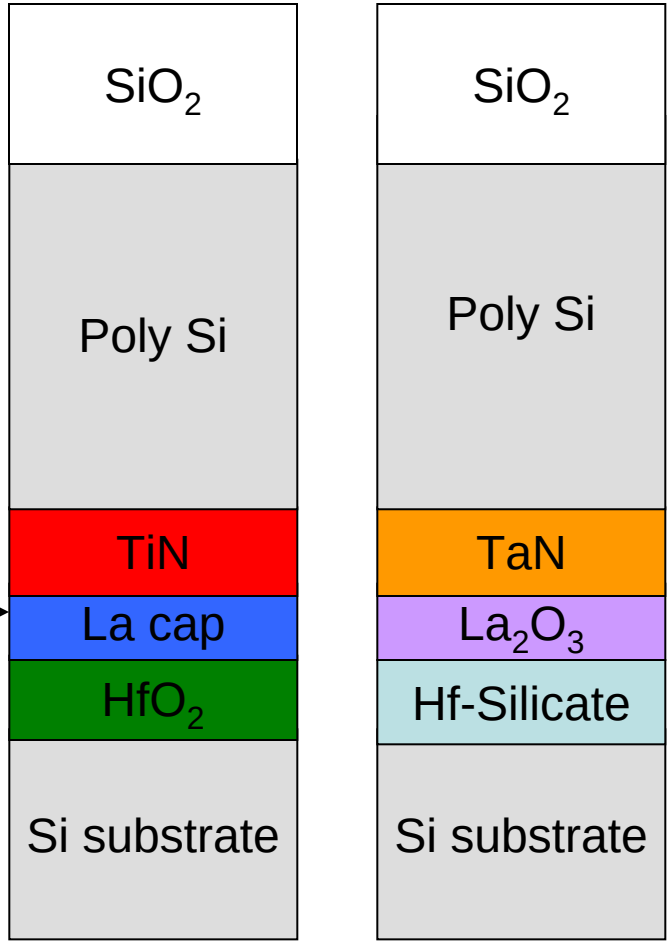


Fig. 9: Backside SIMS showing no La penetration into the Si substrate after high T (1000°C) process

Both La_2O_3 and La-Silicate were used in experiments



IBM Sample

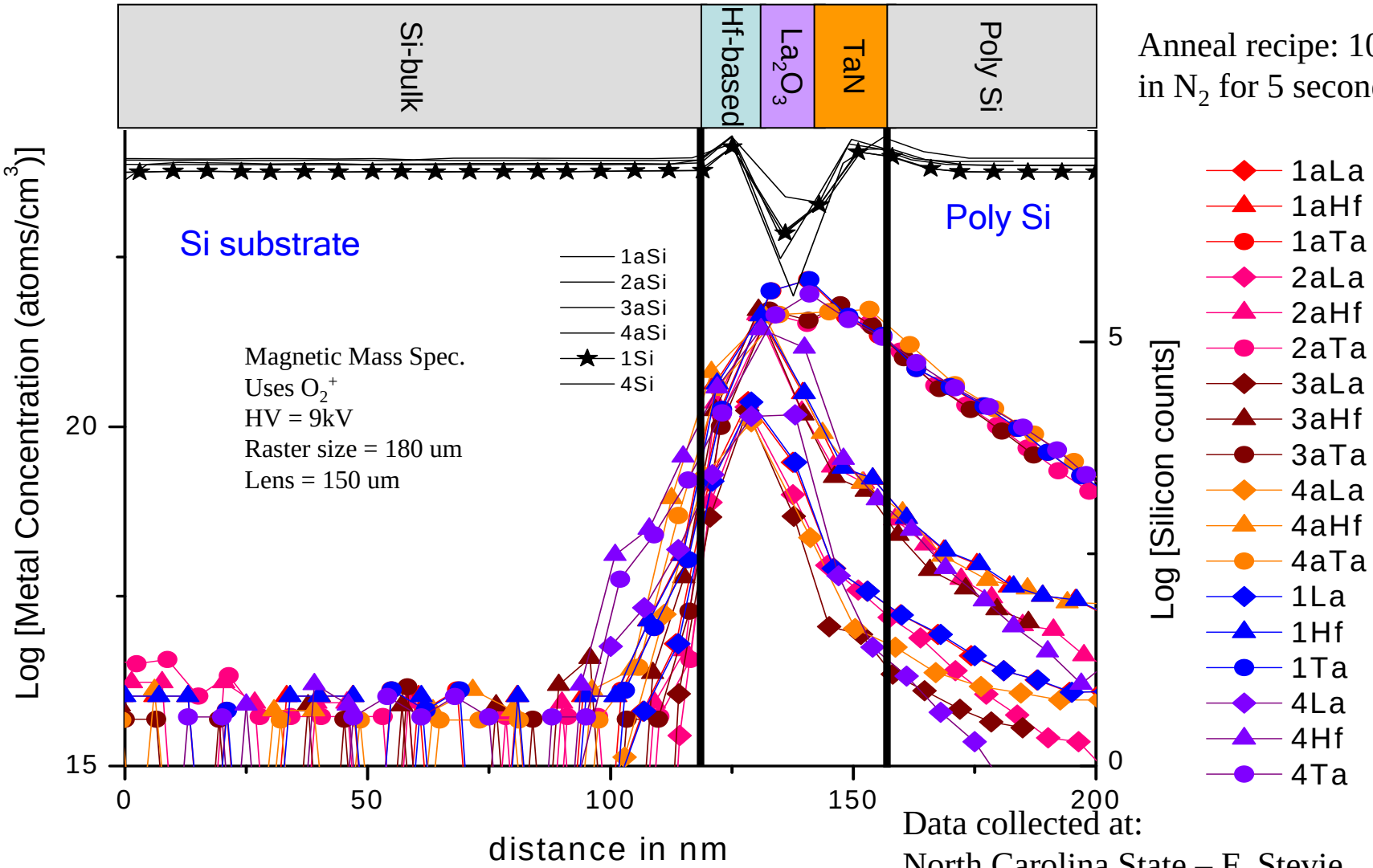
Our Sample

[1]IBM VLSI 2006, V. Narayanan *et. al.*

Diffusion of Ta, La, or Hf into Si bulk

Backside Secondary Ion Mass Spectroscopy: All Samples

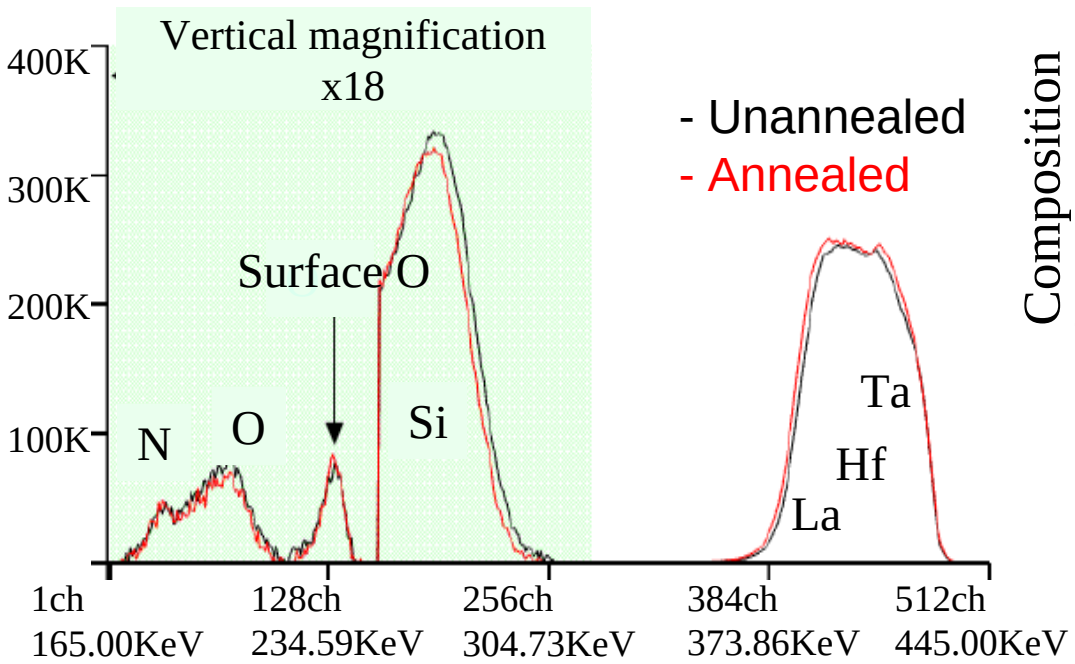
No diffusion of La, Hf, nor Ta, from any of the samples into the substrate.

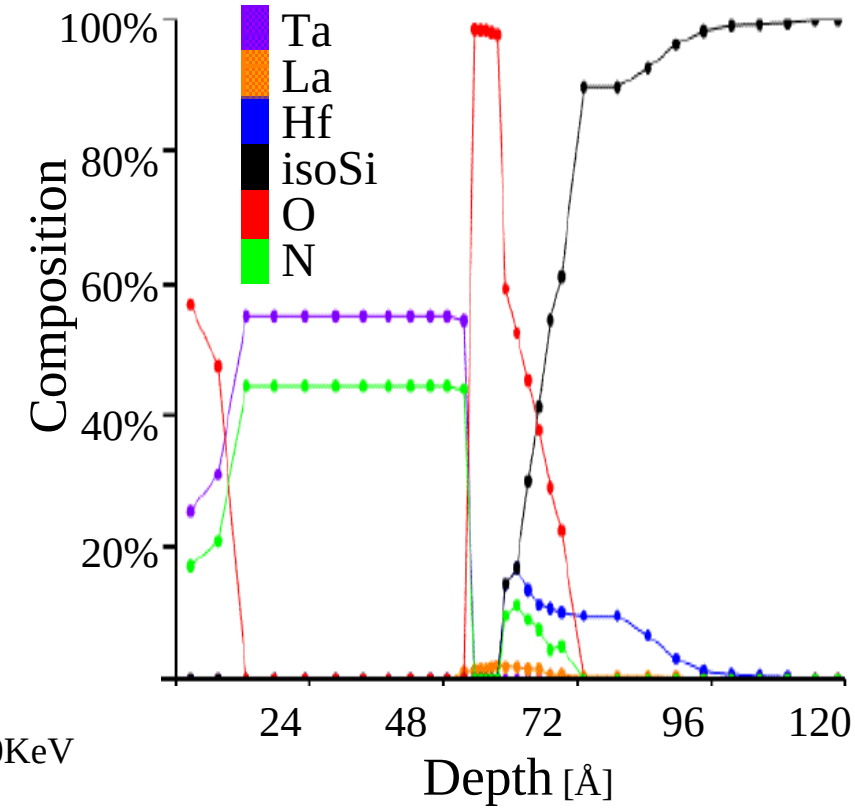


Anneal recipe: 1000ºC in N_2 for 5 seconds.

Data collected at:
North Carolina State – F. Stevie

Sample 1


Vertical magnification
x18
400K
300K
200K
100K
Surface O
N
O
Si
Ta
Hf
La
- Unannealed
- Annealed
1ch
165.00KeV
128ch
234.59KeV
256ch
304.73KeV
384ch
373.86KeV
512ch
445.00KeV


Ta
La
Hf
isoSi
O
N
100%
80%
60%
40%
20%
Composition
24
48
72
96
120
Depth [Å]

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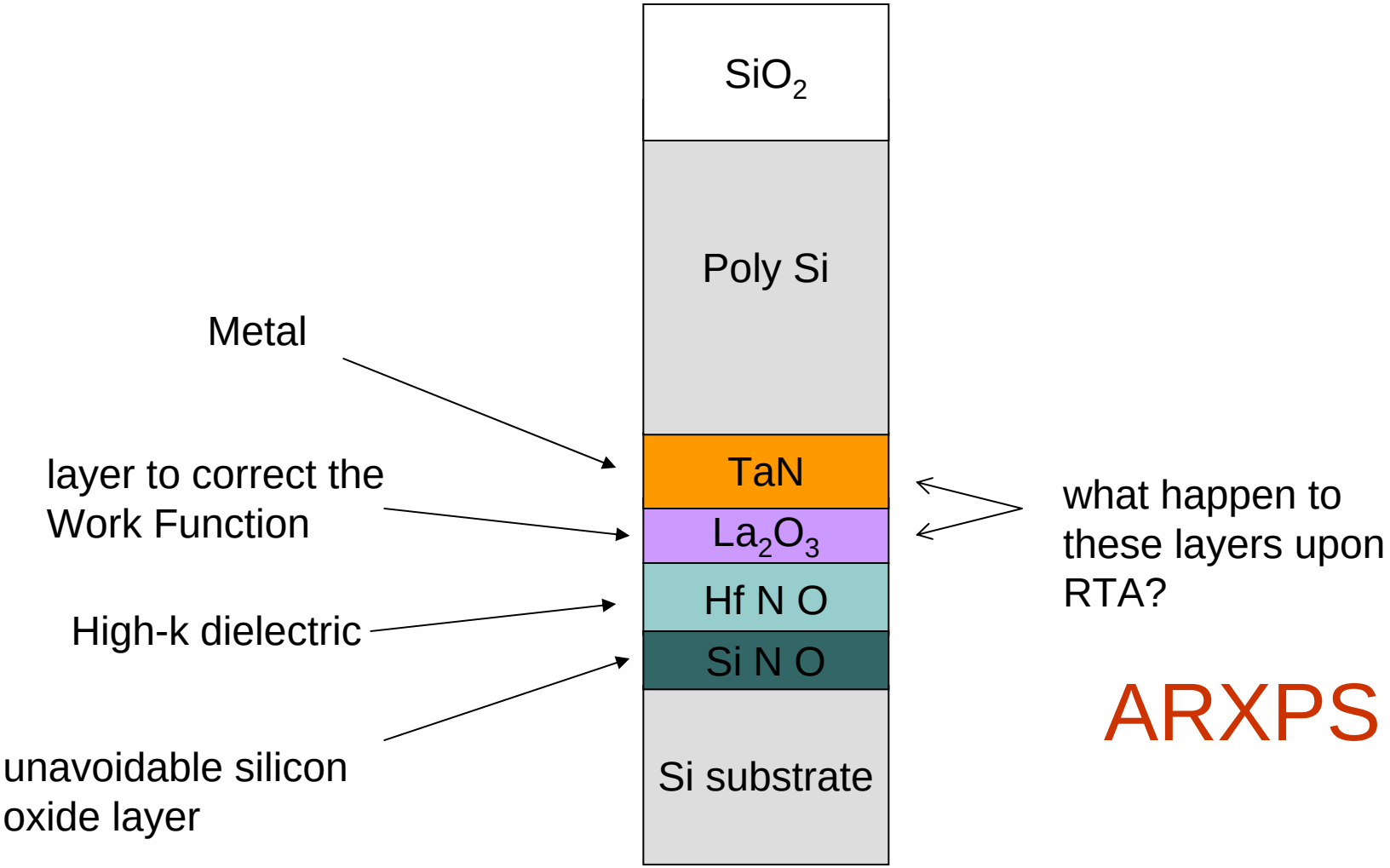
The structure of Hf-based high-k dielectric stacks

Motivation

Thermal stability of dielectric stacks

(Samples grown at Texas Instruments)



ARXPS

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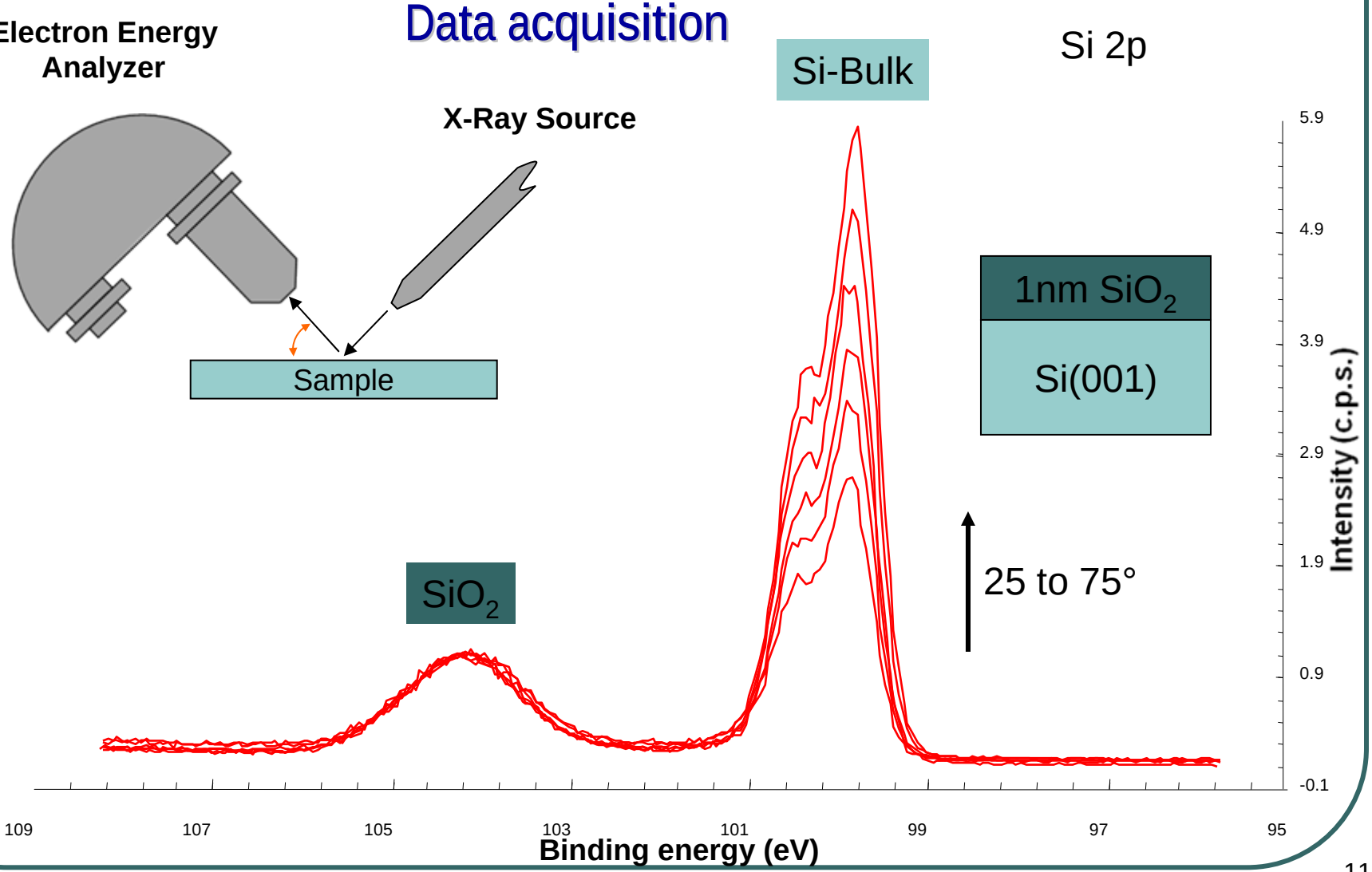
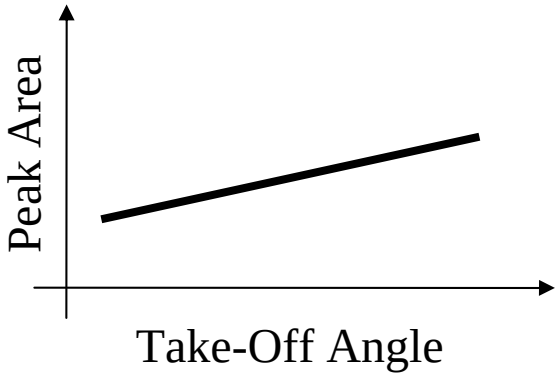
Brief description of ARXPS

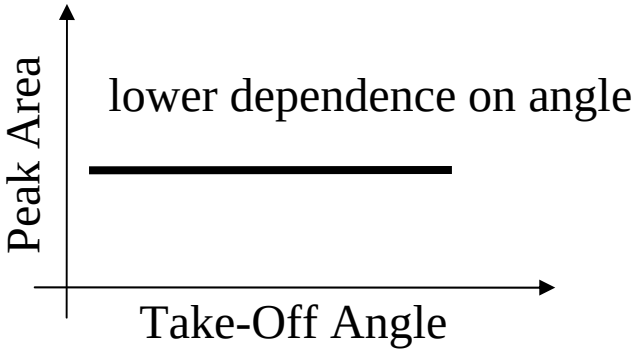
Angle Resolved XPS

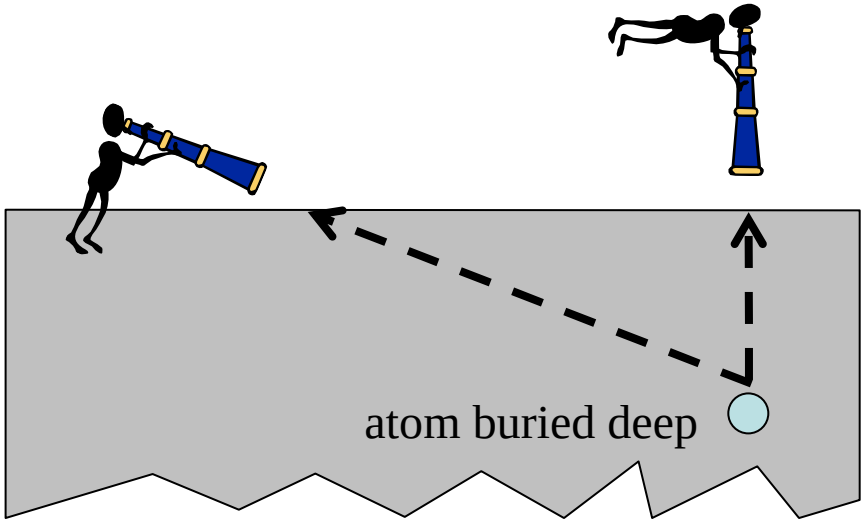
Angle Resolved XPS:

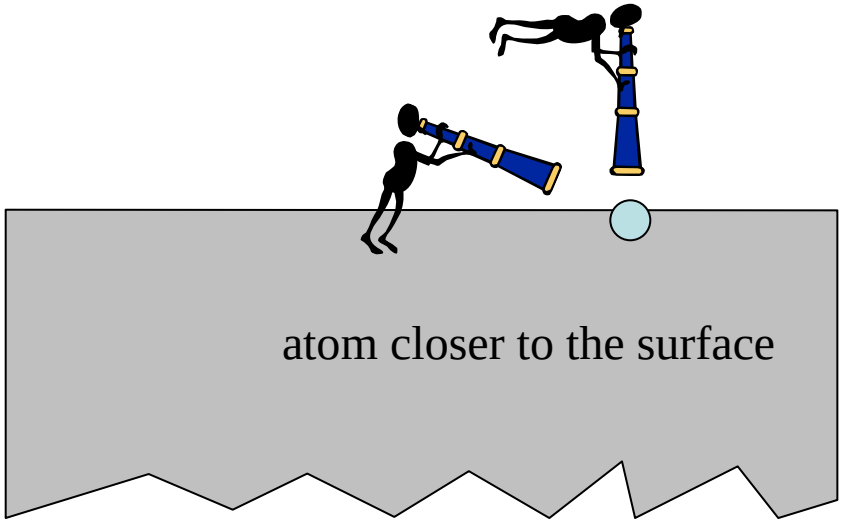
Data acquisition




Peak Area
Take-Off Angle


lower dependence on angle
Peak Area
Take-Off Angle


atom buried deep


atom closer to the surface

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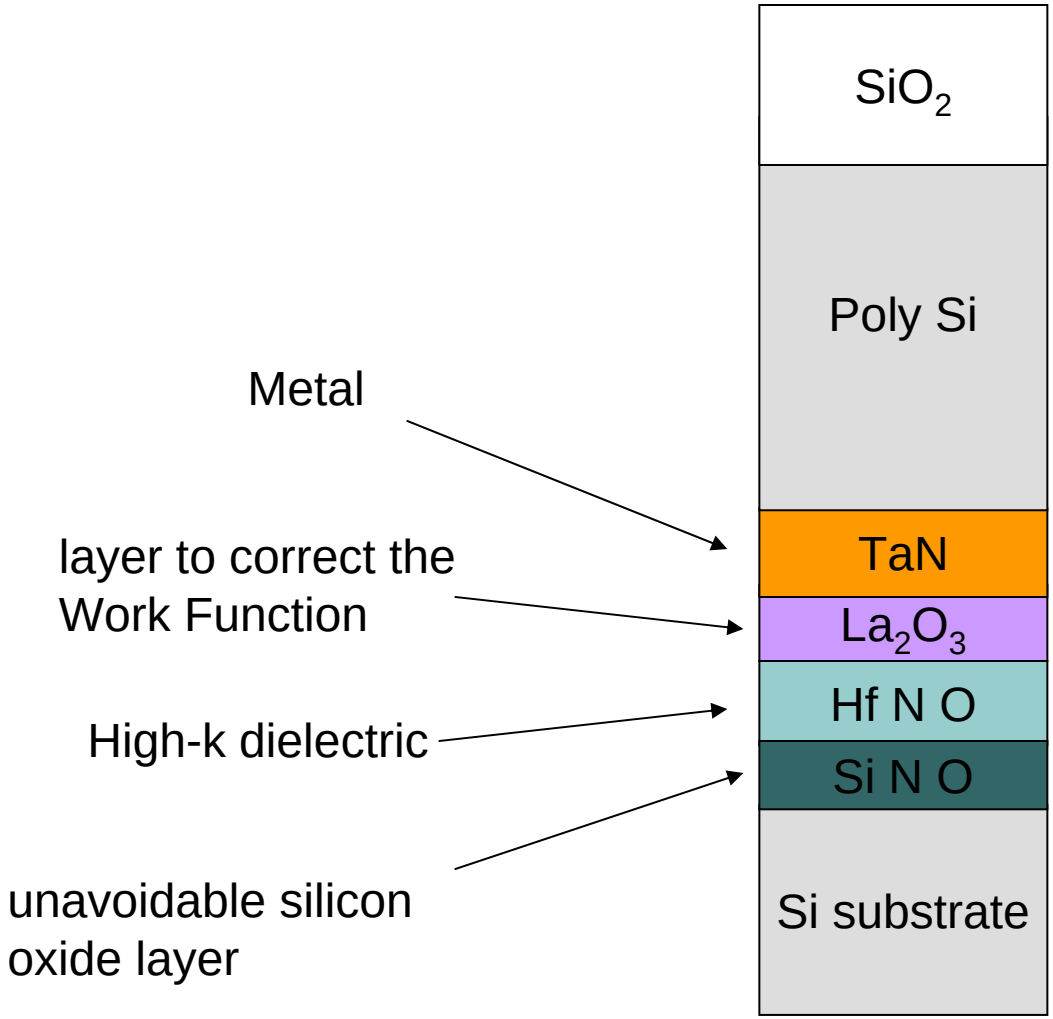
The removal of the upper layers: need longer EAL

Angle Resolved XPS

Thermal stability of dielectric stacks



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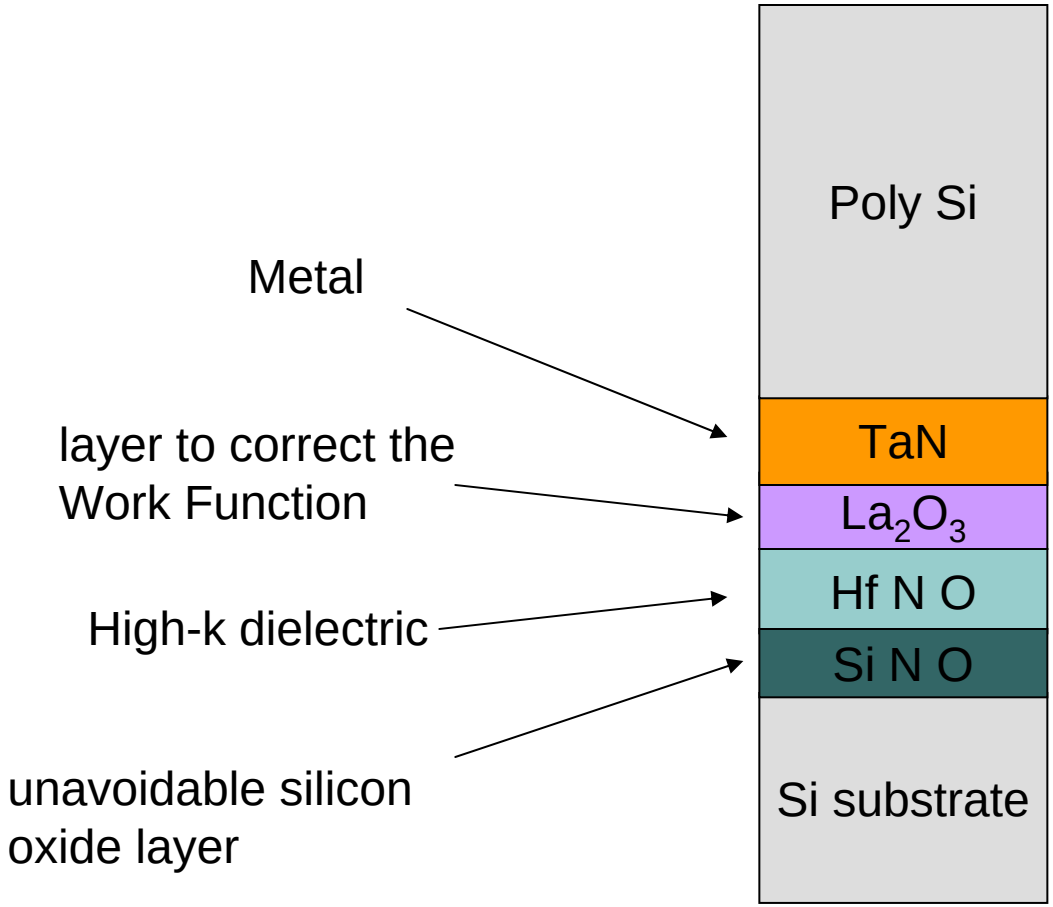
The removal of the upper layers: need longer EAL

Angle Resolved XPS

Thermal stability of dielectric stacks



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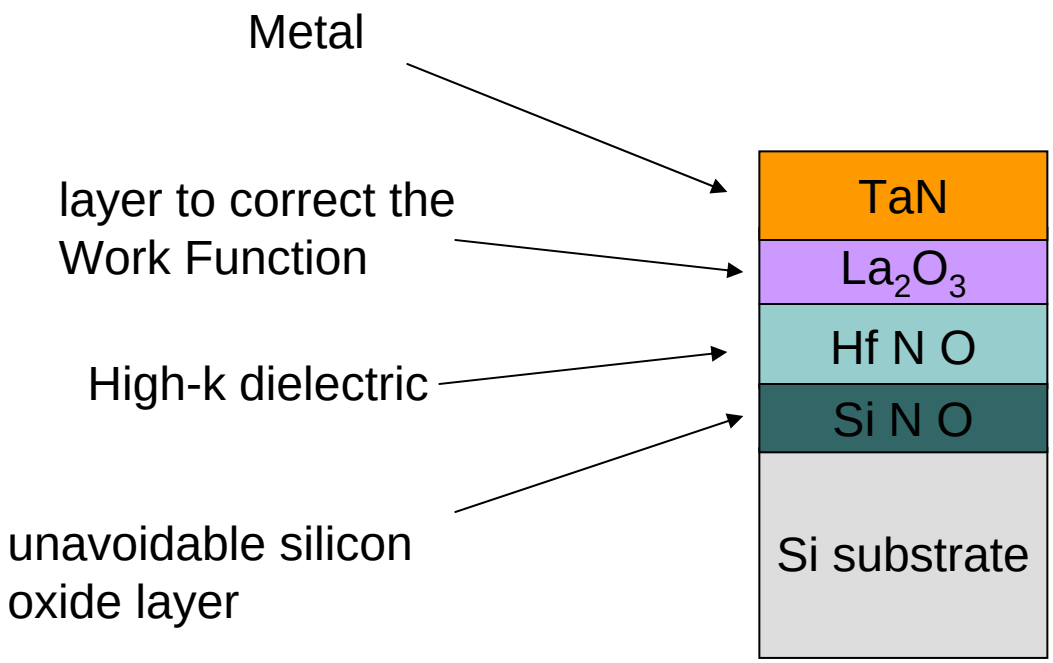
Angle Resolved XPS

Thermal stability of dielectric stacks



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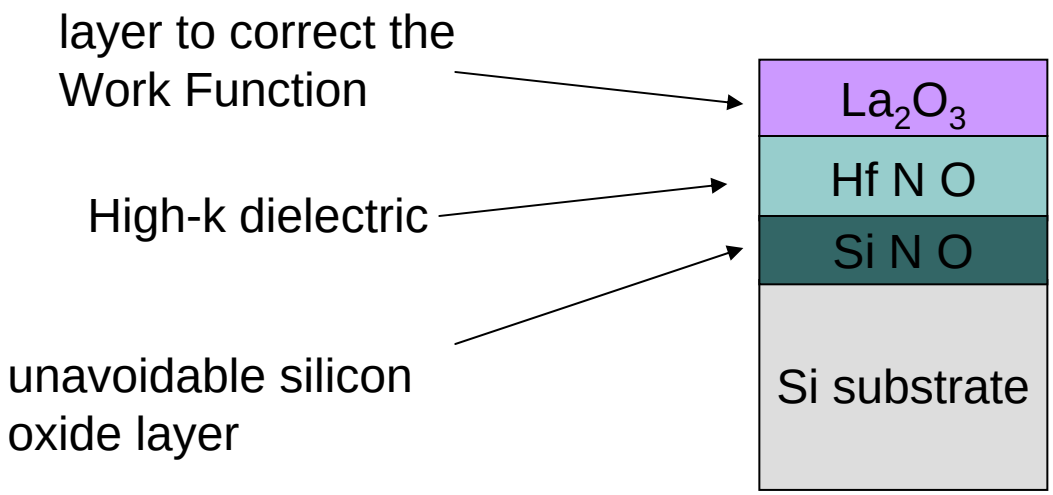
Angle Resolved XPS

Thermal stability of dielectric stacks



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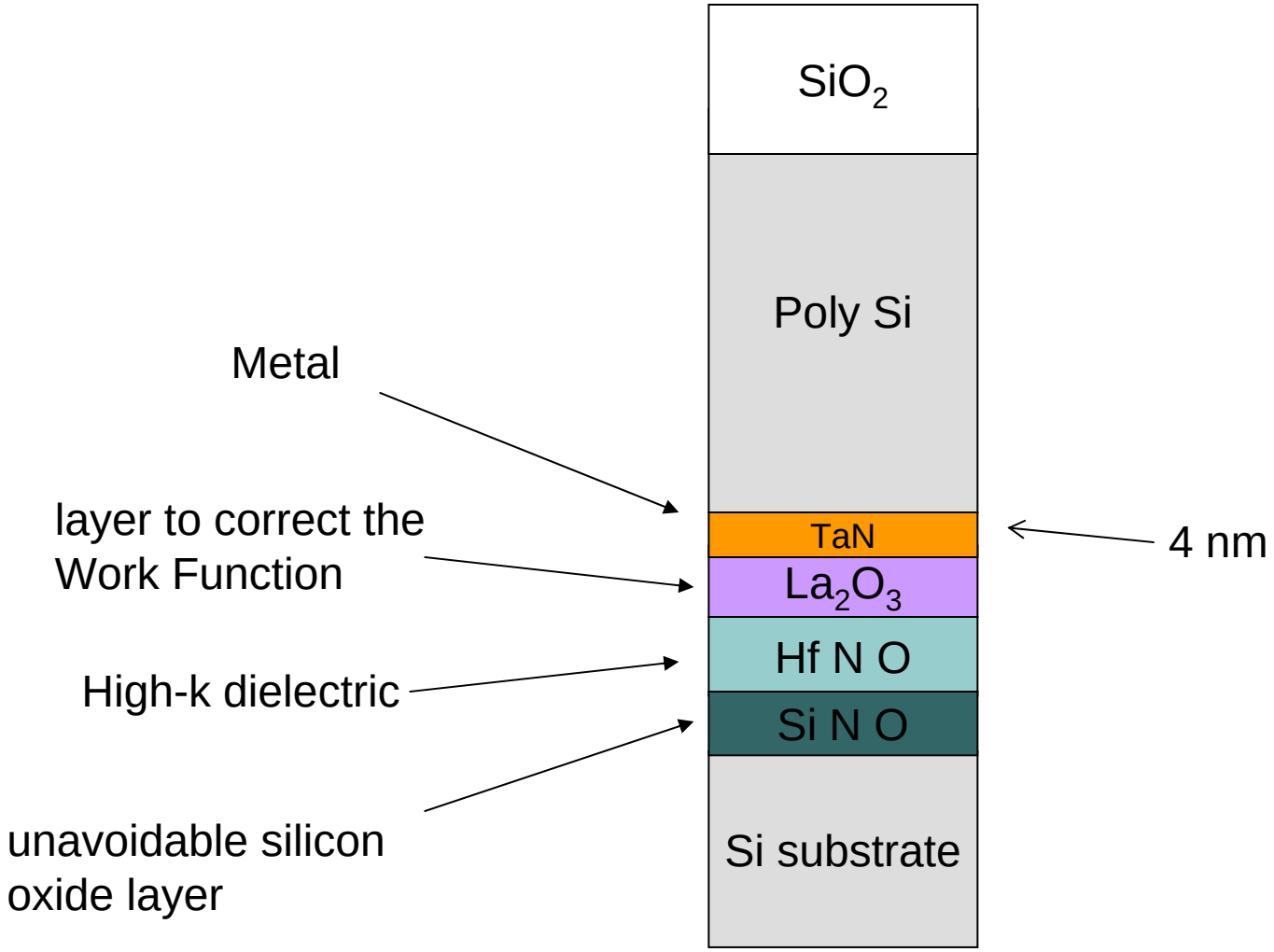
Thermal stability of dielectric stacks



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Angle Resolved XPS

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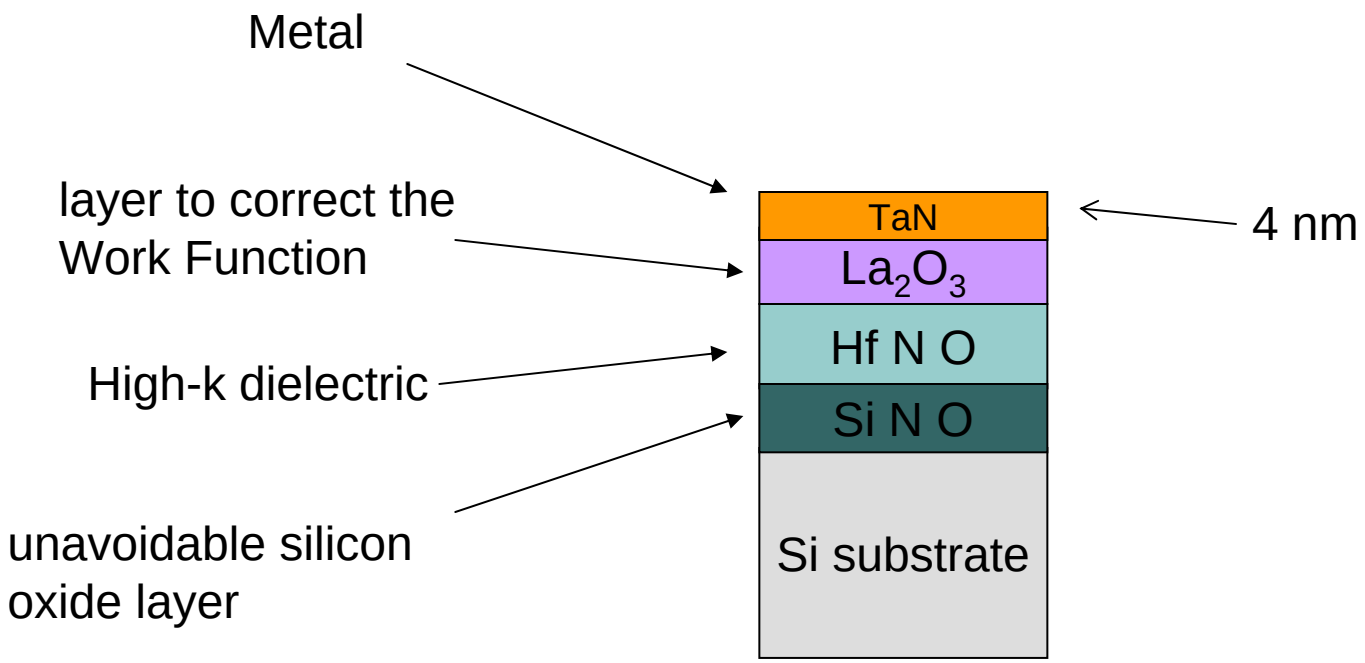

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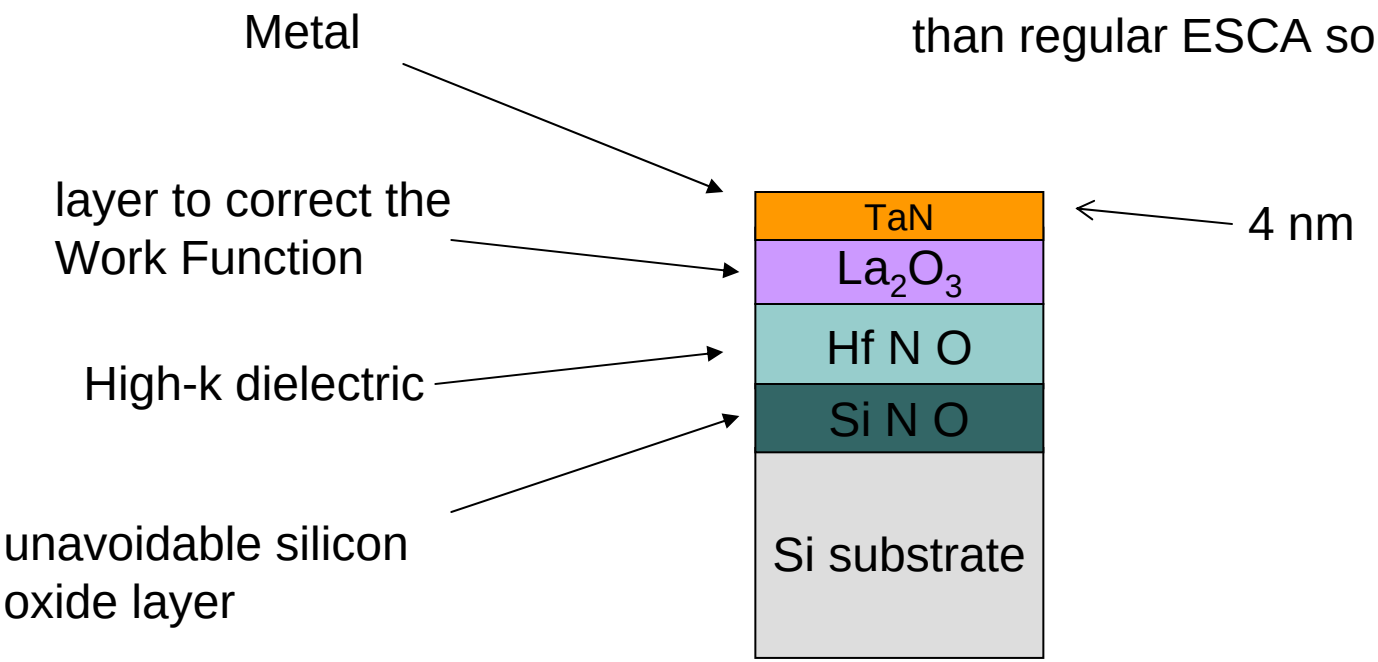
The removal of the upper layers: need longer EAL

Angle Resolved XPS

Thermal stability of dielectric stacks

- **Higher intensity**
- **Higher energy**
- **Higher resolution**

than regular ESCA sources



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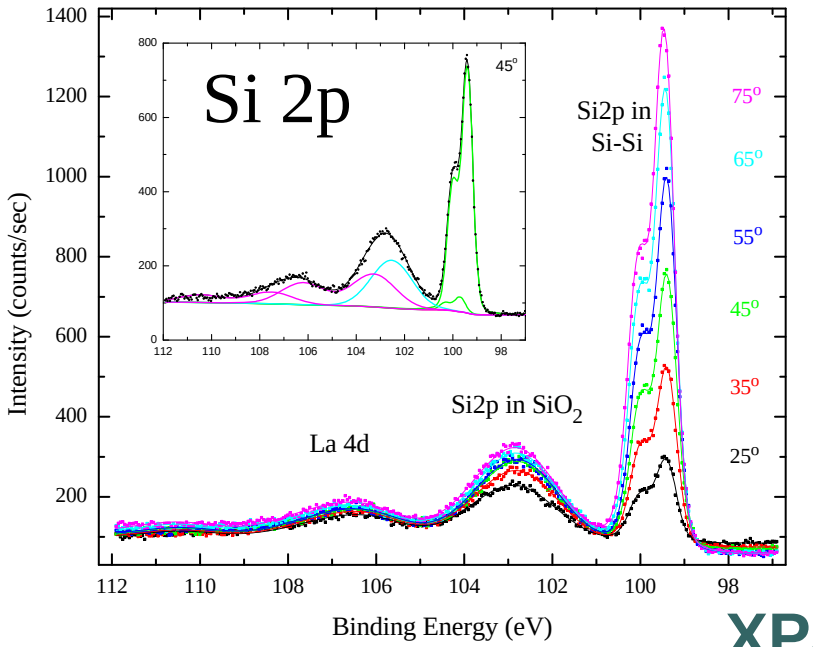
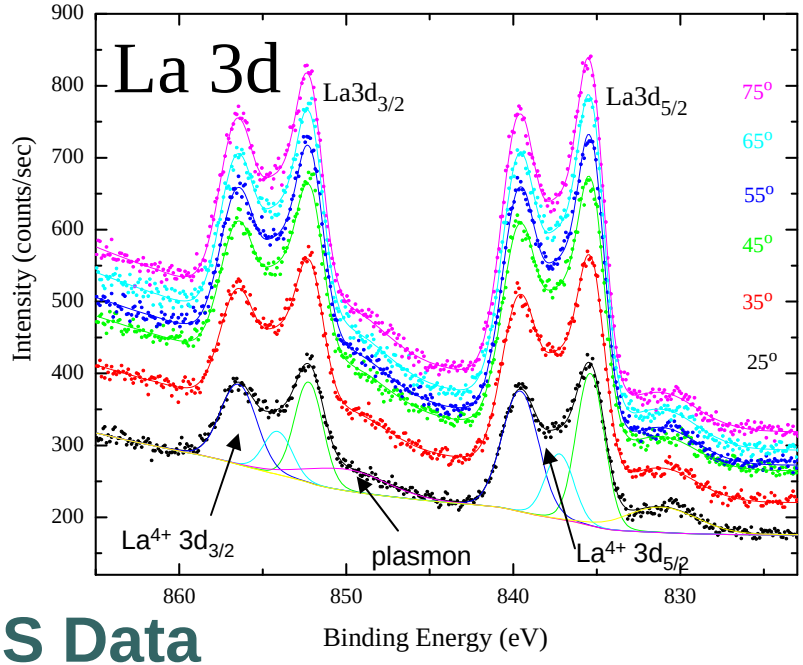
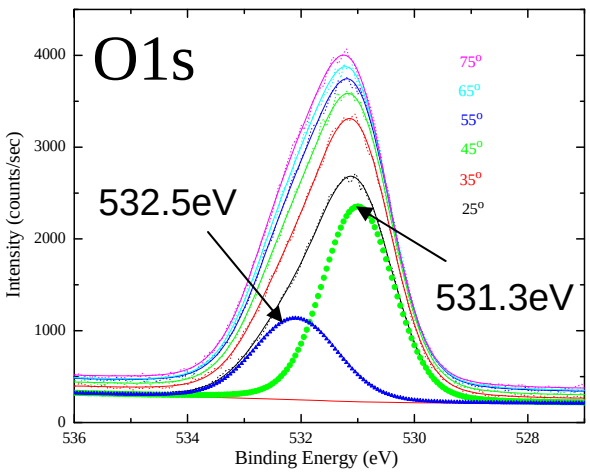
Applying ARXPS and deconvolving the O 1s peak

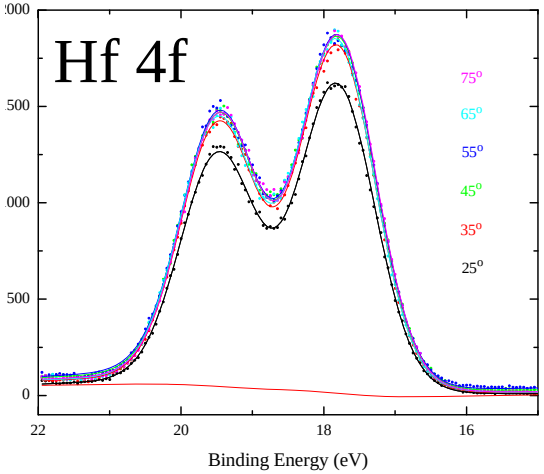
Angle Resolved XPS

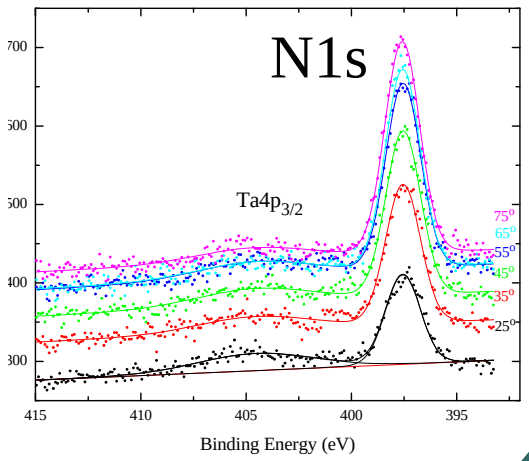




XPS Data







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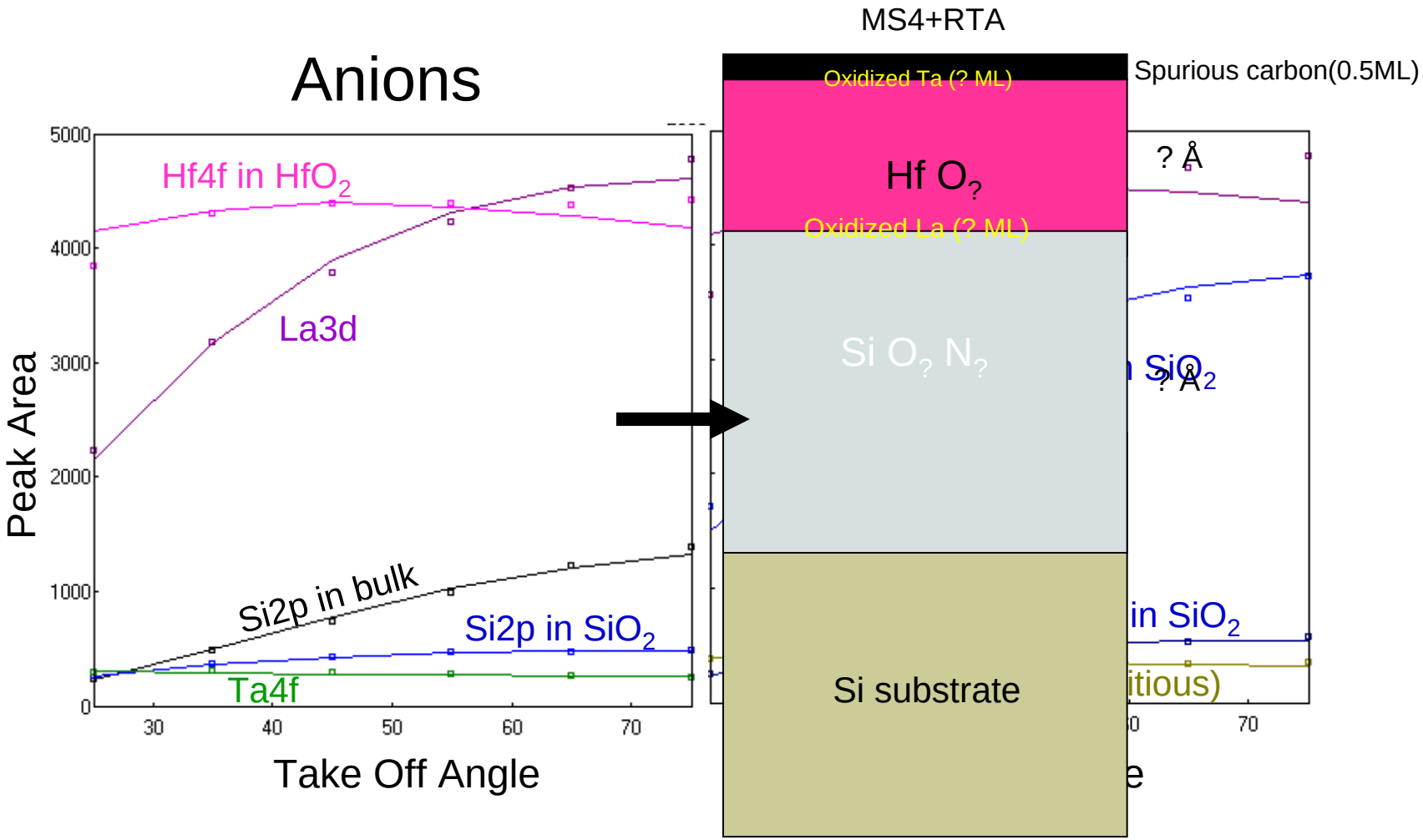
Applying ARXPS and deconvolving the O 1s peak

Angle Resolved XPS



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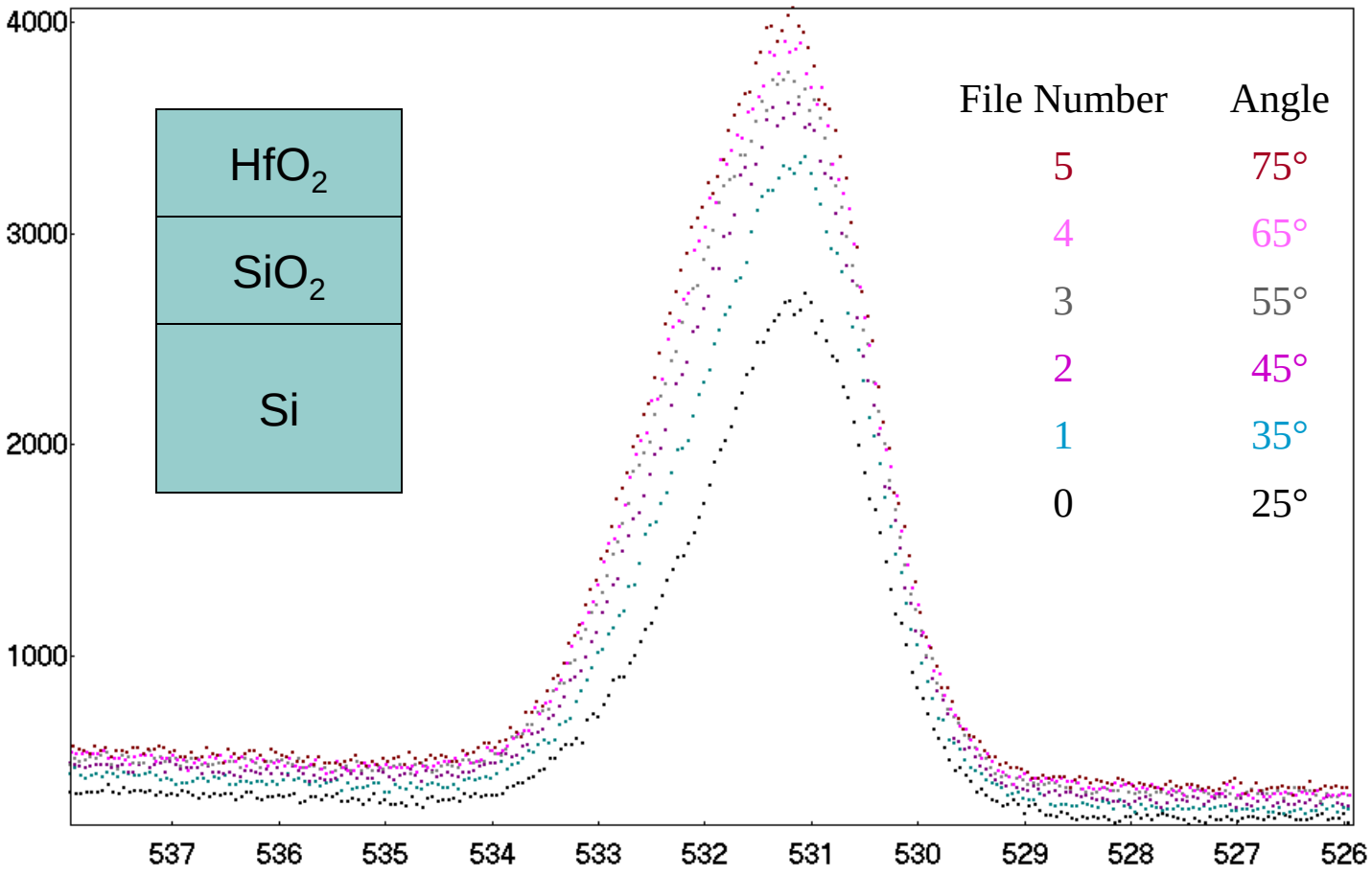
Applying ARXPS and deconvolving the O 1s peak

Angle Resolved XPS

HfO2/SiO2/Si nanofilm



Is it possible to robustly and uniquely get the two peaks?

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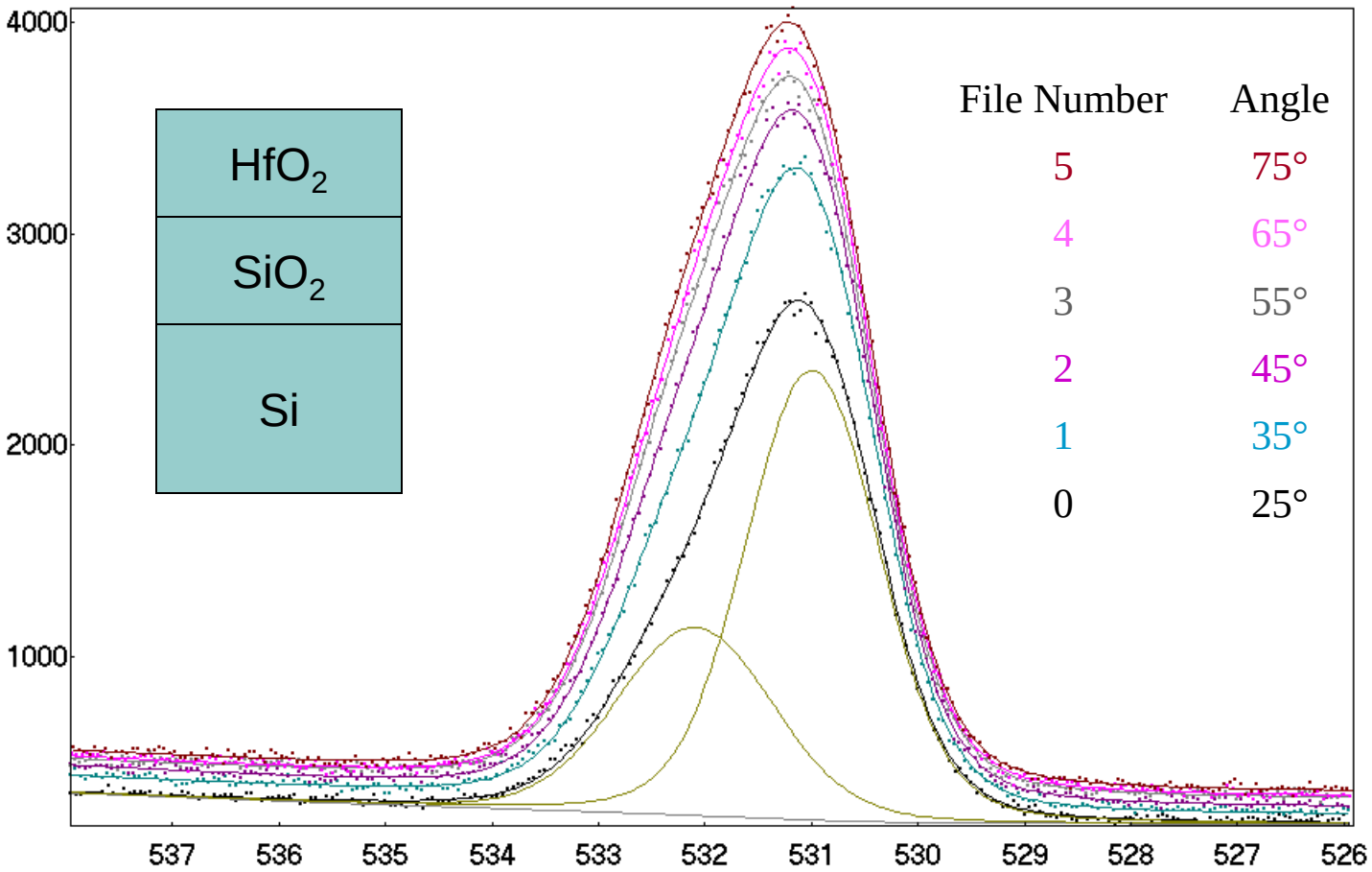
Applying ARXPS and deconvolving the O 1s peak

Angle Resolved XPS

HfO2/SiO2/Si nanofilm



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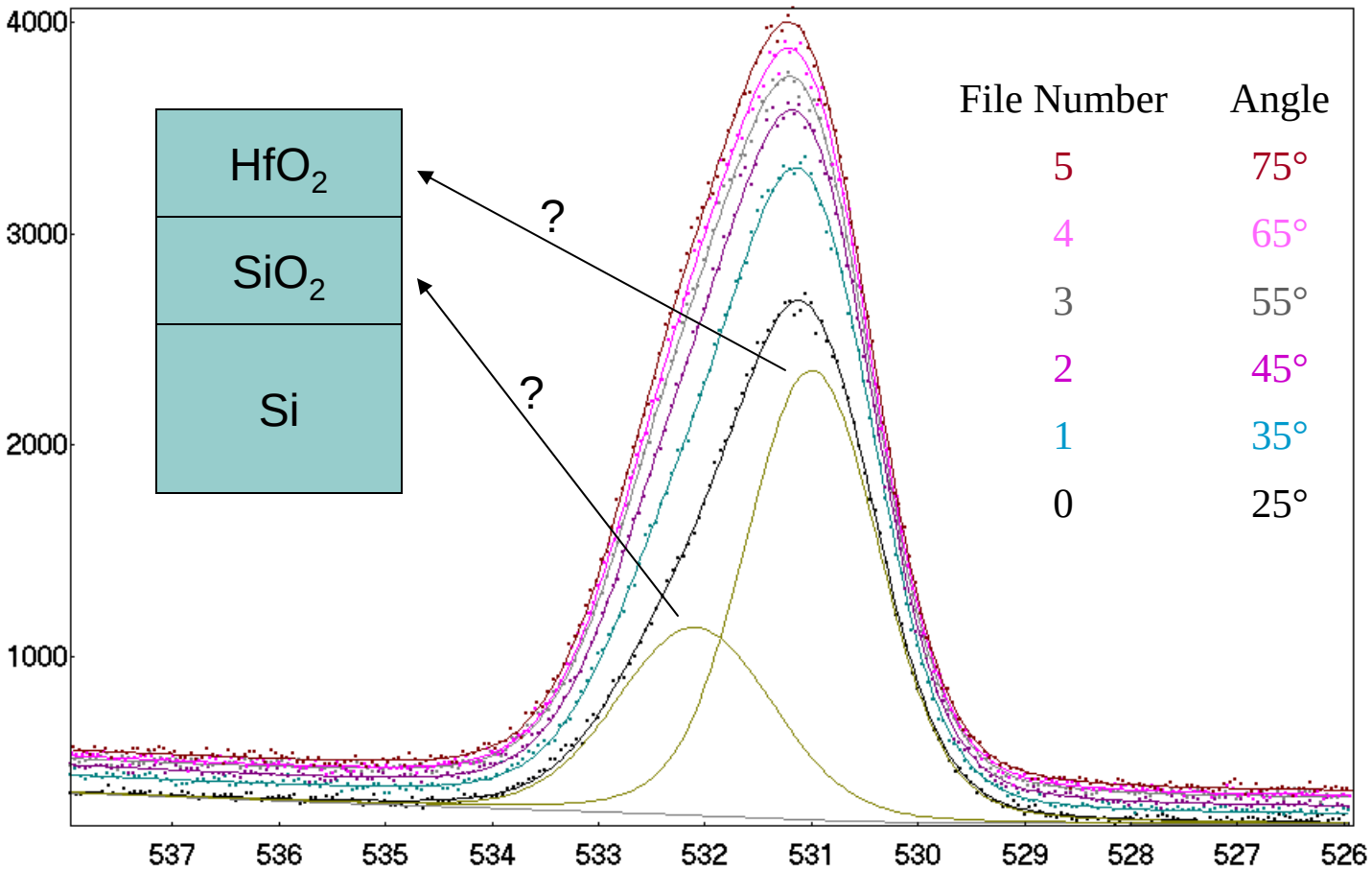

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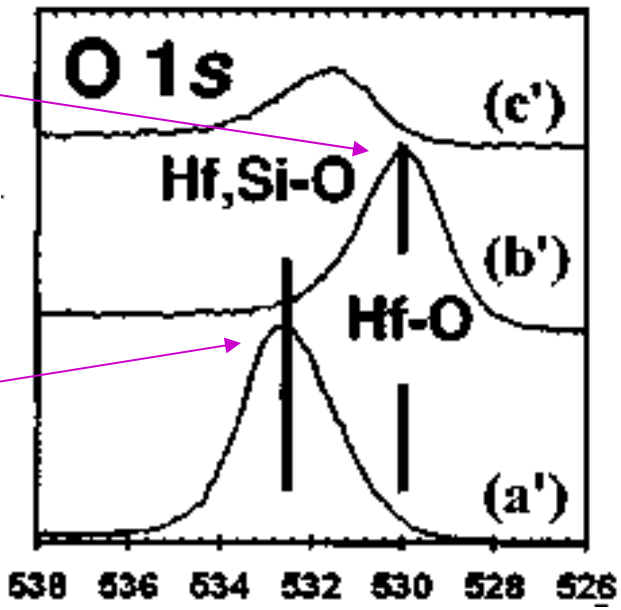
Angle Resolved XPS

Applying ARXPS and deconvolving the O 1s peak

Thick HfO_2

Thick SiO_2



G. D. Wilk, R. M. Wallace, and J. M. Anthony, J. Appl. Phys. **87**, 484 (2000).

O	1s	HfO2	530.00
O	1s	HfO2	530.40
O	1s	HfO2/C	529.60

O	1s	SiO2	532.00
O	1s	SiO2	532.10
O	1s	SiO2	532.27
O	1s	SiO2	532.30
O	1s	SiO2	532.40

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Applying ARXPS and deconvolving the O 1s peak

Angle Resolved XPS

Element	Spectral Line	Formula	Energy (eV)
O	1s	SiO2	532.40
O	1s	SiO2	532.50
O	1s	SiO2	532.50
O	1s	SiO2	532.51
O	1s	SiO2	532.60
O	1s	SiO2	532.60
O	1s	SiO2	532.60
O	1s	SiO2	532.65
O	1s	SiO2	532.70
O	1s	SiO2	532.70
O	1s	SiO2	532.80
O	1s	SiO2	532.80
O	1s	SiO2	532.80
O	1s	SiO2	532.80
O	1s	SiO2	532.80
O	1s	SiO2	532.84
O	1s	SiO2	532.90
O	1s	SiO2	532.90
O	1s	SiO2	532.90
O	1s	SiO2	532.90

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Applying ARXPS and deconvolving the O 1s peak

Angle Resolved XPS

Element	Spectral Line	Formula	Energy (eV)
O	1s	SiO2	532.40
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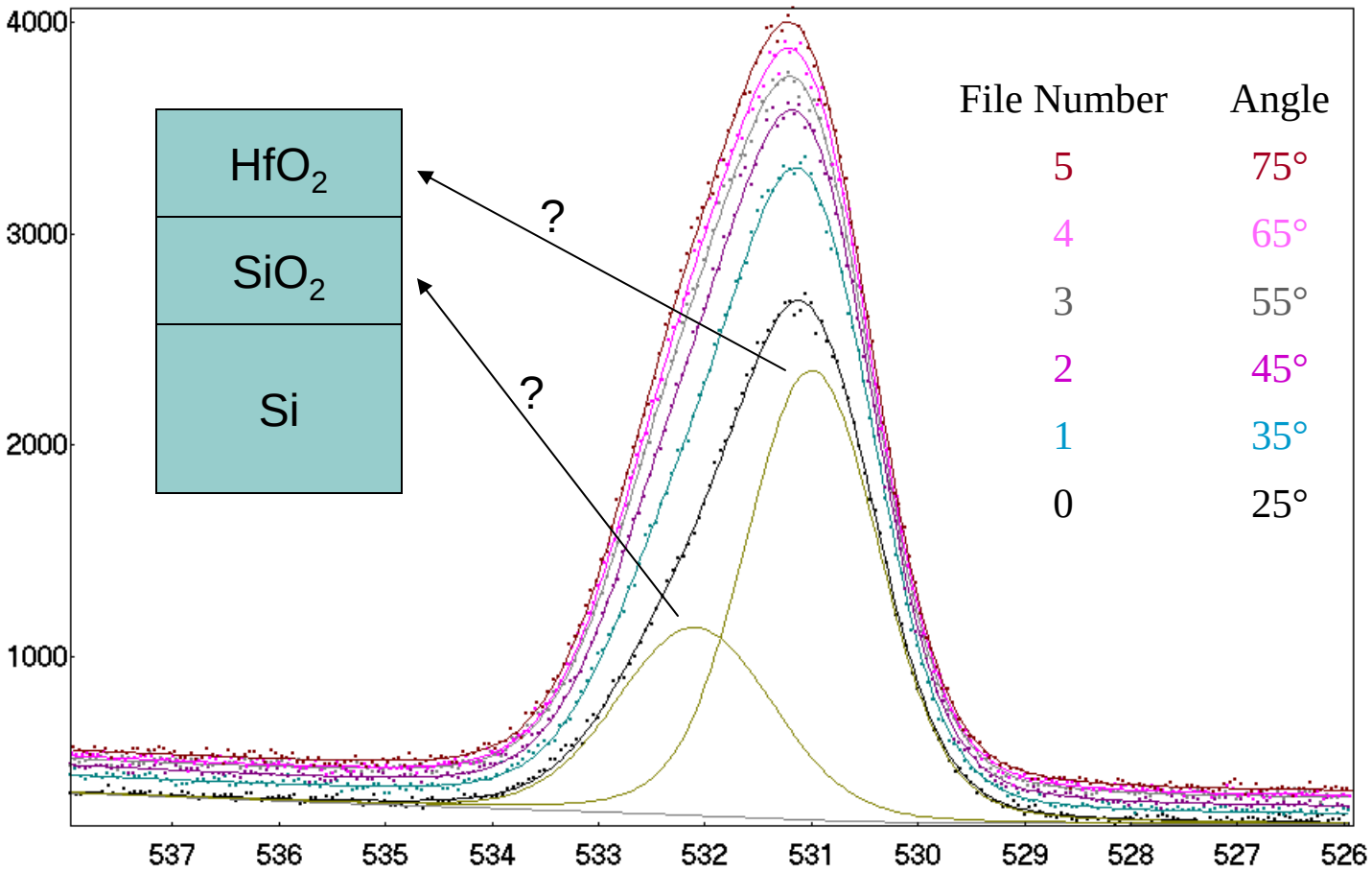
Angle Resolved XPS

HfO2/SiO2/Si nanofilm



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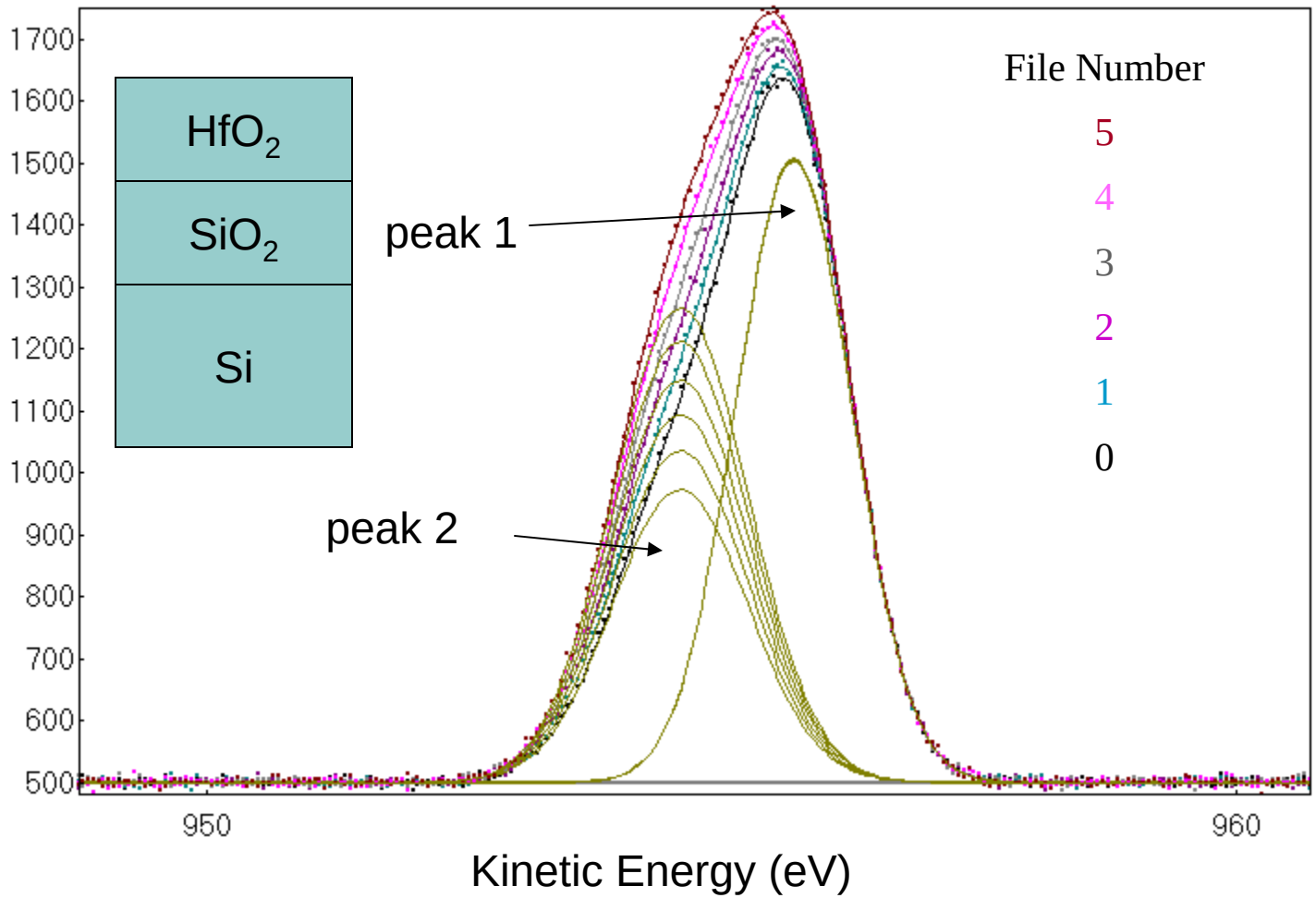
Standard fitting method: Sequential

Simulated data mimicking O 1s from HfO_2 (peak 1) and SiO_2 (peak 2)



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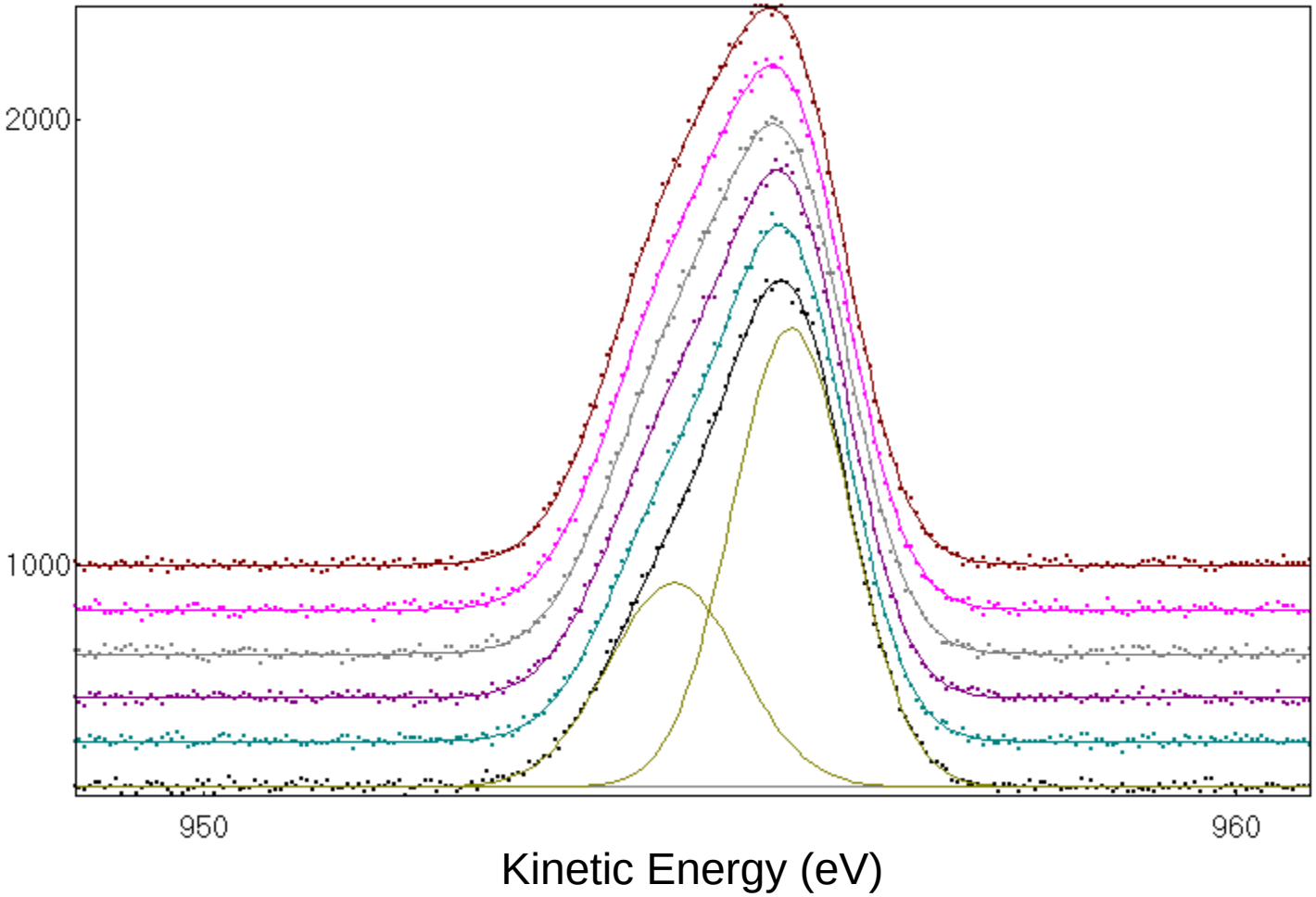

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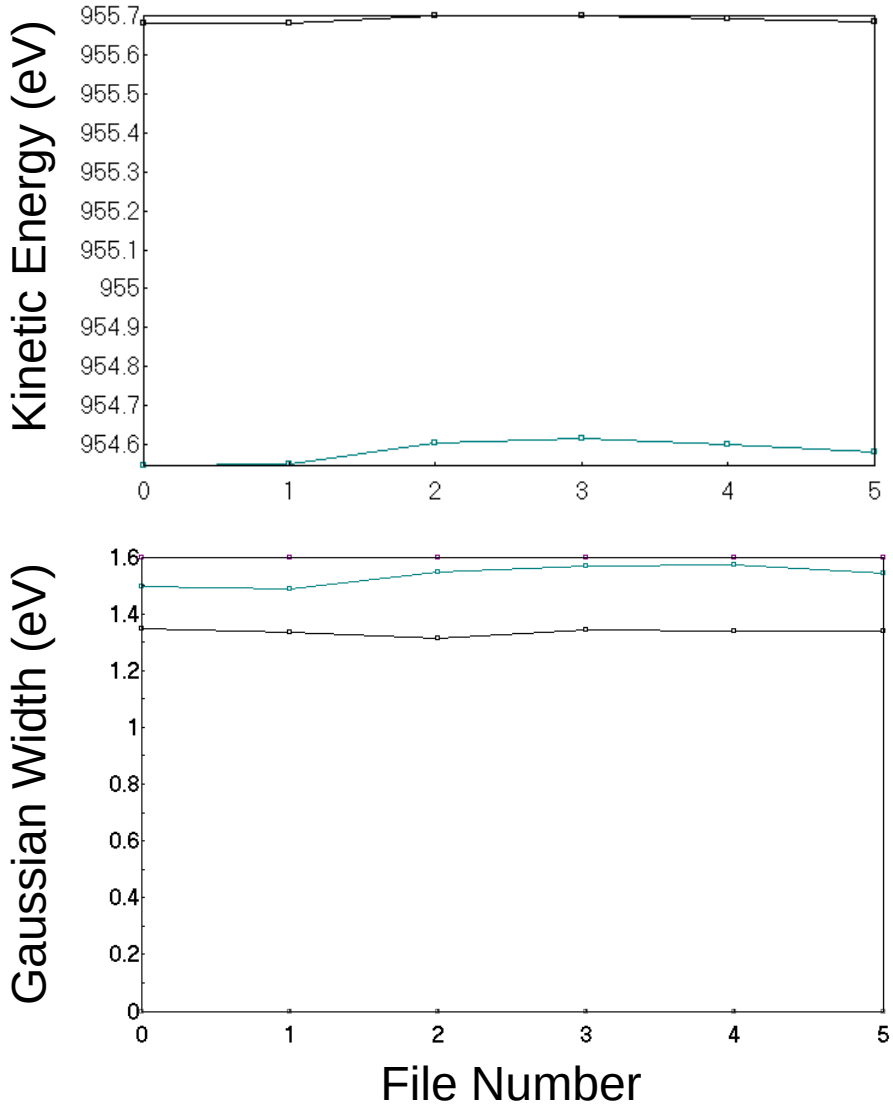
Standard fitting method: Sequential

The importance of noise in XPS data fitting



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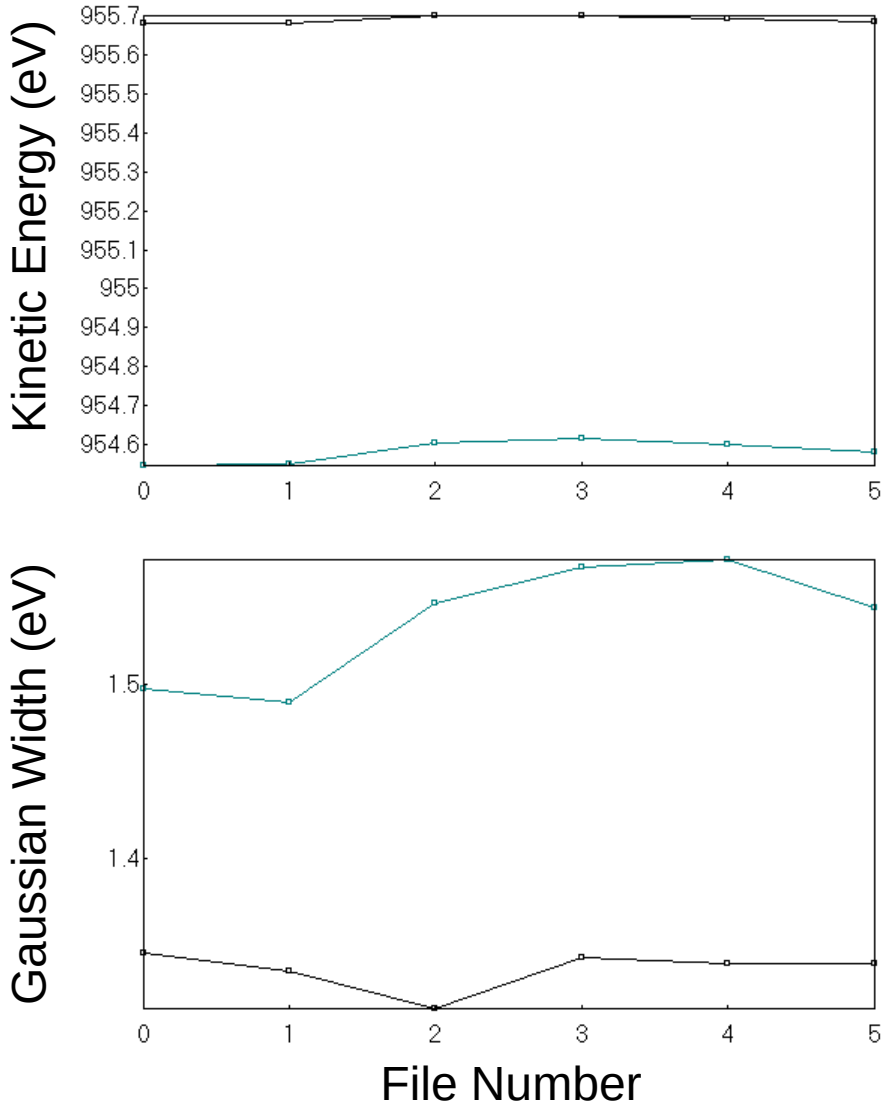

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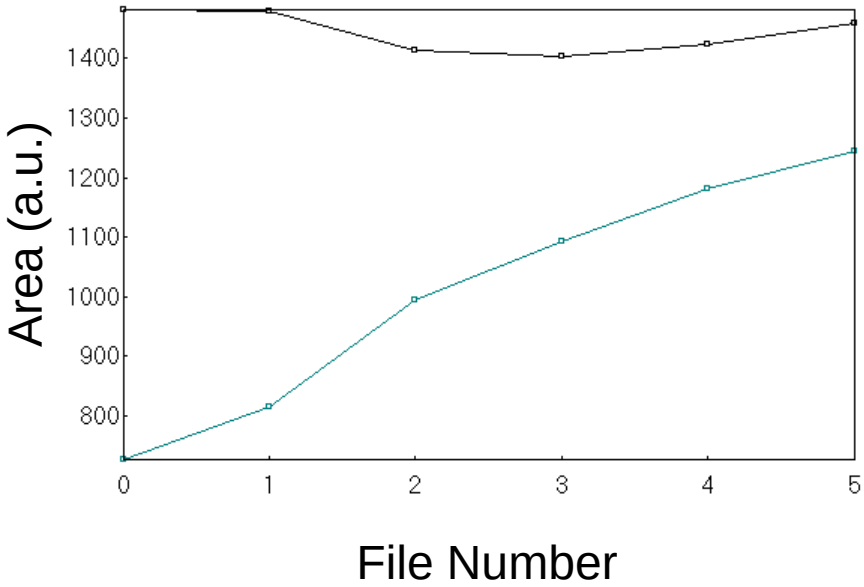

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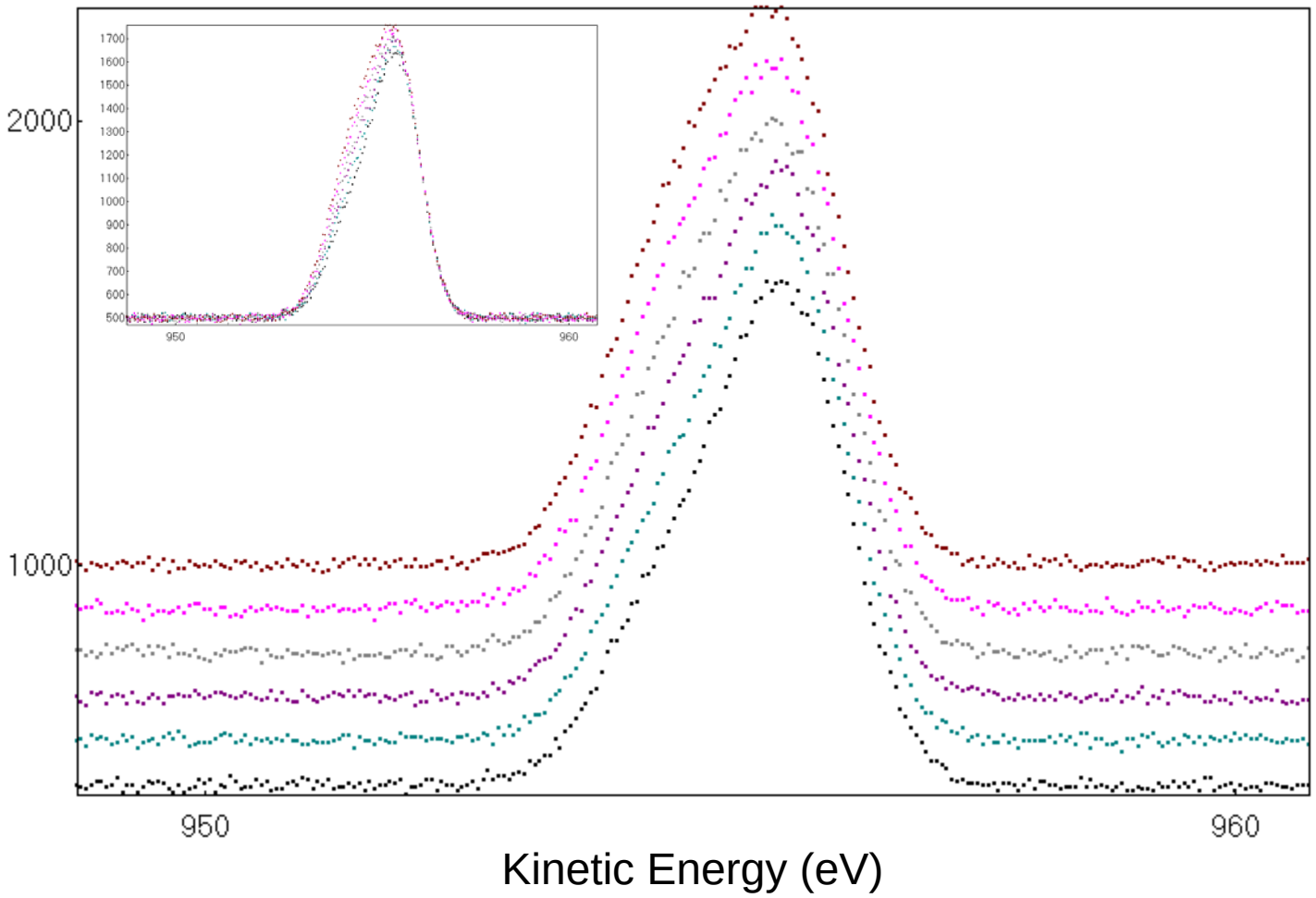
Robust fitting method: Simultaneous



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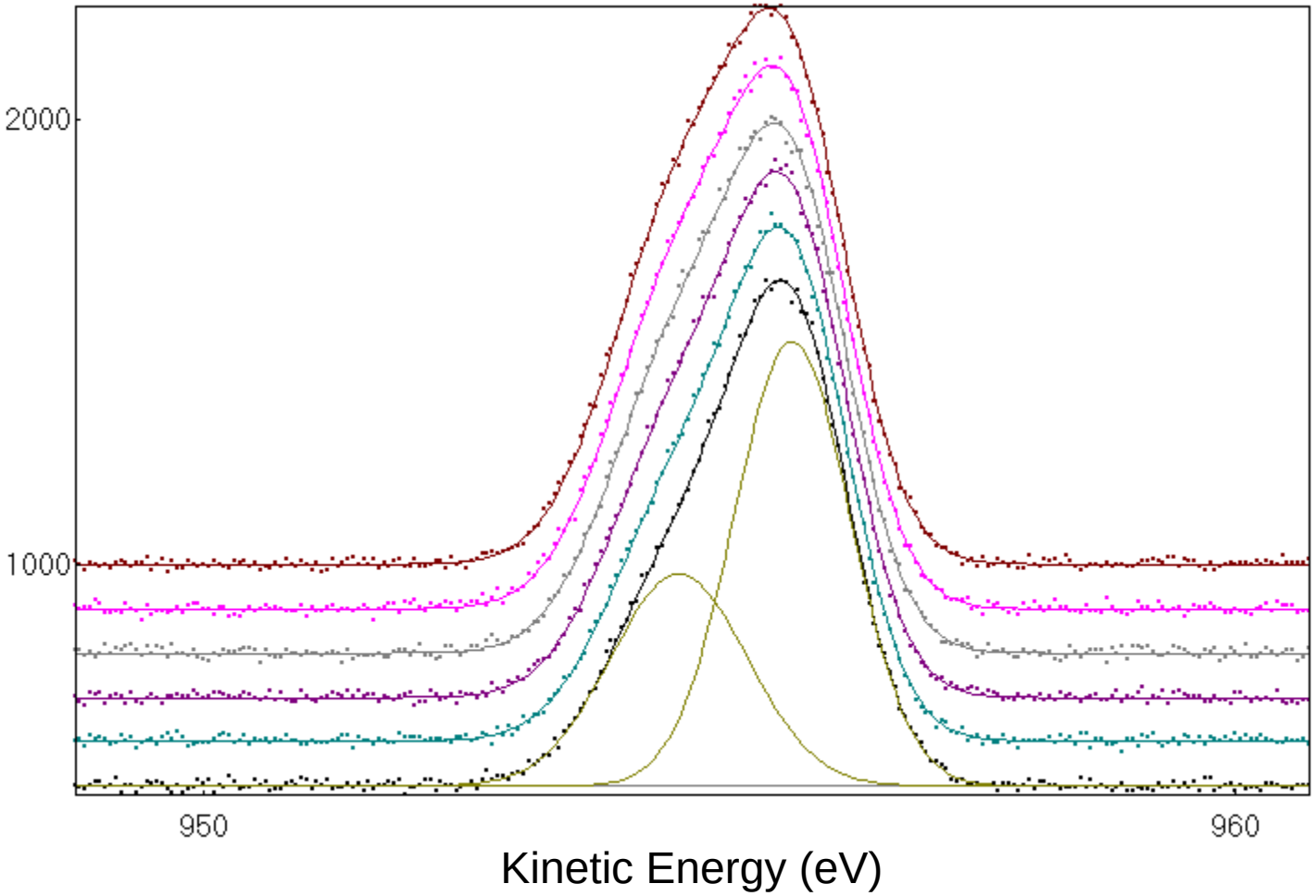

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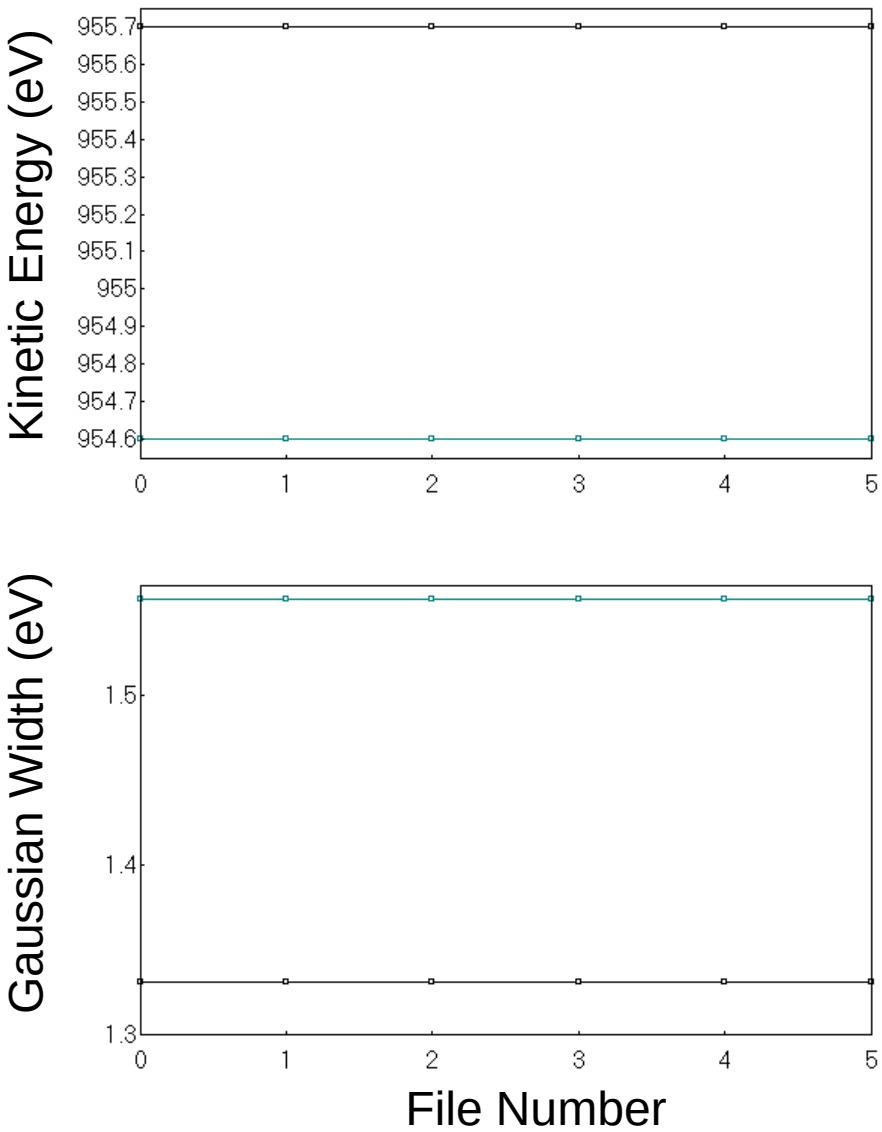

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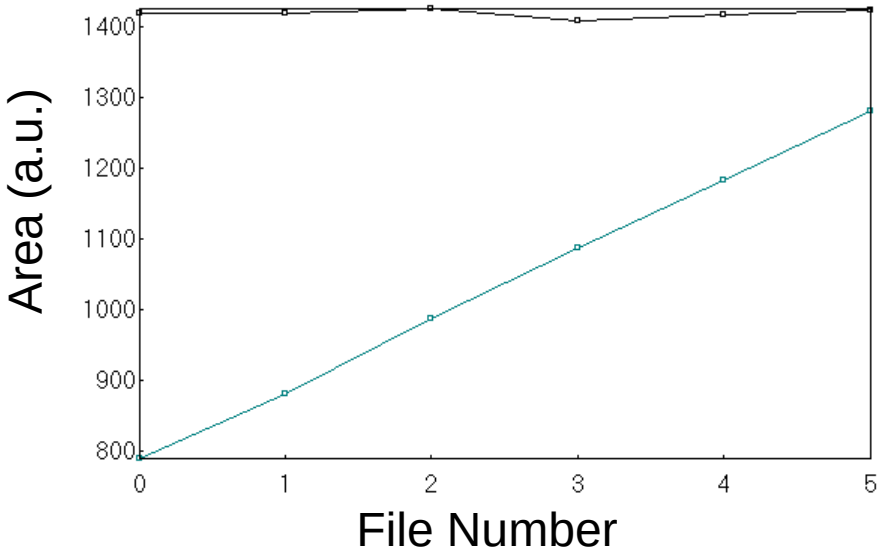

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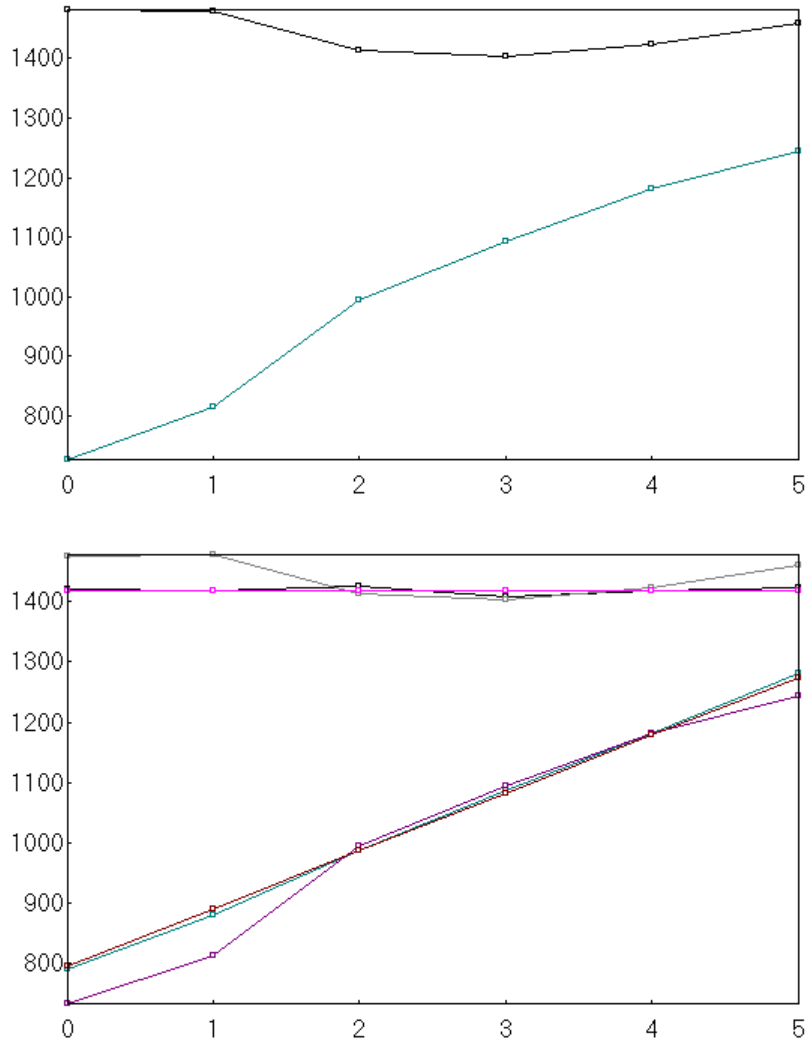
Simultaneous vs Sequential



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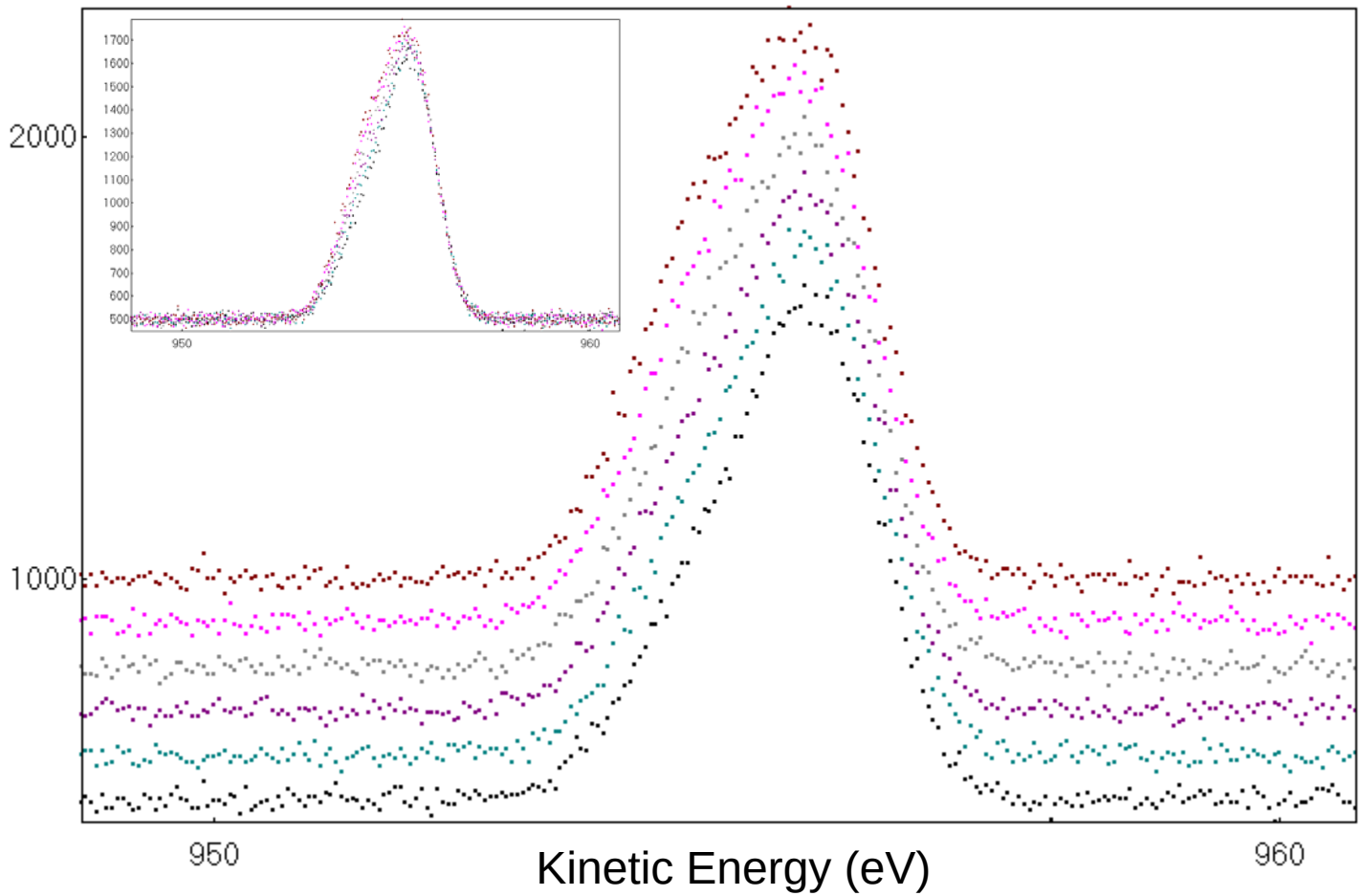
Simultaneous vs Sequential

What happens with slightly larger noise?



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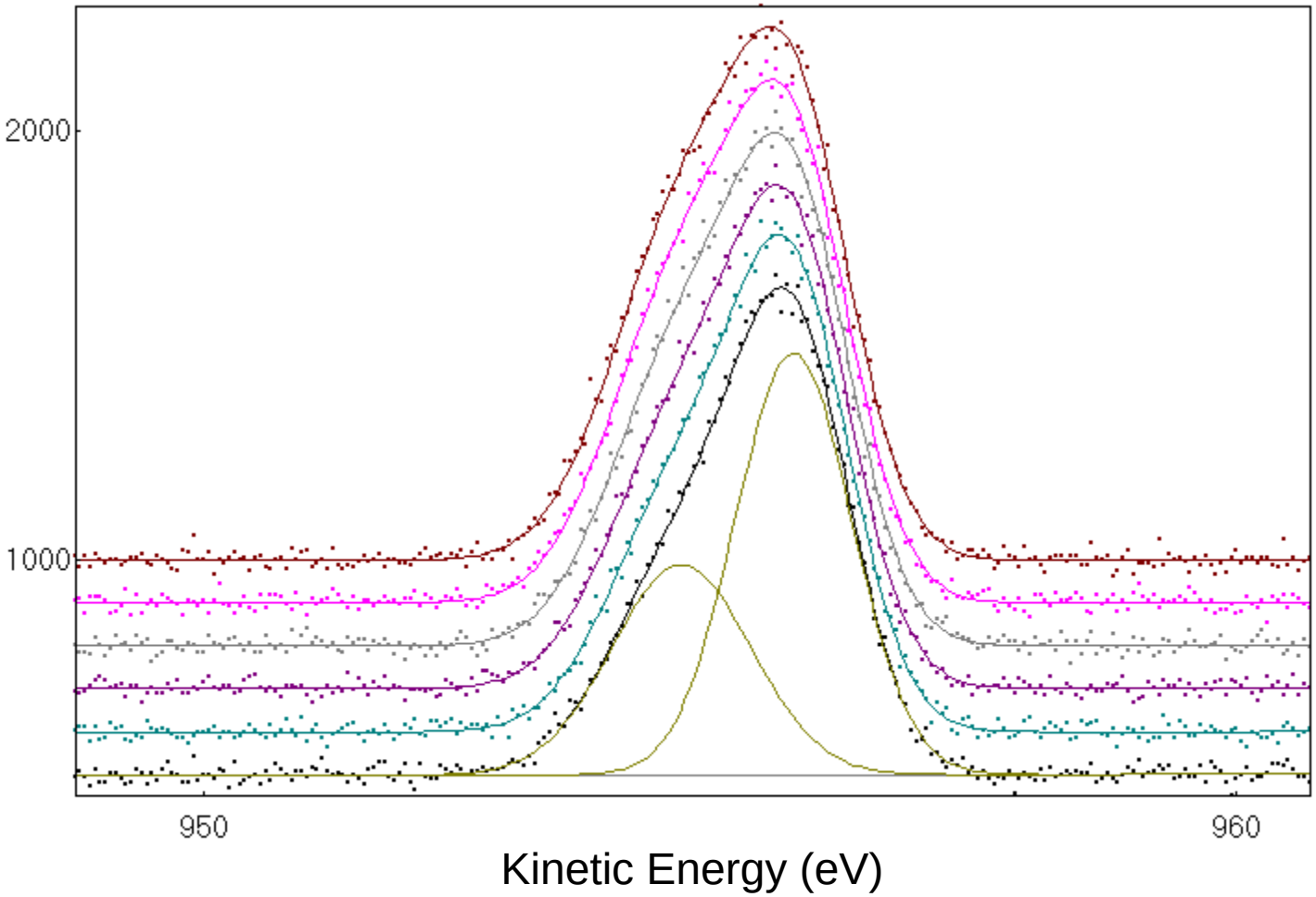
Simultaneous vs Sequential



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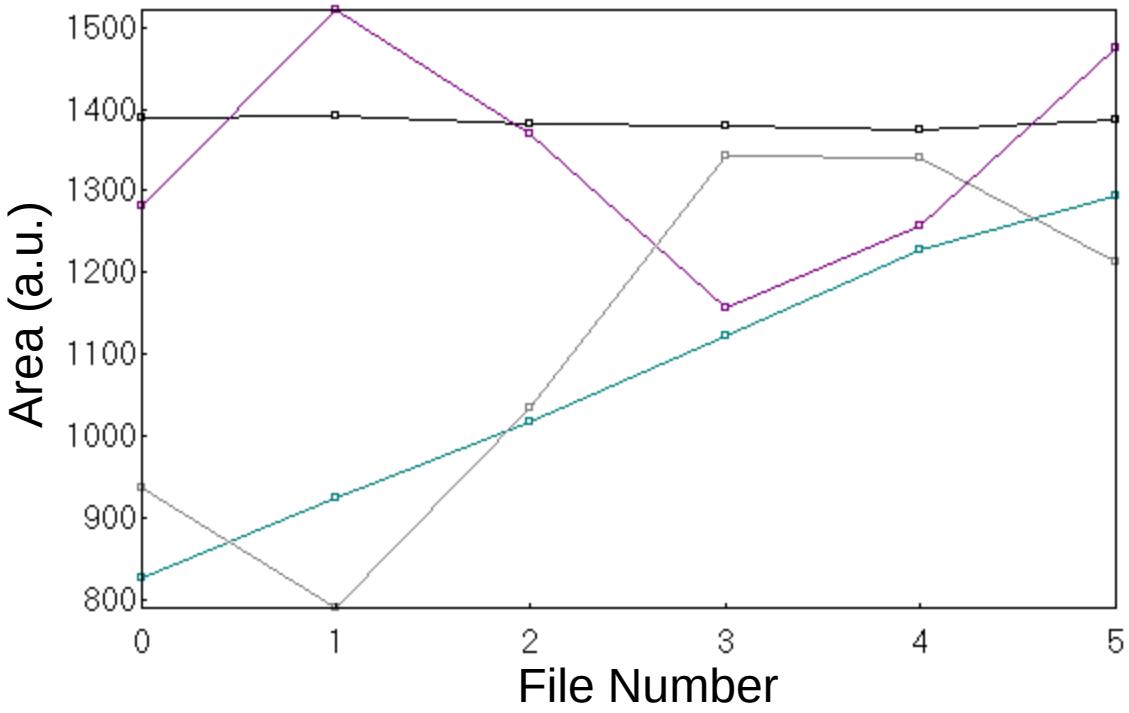
Simultaneous vs Sequential



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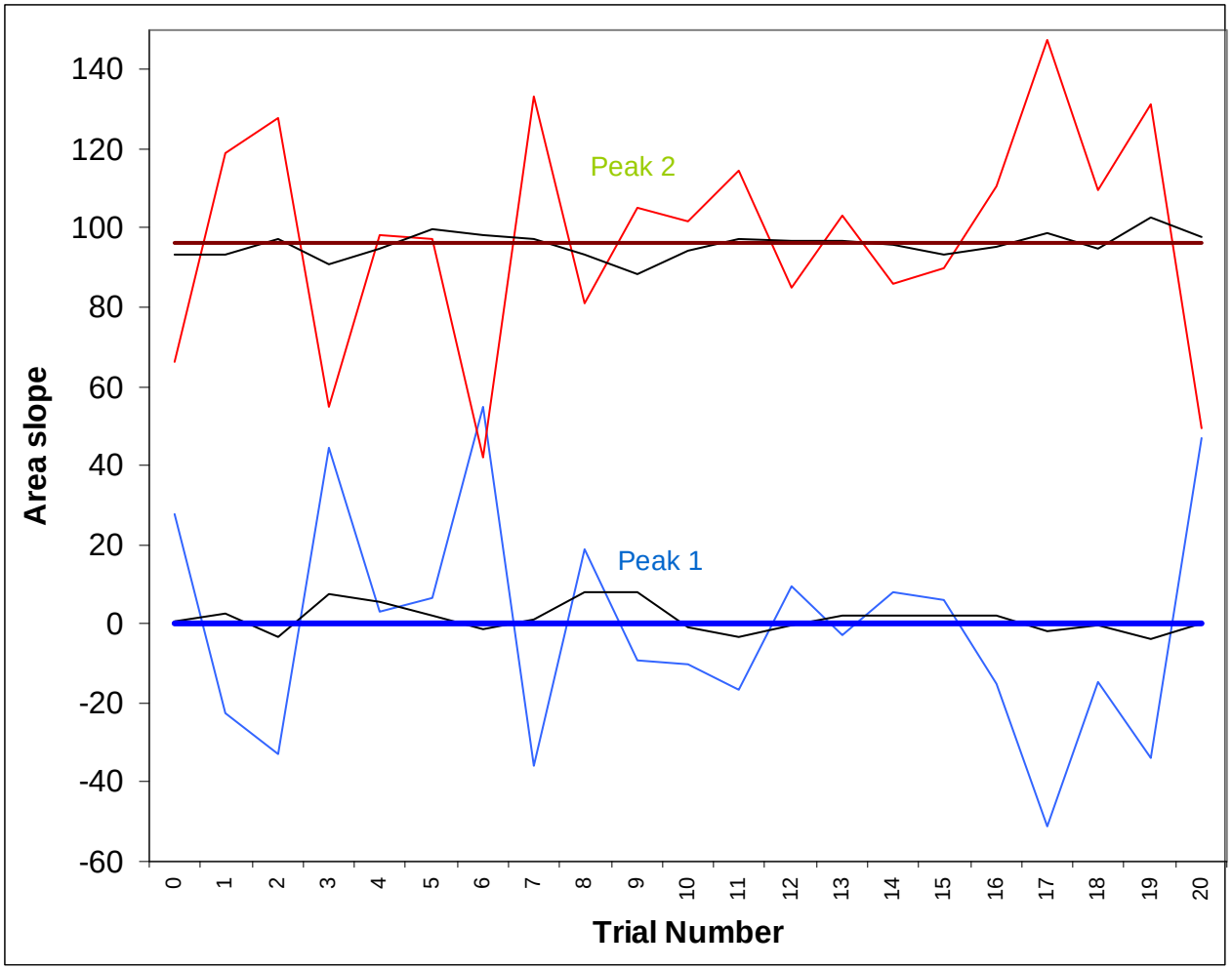
Simultaneous vs Sequential

The importance of noise in XPS data fitting

Noise 1



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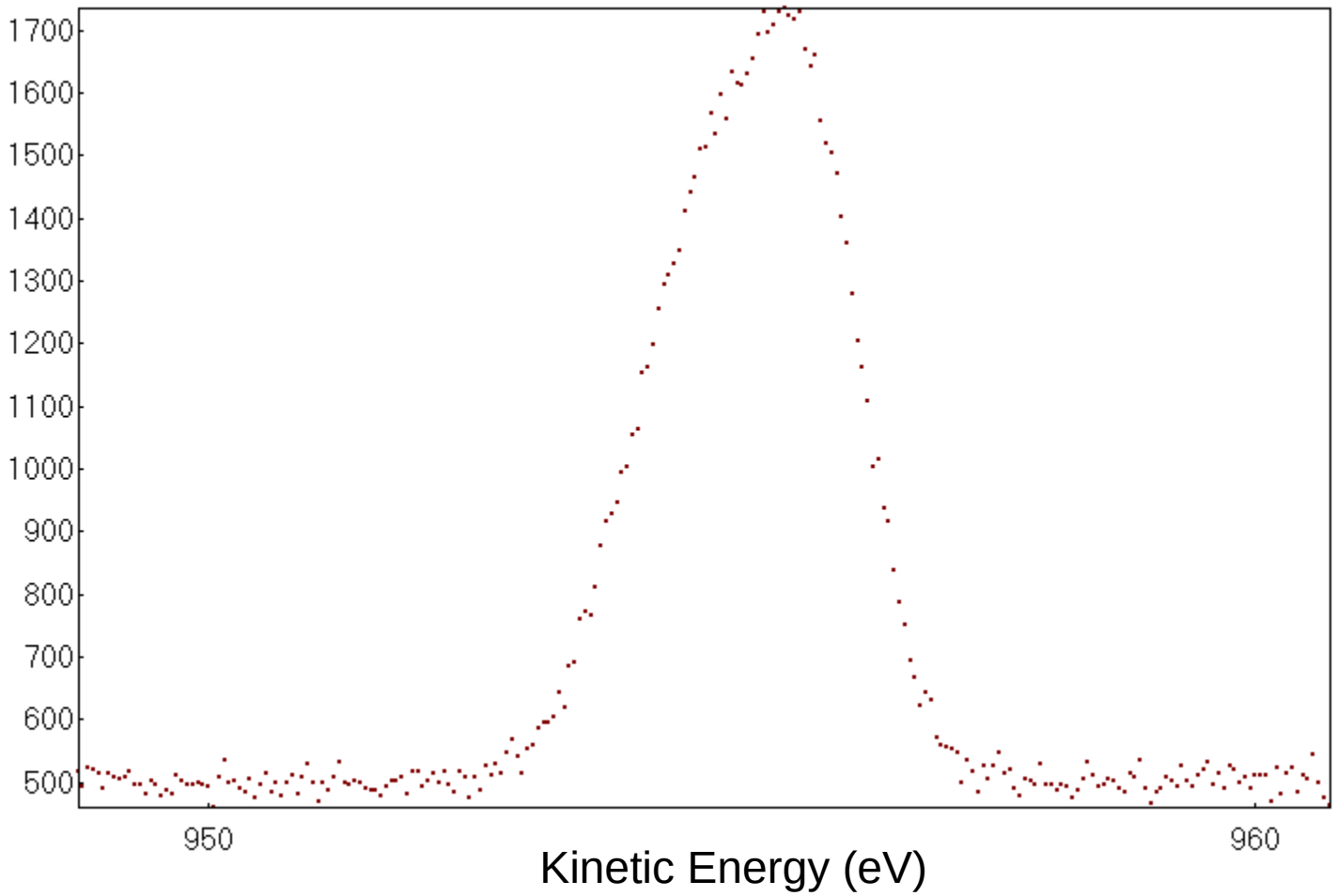
Simultaneous vs Sequential

Noise 1



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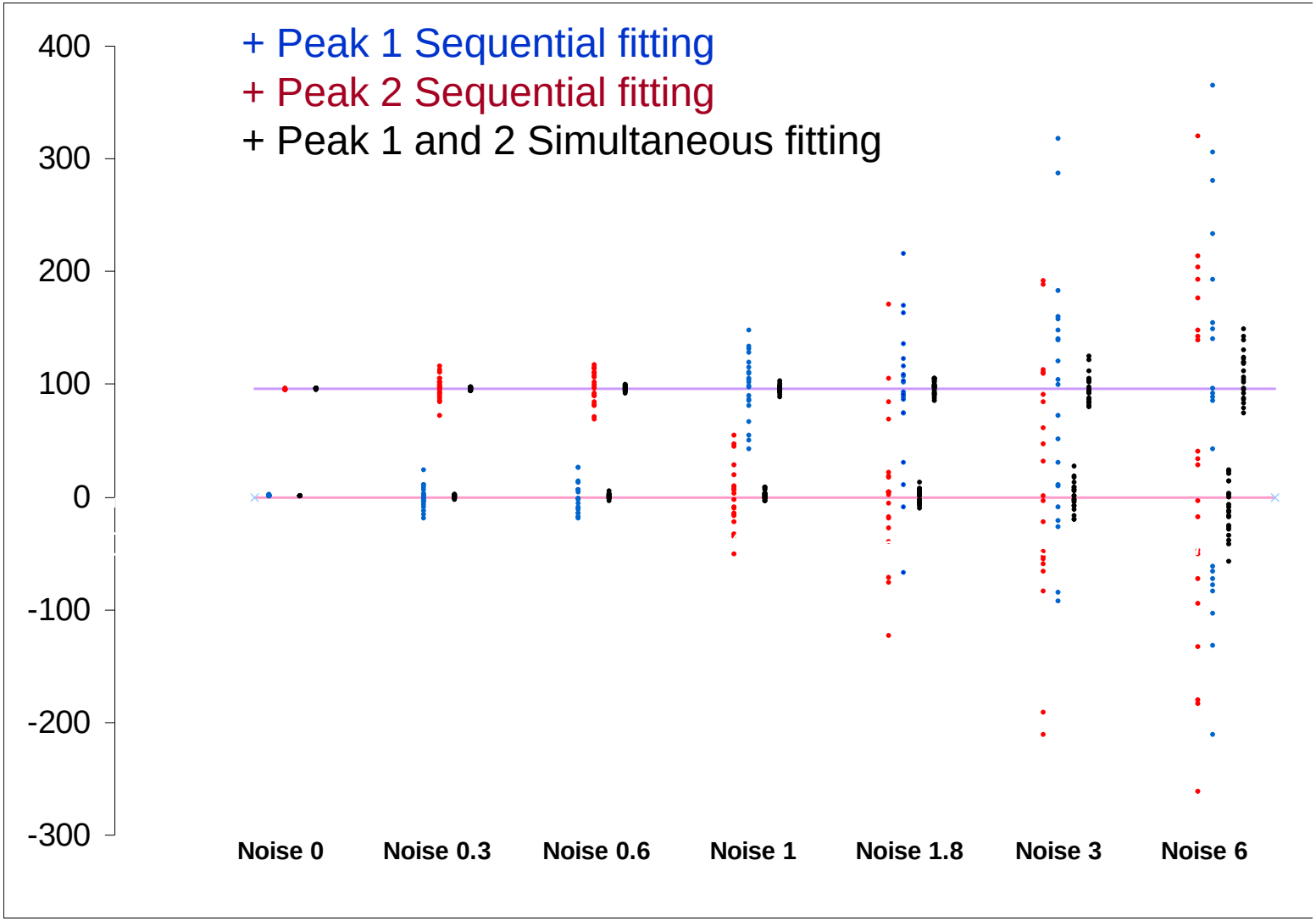
Simultaneous vs Sequential

The importance of noise in XPS data fitting

Slopes of Peaks 1 and 2



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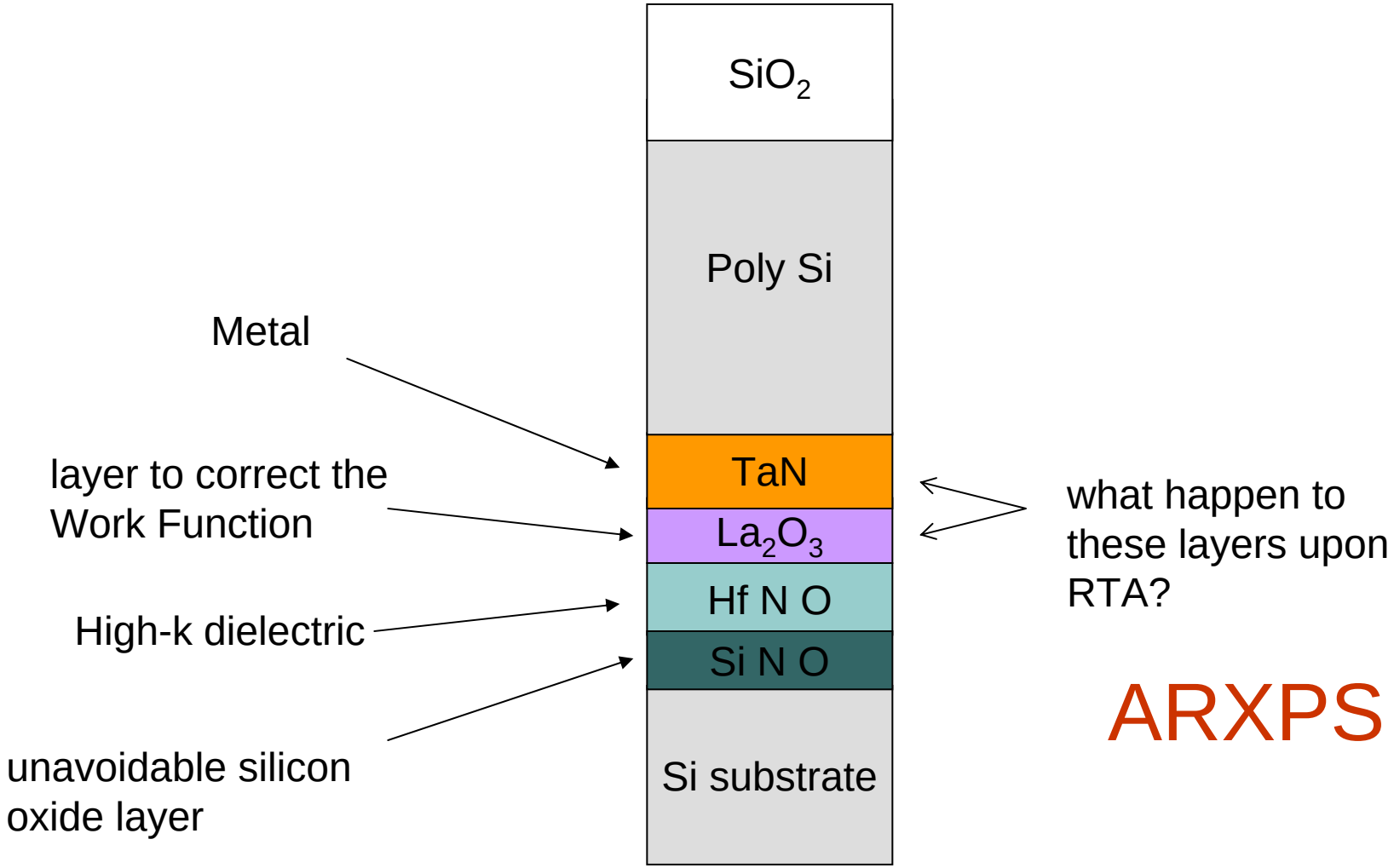
Application

Thermal stability of La in Hf-based MOS stacks

Thermal stability of dielectric stacks



ARXPS

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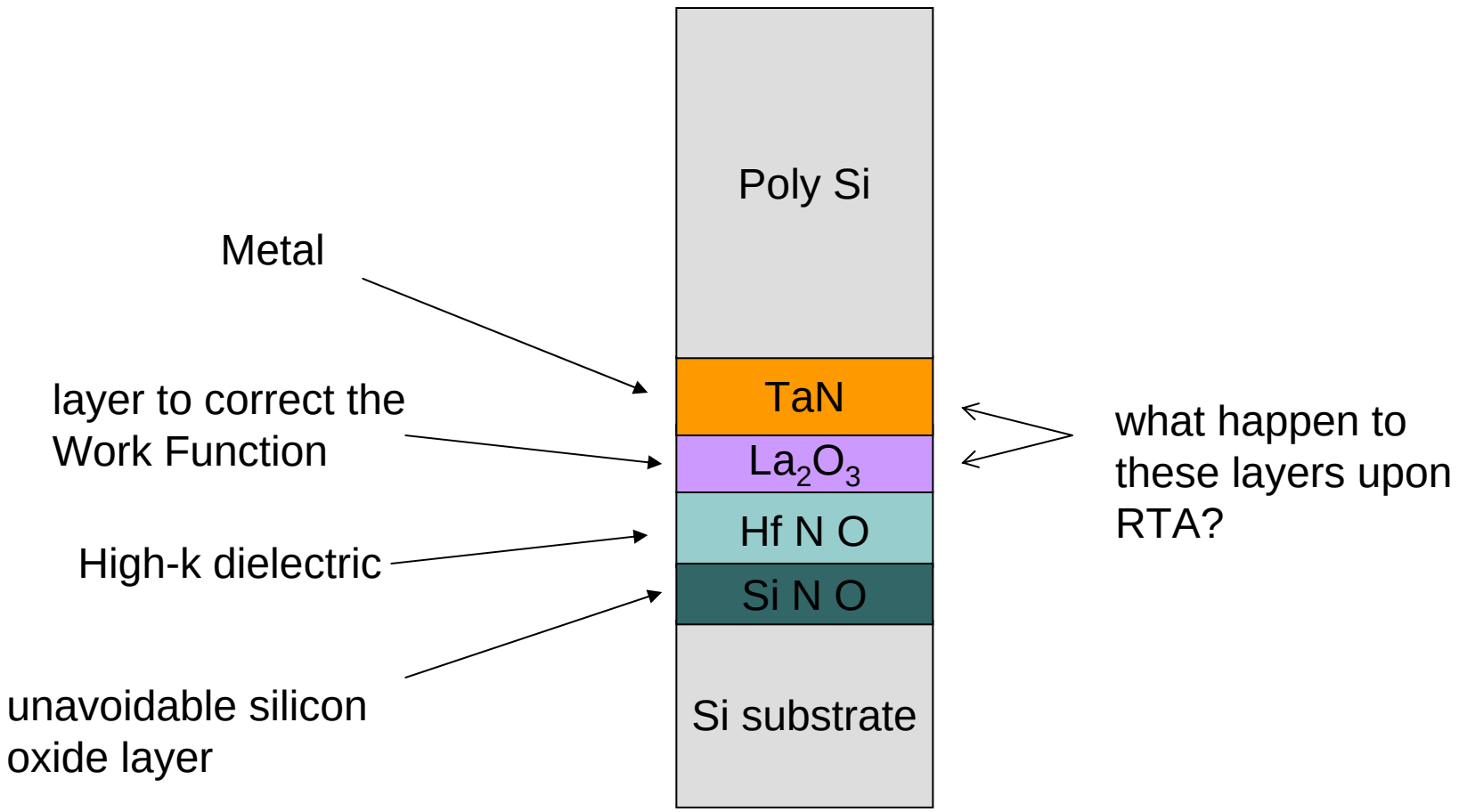
Application

Thermal stability of dielectric stacks



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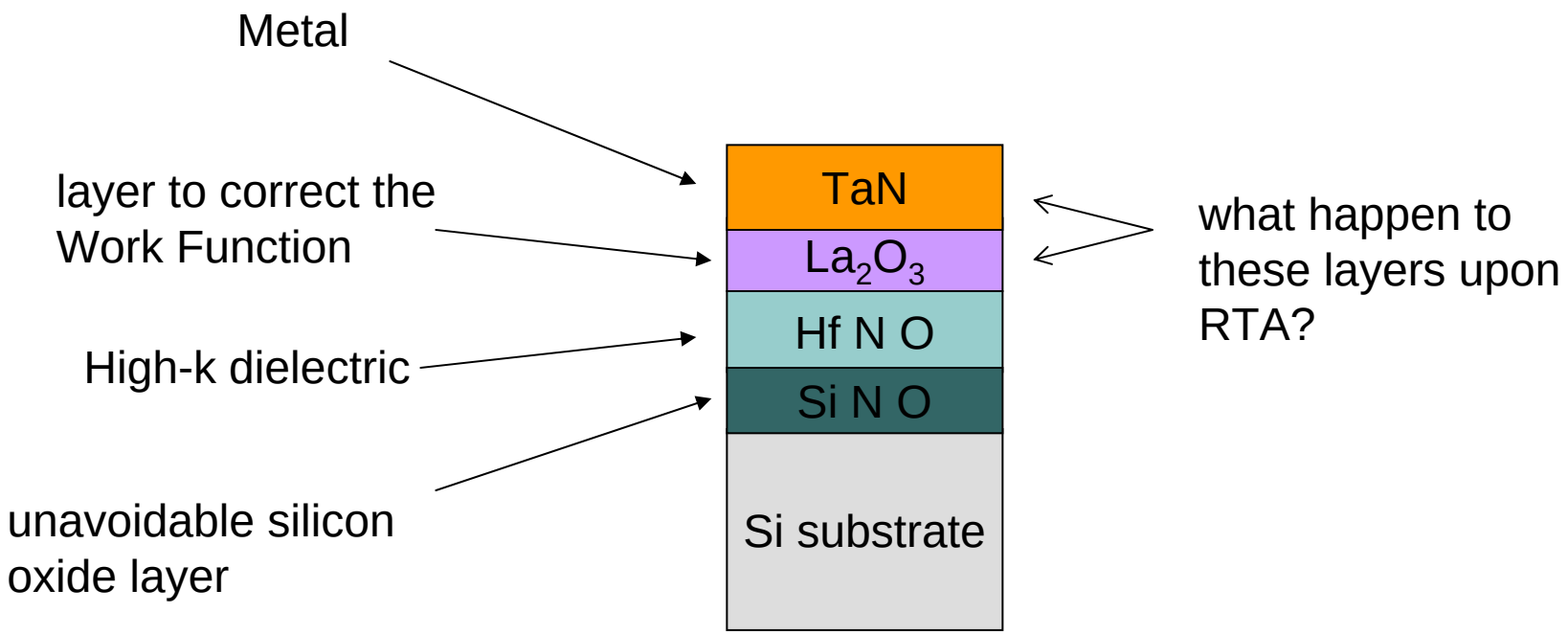
Thermal stability of dielectric stacks



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Thermal stability of La in Hf-based MOS stacks

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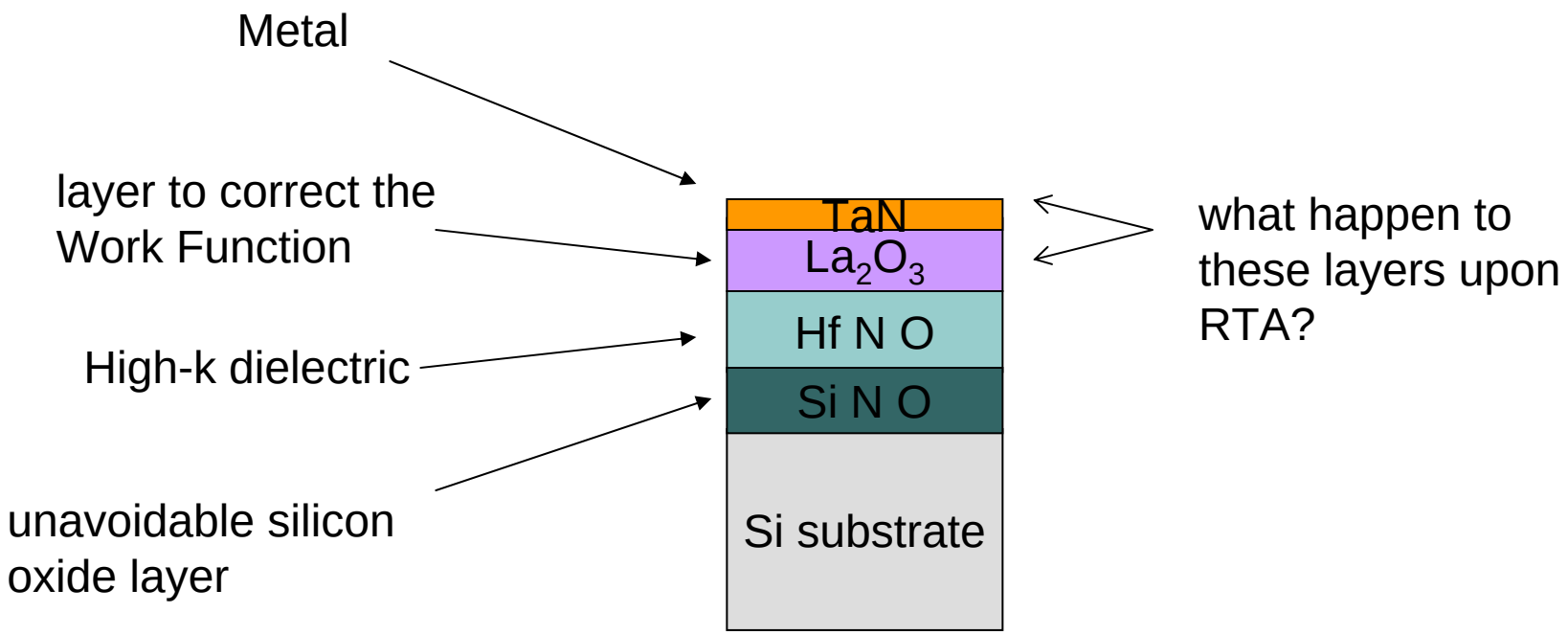

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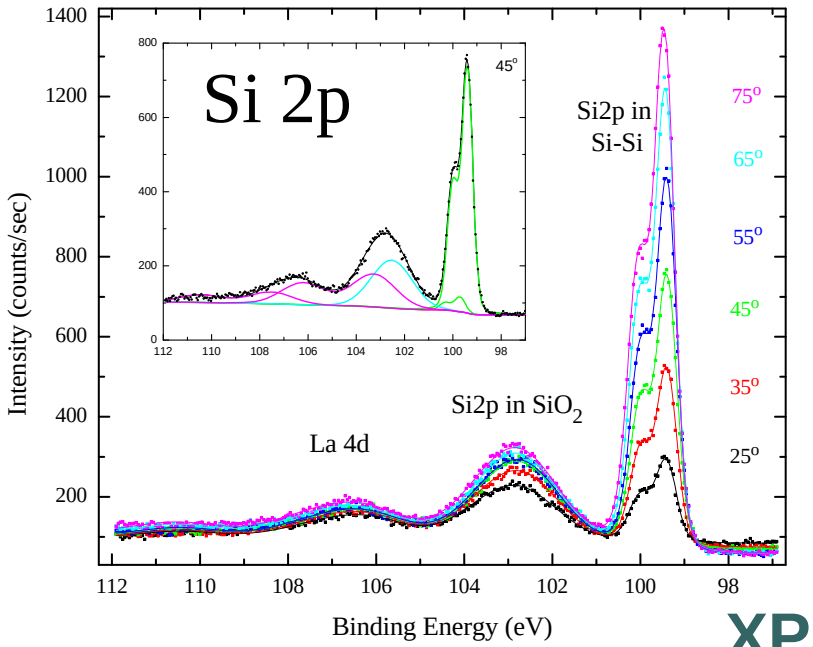
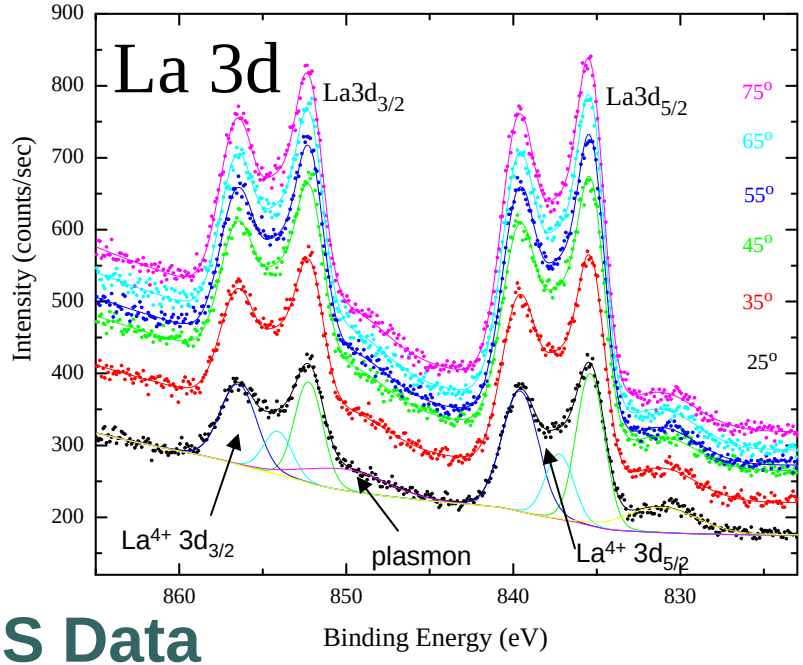
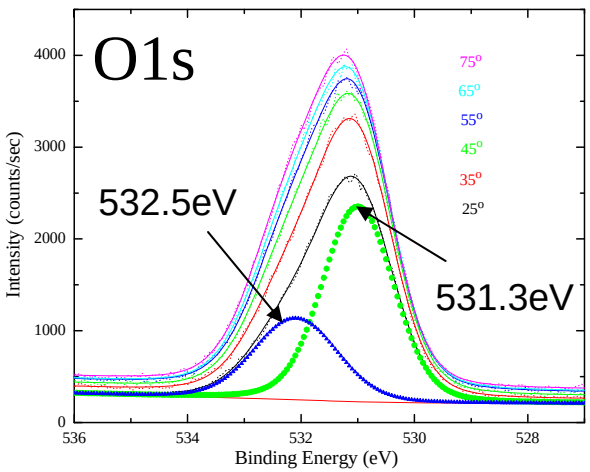
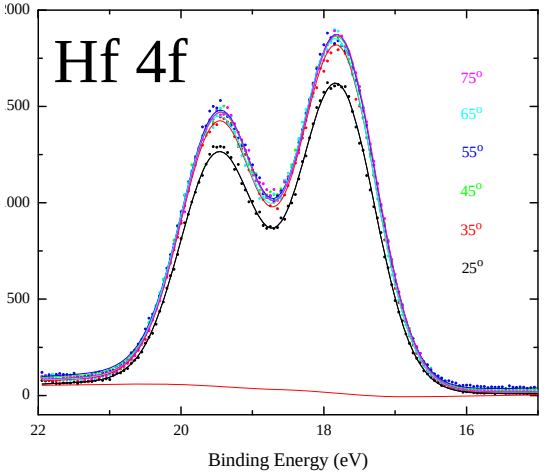
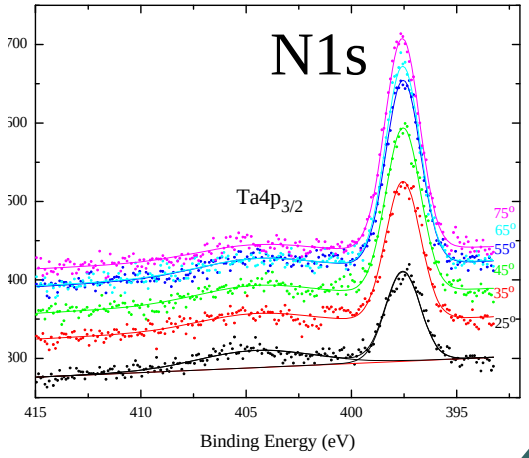




XPS Data







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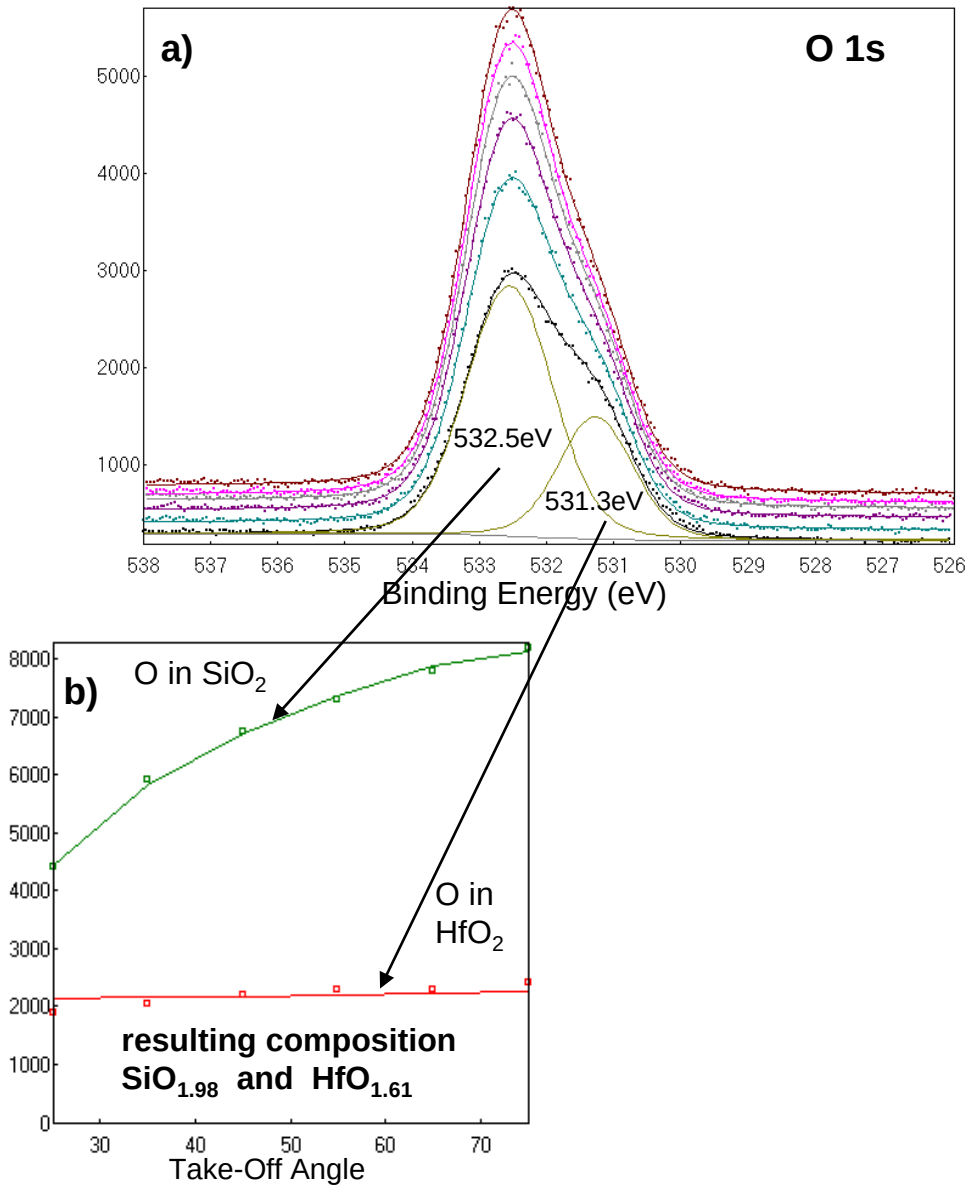
Finding the binding energy of O 1s in HfO_2 and SiO_2

Thermal stability of La in Hf-based MOS stacks



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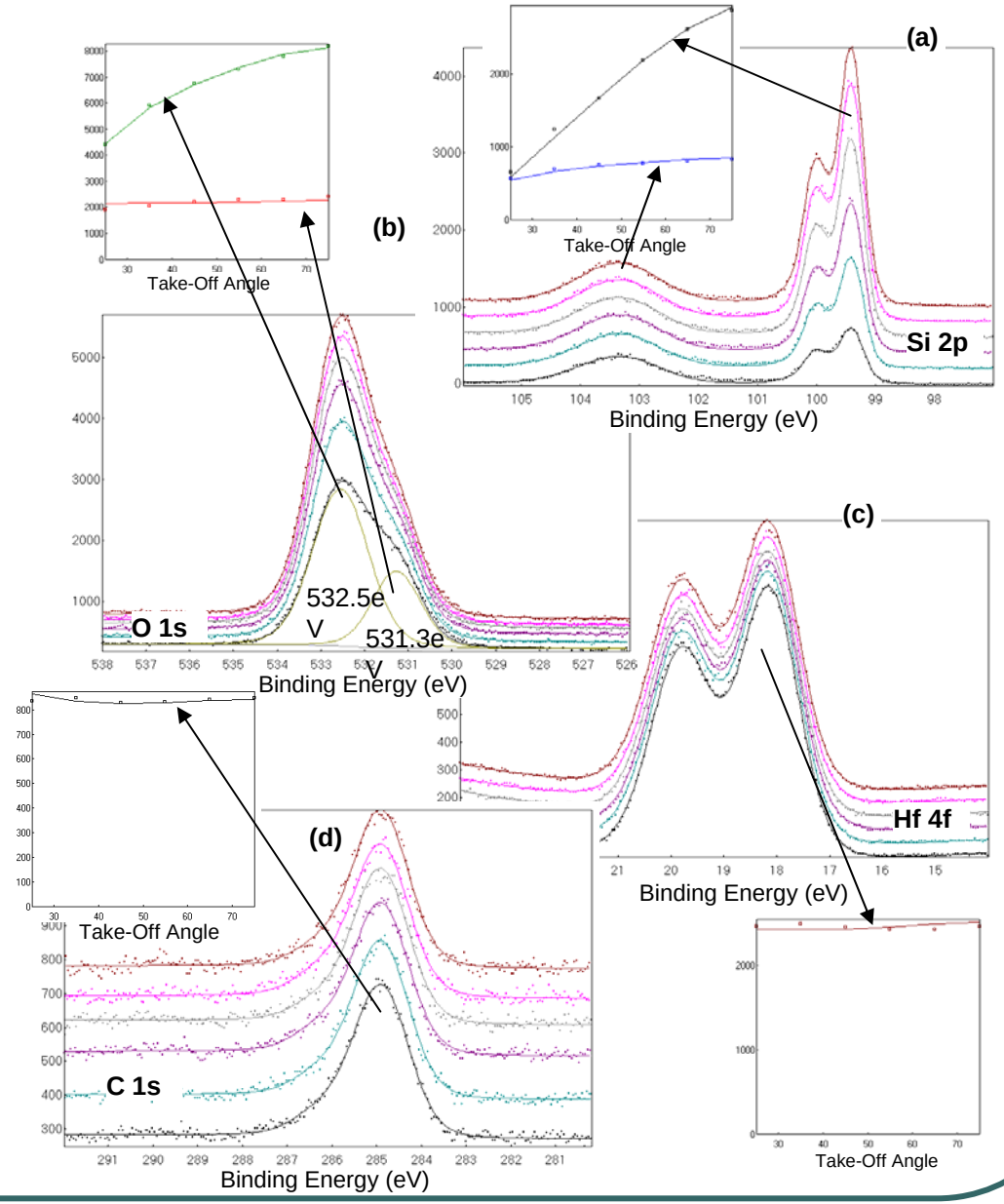
Dealing with straight or ratio data

The Sharp Interface Method

so far we have been dealing with the peak areas directly



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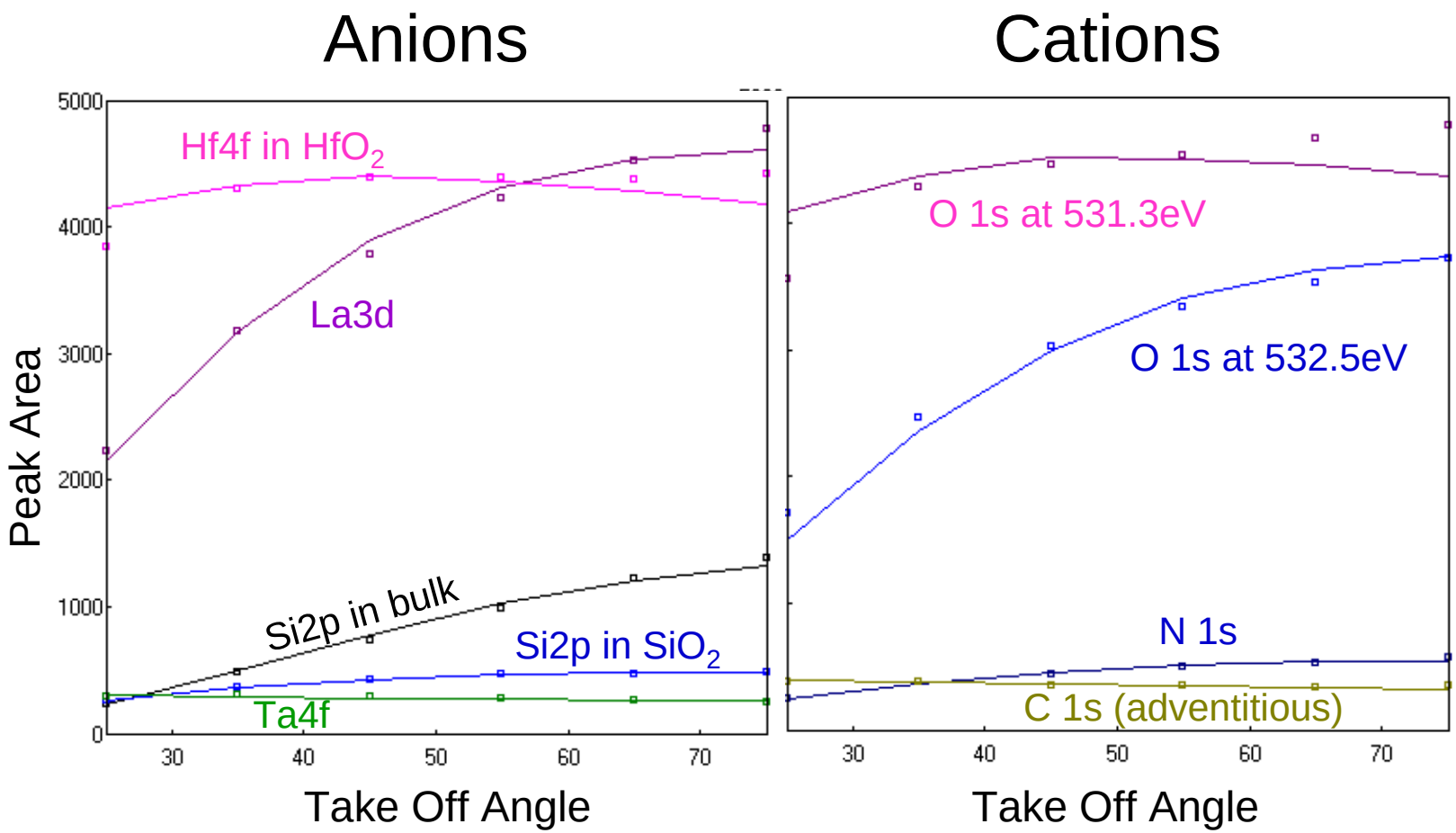
Estabilidad térmica de multicapas

Algunos ejemplos de aplicación de ARXPS

ARXPS Data

The take-off angle dependence of the peak areas (*ARXPS Data*)



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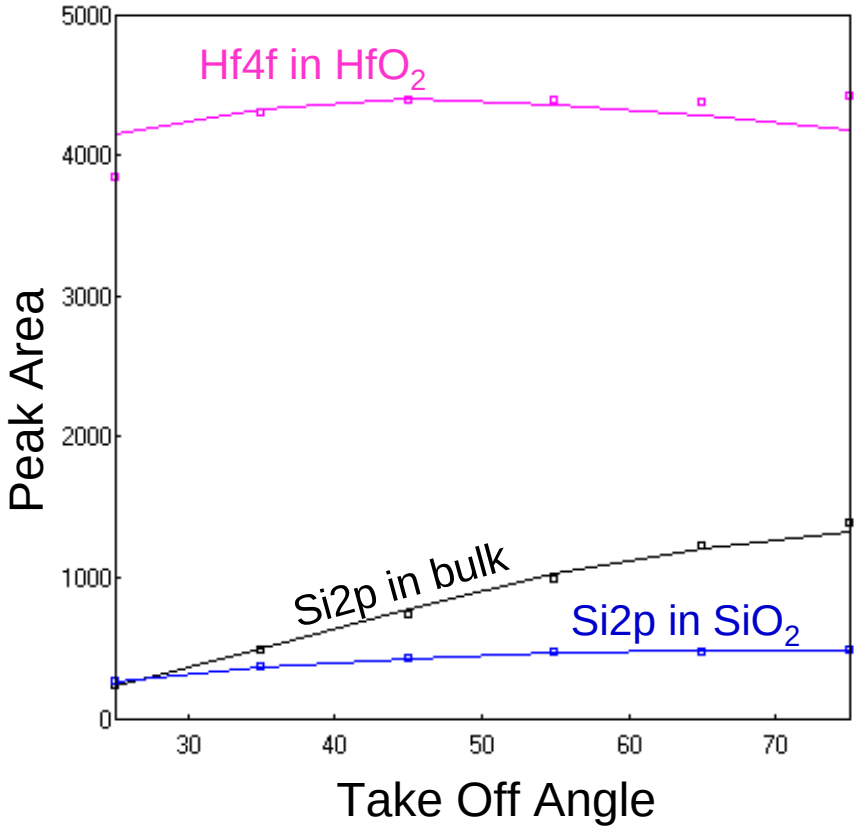
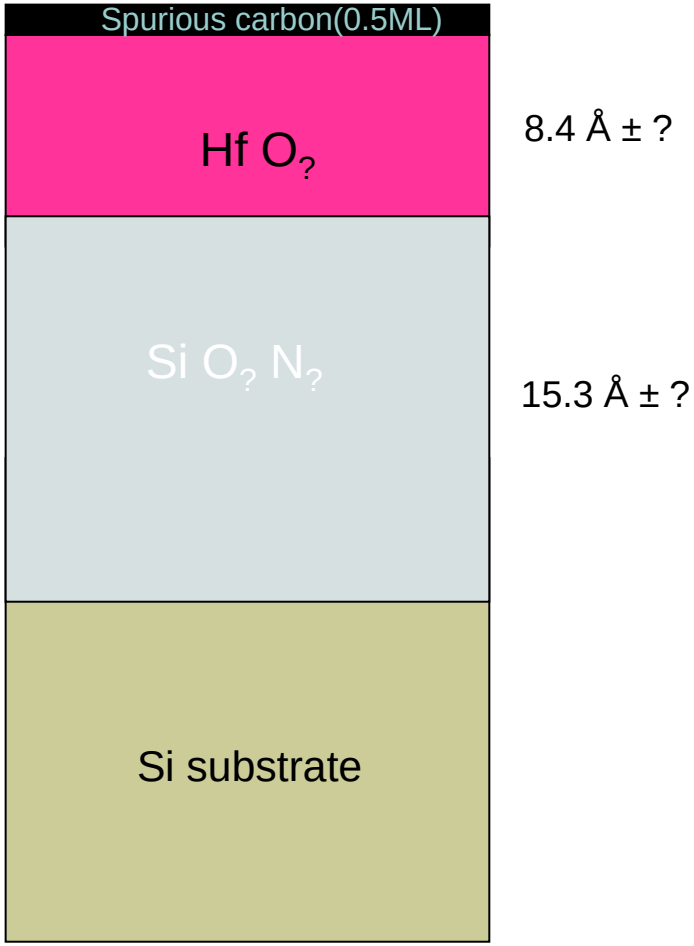
Anions



MS4+RTA



Thickness assuming bulk density

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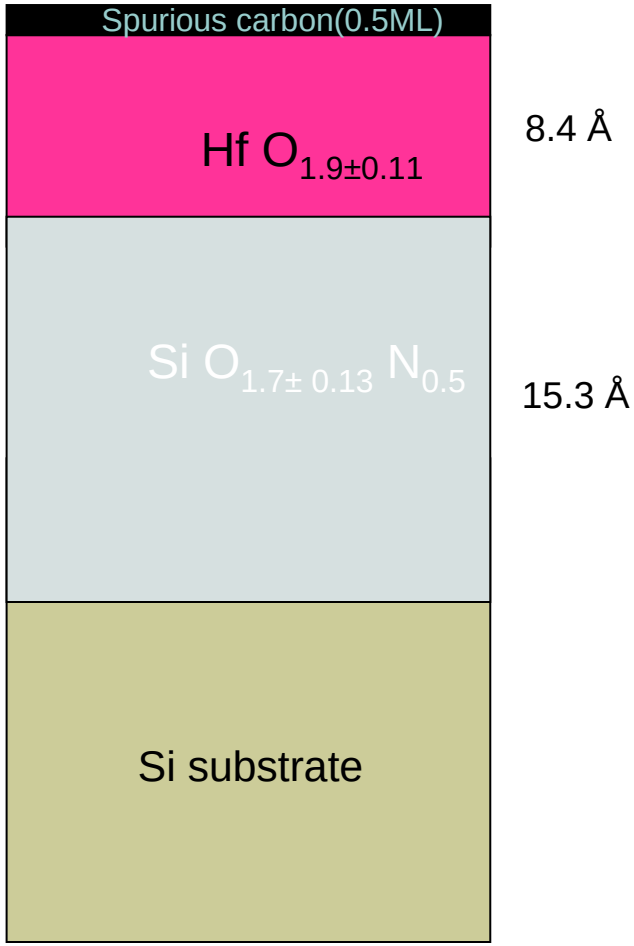
Estabilidad térmica de multicapas

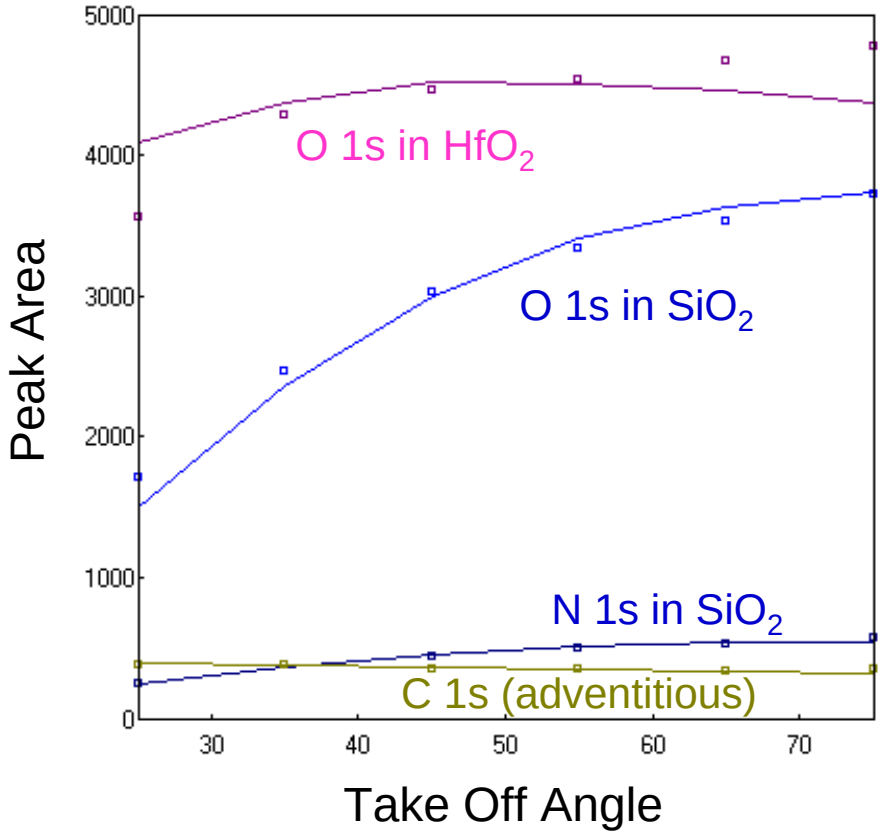
Composition found from the cation data

Algunos ejemplos de aplicación de ARXPS

MS4+RTA



Cations



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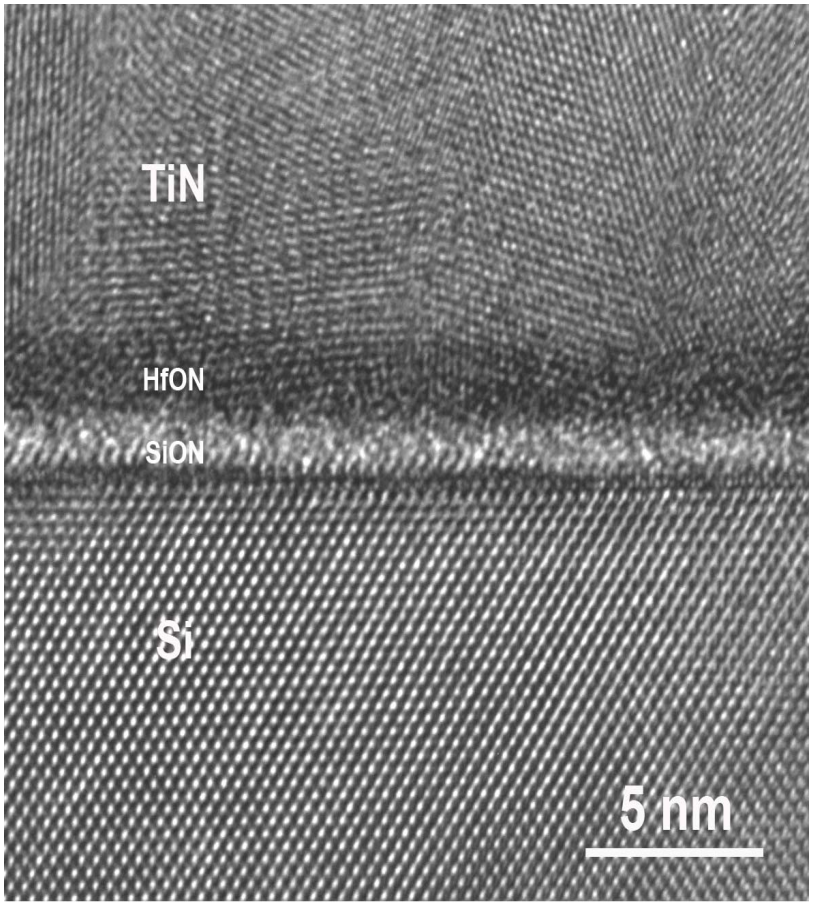
Going around ill-conditioning

The Sharp Interface Method (*SIM*)

Extra information from TEM images



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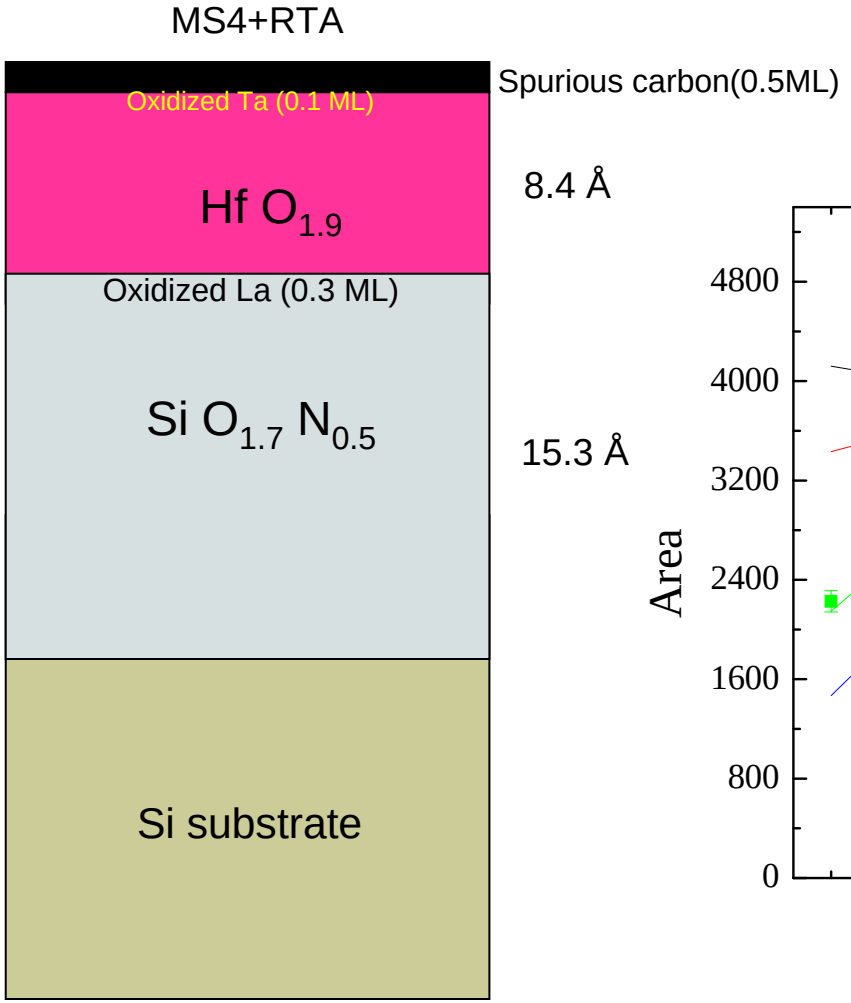
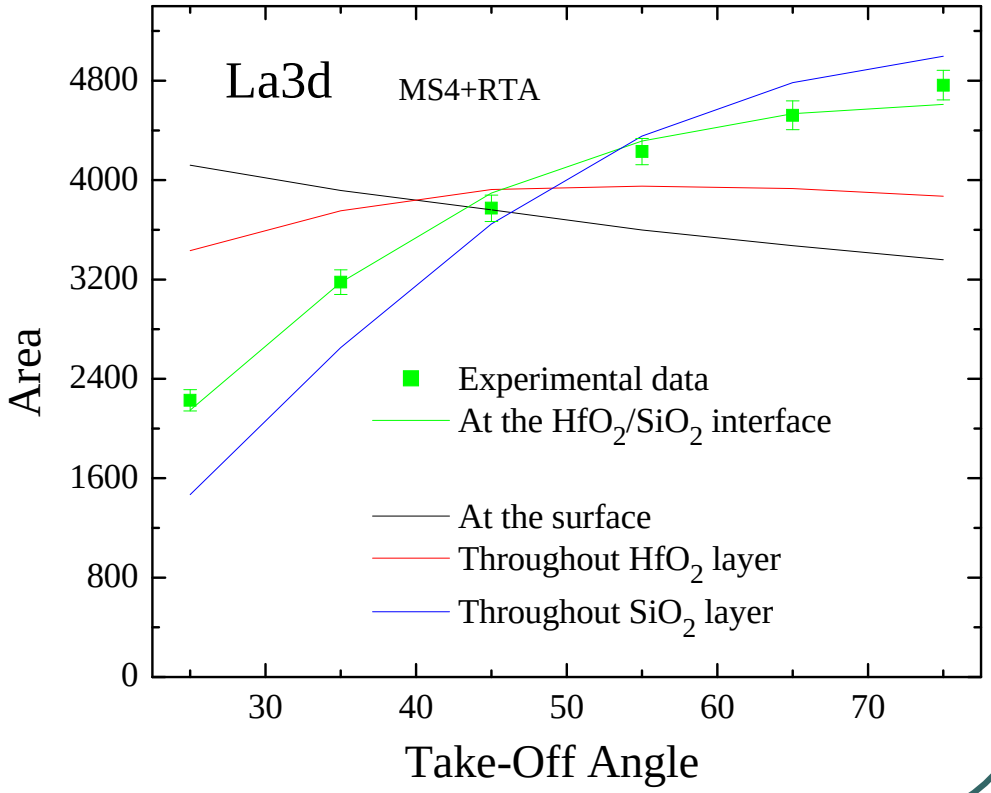
Algunos ejemplos de aplicación de ARXPS

A “difficult problem”: the position of La



La amount and position



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- The characterization of the La depth profile, is a challenging problem. ARXPS is the ideal technique because it provides both chemical and depth profile information.
- The noise could be a killing factor in ARXPS analysis.
- For XPS data peak fitting, simultaneous analysis is (much) more robust than sequential analysis.
- For the recovery of the depth profile from the angle dependence of the peak areas, the parametric methods (such as the Multilayer Model) is more robust and less dependent on noise than the back transform or regularization methods (such as Maximum Entropy).
- By employing robust methods for both XPS data analysis and depth profile analysis, it is possible to recover the structure of the films.
- La diffuses into the HfO2/SiO2 interface

Thank you!