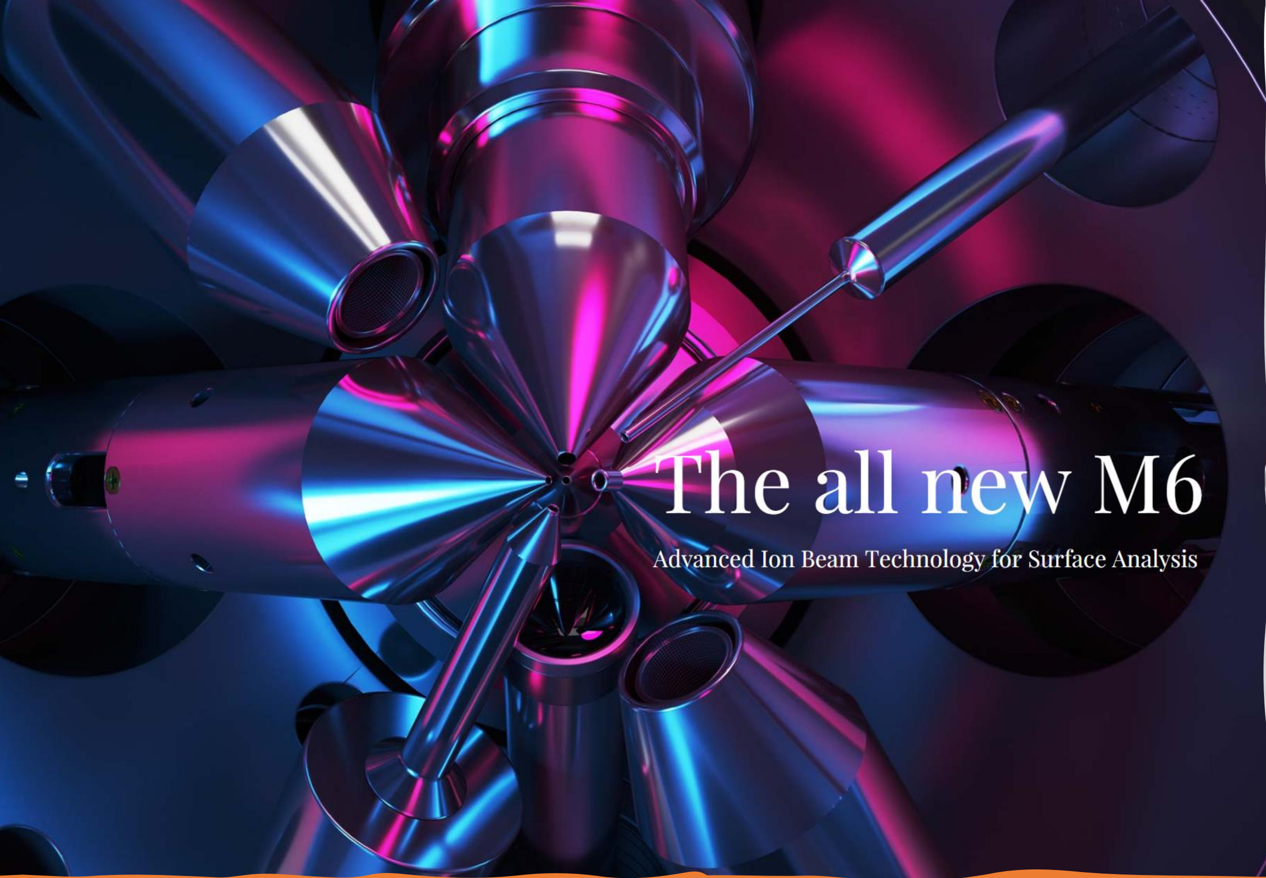
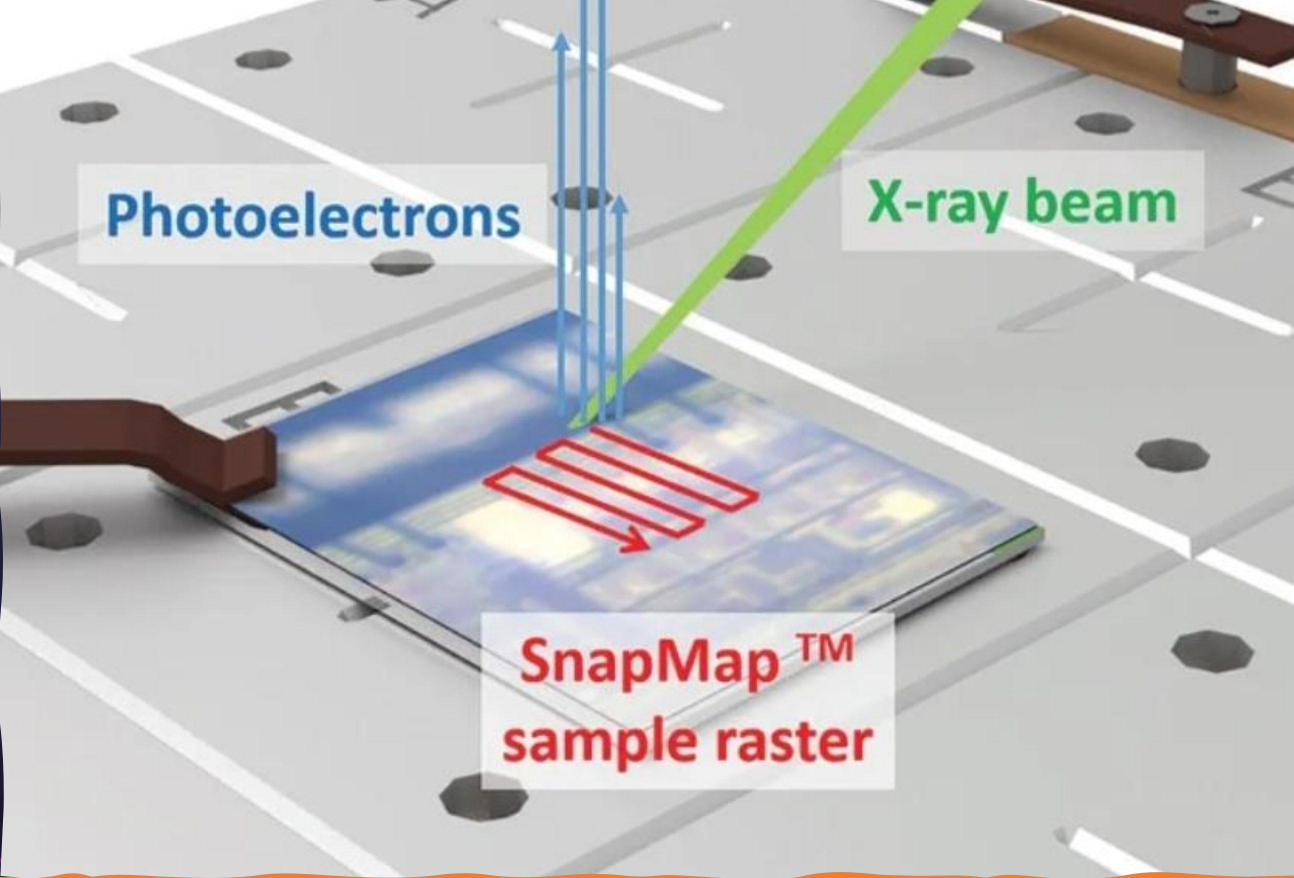




Depth profiling and mapping with XPS and SIMS

- **Xinqi Chen**
- **Nuance Center**
- **Northwestern University**

Destructive depth profiling

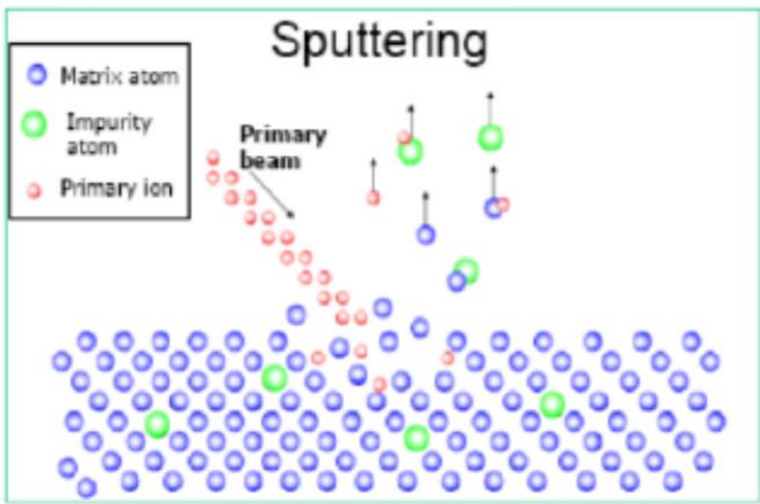
Acquire XPS spectrum

Ar ion sputtering

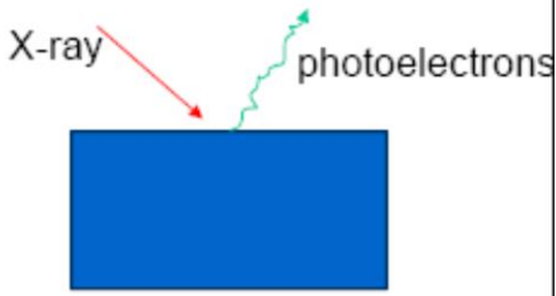
Acquire XPS spectrum

Ar ion sputtering

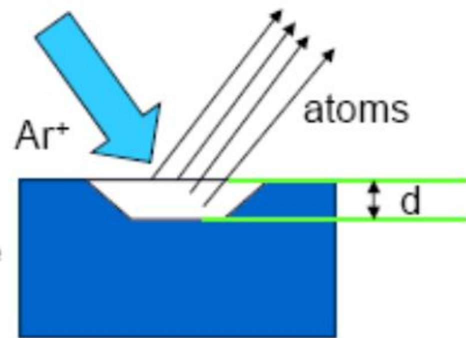
Acquire XPS spectrum



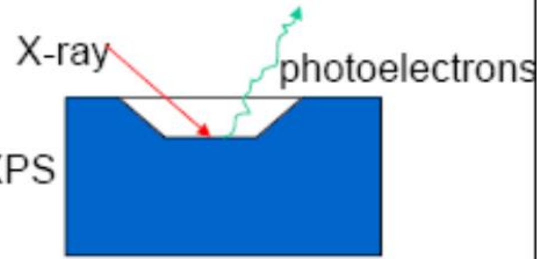
Acquire XPS



Sputter surface



Acquire XPS



XPS:

Sputter beam

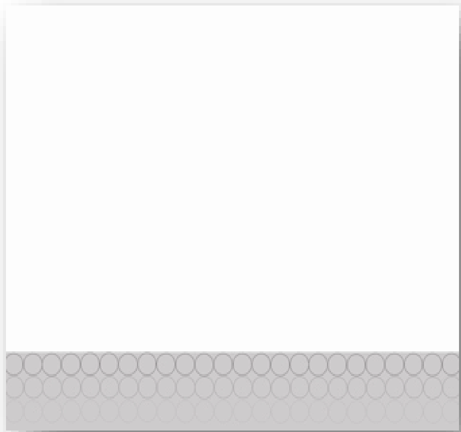
- Ar monatomic gun
- Cluster gun

SIMS:

Sputter beam

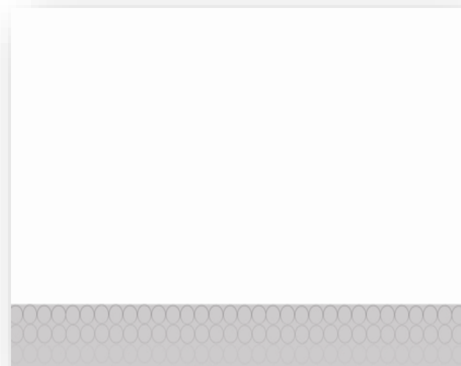
- O_2 gun for positive ion
- Cs gun for negative ion
- Cluster gun for organic samples

Cluster ions v monatomic ions



Monatomic ions (Ar^+)

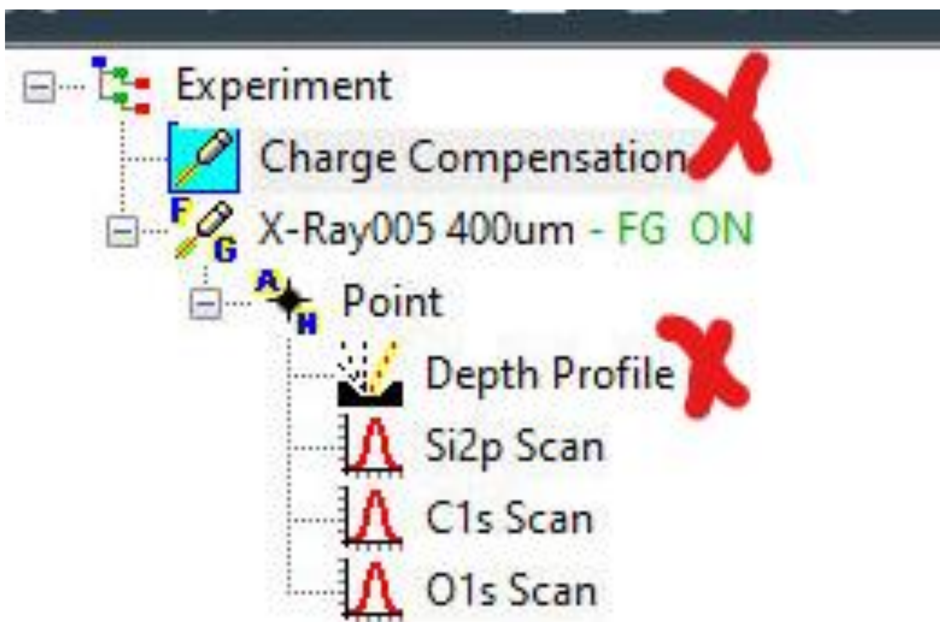
- High energy per atom (200eV – 4keV)
- High etch rate
- Deep surface penetration
- **Can damage surface chemistry**
- Ideal for etching inorganic material

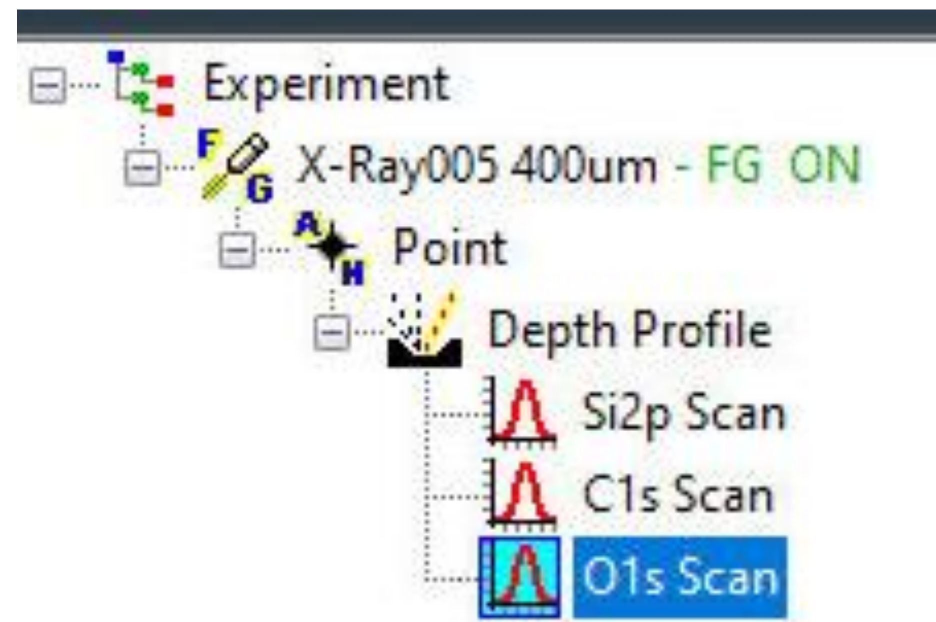


Cluster ions (Ar^+_n)

- Low energy per atom (1eV – 100eV)
- Minimal surface penetration
- **Non-damaging to surface chemistry**
- Low etch rate for large clusters
- Large clusters ideal for etching organic material

XPS Depth Profiling Setup





Profile | Ion Gun | Etch Phases | Etch Position | General

Ion Energy 3000 eV
Mode Monatomic
Current: Low High
Sputter Rate Estimate: Ta2O5 = 0.49 nm / sec
Raster Size: Auto Manual
2.00 mm
Blank X-ray gun during etching
Turn gun off when finished

Depth Profile Object

Profile | Ion Gun | Etch Phases | Etch Position | General

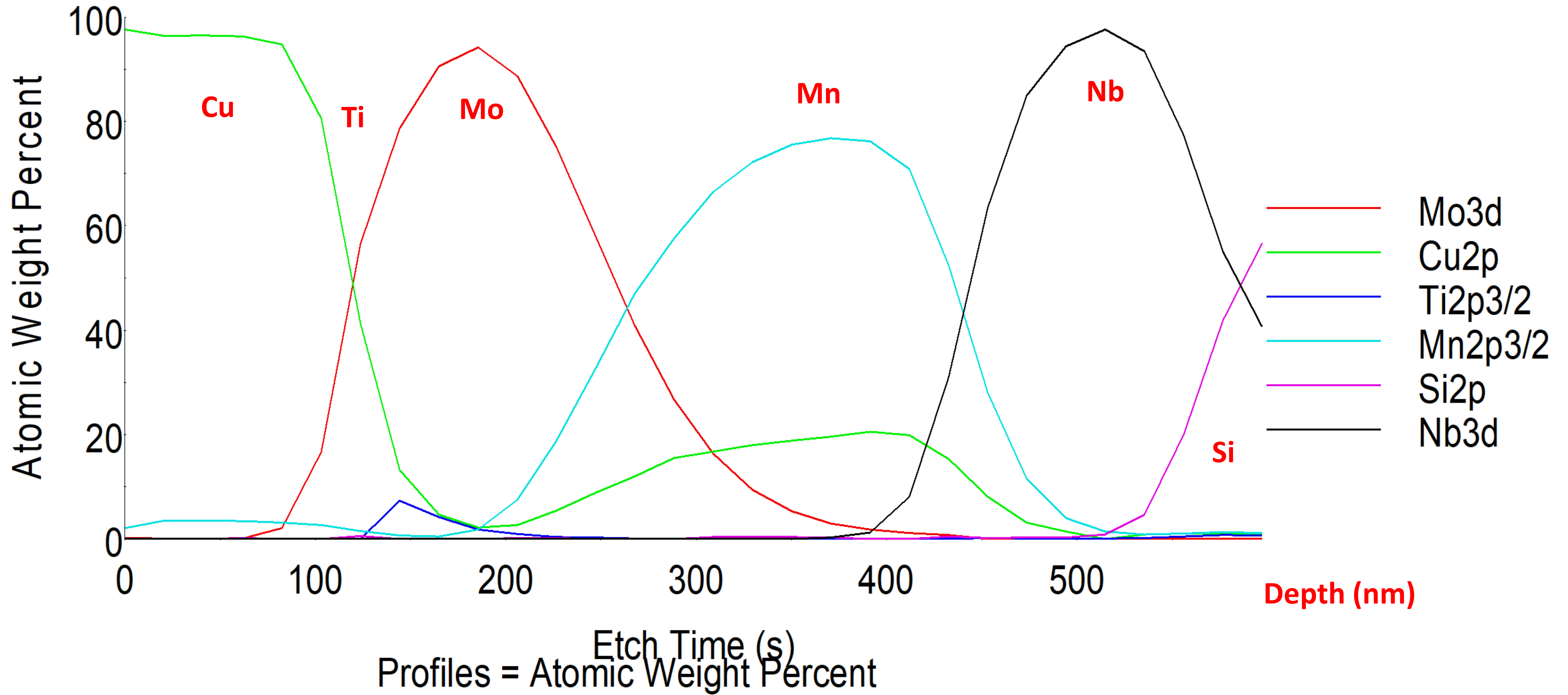
Ion Energy 6000 eV
Mode Cluster
Cluster Size 1000
Raster Size: Auto Manual
2.00 mm
Blank X-ray gun during etching
Turn gun off when finished

Depth Profile Object

Profile | Ion Gun | Etch Phases | Etch Position | General

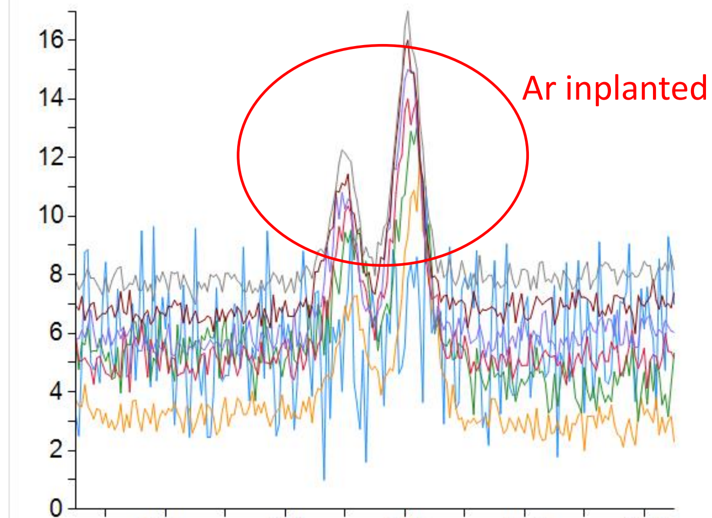
Etching: Move to etch position; Single Phase Etching; Delay after Etch (s) 1
Rotation: None; Azimuthal; Compucentric
Etch Cycle: Time (s) 60; Rotations 1
Termination Condition: Total Levels 20; Total Etch Time 0 Hour 19 Min 0 Sec
Test Rotation | Stop

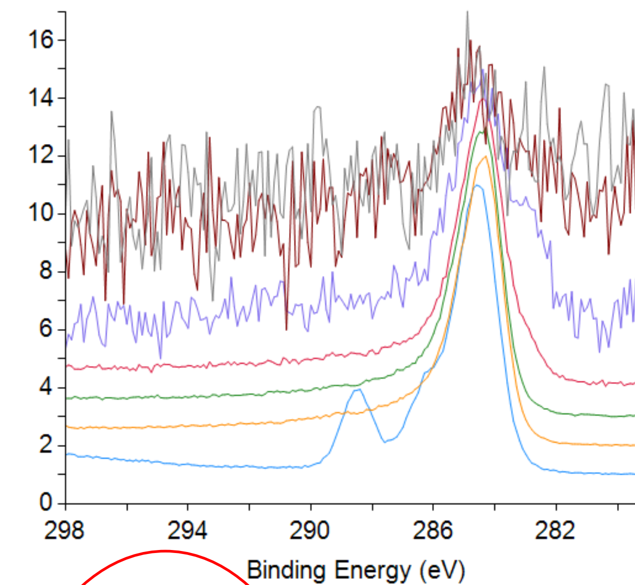
XPS depth profiling

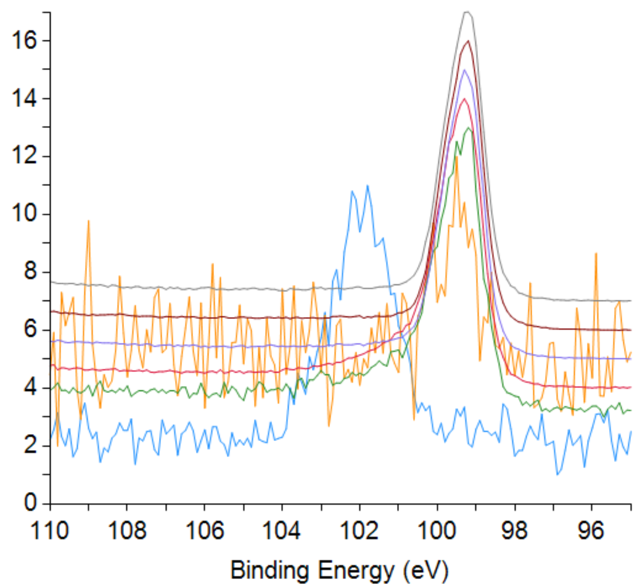


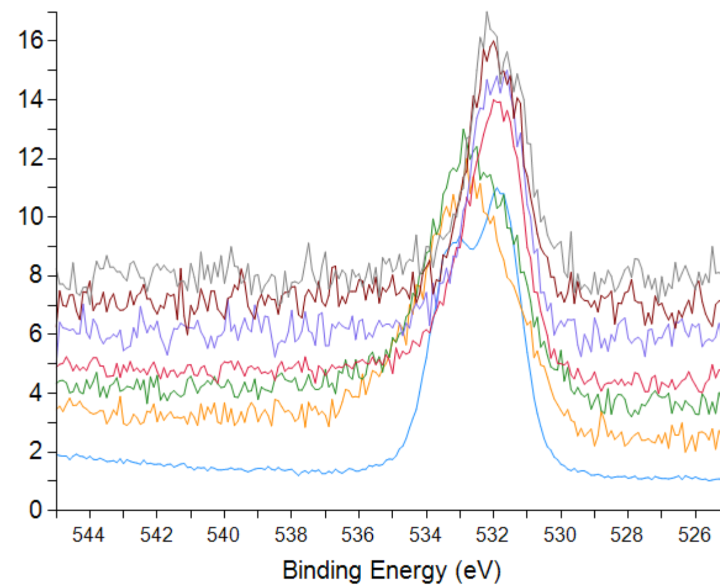
PMMA/Si: Depth profiling

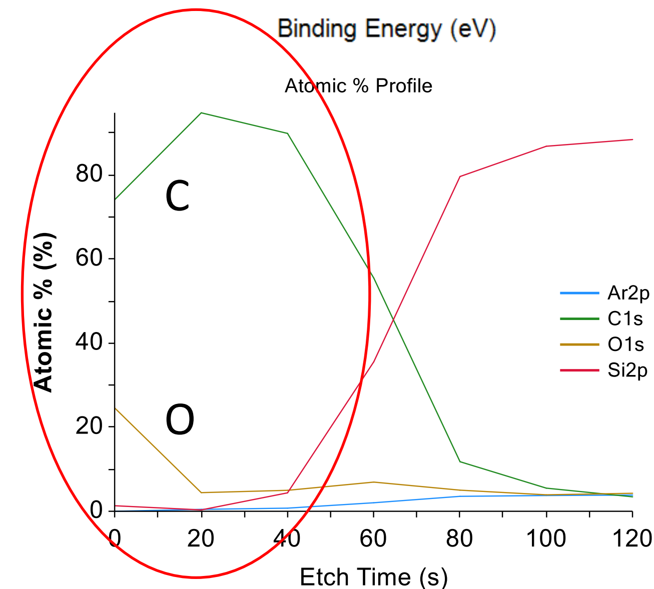
Ar **monatomic** ion
3KV High, 20S,
X-ray: 100um
Sputter: 500 um











Oxygen was preferentially etched and the stoichiometry is changed

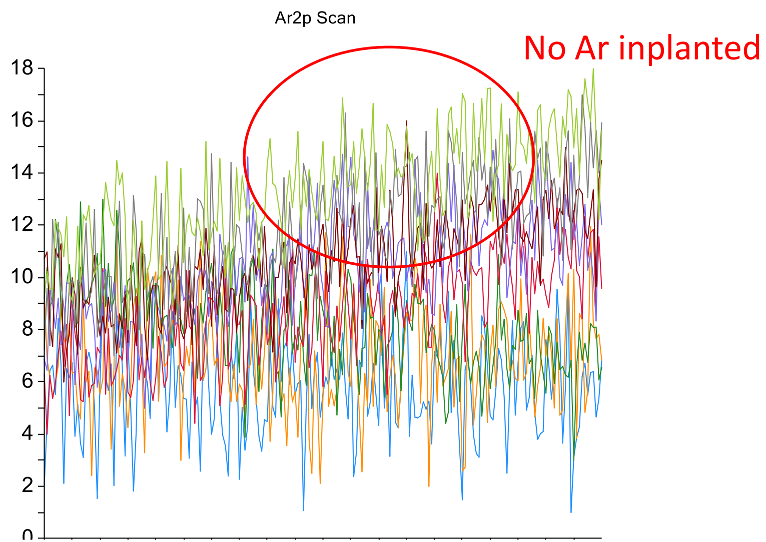
PMMA/Si: Depth profiling

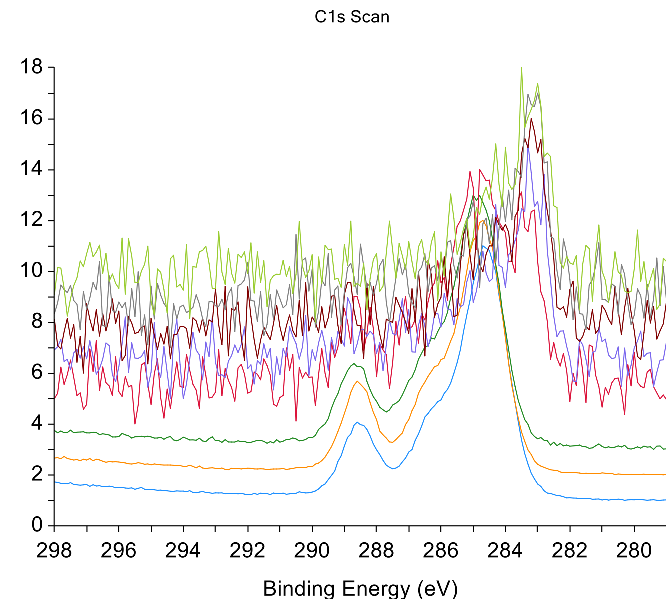
Ar **cluster** ion (Ar_{1000})

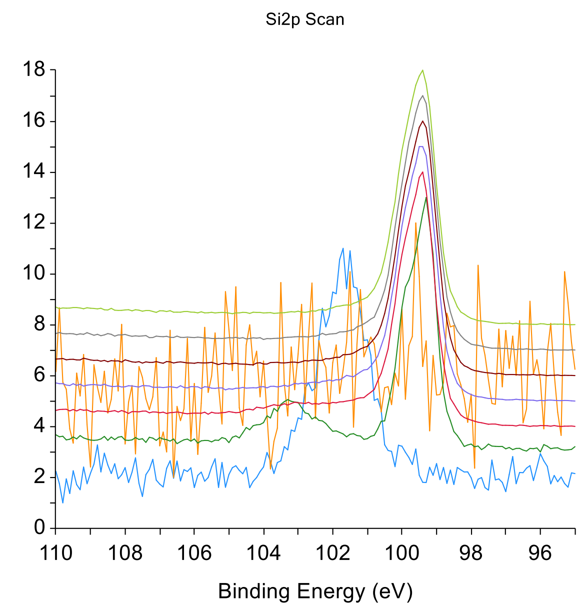
6KV High, 20S,

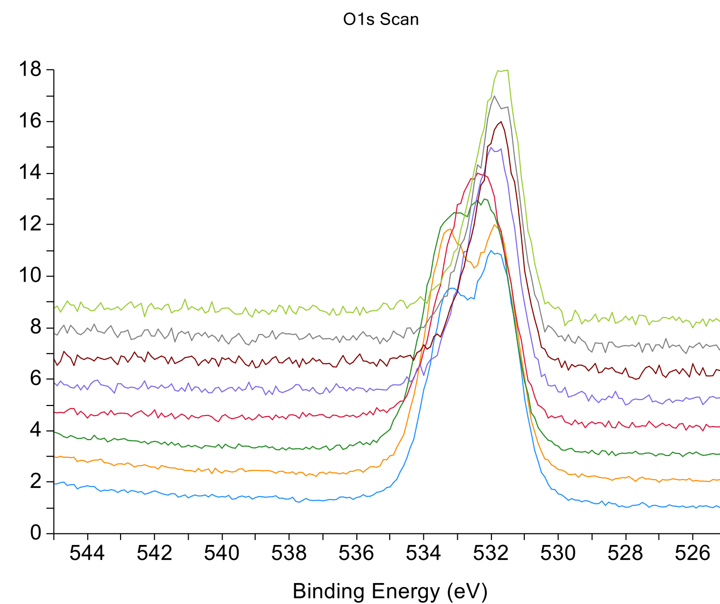
X-ray: 100um

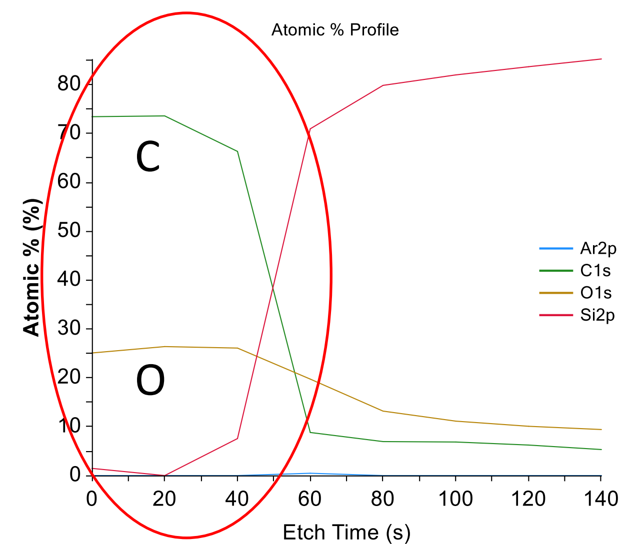
Sputter: 500 um



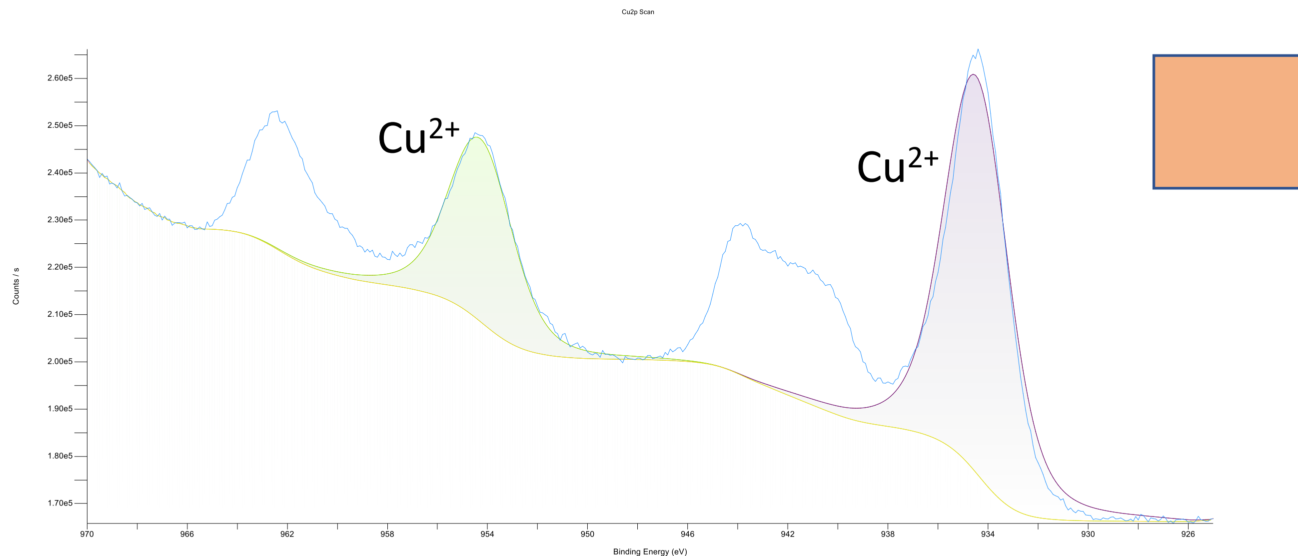


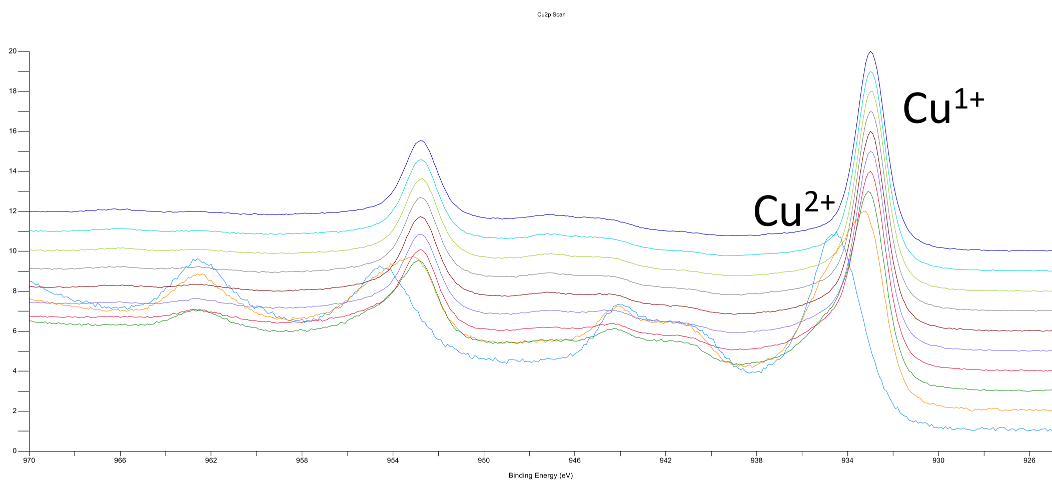


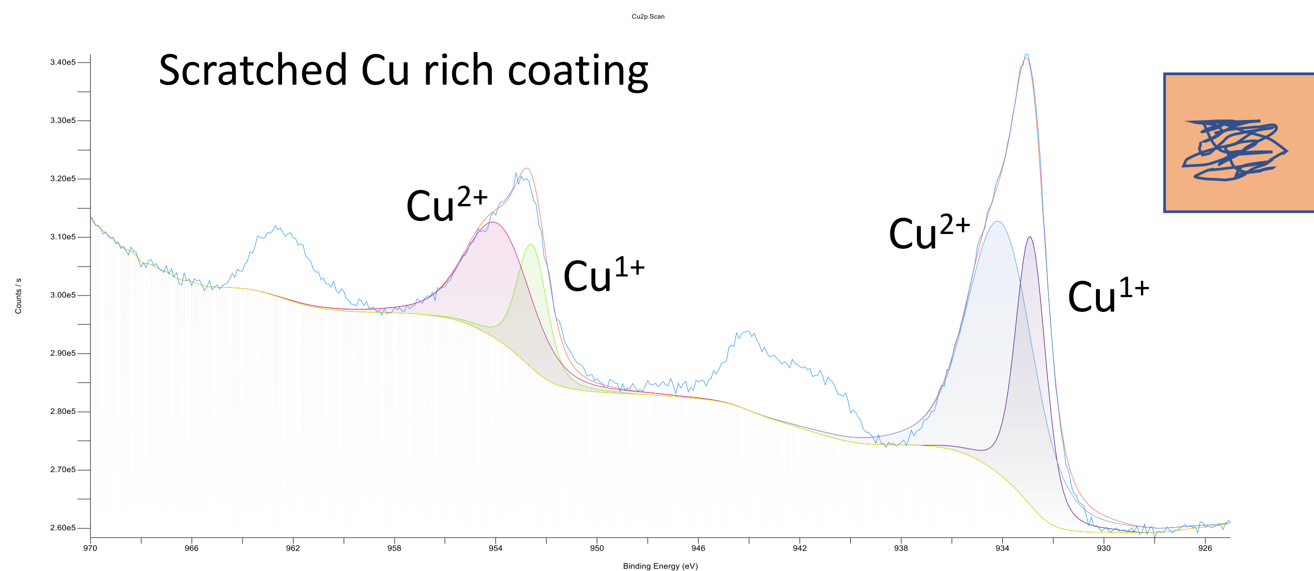




Case study:
Ar Sputtering caused reduction or not on copper sample?



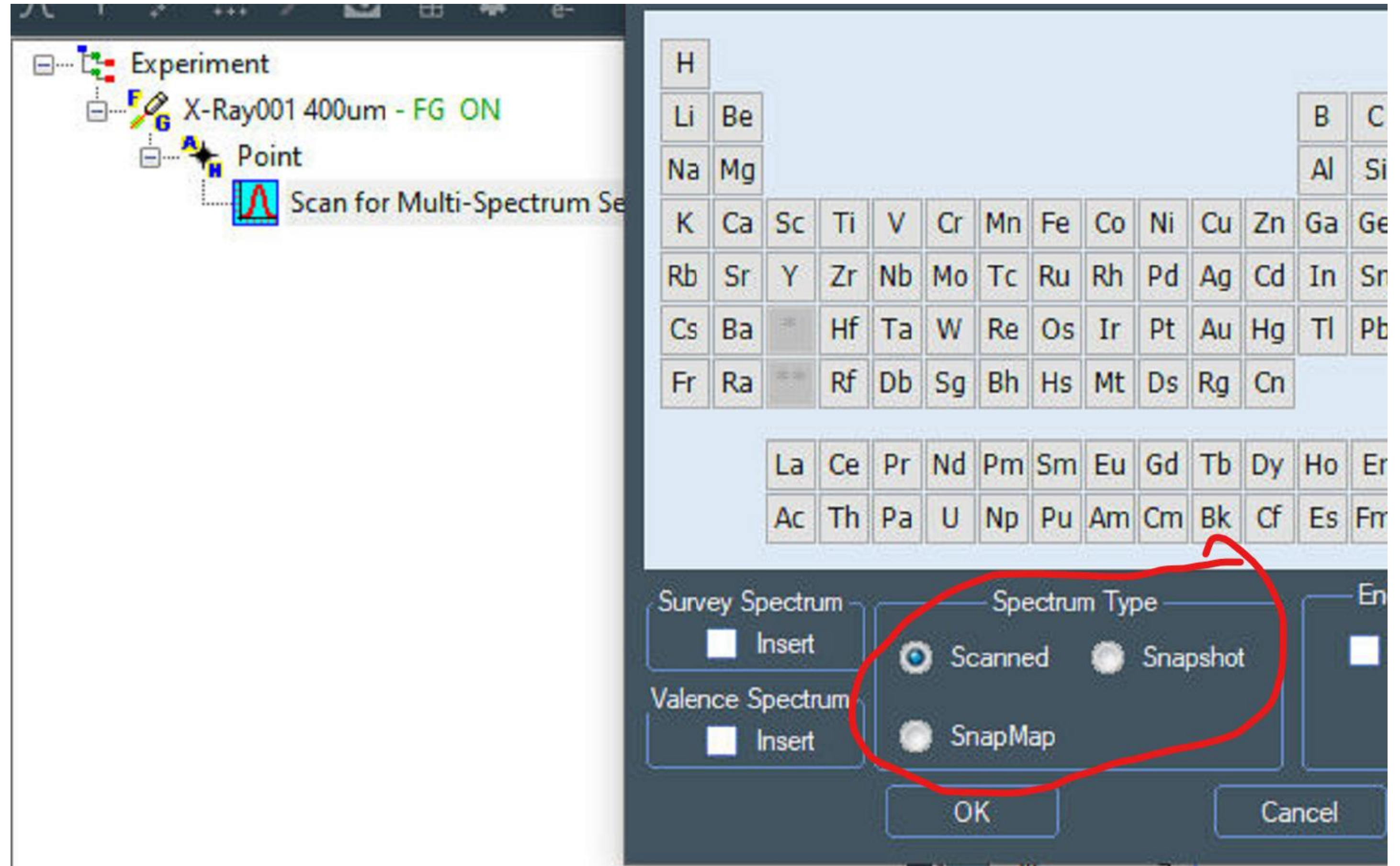




This proves that the depth profiling result is trustable.

Spectrum Type

1. **Scanned**: the highest energy resolution, good for elemental scan
2. **Snapshot**: the fast collection, good for depth profiling
3. **SnapMap**: good for elemental mapping



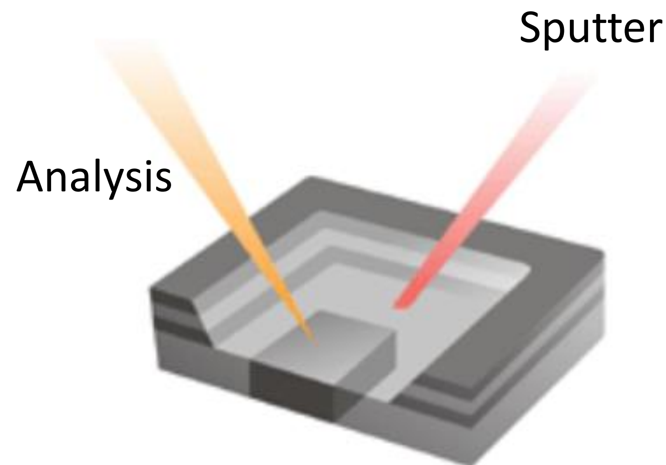
SIMS Depth Profiling

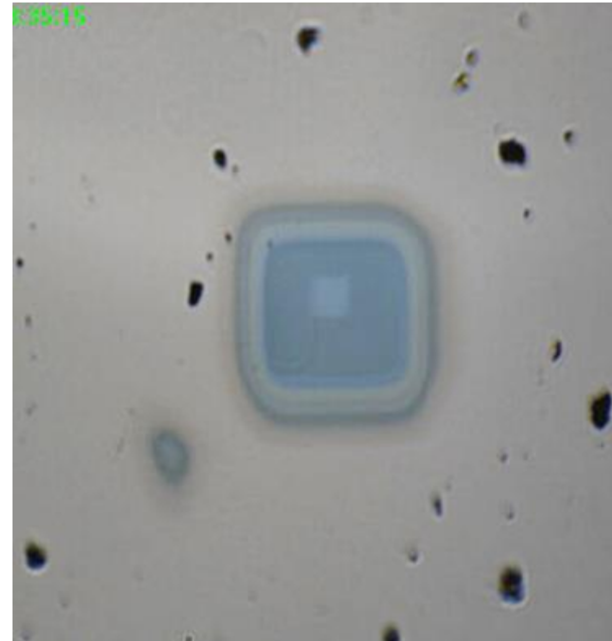
Sputter beam:

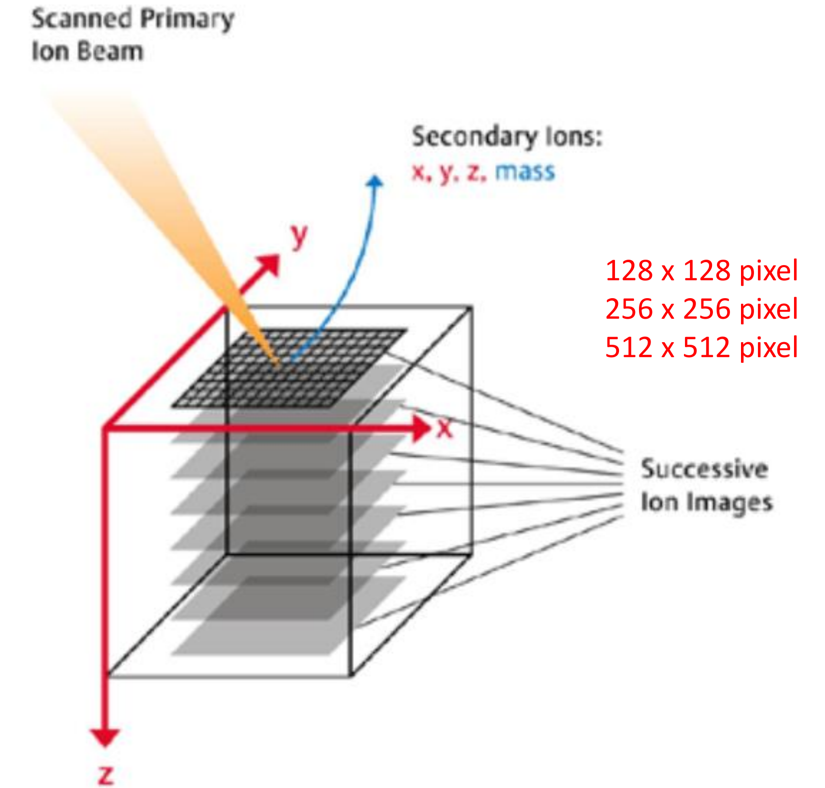
- Oxygen gun for positive ion
- Cesium gun for negative ion
- Cluster Ar ion gun for organic sample

Analysis beam:

- Bi1 and Bi3





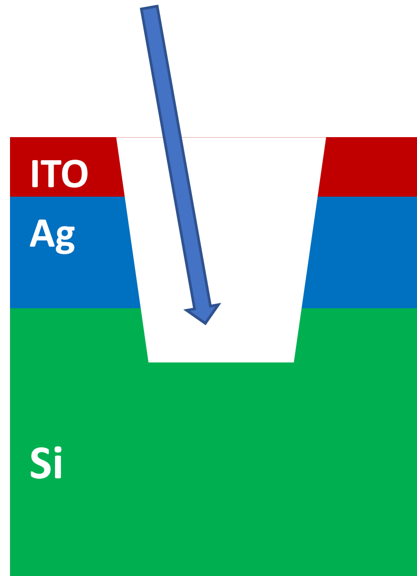


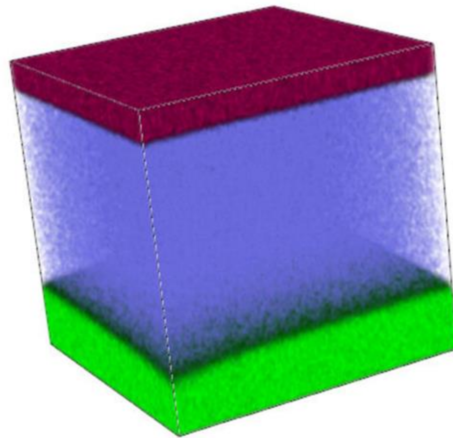
128 x 128 pixel
256 x 256 pixel
512 x 512 pixel

The x, y, z coordinates and mass of every secondary ion reaching the detector are stored.

Depth profiling with oxygen gun

O_2, Cs, Ar





In

Ag

Si

Intensity [counts]

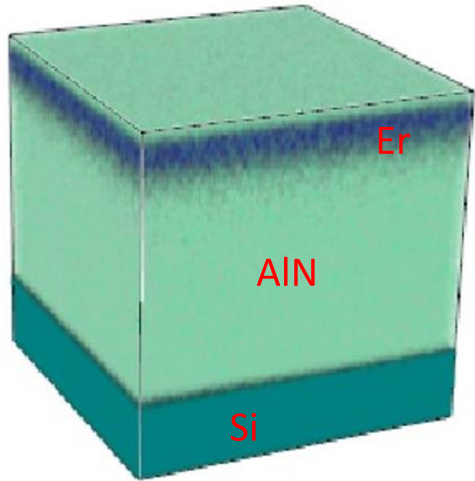
2573.87 s 135233.87

10^5 10^4 10^3 10^2 10^1 10^0

0 250 500 750 1000 1250 1500 1750 2000 2250 2500

Sputter Time [s]

Erbium doped in AlN on Si



Intensity [counts]

Er+
Al2+
Si+
N+

10^5
10^4
10^3
10^2
10^1
10^0

0 200 400 600 800 Depth [nm]

Sample: 15
Comment:
Origin:

File: Good profile-sample-15-500 um3
Date: Tue Aug 23 14:00:29 2022
Polarity: **Positive**

Analysis Parameters:

PI:	Bi1
Energy:	30 keV
Current:	pA
Area:	100 x 100 µm²
PIDD:	Ions/cm²

Sputter Parameters:

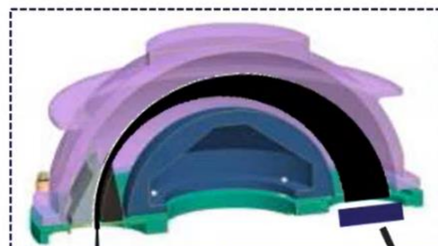
SpI:	O2
Energy:	2 keV
Current:	nA
Area:	500 x 500 µm²
SpIDD:	Ions/cm²

XPS depth profiling	
Strength:	Quantitative analysis
	Chemistry information
	All elements in one profile
	Snapshot mode is available (new)
Weakness:	Sputter crater is bigger (1 or 2 mm square)
	Time consuming for multiple elements
	not for trace elements and very low concentration

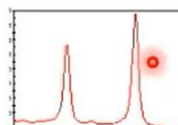
SIMS depth profiling	
Strength:	Fast speed
	Better depth resolution
	Good for trace elements and very low concentration
	3D reconstruction
	Small sputter crater (200 um square)
Weakness:	Qualitative analysis
	No chemistry information
	Only for positive ions or negative ions at a time

XPS Imaging : XPS-SnapMap

2. Photo-electrons from that small area are collected and focussed into the energy selector

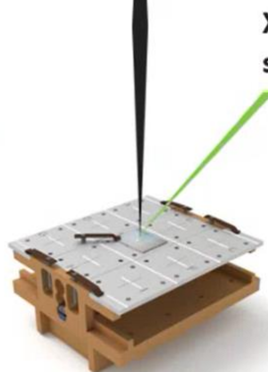


3. Energy filtration is applied, selecting a specified range of electron energies



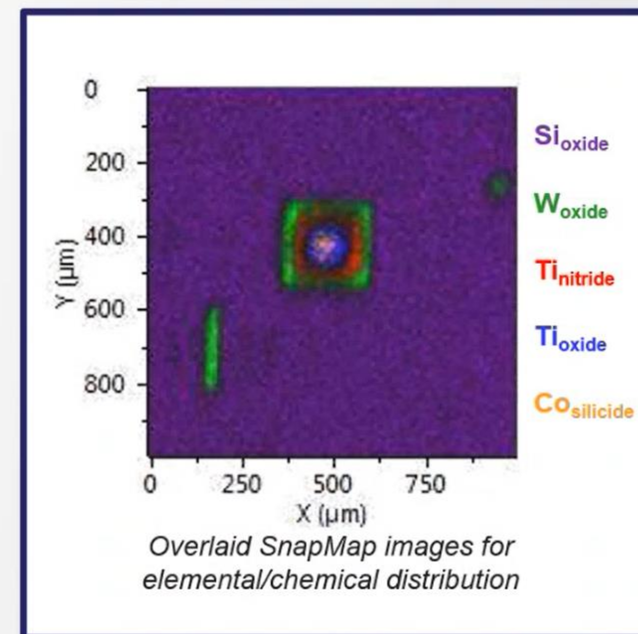
1. Aluminium Kα X-rays illuminate a small area on the sample, freeing electrons from the surface

X-ray source



4. Spectra are continually acquired whilst sample stage is rastered

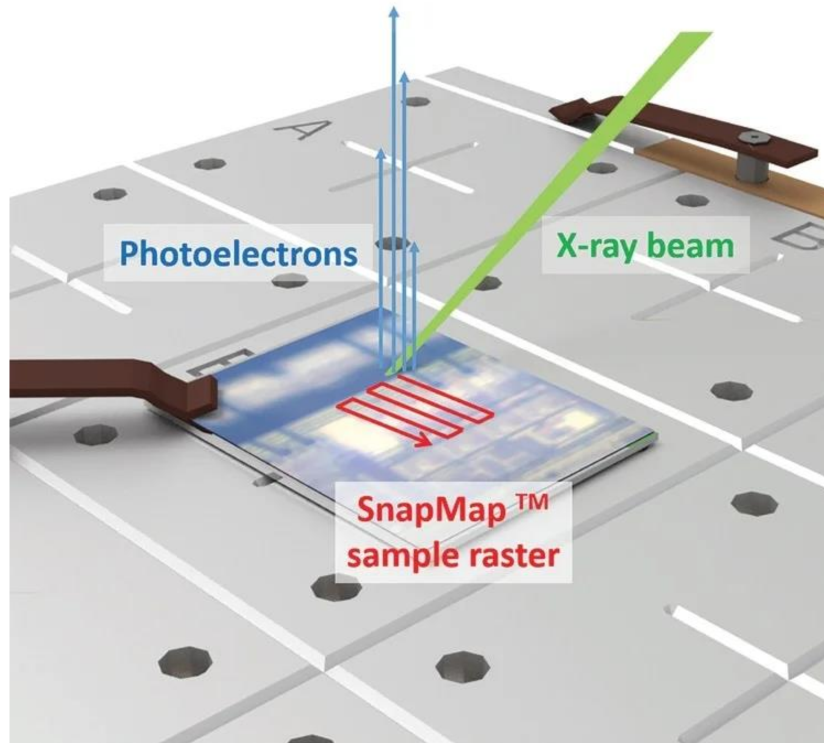
5. Stage position monitored throughout data acquisition, positions used to generate SnapMap

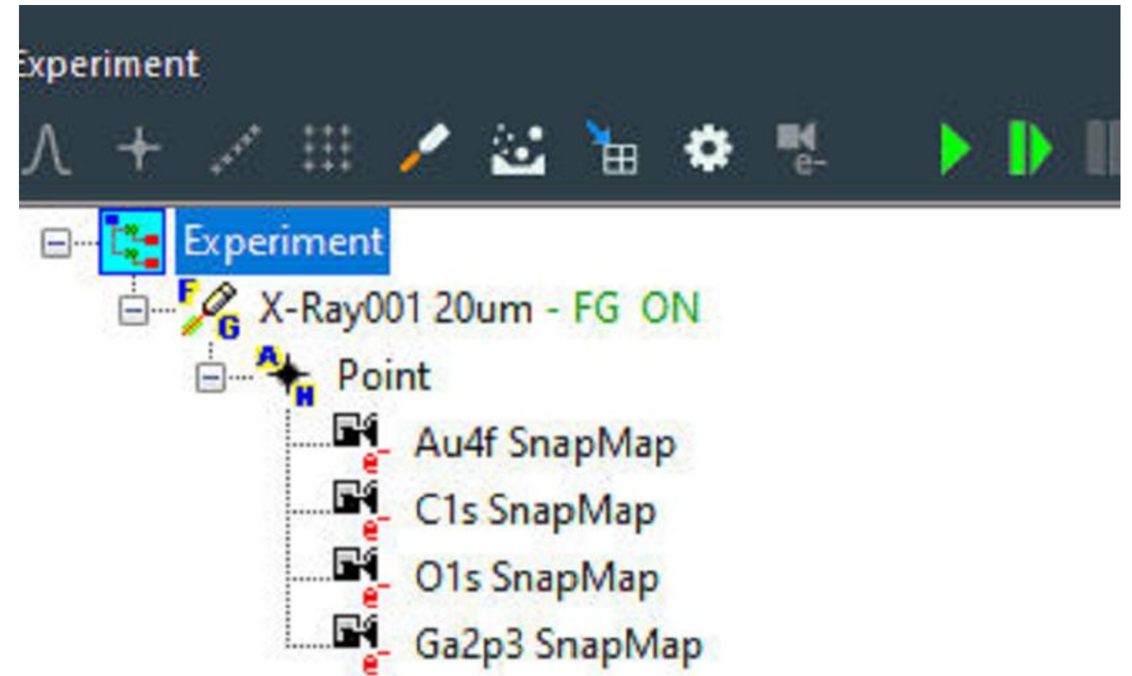


Overlaid SnapMap images for elemental/chemical distribution

www.xps-simplified.com

XPS SnapMap

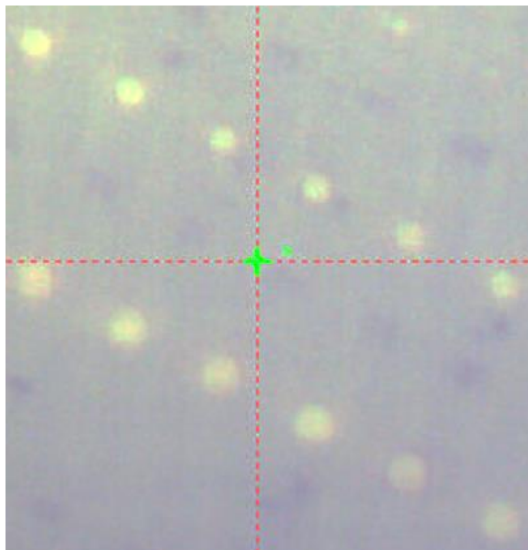


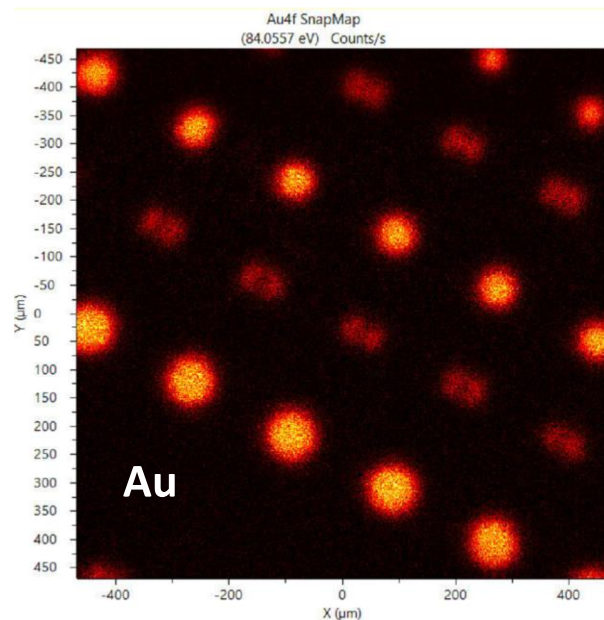


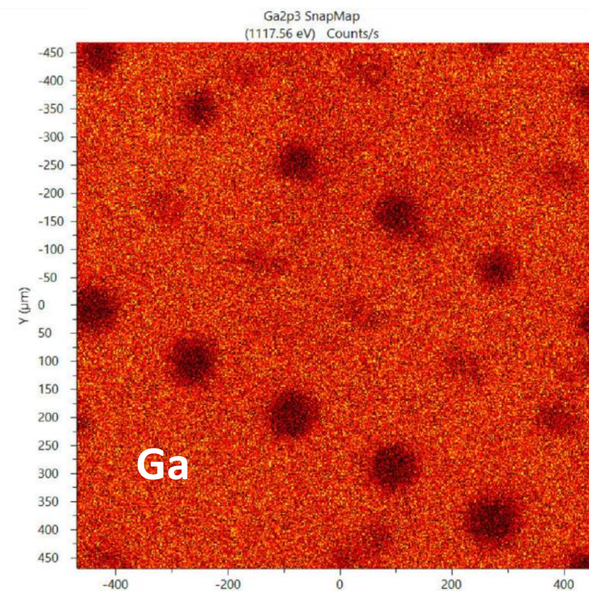
Snapmap mode allows rapid high resolution surface mapping

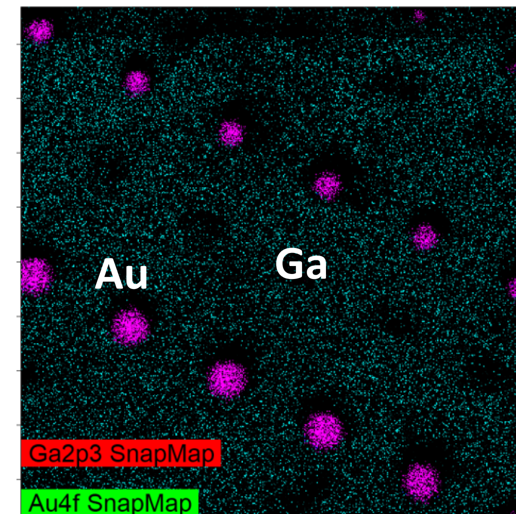
1. Analyzer is fixed on a specific binding energy window (an elemental peak)
2. The sample stage is rastered.
3. The analyzer acquires rapid "snapshot" spectra for each pixel of the image.
4. The pixel resolution is limited only by the minimum X-ray spot size (10 μm).

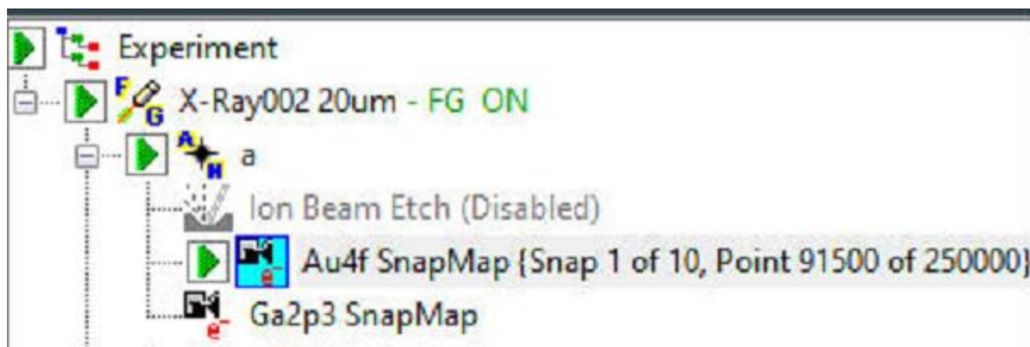
XPS SnapMap (Gold pattern on Ga)-new feature











SnapMap | General

Energy Scale: Kinetic / Binding

Size in mm: 1.0

Centre Energy: 87.586641

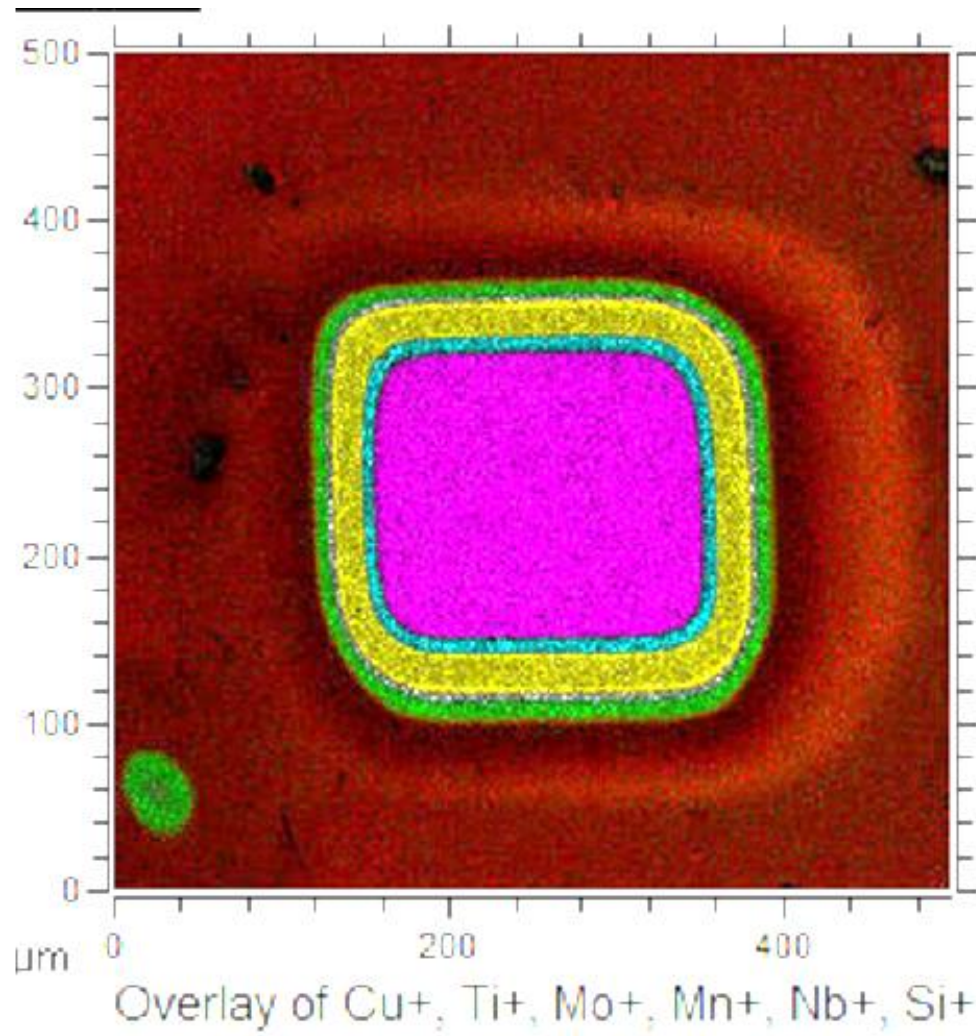
Image Resolution: 500x500

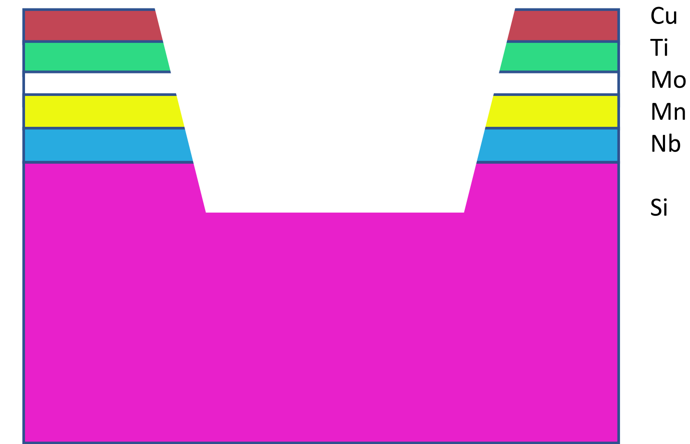
Number of Snaps: 10

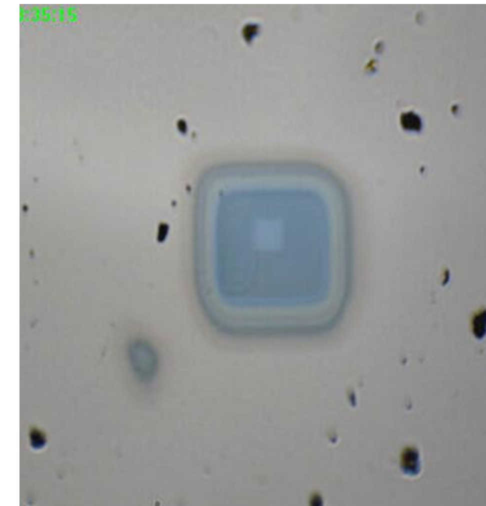
Step Size in mm: 0.002

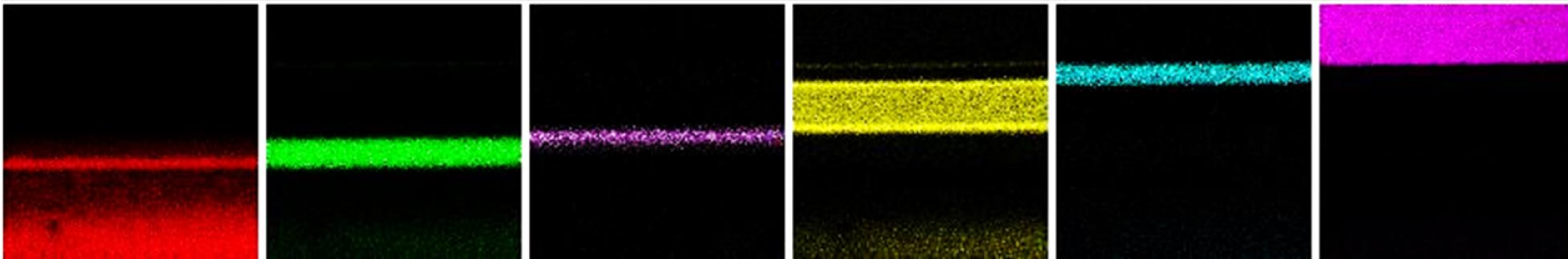
Stage Speed in mm/s: 0.2

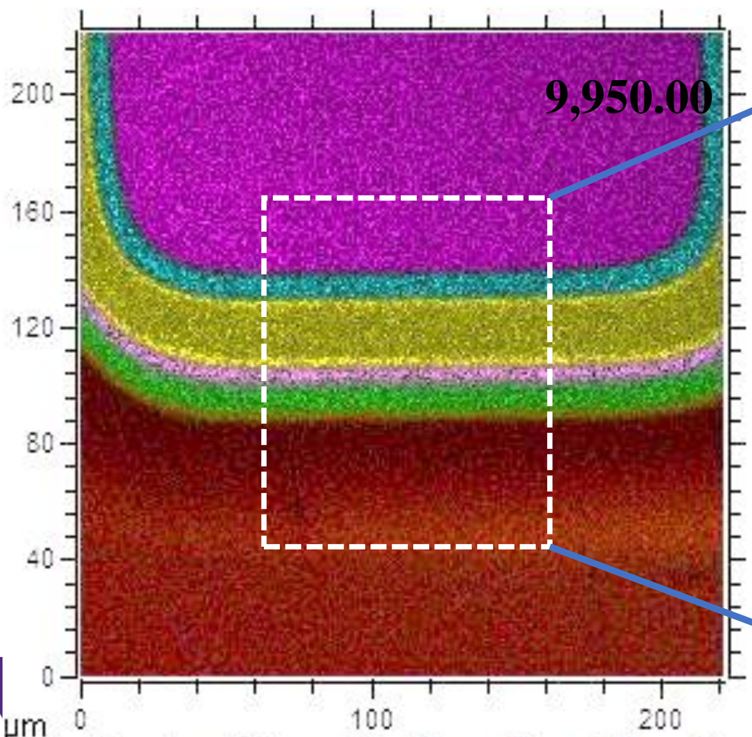
SIMS mapping: Elemental Mapping on Depth Profiling Crater on Cu/Ti/Mo/Mn/Nb on Si Wafer

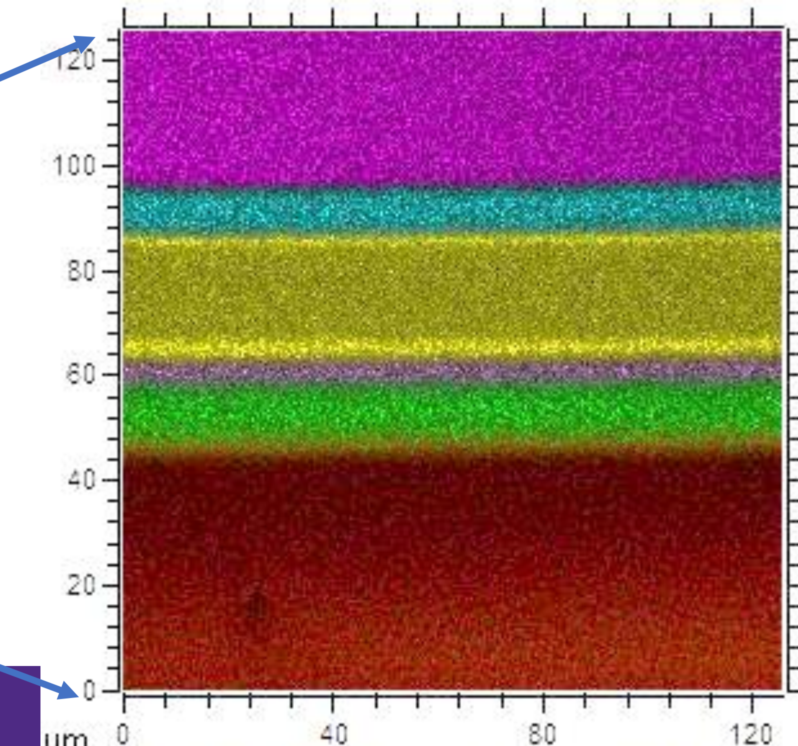


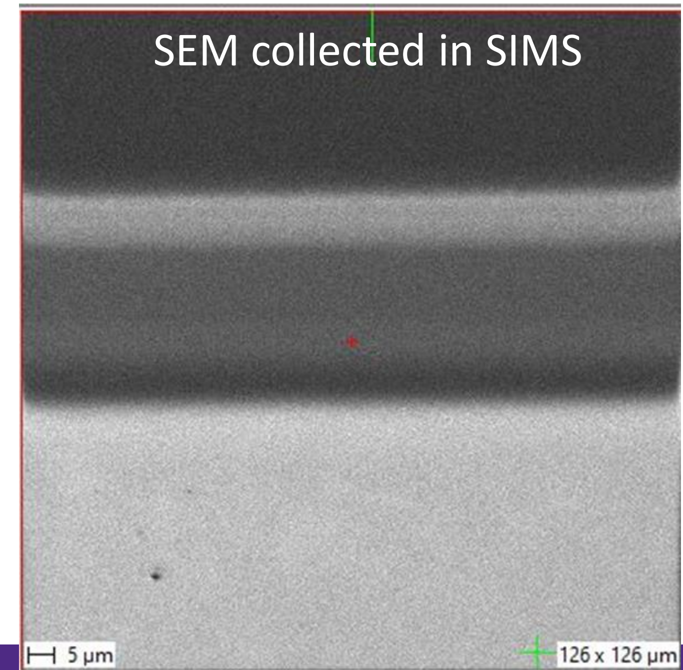





Cu+
Ti+
Mo+
Mn+
Nb+
Si+


9,950.00
200
160
120
80
40
0
µm
0
100
200
Overlay of Cu+, Ti+, Mo+, Mn+, Nb+, Si+


120
100
80
60
40
20
0
µm
0
40
80
120
Overlay of Cu+, Ti+, Mo+, Mn+, Nb+, Si+


SEM collected in SIMS
5 µm
126 x 126 µm

XPS Mapping	
Strength:	Every pixel has a spectrum
	Chemical information
	10 um lateral resolution
	Surface composition distribution
Weakness:	Time consuming for hi-resolution

SIMS mapping	
Strength:	Fast speed
	Better spatial resolution (100 nm)
	Very sensitive (ppm to ppb)
	All elements including H, He, etc.
	Identify isotopes
Weakness:	No chemical information
	Surface destructive

surfsciftp.co.uk/avant6/

Login - NUcore | Webmail : Northwe... | #JoeScarborough:... | NUANCE: Northwes... | Yahoo | GoToAssist Corpo

Secure version of this website at https://www.surfsciftp.co.uk/avant6

Avantage Software

Version 6 - Please read this information carefully before downloading and installing Avantage

*** * * You must have Windows 10 on your instrument PC. * * ***

Version 6

- Currently supports following systems
- Processing-Only installations with Windows 10 [or Windows 11 - limited testing and support] **For Windows 11 22H2 l**

Serial Number begins...	System / Model
A995...	Escalab Xi+
NXA..., NXA2G...	Nexsa, Nexsa G2
KAN995..., KAN2G995...	K-Alpha

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CasaXPS: Processing Software for XPS, AES, SIMS and More

CasaXPS processing software offers powerful analysis techniques for both spectral and imaging data. The system originally designed for XPS and Auger data now offers features covering a wide range of analytical techniques including ToF SIMS, dynamic SIMS and many more.

For further information, please contact neal@casaxps.com.

日本語は、こちら For information in Japanese, please click here.

Data conversion options are standard in CasaXPS, where proprietary file formats are converted to ISO 14976 (VAMAS) allowing results to be easily exchanged between laboratories.

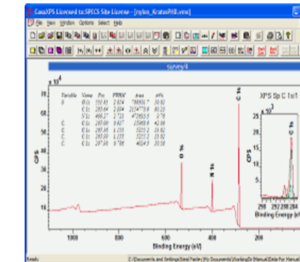
CasaXPS Training and WORKSHOP Events

CasaXPS videos are also available on YouTube under the

CasaXPS Casa Software YouTube Channel

CasaXPS Version 2.3.25 Download

To view the Web Manual, click below



Both Avantage and CasaXPS are freely downloadable and the licenses are also available.

Thanks for your attention

Questions?

xchen@northwestern.edu

847-491-5505