

# Welcome

The presentation will start shortly

# CISA

## Correlative Imaging & Surface Analysis

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Applications Development & Marketing Manager

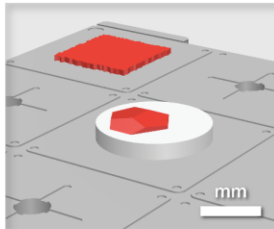
1<sup>st</sup> August 2022

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**CISA mounting**

Samples or SEM stubs mounted on a dedicated sample plate with fiducial marks

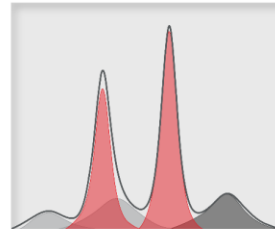


Maintenance of same sample mounting at each step for easy region of interest (ROI) targeting



**XPS**

Quantitative chemical composition of the top 10 nm of the sample surface



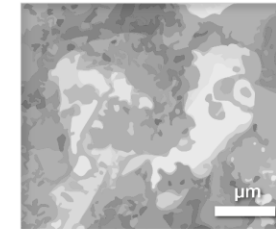
Surface analysis using XPS and related techniques, plus ion beam cleaning

**Avantage Software**



**SEM**

High-resolution imaging of the sample and bulk elemental information with EDS



High-resolution imaging for feature sizing and identification

**ChemiSEM Technology**

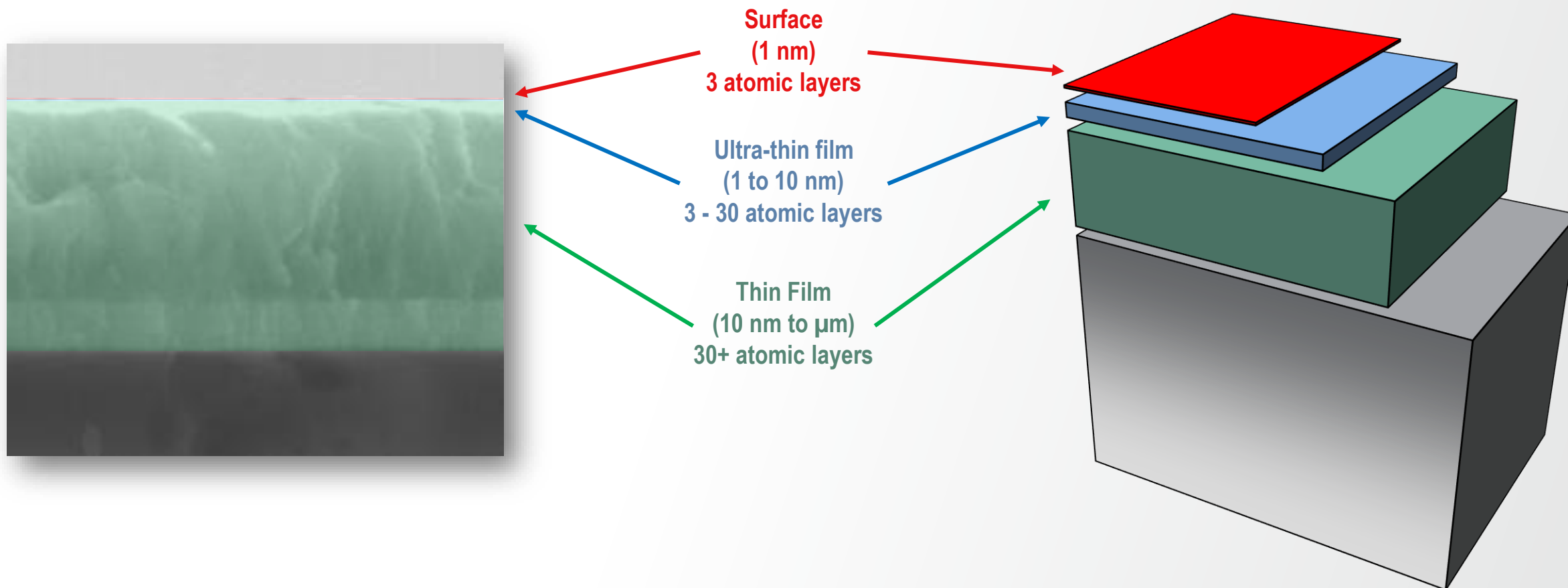
**Maps Software**

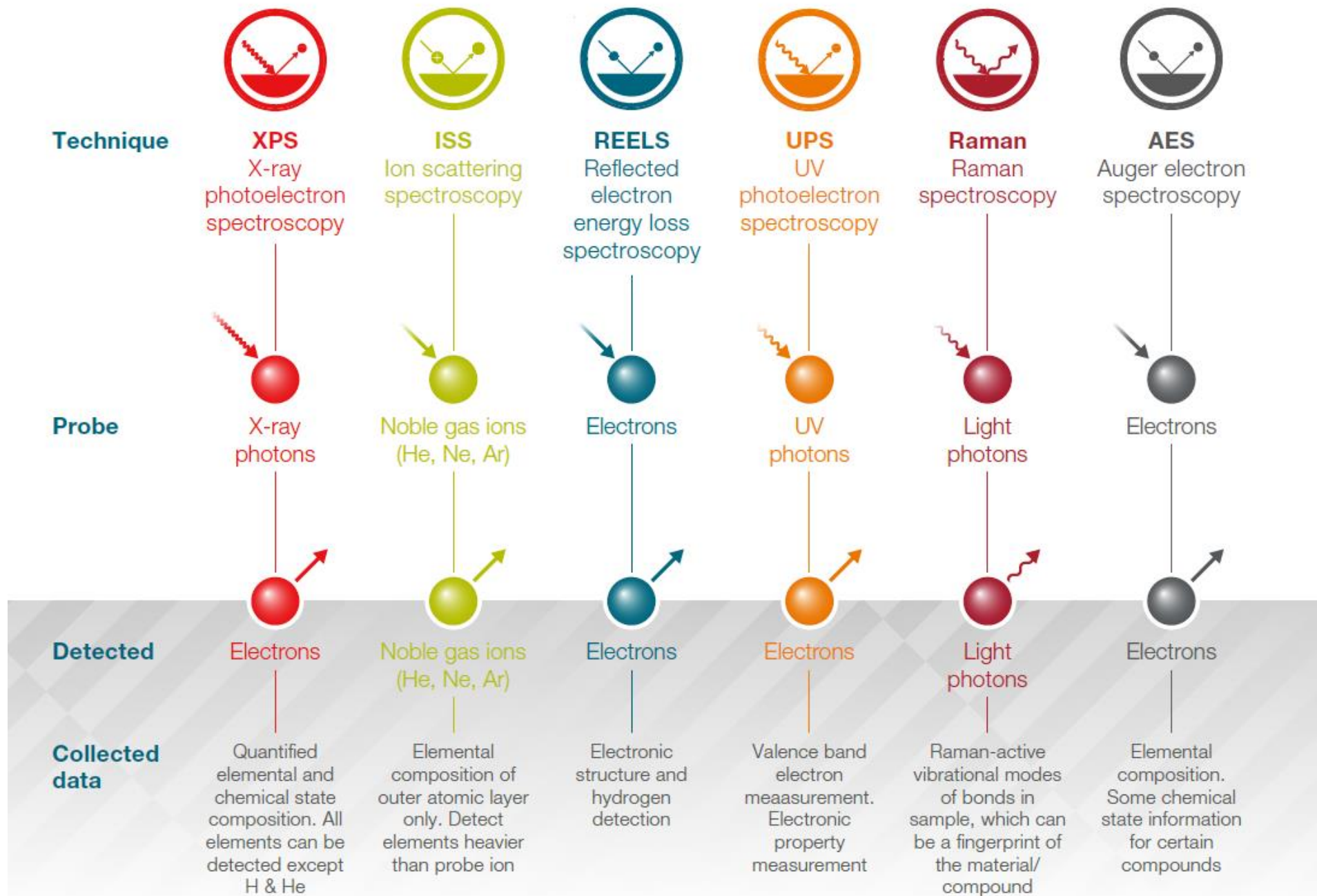


Sample journey

# Definition of a surface

“The Surface” is generally regarded to be the outer 10 nm for scientists using XPS



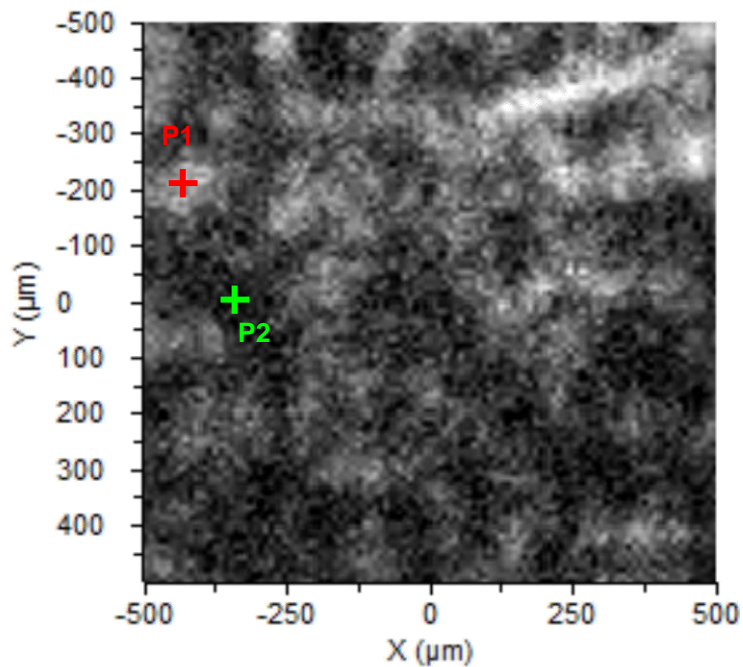


# Case study

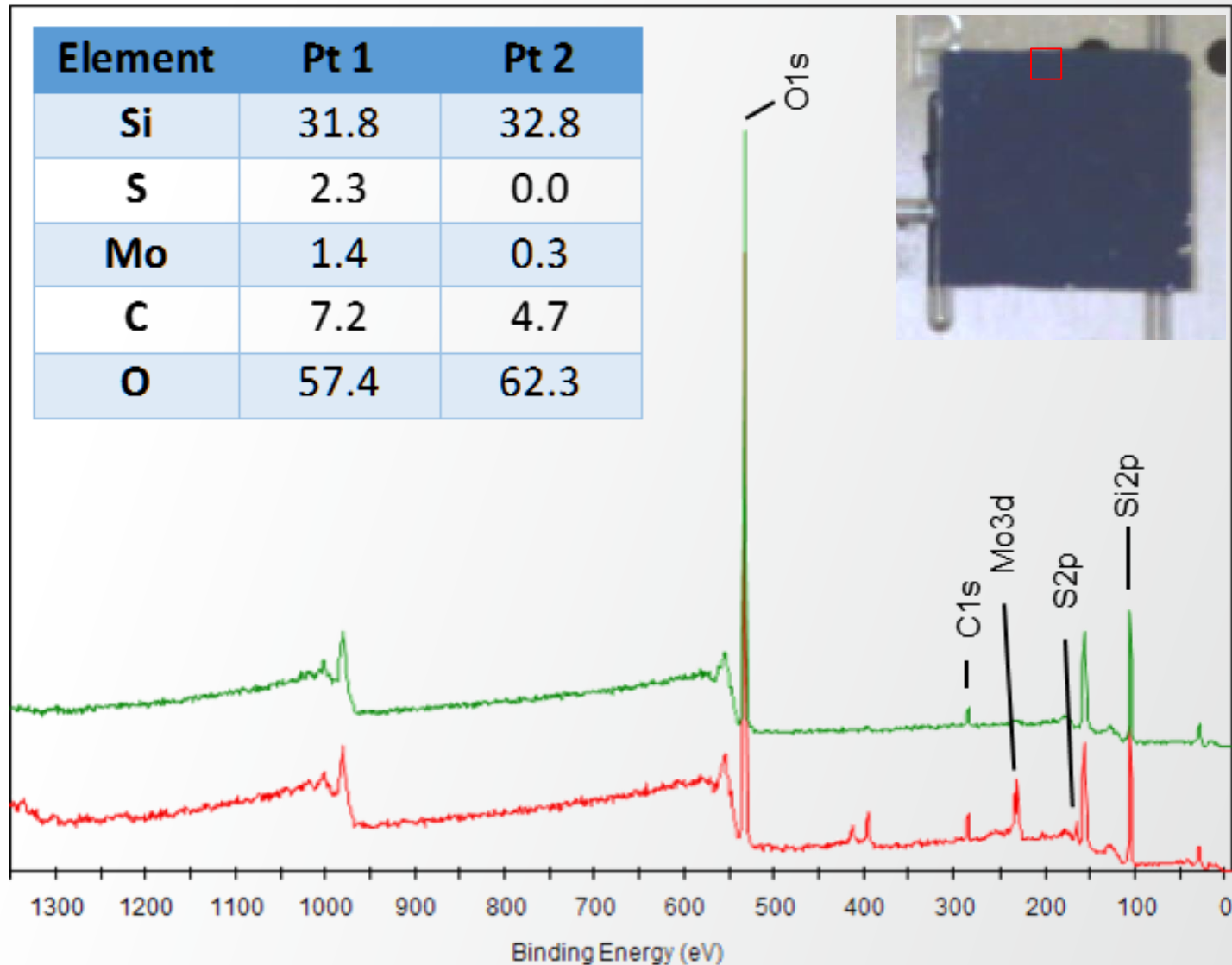
2D Materials

# XPS-Raman analysis of MoS<sub>2</sub>

Elemental quantification with small spot XPS

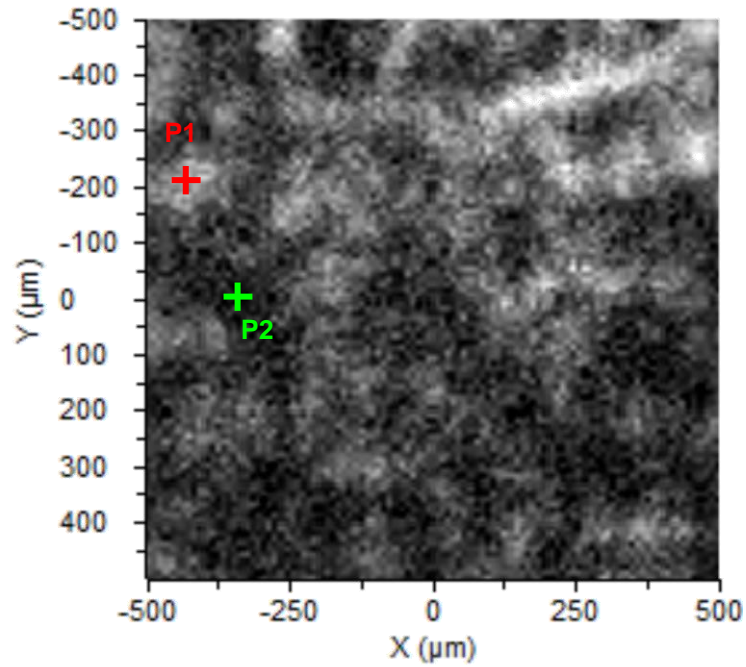


Mo3d SnapMap image

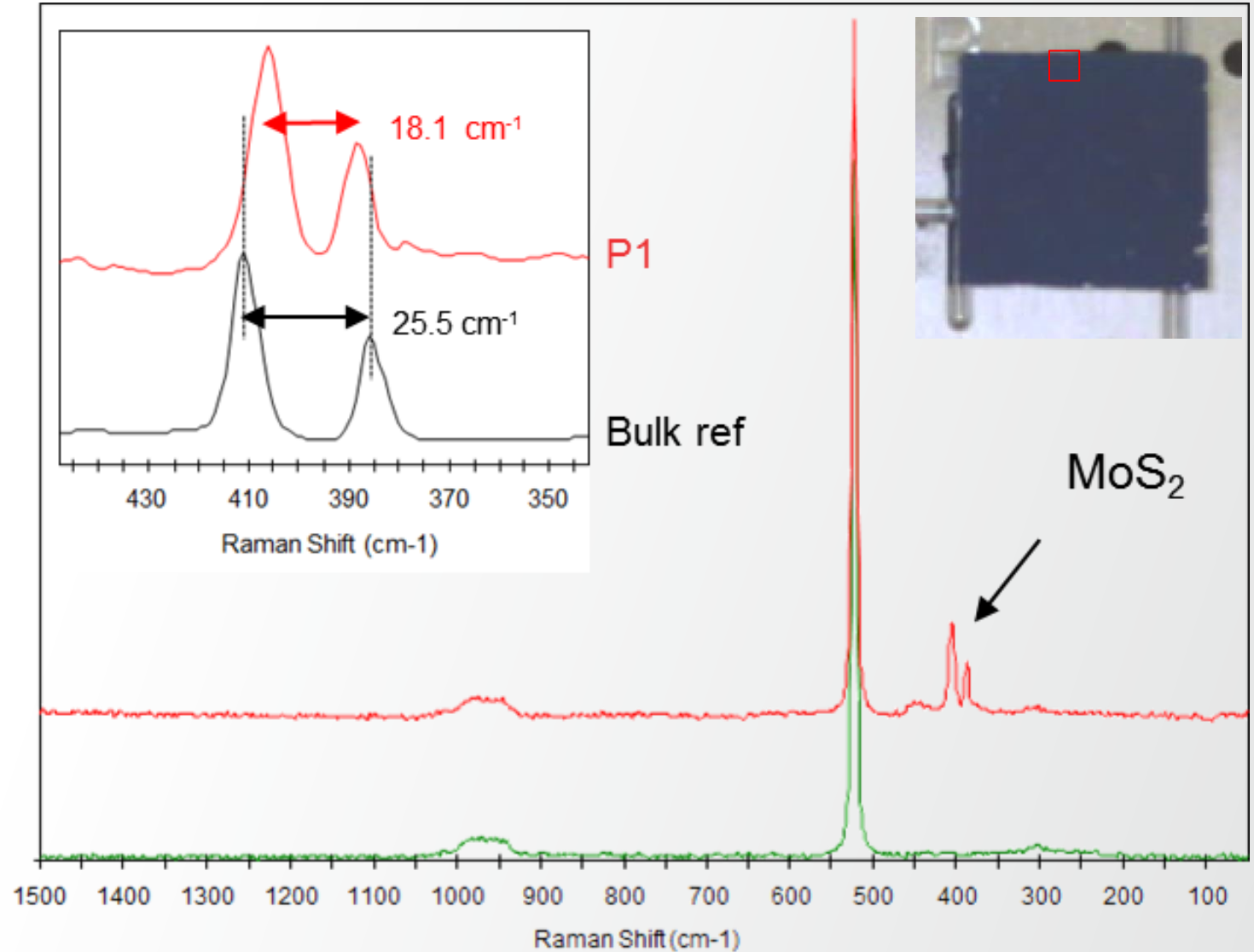


# XPS-Raman analysis of MoS<sub>2</sub>

Layer thickness with Raman spectroscopy

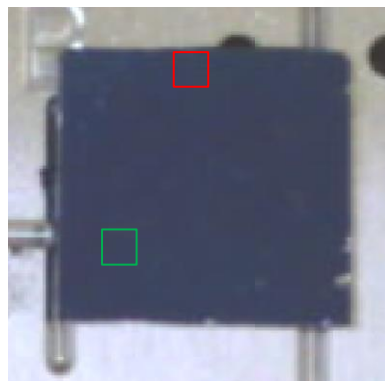
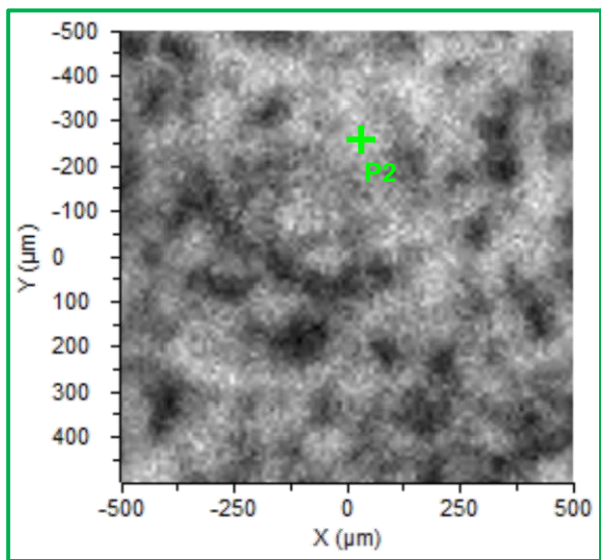
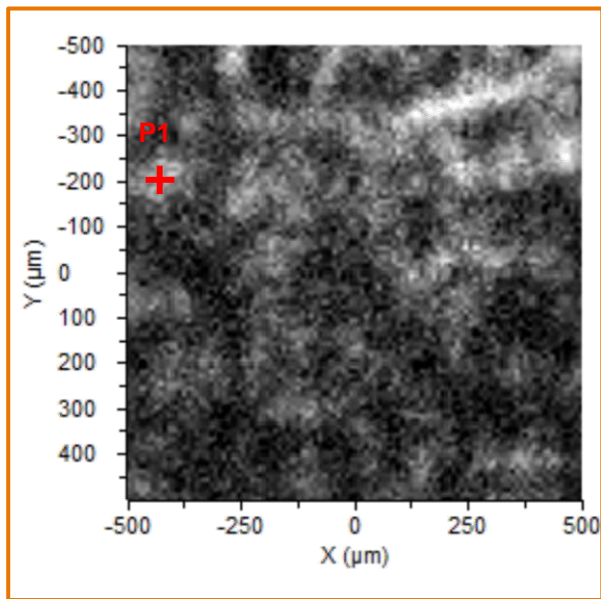


Mo3d SnapMap image

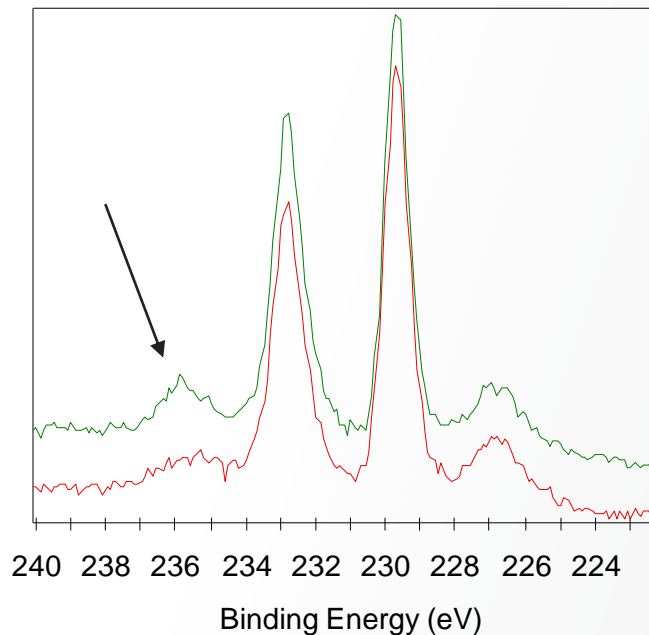




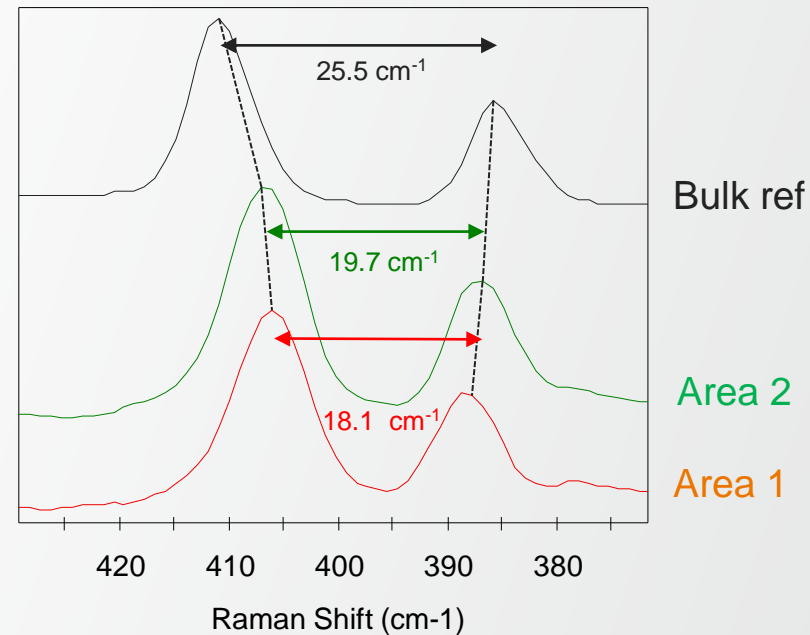
# XPS-Raman analysis of MoS<sub>2</sub>



## Mo3d XPS



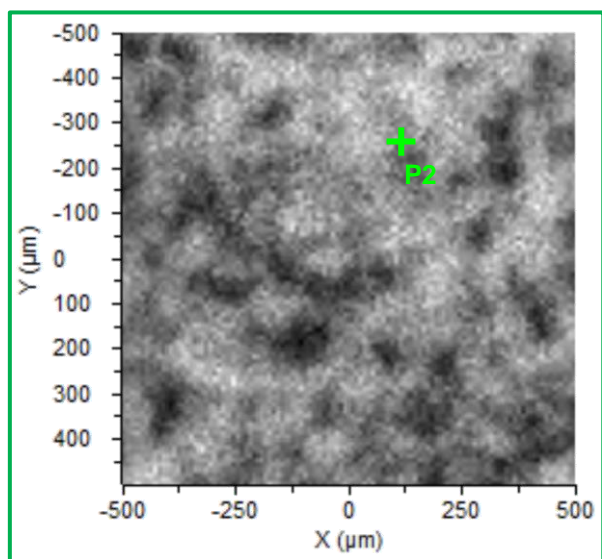
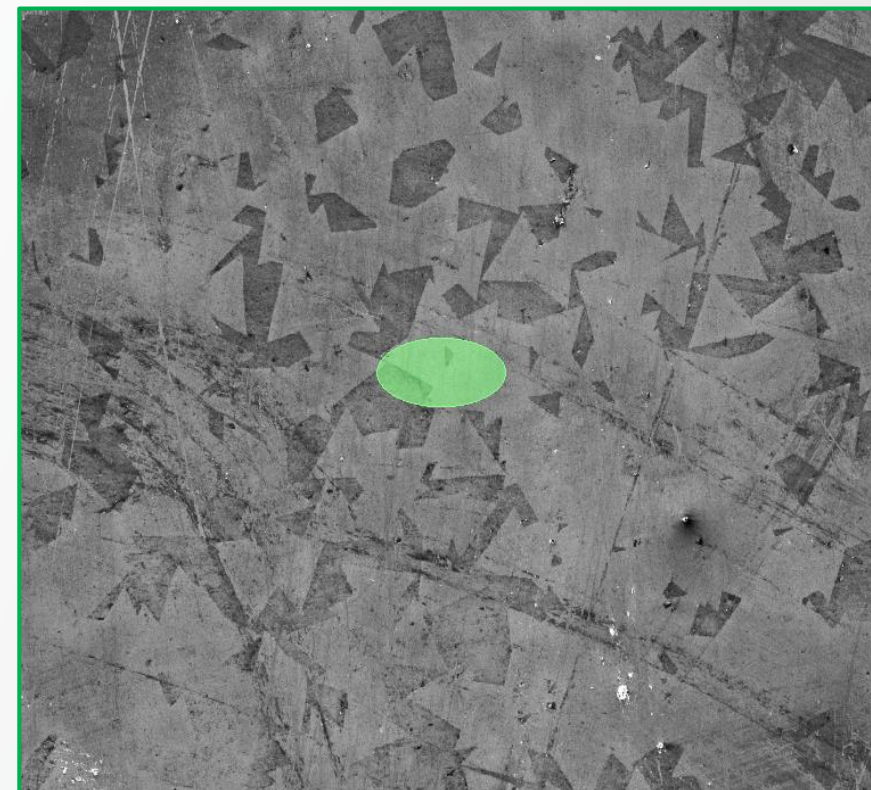
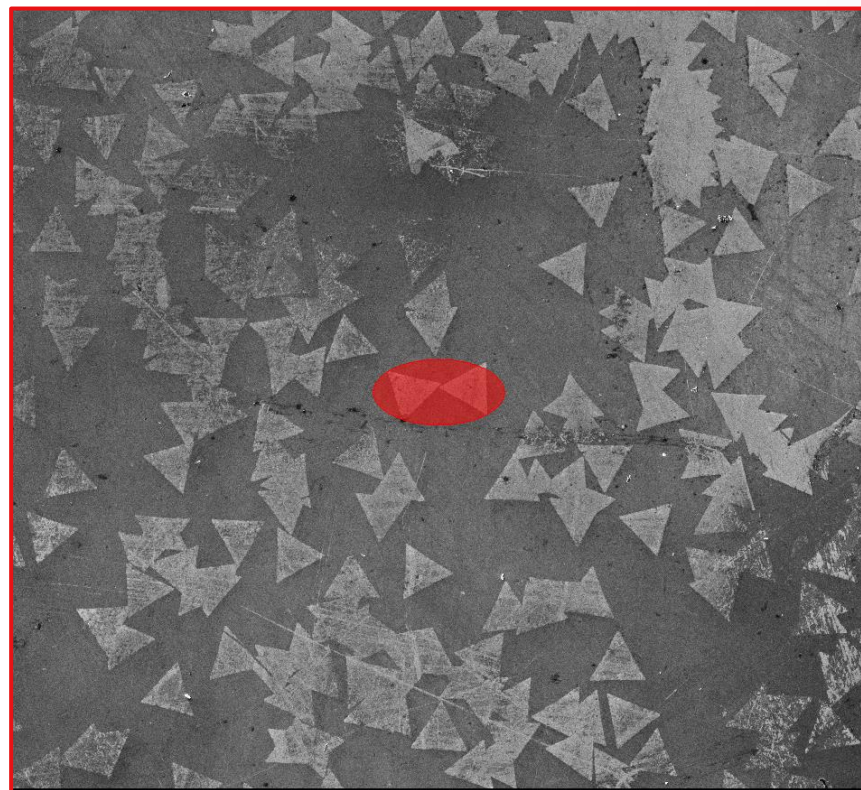
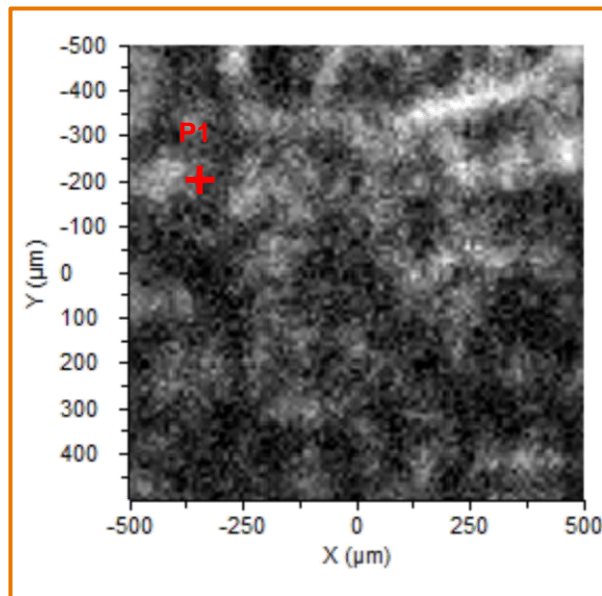
## Raman



- Increased oxidation seen in Area 2 (indicated by arrow)
- Increased separation between two Raman peaks indicates increase in number of layers.

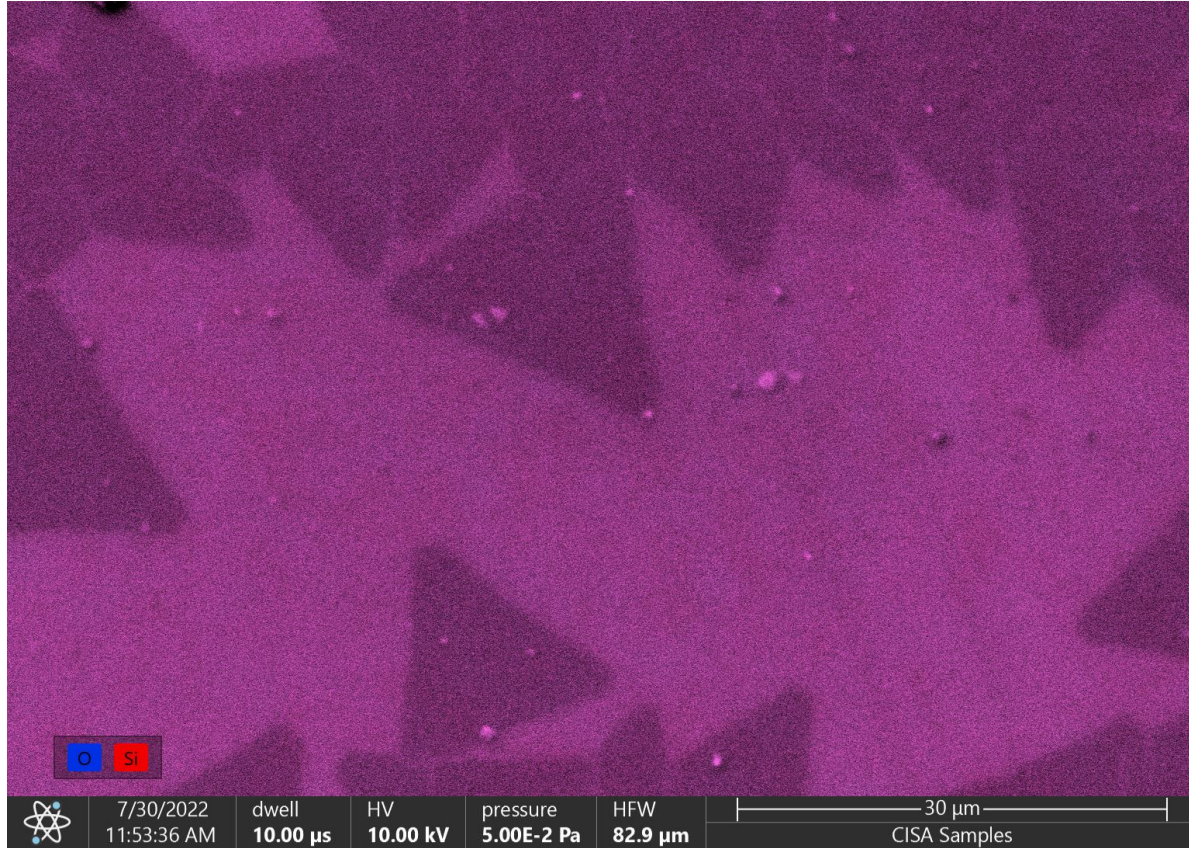
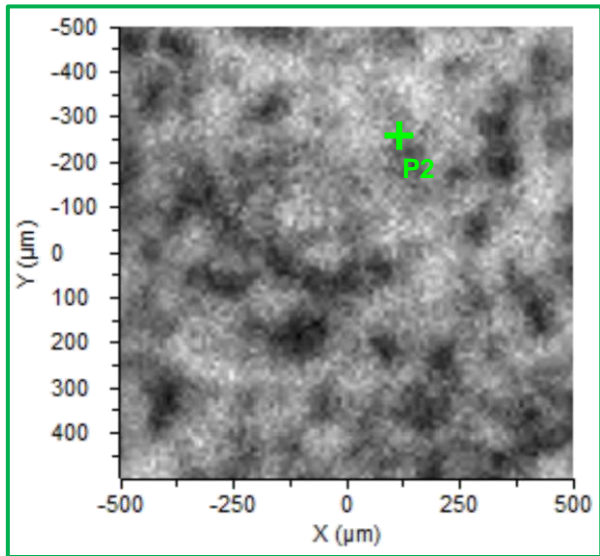
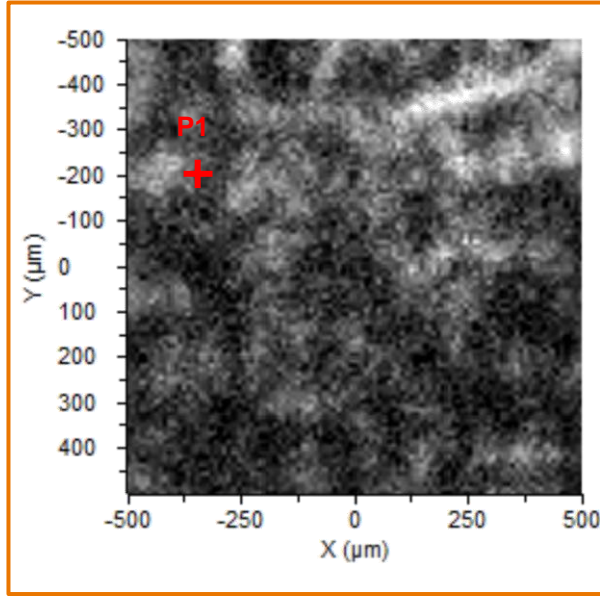


# Correlation with SEM



SEM shows lower density of triangular structures in area with single layer MoS<sub>2</sub>

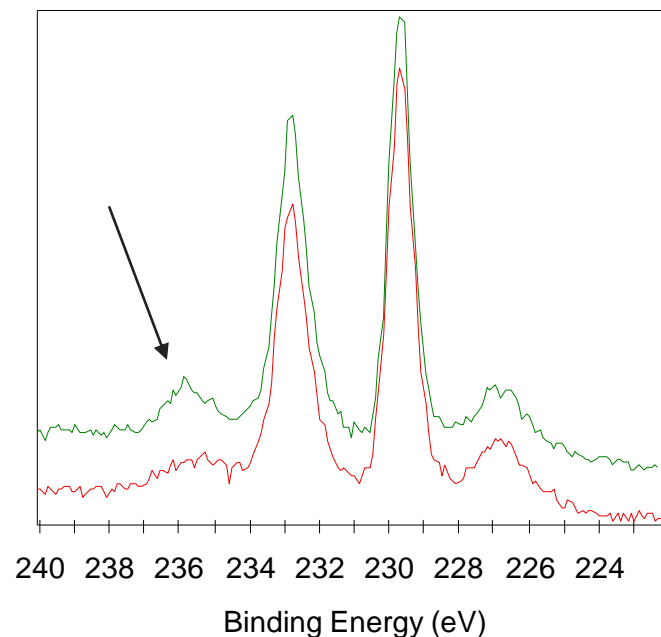
# Correlation with SEM



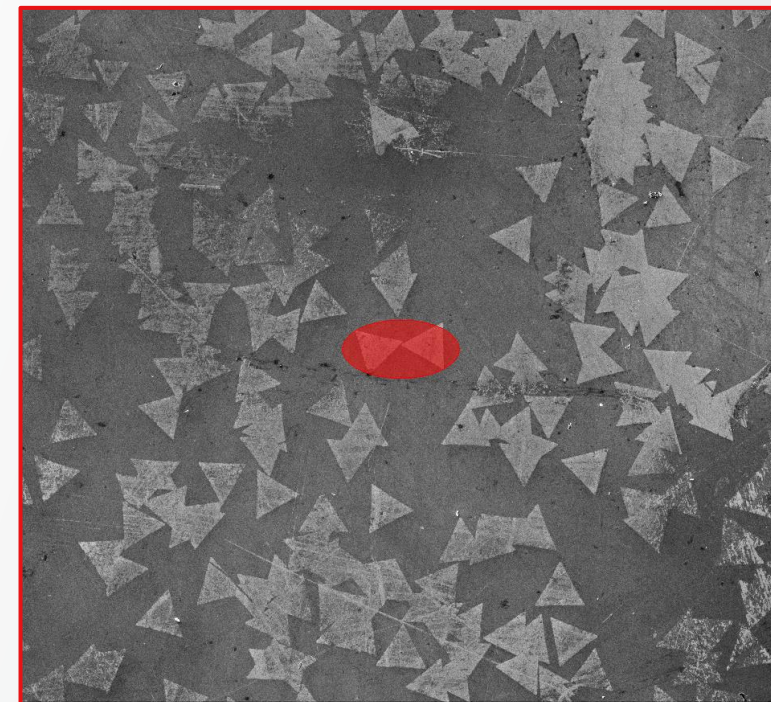
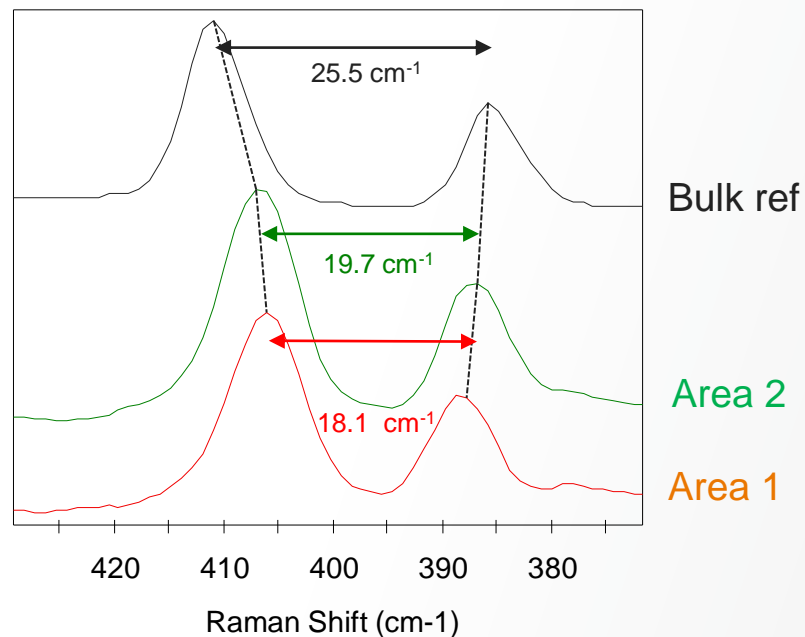
EDS not surface sensitive enough to measure Mo & S signals

# XPS-Raman-SEM analysis of MoS<sub>2</sub>

## Mo3d XPS



## Raman



- Increased oxidation seen in Area 2 (indicated by arrow)
- Increased separation between two Raman peaks indicates increase in number of layers.
- SEM shows varying density of MoS<sub>2</sub> structures in areas 1 & 2

## **Case study**

Anti-bacterial fabrics

# Introduction

## Functionalisation of fabrics

- Lots of examples of processes to impart desirable properties to fabrics
  - Improved fire-retardants
  - Wettability / water repellence
  - Stain resistance
  - Strengthening
  - Anti-bacterial properties
- Coatings
- Nanoparticles

Milošević, Milica, et al. *Journal of Materials Science* 48.16 (2013): 5447-5455

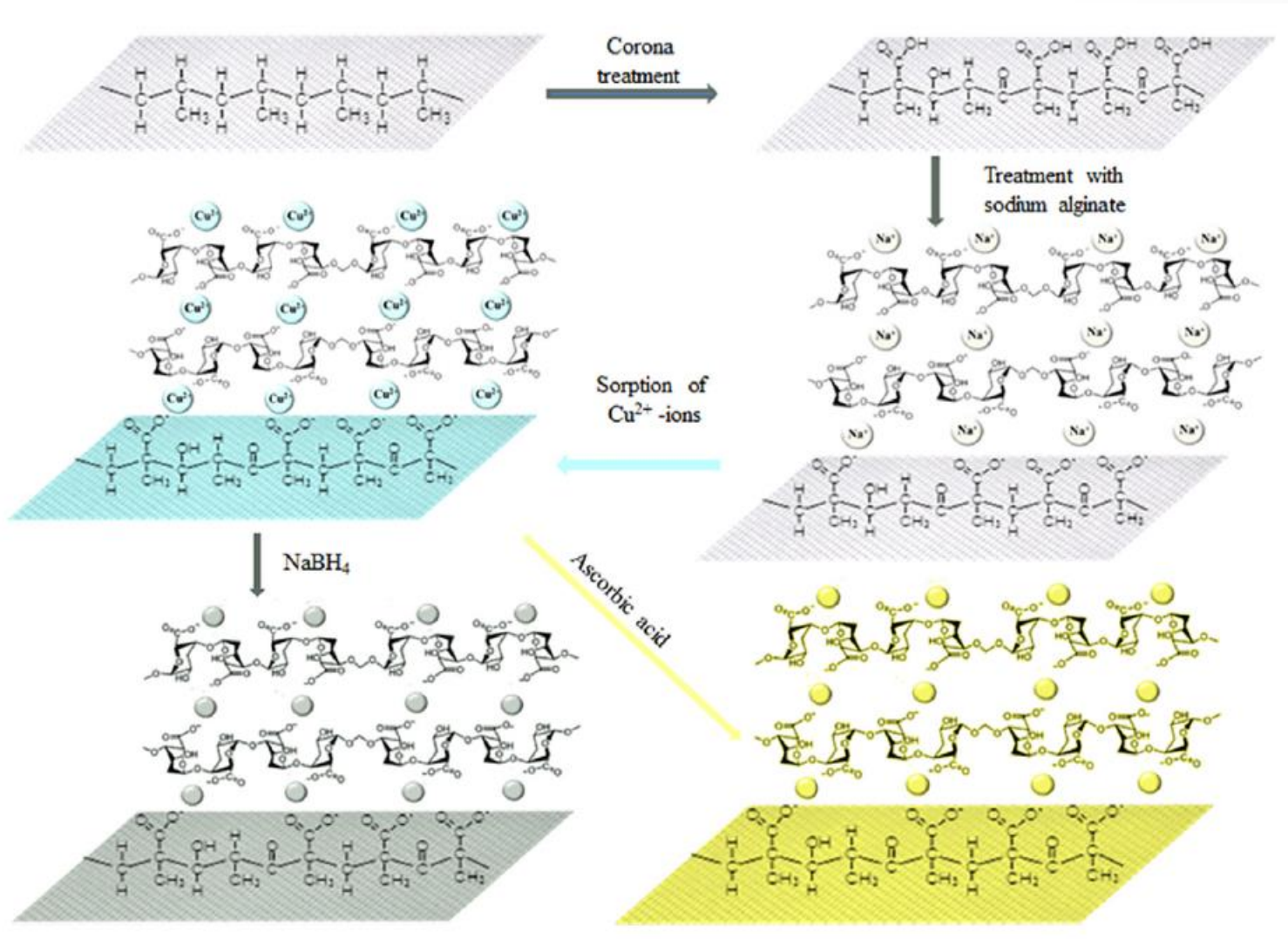
Milošević, Milica, et al. *Cellulose* 21.5 (2014): 3781-3795.

Marković, D., et al. (2018). *Carbohydrate polymers*, 200, 173-182

Marković, Darka, et al. *Fibers and Polymers* 20.11 (2019): 2317-2325.



# Functionalization of medical textiles

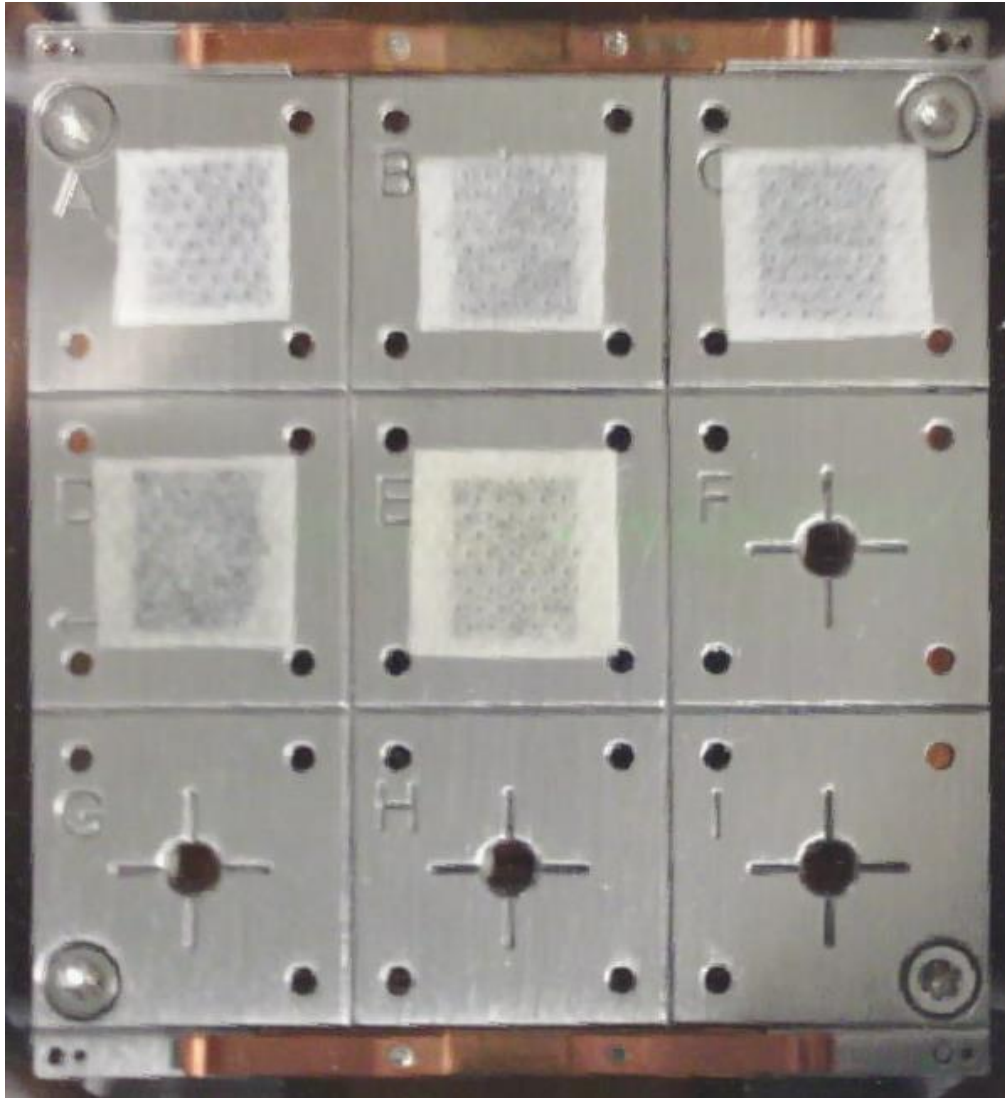


## Scheme:

- Polypropylene raw material
- Corona discharge processing in air
- Treatment with sodium alginate
- Immersion in  $\text{CuSO}_4$  solution
- Reduction step
  - With  $\text{NaBH}_4$
  - With ascorbic acid



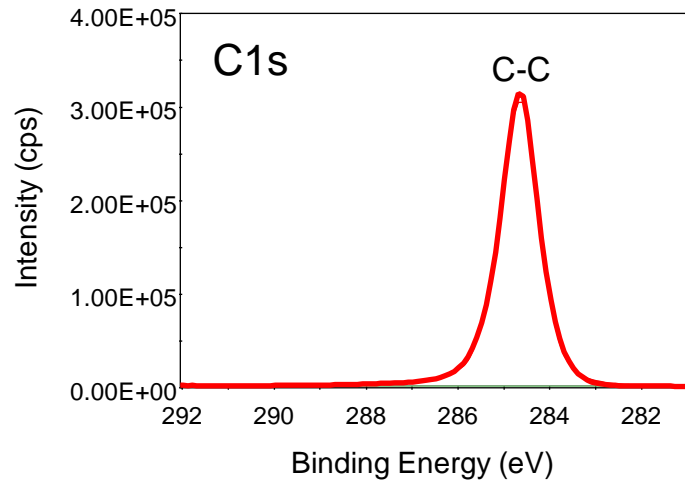
# Sample mounting



Position	Sample
A	Polypropylene
B	After corona treatment
C	After treatment with Na alginate
D	After reduction with $\text{NaBH}_4$
E	After reduction with ascorbic acid

# Effects of corona discharge and alginate treatments on PP

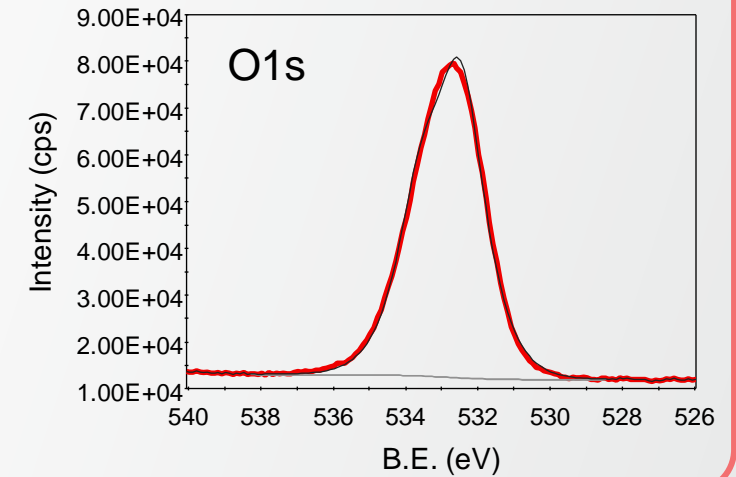
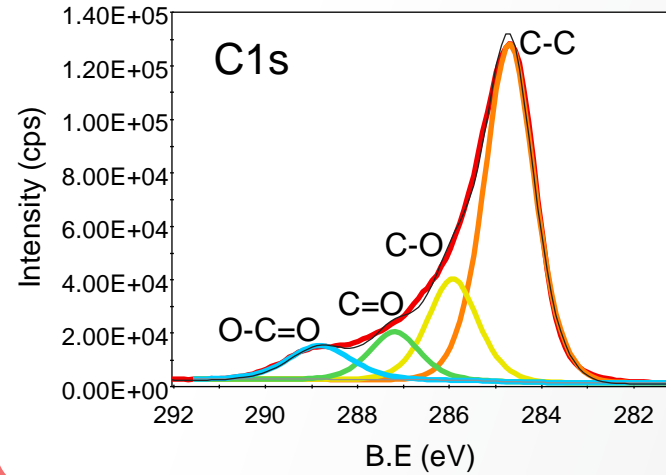
Neat PP



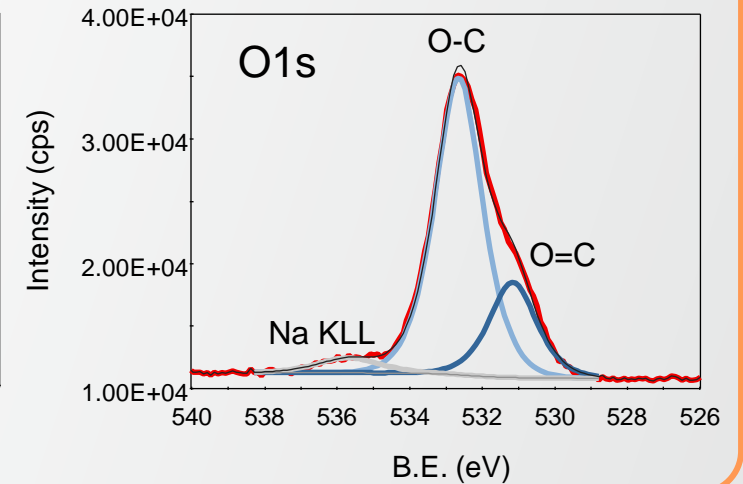
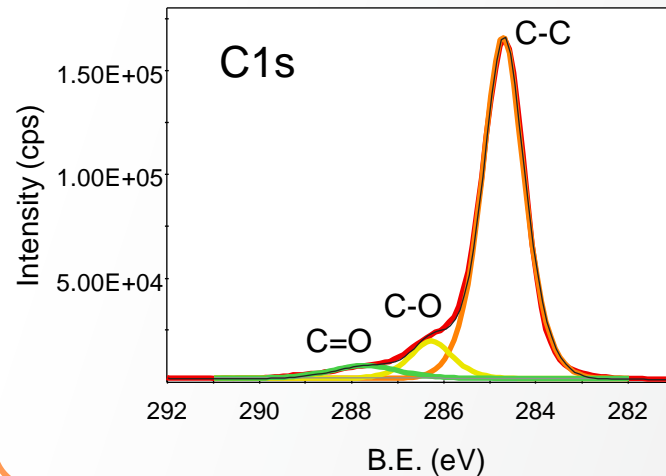
Significant changes to C1s upon corona discharge and alginate treatments

- Hydrophobic → hydrophilic surface

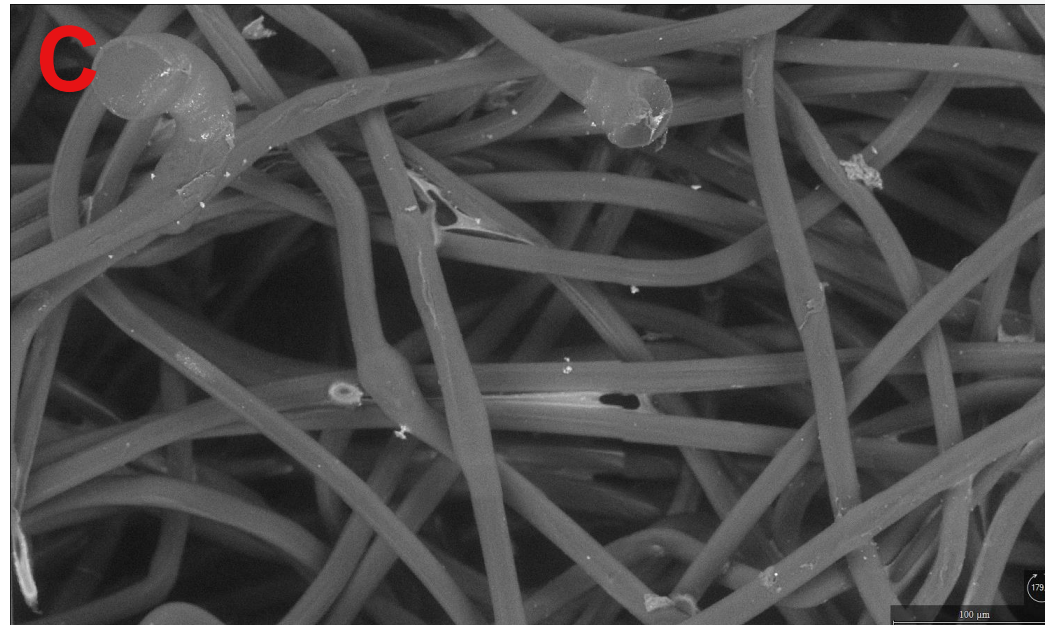
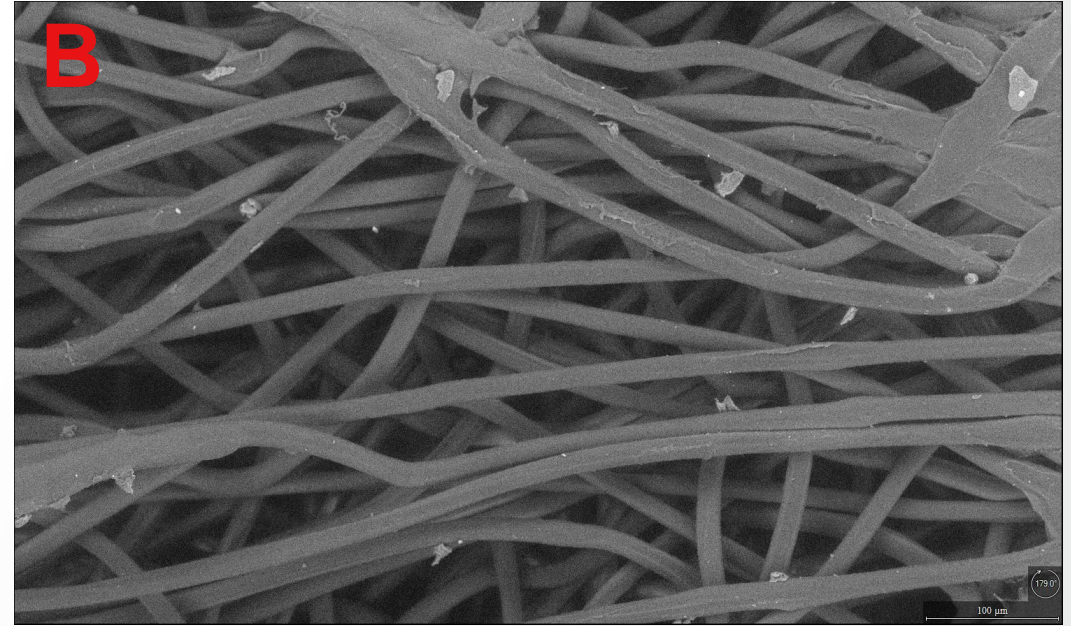
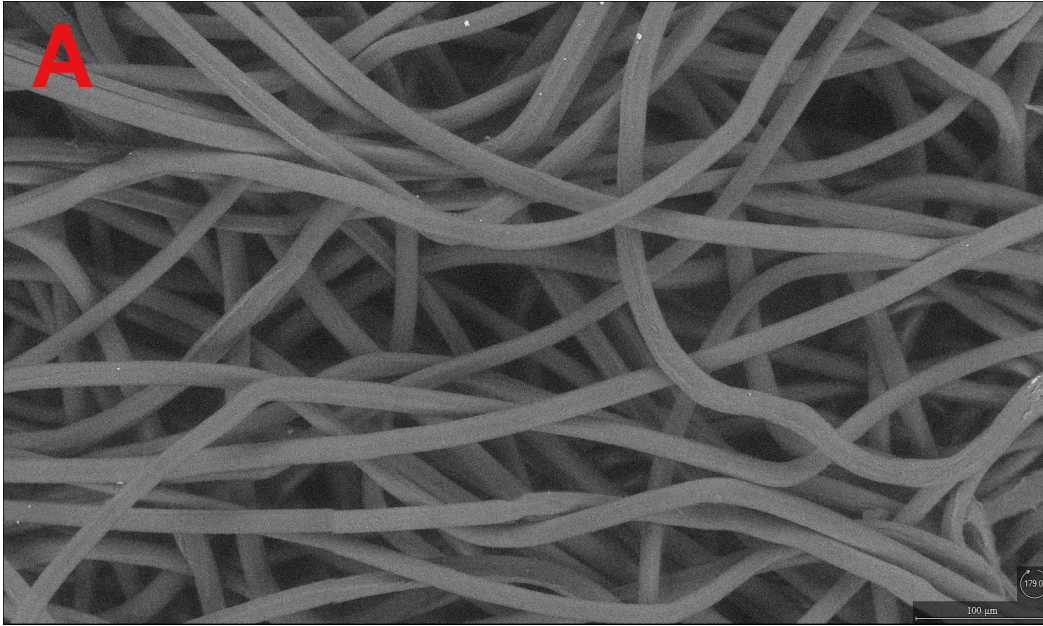
Corona discharge



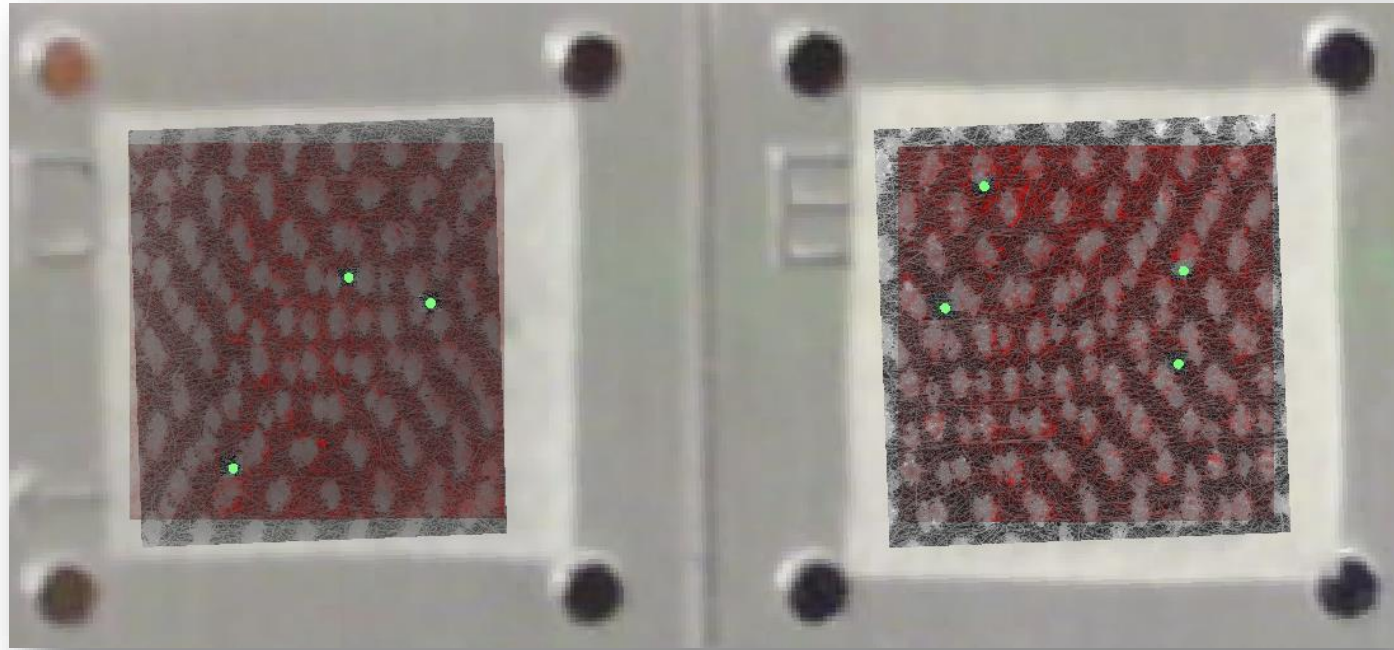
Alginate functionalization



# SEM images of fibres Samples A - C

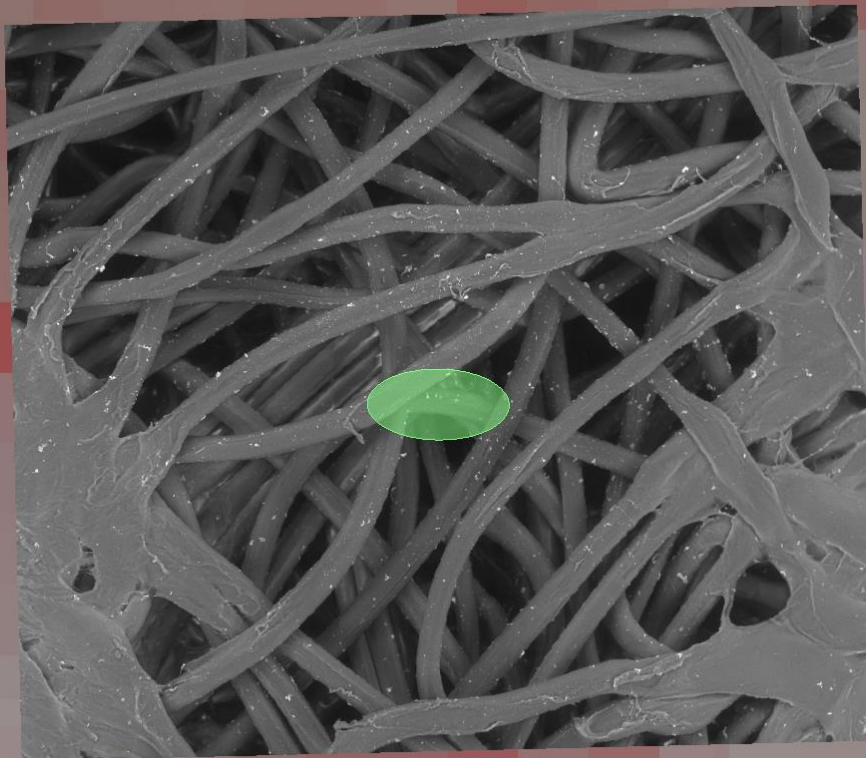


# Cu NP + PP samples

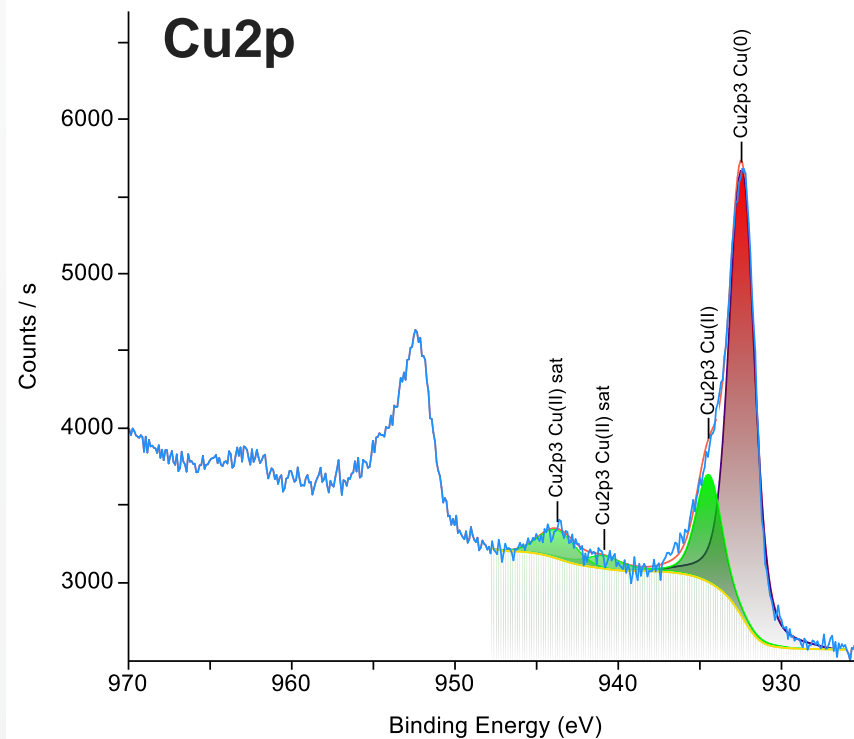
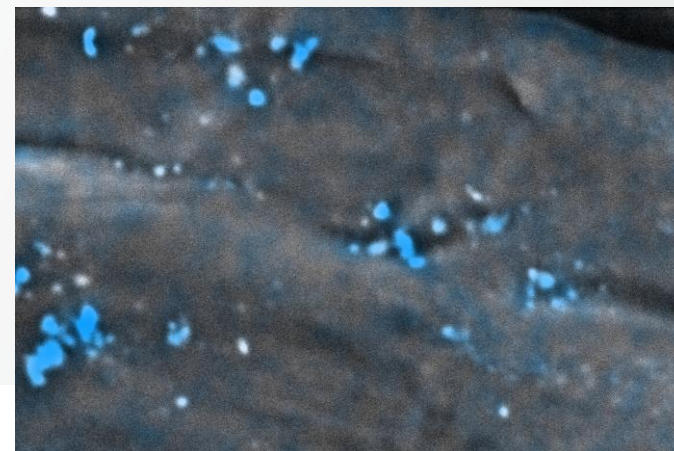


- Sample imaged using SnapMap in the Nexsa G2, identifying variation in Cu concentration
- Data collected at points with high / low Cu
- Same positions imaged in Axia ChemiSEM

# Sample D – aggressive reduction with $\text{NaBH}_4$

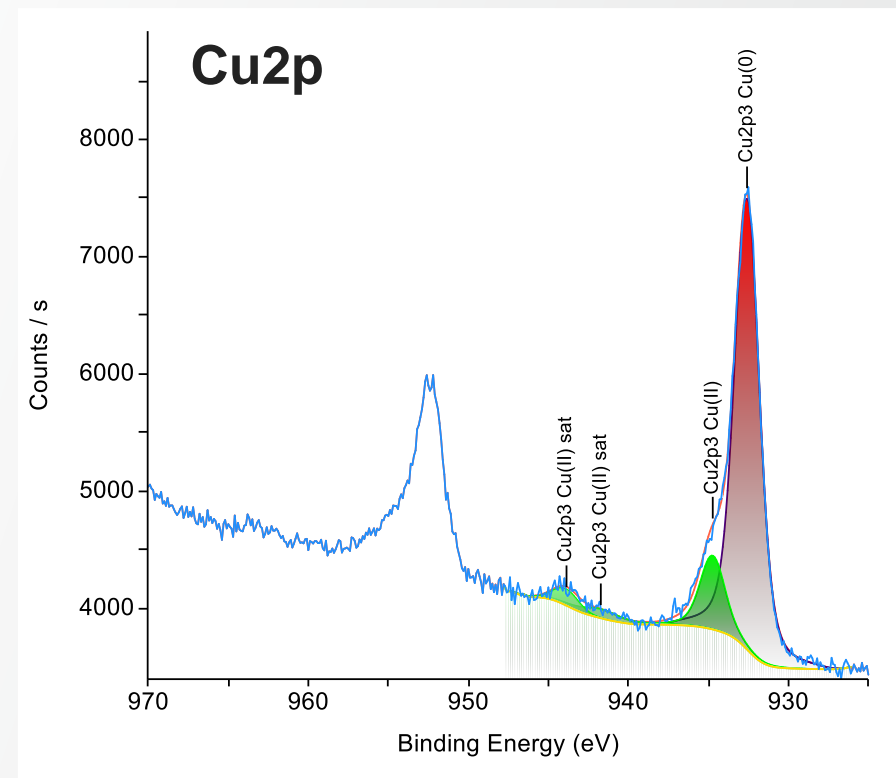
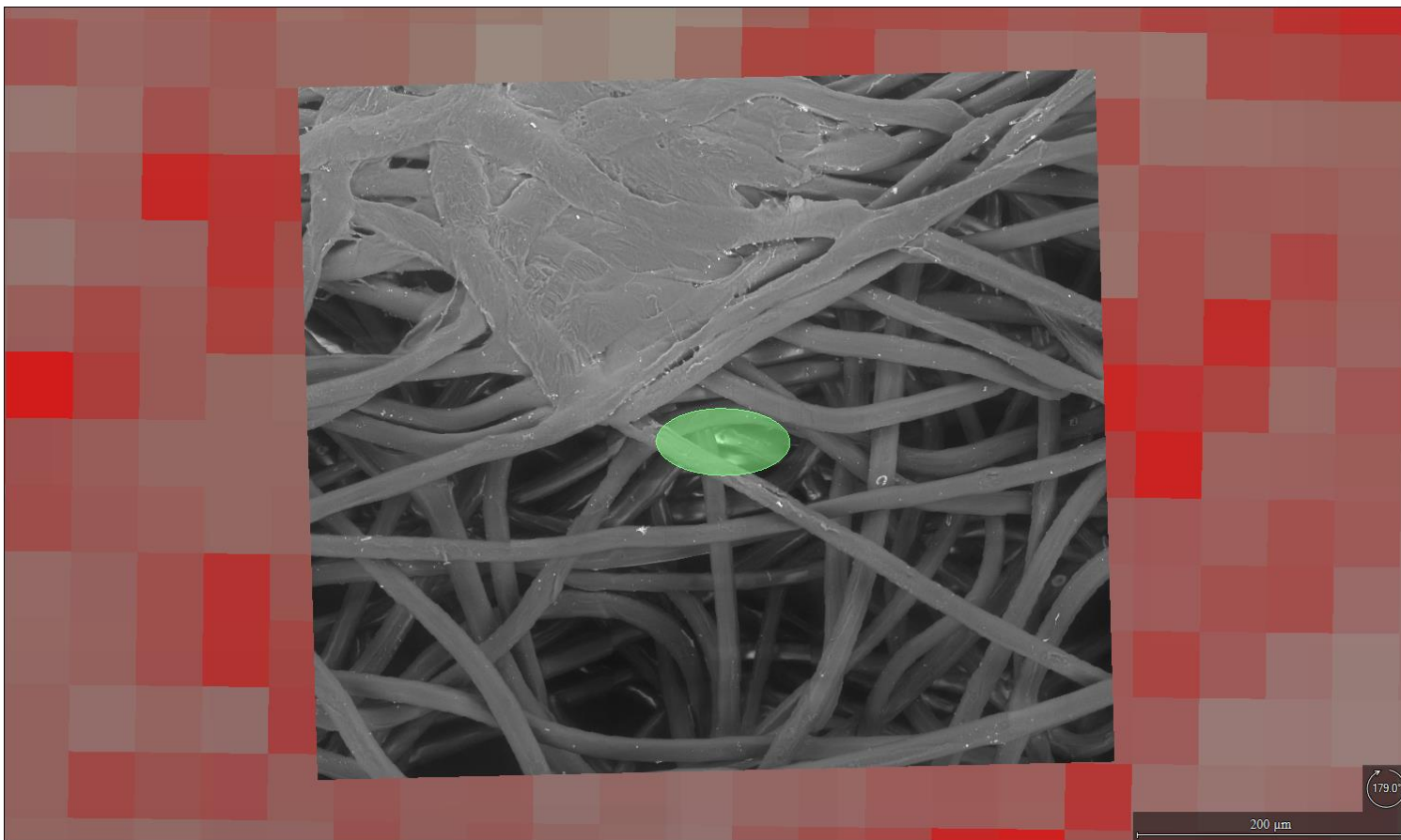


179.0°  
200 μm



Name	Peak BE	Height CPS	FWHM eV	Area (P) CPS.eV	Atomic %
C 1s	285.38	56529.90	1.62	190440.49	87.88
O 1s	532.74	13595.11	3.32	51082.88	9.75
Cu 2p	933.26	11739.93	2.85	76480.98	2.37

# Sample E – milder reduction with ascorbic acid



Name	Peak BE	Height CPS	FWHM eV	Area (P) CPS.eV	Atomic %
C 1s	285.26	80702.33	2.62	258905.31	87.44
O 1s	532.75	20850.79	3.18	75621.07	10.57
Cu 2p	933.27	14312.06	2.86	87859.63	1.99

# Anti-microbial activity of synthesized nanocomposites

Number of microbial colonies (CFU/ml)

Micro-organism	Innoculum	Control PP	CPP + ALG + Cu1	CPP + ALG + Cu2
<b>E. coli (ATCC 25922)</b>	$4.1 \times 10^6$	$1.1 \times 10^6$	<10	<10
<b>S. aureus (ATCC 25923)</b>	$8.0 \times 10^5$	$5.0 \times 10^5$	<10	<10
<b>C. albicans (ATCC 24433)</b>	$6.0 \times 10^5$	$1.0 \times 10^5$	<10	<10

- PP + CPP & PP + CPP + ALG showed similar performance to the Control PP fabric
- Both Cu treatments produce excellent antimicrobial activity
- Ascorbic acid route is effective, even though the Cu loading is ~ 50% of NaBH<sub>4</sub> reduced samples

# CISA workflow

- Enabling analysis at the same regions of interest on the sample in XPS & SEM instruments
- Surface chemistry identification and quantification
- Surface structure visualisation
- Compare bulk & surface chemistry using XPS, ISS and EDX
- Consolidate sample information using Maps





# Thank you

See also: Session A10.6 - Thu 4<sup>th</sup> Aug – 4:15pm

