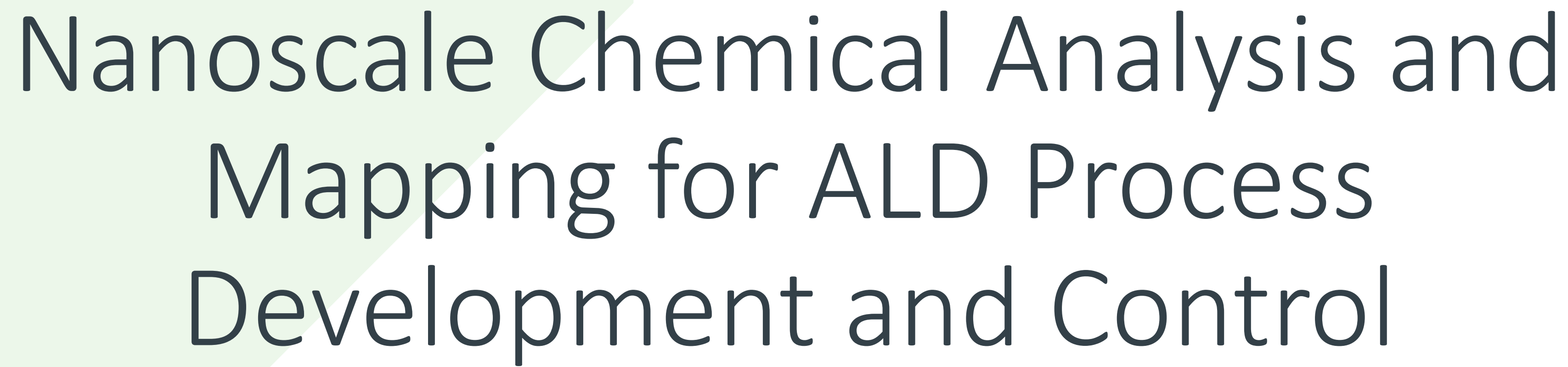
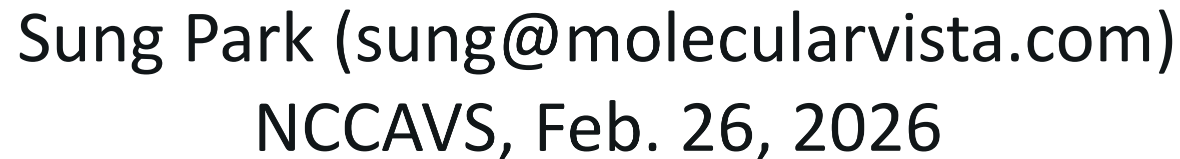


Nanoscale Chemical Analysis and Mapping for ALD Process Development and Control



Sung Park (sung@molecularvista.com)

NCCAVS, Feb. 26, 2026

Complements other standard analytical techniques

	IR PiFM	Raman	FTIR	TOF-SIMS	XPS	TXRF	SEM/EDS	TEM	Auger
Species Detected	**M.I.**	M.I.	M.I.	M.I.	M.I.	E.I.	E.I.	E.I.	E.I.
Chemical Mapping	**Yes**	Yes	Yes	Yes	Yes	Yes (Elemental)	Yes (Elemental)	Yes (Elemental)	Yes (Elemental)
Lateral Resolution	**< 5 nm ~ 20 nm+**	> 0.5 μm	> 10 μm	> 0.2 μm	10 μm – 2 mm	~ 1 mm	1 nm* 0.5μm EDS	0.2 nm* 1 ~ 20 nm EDS	~ 10 nm
Depth Probed	**< 20 nm ~ 500 nm+**	> 500 nm	1 μm	1 nm	10 nm	10 nm	1 μm	~ 100 nm	10 nm

+ bulk mode   * imaging   M.I. molecular information   E.I. elemental information

Color coded for semiconductor applications with small dimensions.

IR PiFM brings molecular analysis to the realm of true nanoscale, providing both IR absorption spectra and chemical mapping with ~ 5 nm spatial resolution and monolayer sensitivity.

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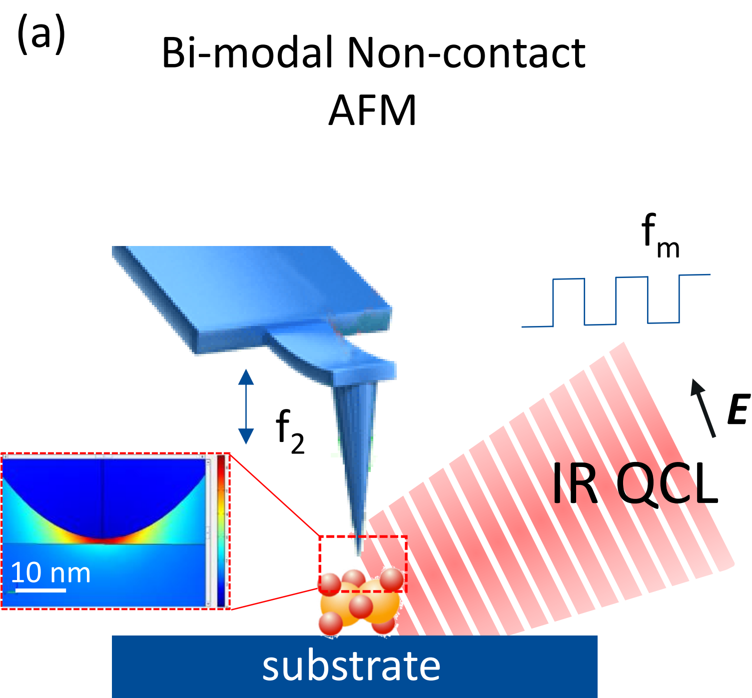
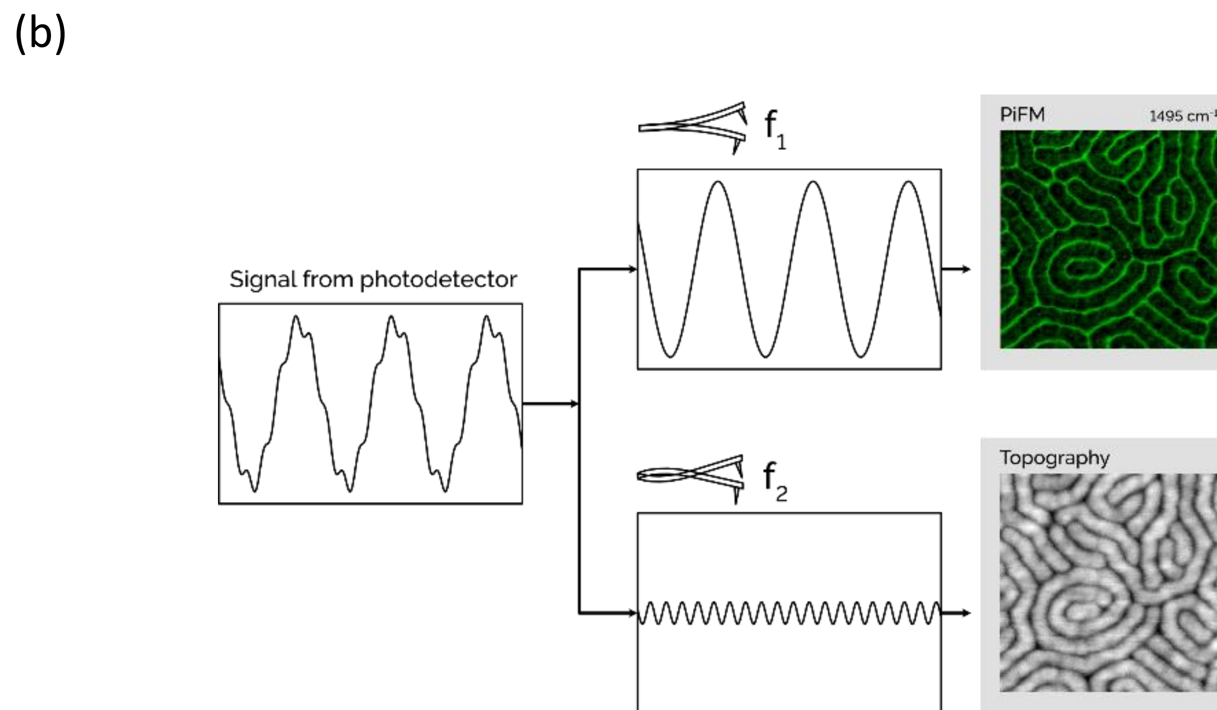
Infrared Photo-induced force microscope (IR PiFM)





A multi-modal AFM approach can collect PiFM chemical maps and topography images simultaneously.

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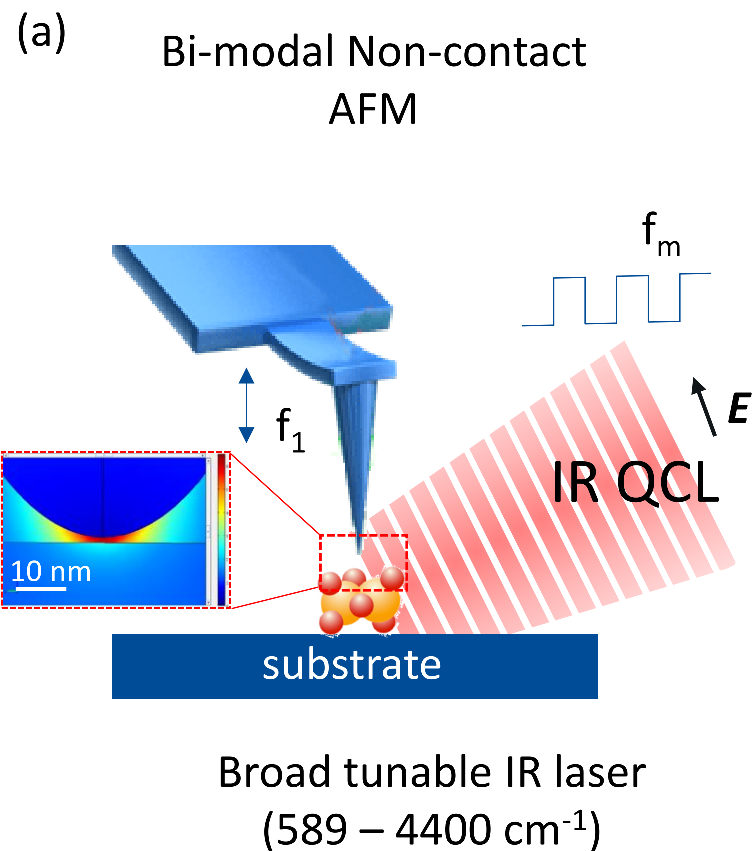
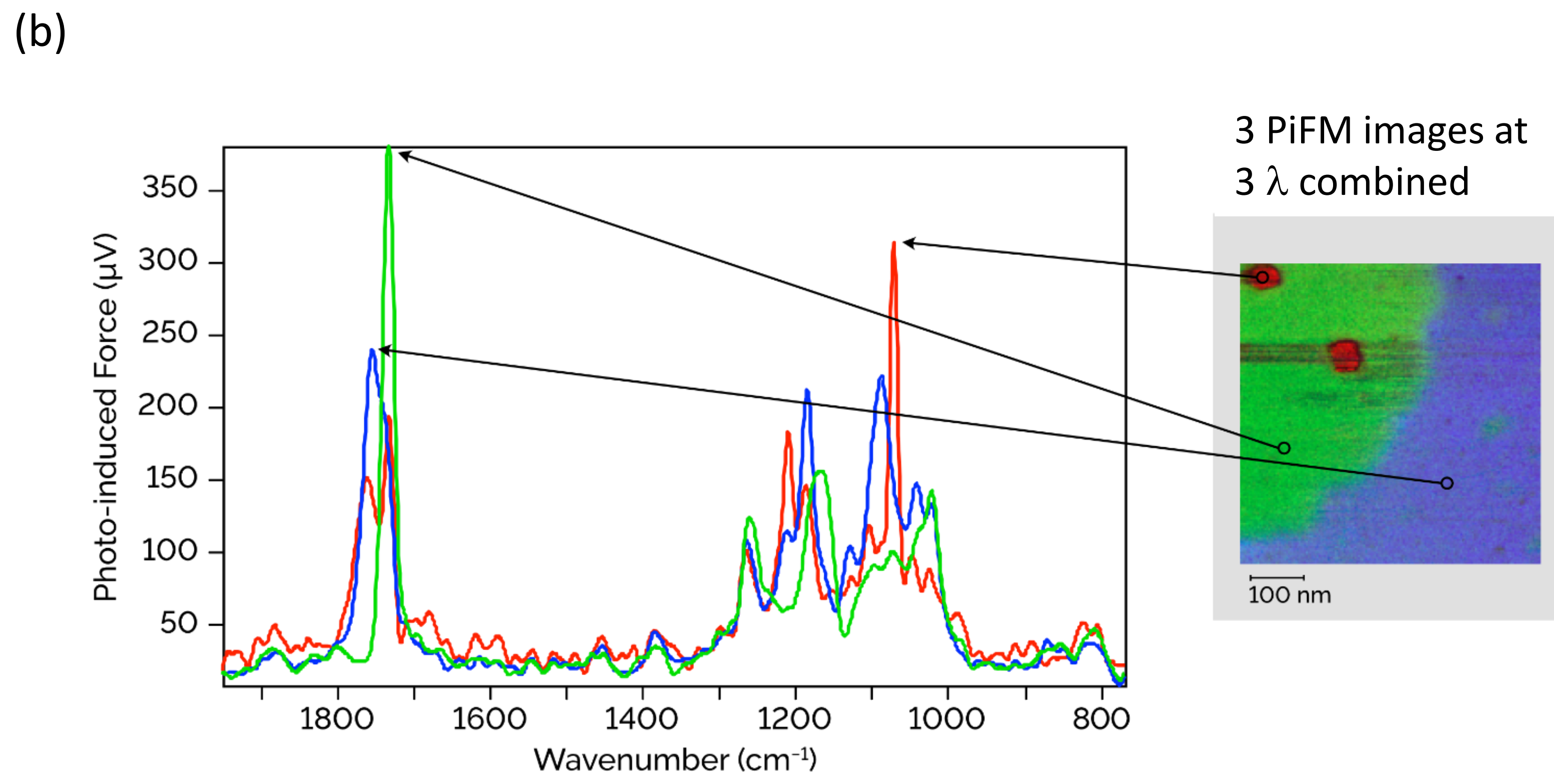
Infrared Photo-induced force microscope (IR PiFM)





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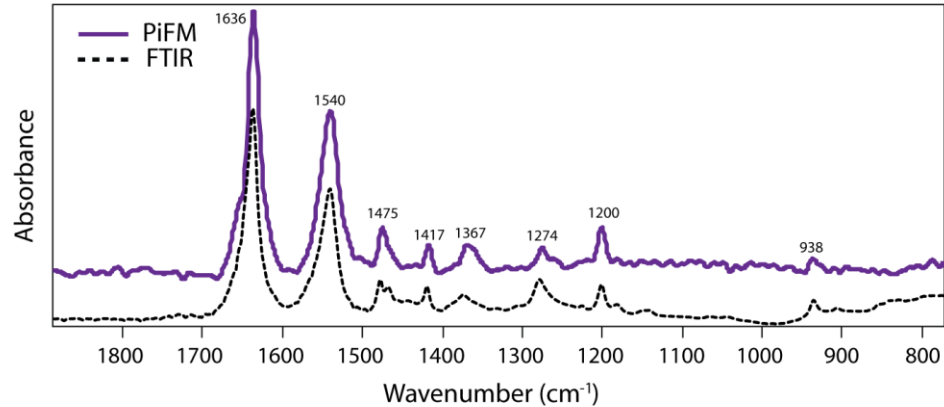
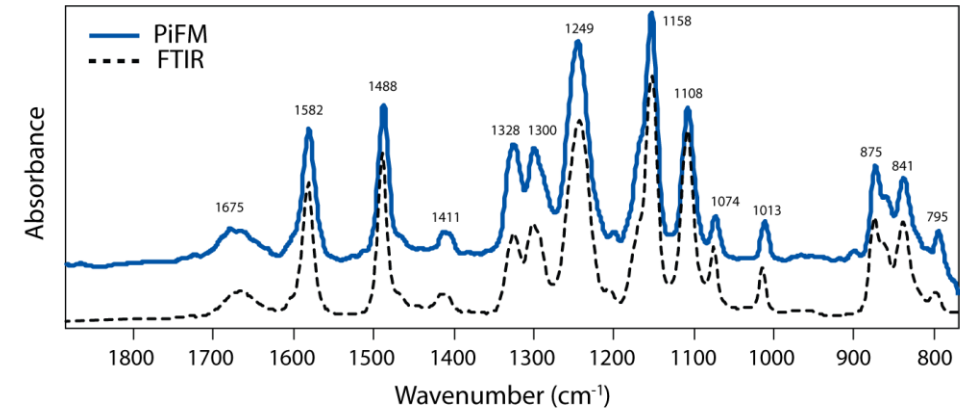
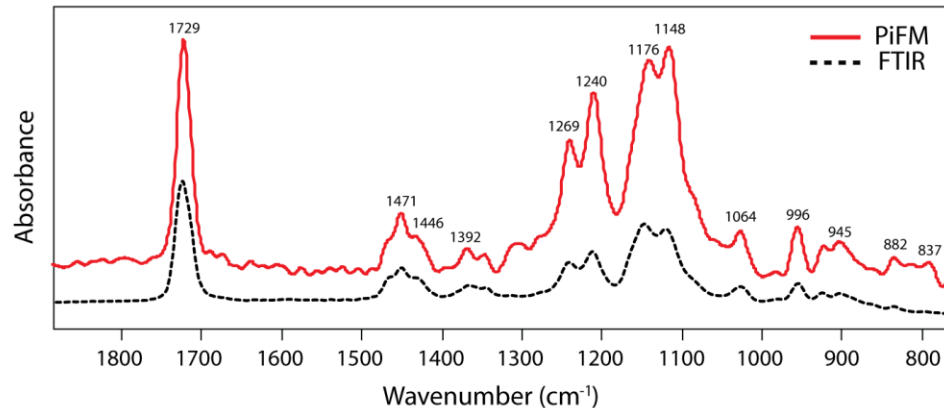
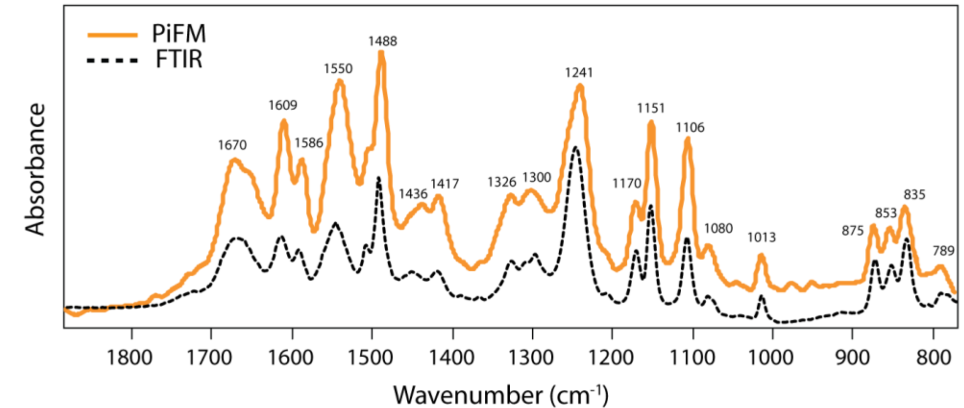
Good agreement between PiF-IR and FTIR spectra









Excellent agreement between PiF-IR spectra and FTIR spectra on homogeneous samples. Allows the use of IR libraries for defect analysis.

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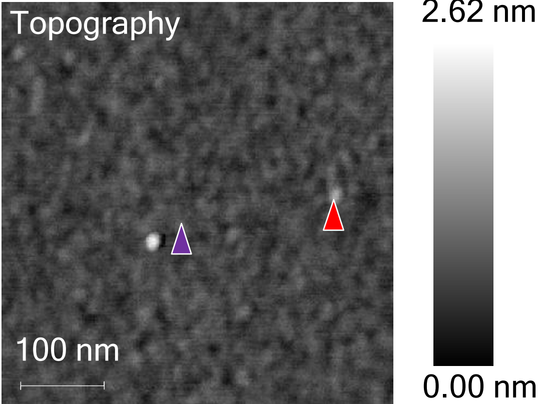
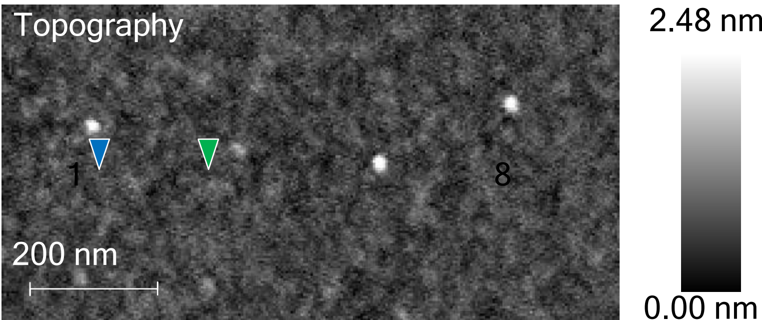
PiFM for Analysis of Sub-20 nm
Organic/Inorganic Defects

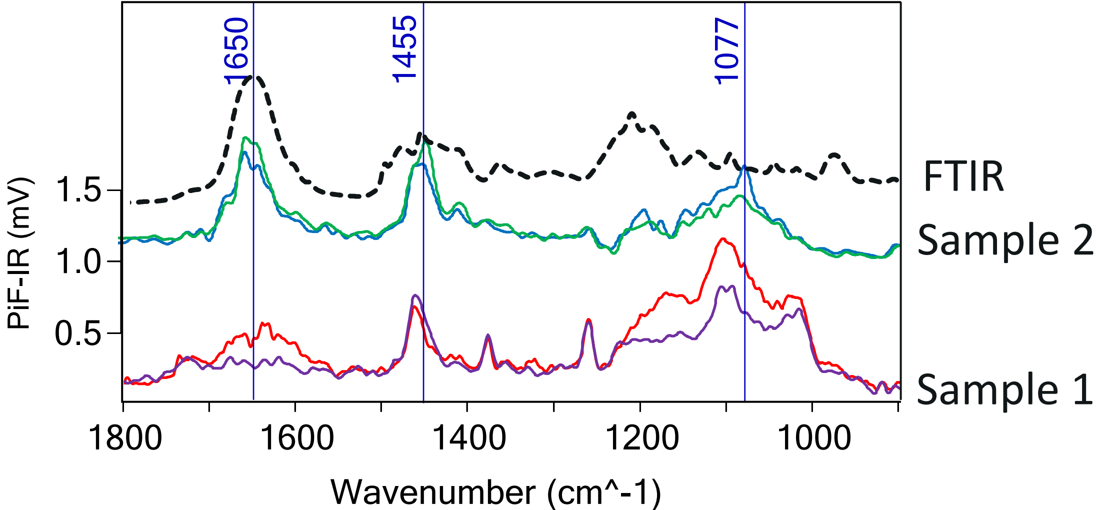
How can you tell if you have a conformal monolayer film?

Sample 1, Carboxylic



Sample 2, Hydroxyl





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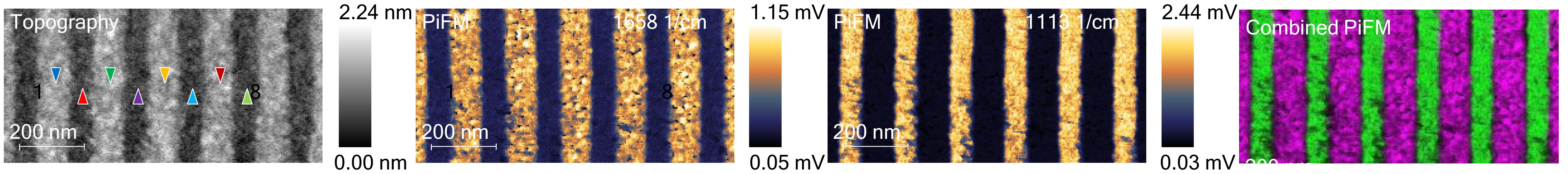
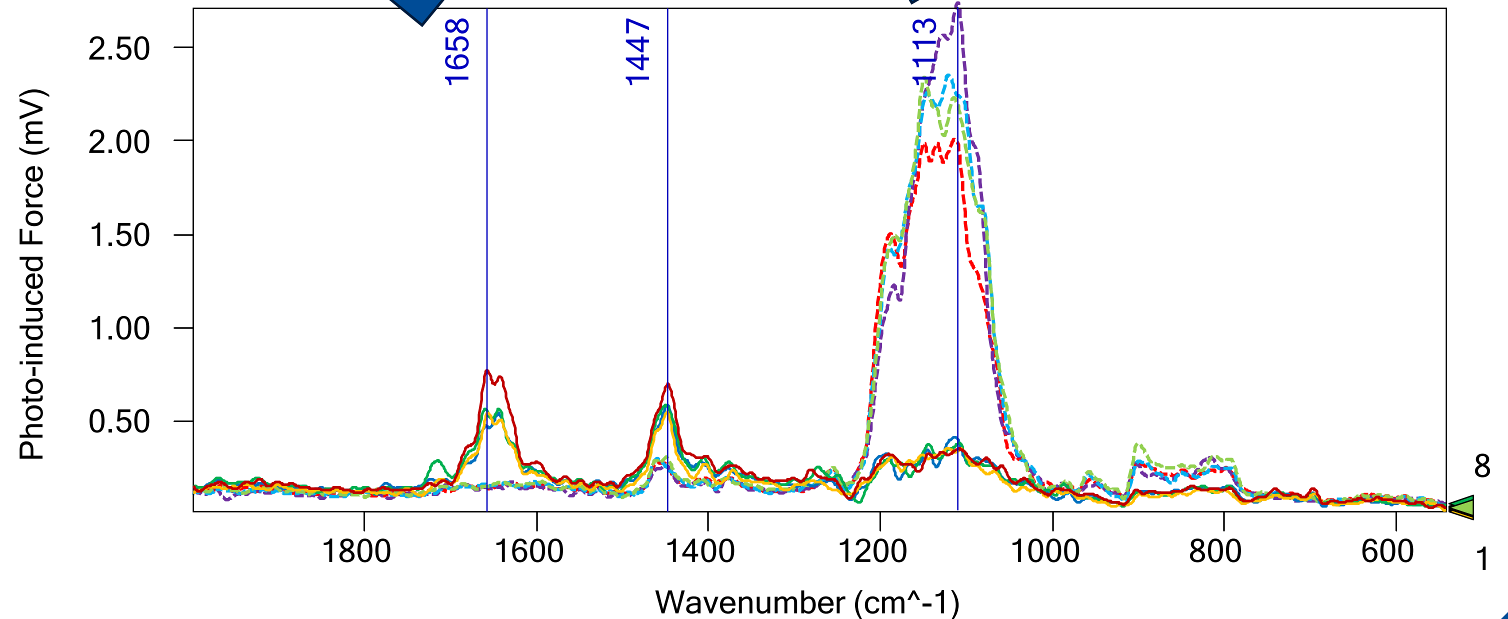
Patterned Polypeptoid Brush Monolayer



Peptoid (1658 cm^{-1})
SiO_2 (1113 cm^{-1})

Only ~ 1 nm thick, pitch of 170 nm



PiF-IR Analysis of ALD Grown Al_2O_3

Photo-induced Force (µV)

800
600
400
200

FTIR

1300 1200 1100 1000 900 800 700 600

Wavenumber (cm^-1)

1 nm 2.5 nm 5 nm 10 nm 25 nm 50 nm

Four spectra (1 µm spacing) are acquired from each sample; they are reasonably repeatable indicating that each sample is homogeneous. Compared to the broad FTIR spectrum, PiF-IR spectra display sharper features that vary with thickness.

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High-K Dielectric

Blue = HK Thk = 0.1 Å
Black = HK Thk = 3.3 Å
Gold = HK Thk = 4.5 Å

Photo-induced Force (mV)
1.50
1.00
0.50
3500 3000 2500 2000 1500 1000
Wavenumber (cm^-1)

PiFM
500 nm
1.09 mV
0.44 mV

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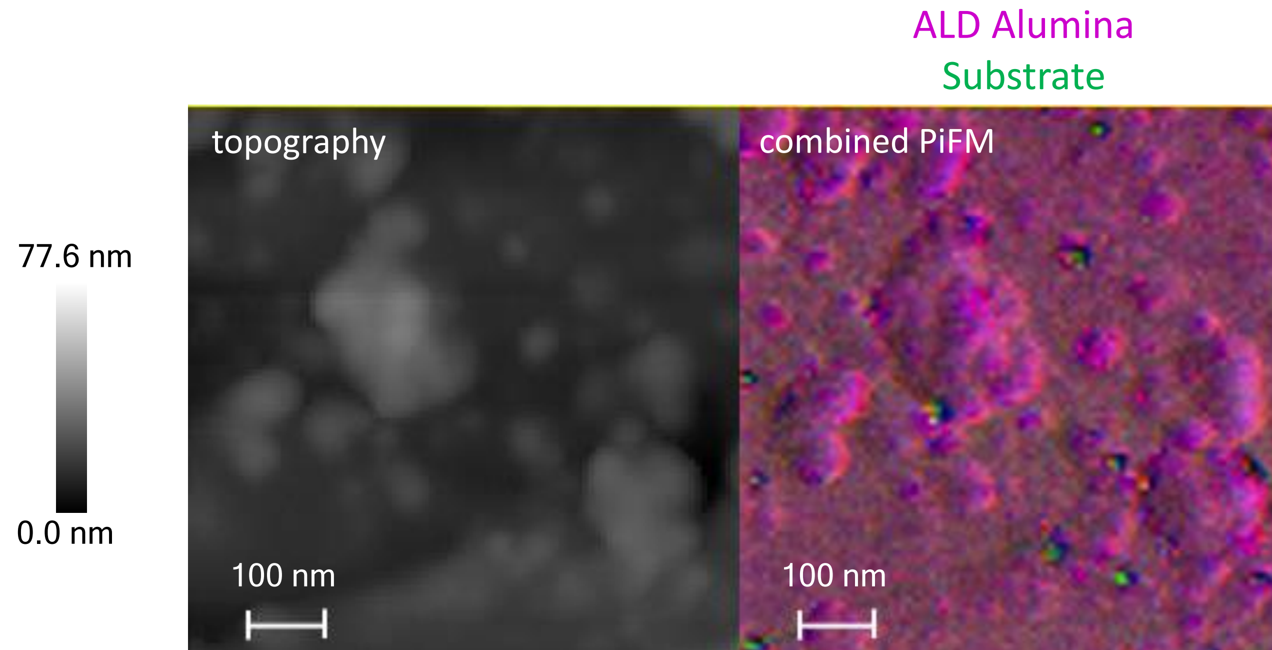
PiFM Image of ALD Grown Al_2O_3



Alumina is grown on top of a substrate material via ALD and examined via IR PiFM. Topography is show on the left. A combined PiFM image consisting of one PiFM image for alumina (purple) and another one for the substrate (green) clearly shows multiple pinholes (~ 10 nm in size) in the alumina layer.

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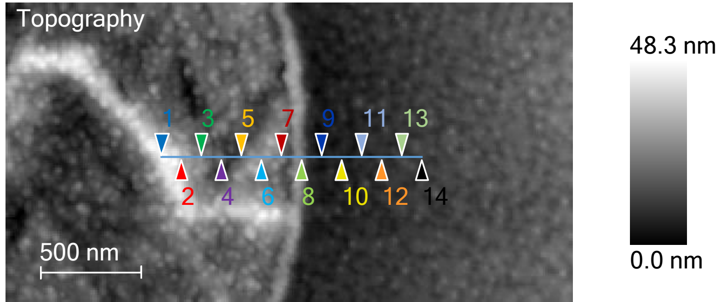
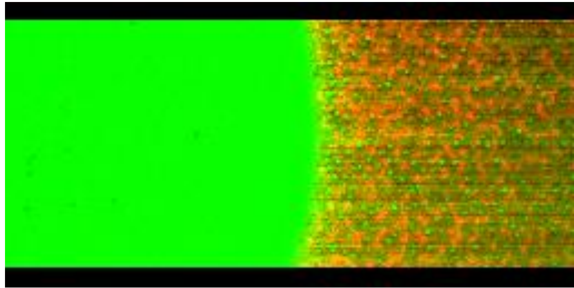
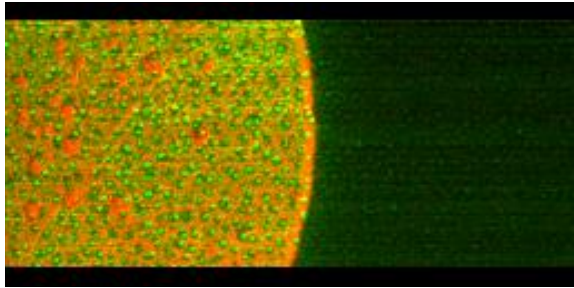
Other Applications

Monitoring Post Glue-clean Wafer





Highlights glue residue at WN1 (green) in the oxide region. The display is adjusted to show the oxide region. Due to the enhanced field enhancement on Cu region, it appears much brighter even at WN1.



Highlights glue residue at WN1 (green) in the Cu pad region

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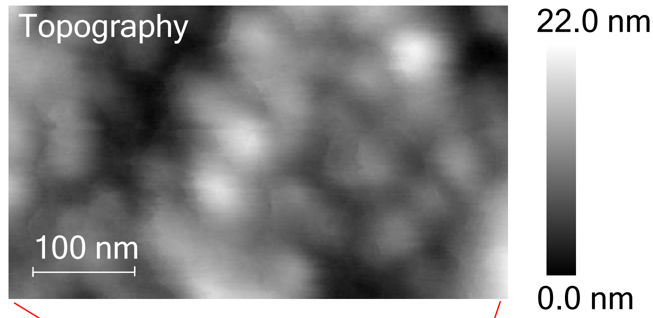
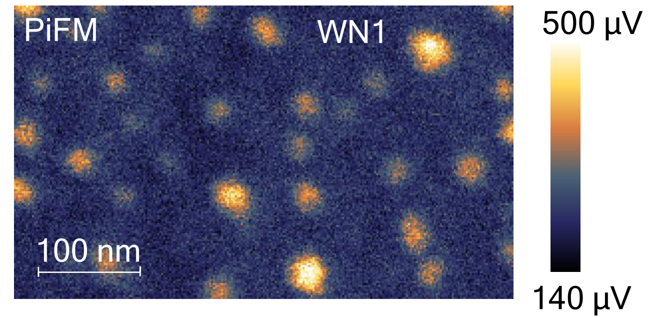
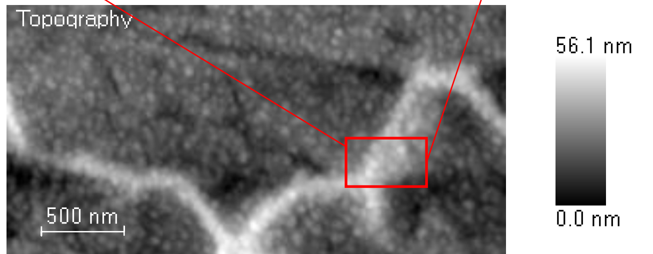
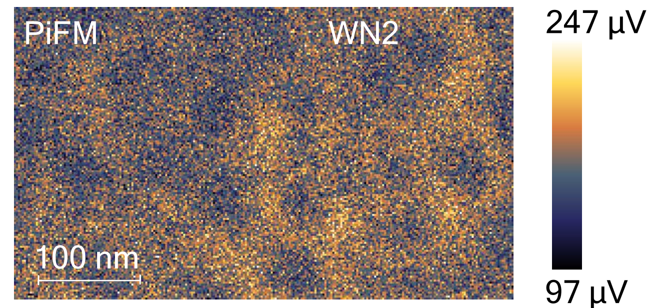
Zoomed-in Cu Pad Region



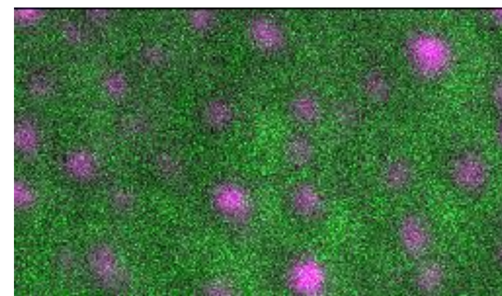


PiFM image at WN1 highlights the glue that remains even after the cleaning process.





PiFM image at WN2 highlights the Cu^{2+} based compounds (includes CuO and Cu-organic) on the copper pad.



Combined PiFM images show the glue residue (purple) on top of the copper pad (green).

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Automated Defect Review via KLARF Coordinates

KLARF Defect Map

C:\Users\James Woo\Downloads\Nathan Jarnagin-160nm_PWP_HTO_BS_s23_26.SurfScan.txt

- [x] DEFECTID
- [x] XTRANSLATED
- [x] YTRANSLATED
- [ ] XREL
- [ ] YREL
- [ ] XINDEX
- [ ] YINDEX
- [ ] XSIZE
- [ ] YSIZE
- [ ] DEFECTAREA
- [x] DSIZE
- [ ] CLASSNUMBER
- [ ] TEST
- [ ] CLUSTERNUMBER
- [ ] ROUGHBINNUMBER
- [ ] FINEBINNUMBER
- [ ] REVIEWSAMPLE
- [ ] CHANNELID
- [ ] ADDERFLAG
- [ ] IMAGEINFO
- [ ] POLARITY
- [ ] AREA
- [ ] DEFECTSIZE
- [ ] HAZEAVERAGE
- [ ] NPPMSIZE
- [ ] POSITION_BOX_THETAEND
- [ ] POSITION_BOX_THETASTART
- [ ] POSITION_RCENTROID
- [ ] POSITION_THETACENTROID
- [ ] RPPMSIZE
- [ ] SN_RATIO

Select All Columns | Select All Rows

DEFECTID	XTRANSLATED	YTRANSLATED	DSIZE
1	150463000	157089000	1668
2	150510000	157175000	26176
3	150536000	157260000	55571
4	150542000	157343000	58358
5	150517000	157435000	42529
6	150525000	157521000	41117
7	161223000	152611000	2292
8	161299000	152655000	76773
9	161387000	152668000	90608
10	161472000	152705000	75958
11	161557000	152721000	72292
12	139857000	162772000	35592
13	139800000	162847000	50783
14	139763000	162918000	102198
15	139716000	163003000	130518
16	139662000	163071000	138305
17	139595000	163130000	122068
18	139523000	163194000	84818
19	139495000	163272000	17347
20	135460000	157638000	18523
21	135400000	157705000	66252
22	135311000	157743000	87538
23	135246000	157811000	135062
24	135163000	157843000	156570
25	135081000	157883000	158978
26	134990000	157892000	115898
27	134152000	157425000	25056
28	134060000	157439000	81941
29	133978000	157461000	101969
30	133870000	157457000	118369
31	133783000	157462000	92523
32	133688000	157475000	59673
33	133621000	157555000	3233
34	133589000	157484000	14166
35	133512000	157517000	10673

3.0E+7

Defect #943

nm 150000000 , 150000000 Select Color Mapping:

Lasso Select

Adjust Point Size: + - 1x 10x 100x 1000x

Density Scan

X Coordinates	Y Coordinates	Density

Cancel | Import

Column to Filter:

Filter Type: Min - Max; Equal

Add Filter | Delete Selected | Delete All

Column	Filter Type	Value

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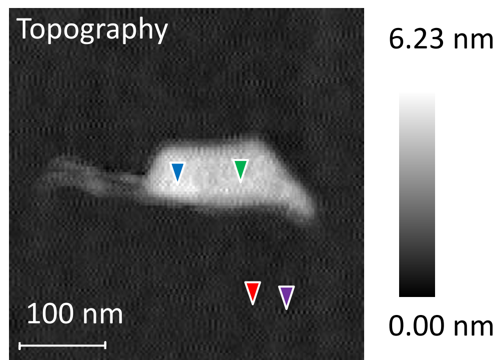
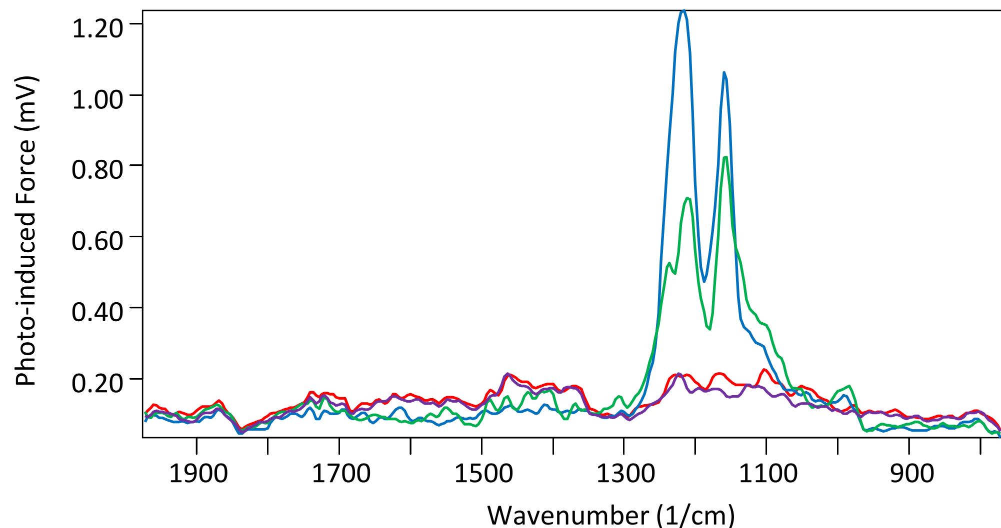
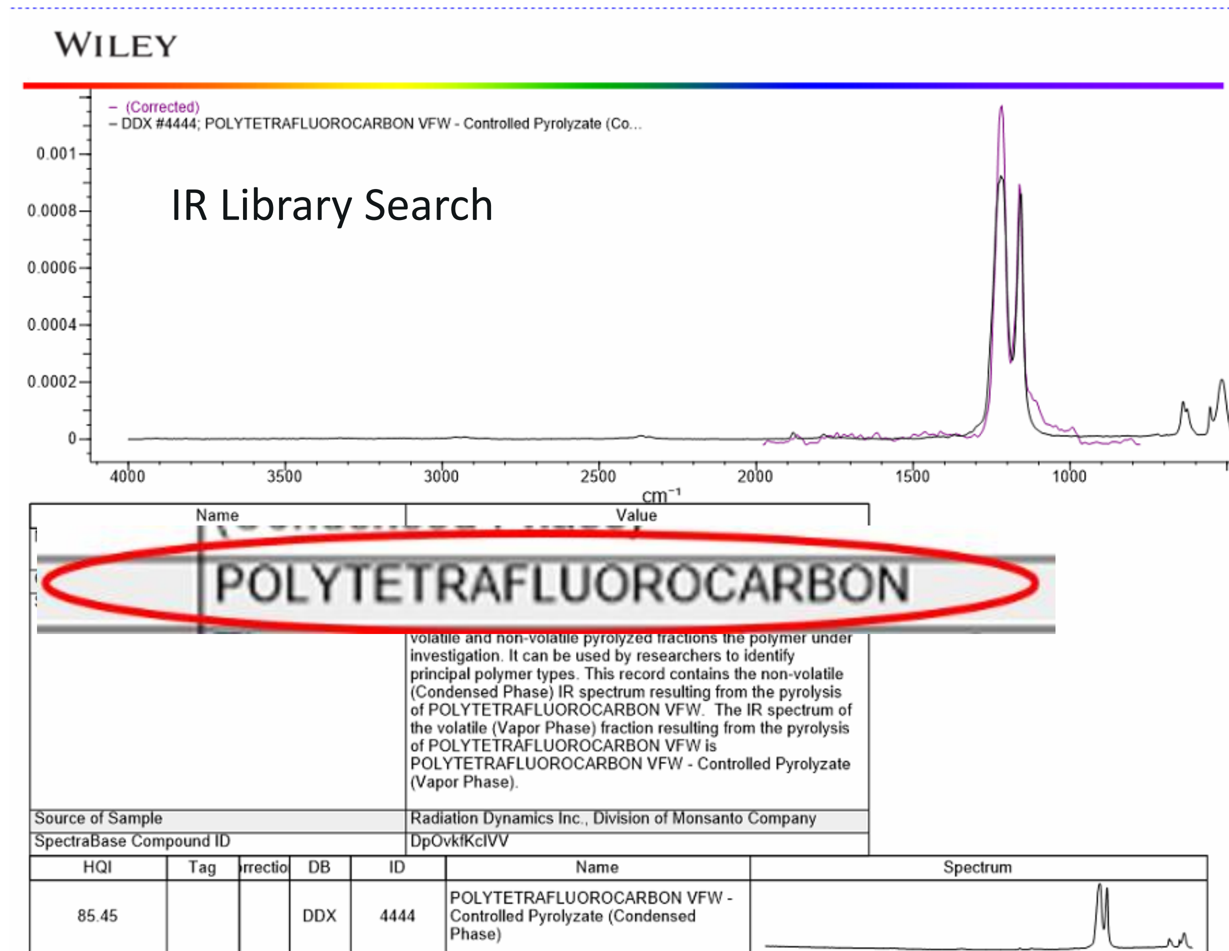
Sub-100 nm Organic Defects on Wafers

~ 5 nm thick, 50 nm x 100 nm







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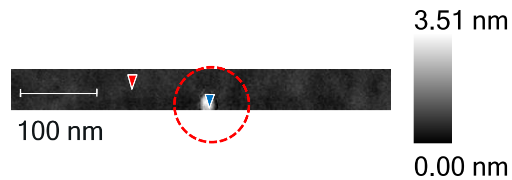
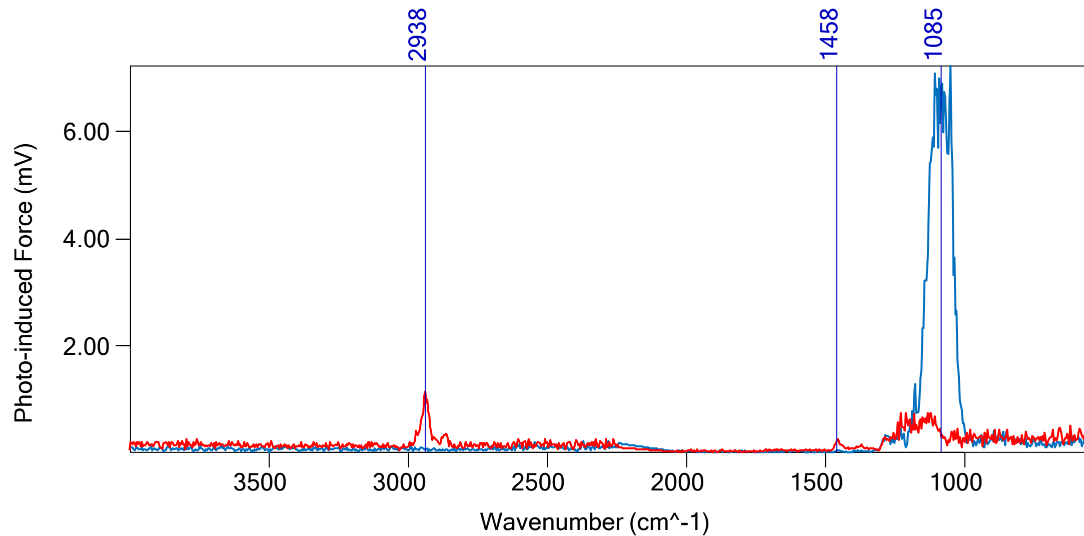
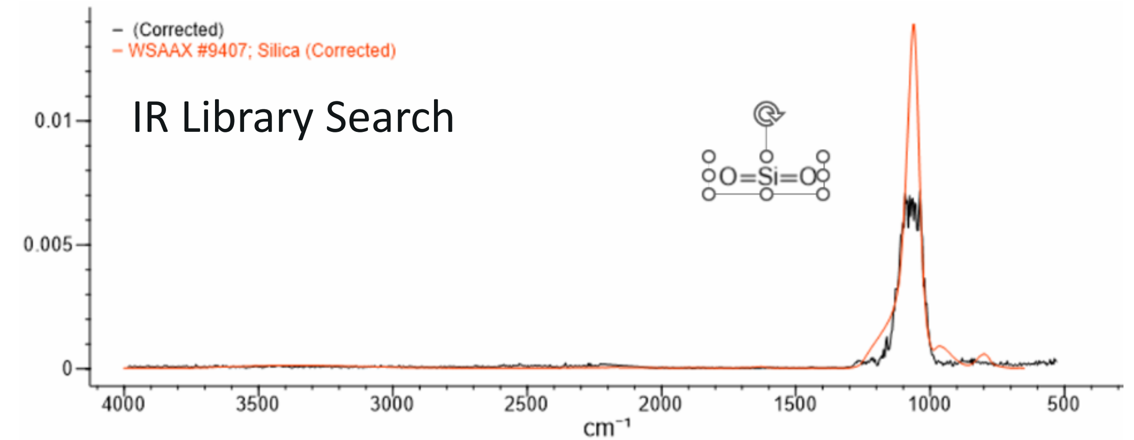
Sub-100 nm Inorganic Defects on Wafers

~ 3 nm particle







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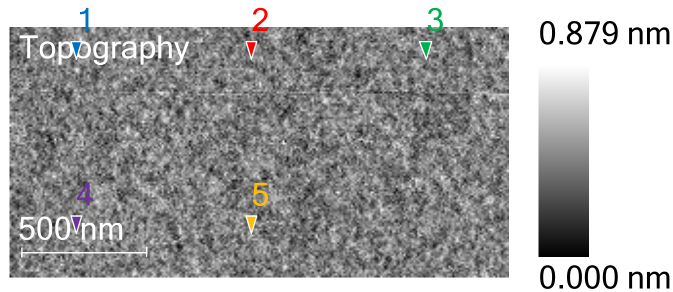
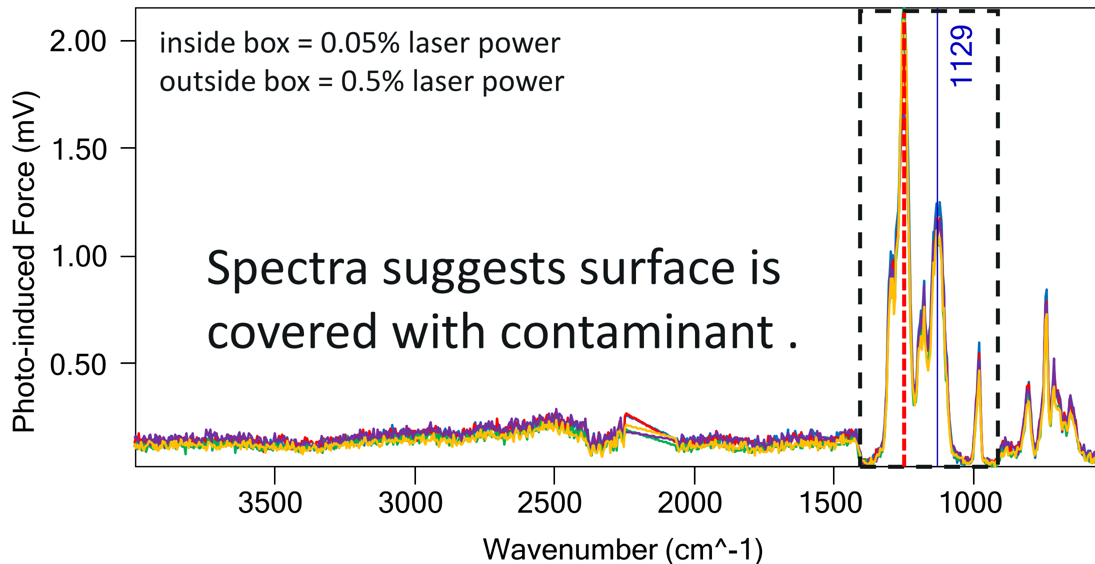
PFPE Contamination Monolayer on a Wafer Surface

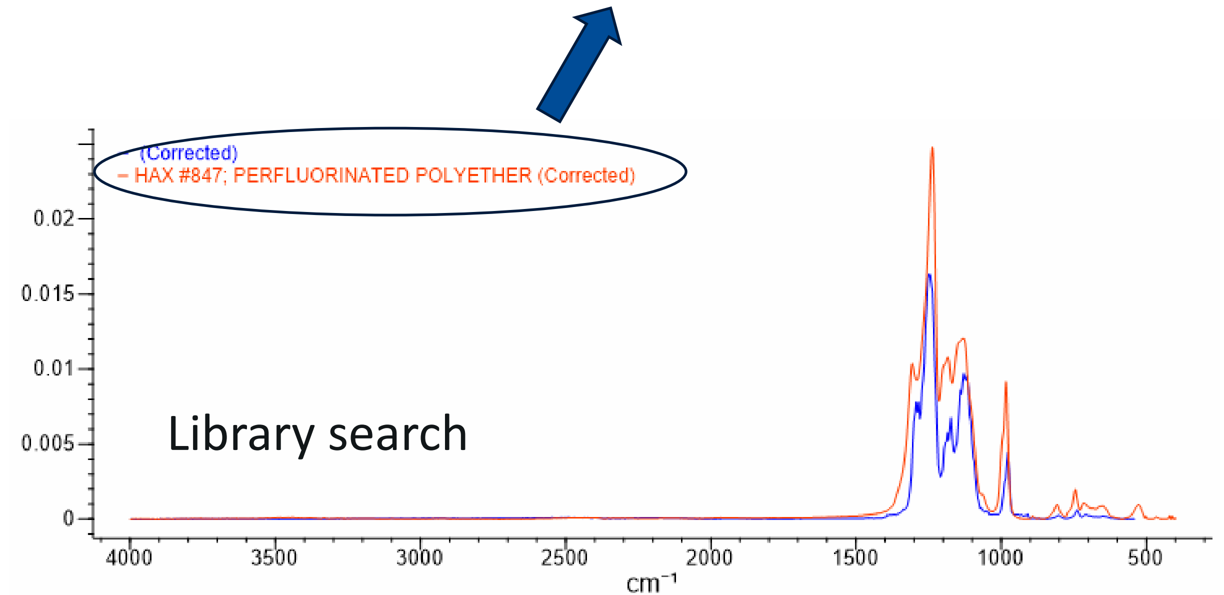
Topography reveals a standard flat wafer surface.





Given the micro-roughness and repeatable PiF-IR spectra, most likely a conforming monolayer.

Contaminated with perfluoro polyether (PFPE)



HQI	Tag	rrectio	DB	ID	Name
83.20			HAX	848	PERFLUORINATED POLYETHER

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Cross-section Analysis

Topography
92.5 nm
0.0 nm
1.00 µm

Topography
7.06 nm
0.00 nm
200 nm

Combined PiFM
200 nm

$1200\ cm^{-1}$
$970\ cm^{-1}$

PiFM 1200 1/cm
1.04 mV
0.12 mV
200 nm

PiFM 970 1/cm
729 µV
141 µV
200 nm

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Summary

- IR PiFM is introduced.
- PiFM measures sample's IR absorption via mechanical force detection.
- It provides for exceptional spatial resolution (< 5 nm), excellent surface sensitivity (monolayer), and ease-of-use.
- Vista-IR is a turnkey PiFM systems with visible to mid-IR lasers.
- Vista-IR can characterize SAM, ALD, and ALE processes based on the materials' IR signatures.
- Applications to monolayer/surface characterization and nano defect analysis were shared.

- Covalent Metrology has one of our tools and can provide analytical services.
- You are welcome to contact us to discuss your application needs: info@molecularvista.com.

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Thank You!

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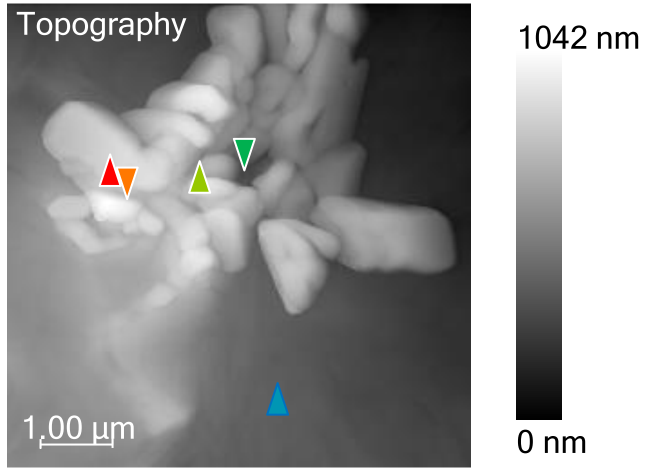
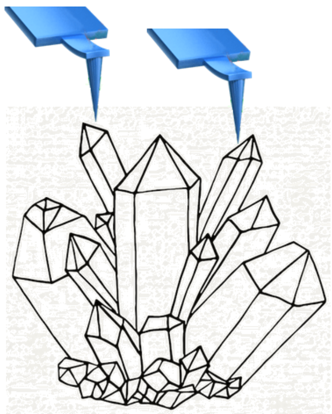
Molecular Orientation and PiFM

Discrimination of molecular orientation





Poly(e-Caprolactone)/Ascorbic Acid (PCL/5AA) – AA Crystals

PCL FTIR

AA FTIR

Photo-induced Force (mV)

1.80
1.60
1.40
1.20
1.00
0.80
0.60
0.40
0.20

1800 1700 1600 1500 1400 1300 1200 1100 1000 900 800

Wavenumber (cm^{-1})

Applications Lab

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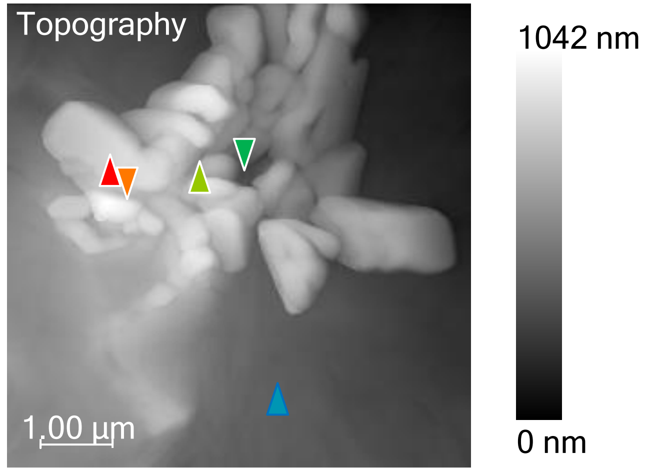
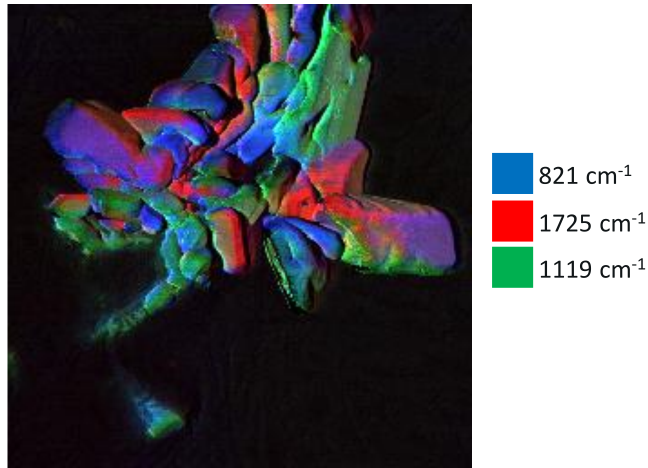
Discrimination of molecular orientation





Ensemble averaged FTIR versus local PiF-IR spectra

PCL FTIR

AA FTIR

Averaged PiF-IR

Photo-induced Force (mV)

1.80
1.60
1.40
1.20
1.00
0.80
0.60
0.40
0.20

1800 1700 1600 1500 1400 1300 1200 1100 1000 900 800

Wavenumber (cm^{-1})

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Combining S/TEM and PiFM: AA Crystallized on TEM Grid

S/TEM

1

100 µm

1

AFM/PiFM

Topography

2.00 µm

45.5 nm

0.0 nm

Topography

10.0 µm

75.9 nm

0.0 nm

Topography

500 nm

12.3 nm

0.0 nm

Combined PiFM

500 nm

1733 cm^{-1}

1670 cm^{-1}

1144 cm^{-1}

1732 1671 1143

Photo-induced Force (mV)

Wavenumber (1/cm)

Understanding & Modelling of IR Peaks of AA

We are planning scanning nanobeam electron diffraction (SNBED) analysis on the same measurement locations to correlate local crystal structure to phonon vibrations.

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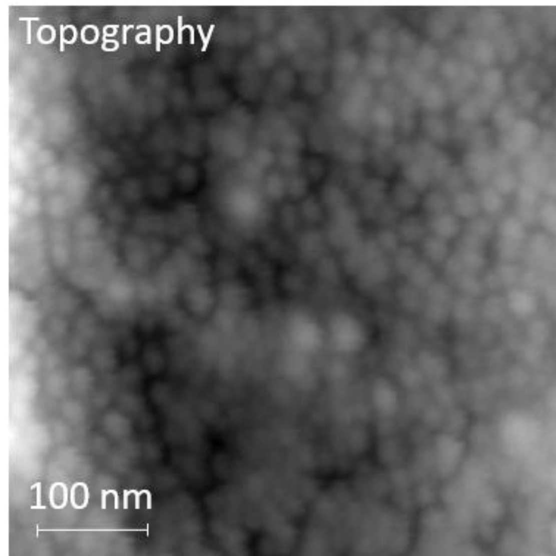
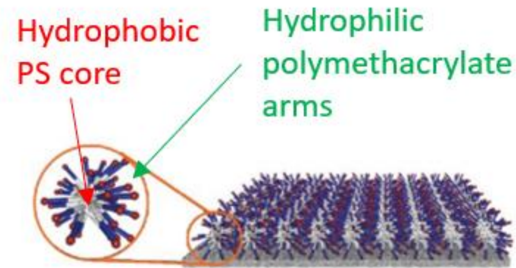
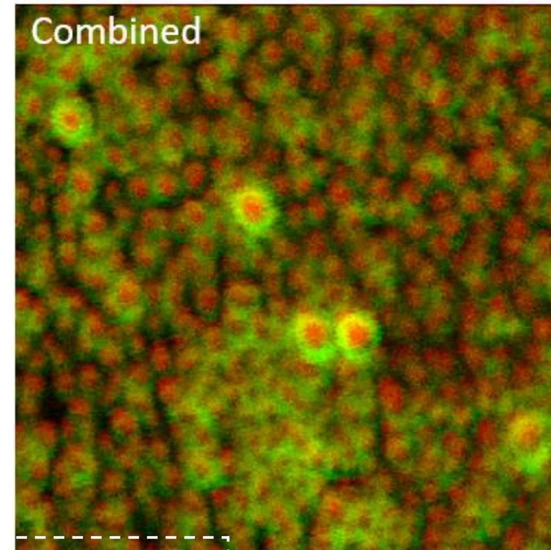
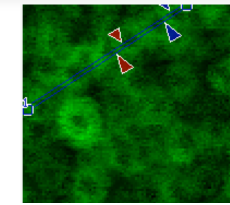
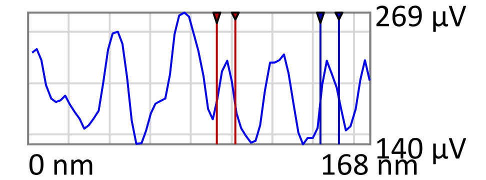
Core-Shell Structure

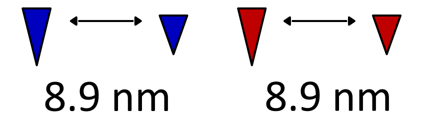


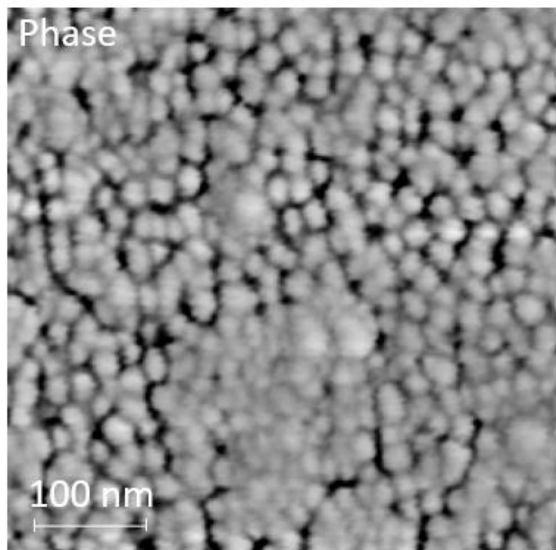


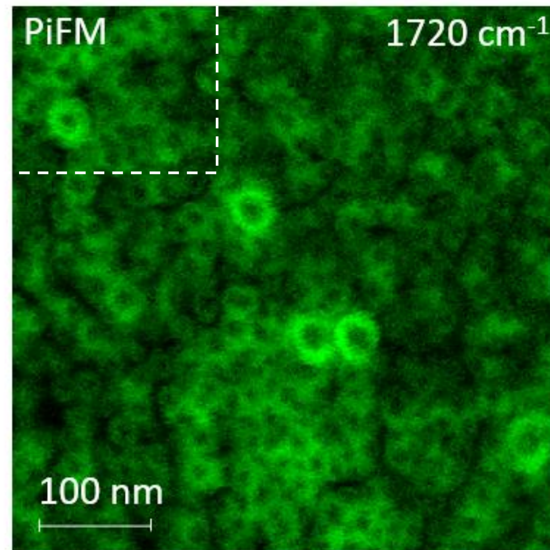


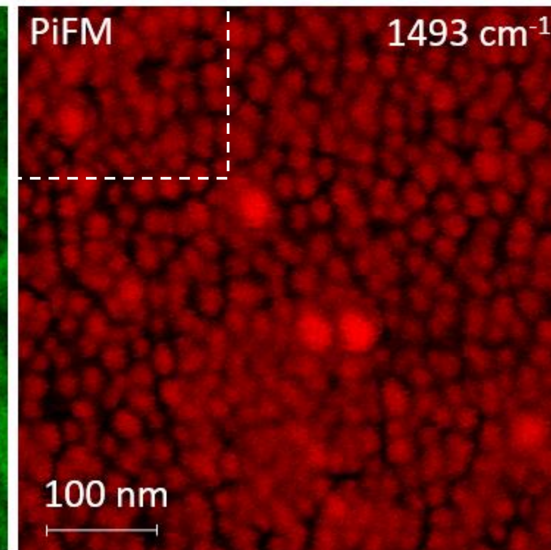


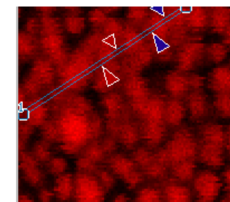


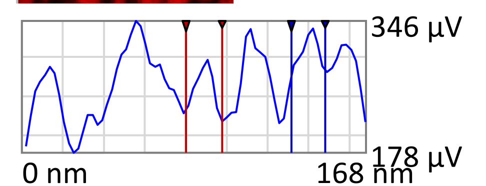


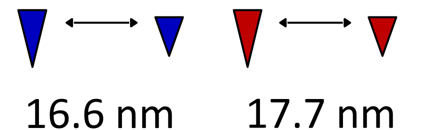












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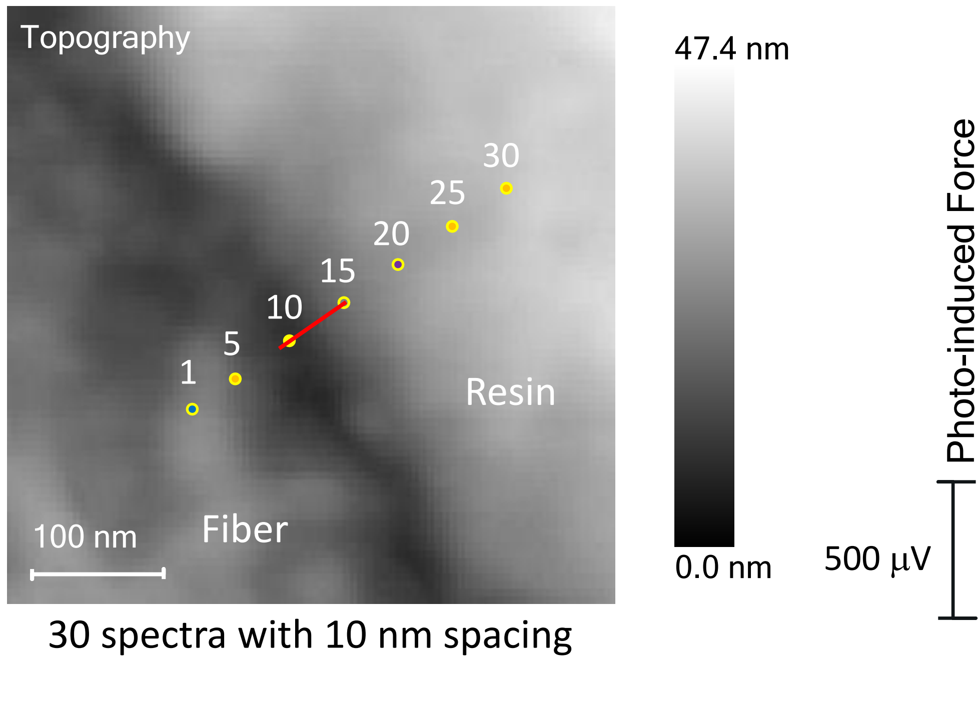
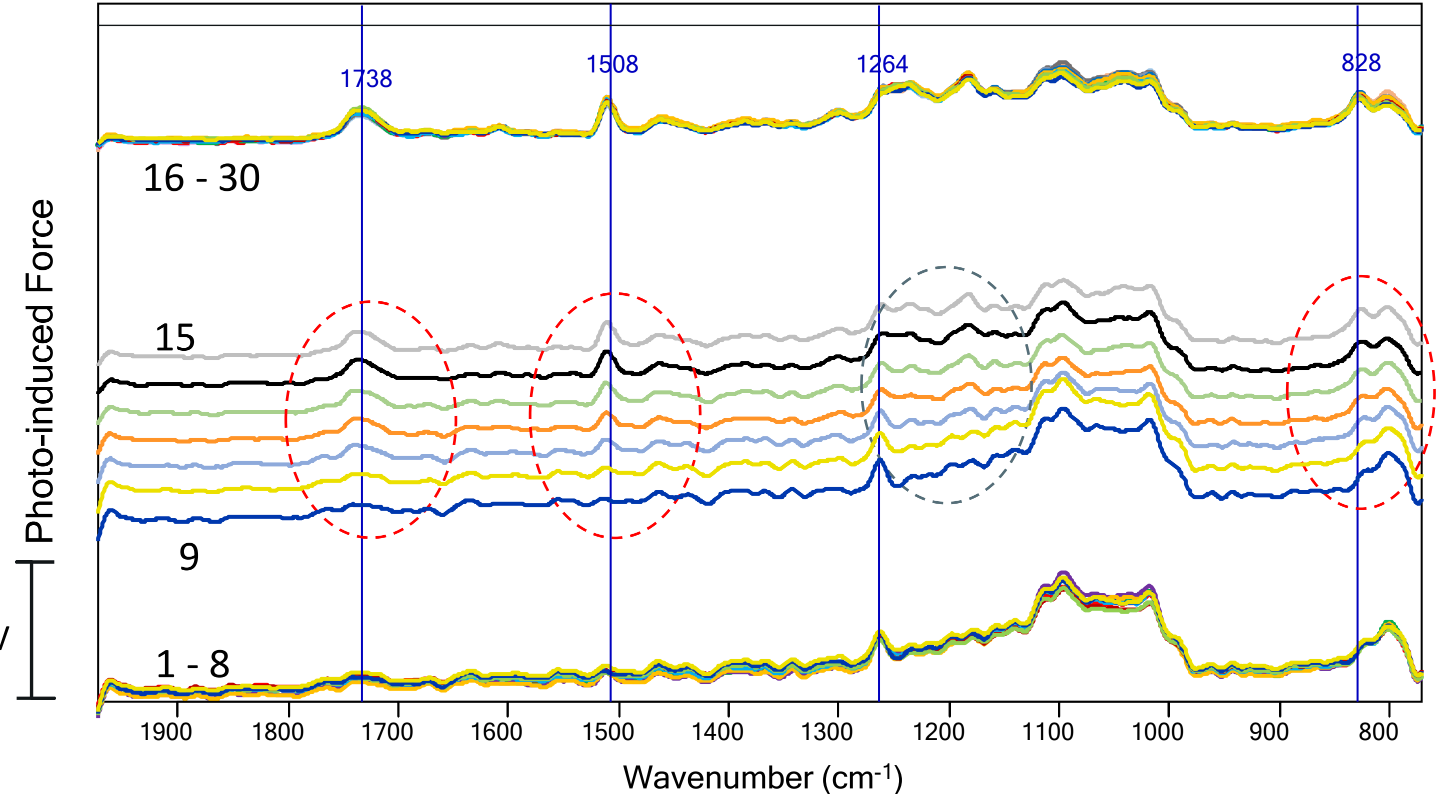
Excellent Repeatability



30 spectra with 10 nm spacing



The interface between a carbon fiber and resin are analyzed by preparing a cross-section of the composite fiber via microtome. 30 PiF-IR spectra are acquired across the interface with ~ 10 nm spacing. The first 8 spectra in the carbon fiber and the last 16 – 30 spectra are very repeatable, with all the spectral changes taking place over 9 – 15 spectra. The SNR is good enough to discern chemical changes for each 10 nm changes in spatial location.

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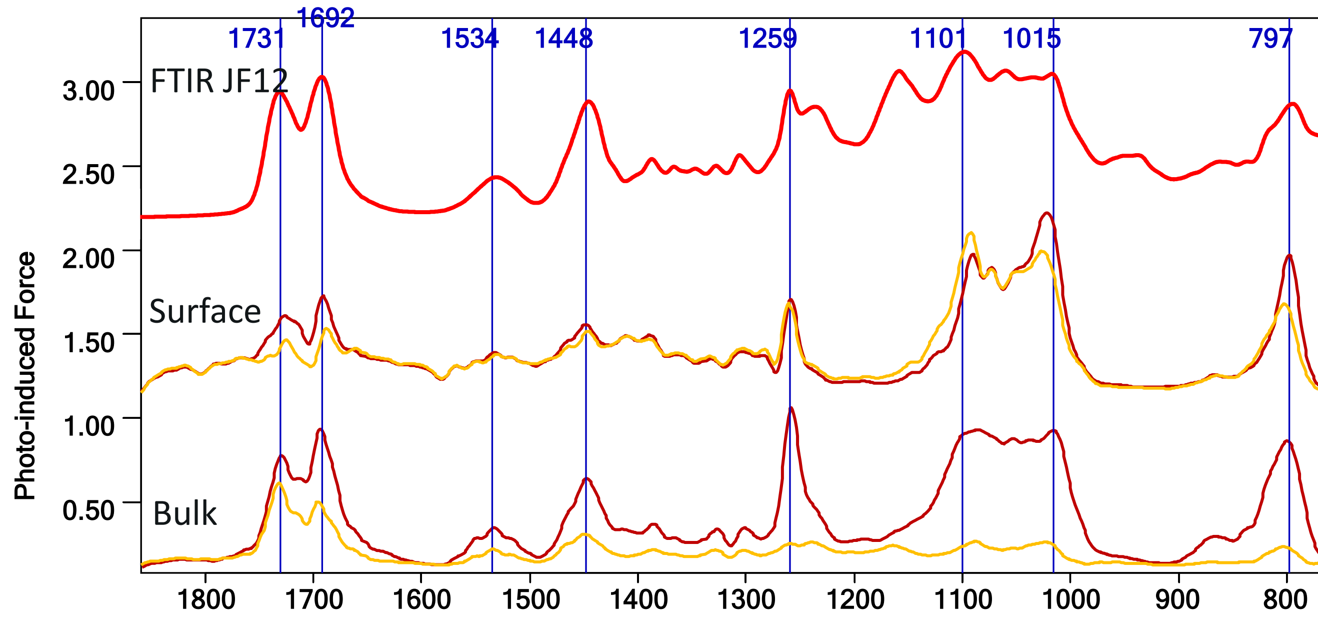
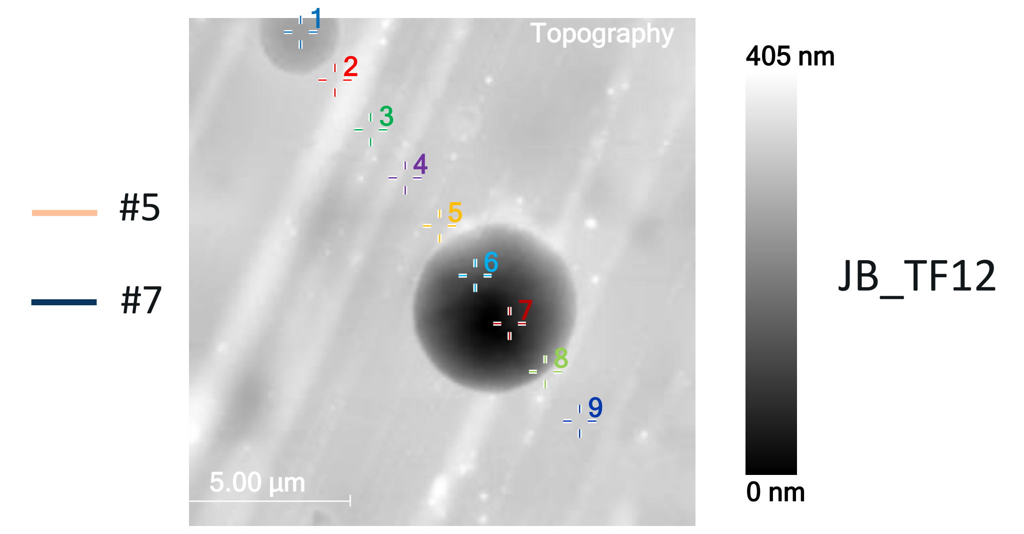
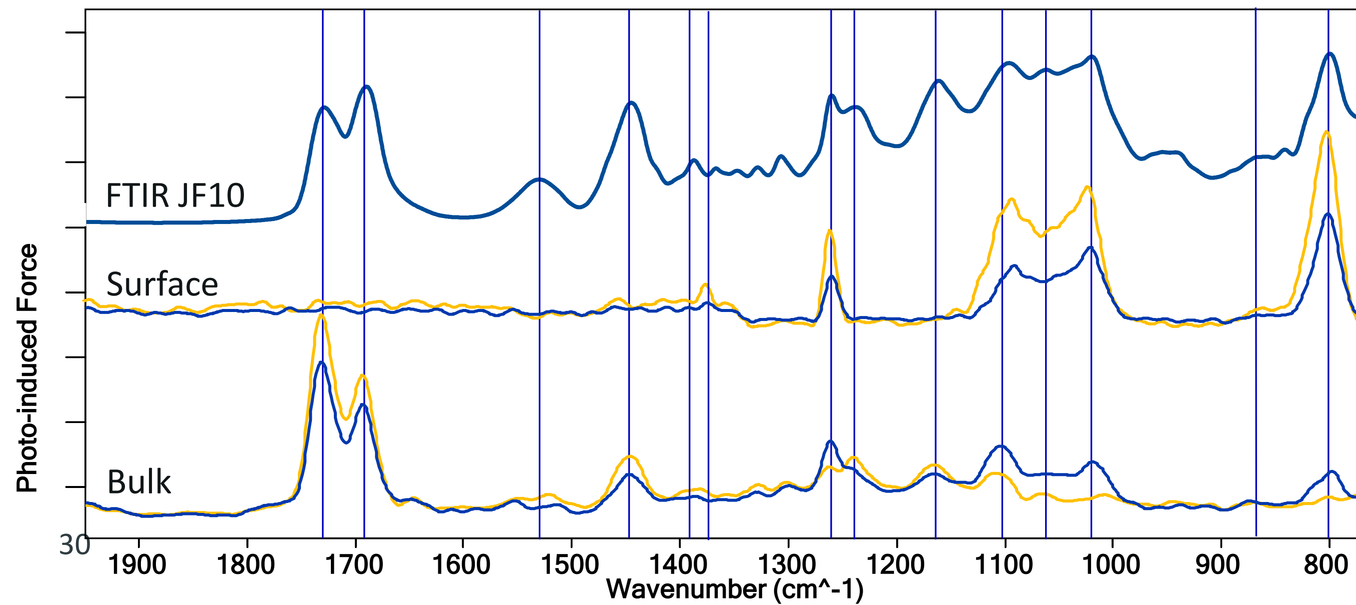
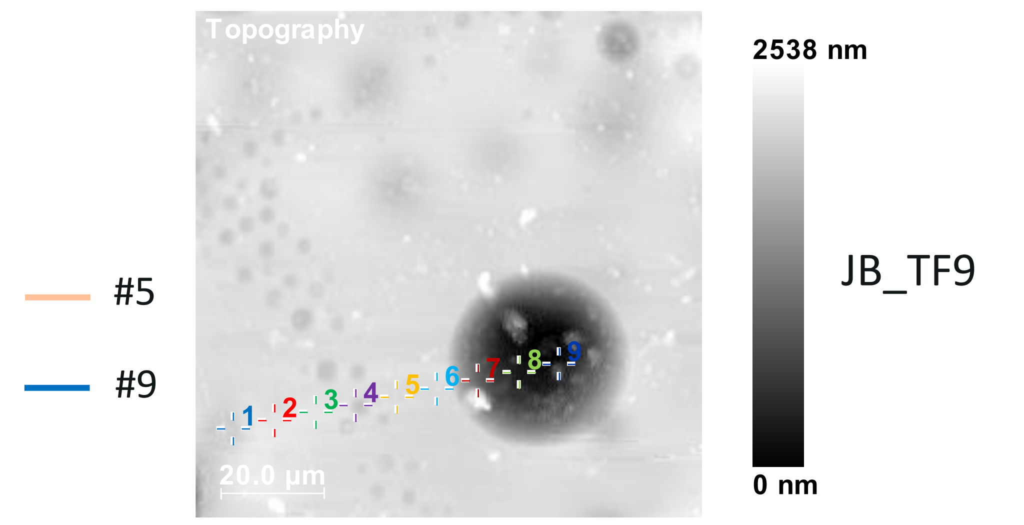
3D Chemical Analysis with Combined Surface and Bulk Modes









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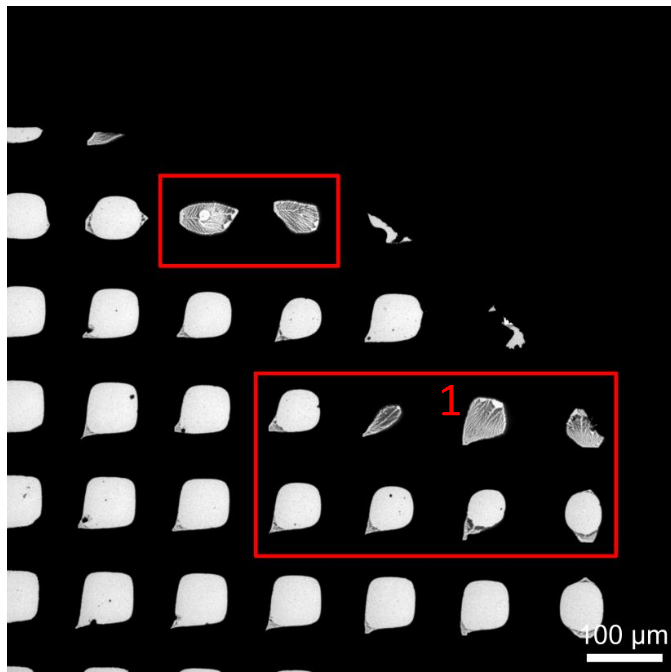
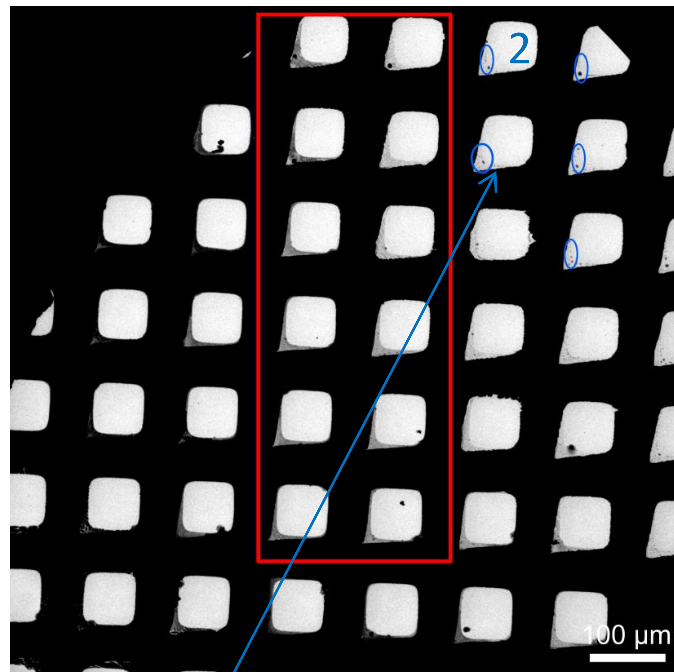
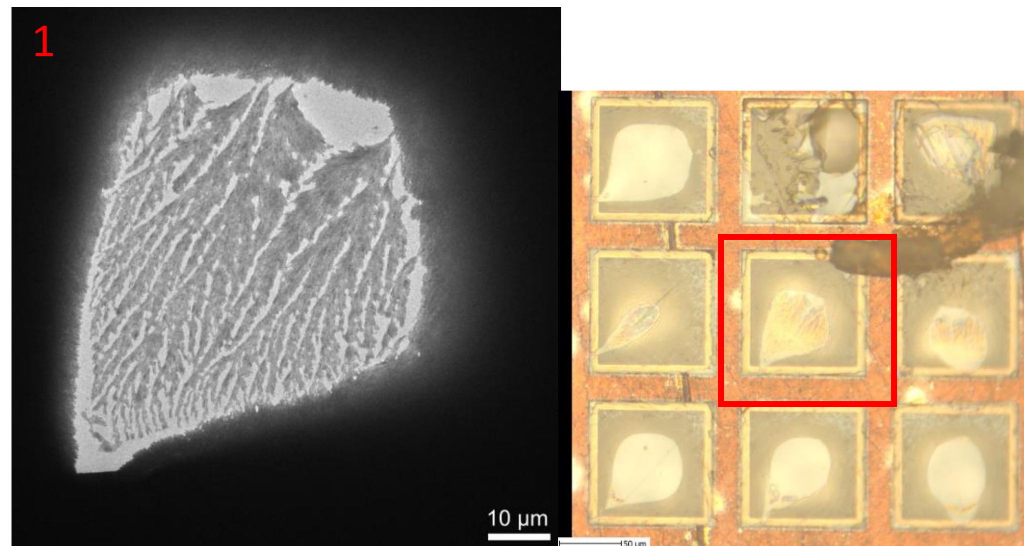
Sample Information

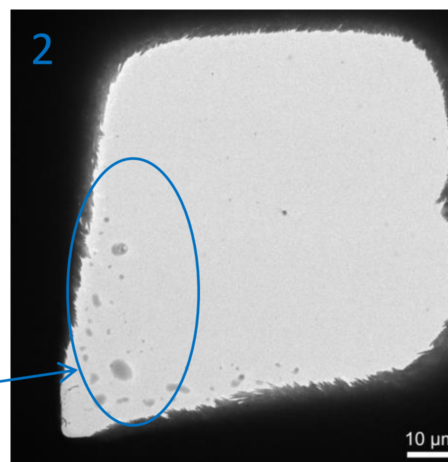
Sample description (Grid 2)

The majority of the grid spaces area are usable, but most of the crystals were recyrstallized only along the edge of the grid spaces. I highlighted some areas that contain more crystals (red boxes).









non-crystalline contamination

The optical image from Molecular Vista Microscope is shown above. The sample we measured is the Grid 2, same area with the No. 1 as the sample information shows.

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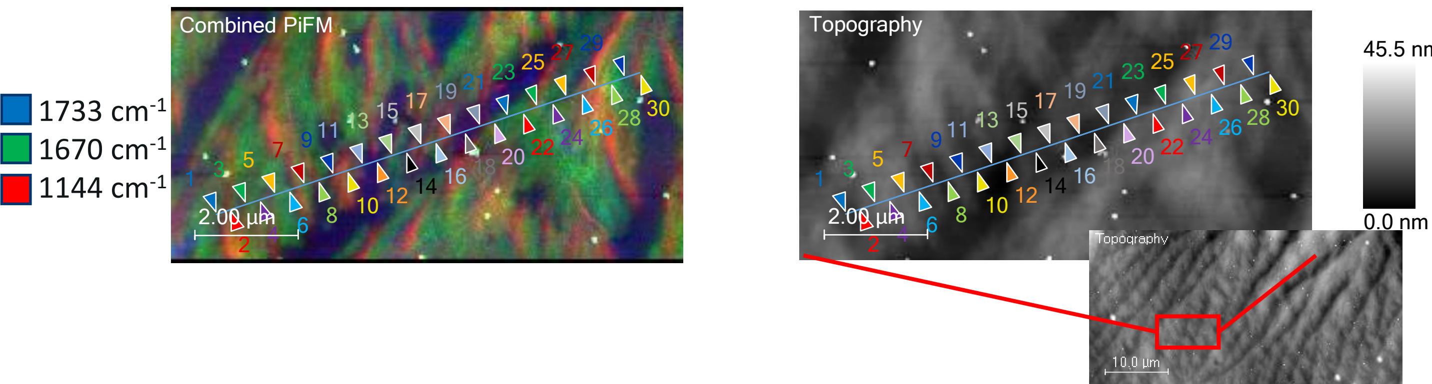
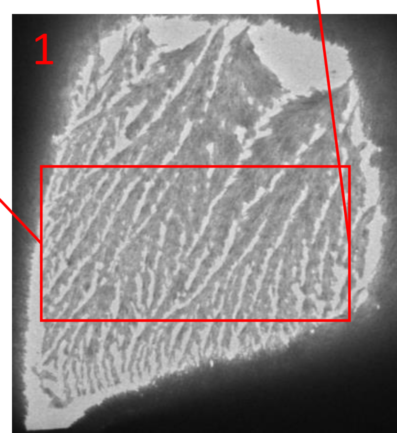
PiF-IR: 10 x 5 μm²

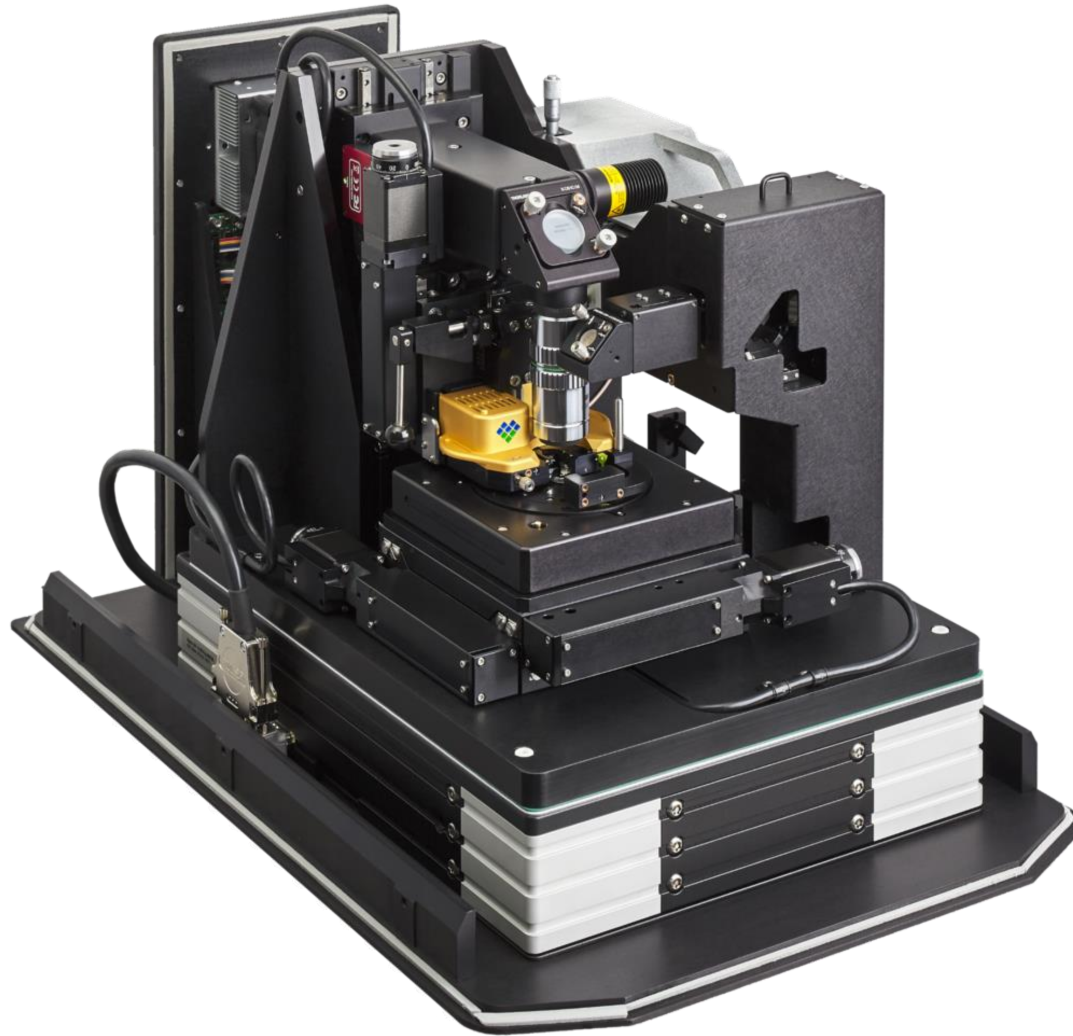




The combined PiFM image shows the crystals are highlighted with two different colors: red and green. And the lower regions are highlighted as blue and purple.

The line spectra with 300nm pitch were taken across the features. The green highlighted regions show peaks around 1751, 1733, 1683, 1673, 1339, 1317, 1252, 1143, 1119, and 1022 cm^{-1}. However, the red highlighted regions show a stronger peak around 1143 cm^{-1} than the green highlighted regions. The blue and purple regions show distinct decreasing of IR signal around 1683 and 1673 cm^{-1}, but a little bit stronger peak around 1733 cm^{-1}.

Nano-scale FTIR & AFM



Vista 75:

Smaller Foot-print for the Same Great Nano-chemical Metrology (for samples up to 100 mm in size)

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Nano-scale FTIR & AFM

Vista 300:

The Most Advanced Nano-Chemical Metrology Instrument
for R&D and Defect Analysis in Nanofabrication
(for samples up to 300 mm wafer)

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Chemical Mapping: SEM/TEM EDX

Atomic Resolution Elemental Mapping on $SrTiO_3$ crystal by Super X EDS (EDX) system on Titan 80-300 Aberration Corrected Scanning Transmission Electron Microscope

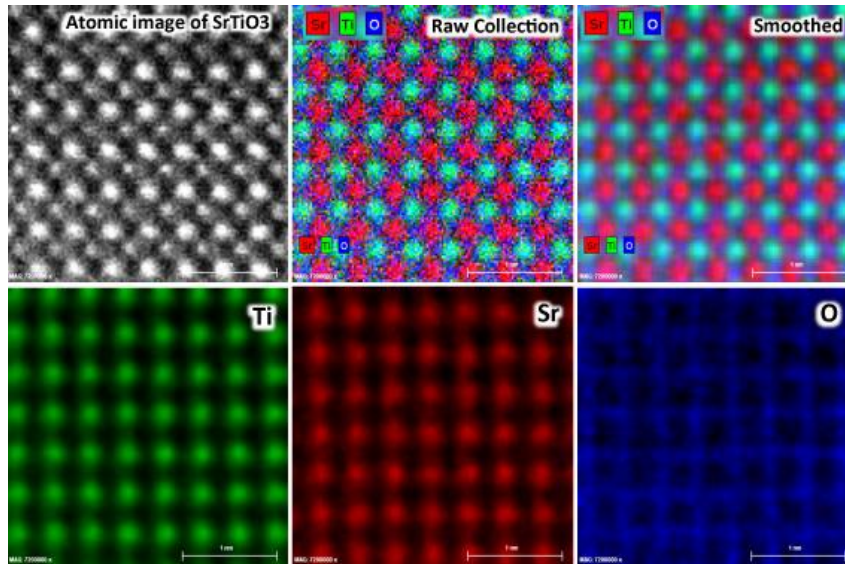

Image credit: North Carolina State Univ. Analytical Instrumentation Facility
https://www.aif.ncsu.edu/tem-lab/

Elemental mapping of a device structure by EDS (EDX)

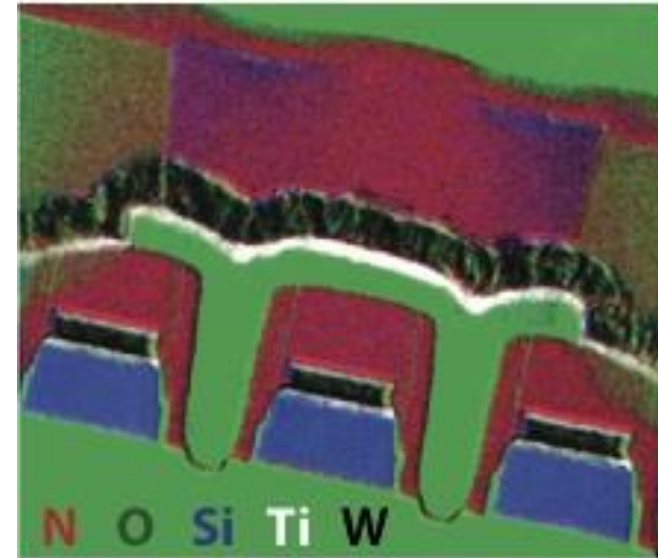

Image credit: Nanolab Techologies
http://www.nanolabtechnologies.com/TEM-STEM-EELS-EDS

- Advanced capability for elemental mapping, but
- No molecular information
- Not effective for organic materials

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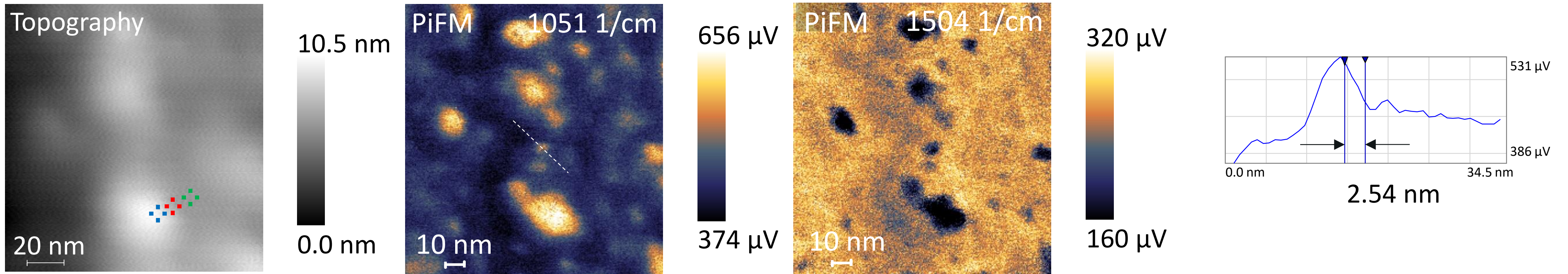
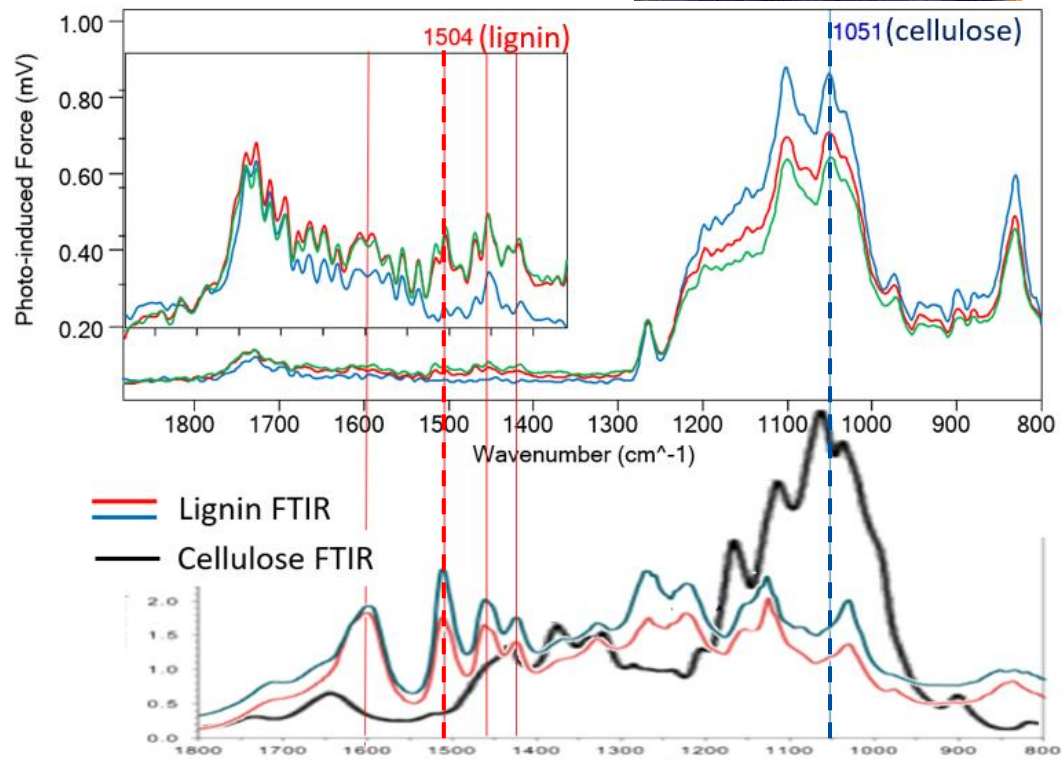
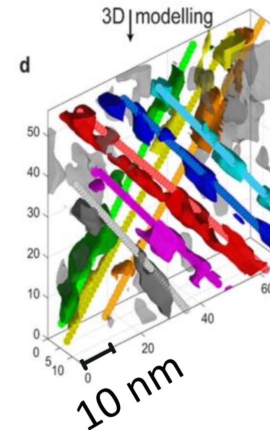
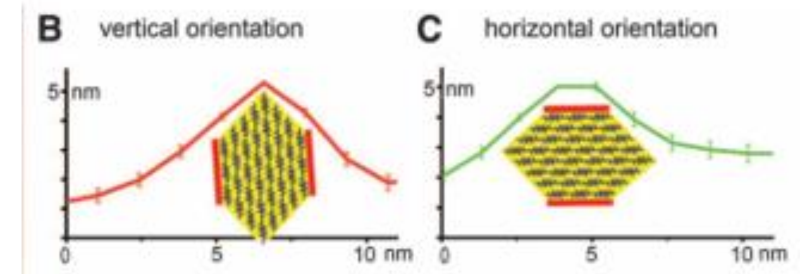
AFM vs IR PiFM of Norway spruce wood cell wall





TEM Model





cellulose elementary fibril

Sub-5 nm spatial resolution!

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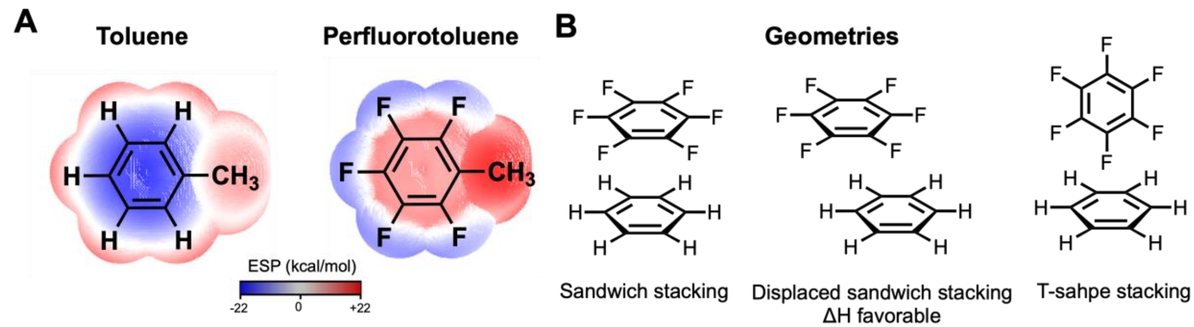
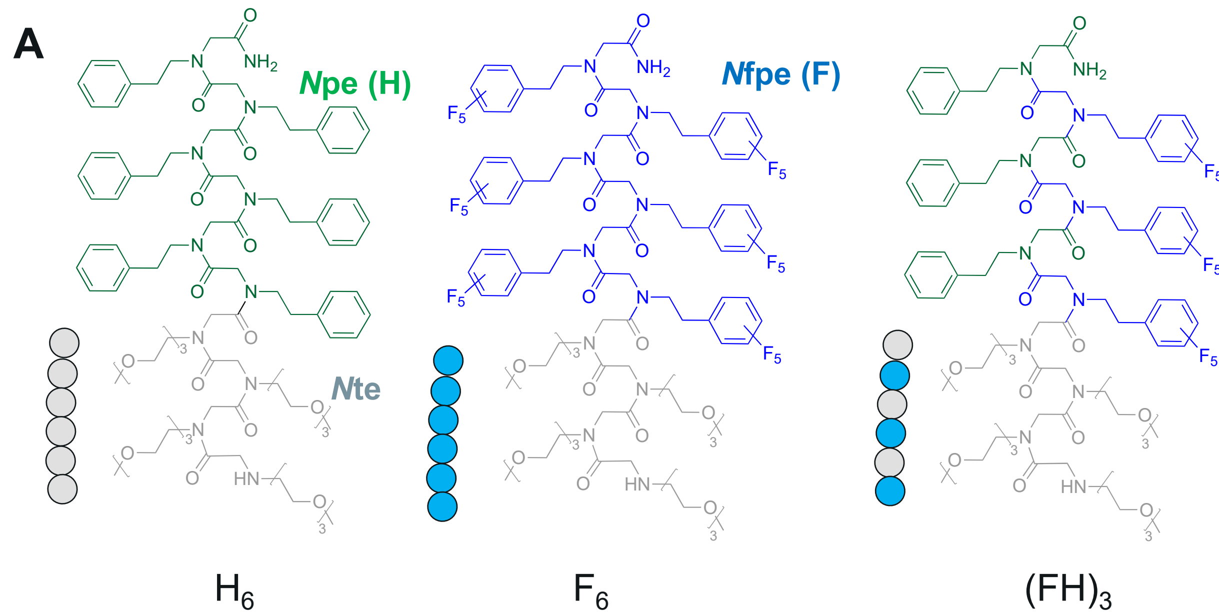
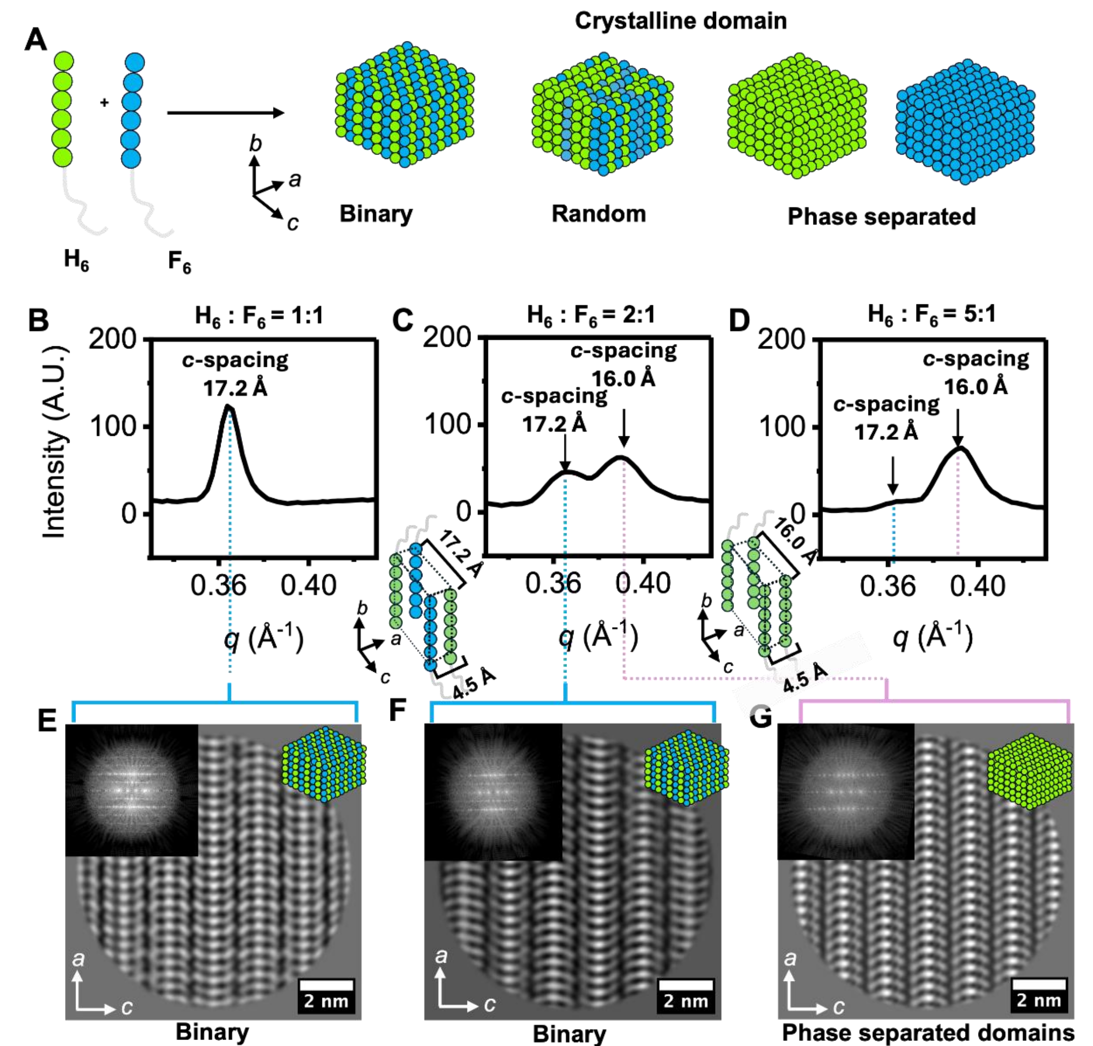
Sub-5 nm Spatial Resolution

Perfluorophenyl-phenyl interactions in nanosheets







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3:1 (Non-substituted:F-substituted derivative)

Topography
1.00 µm
Topography
2.00 µm
6.51 nm
0.00 nm
Topography
100 nm
4.87 nm
0.00 nm
6.22 nm
0.00 nm
Combined PiFM
100 nm
1658 cm⁻¹
1506 cm⁻¹
PiFM
100 nm
1506 1/cm
798 µV
178 µV
741 µV
181 µV
0.0 nm
41.0 nm
Copy
Length (nm) 0.842

Photo-induced Force (mV)
Wavenumber (1/cm)
1691 1664 1507 1461 1378 1218 1211 955 850

Applications Lab

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3:1 (Non-substituted:F-substituted derivative)

F- and non-substituted | **F-substituted only** | **Non-substituted only**

Topography 2.00 µm | 6.22 n – 0.00 n | PiFM 1658 1/cm 2.00 µm | 1.12 m – 0.27 m | PiFM 1506 1/cm 2.00 µm | 1.46 mV – 0.20 mV | 2.00 µm | 2.97 V – 0.51 V

Topography 1.00 µm | 6.51 n – 0.00 n | PiFM 1658 1/cm 1.00 µm | 946 µV – 212 µV | PiFM 1506 1/cm 1.00 µm | 1.03 mV – 0.14 mV | 1.00 µm | 3.04 V – 0.84 V

Topography 100 nm | 4.87 n – 0.00 n | PiFM 1658 1/cm 100 nm | 867 µV – 226 µV | PiFM 1506 1/cm 100 nm | 827 µV – 153 µV | 100 nm | 4.12 V – 0.87 V

Topography 500 nm | 7.17 n – 0.00 n | PiFM 1658 1/cm 500 nm | 693 µV – 195 µV | PiFM 1506 1/cm 500 nm | 886 µV – 192 µV | 500 nm | 2.75 V – 0.99 V

Topography 50.0 nm | 6.99 n – 0.00 n | PiFM 1658 1/cm 50.0 nm | 821 µV – 223 µV | PiFM 1506 1/cm 50.0 nm | 935 µV – 166 µV | 50.0 nm | 3.09 V – 0.91 V

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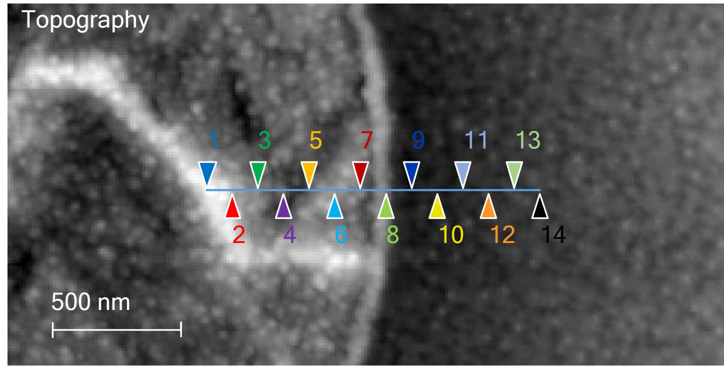
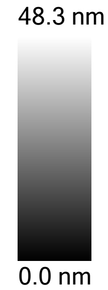
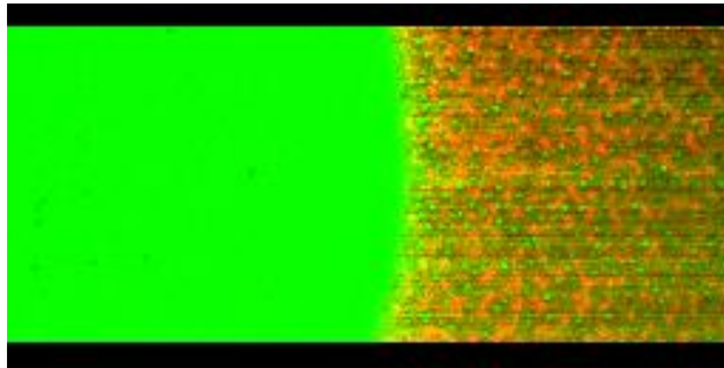
Monitoring Post Glue-clean Wafer

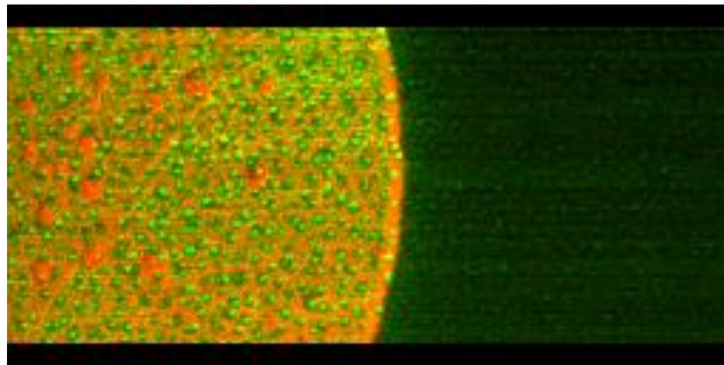
The selection of the wavenumbers for PiFM images are based on PiF-IR spectra acquired on the samples. The triangles show the location of PiF-IR acquired on this sample. The spectra are withheld due to NDA.







Highlights glue residue at WN1 (green) in the oxide region. The display is adjusted to show the oxide region. Due to the enhanced field enhancement on Cu region, it appears much brighter even at WN1.



Highlights glue residue at WN1 (green) in the Cu pad region

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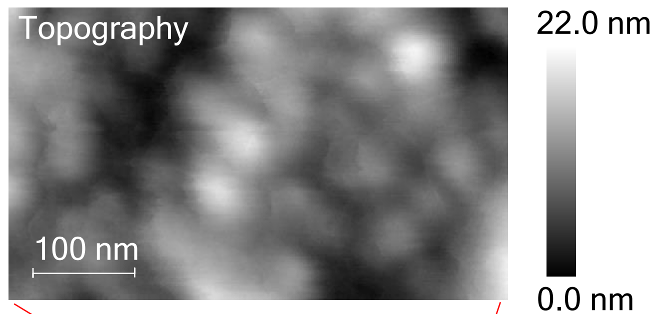
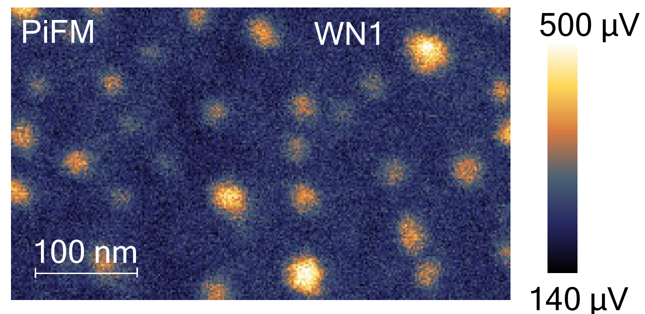
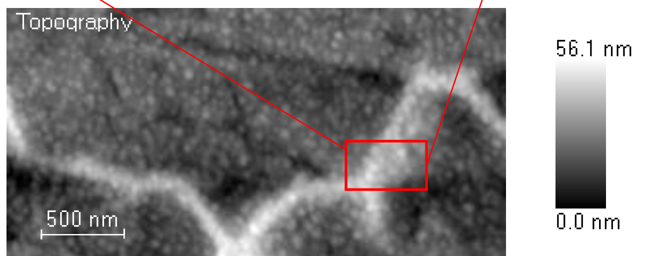
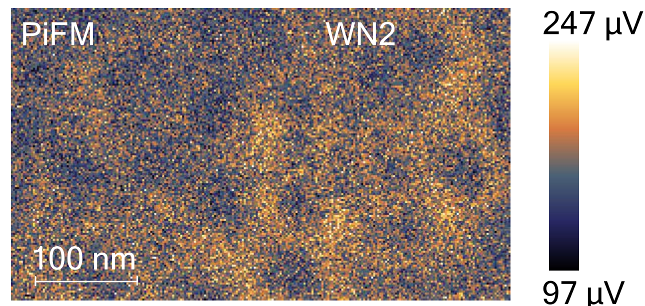
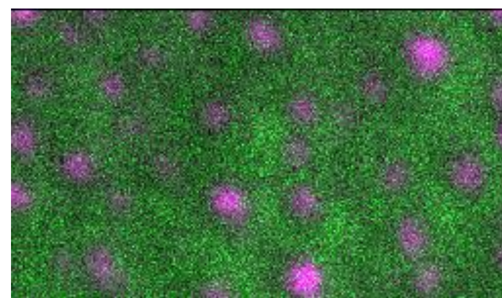
Zoomed-in Cu Pad Region











PiFM image at WN1 highlights the glue that remains even after the cleaning process.

PiFM image at WN2 highlights the Cu1+ based compounds (includes Cu_2O and Cu-organic) on the copper pad.

Combined PiFM images show the glue residue (purple) on top of the copper pad (green).

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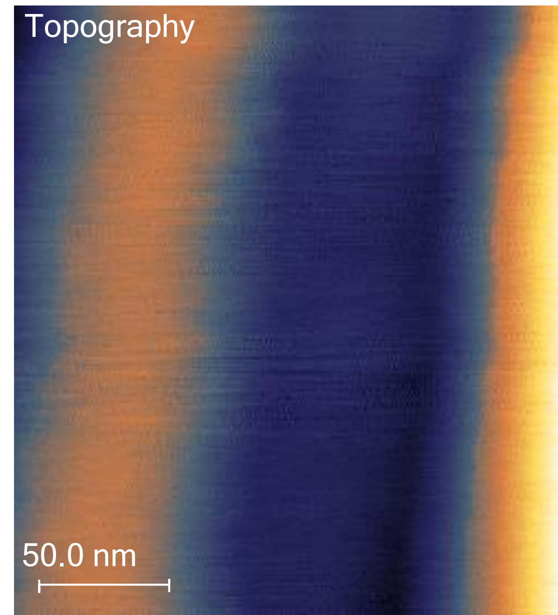
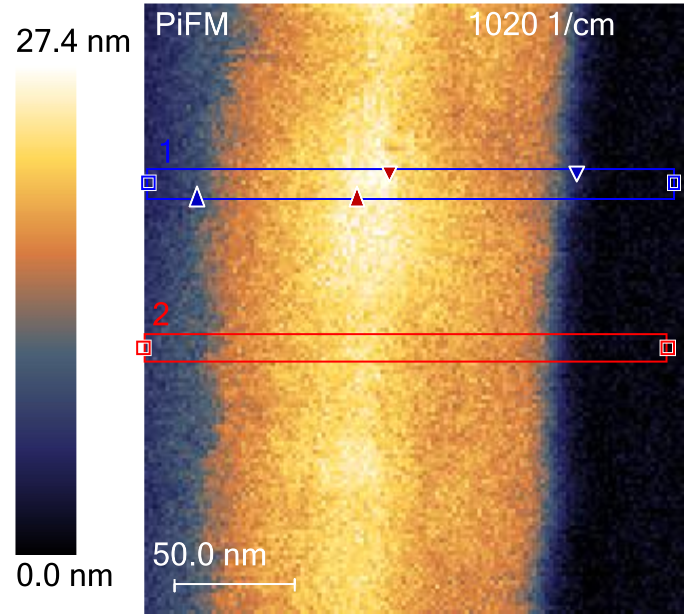
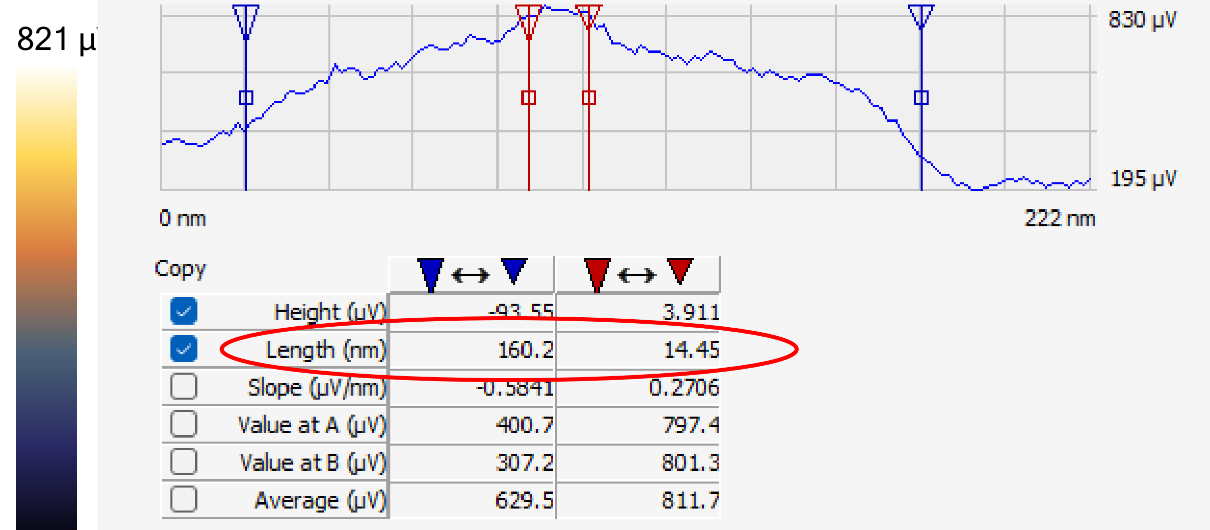
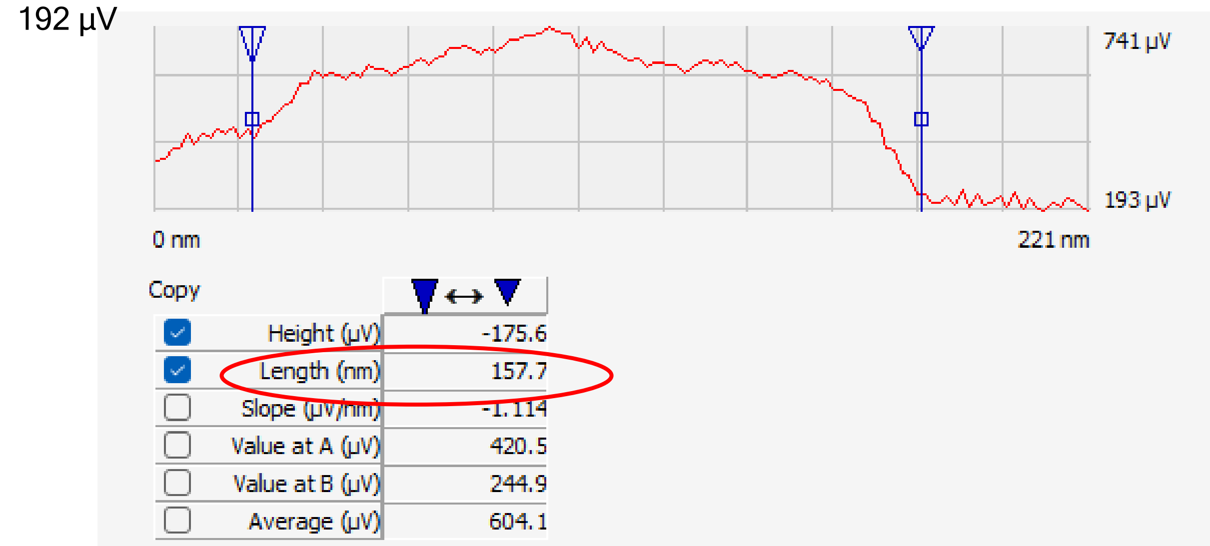
Monitoring Si-O-Si Bonding in Hydrogen Bonded Dielectric Layers



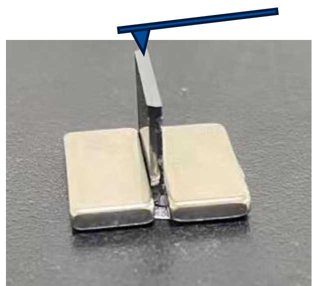


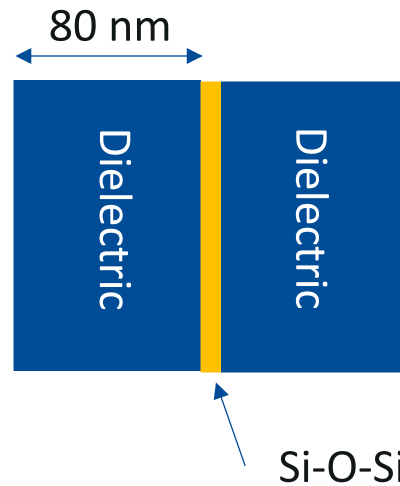


		▼↔▼ (blue)	▼↔▼ (red)
☑	Height (µV)	-93.55	3.911
☑	Length (nm)	160.2	14.45
☐	Slope (µV/nm)	-0.5841	0.2706
☐	Value at A (µV)	400.7	797.4
☐	Value at B (µV)	307.2	801.3
☐	Average (µV)	629.5	811.7



		▼↔▼
☑	Height (µV)	-175.6
☑	Length (nm)	157.7
☐	Slope (µV/nm)	-1.114
☐	Value at A (µV)	420.5
☐	Value at B (µV)	244.9
☐	Average (µV)	604.1





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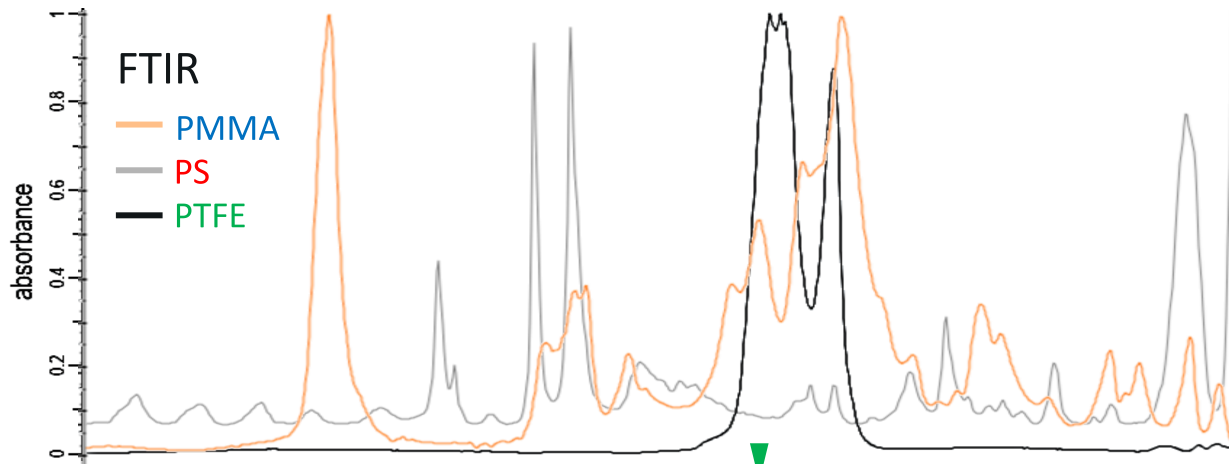
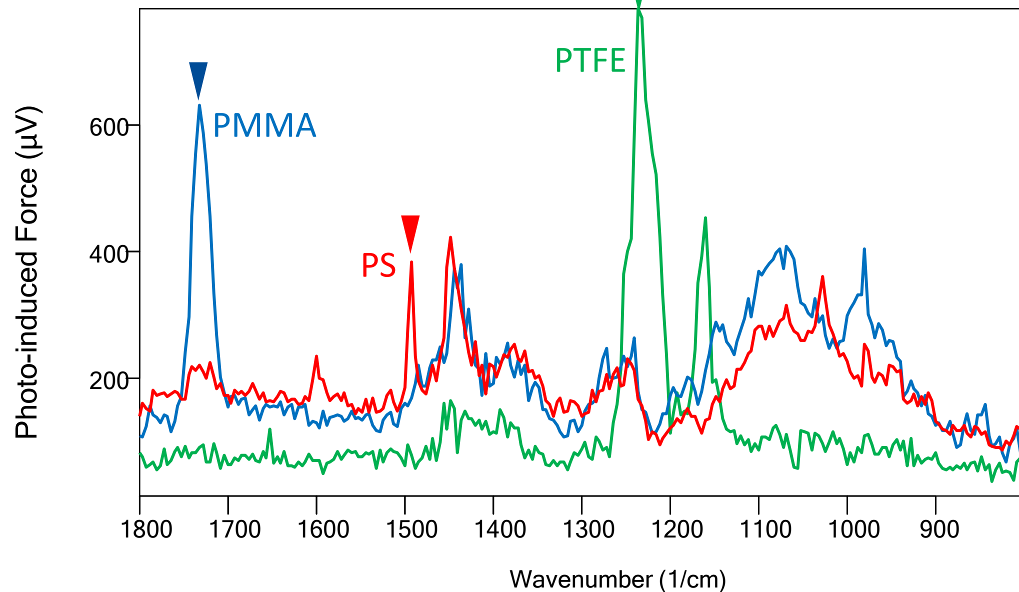
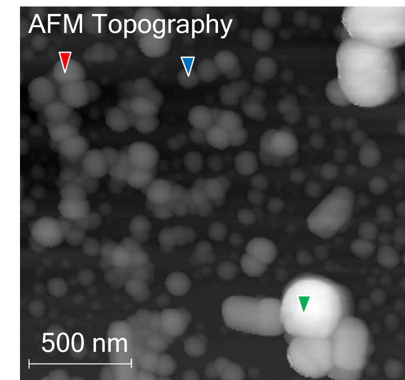
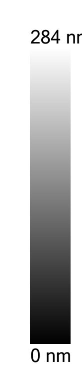
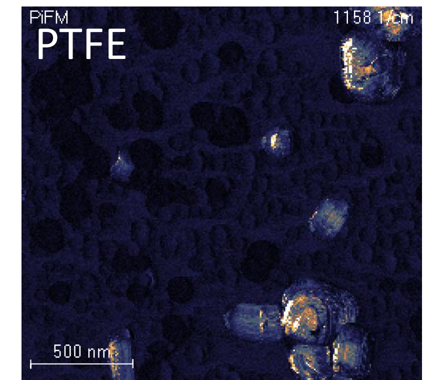
Chemical Analysis of Sub-100 nm Particles

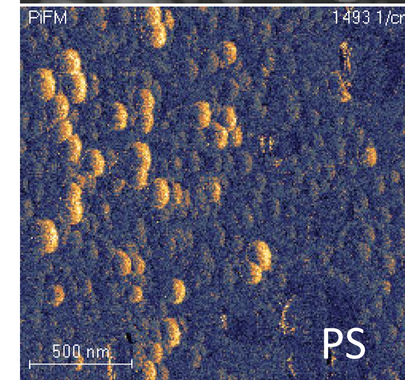


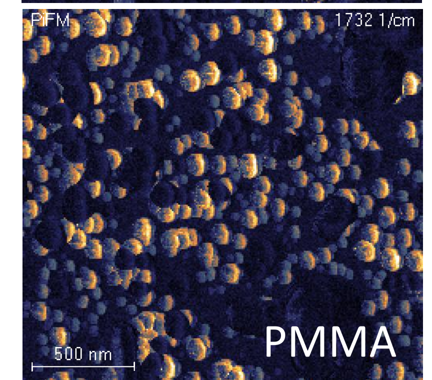


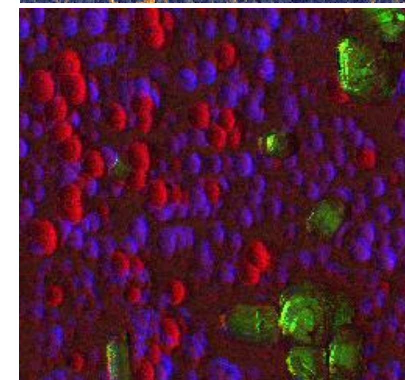












PMMA (1732 cm^{-1}) ~ 40 nm
PS (1493 cm^{-1}) ~ 70 nm
PTFE (1158 cm^{-1}) ~ 190 nm

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Characterization of -OH Bonds on Clean SiO_2 Surfaces

Applications Lab

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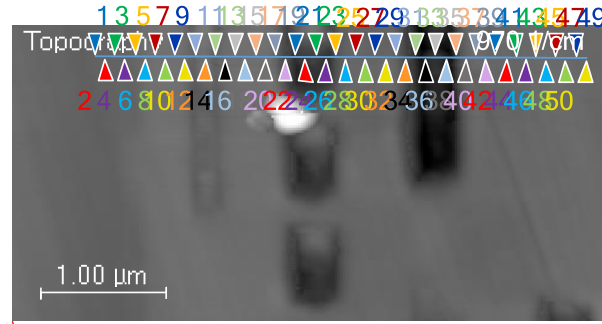
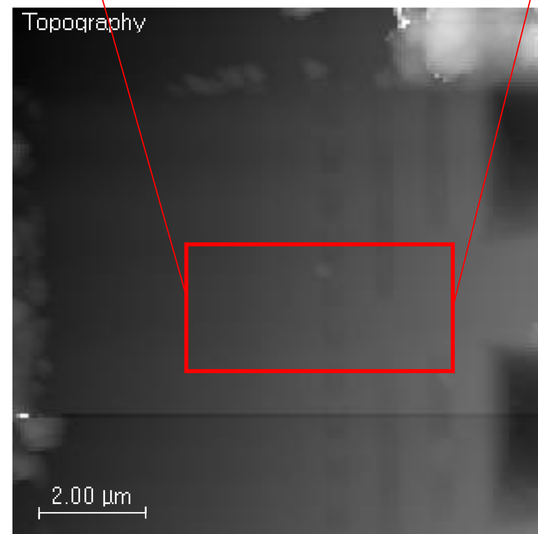
Cross-section Analysis

- The PDMS contamination (1262, 1108, 1021 and 803 cm^{-1}) and hydrocarbon contaminants (1462 cm^{-1}) were found across the sample surface. The PDMS might be from the container of the sample.
- The Si between the metal lines show strong peak around 1116 cm^{-1}.
- There are some variations on spectra around 1200, 1072, and 970 cm^{-1}.



92.5 nm

0.0 nm



1211 nm

0 nm

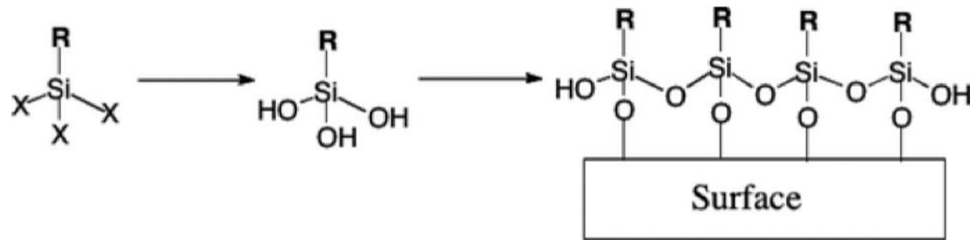
1462 1262 1200 1116 1108 1072 1021 970 803

Photo-induced Force (mV)

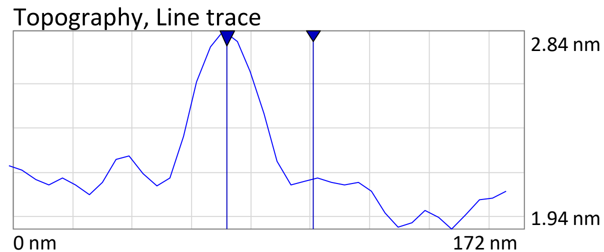
Wavenumber (1/cm)

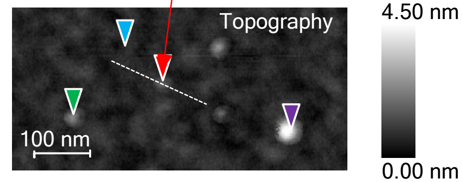
Failed Aminosilane Functionalization

Ideal functionalization should result in a monolayer



Only ~ 0.65 nm tall!

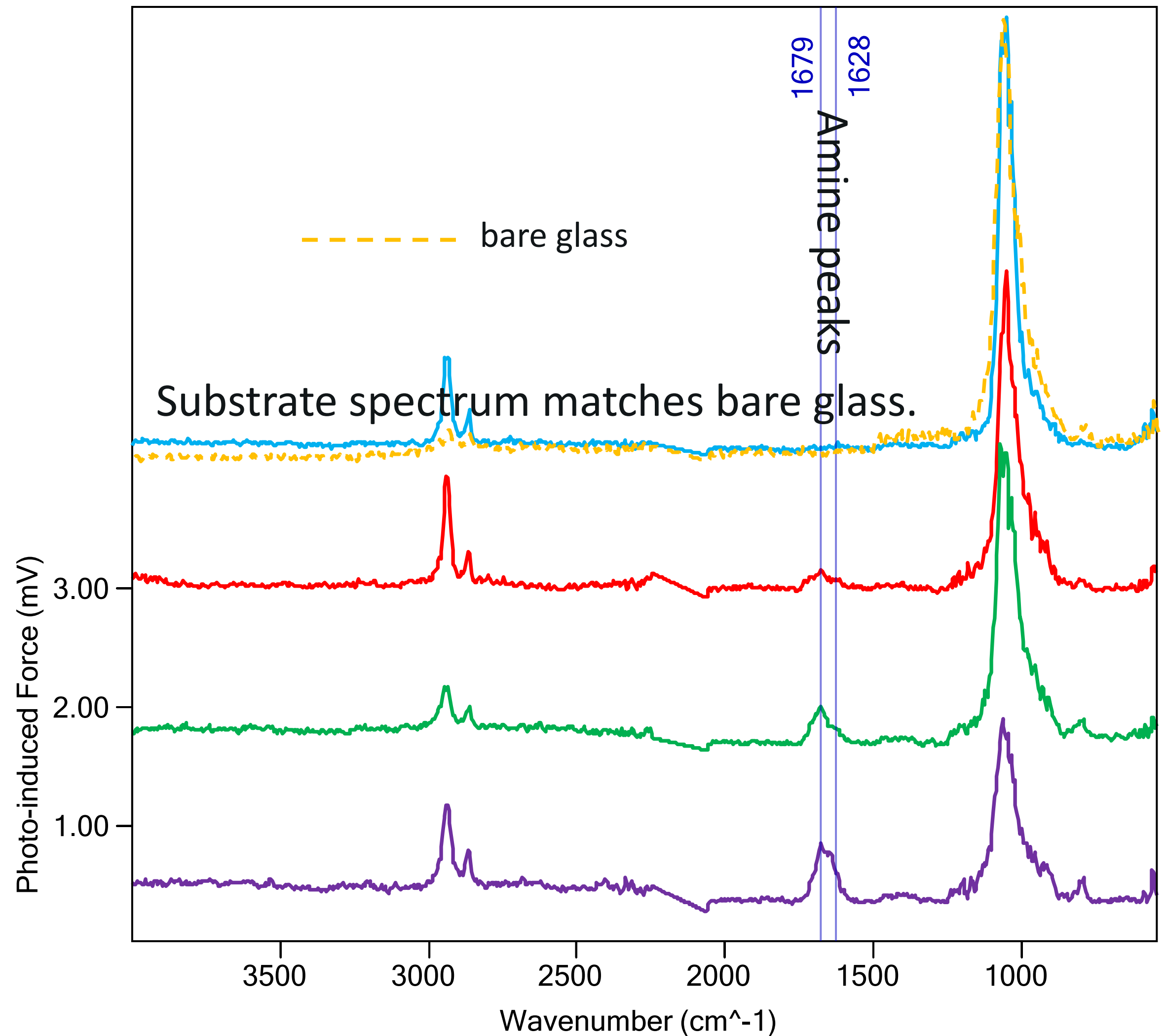




Shows aggregates.

Only aggregates show amine peaks.

Aminosilane molecules are mapped.



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PiFM for Surface Chemistry Analysis

Role of Cu Oxidation in Low T Cu-Cu Hybrid Bonding

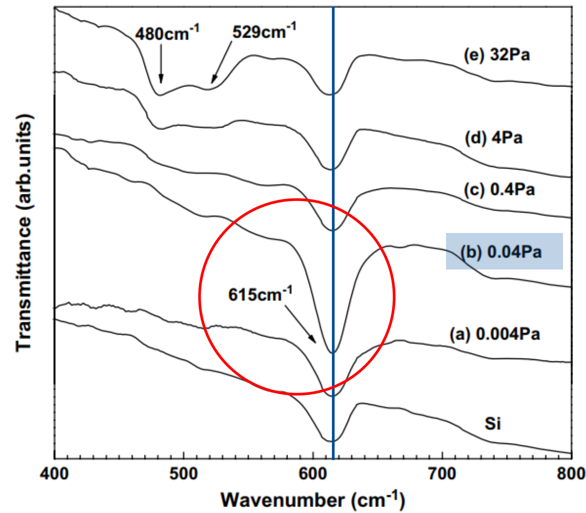

Fig. 3. The FTIR spectra of copper oxide films prepared at 500 °C under oxygen pressure of (a) 0.004 Pa, (b) 0.04 Pa, (c) 0.4 Pa, (d) 4 Pa and (e) 32 Pa.

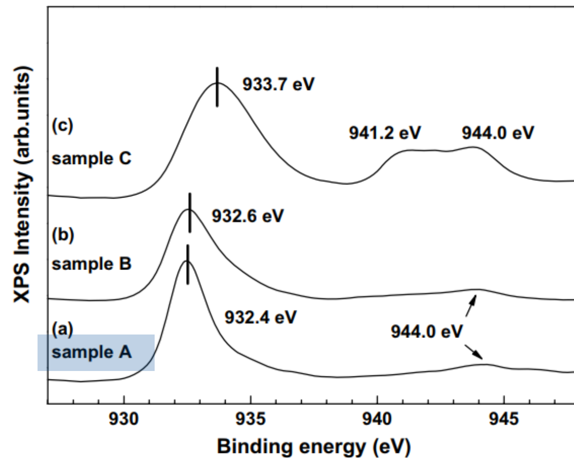

Fig. 4. XPS $Cu2p_{3/2}$ core-level spectra of samples A, B and C.

surface of the films, three typical samples deposited under 0.04 Pa (sample A), 0.4 Pa (sample B), 32 Pa (sample C) were determined by

Impact of Plasma Activation on Copper Surface Layer for Low Temperature Hybrid Bonding

Christopher Netzband, Dylan Burns, Kandabara Tapily, Son Ilseok, Cory Wajda
TEL Technology Center, America, LLC
Albany, NY, United States

Table 1: Diffusion lengths of Cu during a 30min anneal

Species	Diffusion Length of Cu Through Different Species at Varying Temperatures (nm)				
	100°C	150°C	200°C	250°C	300°C
Cu2O	0.17	1.5	8.45	34.16	108.38
CuN	0.0005	0.01	0.15	1.14	6.13
CuO	0.0003	0.006	0.05	0.33	1.48

How to monitor for Cu+1 (Cu_2O) with PiFM?

Use the IR peak at ~ 615 cm^{-1}!

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Mapping Cu Oxide State for Hybrid Bonding

Table 1: Diffusion lengths of Cu during a 30min anneal

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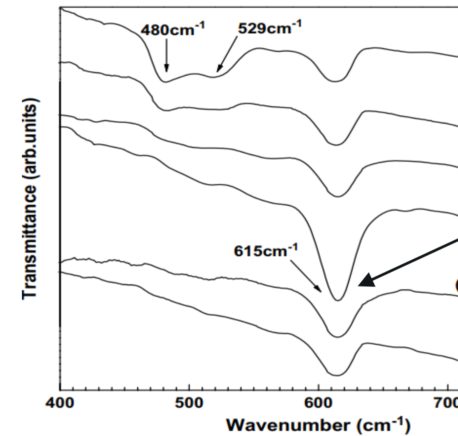
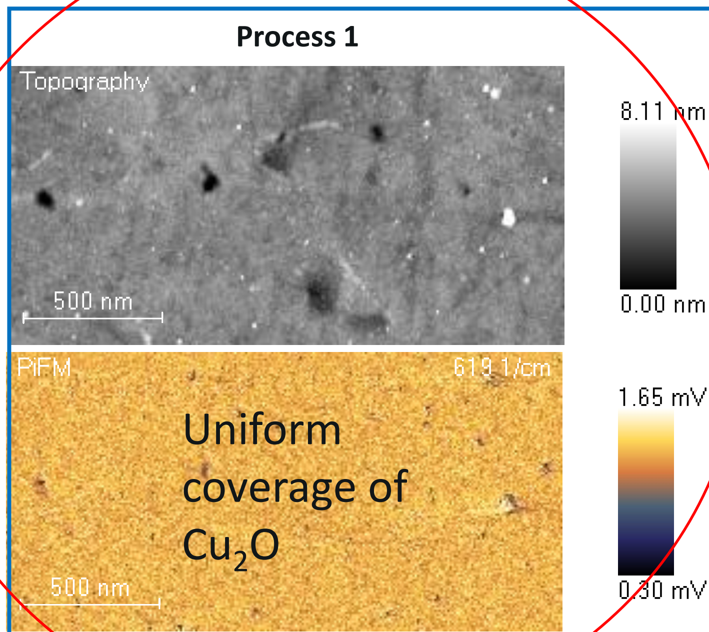
Cu_2O better than CuO for low-T Cu-Cu Hybrid Bonding

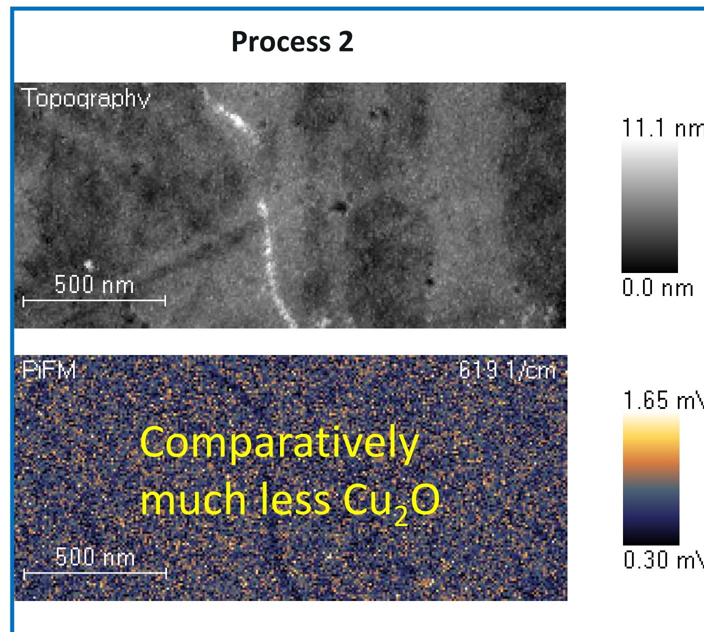
2023 IEEE 73rd Electronic Components and Technology Conference (ECTC), p351

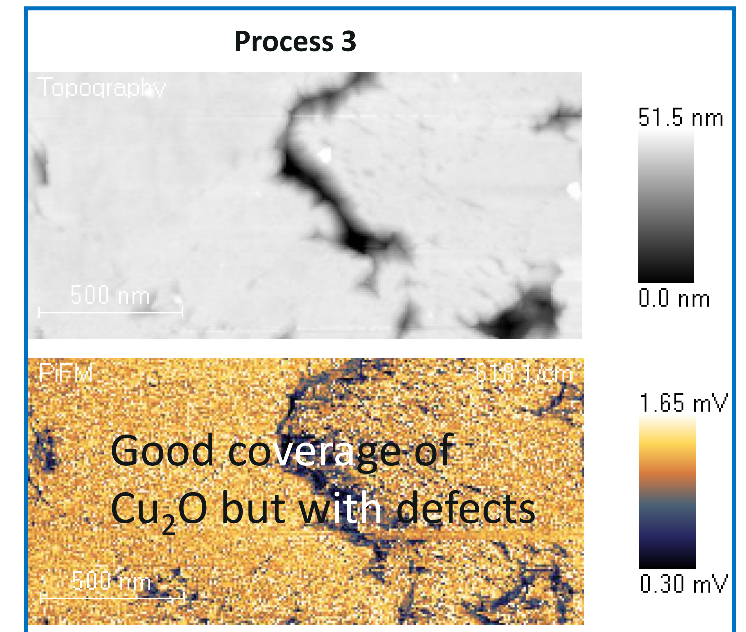


Cu_2O characterized by a peak at ~ 615 cm^{-1}.

Mapping the amount of Cu+1 (Cu_2O) state with PiFM at 619 cm^{-1}.







BEST!

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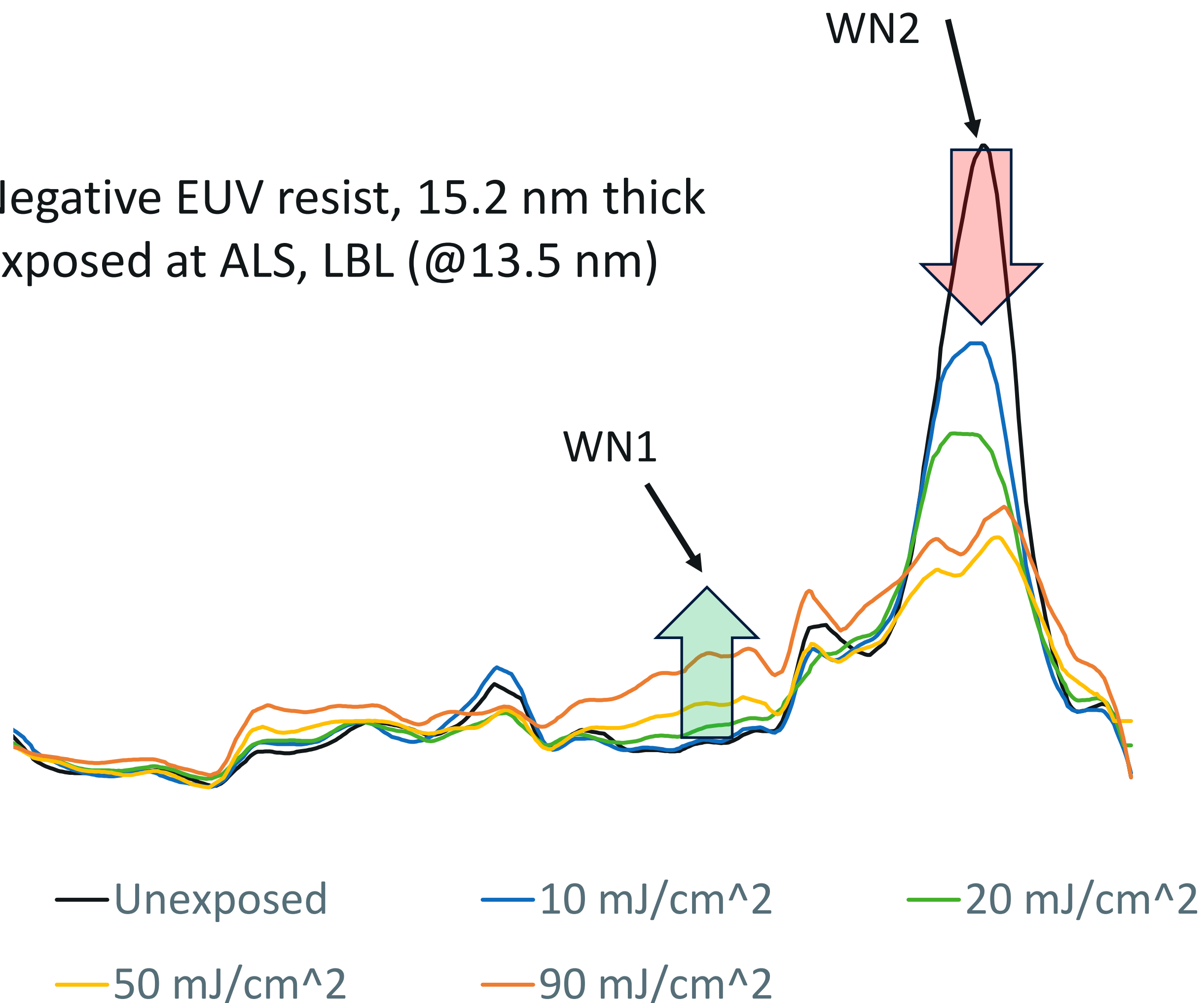
PiFM for EUV Lithography

Changes to IR Peaks upon EUV Exposure

- Negative EUV resist, 15.2 nm thick
- Exposed at ALS, LBL (@13.5 nm)



Nano IR spectra at different dose.

Nano IR signal changes upon EUV exposure.

PiFM images @ WN2 and WN1 will provide latent (undeveloped) images based on *chemical change*.

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Latent Images due to Chemical Changes upon EUV Exposure

- Commercial EUV resist, 15.2 nm thick
- Exposed at ALS, LBL (@13.5 nm)
- 16 nm half-pitch

WN2

WN1

PiFM

Unexposed to EUV

2.06 nm

Topography

0.00 nm

PiFM

Exposed to EUV

50.0 nm

—Unexposed —10 mJ/cm^2 —20 mJ/cm^2
—50 mJ/cm^2 —90 mJ/cm^2

Chemical changes due to EUV exposure imaged without development!

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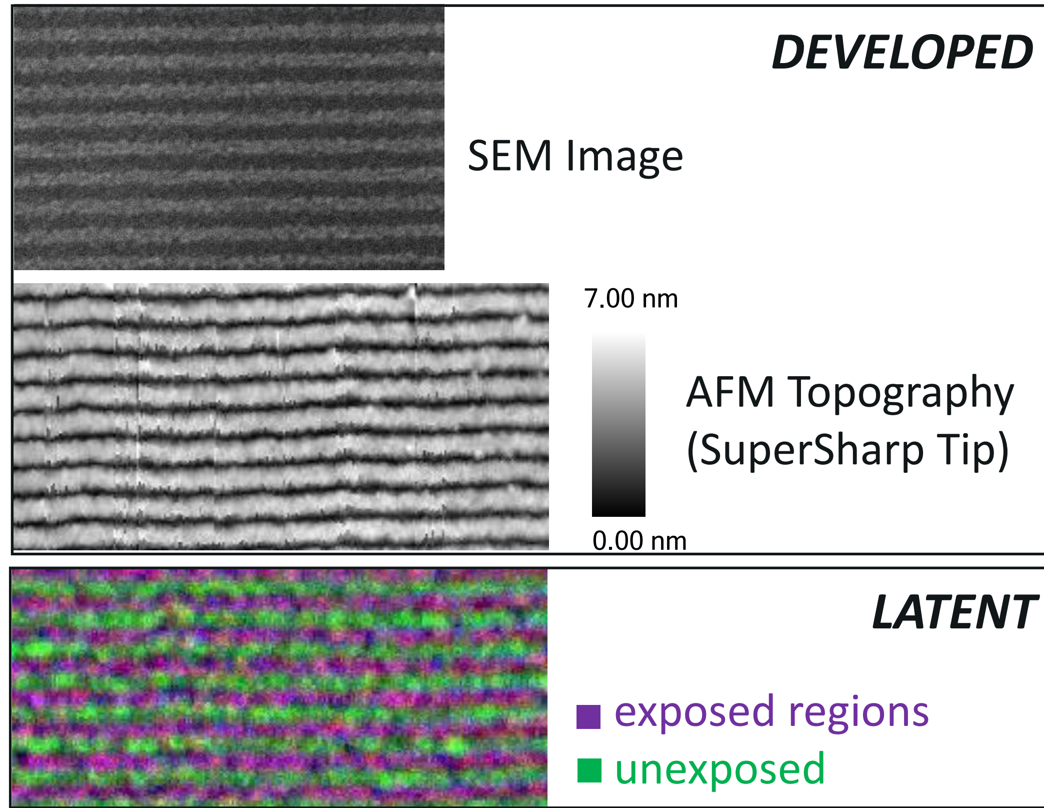
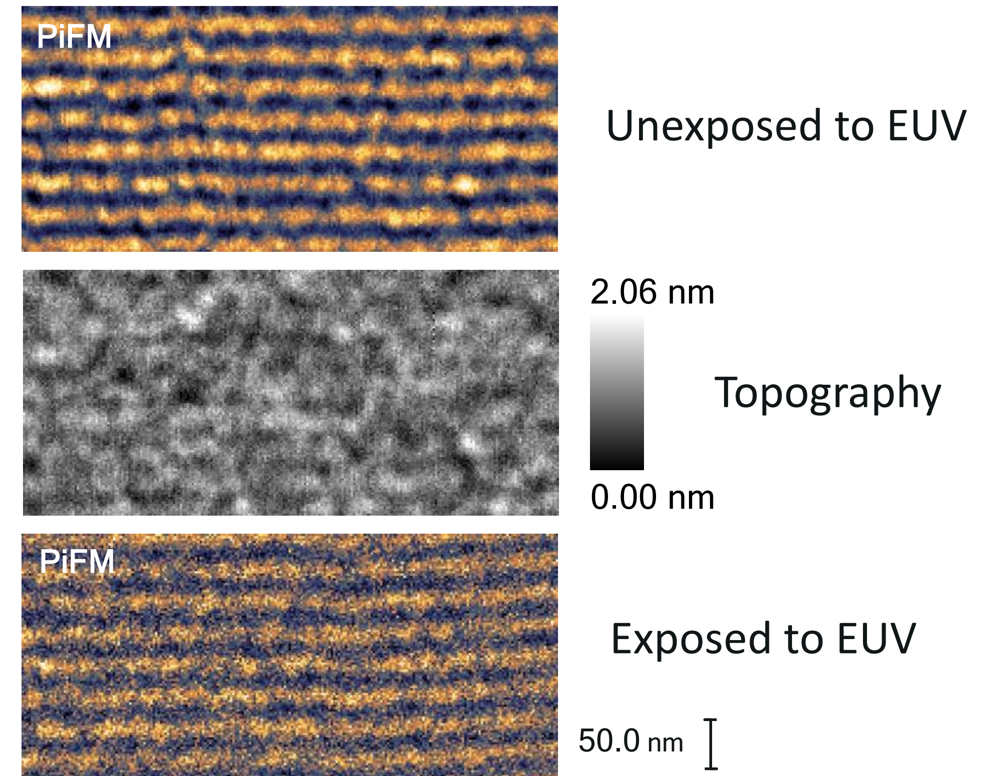
Comparing Latent & Developed Images





Developed pattern compared to the latent image.

Chemical changes due to EUV exposure imaged without development!

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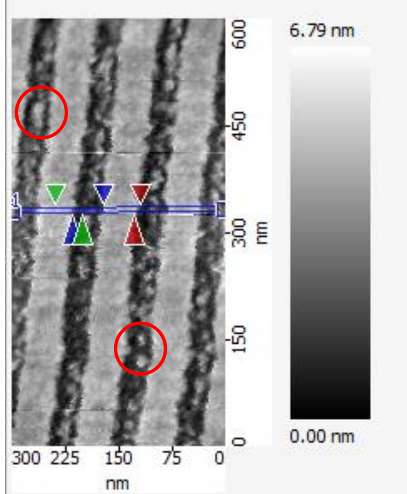
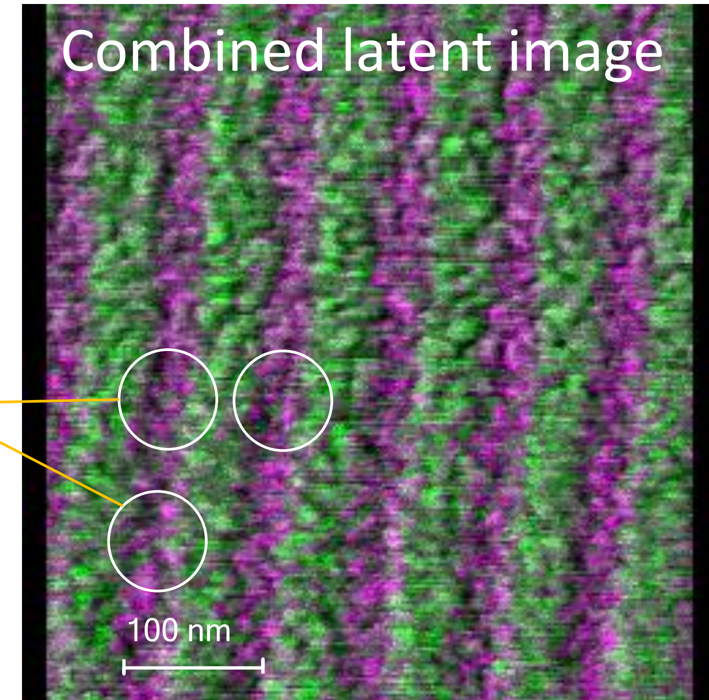
Residues in Developed Pattern Predicted in Latent Images

Developed pattern



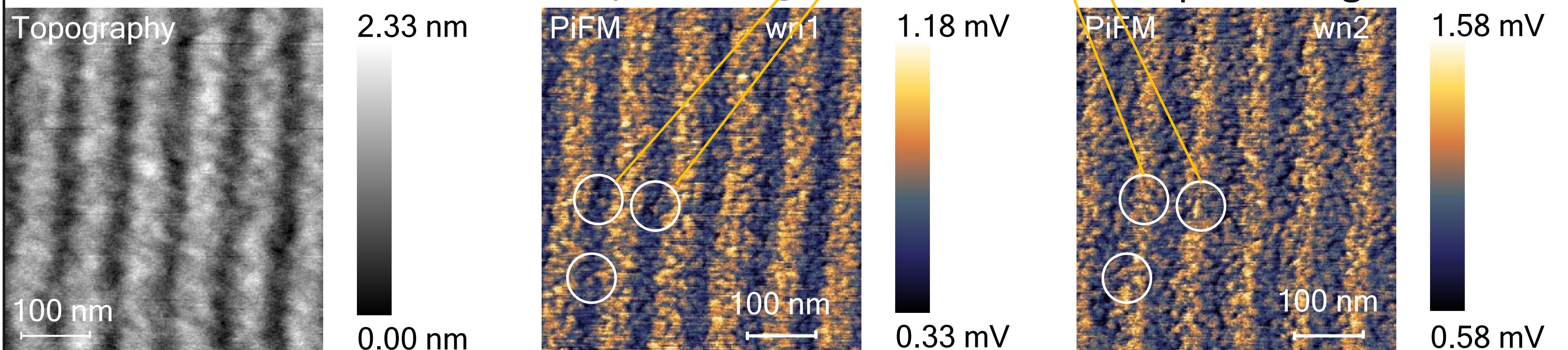
Negative resist → exposed regions remain. Visible residues.

Some unexposed regions look as if chemically exposed → lead to residue?



Exposed Unexposed

Latent Images



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PiFM for EUV Lithography

- Latent imaging – study impact of many parameters on LER and LWR upon EUV exposure without developing but purely based on chemical signature
  - Chemical composition
  - Underlayer
  - Thickness
  - Dose
  - Post exposure processing

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