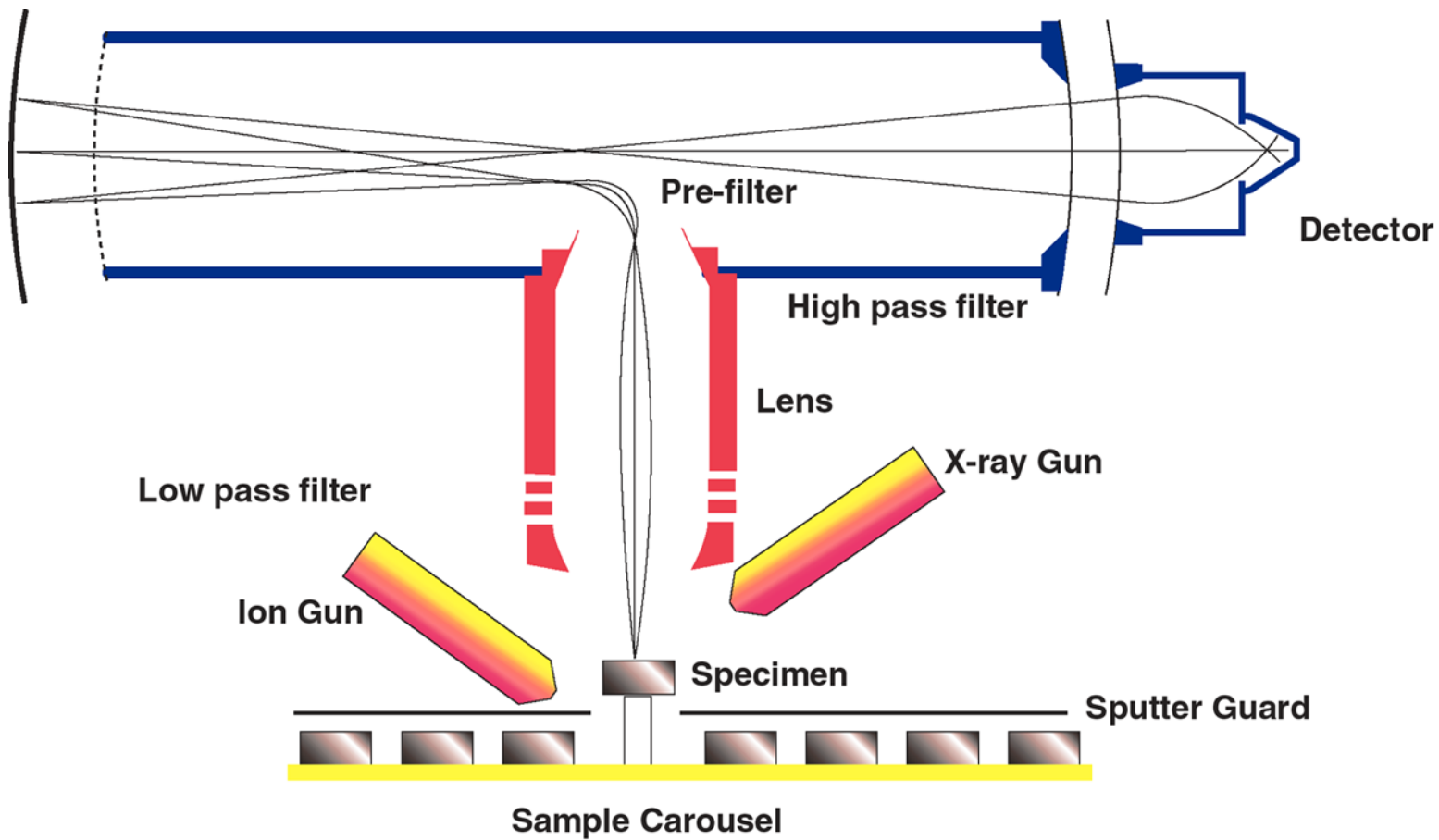


# AMICUS Sample and Analyser Geometry



# Microscopic Examination

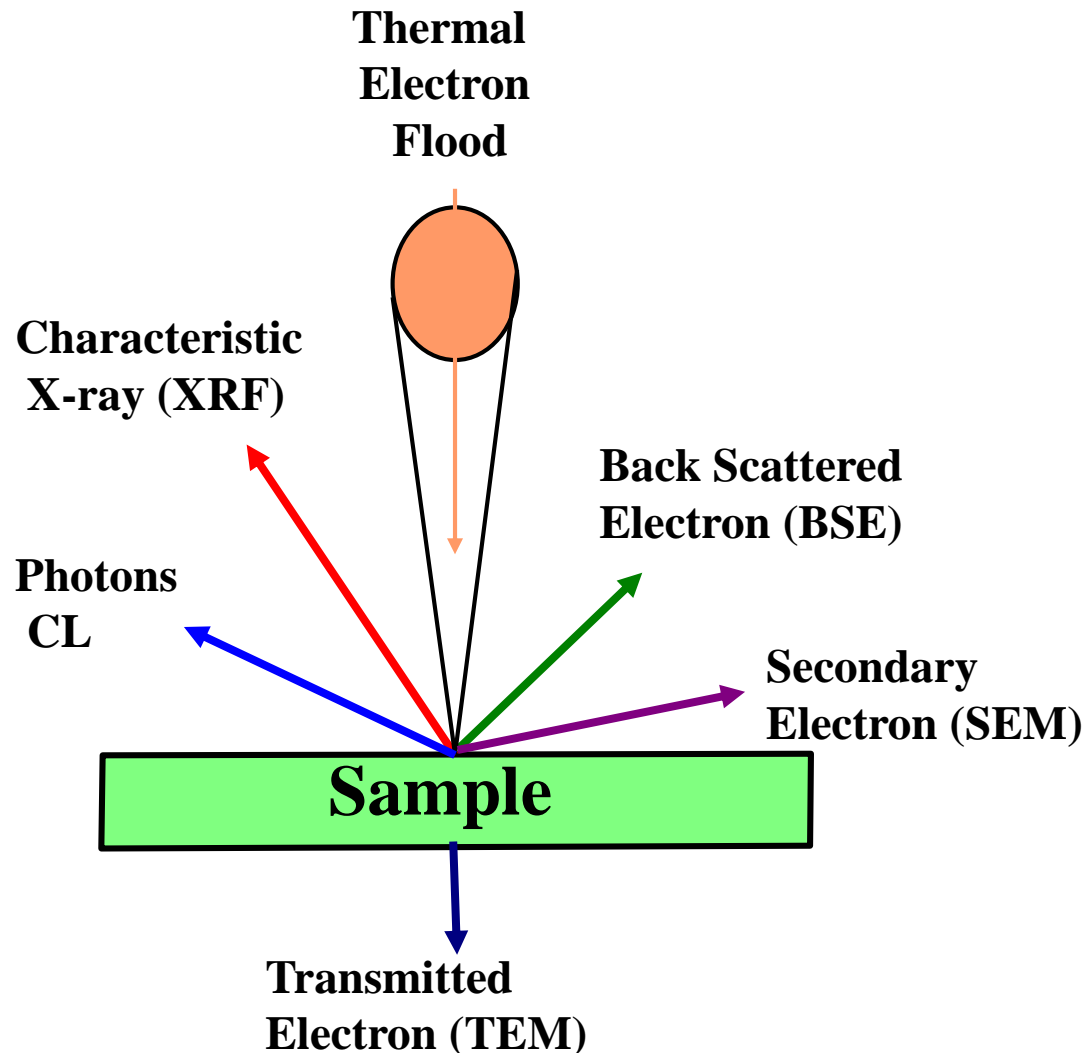
---

## EPMA

Electron Probe Microscope

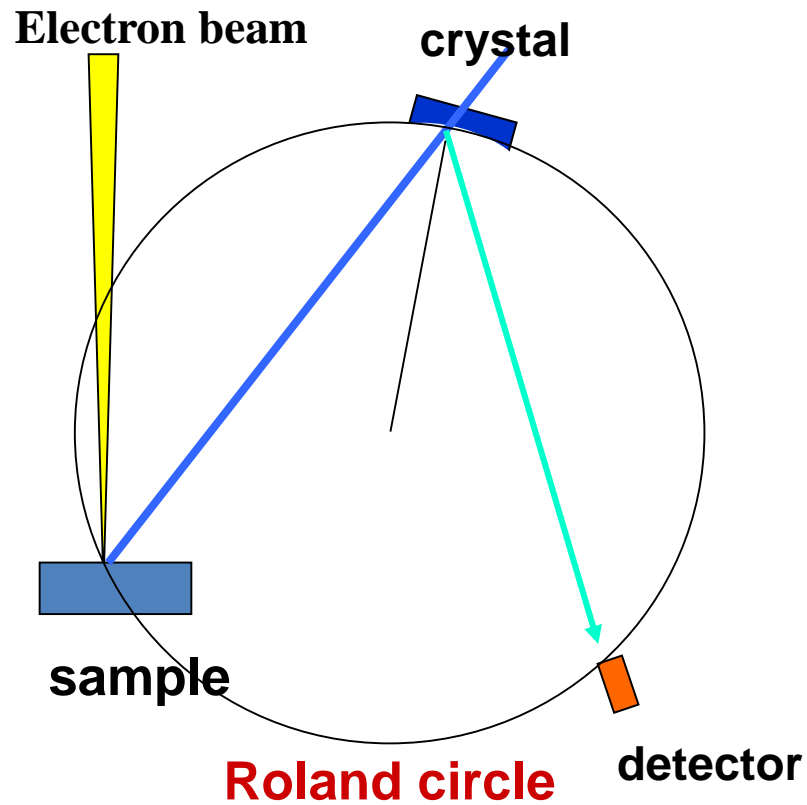
SEM + WDX + CL

# Electron Probe Micro-Analyzer

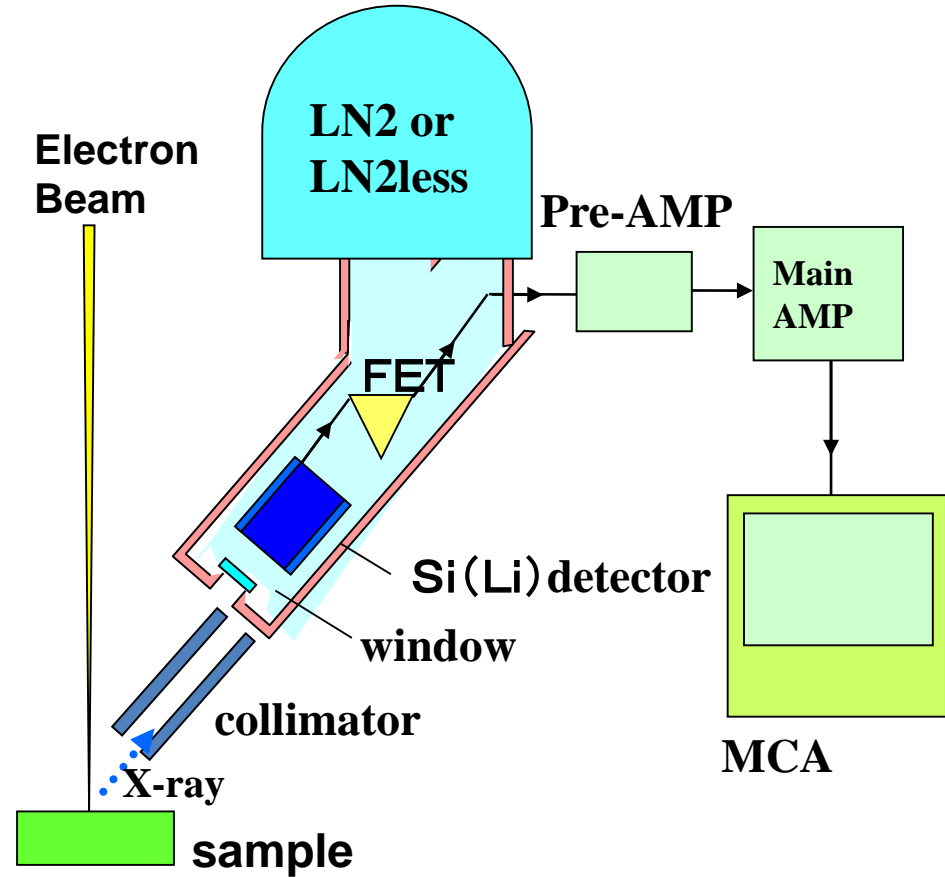


- Imaging down to 3nm Resolution
- Elemental mapping resolution down to a  $0.1\mu\text{m}$
- Elemental Range Be to U
- Light element detection down to 10 ppm.

# Comparison between WDS & EDS

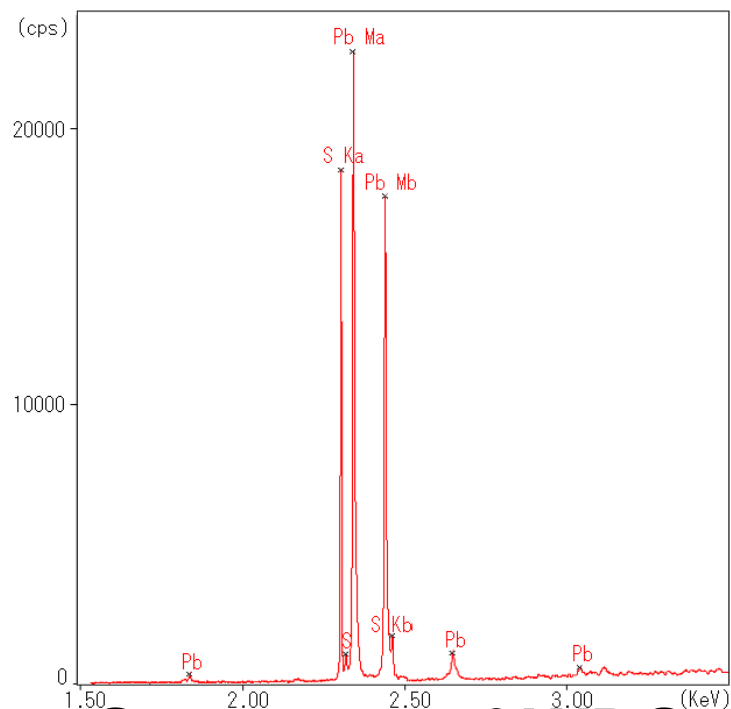


**WDS (Wavelength Dispersive Spectrometer)**

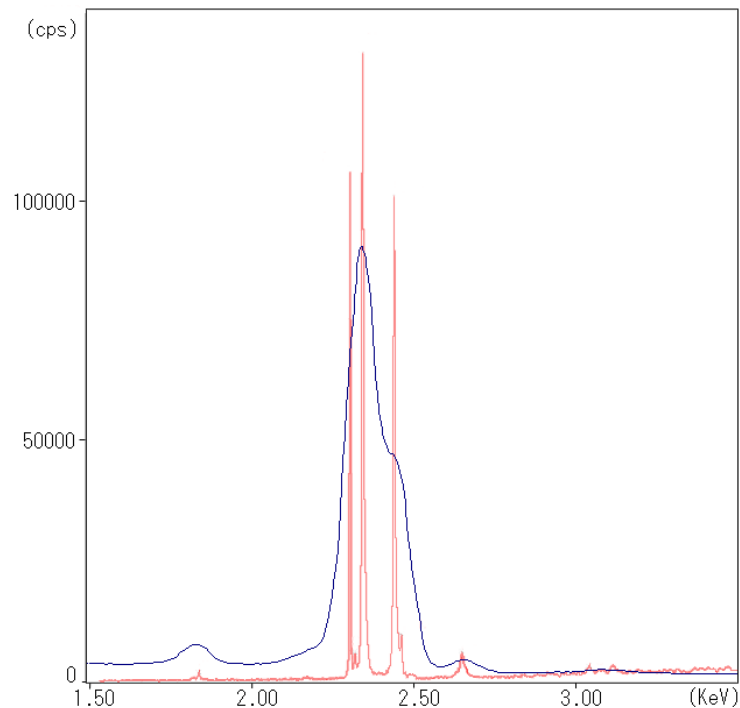


**EDS (Energy Dispersive Spectrometer)**

# Advantages for WDS



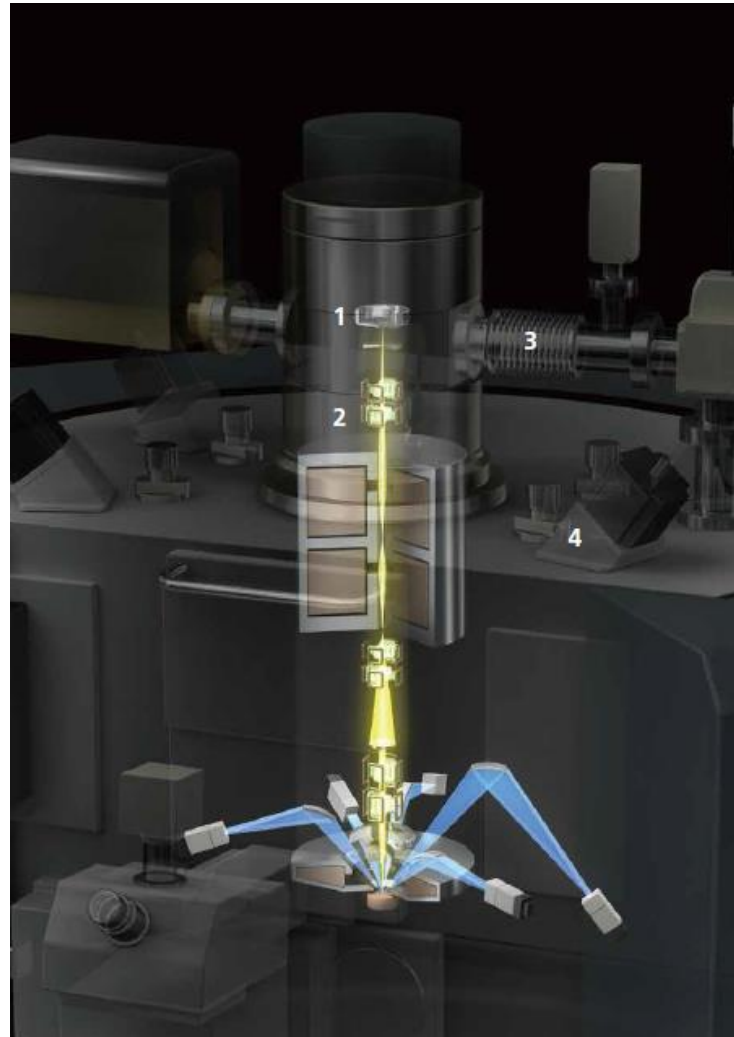
**Spectrum of WDS**



**Spectrum of EDS**

- ① Higher resolution (brings precise identification)
- ② Higher Signal/Noise ratio (Good for lower quantity)
- ③ Never saturate under big current condition ( Can analyze from 100% to very small quantity)

# Electron Probe Micro-Analyzer



# Electron Probe Micro-Analyzer



**EPMA-1720H**



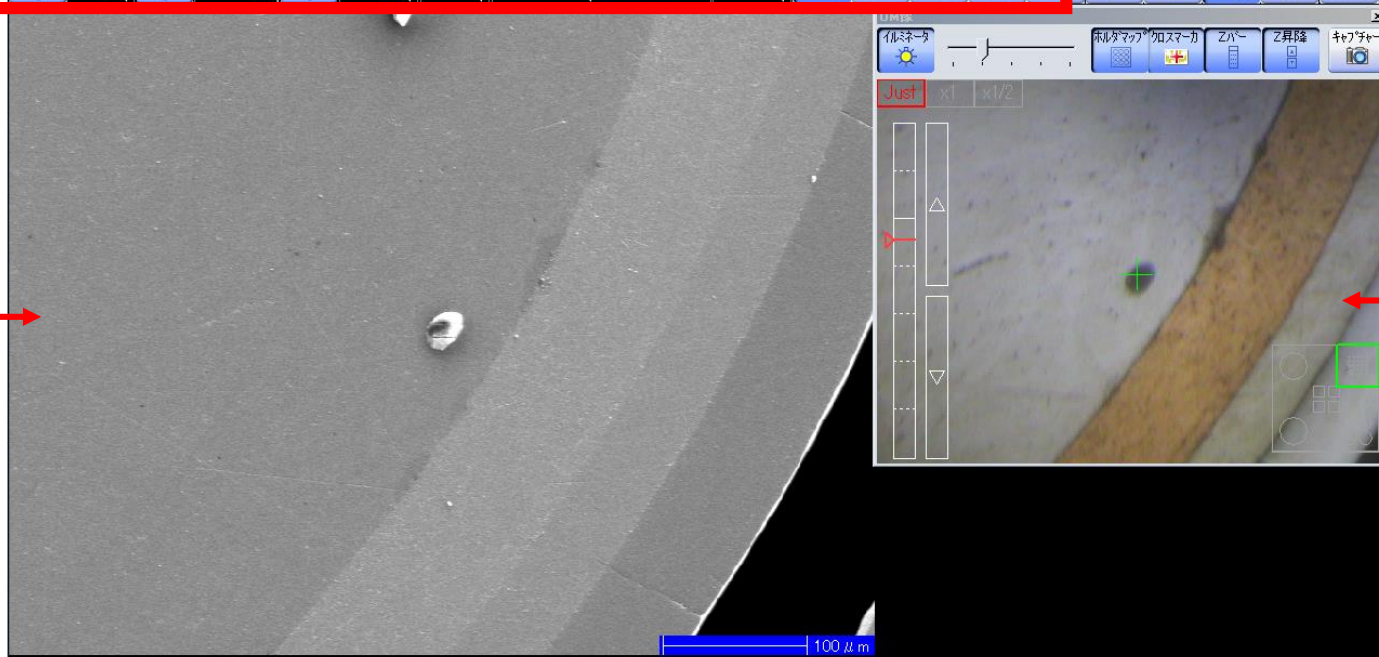
**EPMA-8505G**

# Observation GUI

## Beam Condition

ON	15.00 kV	ON	18.90 $\mu$ A	ON	5.19 nA	Min	Area	Point	Window	200	TV1	Fast1	Slow1	Slow3	Slow
----	----------	----	---------------	----	---------	-----	------	-------	--------	-----	-----	-------	-------	-------	------

## EM Image



## OM Image



パイアス: 115 対物レンズパワー: 55.4710 % フライメント電流: 79.54 % ローテーション: 0.0 deg フライメント点灯時間: 271.29 X: 0.0000 $\mu$ m Y: 0.0000 $\mu$ m プローブコントロール: 35.7574 % ヒステリシス除去	初期化... 移動... ピークサーチ... PHA... X線強度モニタ... スケール... 停止	<table border="1"> <tr> <th>CH1</th> <th>CH2</th> <th>CH3</th> <th>CH4</th> <th>CH5</th> </tr> <tr> <td>RAP</td> <td>PbST</td> <td>LIF</td> <td>LIF</td> <td>LIF</td> </tr> <tr> <td>LSA70</td> <td>LSA120</td> <td>PET</td> <td>ADP</td> <td>PET</td> </tr> <tr> <td>Cr La</td> <td>C Ka</td> <td>Fe Ka</td> <td>Cu Ka</td> <td>Ni Ka</td> </tr> <tr> <td>21.7016</td> <td>44.6996</td> <td>1.9370</td> <td>1.5365</td> <td>1.6579</td> </tr> </table>	CH1	CH2	CH3	CH4	CH5	RAP	PbST	LIF	LIF	LIF	LSA70	LSA120	PET	ADP	PET	Cr La	C Ka	Fe Ka	Cu Ka	Ni Ka	21.7016	44.6996	1.9370	1.5365	1.6579	ボジション... B.L除去 定型移動... 初期化 ホルダ情報... 表示設定 停止	No.3 Plating  X: 71.593 Y: 23.485 Z: 4.544 試料交換
CH1	CH2	CH3	CH4	CH5																									
RAP	PbST	LIF	LIF	LIF																									
LSA70	LSA120	PET	ADP	PET																									
Cr La	C Ka	Fe Ka	Cu Ka	Ni Ka																									
21.7016	44.6996	1.9370	1.5365	1.6579																									

## Electron beam control

## WDS control

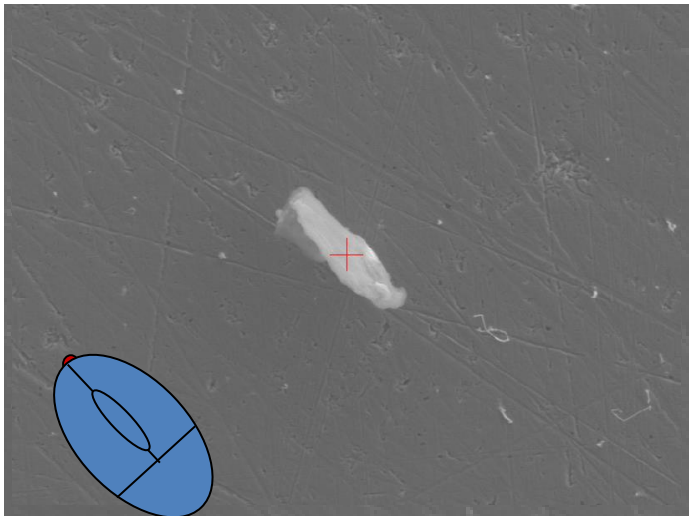
## Sample stage control

シグナル SE	Gain 3	コントラスト 0.217	フィルタ	ブライトネス 0.278	C&B	フォー	スティア	SEM	情報	ゼンマ	OM像	制御画面
---------	--------	--------------	------	--------------	-----	-----	------	-----	----	-----	-----	------

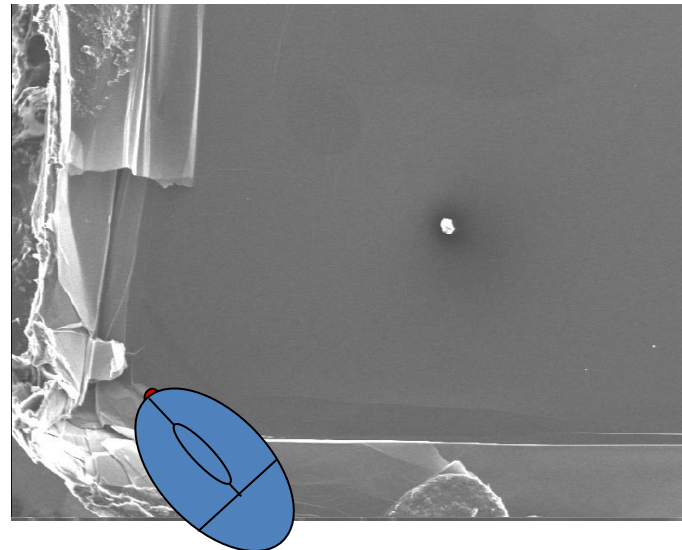


# Sample Stage movement by mouse

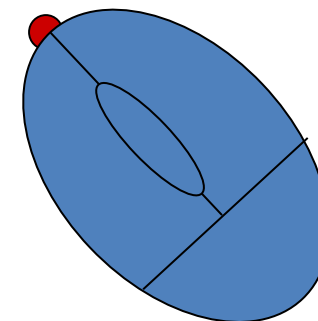
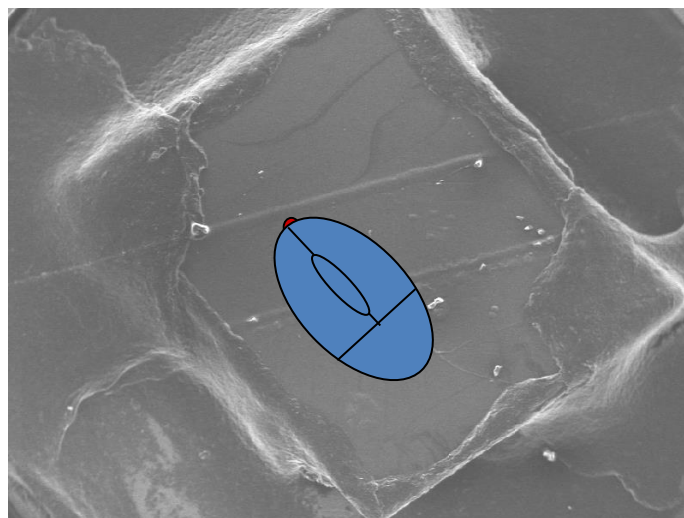
Centering by double clicking



Sample movement by drag & drop

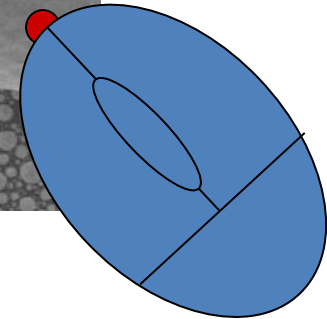
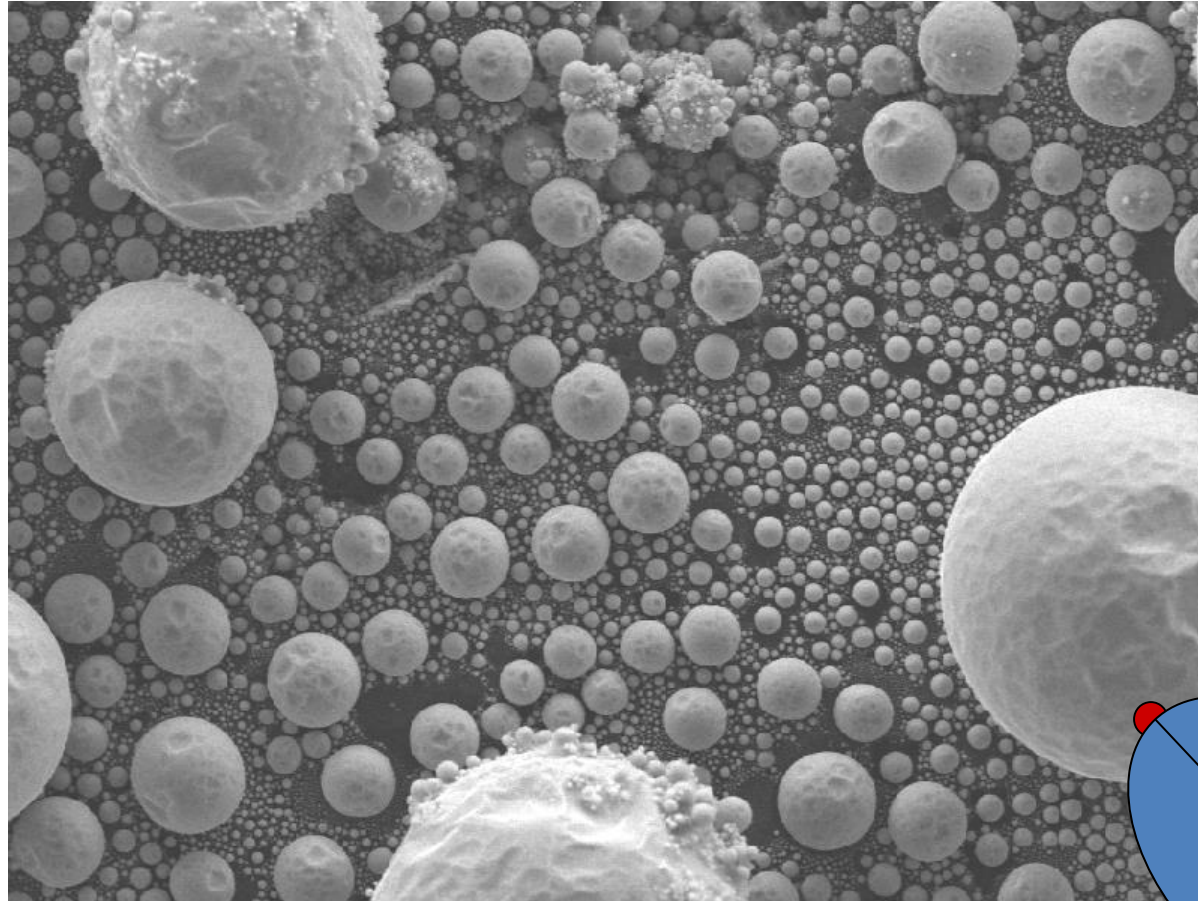


Sample movement by joystick



Mouse operation

# Focusing

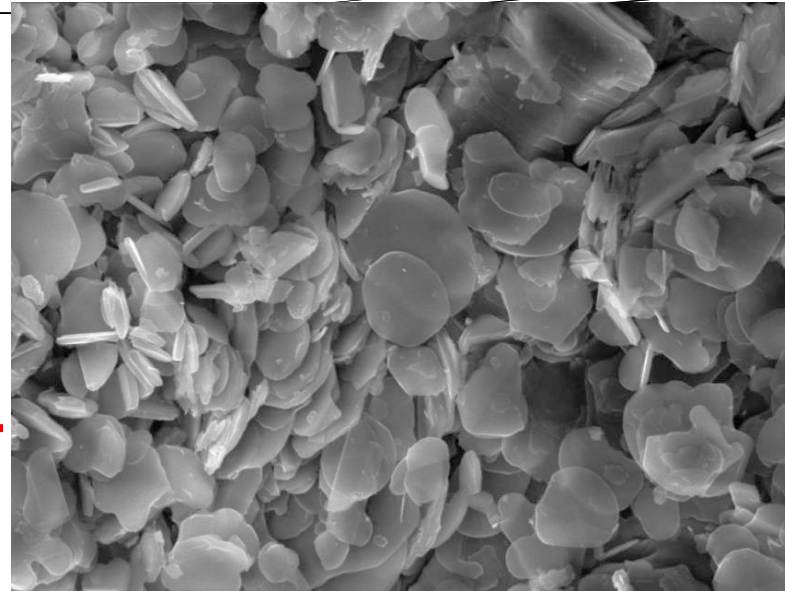
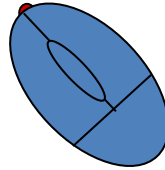


**Mouse  
Operation**

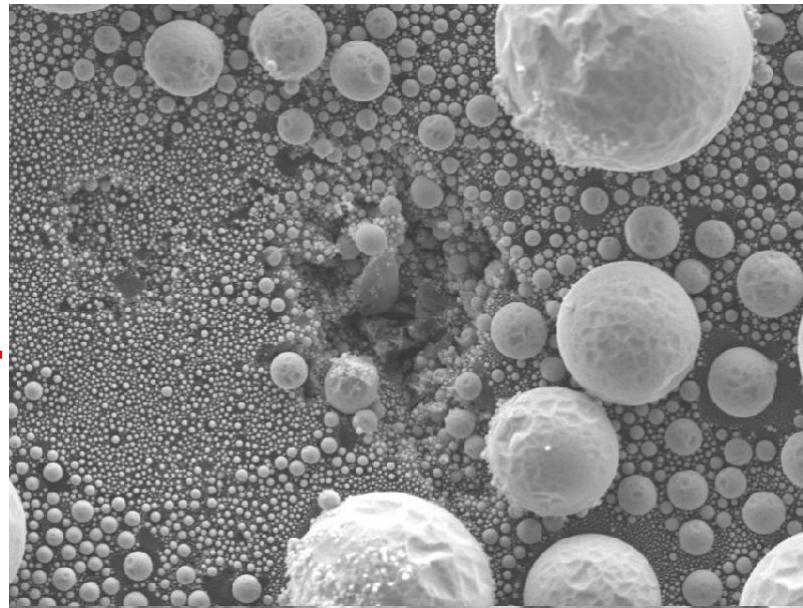
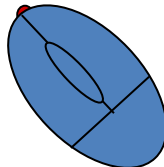
# Auto Functions



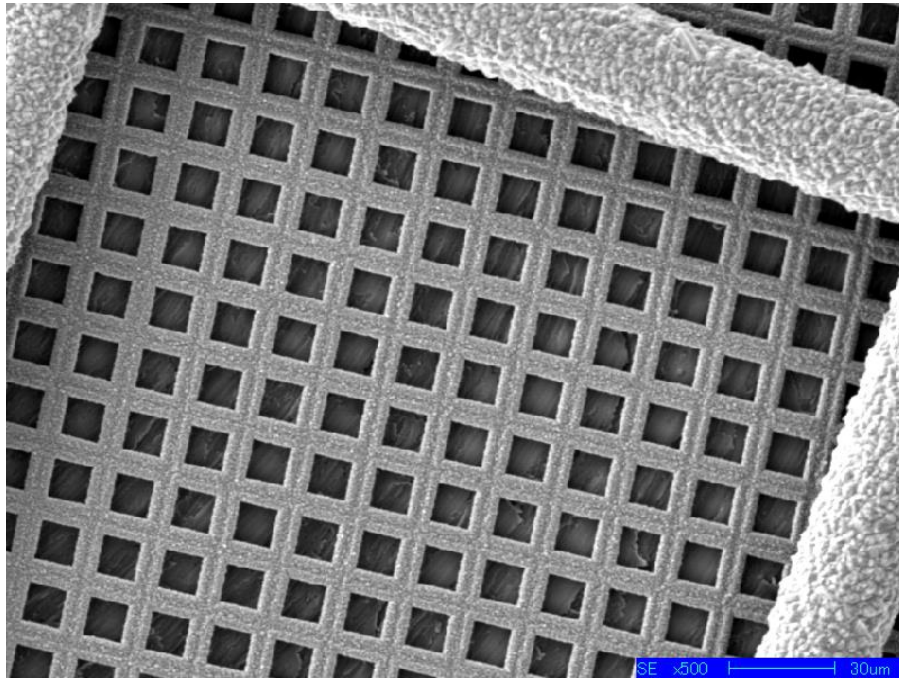
**Auto Contrast & Brightness**



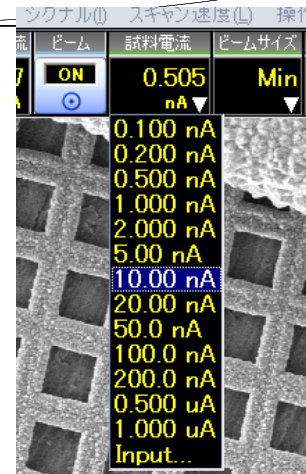
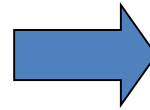
**Auto Stigma & Focusing**



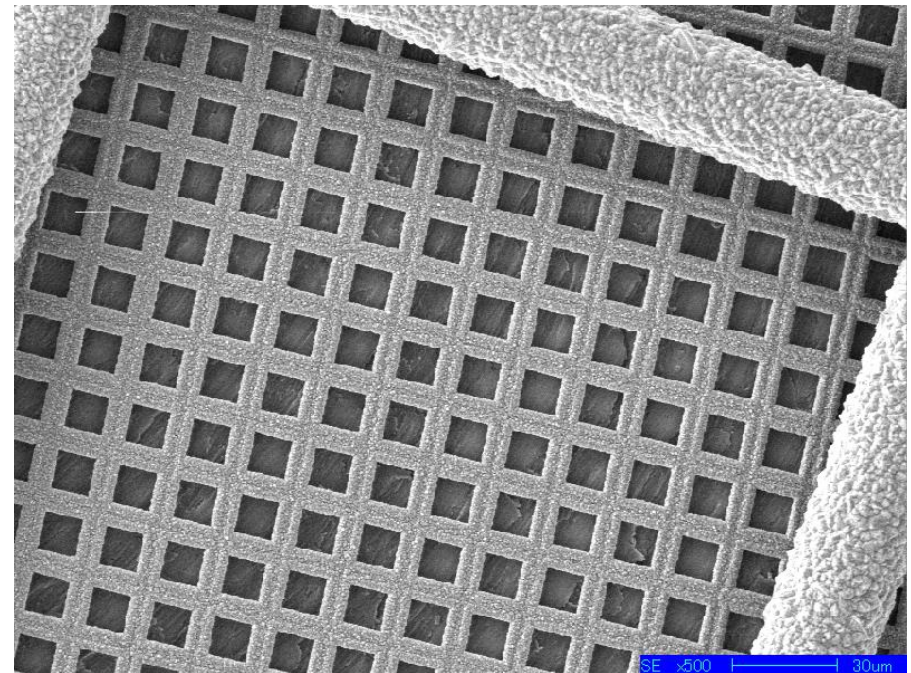
# Change for Beam Current



Beam Current 500pA

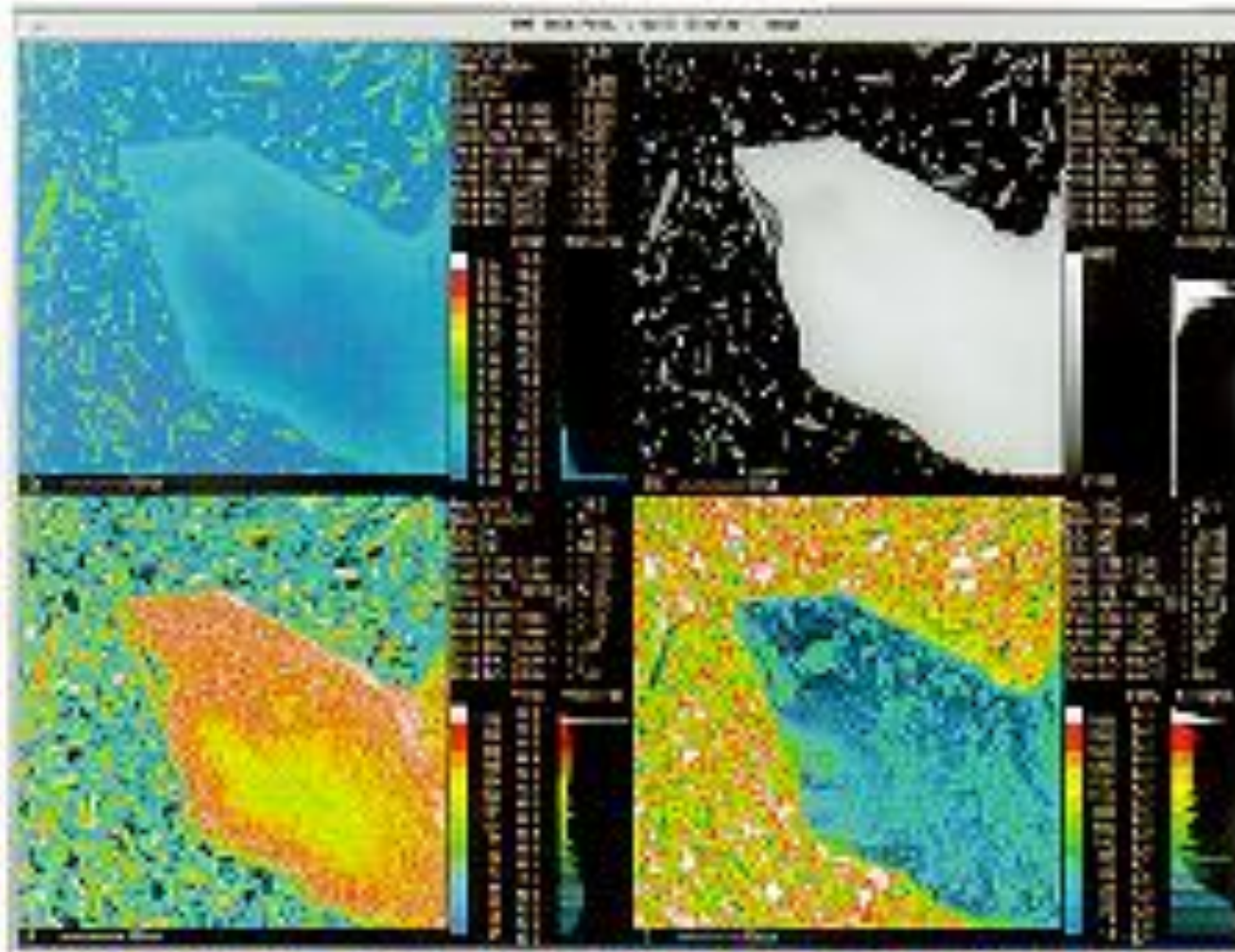


Beam Current 10nA



# Electron Probe Micro-Analyzer

Ca



BSE

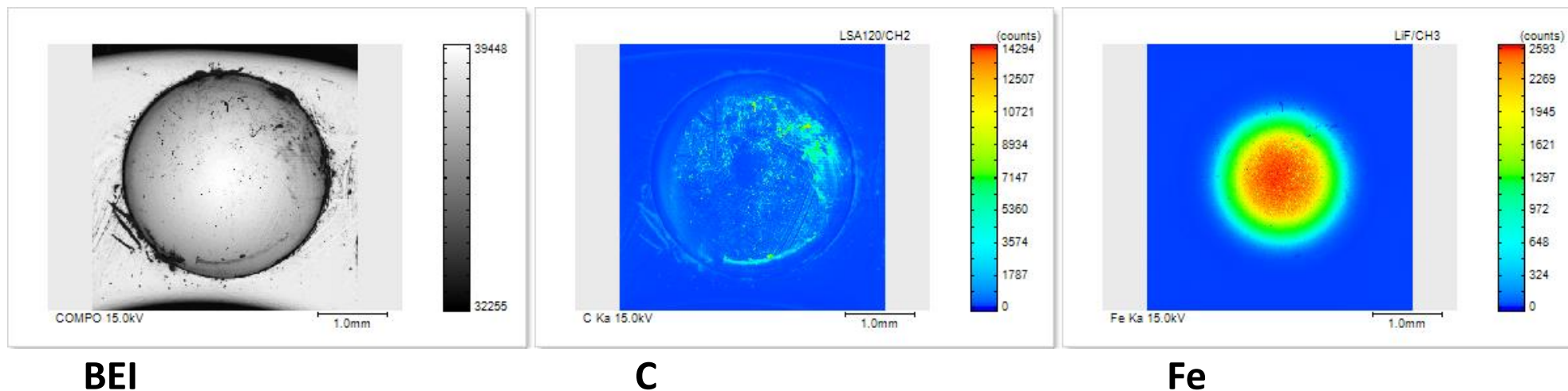
O

C

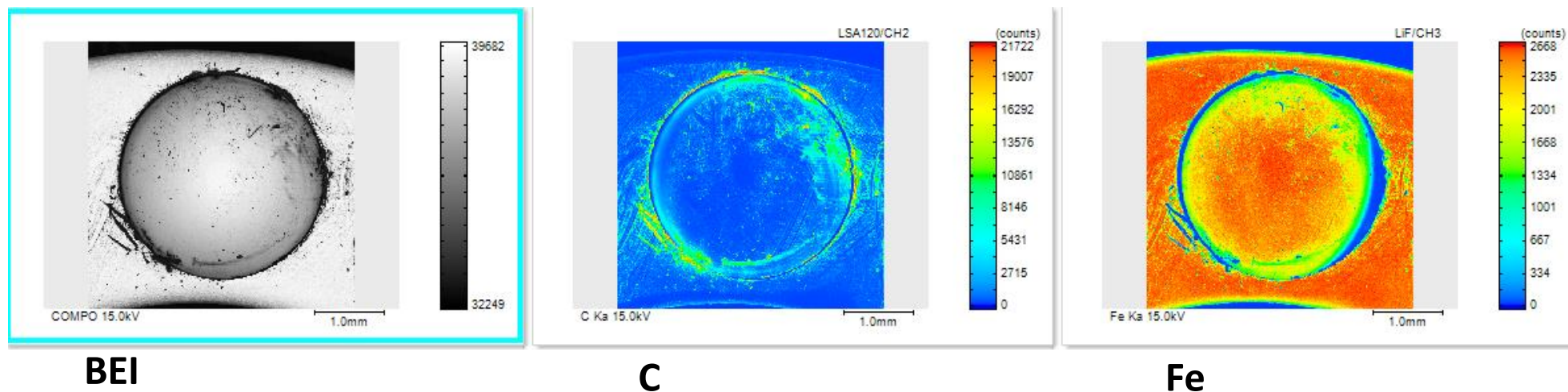
**EPMA 1720 mapping - 500x500  $\mu\text{m}^2$**

# Trace mapping results

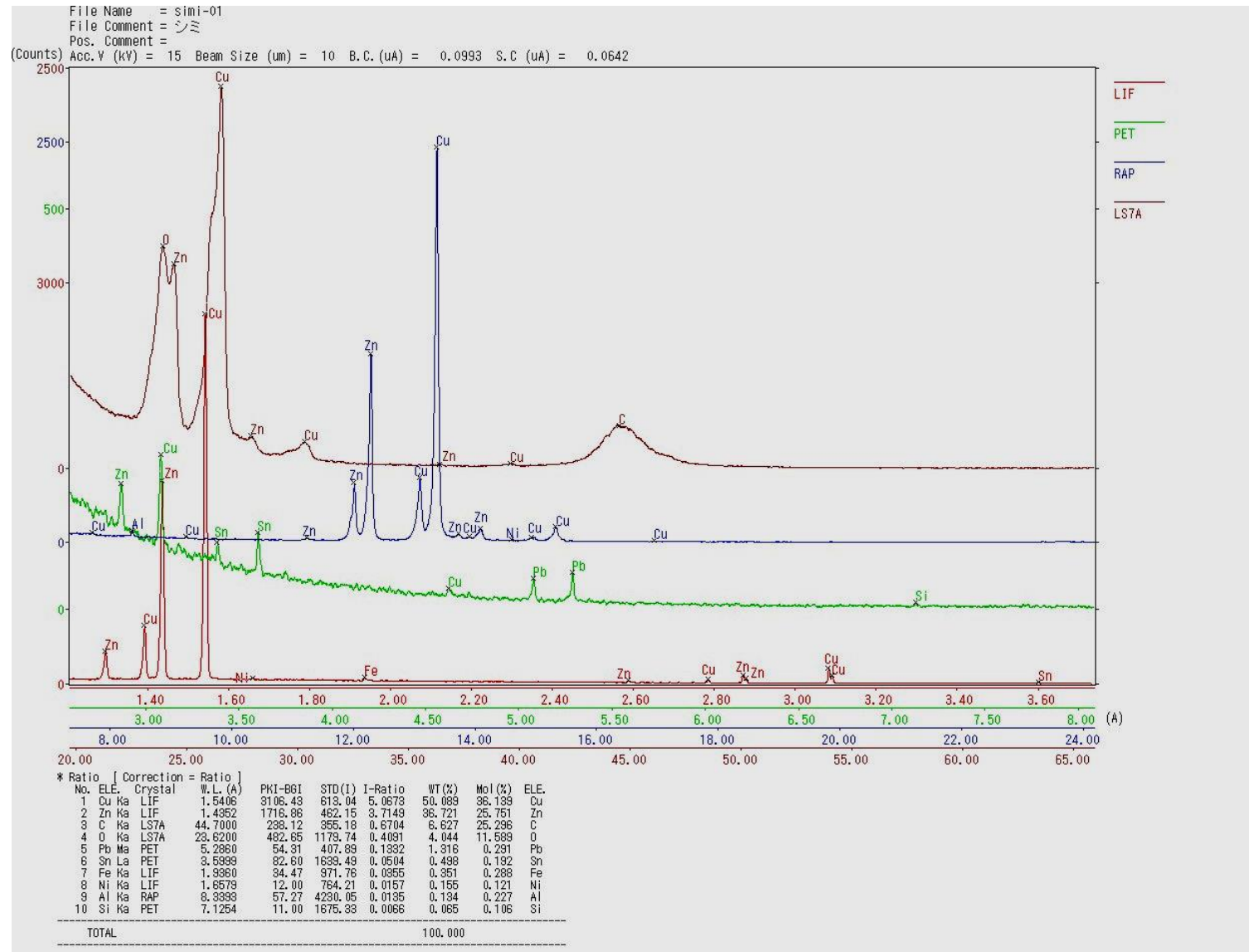
## Trace mapping OFF



## Trace mapping ON

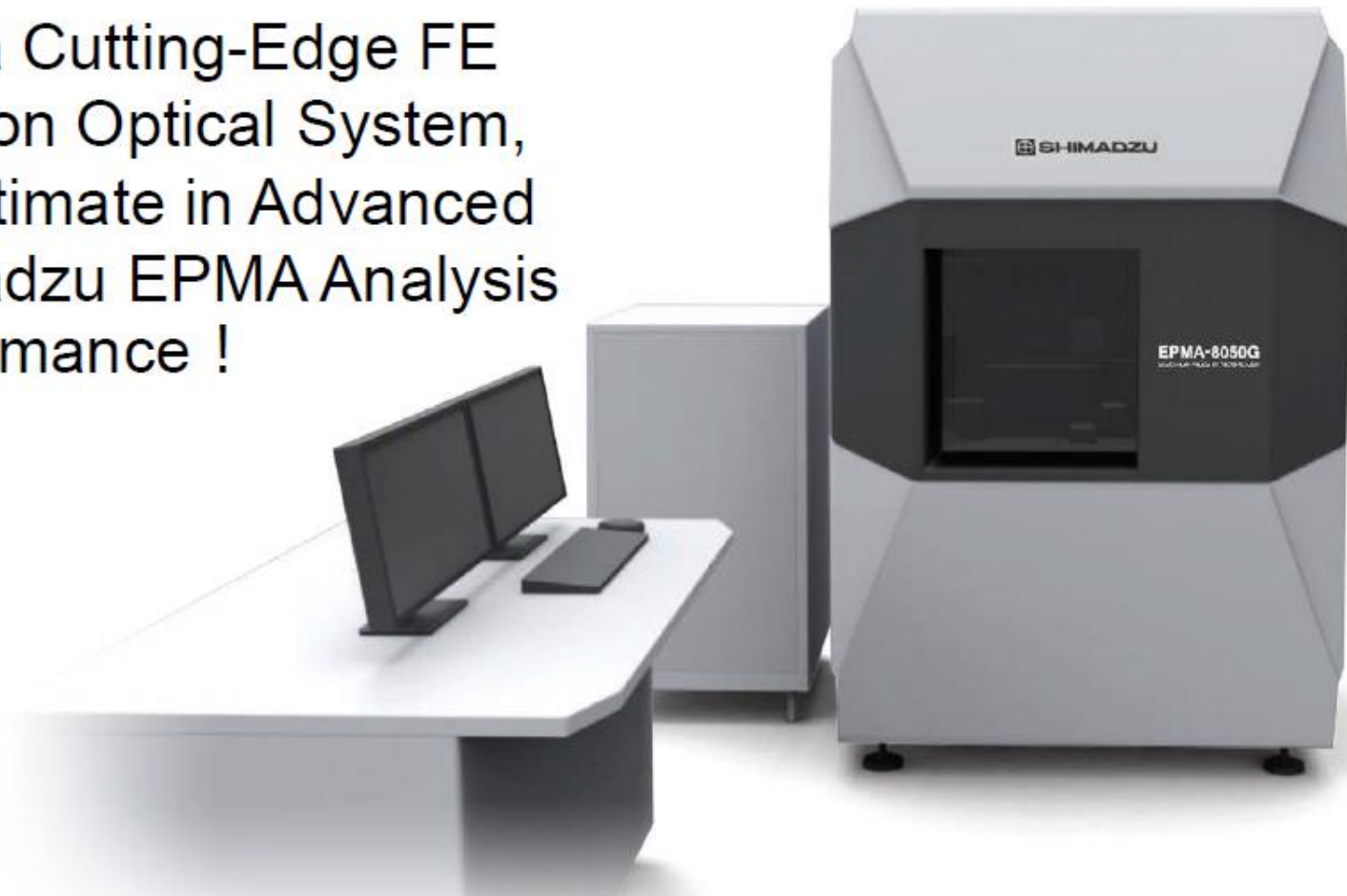


# Qualitative Analysis by WDS



# Debut of "the Grand EPMA"

With a Cutting-Edge FE  
Electron Optical System,  
the Ultimate in Advanced  
Shimadzu EPMA Analysis  
Performance !





# Debut of "the Grand EPMA"

## Refined stylish form !

The latest model of the full-faced-mask which only Shimadzu adopted in the world.

The solid and novel design appropriate for The high-end model brings necessary influence for EPMA including shield and temperature control as well as seamliness.

## and

Shimadzu EPMA-8050G is an energy saving design!

Our cut consumption electricity more than 30% while being a superlative degree product giving the world's best performance !

(Comparison with EPMA-1720 series)

**Shimadzu announces eco-friendly EPMA with a person by prominent operability and energy saving design.**



# The high-end model in pursuit of the beauty of the image

## New electronic optical system & vibration insulation mechanism & Magnetic shield deployment

The secondary-electron image resolution of 3 nm (30 kV accelerating voltage) is the highest level for an EPMA system.

### Comparison of SEM image EPMA-8050G VS. EPMA-1720H

EPMA-8050G

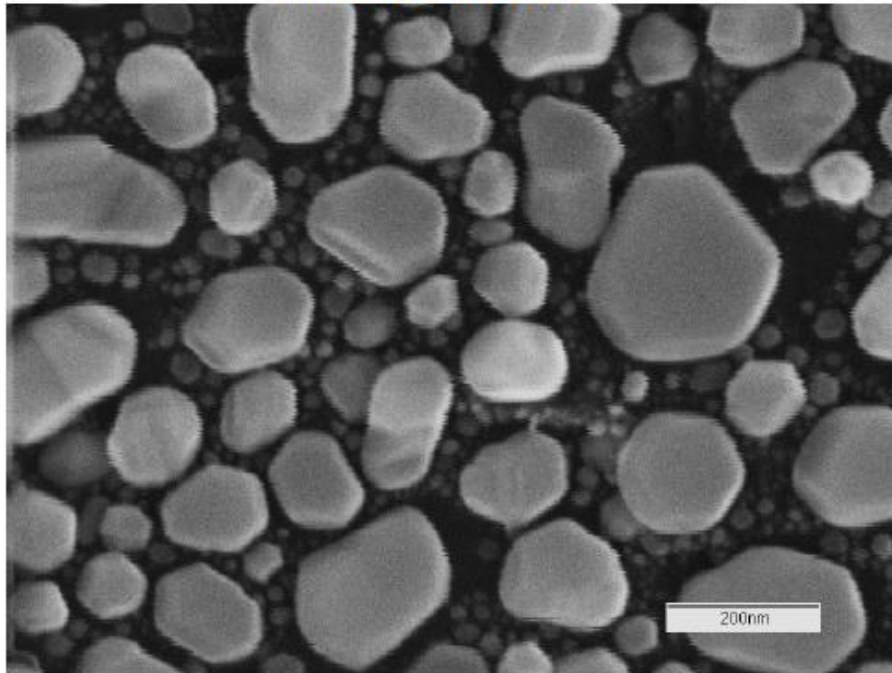


Image resolving power : 3 nm

EPMA-1720H

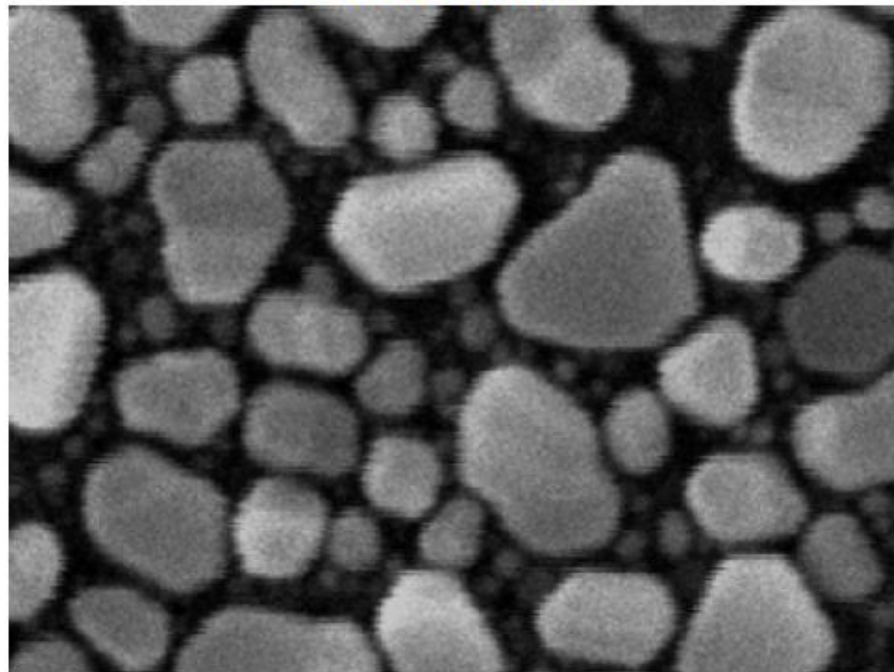
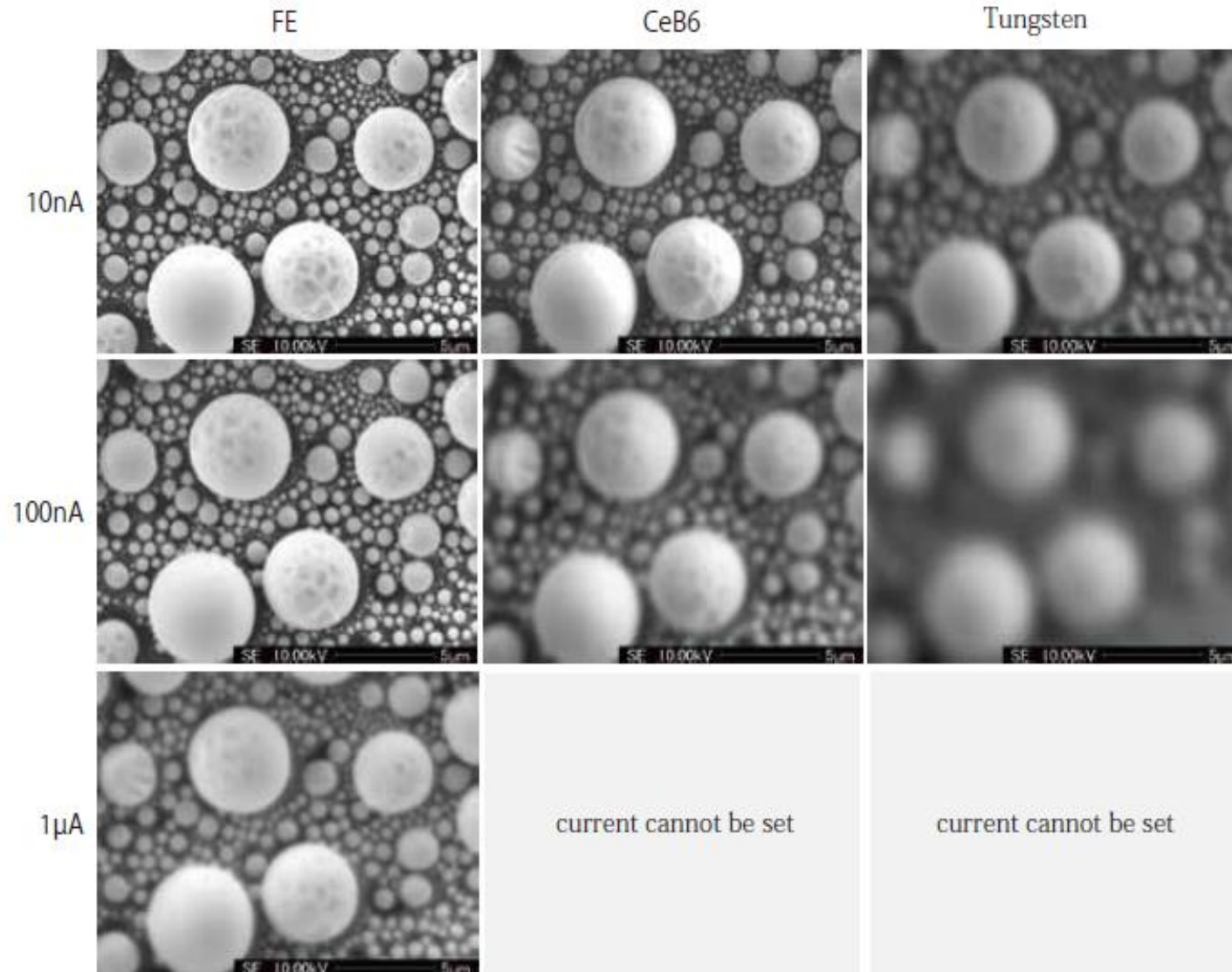


Image resolving power : 5 nm

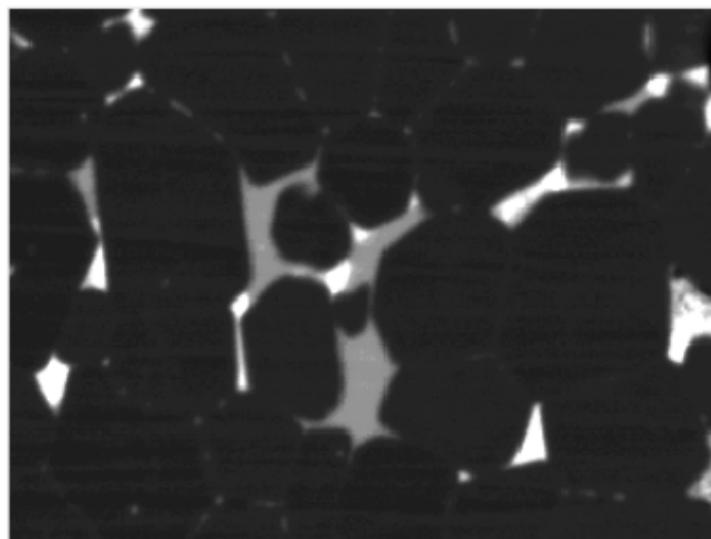
# Image Resolution



Comparison of Electron Gun Beam Characteristics (10 kV accelerating voltage)

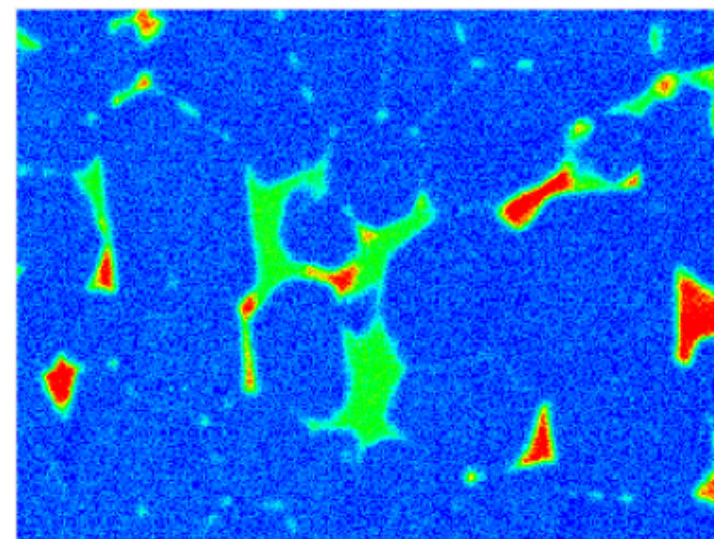
## Ultra High Sensitivity Mapping

The neodymium magnet contributes to energy saving and the downsizing of the product by a strong magnetic field. However, it is dysprosium that is added to raise heat resistance because there is a fault that the neodymium magnet is vulnerable to heat. But most of dysprosium depend on the import from China. Therefore I can plan reduction of dysprosium by reducing alloy powder before baking a magnet, and hardening it conventionally, and optimizing the manufacture process so that it is thin, and an interface (neodymium aspect) surrounds it around a crystal particle uniformly. A characteristic (coercive force) evaluation was carried out conventionally, but, by these data, was able to confirm the neodymium aspect of the crystal interface visually for the first time.



COMPO 10.0kV x6000

5μm



Nd La 10.0kV x6000

5μm

# Specifications (EPMA-8050G)

<b>Electron Source</b>	<b>Schottky emitter</b>
<b>Secondary-Electron Image Resolution</b>	3 nm (30 kV accelerating voltage)
<b>Analysis Conditions for Secondary-Electron Image Resolution</b>	(10 kV accelerating voltage) 20 nm (10 nA beam current) / 50 nm (100 nA beam current) / 150 nm (1 $\mu$ A beam current)
<b>Accelerating Voltage</b>	0.5 kV to 30 kV (in 0.1 kV increments. At 5 kV or less, can be set in 10 V units.)
<b>Beam Current</b>	0.2 nA to 3 $\mu$ A (30 kV accelerating voltage)
<b>Beam Current Stability</b>	$\pm$ 0.3 %/h (Beam current: 50 nA, accelerating voltage: 10 kV)
<b>Magnification</b>	40 $\times$ to 400,000 $\times$
<b>Back-Scattered Electron Detector</b>	4-block, semiconductor detector
<b>Objective Aperture</b>	Selection not required
<b>Vacuum Level</b>	Analysis Chamber: $1.0 \times 10^{-3}$ Pa max. Electron Gun Chamber: $3.5 \times 10^{-7}$ Pa max.
<b>Evacuation Pump</b>	Turbo molecular pumps: One unit for main evacuation; one unit for preliminary evacuation Rotary pumps: One unit for main evacuation; one unit for preliminary evacuation Ion pumps: Two units for the electron gun chamber; one unit for the intermediate chamber
<b>Vacuum Detection</b>	Penning gauge, Pirani gauge, and ion gauge
<b>Automated Functions</b>	Automatic evacuation (main chamber evacuation, shut-down, sample loading chamber evacuation), safety operations via error detection
<b>PC</b>	PC/AT compatible; main memory 8 GB or more; HDD 1 TB or more
<b>Display</b>	23-inch touch panel LCD (Full HD, 1,920 pixels $\times$ 1,080 pixels), two monitors
<b>OS</b>	Windows 7

# Topography Examination

## Scanning Probe Microscope

AFM + LFM+KFM + STM+MFM+FMSPM

### Imaging of Interaction

Signals : Atomic Force (AFM)

Phase

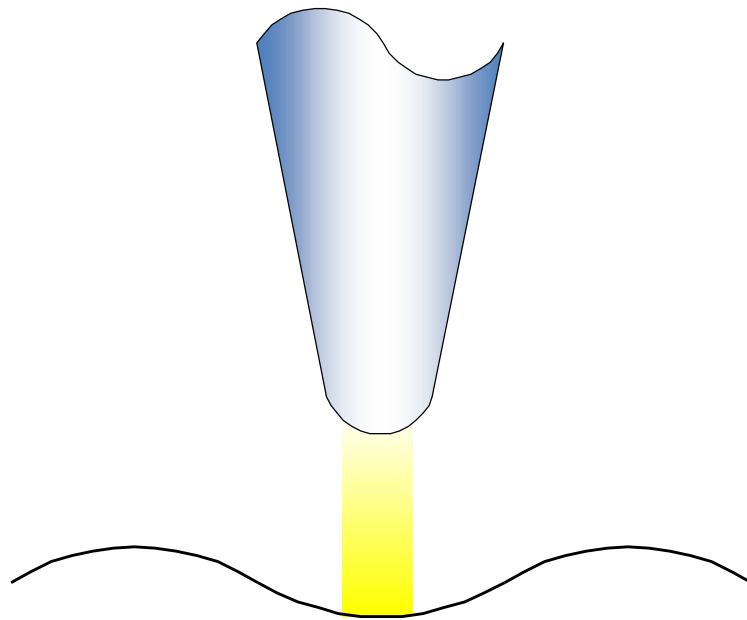
Force Modulation

Current

Magnetic Force (MFM)

Surface Potential (KFM)

Lateral Force (LFM)



**Sample Surface**

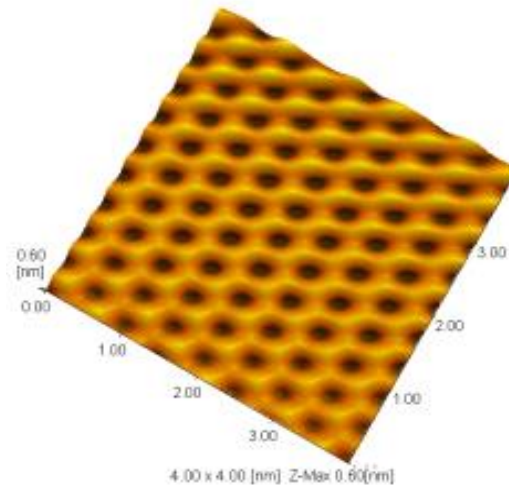
# Scanning Probe Microscopy



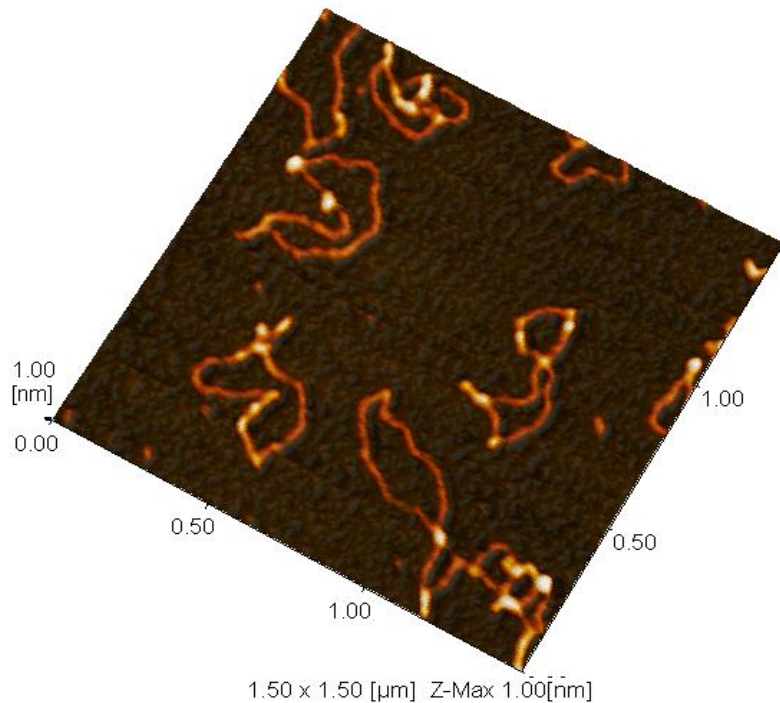
**SPM-9700**

# Function SPM 9700

## High Resolution



mica

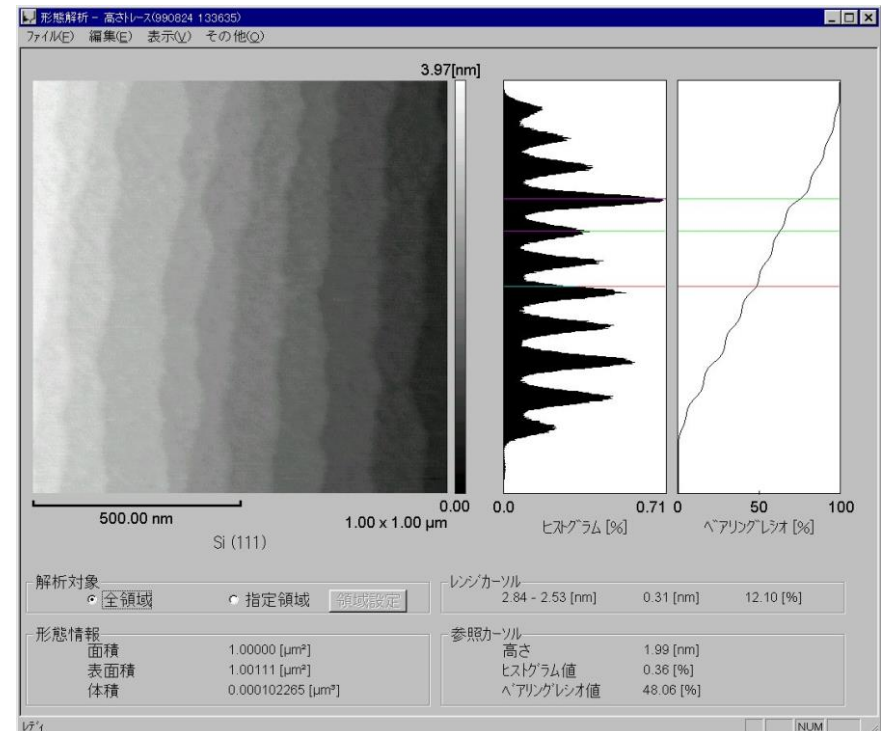


plasmid DNA

## Digital Control

**XY : Full-time 16bit accuracy**

**Z : max. 22bit accuracy**

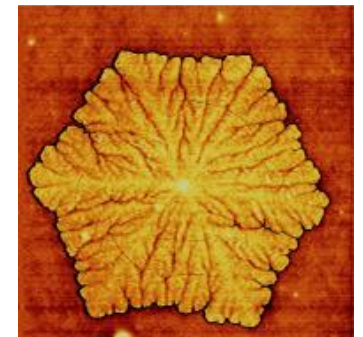


Si (111)



# SPM Application

- Observation of various sample such as semiconductor, thin film, powder and bio samples.
- Shape measurement, Step measurement, Micro roughness measurement
- Physical properties measurement, Electric and Magnetic measurement, Dynamic measurement



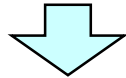
# Function

## Particle Analysis Software

Particle or hole shapes are extracted from image



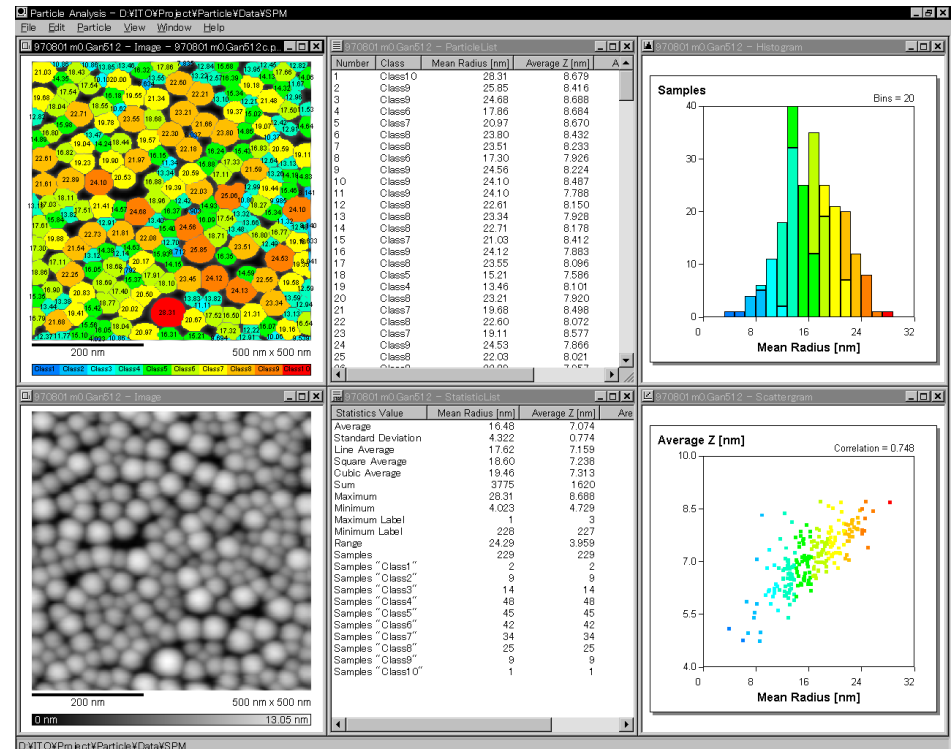
The features of individual particles is determined.



Statistic Processing  
Classification of particles



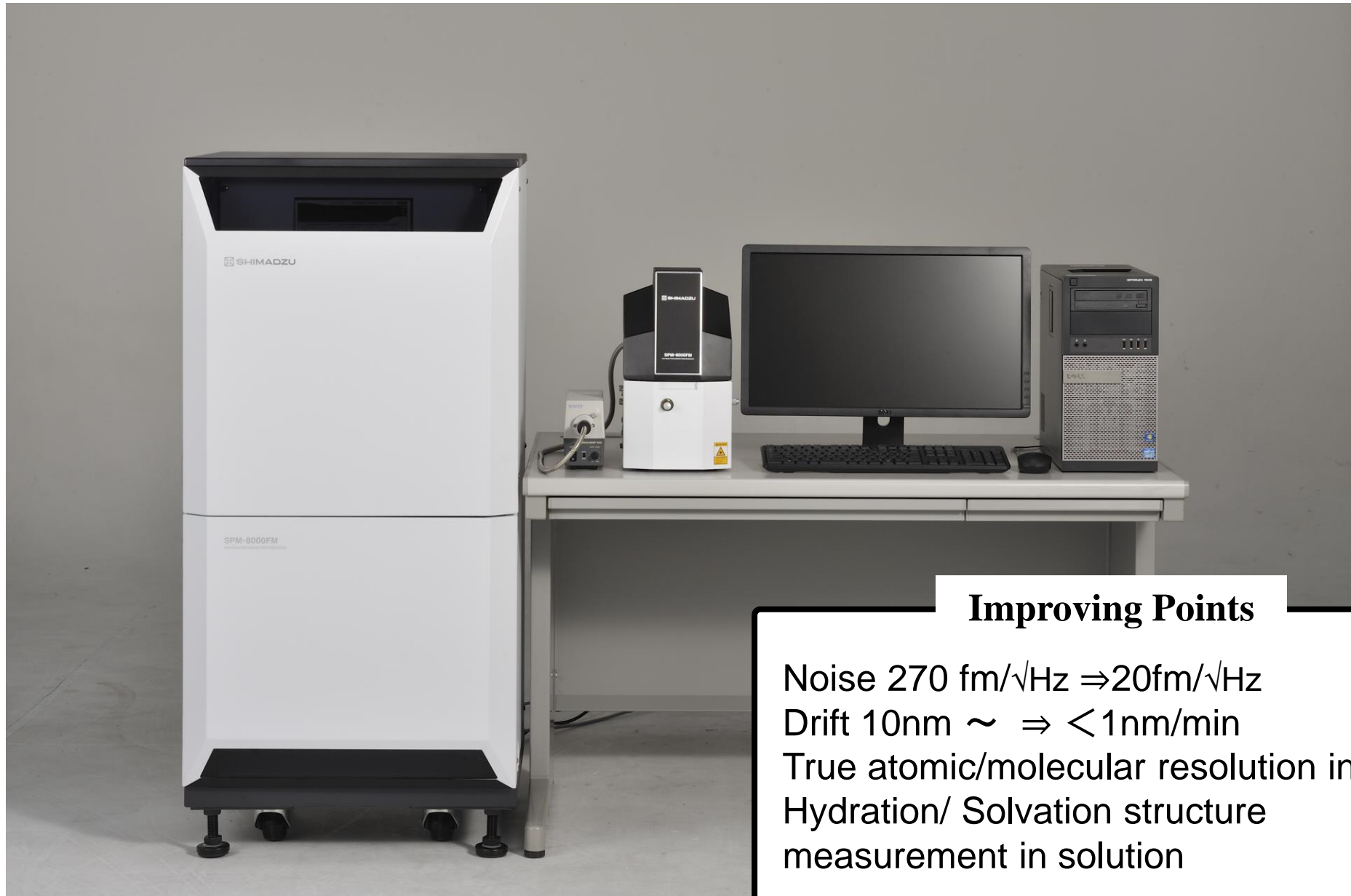
Features: 29types (Radius, Height, Surface Area, Area, Volume, etc)



# SPM - 8000FM



# High Resolution Scanning Probe Microscope SPM-8000FM



## Improving Points

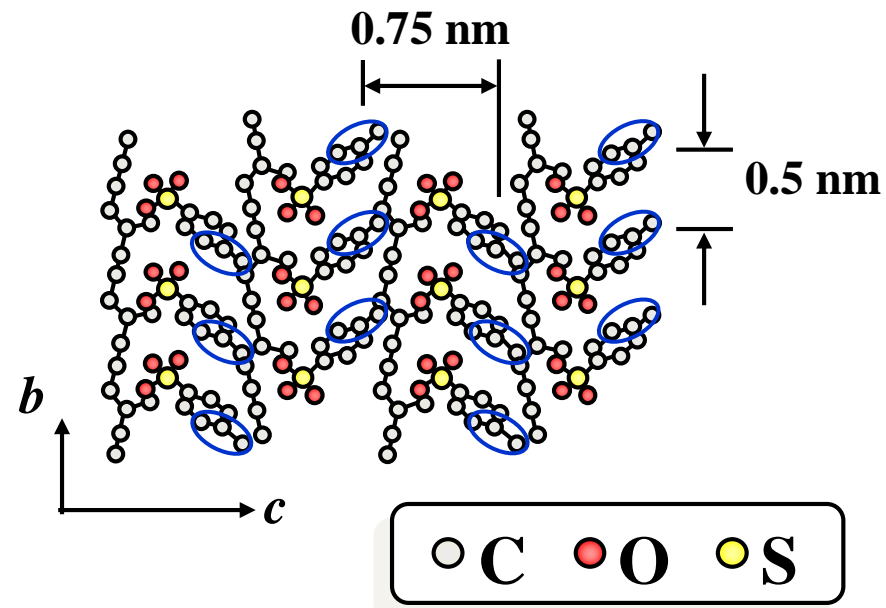
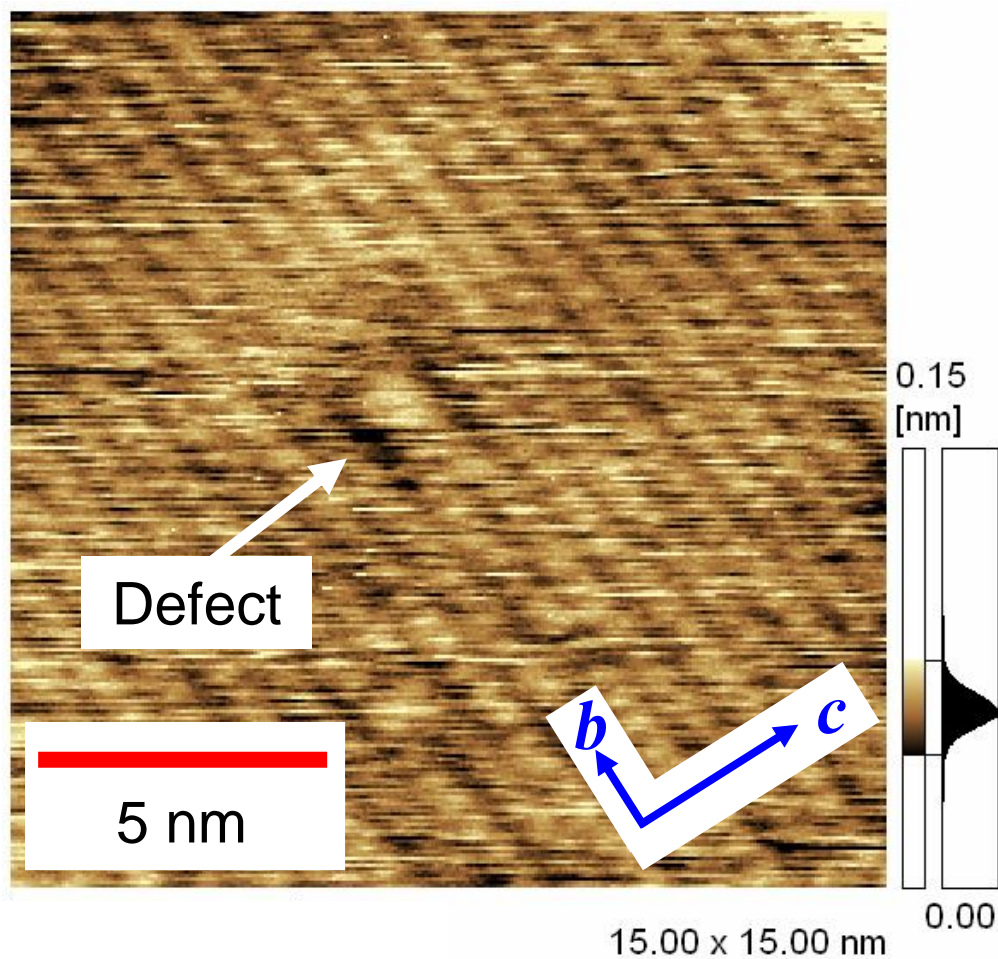
Noise  $270 \text{ fm}/\sqrt{\text{Hz}} \Rightarrow 20 \text{ fm}/\sqrt{\text{Hz}}$

Drift  $10 \text{ nm} \sim \Rightarrow < 1 \text{ nm}/\text{min}$

True atomic/molecular resolution in air  
Hydration/ Solvation structure  
measurement in solution

# High Resolution Topography in Air

## Poly diacetylene single crystal



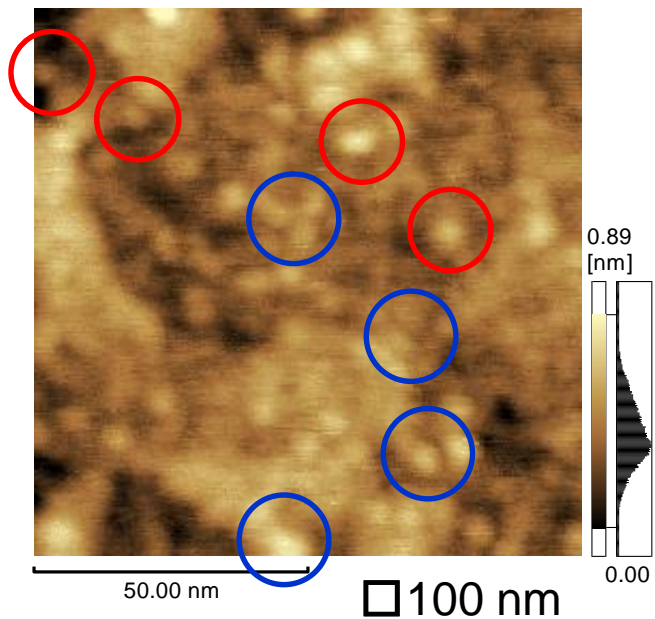
The side chains on the topmost surface

# Physical Properties Measurements

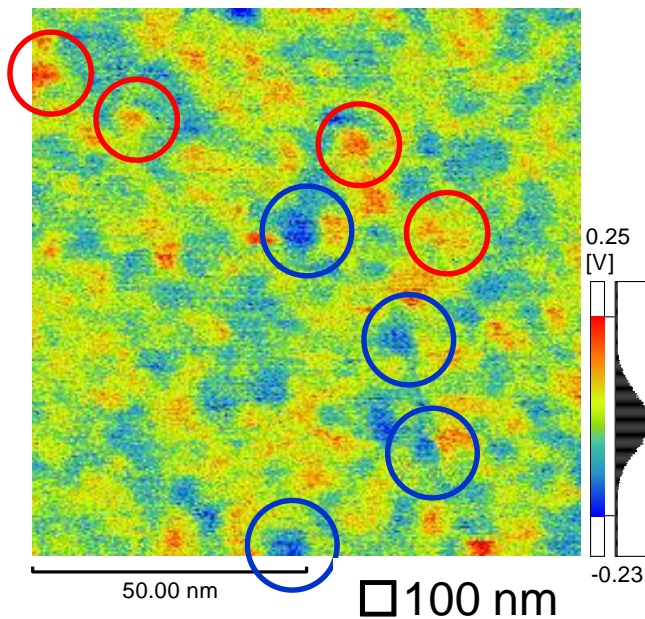
## Surface Potential Measurement in Gas

# Contact Potential Deference Distribution in Gas

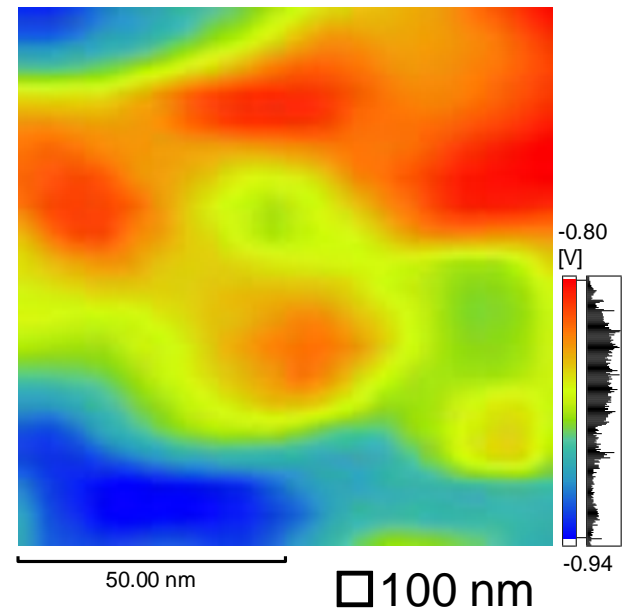
## Pt clusters / TiO<sub>2</sub>



Topography

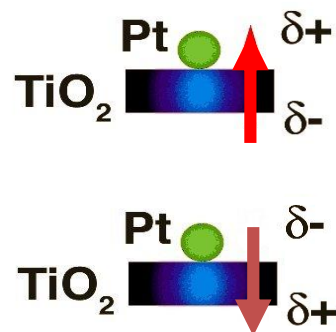


CPD by KFM

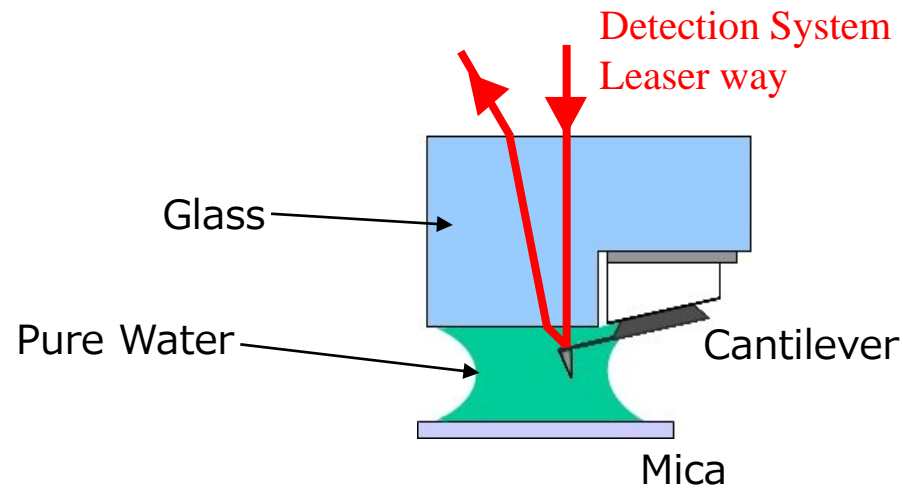


Conventional KFM

Single-nm resolution image observed in an atmospheric pressure N<sub>2</sub> gas environment



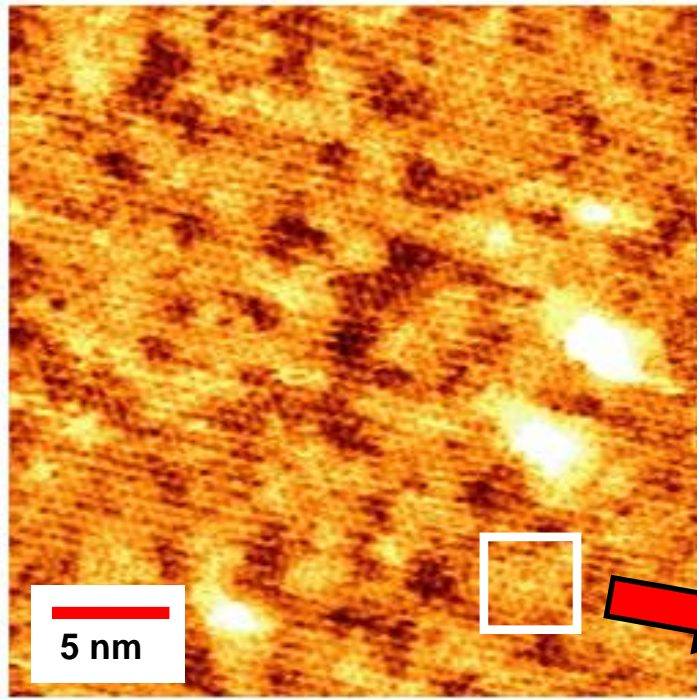
# High Resolution Observation in Solution



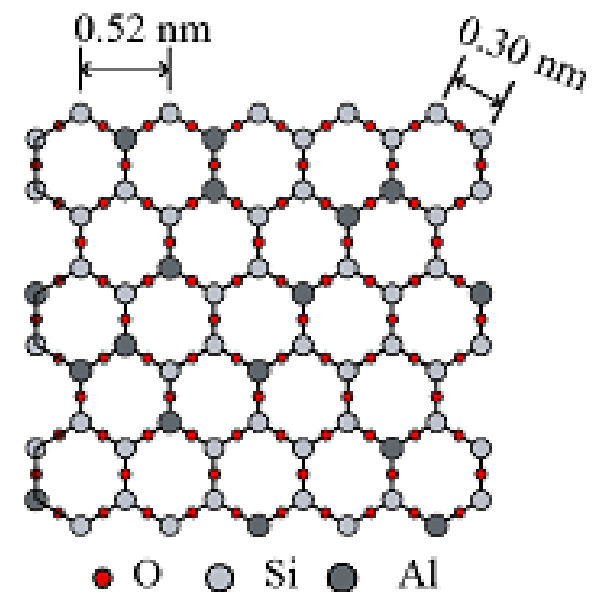
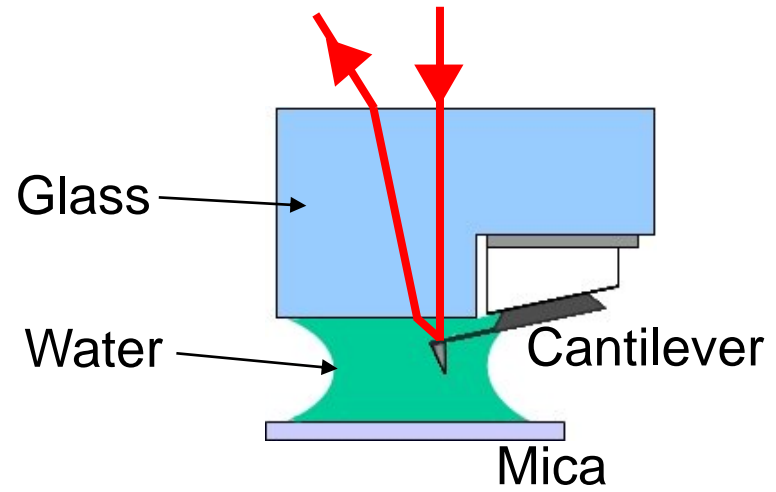
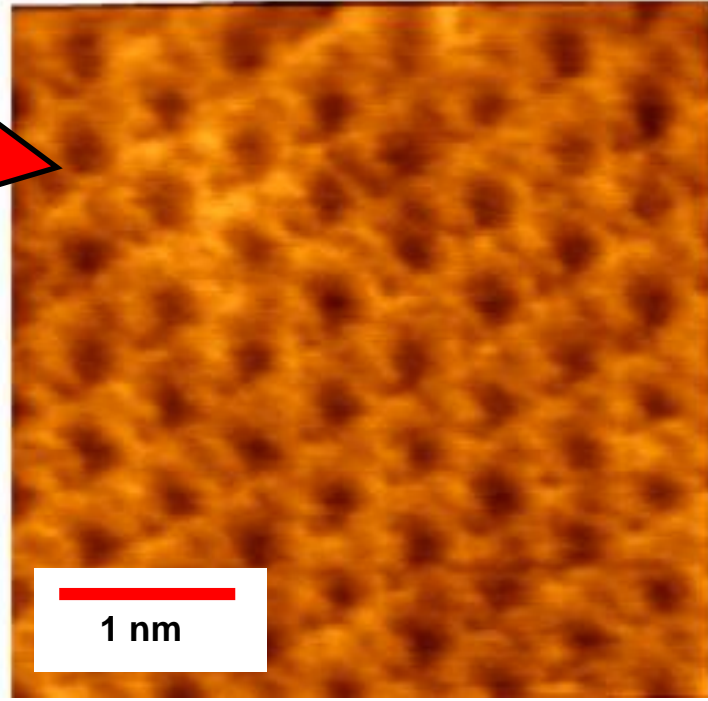


# High Resolution Topography in Water

## Muscovite mica

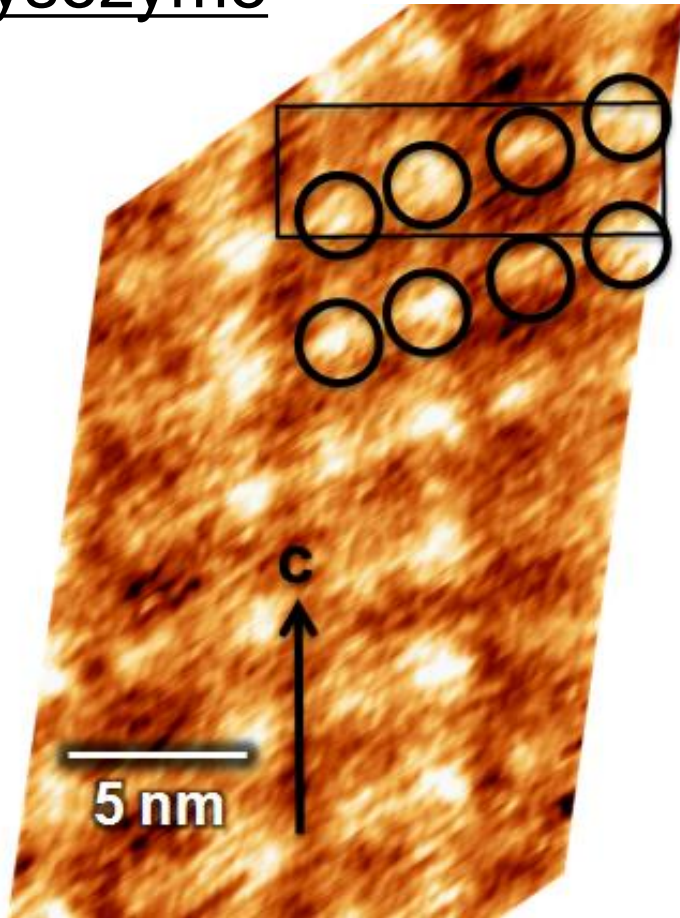


Cleavage plane immersed in water



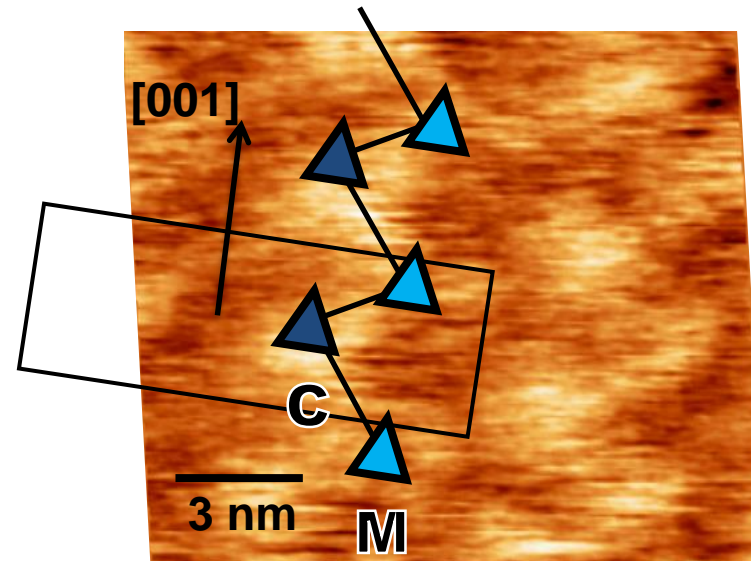
# Molecular Resolution Observation of Soluble Protein Crystals

## Lysozyme

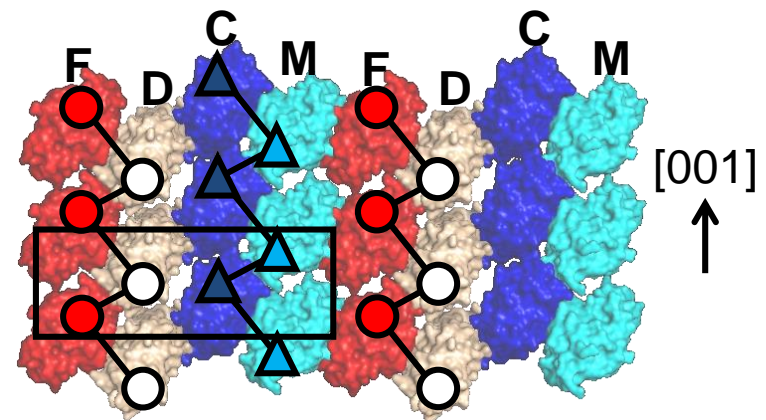


The rectangle represents a surface unit cell with the arrow indicating the c-axis.

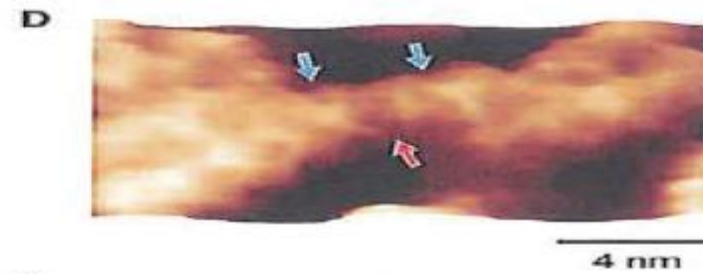
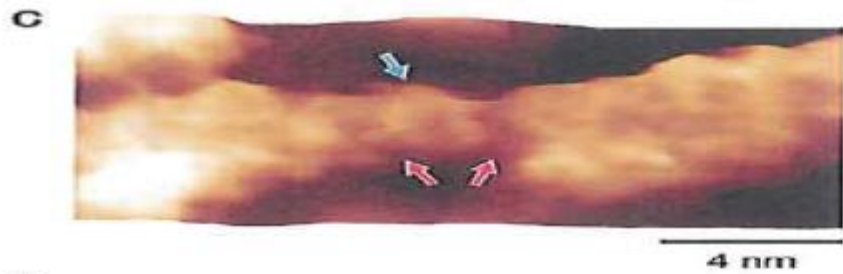
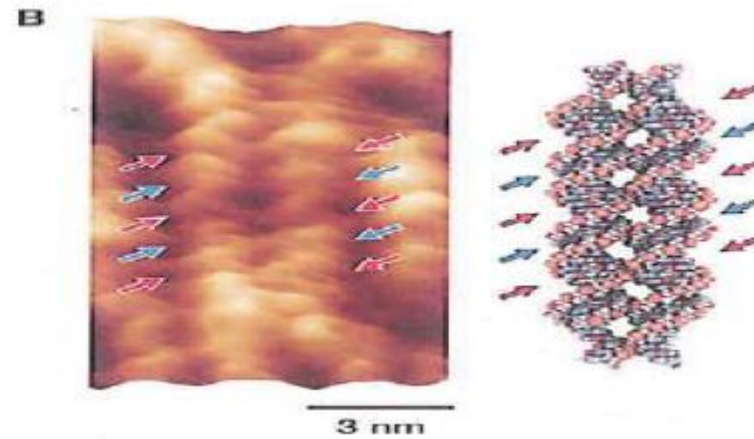
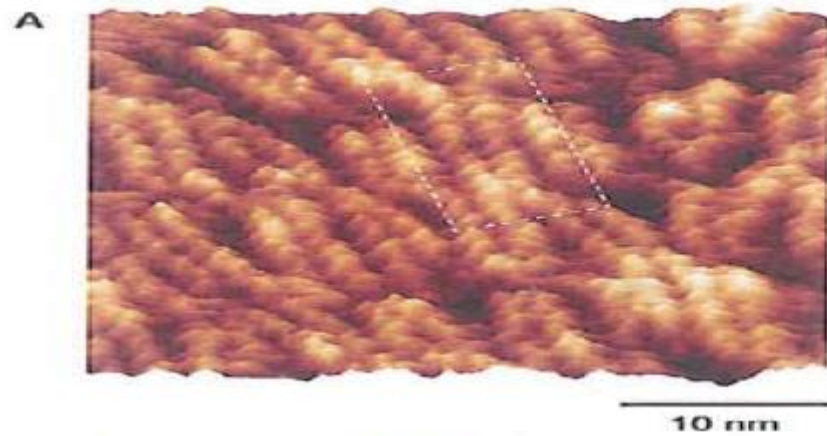
Molecular resolution image observed in a saturated solution



Tetragonal lysozyme (110) face



# Beyond the Helix Pitch: Direct Visualization of Native DNA in Aqueous Solution



# Antibody Observation reported in Japanese newspaper and TV

**Y字の抗体じゅうたん模様**  
京大チーム観察

原子間力顕微鏡で観察した抗体  
規則正しく並ぶ

1個の抗体はY字形

溶液の成分を調節すると、抗体が6個1組で環状に集合

50ナノメートル

5ナノメートル

画面上では映りていない

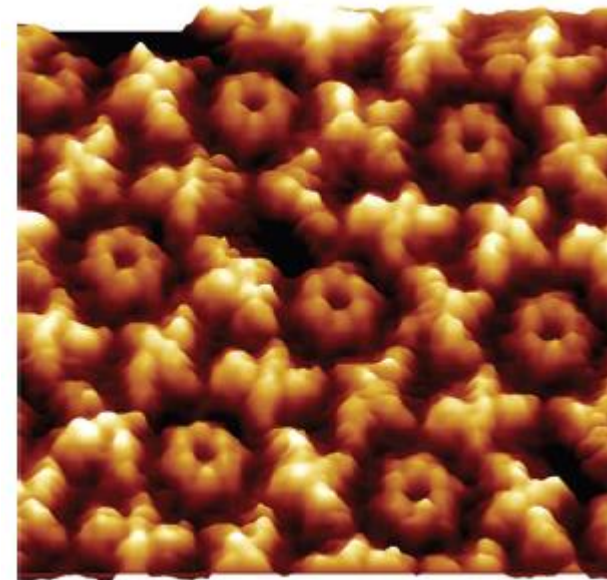
(写真は山田准教授提供)

体内に侵入した病原体から体を守る「抗体」の構造を、高精度の顕微鏡を使って詳細に観察することに成功したと、京都大の山田啓文准教授らのチームが発表した。抗体に特有なY字形がはっきりと見え、抗体がじゅうたんの模様のように整然と並ぶこともわかった。20日の科学誌ネイチャー・マテリアルズ電子版に論文が掲載される。

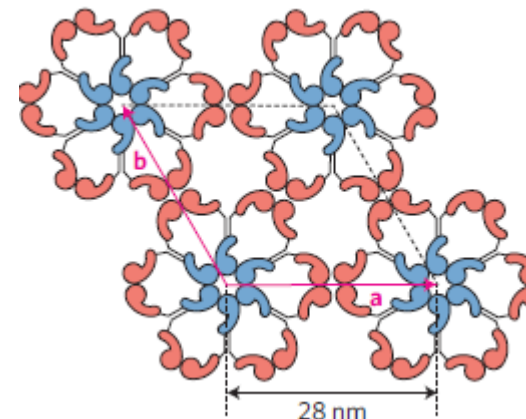
抗体はY字形をしており、2本の腕がウイルスなどのたんぱく質を捉える。チームは、超極

細の針先で物質表面をなぞって細かな形を画像化する「原子間力顕微鏡」を利用。針先の感度を高め、マウスの抗体(長さ約10~20ナノメートル、ナノは10億分の1)を生体内に近い溶液に浸した状態で観察した。さらに、溶液の成分を調節すると、抗体は6個1組で環状に集まり、規則正しく並んだ。免疫反応を高めるためグループ化するとみられる。

山田准教授は「高い密度で集合した抗体は、ウイルスの有無を調べる高感度センサーとして利用できるだろう」と話している。



20 nm

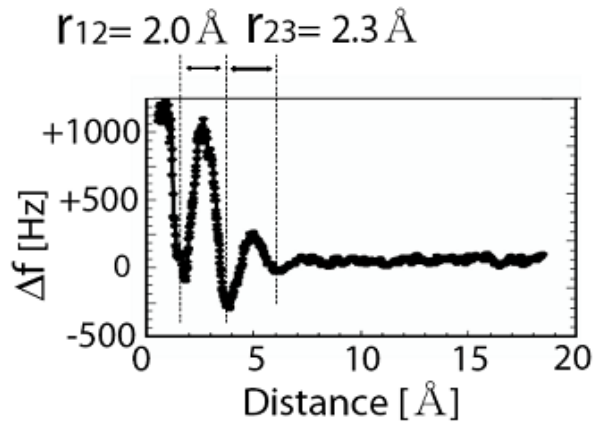
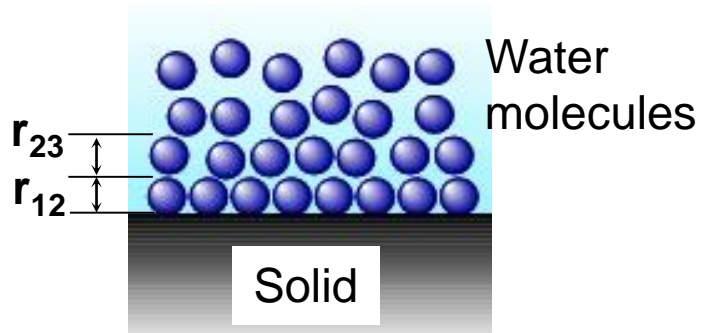


# Hydration / Solvation structure measurement

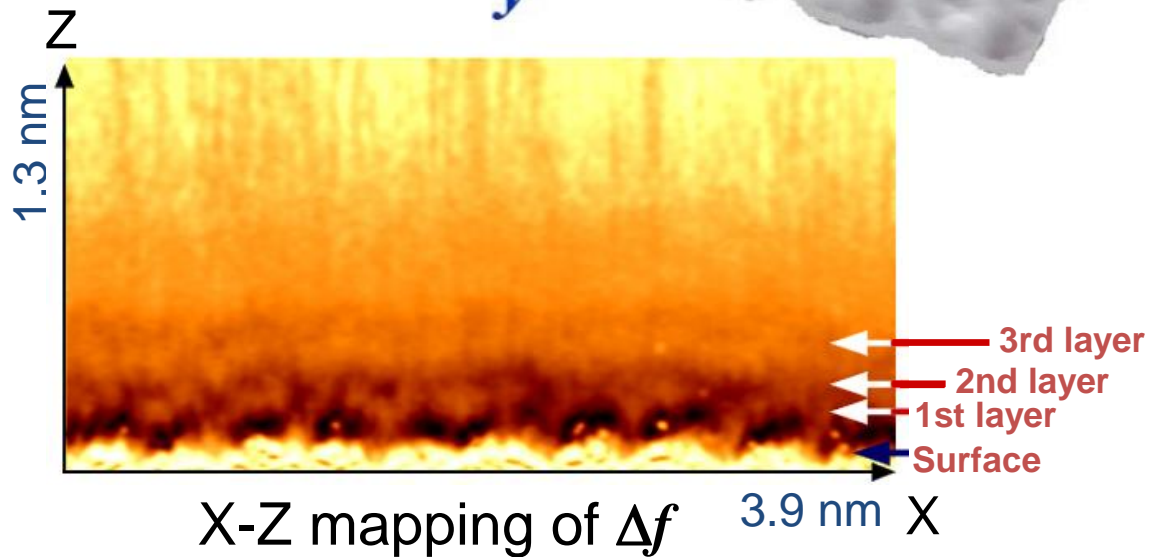
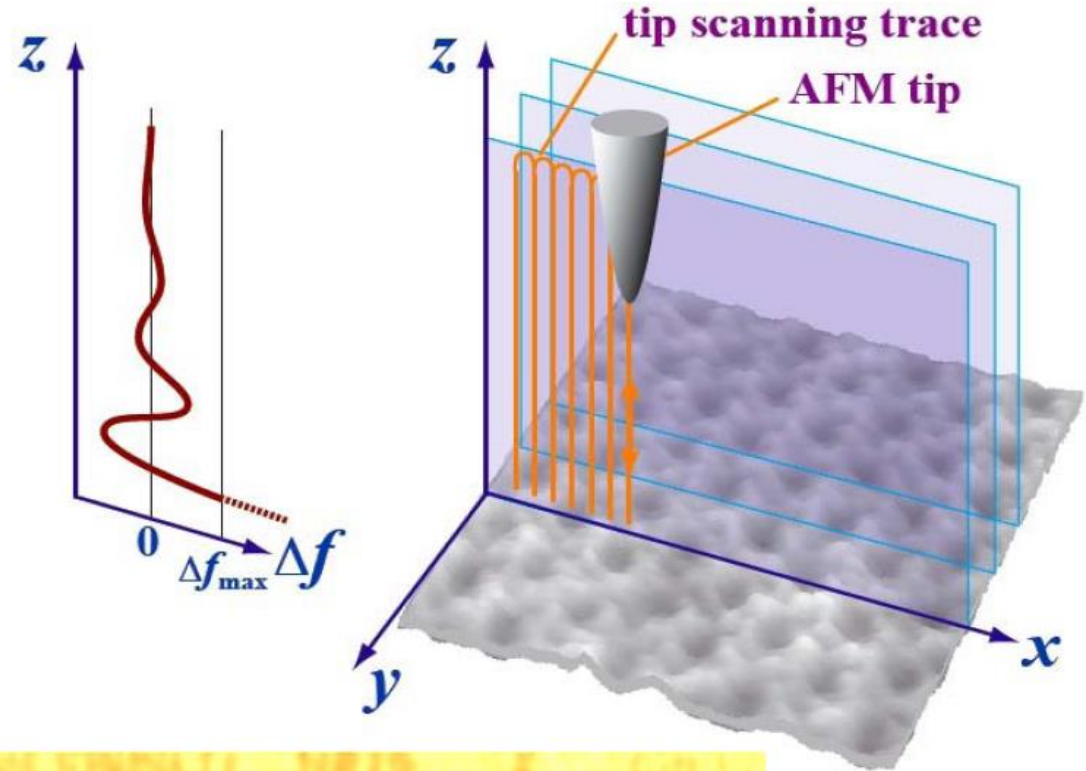
# Water Structure at Interfaces

## Structure of water

### Hydration structure



$\Delta f$  vs distance curve



# Nano Search Microscope OLS4500

From millimeter to nanometer size observation in one.

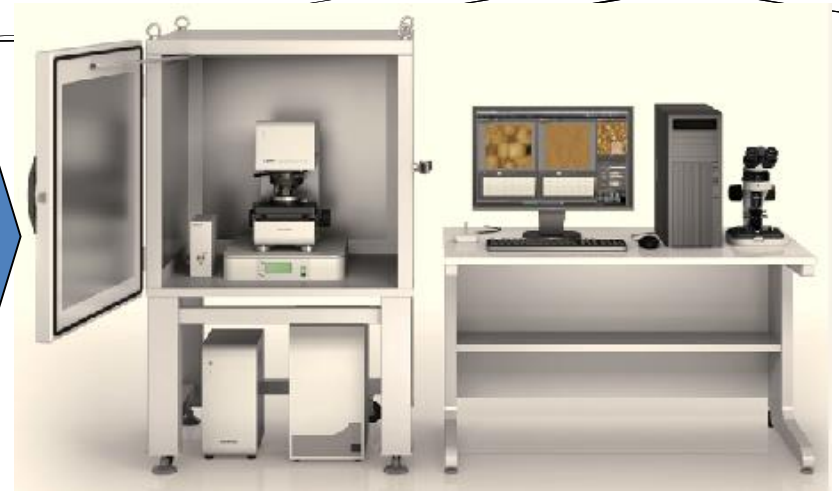
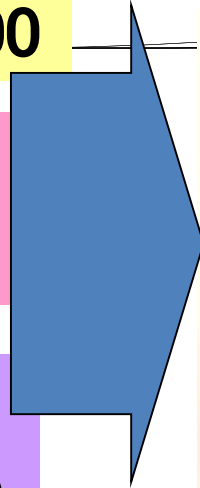


# Nano-Search Microscope

Optical Microscope :  $\times 100 \sim \times 2000$

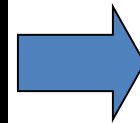
Laser Scanning Microscope (LSM)  
Magnification :  $\times 100 \sim \times 14000$

Atomic Force Microscope (AFM)  
Magnification :  $\times 1000 \sim \times 1000000$



Hybrid Microscope

Coaxial Object lens and AFM Tip



From millimeter to nanometer  
size observation in one.

Never miss the destination.  
LSM  $\longleftrightarrow$  AFM

In Air  
No Coating

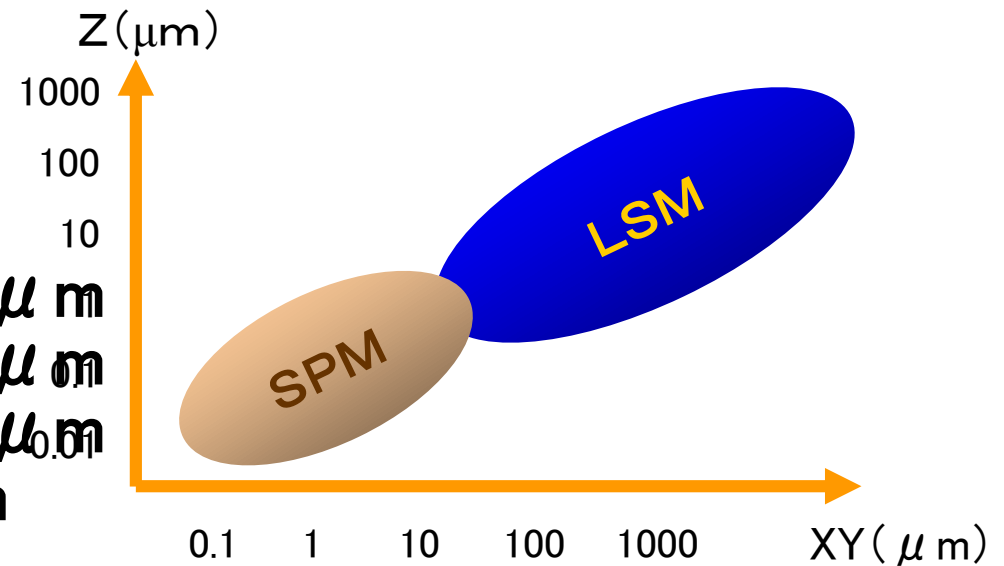


# From mm to nm size observation in one

- Continuous Observation

- Objective Lens

- 5x : 2600  $\mu\text{m}$  ~ 430  $\mu\text{m}$
    - 20x : 640  $\mu\text{m}$  ~ 110  $\mu\text{m}$
    - 100x : 130  $\mu\text{m}$  ~ 20  $\mu\text{m}$
    - SPM : 30  $\mu\text{m}$  ~ sub  $\mu\text{m}$



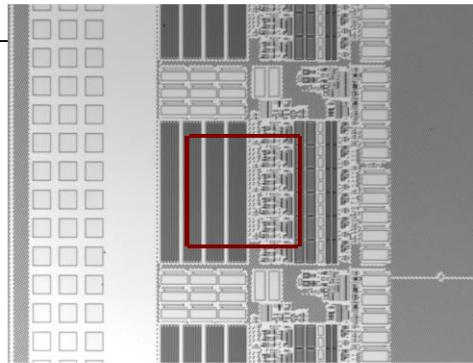
- Object lens and AFM Tip is coaxial

- **SPM Tip Position is strictly inside in 100X Objective Sight.**

# Wafer (Mag.: 200x ~ 1,000,000x)

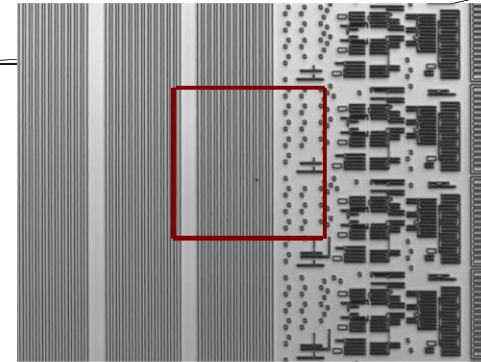
256 × 192 μm

LSM



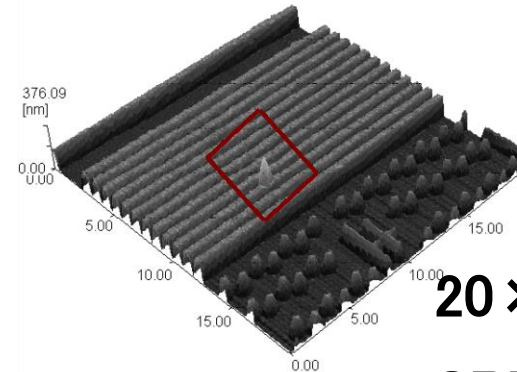
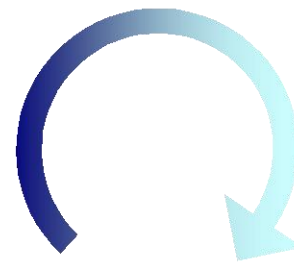
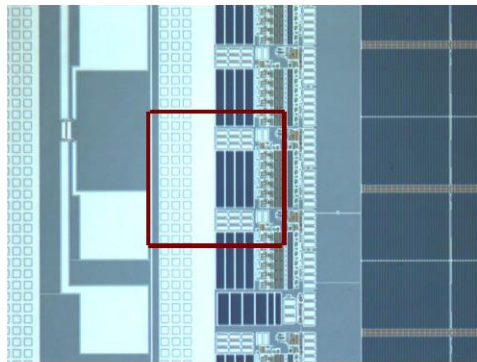
64 × 48 μm

LSM



640 × 480 μm

OM Image



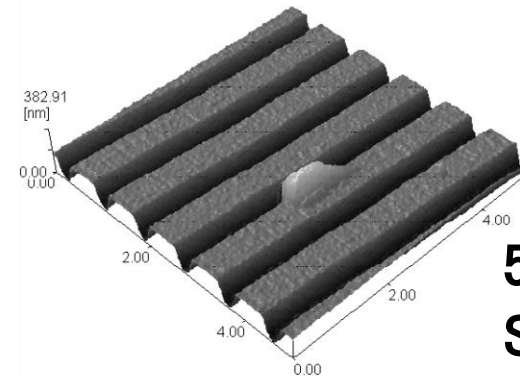
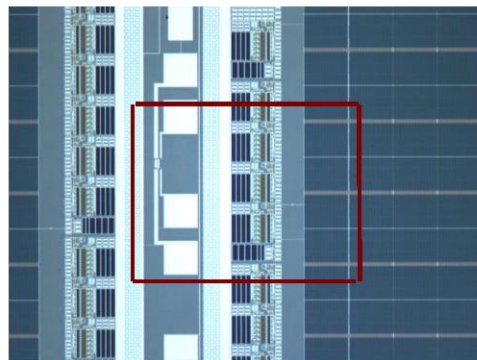
20 × 20 μm

SPM

20.00 x 20.00 [μm] Z 0.00 - 376.09 [nm]  
ウエハ上異物 測定範囲: 20 μm □

1280 × 960 μm

OM Image

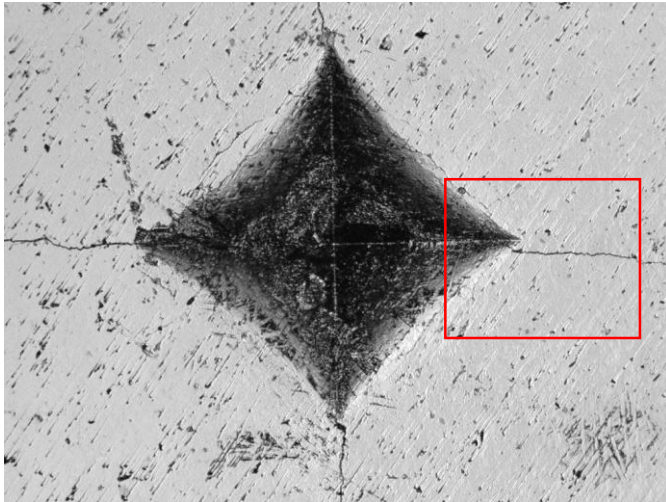


5 × 5 μm

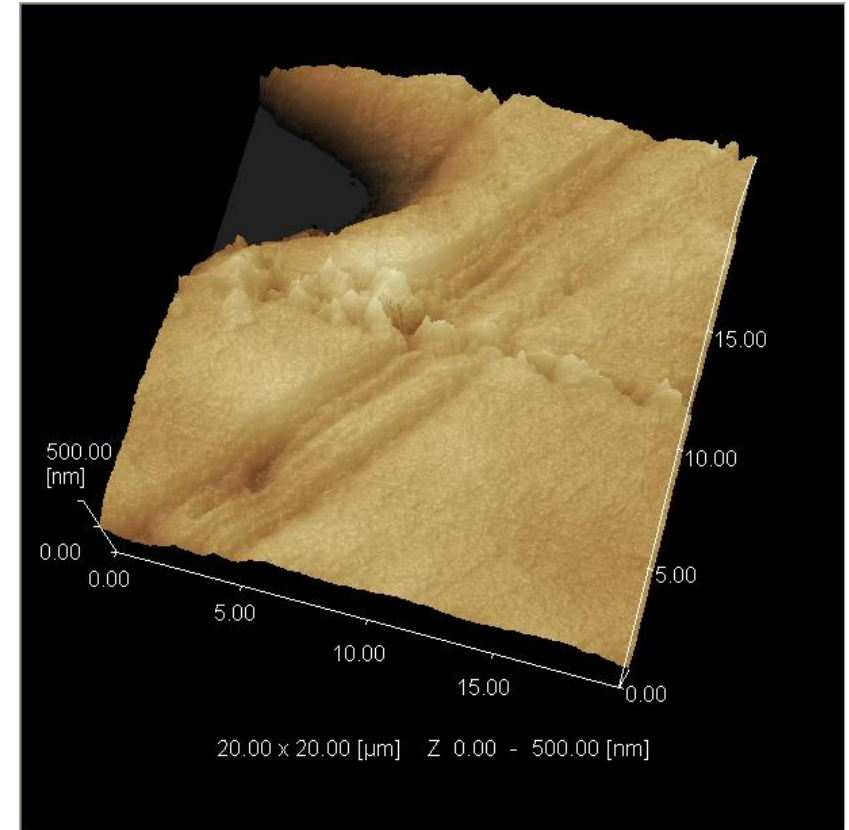
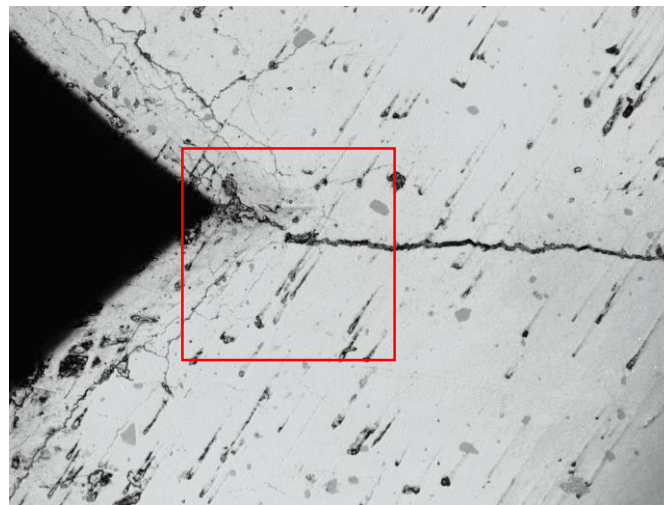
SPM

5.00 x 5.00 [μm] Z 0.00 - 382.91 [nm]  
ウエハ上異物 測定範囲: 5 μm □

# A mark of Micro Vickers



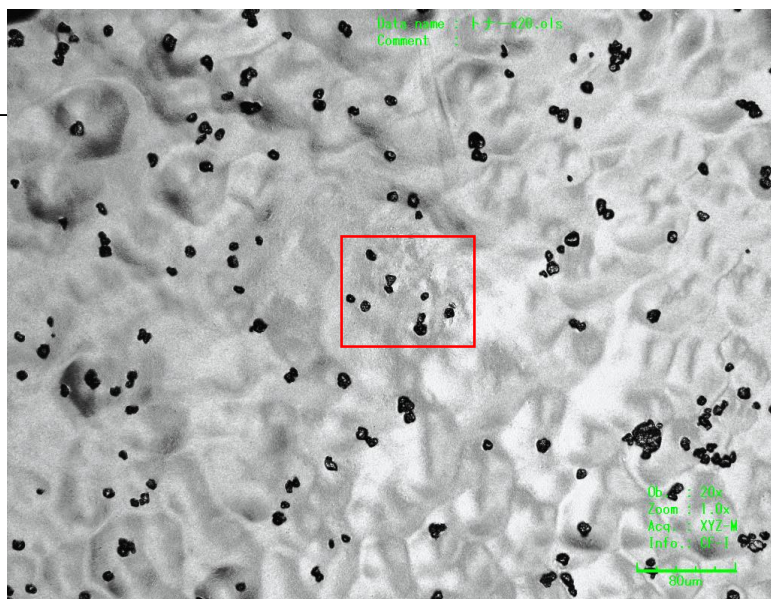
LSM Image  
 $400 \times 300 \mu\text{m}$  ↑  
 $64 \times 48 \mu\text{m}$  →



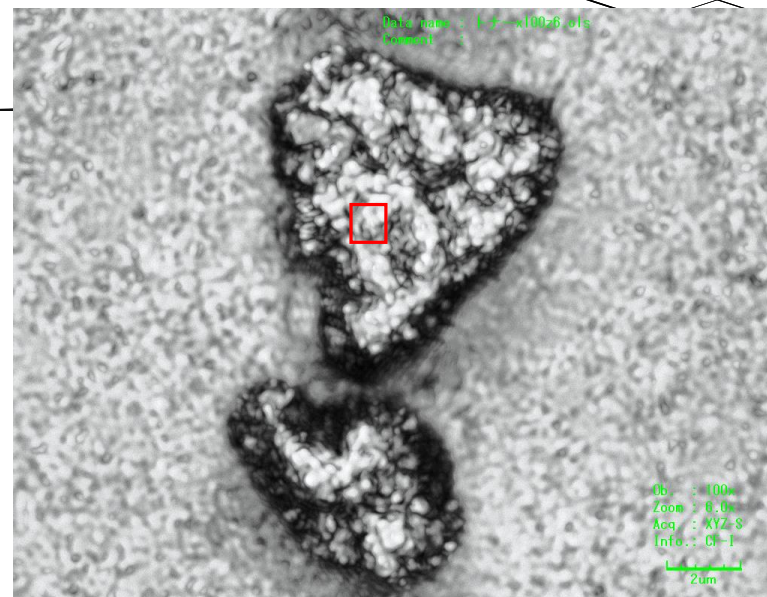
SPM Image  $20 \times 20 \mu\text{m}$  3D

**The change in height that built on both sides of the crack can be measured.**

# Toner



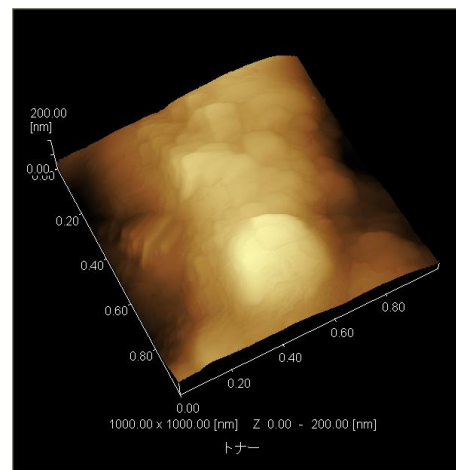
LSM 640 × 480µm



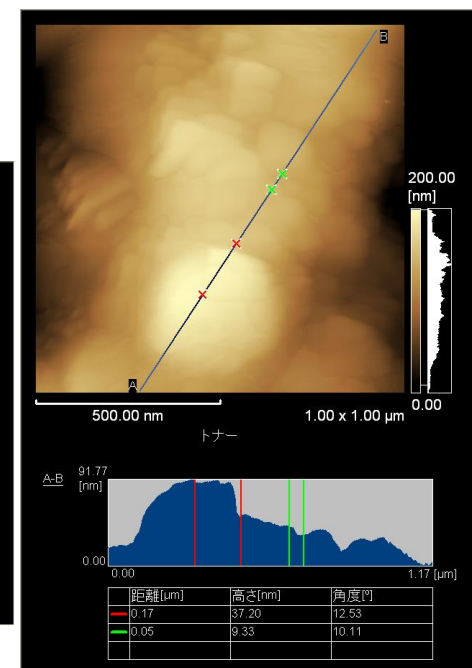
LSM 21 × 16µm



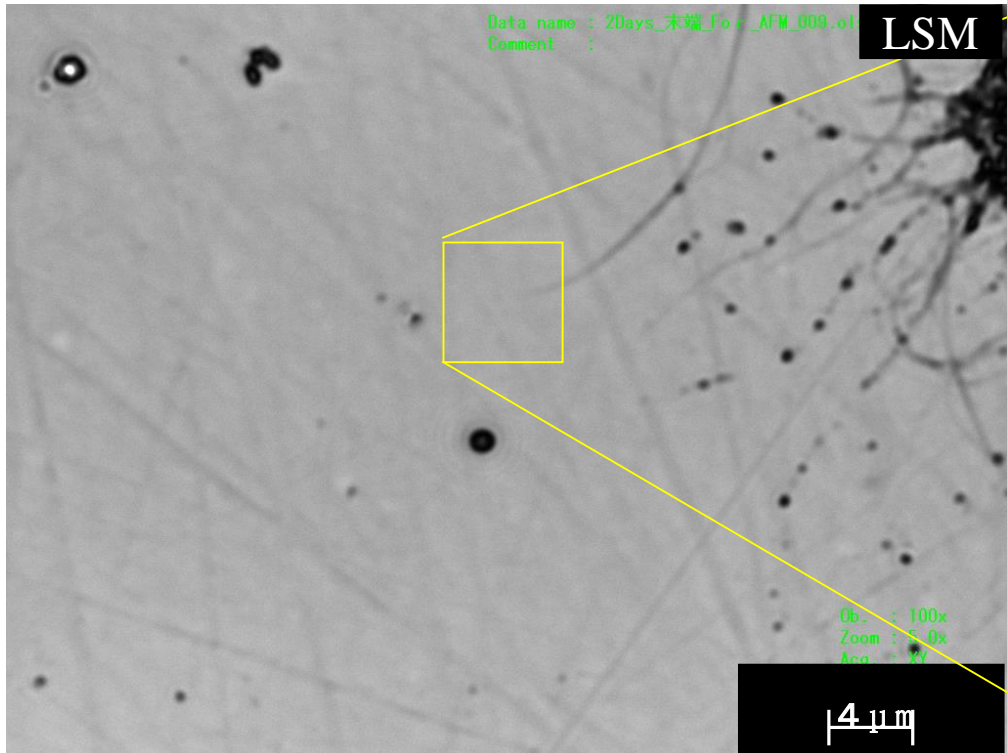
LSM 128 × 96µm



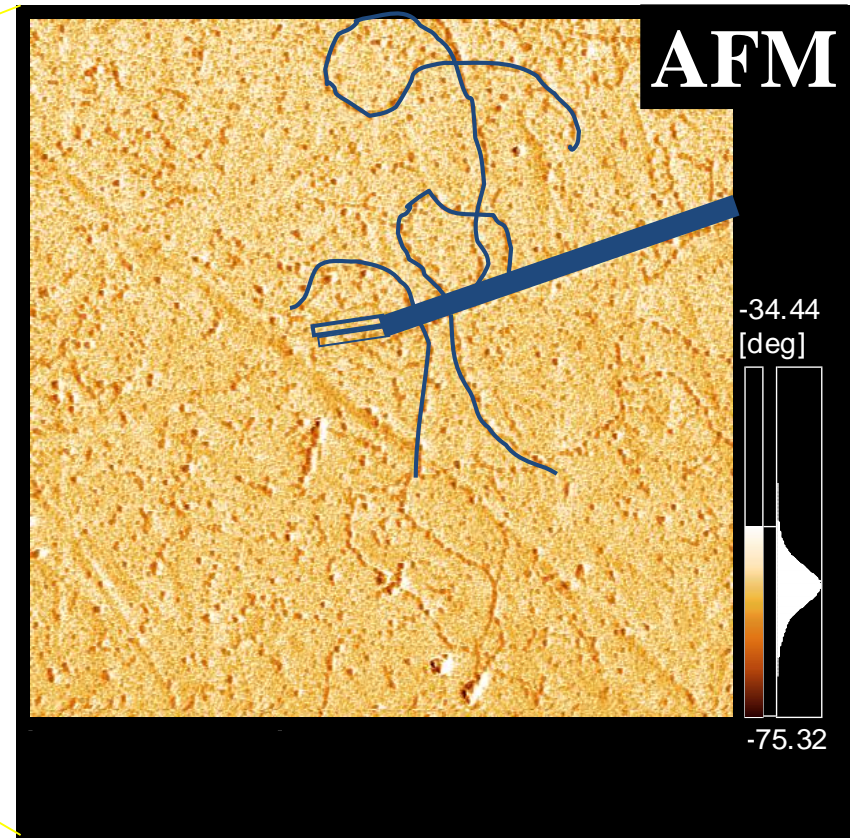
SPM 1 × 1µm



# Observation of Collagen End



Magnification x5000



Mag. x25000

**A thinner fiber can observe at the collagen end.**

# Material Characterisation Tools

## Elemental Analysis

AA AA+GF ICPS ICPMS

EDX XRF EPMA XPS

## Compound Analysis

GC LC GCMS LCMS MSMS

# Material Characterisation Tools

## Structural Analysis

XRD FTIR SALD DTG TGA

MALDI XPS

## Imaging

SPM FMSPM Nanoserach

# Material Characterisation Tools

## Failure Analysis

Servo hydraulic machines

Universal Testing Machines

Hardness Testers

X-Ray Inspection

**XPS**   **EPMA**   **FTIR-Microscope**



Shimadzu Provides ALL

THANK YOU

Mahendra Chaudhari