

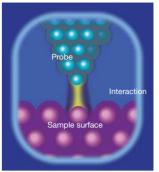






## **Principle of Scanning Probe Microscope**

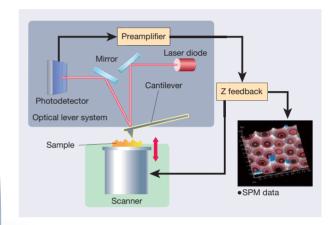
Scanning Probe Microscope (SPM)
represents a particular family of advanced
microscopy techniques that allow the
simultaneous measurements of surface
topography as well as a wide variety of
material properties at nanometer scale.
The two key members in this family
are Scanning Tunneling Microscope
(STM) and Atomic Force
Microscope (AFM).



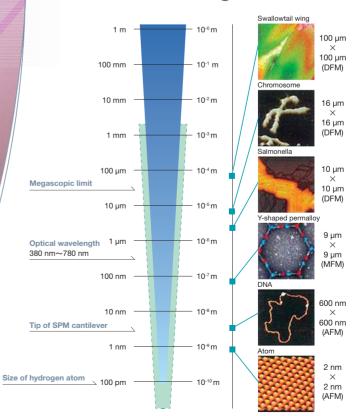
Schematic Diagram of SPM

# AFM5300E 815

**Environment Control Unit** 



## **Observable Range of SPM**





# GUI (Graphical User Interface)

Intuitive and logical control icons balance the screen layout with information provided for ultraefficient, productive, and easy SPM operation for all levels of users.



Analysis



Mode Selector



Operation Instructions

Measurement and analysis tabs provide organized and spacious work areas on the monitor display.



## Auto Tuning Functions for Optimal Measurement Parameters

The improved auto tuning function systematically and efficiently monitors sample topography, scanning area, the cantilever, and the scanner to determine the best operating conditions. As the measurement parameters are optimized, the cantilever's vibration amplitude and operation frequency are automatically adjusted based on the sample and cantilever type. The new and improved auto tuning algorithm offers reliable and precise images with a simple point-and-click!

#### **One-click automatic measurement**

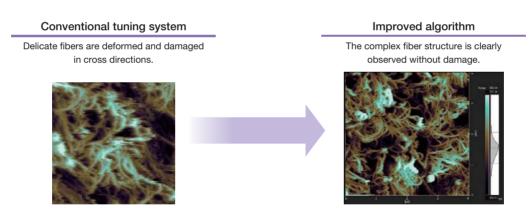


#### **Automatic tuning function examples**



#### Fibrous carbon nano-tube structure

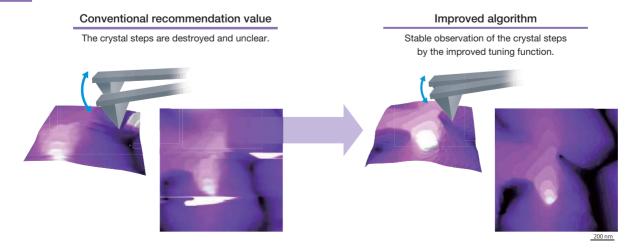
(Gecko adhesive tape, sample generously provided by Nitto Denko Corporation)



Example 2

#### Polycrystalline organic thin-film transistor

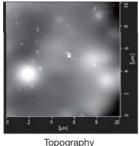
(Polycrystalline pentacene thin film, sample generously provided by Kitamura Laboratory, Kobe University)

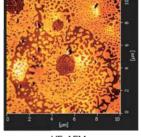


# Various Analysis Functions

#### 3D overlay

The 3D overlay image of an oil film on a polyethylene sheet's topography and VE-AFM (Viscoelastic-AFM).







Topography

VE-AFM

3D overlay image



#### Roughness and cross-section profile analysis

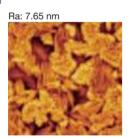
The 3D Overlay Function enables the observation of "cause and effect relationship" between topography and physical properties. A variety of other functions, such as roughness and cross-section analysis, are also standard tools.

# Tip Calibration

Tip Calibration improves reliability and validity of obtained data, since the resolution of SPM data greatly depends on the sharpness of the tip. When the tip is deformed or contaminated, its shape affects the result of topographic image.

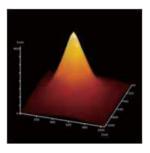


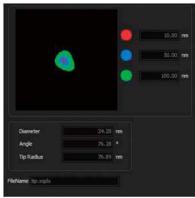
### ITO thin films scanned with a new probe (left) and worn out probe (right)



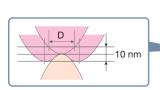


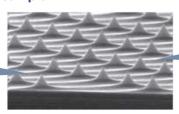
#### Tip calibration result

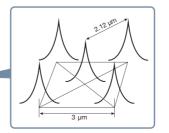




#### Tip calibration standard sample







## Q Control

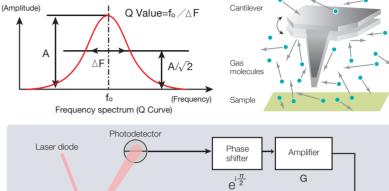


- High sensitivity measurement both in air and vacuum
- High speed response in vacuum
- Enhanced force measurement in solution

Sharpness of the frequency spectrum for an oscillating cantilever can be characterized and quantified by the Q factor, which is proportional to the sensitivity of the force gradient detection. Higher Q values imply an enhanced sensitivity to the variations of the tip-to-surface forces as well as a faster response of the system. Typically, the hydrodynamic damping with the medium reduces the Q. Therefore, Q control can be used to improve the performances in a medium.

Q control uses a phase shifter and amplifier to regulate the actual oscillation of a cantilever.

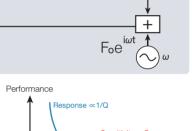
The cantilever signal is amplified, phase shifted by  $\pi/2$ , and then feedback for cantilever excitations, thus to modify its vibration. When the amplification factor "G" is positive, it leads to an increase of the Q factor.



Piezoelectric device

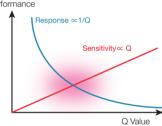
Cantileve

Z(t) (



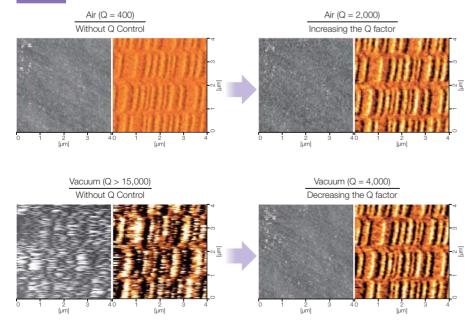
Q Control balances both sensitivity and responsiveness.

Measurements under vacuum are best suited for electromagnetic modes and avoiding the influence of adsorbed water. However, when the Q factor is too high, it reduces the responsiveness (stability).



# Example 1

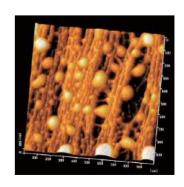
#### MFM observation of magnetic recording on a hard disk





# **DFM** observation of hollow fiber membrane in liquid

Showing below is a DFM topography image of a hollow fiber membrane that is widely involved in medical applications. The surface contains some very soft hydrated film protrusions. Despite the extreme challenges, such delicate and fragile features can be resolved clearly by gentle imaging with enhanced force detection via effective Q control.



# Measurement Modes

## Topography

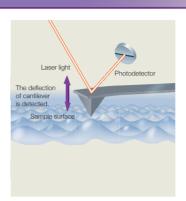
# Atomic Force Microscope (AFM) / Contact Mode



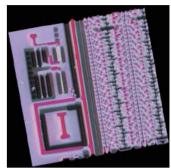




For contact mode AFM, the force between probe and sample is detected and measured via cantilever's deflection. A feedback system will maintain this deflection constant while scanning the sample surface to observe topography.



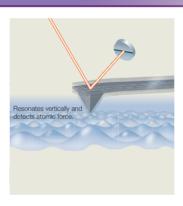
Semiconductor circuit



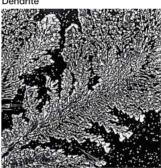
Scan area: 100 µm

## Dynamic Force Microscope (DFM)

For DFM, the cantilever is oscillating while it approaches to the sample surface. The force between probe and sample is reflected by cantilever amplitude change and maintained to be constant while scanning and observing the sample surface.



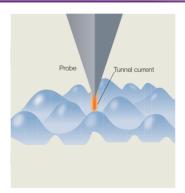
Dendrite

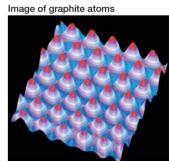


Scan area: 30 µm

## Scanning Tunneling Microscope (STM)

A tunnel current flowing between probe and sample is detected (controlled so that the tunnel current is fixed and sample surface is scanned) by applying a bias voltage between a metallic probe and conductive or semiconductive sample as the distance between them approaches less than several nm. Sample topography as well as its electronic state are observed.





Scan area: 1.3 nm











## Sampling Intelligent Scan (SIS)









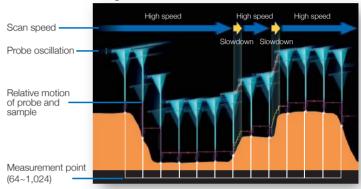
In SIS mode, probe approaches each measurement point and acquires topography and physical property information, followed by retracting from the sample and moving to the next measurement point. It is an intelligent measurement mode that can adjust the scan speed according to sample surface. SIS solved problems occurred in conventional SPM by reducing the lateral tip/sample interactions. This enables stabilized measurements, particularly on soft and adhesive samples as well as samples with high aspect ratio structures.

When used in the Current Mode for a soft material, SIS allows the stable acquisition of topographic image without damaging the sample. SIS is also effective in the Phase Mode (PM): SIS-PM eliminates the effects of sample topography, which may cause artifacts in PM imaging.

#### Conceptual diagram of SIS-Topo\* scanning movements

Probe and sample make contact only when getting data. Aside from this, scanning speed drops when moving at high speeds in the horizontal direction while on stand by in mid-air and when in contact with the sample surface; and shelter motions that rise from the sample surface are automatically performed.

#### Image of SIS mode

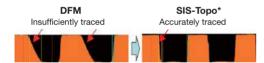


#### SIS-Topo\* Examples

#### Measuring deep trenches



Comparison between DFM and SIS-Topo: Measuring deep trenches by using conventional DFM cantilevers

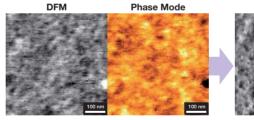


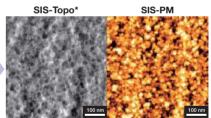
SIS-Topo\* eliminates effects from horizontal direction and accurately scans trench walls.

#### Observation of adhesives



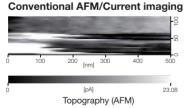
DFM cannot get a clear image of the phase or the topography of soft and adhesive surfaces; however, SIS-Topo clearly observes both topography and phase images.

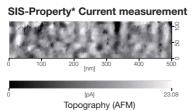


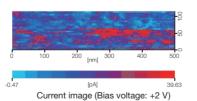


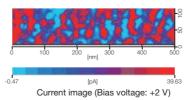
#### SIS-Property\* Example

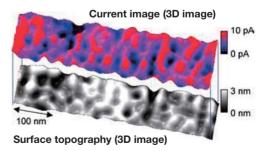












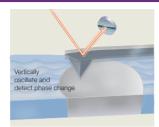
The conventional current and AFM modes deform samples, resulting in an unstable image reflected in the scan direction for current distribution. In contrast, SIS-Property\* (Current) performs stabilized measurements for both current and topographic images without deforming the sample.

\*SIS-Topo is for observing topography and phase mode, while SIS-Property is for mechanical and electromagnetic property measurements.

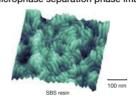
## Mechanical Property

### Phase Mode (PM)

The DFM measurement detects phase lag in oscillation of the cantilever depending on the size of adsorptive power or hardness and softness, and observes differences in physical properties of the sample surface.



Microphase separation phase image

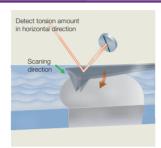


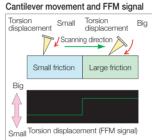
Polymer blend phase image



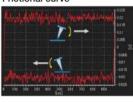
## Friction $\mathsf{F}$ orce $\mathsf{M}$ icroscope (FFM)

The sample is scanned in the direction in which the cantilever is twisted. Frictional force, occurring between probe and sample, is converted into cantilever's torsion which is simultaneously detected as frictional and topographic images.



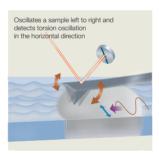


Frictional curve

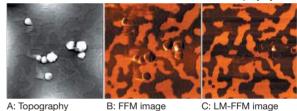


## ateral Modulation FFM (LM-FFM)

Friction image that does not rely on surface unevenness or scan direction by adding micro oscillations in the horizontal direction (direction of cantilever deflection) to the sample is observed and the torsion oscillation of the cantilever is detected.



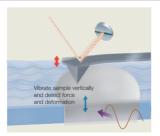
Friction distribution measurement of silicon oil film on polystyrene

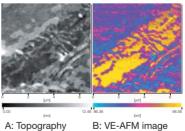


Friction of oil film is less, resulting in dark contrast in FFM and LM-FFM images.

## isco Elastic AFM (VE-AFM) / Force Modulation Microscope

VE-AFM observes viscoelasticity image by adding vertical micro-oscillations in a sample and detects deflection oscillations (oscillation in the vertical direction) of the cantilever that change by differences in surface viscoelasticity.





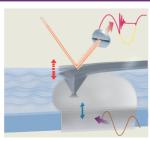
Viscoelasticity distribution measurement of dispersed coating on a polyethylene film

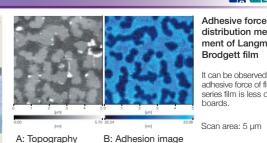
The polystyrene substate is harder than the coating.

Scan area: 7 µm

## **Adhesion**

It micro-oscillates the sample in the vertical direction; repeats the movement that the probe and sample contact and separate periodically; detects the deflection of the cantilever at the moment that the probe is separated from the sample; and observes adsorption power distribution.





distribution measurement of Langmuir-Brodgett film

It can be observed that adhesive force of fluorine series film is less on Si

Scan area: 5 um











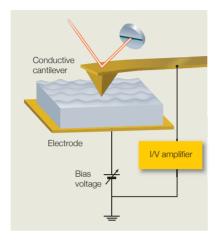


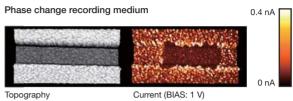
## **Electromagnetic Property**

## **Current/Pico-current**



Scans in the horizontal direction with a bias voltage applied to the sample, detects the current that flows between probe and sample, and observes current distribution.

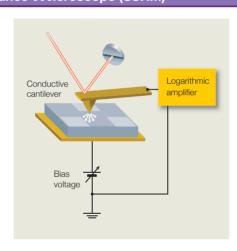


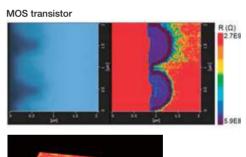


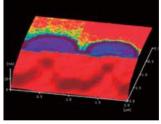
# Scanning Spread Resistance Microscope (SSRM)



Local resistance distribution on the sample surface at the wide range amplifier greater than 6th order is observed by using a hard cantilever of high conductivity and measuring the micro-current at the contact position with the probe by applying a bias voltage to the sample. The practical semiconductor dopant concentration range is sufficiently covered.





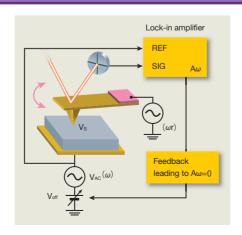


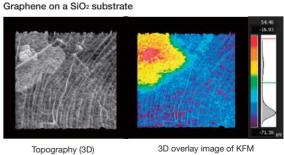
## ${\sf K}$ elvin Probe ${\sf F}$ orce ${\sf M}$ icroscope (KFM)



Measures the surface potential by the feedback DC voltage by applying AC as well as DC voltages between a conductive cantilever and sample so that the amplitude of the static force component from the AC voltage is zero.

- ·CC-KFM (Cyclic contact KFM)
- •NC-KFM (Non-contact KFM)





and Topography

## **Electromagnetic Property**

## Electrostatic Force Microscope (EFM)

Applies an AC or DC voltage between a conductive cantilever and sample and creates an image of the electrostatic force components (amplitude component and phase component) by an AC voltage.

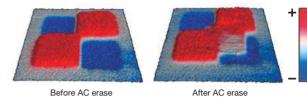
- •EFM (AC)...AC field response
- •EFM (DC)...DC field response

KFM directly detects potential of sample surfaces.

EFM does not directly detect surface potential but has better responsiveness than KFM and is convenient for imaging qualitative electrical properties.



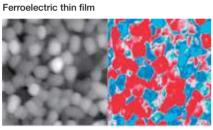
#### Ferroelectric polarization patterns



## Piezo-Response Microscope (PRM)

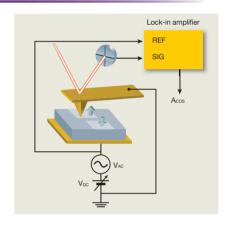


Applies an AC current between the probe and sample, and by scanning, observes strain distribution of the sample while detecting the ferroelectric strain component.



PRM

3D overlay image

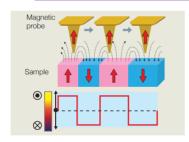


## Magnetic Force Microscope (MFM) [Received the Magnetic Society of Japan Best Paper Award (2003 & 2005)]

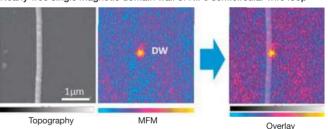


A magnetic action between magnetic probe and sample is produced and imaged as potential changes in cantilever oscillation. High sensitivity and high resolution magnetic domain imaging is possible by measuring in vacuum.

Topography

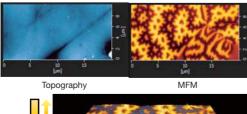


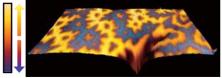
#### Nearly-free single magnetic domain wall of NiFe semicircular wire loop



Joint research with Keio University Nature 432 (2004) 203-206

#### Magnetic domains of a theramally demagnetized Nd-Fe-B sintered magnet

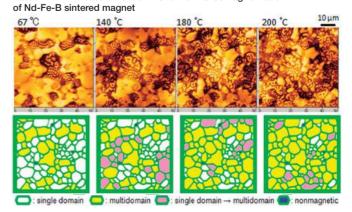




3D overlay image

Sample generously provided by Kobayashi Laboratory, Shizuoka Institute of Science & Technology

Vacuum MFM measurement of in situ thermal demagnetization









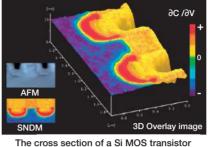


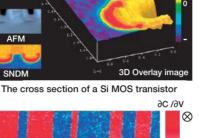


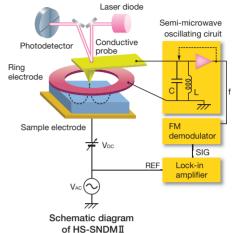
## f High f Sensitivity f Scanning f Nonlinear f Dielectric f Microscope f I (HS-SNDMf I)

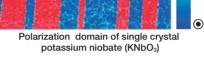


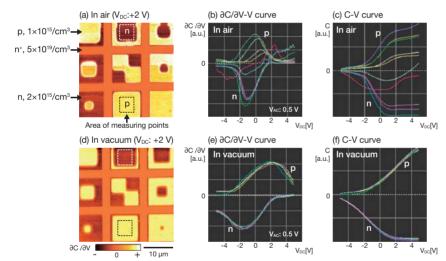
Scanning nonlinear dielectric microcopy (SNDM) is a Hitachi licensed AFM technique for 2D dopant profiling of semiconductor devices and characterization of ferroelectric materials. Similar to the well-known scanning capacitance microscopy (SCM) that is equipped with a GHz resonant capacitance sensor, SNDM can measure the first order capacitance variation in response to an applied AC voltage. However, SNDM instrumentation is with a special design to reduce undesired stray capacitance and boosts the detection of local tip/sample interactions. Therefore, SNDM exhibits a much higher sensitivity to capacitance measurements than SCM. When combined with Hitachi high vacuum and environmental control AFM5300E, SNDM measurements with further enhanced sensitivity, accuracy, and repeatability can be achieved. For instance, it has been verified that vacuum SNDM can resolve domains with doping concentration down to the level of 1013 atom/cm3.











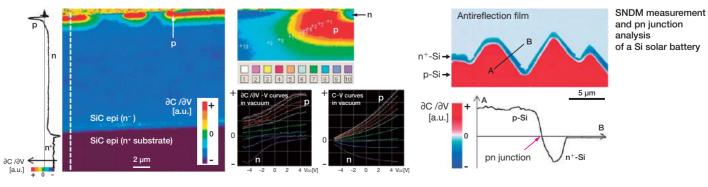
#### An example showing the benefits of SNDM technique with environmental control

Vacuum SNDM eliminates the interference of adsorbed water on fabricated p-n structures and enables more stable and better  $\partial C/\partial V$ -V curve and C-V curve measurements than the ones acquired in air.

The small n-type spot appeared on the vacuum SNDM image (d) was not observed in the SDNM image in air (a).  $\partial C/\partial V$ -V curve and C-V curve (e, f) were separated in accordance with two different concentration levels of p-type and n-type in vacuum. In comparison, curves acquired in air (b,c) are inconsistent.

#### Vacuum SNDM measurements of a SiC Power MOS FET and C-V analysis near its pn junction

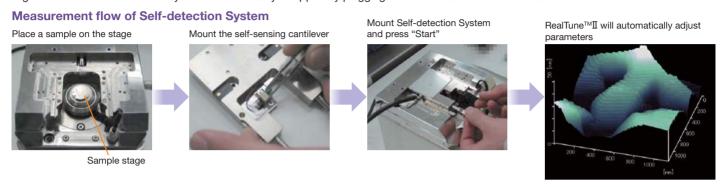
Carrier concentration mapping of a SiC Power MOS FET in accordance with the device structure was obtained using vacuum SNDM. Throughout the 10 points of C-V curve measurements across from p to n regions, the changes in the depletion layer is clearly obtained in response to concentration and domain. The SNDM image and its signal cross section of a Si solar battery clearly indicates the location of the pn junction.



\*We have received guidance from Professor Cho at Tohoku University, the inventor of SNDM, to develop SNDM as a product.

## **Detection System**

The AFM5100N offers both Optical Lever System and Self-detection System. Optical Lever System supports electromagnetic and mechanical modes and is compatible with environmental controls, such as in-liquid and temperature control. You can achieve multiple measurements without exchanging the cantilever holder, since it can cover all measurements mode except STM and in-liquid imaging. Self-detection System simplifies difficult SPM operations. Its self-sensing cantilever has a sensor on itself, therefore this detection method does not require laser alignment. These two detection systems can be easily swapped by plugging in/out their cables to the main SPM unit.



The self-sensing cantilever has a piezoresistive sensor, assembled by MEMS technology. The cantilever can be easily exchanged, since it is mounted on a substrate, which makes it easy to grip the cantilever. The cantilever bends from a force that acts on the probe, changing the resistance of the piezoresistive sensor in the narrow part of the cantilever. Cantilever's deflection changes the resistance, which is detected by the bridge circuit together with the resistance of the temperature compensator dummy resistor.

## Optical Microscope

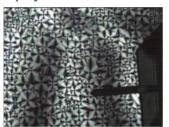
#### **Metallurgical Microscope**

Metallurgical Microscope enables precise positioning of a cantilever.

#### **Crystallinity in polymer**

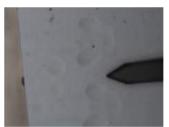


Optical microscope (Epi-illumination)



Metallurgical microscope (Polarized light)

#### Patterns on a silicon wafer



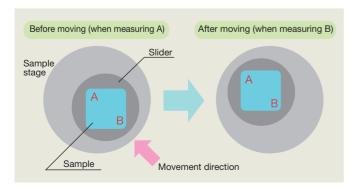
Optical microscope (Epi-illumination)

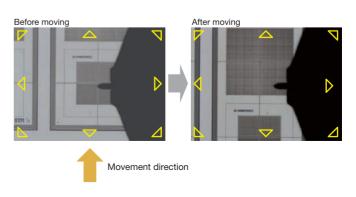


Metallurgical microscope (Polarized light)

## Impact Stage (optional)

The impact stage is a function that can easily change measurement positions by the operation on the screen display. This greatly improves operability for measuring multiple locations on the same sample.



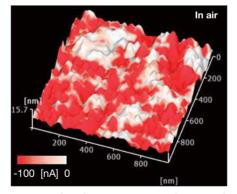


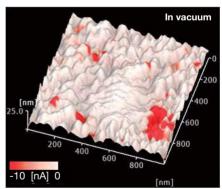


# High Vacuum System

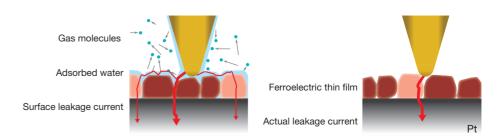
AFM5300E's vacuum chamber meets the demand of advanced environment control needs. Some advanced materials and precise measurements of electromagnetic properties require a vacuum environment where adsorbed water and gas molecules are reduced.

Leakage current observations of a ferroelectric thin film on the right is an example of how vacuum environment can enhance the accuracy of current measurements.





Leakage current observations of a ferroelectric thin film

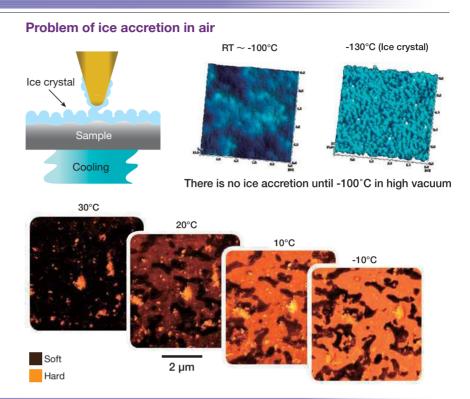


## **Temperature Control**

When the temperature in air falls below the dew point, ice will form on the surface. Even with a dried gas that has as little water vapor as possible, ice gradually forms when cooled below 0°C. In a vacuum environment created by the AFM5300E's turbo molecular pump, changes in topography and physical properties can be investigated while cooling down the sample to -120°C.

Cooling of polymer samples can also reveal unique characteristics of polymer nanostructures.

Phase images on the right show thermal behavior of blended polymer at different temperatures. There is no distinctive differences between natural and synthetic rubbers. At -10°C, however, the natural rubber becomes harder than artificial one which visualize the distribution.



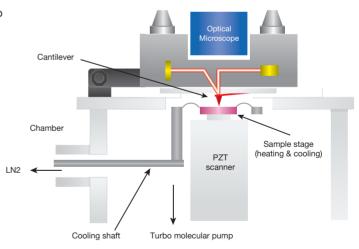
## Simple and Easy Operation

A tool-free open-close mechanism is employed. It is not necessary to align the laser after removing and mounting another sample and to exchange the cantilever holder when switching the measurement modes.\*

\*Except STM, SNDM, and in-liquid imaging.

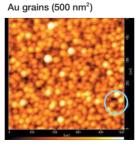


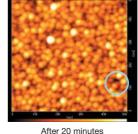


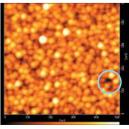


# Established Excellent High Performance

Swing Cancellation Mechanism achieved drastic reduction of drift. This mechanism improves and stabilizes SPM data.







Right after approaching

utes After 40 minutes

# In-situ Observations Optional Accessories

The AFM5300E supports various environments including in air, vacuum, liquid, temperature control, and humidity control.

In Liquid

Unlike most conventional UHV systems, AFM53000E supports in-liquid imaging by mounting the cantilever holder expansion flange and exchanging the cantilever holder.



# **Control**

Temperature AFM5300E enables wide range of temperature control from -120°C to 300°C/ from room temperature to 800°C. Its vacuum system helps temperature control to be stable.



Temperature Controller



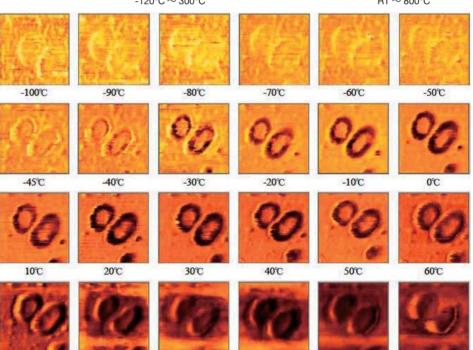
Heating and Cooling Temperature Control Module -120°C ∼ 300°C



High Temperature Sample Stage  $RT \sim 800$ °C

VE-AFM image of polypropylene block copolymer

Soft Hard



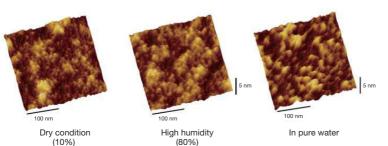
100℃

### **Humidity** Control

Observations of solid polymer electrolyte membrane for fuel cell in dry condition, high humidity and liquid.

90℃

80°C



70℃



120℃

110℃

## Air Protection Sample Holder Unit

Some materials are prone to oxidation or changing chemical/physical state under atmospheric conditions. The AFM5300E with optional Air Protection Sample Holder Unit offers imaging without exposure to the atmosphere. This unit has a vacuum enclosure that is sealed in situ allowing for safe and easy transfer to the





Sample Holder

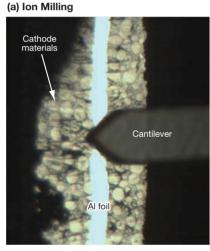
SEM\*/ the Ion Milling System\*\* within an inert or vacuum environment.

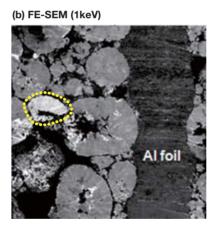
- \*Compatible with Hitachi FE-SEM with Air Protection Specimen Exchange Chamber.
- \*\*Compatible with Hitachi IM4000 Ion milling System with Air Protection Holder Unit.

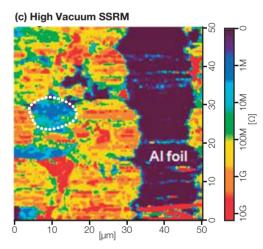


#### Observation of the same milled area of Li-ion battery cathode materials with FE-SEM and SSRM

- The electric resistance distribution of highly conductive Al foil and various cathode materials (micro-cathode active materials, a conductive assistant, and resin binders) on both sides are clearly observed in the SSRM image.
- The correlation between SSRM and FE-SEM images is shown. The active material, indicated by the dotted circles on (b) and (c), exhibits a brighter contrast on the SEM image and lower resistance on the SSRM image.
- The image (a) is captured by the metallurgical microscope's video camera attached to the AFM5300E. The top-view and high-magnification optical microscope significantly enhances the ease of use and enables SEM-SPM measurements of the same area.









# Specifications



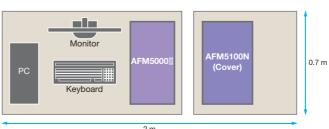
# Probe Station AFM5000II

	AFM5000Ⅱ			
os	Windows®7			
Compatible Units	AFM5100N AFM5300E			
RealTune™ II	Automatic tuning of cantilever amplitude (DFM), contact force, scan speed, and feedback gains (Various tuning modes including Auto, Fast, Soft, Rough, Flat, and Point)			
Various Functions	Operating instructions; Tab structure (Measurement/ Analysis);  Measurment area indicator/ Measurement area tracking window; Batch processing; and Tip calibration			
Operating Voltage	XY (±200 V/18 bit) Z (±200 V/26 bit)			
Multi Channel (Data Points)	4 channels (max. 2,048 × 2,048) 2 channels (max. 4,096 × 4,096)			
Rectangular Scan	1:1, 2:1, 4:1, 8:1, 16:1, 32:1, 64:1, 128:1, 256:1, 512:1, 1,024:1			
Analysis Software	3D display and overlay, Roughness, Cross-section, Average cross-section			
Size	220 mm(W) × 500 mm(D) × 385 mm(H), approximately 15 kg			
Power Supply	AC 100 V ~ 240 V±10%			

### Installation layout

#### **AFM5100N**

(Floor model anti-vibration table)



# **AFM5100N**







	AFM5100N	AFM5100N Optional Accessories	AFM5300E	AFM5300E Optional Accessories
Manual Stage	XY ±2.5 mm	Impact Stage	XY ±2.5 mm	_
Sample Size	35 mm (diameter) Thickness: 10 mm	2 inch Adjustment Block 50.08 mm x 50.08 mm x 20 mm	25 mm (diameter) Thickness: 10 mm	_
Scan Range (Select at least one)	20 μm x 20 μm x 1.5 μm 100 μm x 100 μm x 15 μm 150 μm x 150 μm x 5 μm	Closed loop scanner 110 µm x 110 µm x 6 µm	20 μm x 20 μm x 1.5 μm	100 μm x 100 μm x 15 μm 150 μm x 150 μm x 5 μm Closed loop scanner XY: 15 μm
Detection	Self-Detection/ Optical Lever (Select at least one)	_	Optical Lever (Low-coherence light)	
Optical Microscope (Select at least one)	Optical Microscope (Lens magnification: x4) Desktop Zoom Microscope (Lens magnification: x7) Zoom Microscope (Lens magnification: x7) Metallurgical Microscope (Lens magnification: x5, x20, x50)		Zoom Microscope (Lens magnification: x7) Metallurgical Microscope (Lens magnification: x5, x20, x50)	
Anti-vibration	Desktop Floor model (Select at least one or supply equivalent table top vibration isolation.)		Floor model  (The main AFM unit is integrated with the vibration isolation table.)	
Temperature Control	_	In air: RT $\sim$ 250°C In liquid: RT $\sim$ 60°C	-	-120°C $\sim$ 300°C RT $\sim$ 800°C In liquid: RT $\sim$ 60°C
In Liquid	_	✓	_	✓
<b>Humidity Control</b>	_	_	_	✓20 ~ 80%RH
Vacuum	_	_	-	Turbo-molecular Pump & Rotary Pump (9.9 x 10 <sup>-5</sup> pa)

#### AFM5300E





#### **Science Ring**

This logo symbolizes Scientific and Analytical instruments of Hitachi High-Tech Group. It is composed with an "S", standing for "Science", our technology core competency, and with a ring that represents close connection we make with our customers. This "Science Ring" shows how we are committed to create new values by strengthening ties between Science and Society.

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  - System specifications are subject to change without notice.
    - The PC monitor shows composite images
    - "Windows" is a registered trademark of Microsoft Corporation in the United States and other countries.
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