



Scanning Probe Microscope





Probe Station AFM5000II

Control System and Software P3 Measurement Modes P7



AFM5000 Series



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



Observable Range of SPM



Environment Control Unit

-



GUI (Graphical User Interface)

Intuitive and logical control icons balance the screen layout

Analysis





Operation Instructions

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

3

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)

Conventional tuning system

Delicate fibers are deformed and damaged in cross directions.



Improved algorithm

The complex fiber structure is clearly observed without damage.





Polycrystalline organic thin-film transistor

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

Conventional recommendation value

The crystal steps are destroyed and unclear.

Improved algorithm

Stable observation of the crystal steps by the improved tuning function.







Various Analysis Functions

3D overlay

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





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 standard sample





Tip calibration result







Q Control

Benefits

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.

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).





Cantileve

Performance Response ∝1/Q Sensitivity∝ Q

Q Value

Example 2

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.

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.

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.

Laser ligh The deflection f cantiles Indat

Semiconductor circuit







Scan area: 30 µm





Scan area: 1.3 nm





Probe

Tunnel current



💦 Q Control 🛒 🦳 In liquid 🕅 In vacuum

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.



SIS-Topo* Examples

Measuring deep trenches



SIS-Topo: Measuring deep trenches by using conventional DFM



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

SIS-Property* Example



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-PM

10 pA

fl nA

3 nm

0.000

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



Frictional curve

riction Force Microscope (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.



Cantilever movement and FFM signal Torsion Small Big displacement Scanning direction Small friction Large friction

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



A: Topography

Scan area: 2.5 um

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

C: LM-FFM image

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.





A: Topography B: VE-AFM image Viscoelasticity distribution measurement of dispersed coating on

a polyethylene film The polystyrene substate is

harder than the coating.

Scan area: 7 um

Adhesion

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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 boards

Scan area: 5 um

A: Topography

B: Adhesion image





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.



Phase change recording medium

Q Control In air In liquid In vacuum



Topography

Current (BIAS: 1 V)

Temperature control

0.4 nA

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.







Kelvin Probe Force Microscope (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)

Lock-in amplifier REF SIG (ωr) Feedback $V_{AC}(\omega)$ leading to $A\omega = 0$ Voff

Graphene on a SiO2 substrate



Topography (3D)

3D overlay image of KFM and Topography

Electromagnetic Property

Electrostatic Force Microscope (EFM)

Joint research with Keio University

Nature 432 (2004) 203-206

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



${\sf P}$ iezo- ${\sf R}$ esponse ${\sf M}$ icroscope (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.



Topography





AC erase



agnetic 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.



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



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



Topography



3D overlay image Sample generously provided by Kobayashi Laboratory, Shizuoka Institute of Science & Technology

Vacuum MFM measurement of in situ thermal demagnetization of Nd-Fe-B sintered magnet



🗔 single domán 🧔 maltidoman 🥮 single domain --- multidomain 👹 atomaignetic

High Sensitivity Scanning Nonlinear Dielectric Microscope II (HS-SNDMI)

Temperature control

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 strav 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.



💦 Q Control 🛛 🧖 In air

In liquid

The cross section of a Si MOS transistor



Polarization domain of single crystal potassium niobate (KNbO₃)



In vacuum



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.



Select your own configuration.



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.

Measurement flow of Self-detection System

Place a sample on the stage



Sample stage



Mount Self-detection System and press "Start"



RealTune[™]II will automatically adjust parameters



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**



Impact Stage (optional)

Optical microscope (Epi-illumination)

Metallurgical microscope (Polarized light)



Optical microscope (Epi-illumination)

Patterns on a silicon wafer



Metallurgical microscope (Polarized light)

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.









Environment Control Unit

The AFM5300E is a high vacuum scanning probe microscope which provides various measurement conditions, such as air, liquid, vacuum, humidity control, and temperature control. Its vacuum chamber enables clear and high resolution observations of electromagnetic properties and in-situ observations at high and low temperatures.

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.

Au grains (500 nm²)





Right after approaching

After 20 minutes

After 40 minutes



Problem of ice accretion in air

Sample

Coolina

Ice crystal





There is no ice accretion until -100°C in high vacuum

Versatility

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

VE-AFM image of polypropylene block copolymer









High Temperature Sample Stage $RT \sim 800^{\circ}C$



Humidity Control

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



Dry condition (10%)







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



*Compatible with Hitachi FE-SEM with Air Protection Specimen Exchange Chamber.

**Compatible with Hitachi IM4000 Ion milling System with Air Protection Holder Unit.

Example

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

- The electric resistance distribution of highly conductive AI 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.







Sample Holder

Specifications

Probe Station



	AFM5000II		
OS	Windows®10		
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) \times 500 mm(D) \times 385 mm(H), approximately 15 kg		
Power Supply	AC 100 V \sim 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		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
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 ~ 300°C RT ~ 800°C In liquid: RT ~ 60°C
In Liquid	-	\checkmark	-	\checkmark
Humidity Control	-	—	-	✓20~80%RH
Vacuum	_	-	-	Turbo-molecular Pump & Rotary Pump (9.9 x 10 ⁻⁵ pa)

AFM5300E



Hitachi High-Tech Science

In 2013, Hitachi High-Tech Science Corporation has made a new start as a member of the Hitachi High-Technologies Group.

Since opening its doors in the 1970s, Hitachi High-Tech Science has cultivated unique technologies

and produced numerous innovative analytical and measurement instruments.

Those products based on our high precision technologies are used in industrial fields that include electronic devices, environment, materials, and clean energy, as well as for research support at universities and research institutes.

Our aim as a member of the Hitachi High-Technologies Group is to be a successful enterprise, one trusted by our stakeholders, providing advanced products that satisfy market needs.

Hitachi High-Tech Science is actively pursuing development that skillfully balances scientific technologies with the global society.



History						
1070-	Hitachi High-Tech Science Corporate History	Hitachi High-Tech Science SPM Product History	History of SPM			
1970s -	1970 Daini Seikosha, current Seiko Instruments Inc., established an R&D Center and entered into the business of scientific instruments					
19805		 1985 STM research & development with the National Institute of Advanced Industrial Science and Technology (AIST) 1986 First AFM observation (NbSe₂) in Japan by AIST Image: State of Advanced Industrial Science and Technology (AIST) 1988 Commercialization of Japan's first STM (SAM3000) 	 1981 First STM observation of atoms by Dr. Binnig, Dr. Rohrer (IBM Zurich Laboratory) 1986 AFM development by Dr. Binnig (IBM), Dr. Quate (Stanford University) Dr. Binnig, Dr. Rohrer received the Noble Prize in Physics 			
1990s ←		1991 Japan's first AFM (SFA300) 1992 Japan's first SPM (SFA300) Main unit: Controller: SPA-400 SPI3600 SPA-300HV SPI3700 SPA-250, SPA-260, SPI3800 SPA-270, SPA-500				
20003	 2000 Seiko Instruments founded SII Microscope Inc. 2003 SII Microscope Inc. changed company name to SII NanoTechnology Inc. SII NanoTechnology succeeded the scientific instruments business of Seiko Instruments by partition of corporation 	Main unit: Controller: Nanocute SPI4000 S-image NanoNavi E-sweep NanoNavi∏ L-trace L-trace				
2010s —	 2013 Became a member of Hitachi High-Technologies Group and officially changed company name to Hitachi High-Tech Science Corporation. Company head office moved to Minato-ku, Tokyo Hitachi High-Tech Science succeeded Design & Development, QA and Domestic Sales section of analytical instruments business from Hitachi High-Technologies 	Main unit: Controller: AFM5100N AFM5000 AFM5200S (NanoNavi Real) AFM5300E AFM5000II AFM5400L				



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.

The above logo is a registered trademark of Hitachi High-Tech Corporation in the United States and other countries.

- Note: •To ensure safe operation, follow the instruction manual when using the instrument.
 - 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.
 - "RealTune" is a registered trademark of Hitachi High-Tech Science Corporation in Japan, the United States, and the European Union.

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