



Scanning Probe Microscope SPM-9700



Making the Unknown Visible Scanning probe microscope (SPM) is a generic term for microscopes that scan sample surfaces with an extremely sharp probe to observe their three-dimensional images of local properties at bigh magnifications. The SPM-9700 offers higher performance, faster speeds, and easier operation. image or local properties at high magnifications. SPM-9700 SCANNING PROBE MICROSCOPE

SPM-9700

00000 0000 PM CONTROL UNIT Det. Functionality and Expandability to Meet a Wide Variety of Requirements P.4 Head-Slide Mechanism – High Stability P.6 Ease of Operation Minimizes Distraction from Observation to Analysis P.8 Head-Slide Mechanism – High Throughput P.7 Wide Variety of 3D Rendering Functions Using Mouse Operations P. 10 Installation Specifications P.21 Particle Analysis Software P. 11 SPM Data Room Website P. 12 P.14 P.16 SPM Unit WET-SPM Series

Functionality and Expandability to Meet a Wide Range of Requirements







Head-Slide Mechanism – High Stability



Allows Sliding the Entire Optical Lever System as a Single Unit, While Maintaining High Rigidity.

- The laser remains stable and irradiates the cantilever even while replacing samples.
- Design is resistant to vibration, noise, wind, and other external disturbances, so a specialized enclosure is not necessary.
- The main unit includes a built-in vibration isolator.



Secret to the High Stability of the SPM-9700

Remarkable Mechanism Maintains High Performance — Comparison of Stability for Different Laser Irradiation —



Head-Slide Mechanism – High Throughput

Successfully Opened Up the Area Around the Sample While Maintaining High Rigidity

- Samples can be replaced without removing the cantilever holder.
- Samples can be accessed even during SPM observation.
- Samples are approached automatically, regardless of thesample thickness.

thickness. (Japanese Patent No. 2833491)



SPM-9700 Scanning Probe Microscope

Ease of Operation Minimizes Distraction from **Observation to Analysis**

A revolutionary layout-free graphical user interface (GUI) provides borderless support for operations ranging from online observation to offline analysis. This means the SPM can be operated from observation to analysis without confusion.



Operate Without Confusion

From startup to observation and analysis, the SPM can be operated using only mouse clicks; no complicated settings are required



Startup





automatically, from approach to observation.

Select the observation mode in the manager window.

Follow the steps indicated in the guidance window to easily complete setup.

Determine the Observation Position Without Confusion



1 Observation Window

Up to 8 images can be displayed simultaneously. This means the surface shape and physical properties can be compared in multiple images, while scanning.



2 Navigator

The Navigator allows freely navigating from a broad area to any specific area desired.

Saved image data can be displayed as reference as well.

Obtain Observation Results Without Confusion



Online Profile

Cross-section profiles can be measured in the online window while observing samples.



4 Image History

Past image data can be displayed next to current observation images for comparison.

Wide Assortment of Scanning Functions



5 Force Mapping

(special order) A force curve can be measured for each point in observed image data to acquire a distribution of sample mechanical properties or adhesion force.

| Command | | | Automos. |
|--------------|--|------|----------|
| Scarerer X | 0.000 pm | | bitules. |
| Scarner'Y | 0.000 µm | | |
| Continuously | 399 | Stop | |
| - | it. | | |
| Red . | Long de la constante de la con | | |
| Command | | | 64 |
| and a | . or Smith 11 | | |

Vector Scanning (special order)

The scanning direction, force between the probe and sample, or the applied voltage can be programmed to allow scanning according to a program.



4 **Display** Image data observed in the past can be viewed without switching offline.



Offline Analysis

A wide selection of functions for displaying, processing, and analyzing images are available for expressing observation results more attractively and quantitatively. Improved Usability!

Wide Variety of 3D Rendering Functions Using Mouse Operations

Use the mouse to freely rotate images, zoom, or change the Z-axis magnification. This enables expressing image data in a variety of ways while confirming the data in real time.



Texture Function

Height information can be displayed overlaid with information about other physical properties. This allows clearly showing the relationship between both parameters.



3D Image



Overlay of Topographic Image and Phase Image

3D Cross-Section Profile Analysis

Cross-section profiles can be analyzed in 3D images. If physical property information is expressed in terms of texture, respective cross-section profiles can be displayed and analyzed in the same location.



Particle Analysis Software (option)

16 Average Z

20 Surface Area

22 Pattern Direction

24 Area / Feret Area

27 Circular Degree

23 2nd Moment Direction

25 Particle Area / All Area

21 Volume

26 Distortion

28 Roughness

29 Thin Degree

17 Average Round Z

18 Area excluding Holes

19 Area including Holes

The particle analysis software extracts multiple particles from SPM-9700 image data and calculates feature values for each particle, then analyzes and displays them. This is especially useful for processing data statistically. The following wide selection of feature values and their corresponding statistical quantities can be calculated, tabulated, sorted, or graphed. Numerical data can be exported.

Feature Parameters

- 1 Center X
- 2 Center Y
- 3 Maximum Diameter
- 4 Pattern Width
- 5 Horizontal Feret Length
- 6 Vertical Feret Length
- 7 Radius as Circle excluding Hole
- 8 Radius as Circle including Hole
- 9 Mean Radius
- 10 Mean Radius Variance
- 11 Nearest Distance
- 12 Perimeter
- 13 C Perimeter
- 14 Maximum Z
- 15 Minimum Z

Analysis Example



Thin Film (5 µm square)



E. Coli Bacteria (30 µm square)



Particle Extraction and Classification Results



Particle Extraction and Labeling Results

Statistical Values

Average
 Standard Deviation
 Line Average
 Square Average
 Cubic Average
 Sum
 Maximum
 Maximum
 Maximum Label
 Minimum Label
 Range
 Samples



Histogram of Mean Radius



Graph of Correlation Between Maximum Diameter and Thin Degree

SPM Data Room Website

The SPM Data Room website includes examples of new observation data, applications, a list of scientific articles, and a list of presentations.



Boundary Surface of Plating Layer



A cross-section of a copper (Cu) plated iron (Fe) sample was prepared, and the electric potential measured along the boundary surface. The topographic image on the left does not show any change in thickness, but the electric potential image on the right shows that the iron portion has a potential that is about 90 mV higher.

04 Minerals

Observation of Calcite in Solution



The crystal dissolution process of calcite in solution was observed. Propagation steps of about 0.3 nm, due to dissolution, were observed. About 10 minutes elapsed between (b-1) and (b-3).

(Data provided by Dr. Kagi, School of Science, The University of Tokyo)

05 Ceramics

Film Dispersed with Silica



Film material with mono-dispersed spherical silica dispersed in an organic binder. This clearly shows how the binder binds the spherical particles. (Data provided by Japan Fine Ceramics Center (JFCC))

06 Polymers Li-lon Battery Separator



The separator surface was observed after removal from the lithium-ion battery. Heated observation shows how the fiber swells at high temperatures and fills the pores.

07 Powders Toner Particle



The top part of one toner particle was observed at high magnification. A topographic image of the surface is shown on the left. Phase and surface potential (KFM) images are shown on the right. The images on the right show how comparing images of different properties in the same field of view allows correlating the distribution of toner material and external additives with the corresponding electric potential distribution.

08 Nanotechnology

Rendering Images Using Electric Potential



Vector scanning was used on a gold vapor deposition surface on a silicon substrate to render the trace shown in Fig. 1. A conductive cantilever was used to apply a tiny electric potential between the sample and probe. After rendering, simultaneous AFM and KFM measurements showed no change in the shape of the AFM image (Fig. 2), but the potential measured along the trace in the KFM image (Fig. 3) was about 50 mV lower than the surrounding area.

09 Thin Films

Cross-Section of Thin Film



A cross-section of an organic thin film vapor-deposited on a silicon substrate was observed with the SPM by turning the sample so the cut edge faced upward.

The boundary can be clearly observed. This shows that about the top 1/3 is the organic film layer, which is 390 nm thick. This application example is only possible because of the stable probe control provided by the SPM-9700.

10 Semiconductors

Electric Potential Analysis of Organic Thin Film Transistor (FET)

This is an example of analyzing the shape and electric potential of organic thin film transistors, which have gained attention for their use in flexible displays and other applications. The film material is P3HT (3-hexylthiophene), which provides high electron mobility. To use the SPM for actual measurement, the source electrode was grounded and an electric potential was applied independently to the gate



and drain electrodes, then the variation in surface potential on the gate was determined. (Data provided by Dr. Fukuda, Department of Information and Electronic Engineering, Muroran Institute of Technology)

11 Coatings

Baking Finished Surface



The coated surface shows many holes from outgassing. The metallic painted surface (left) shows it contains metal fibers.



SPM-9700 Scanning Probe Microscope SPM Unit



Specifications for SPM Unit

| Resolution | XY: 0.2 nm, Z: 0.01 nmm | | | |
|----------------------------------|---|--|---|--|
| Max. Scanning Range (X, Y, Z) | Standard scanner Wide range scanner Deep scanner Narrow range scanner | X and Y: 30 µm X and Y: 125 µm X and Y: 55 µm X and Y: 2.5 µm | Ζ: 5 μm Ζ: 7 μm Ζ: 13 μm Ζ: 0.3 μm | |
| Stage | Max. sample size: 24 mm dia. × 8 mm Sample replacement method: Head-slide mechanism with integrated displacement detection system and cantilever Samples can be replaced without removing cantilever. Sample securing method: Secured with magnets | | | |

Consumable Parts

| Cantilever for contact mode | SiN | Set of 34 chips |
|---|-----|-----------------|
| Cantilever for dynamic mode | Si | Set of 20 chips |
| Cantilever for magnetic force mode (MFM) | Si | Set of 20 chips |
| Cantilever for current mode | Si | Set of 20 chips |
| Cantilever for surface potential mode (KFM) | Si | Set of 20 chips |

* Many other types of cantilevers are also available. Contact your Shimadzu representative for details.

Standard Functions

Contact Mode

This mode creates an image of displacement in the sample height direction by scanning the sample surface with the repulsive force acting between the cantilever tip and sample kept constant. Force curves can be measured as well.



Dynamic Mode

This mode vibrates the cantilever near its resonant frequency. Since the amplitude changes as the cantilever approaches the sample, a sample height displacement image can be created by moving the probe to keep the amplitude constant.

Force curves can be measured as well.

Phase Mode

This mode detects the phase shift delay in the cantilever vibration during dynamic mode scanning. This allows creating an image of differences in sample surface properties.

Optional Functions

Current Mode

This mode applies a voltage between a conductive cantilever and sample during contact mode scanning and creates an image from the distribution of current flows. *IV* measurement is also possible.

Surface Potential Mode (KFM)

An image can be created from the electric potential of the sample surface by applying an alternating current electrical signal to a conductive cantilever and detecting the static electric force acting between the sample surface and cantilever.

Magnetic Force Mode (MFM)

This mode scans the sample with a magnetic tipped cantilever kept at a constant distance from the sample. An image can be created from magnetic information of the sample surface obtained by detecting the magnetic force from the magnetic leakage field.

Petri Dish Type Solution Cell

The sample is attached to the bottom of a small petri dish, which is then filled with solution. By scanning with the cantilever immersed in solution, AFM observations can be performed in solutions.









Lateral Force Mode (LFM)

By detecting the amount of twist in the cantilever during contact mode scanning, an image can be created from information corresponding to lateral forces (friction) acting between the sample and cantilever.



Force Modulation Mode

This mode vibrates the sample at constant amplitude and frequency during contact mode scanning. The cantilever response is detected by separating it into its amplitude and phase components. This allows creating an image of differences in sample surface properties.



Force Mapping (special order)

A force curve can be measured for each point in observed image data to observe a distribution of sample mechanical properties or adhesive strength.



Vector Scanning (special order)

The scanning direction, force between the probe and sample, or the applied voltage can be programmed to allow scanning according to a program.



Electrochemical Solution Cell

This cell is used for AFM observations of sample surface changes while an electrochemical reaction occurs in an electrolytic solution. The cell includes three standard electrodes (working, counter, and reference) and includes a petri dish type solution cell.

(Does not include the separately-ordered electrochemical controller (potentiostat).)



Environment Controlled Scanning Probe Microscope WET-SPM Series



SPM Observations in a Controlled Environment

By adding an environment controlled chamber, the SPM-9700 scanning probe microscope can be upgraded to a WET-SPM series system. This is only possible for the SPM-9700, which was optimized for operating within a controlled chamber, by including features such as a Shimadzu proprietary head-slide mechanism, operation from the front panel, fully automatic approach, and open head design.

This is especially ideal for samples vulnerable to air or moisture.



Environment Controlled Chamber CH-I/CH-II

These environment controlled chambers, CH-II (without TMP) and CH-III (with TMP), were designed specifically for the SPM-9700 series as a chamber system with a built-in vibration damper. Since this enables controlling both the sample and surrounding environment, the SPM can be used to directly observe samples processed in a controlled environment (Japanese Patent No. 2612395, US Patent No. 5200616). A large view port and dual glove ports allow pretreating samples inside the chamber. Adding the option for in-situ SPM permits real-time investigation of surface changes due to changes in physical parameters such as temperature, humidity, pressure, luminescence, and concentration. The SPM unit can be easily loaded into and unloaded from the chamber from the rear, allowing it to be used for both ambient atmosphere and controlled environment observations.





| Specifications | |
|---------------------------------|---|
| Port | Glove port2Large view port1Unit loading port1Sample loading port1Pumping port1Spare port4 |
| Pumps Used for Vacuum System | Rotary pump (160 L/min) Turbomolecular pump (50 L/sec) (CH-III only) |
| Gas Introduction Mechanism | Single-circuit automatic control |
| Current Input Terminals (7-pin) | 16 (including spares) |
| Vibration Damper | Integrated air-spring vibration damper |

SPM-9700 Scanning Probe Microscope

WET-SPM Series Options

Temperature and Humidity Controller

Controller is attached to an environment controlled chamber to control the temperature and humidity inside the chamber.





Humidified Gas Generator

FC Film Observation with Environmentally Controlled Temperature and Humidity



Low Temperature High Temperature Variations in the surface shape of Nafion film due to changes in humidity were observed.

In each case, microscopic features of about a few nm in height were observed, but the images show that increasing the humidity results in smoother features and more swelling.

Polymer Film



Low Temperature High Temperature Variations in the shape of polymer film were observed using a controlled temperature and humidity environment.

Gas Spray Unit

The gas spray unit is attached to a spare port to spray small amounts of gas on the sample.



Real Time Observation of Nickel Surface Variations



The nickel surface's reaction to gas was observed continuously in real time. When the clean surface after reduction (left) started being sprayed with carbon monoxide, the change in shape was observed as carbonyl complexes were formed (right). (Data provided by former National Institute of Materials and Chemical Research)

Sample Heating and Cooling Unit

The sample can be loaded into the unit and heated or cooled.



Observation of Cooled Plastic



Room Temperature Two separate phases were observed in the viscosity image



Cooled to -30 °C After cooling, there were almost no visible differences in viscosity.

Sample Heating Unit

The sample can be loaded into the unit and heated. The unit can even be operated in atmospheric conditions, depending on the sample.



Temperature Controller



Heated Holder Installed in Scanner

Observation of Heated Polymer Film







50° The phase image (right) clearly shows the changes in sample surface physical properties as the sample is heated.

Light Irradiation Unit

This unit enables the use of a fiber optic light to irradiate sample surfaces. It does not include the light source or the optical fiber. It can be operated in atmospheric conditions.



Observation of Ultraviolet Light Irradiating Pentacene Thin Film on SrTiO₃





Before Irradiation

40 Minutes After Irradiation

The pentacene thin film was formed as a cluster of two or three 1.6 nm thick layers. When irradiated with 365 nm wavelength ultraviolet light, the cluster structure gradually started breaking apart. After 40 minutes, the thin film cluster was mostly gone. During this time, there is negligible drift and observation is possible using the same field of view. (Data provided by Dr. Yuji Matsumoto, Frontier Research Center, Tokyo Institute of Technology)

> SPM-9700 Scanning Probe Microscope

Specifications

1. SPM Unit

| Observation | Standard | Contact | | |
|---------------------|----------------------------------|--|--|--|
| Modes | | Dynamic | | |
| | | Phase | | |
| | | Lateral Force (LFM) | | |
| | | Force Modulation | | |
| | Optional | Magnetic Force (MFM) | | |
| | | Current | | |
| | | Surface Potential (KFM) | | |
| Resolution | Х, Ү | 0.2 nm | | |
| | Z | 0.01 nm | | |
| SPM Head | Displacement detection system | Light source/Optical lever/Detector | | |
| | Light source | Laser diode (ON/OFF) | | |
| | 5 | Irradiates cantilever continuously, | | |
| | | even while replacing samples. | | |
| | Detector | Photodetector | | |
| Scanner | Drive element | Tube piezoelectric element | | |
| | Max. scanning | 30 μm × 30 μm × 5 μm | | |
| | size (X, Y, Z) | 125 μm × 125 μm × 7 μm (optional) | | |
| | | 55 μm × 55 μm × 13 μm (optional) | | |
| | | 2.5 μm × 2.5 μm × 0.3 μm (optional) | | |
| Stage | Max. sample | 24 mm dia. × 8 mm | | |
| 5 | size | | | |
| | Sample | Head-slide mechanism with integrated | | |
| | replacement | displacement detection system and cantilever | | |
| | method | Samples can be replaced without removing cantilever. | | |
| | Sample securing | Magnet | | |
| | method | | | |
| Z-Axis Coarse | Method | Automatic, using stepping motor | | |
| Adjustment | | Fully automatic, regardless of sample thickness | | |
| Mechanism | Max. stroke | 10 mm | | |
| Signal Display | Displayed | Total incident light to detector (digital display) | | |
| Panel | quantity | ······································ | | |
| Vibration Isolation | Vibration | Built into SPM unit | | |
| System | Damper | | | |
| Optical Microscope | Method | Beam-splitter slide mechanism | | |
| Observation | | | | |
| Specialized | Method | Not necessary or environment controlled chamber | | |
| enclosure | | is used. | | |
| Environment | Method | Chamber can be added without modifying SPM unit. | | |
| Control | | | | |

2. Control Unit

| Scan Controller | X/Y-axis control | ±211 V, full time 16-bit accuracy | | |
|--------------------------------|------------------|--|--|--|
| | Z-axis control | ±211 V, max. 26-bit accuracy | | |
| Feedback Controller | Control system | Digital control by DSP | | |
| Data Acquisition Controller | Input signal | 5 channels (standard) 7 channels (optional) | | |
| Communications | Protocol | ТСР/ІР | | |

3. Data Processing Unit

| Host Computer | Operating system | Windows 7 Professional (32-bit), English version | | |
|-----------------------------|---------------------|---|--|--|
| | RAM | 2GB min | | |
| | Strorage | HDD 160GB min. CD-RW drive | | |
| | Graphics | memory : 256MB min. | | |
| Monitor | Panel | Flat panel display Display resolution : 1920 × 1080 pixels | | |
| Communications Interface | Protocol | ТСР/ІР | | |



4. Software

| Online | Input Signal | Select from up to 6 signals. |
|---------|-----------------------|--|
| | Image data | Maximum 8 images can be displayed simultaneously. |
| | display | |
| | Scanning direction | Trace/retrace (simultaneous observation possible) Angle setting can be changed. |
| | Scanning size | 0.1 nm to max. scanning size (depending on scanner type) Offset setting can be changed. |
| | Number of pixels | 2048 × 2048, 1024 × 1024, 512 × 512, 256 × 256, 128 × 128, 64 × 64, 32 × 32 |
| | Data size | Approx. 16 MB to 64 KB/data |
| | Observation | Multiple frames display: 1 frame, frames, 2 frame, frames |
| | window | (Vertical or Horizontal), or 4 frame, frames |
| | | Z-axis display range settings (display range, offset) |
| | | Color palette settings (400 types) |
| | | Tilt correction setting |
| | | Image history display modes (list, single screen) |
| | Profile display | Display cross-section profile during scanning, and save |
| | | Display cross-section profile at scanning position, analyze profile |
| | | between any two points. |
| | Status display | Display the operating status of the main unit. |
| | Preset | Register and retrieve parameter settings. |
| | Calibration | Independent calibration of each axis (X, Y, and Z) |
| | Scanning | Switch XY-scanning ON/OFF |
| | | Switch Y-scanning ON/OFF |
| | | Y-scanning can be restarted. |
| | | Y-scanning start position can be changed (top, center, or bottom). |
| | Signal display | Display detector vertical/horizontal variation signal. |
| | | Display feedback signal. |
| | Nevientez | Display laser intensity. |
| | Navigator | change angle. Load and display image data. |
| | Image history | Display list of saved images or display saved images. |
| | Cuidenee | Display cross-section profile or analyze profile between any two points. |
| Offline | Browcor | List in thumbrail mode |
| Omme | DIOWSEI | Delete conv move or search data |
| | | Change group names or data names. |
| | | Create/delete folders. |
| | lmage data | Variable shade image (top view) display |
| | display | (length measurement possible) |
| | | Pseudo-3D display, 3D display |
| | | Zoom in/out or rotate 3D display (mouse operation possible) |
| | | Analyze cross-section profile of 3D display. |
| | | Set light source, view angle, and gloss settings for 3D-image display. |
| | | Display contour lines. |
| | | Change 7-axis range setting set 7-axis units |
| | | Reduce/enlarge image create as icon |
| | | Display image data information |
| | | (parameters, image processing history, comments). |
| | | Enter and display comments. |
| | Line data | Overlay, tile, overwrite. |
| | display | Line colors can be changed. |
| | | Reduce/enlarge image, create as icon. |
| | Image data | Flatten, erase noisy lines. |
| | processing | Local filter, spectrum filter |
| | | Zoom, invert, and rotate image. |
| | | Resample, extract lines, use macro functions. |
| | Image data | Profile analysis, line roughness analysis. |
| | andiysis | surrace roughness analysis, topography analysis, step |
| | | Power spectrum analysis autocorrelation analysis |
| | | Fractal analysis, line length analysis, line roughness analysis |
| | File output | DIB formant (bitmap) |
| | | TIFF format, ASCII format |
| - | | |

* Windows is a registered trademark of Microsoft Corporation in the United States and/or other countries.
* Other company names and product names mentioned in this document are trademarks or registered trademarks of their respective companies.
* TM and ® symbols are omitted in this document.

Installation Specifications

Installation Environment

The following conditions are appropriate for the room where the SPM is installed. Temperature $: 23 \text{ °C} \pm 5 \text{ °C}$ Relative Humidity : 60 % max.

Power Supply

The following power supply is required to operate the SPM-9700.

SPM-9700

Single-phase 100 - 120 V / 200 - 240 V, 50/60 Hz, 15 A - 2 circuits Grounding Resistance: 100 $\,\Omega\,$ max.

* The power supply indicated above is for a basic configuration of the SPM-9700 and can vary depending on the options included. Please see specifications for details.

Environment Controlled Chamber Single-phase 100 - 120 V, 50/60 Hz, 15 A - 2 circuits Grounding Resistance: 100 Ω max.

Size and Weight of Units

| SPM Unit | W180 | × D255 × H260mm | 5.5kg |
|--------------------------------|-------|--------------------|--------|
| Controller | W250 | × D420 × H454mm | 18.5kg |
| Environment Controlled Chamber | W1170 |) × D725 × H1055mm | 210kg |





Installation Example * Figure shows example of one possible configuration.

800mm



▶ |⊲

* Dimensions for the computer table and desk-type air-spring

vibration damper are only indicated for reference purposes.

1200mm







* Dimensions for the computer table are only indicated for reference purposes.



725mm



Shimadzu Corporation www.shimadzu.com/an/ Company names, product/service names and logos used in this publication are trademarks and trade names of Shimadzu Corporation or its affiliates, whether or not they are used with trademark symbol "TM" or "@". Third-party trademarks and trade names may be used in this publication to refer to either the entities or their products/services. Shimadzu disclaims any proprietary interest in trademarks and trade names other than its own.

For Research Use Only. Not for use in diagnostic procedures. The contents of this publication are provided to you "as is" without warranty of any kind, and are subject to change without notice. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication.

> © Shimadzu Corporation, 2012 Printed in Japan 3655-10205-15ANS