Version 2.3.2

Date: 2018.11.22

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NX10 USER'S MANUAL



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Preface

The Scanning Probe Microscope (SPM) is not only leading the list of equipment pioneering the nano scale world, it is also the most fundamental technology. Subsequent to the first generation optical microscope, and the second generation electron microscope, the SPM has every right to be recognized as a "third generation" microscope since it enables users to experience and have a glimpse of the nano scale world. In addition, it has significant advantages compared with manual microscopes which passively observe samples. The SPM is like a miniature robot, fabricating specific structures by manipulating atoms on the sample surface and using a probe tip to take measurements of those structures.

The SPM originated with the invention of the Scanning Tunneling microscope (STM). The STM uses a tunneling current between a probe tip and a sample in a vacuum state to measure surface topography. As a result, it is limited in that it can only measure conductor or a semiconductor sample. Once the Atomic Force Microscope (AFM) was developed, however, a whole new range of measurement capabilities became possible. Now, it is not only capable to measure non-conductors in air, but also capable to measure the physical, chemical, mechanical, electrical, and magnetic properties of a sample's surface, and even measure live cells in solution.

The SPM is indeed the key toward the world of nano technology that has yet to flourish. Also, it is an essential equipment for various researches in the field of basic sciences – physics, chemistry, and biology - and in applied industry - mechanical and electrical engineering.

The importance of the SPM attests to grow greater and greater in the future.

Safety Precautions of System

This preview section describes some safety requirements and procedures related to the general operating of the NX10 in detail. This section should be thoroughly und erstood before operating the NX10 for your safety.

CAUTION!

If the user operates the NX10 in a manner not specified in this User's Manual, serious damage to the instrument may result.

1. Hazard Labels

On the NX10 system, there are hazard labels on the position for hazard possibilities. Caution must be taken for the each hazard label warning.

Symbol	Description
	"ON" (power) To indicate connection to the mains, at least for main switches or their positions, and all those cases where safety is involved.
\bigcirc	"OFF" (power) To indicate disconnection from the mains, at least for main switches or their positions, and all those cases where safety is involved.
\triangle	"Caution, Risk of Danger" This symbol denotes conditions or activities that could cause damage to the equipment.
Λ	"Caution, Risk of Electric Shock" This symbol denotes conditions or activities that could cause electrical shock or burns.
	" Protective Earth(Ground) " This symbol denotes a need for protective grounding of equipment.

Table. Hazard label List

1-1. Electrical Hazard Label



The Electrical Hazard label notifies the area that might cause electrical damage t

o the system of to the personnel. Care must be taken.

The Electrical Hazard label is attached to the areas listed below.

- AFM Controller
- AFM Head
- XY scanner
- Computer
- Monitor

1-2. Protective Ground label



The protective ground label indicates that this equipment needs to be electrically grounded.

2. Operating Safety

2-1. Definition of safety symbols

Table shown below explains the meaning of the safety symbols -

WARNING, CAUTION, NOTE.

Table. Safe	y terms	and	their	meanings
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Symbols	Meaning
WARNING!	Alerts Users to potential danger. Consequences and countermeasures are described. If users fail to follow the procedures described in this manual, serious injury or instrument damage may occur. Such damage will NOT be covered by warranty.
CAUTION!	Calls attention to possible damage to the system that may result if users do not follow the procedures described in this manual.
NOTE!	Draws attention to a general procedure that is to be followed.

Please understand these safety terms thoroughly, and follow the associated instructions. It is important that you read all safety terms very carefully. **WARNING**s, **CAU TION**s, and **NOTE**s include information that, when followed, ensure the operating safety of your NX system.

2-2. General Operating Safety

The following are most of the **WARNING**s, **CAUTION**s, and **NOTE**s necessary to operate the NX10 safely.

WARNING!

The NX10 should be grounded before its components are connected to electric power. The main power plug needs to be connected to a three-prong outlet which includes a protective earth ground contact.

WARNING!

Before the power is turned on, the power selections for the individual components need to be inspected. The voltage selector switch is located on the rear panel of the NX10 Control Electronics, and it can be set to the following voltages: 100 V, 120 V, 230 V, or 240 V.

WARNING!

Do not open the NX10 Control Electronics or the AFM head. Doing so may result in serious electrical shock, as high voltages and electrostatic sensitive components are used in the NX10 Control Electronics and the AFM head.

WARNING!

Be cautious when loading large modules such as EC Cell or ULC onto the NX sample chuck. They may collide with the motorized XY stage when it is moved through a large range, and the modules and/or NX system may be damaged.

CAUTION

Check regularly to ensure that the NX10's cables are free from damage and that all connections are secure. If any damaged cables or faulty connections are found, contact your local Park Systems service representative. Never try to operate the equipment under these conditions.

CAUTION!

All parts in the NX10 system should be handled with extreme care. If not handled properly, these parts can be easily damaged as they are made of fragile electromagnetic equipment.

CAUTION!

An EMI filter must be installed to maintain operating safety and meet EMC (Electromagnetic Compatibility Compliance).

CAUTION!

The AFM head should always be handled with care. When removed from the AFM, the AFM head needs to be carefully placed on a flat surface. This will protect the scanner, the cantilever, and the beam alignment knobs. Never allow anything to impact the AFM head. When separated from the main frame, it is safe to keep the head in its storage box.

CAUTION!

Before the AFM head is mounted or unmounted from the Z stage, the on/off switch for the AFM beam must be turned off. Otherwise, the AFM beam diode in the AFM head may be damaged.

CAUTION!

When the AFM head is mounted or unmounted from the Z stage, ensure that the AFM head does not sustain any damage, and that it is properly grounded. The AFM head is extremely sensitive to electrostatic discharge.

CAUTION!

To meet the EMC guidlines, the Acoustice Enclosure should be closed while making measurements with the NX10.

3. Compliance

3-1. FCC

FCC Label(Part 15 sec.15.19)

Model Name: NX10

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This device complies with Part 15 of the FCC Rules. Operation is subject to the following two conditions: (1) this device may not cause harmful interference, and (2) this device must accept any interference received, including interference that may cause undesired operations.

Made in Korea

This equipment has been tested and found to comply with the limits for a Class A digital device, pursuant to part 15 of the FCC Rules. These limits are designed to provide reasonable protection against harmful interface when the equipment is operated in a commercial environment. This equipment generates, uses, and can radiate radio frequency energy and, if not installed and used in accordance with this User's manual, may cause harmful interference to radio communications. Operation of this equipment in a residential area is likely to cause harmful interference in which case the user will be required to correct the interference at his own expense.

WARNING!

Any changes or modifications not expressly approved by the manufacturer could void the user's authority to operate the equipment.

3-2. CE

A1 / 04.11

CE Certification



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Chapter 1. Introduction to NX10

1-1. Scanning Probe Microscope

The Scanning Probe Microscope (SPM) proved a prevailing concept wrong, that an atom is too small to be observed with even the best microscope. Now, it has every right to be identified as the third generation microscope, with optical and electron micr oscopes being the first and second generations respectively. Whereas the maximum magnifying power of an optical microscope is several thousands and that of a scannin g electron microscope (SEM) is tens of thousands, an SPM has the magnifying power of tens of millions, enough to observe individual atoms. Even though a transmission electron microscope (TEM) has the lateral resolution high enough to image at the ato mic level, its vertical resolution is much weaker at observing individual atoms. On the other hand, the vertical resolution of SPM is even better than its horizontal resolution, making it possible to measure on the scale of fractions of the diameter of an atom (0. 01nm).

The SPM, with its exceptional resolution, not only makes it possible to understan d the various nano scale worlds which heretofore were not completely revealed, but al so brings the unbelievable into reality. It provides such capabilities as allowing a user to change the position of individual atoms or to write letters by transforming the surfa ce of a material at the atomic level.

1-2. Atomic Force Microscope

Among SPMs, the first to be invented was the Scanning Tunneling Microscope (S TM). The STM measures the tunneling current between a sharp, conducting tip and a conducting sample. The STM can image the sample's topography and also measure t he electrical properties of the sample by the "tunneling current" between them.

The STM technique, however, has a major disadvantage in that it cannot measur e non-conducting material. This problem has been solved by the invention of the Atom ic Force Microscope (AFM) which may be used to measure almost any sample, regar dless of its electrical properties. As a result, the AFM has greatly extended the SPM's applicability to all branches of scientific research.



Figure 1-1. Diagram of Conventional AFM's Scanning

Instead of a conducting needle, the AFM uses a micro-machined cantilever with a sharp tip to measure a sample's surface. See "Chapter 4. Cantilever Selection" for m ore information on the cantilever. Depending on the distance between the atoms at th e tip of the cantilever and those at the sample's surface, there exists either an attracti ve or repulsive force/interaction that may be utilized to measure the sample surface. See the "AFM in Contact Mode" and AFM in "Non-Contact Mode" chapters for a furthe r discussion on utilizing the atomic forces.

Figure 1-1 displays the basic configuration for most AFMs. This scanning AFM is typically used to measure a wide variety of samples, which have relatively small roug hness. The force between the atoms at the sample's surface and those at the cantilever's tip can be detected by monitoring how much the cantilever deflects. This deflection n of the cantilever can be quantified by the measurement of a beam that is reflected o ff the backside of the cantilever and onto the Position Sensitive Photo Detector (PSP D).

The tube-shaped scanner located under the sample moves a sample in the horiz ontal direction (X-Y) and in the vertical direction (Z). It repetitively scans the sample li ne by line, while the PSPD signal is used to establish a feedback loop which controls t he vertical movement of the scanner as the cantilever moves across the sample surfa ce.

The AFM can easily take a measurement of conductive, non-conductive, and eve n some liquid samples without delicate sample preparation. This is a significant advan tage over the extensive preparation techniques required for TEM or SEM.

Despite its many advantages, the AFM does have some drawbacks as well.

- In general, the scanners used in AFMs are piezoelectric ceramic tubes (Figure 1-1). Due to the non-linearity and hysteresis of piezoelectric materials, this may result in measurement errors as shown in Figure 1-2.
- The geometrical and structural restraints imposed by the tube type scanner results in cross coupling of the individual scan axes. Thus, independent movement in the x, y, and z directions is impossible.
- 3. Since the tip has a finite size, it is very difficult and sometimes impossible to measure a narrow, deep indentation or a steep slope. Often, even though such a measurement may be possible, the convolution effect due to the shape of the tip and the sample profile may result in measurement errors.
- Since the tip has to mechanically follow a sample surface, the measurement speed of an AFM is much slower than that of an optical microscope or an electron microscope.

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Since the tip has a finite size, it is very difficult and sometimes impossible to mea sure a narrow, deep indentation or a steep slope. Often, even though such a measure ment may be possible, the convolution effect due to the shape of the tip and the samp le profile may result in measurement errors.

The most inconvenient aspect of using the AFM is its slow speed. As mentioned above, since the image is obtained by the tip's mechanically following a sample's surf ace, it is much slower than other microscopes that use electrons or light. The main fac tors slowing the speed of the AFM are the Z scanner's response rate and the respons e rate of the circuit which detects changes in the cantilever's resonant frequency. The

resonant frequency of the typical tube scanner is several hundred Hz. In order to accurately measure a sample area with 256×256 pixels (data points), it is necessary to scan at a rate of about one line per second. Thus, it takes approximately 4 minutes to acquire an image.

For most cases, the second and third problems listed above can be minimized by software calibration. This is a reasonably simple and inexpensive procedure that invo lves imaging a standard sample, (usually a grid structure with a known pitch) in order t o create a calibration file that will be used to control the scanner's movements when u nknown samples are imaged. Correction using software, however, still depends heavil y on the scan speed and scan direction, and such a correction becomes accurate onl y when the center of the scan range used to measure an unknown sample coincides exactly with the center of the scanning range that was used to image the standard sa mple and to create the calibration file.

Figure 1-2. Nonlinearity and Hysteresis (a), and Cross Coupling (b) Observed in Piezoelectric Tube Scanners



1-3. Park Systems AFM

on axis

Figure 1-3. Z Scanner Separated from X-Y scanner

Since the conventional tube type scanner cannot move in one direction independ ently from other directions, movement in one direction will always simultaneously affe ct the scanner's movement in other directions. This cross talk and non-linearity cause d by the scanner's three axes being non-orthogonal to another has a more pronounce d effect in the case of measuring larger areas or flat samples. This intrinsic problem c an be eliminated completely, however, by physical separation of the Z scanner from th e X-Y scanner (see Figure 1-3).

The breakthrough that eliminated these cumbersome problems came when the P ark Systems SPMs introduced a new concept of separating the Z scanner from the X-Y scanner. The NX system is designed so that the X-Y scanner scans a sample in two -dimensional space, while the Z scanner moves the tip only in the z direction. Figure 1 -3 shows a diagram of the NX system, in which the Z scanner separated from the X-Y scanner. The symmetrical flexure scanner used in the NX system moves only in the X-Y plane, and has superb orthogonality. This scanner's design also makes it possibl e to place much larger samples on the sample stage than could normally be accomm odated for by a piezoelectric tube type scanner. Furthermore, since the flexure scanner only moves in the X-Y direction, it can be scanned at much higher rates (10~50 Hz) than would be possible with a standard AFM. Because the stacked piezoelectric actu

ator used for the Z scanner has a very fast response speed, at least 5 kHz, it is able t o respond to topographic changes on the sample surface more than 10 times faster th an is possible with a conventional tube type scanner.

Having the X-Y scanner separated from the Z scanner in the uniquely designed N X system not only increases the data collecting speed by at least 10 times compared t o a conventional tube type scanner, but also isolates the vertical and horizontal scan axes, completely eliminating cross coupling, resulting in a very accurate measuremen t. Moreover, this independent scanning system improves the error due to the inherent non-linearity of the scanner itself. Figure 1-4 compares the background image of a co nventional tube scanner compared to that of the new NX scan system.

Figure 1-4. Background Flatness Images from a Conventional AFM (a) and Park Systems AFM (b)



Figure 1-5 shows a diagram that explains the cantilever movement detection mec hanism used in the NX system. This SLD Beam & PSPD configuration, which permits the accurate acquisition of stable images at high measurement speeds, satisfies the f ollowing two important imaging conditions:

First, the PSPD should be able to measure only the deflection of the cantilever wi thout interference from the Z scanner.

Second, to improve the response rate in the Z direction, the weight of the Z scan ner must be minimized.





The cantilever and the PSPD move together with the Z scanner while the SLD be am, a steering mirror and a fixed mirror are fixed relative to the scanner frame. The S LD beam, positioned in front of the Z scanner, is aimed at a fixed mirror that is situate d above the cantilever. The mirror reflects the beam downward and onto the back surf ace of the cantilever. The SLD beam will always hit the same spot on the cantilever's surface since the Z scanner only moves vertically. Therefore, once the SLD beam is a ligned, there is no need to realign the SLD beam, even after the Z scanner has been moved up and down to change samples. The steering mirror, located at the front of th e Z scanner assembly, adjusts the reflection angle of the SLD beam to the PSPD. An other feature of this alignment design is that as a result of placing the PSPD onto the scanner frame, it allows a change of the Z scanner position without having to realign t he PSPD. Therefore, only the deflection of the cantilever will be detected, independen t of the Z scanner movement.

Since there is nothing obstructing the view above the cantilever in the structure, t he optical microscope is located on the same axis as the SLD beam that is reflected a t the fixed mirror.



Figure 1-6. Captured Optical Microscope Image

Figure 1-6 shows the cantilever with the SLD beam focused on it, as it is displaye d on the vision software. Since the CCD camera is aligned directly with the cantilever with nothing blocking its view, it is very convenient to focus on or to observe the samp le while moving the camera up and down. This view also provides superb quality for a n optical microscope.

The SLD beam using in NX head has low coherence. This enables accurate ima ging of highly reflective surfaces and precise measurements for approach spectrosco py. An additional advantage the SLD head is its compatibility with experiments that util ize light in the visible region of the spectrum. The unique head design allows for wide open side access to a sample and the tip.

In addition, the head is easily inserted by sliding it along a dovetail rail and autom atically locking into its pre-aligned position with a convenient turn of two thumb locks. There are no additional knobs, springs or cables to adjust as is common with other de signs.

Figure 1-7. Lock Head



The NX system not only achieved a structural design change that yielded exempl ary SPM efficiency, but also brought many improvements to the electronic controller a nd to the supporting software.

The AFM control unit has a fast, powerful Digital Signal Processor (DSP), high sp eed ADC/DACs and offers built-in support for Digital lock-in and digital Q control functi ons without the need for additional instruments.

The NX Control Electronics are designed to enable the scanner, the core unit of t he AFM, to provide efficient, accurate and fast control, and to facilitate the acquisition of a stable image even beyond a scan speed of 10Hz. In addition, the controller contai ns input/output terminals that provide a simple means for users to design advanced e xperiments that extend far beyond and are much more complicated than obtaining ba sic images.

Furthermore, the up to date computer is equipped with the most recent high-pow er Intel Core i5-3570 and Windows 10 system. Two 23" LCD monitor displays crystal clear images using a DVI (Digital Video Interface). All necessary software, including SmartScan[™], the Data Acquisition program, and XEI, the Image Processing program, is installed on the computer. Figure 1-8 shows the SmartScan[™] program's clean and easy-to-use interface, complete with safety functions and various measurement capa bilities that are required to perform advanced applications. Figure 1-9 shows the XEI program that is used to convert acquired data into an image and to perform various a nalyses that meet the user's requirements.



Figure 1-8. SmartScan[™] - Data Acquisition Program




Chapter 2. Components of NX10

The NX10 SPM System consists of four primary components: the NX10 main sys tem; the NX10 control electronics; a computer; and twin monitors.



Figure 2-1. NX10 System

The NX10 main system is where actual measurements are made, and the NX10 control electronics control the movement of the NX10 main system according to the c ommands from the computer. All necessary software, including SmartScan[™] (the Dat a Acquisition program), XEI (the Image Processing program) and the vision program, i s installed on the computer.

2-1. NX10 Main System

The NX10 main system is divided into three components as shown in Figure 2-2. The following sections explain each component in more detail.



Figure 2-2. NX10 Scanning Probe Microcope

2-1-1. Z Stage

The Z stage head assembly controls the coarse vertical positioning of the Z scan ner with a stepper motor, and is used to approach the cantilever near the sample. The max working range of the Z stage is 25mm (max speed 0.8mm/s, resolution 80nm). The Z stage is software controlled.

NX10 Head

The NX10 head is the component which actually interacts with the sample and ta kes measurements. The NX10 provides the Standard Head by default. The Long Trav el Head can be provided optionally. The following table is the specification of the two heads. Please use the head appropriate for your measurement.

Figure 2-3. (a) Standard Head, (b) Long Travel Head,



Table 2-1. Specifications of NX10 Head

Items	Standard Head	Long Travel Head
Compatible Objective Lens	UL10x	UL10x, EL20x
Z Scanner Stroke	> 15 µm	> 38 µm

The NX10 head is a core component of the Park Systems AFM and performs the following functions:

- Cantilever Mount
- Cantilever Modulation
- ➢ Beam Detection
- ➢ Movement in Z axis

Figure 2-4. Structure of NX10 Head



WARNING!

Do not disassemble the NX10 head on your own. Park Systems will not be responsible for any personal, physical damage or degraded performance that may result from unauthorized disassembly.

Probe hand

The probe hand is the part of the AFM head which holds the cantilever. The desi gn of the probe hand depends on the head mode (operating mode) of the AFM. This means that the appropriate probe hand must be selected by the user according to thei r application. The standard probe hand provided with the NX10 system (Figure 2-5) h as a bimorph for vibrating the cantilever in non-contact mode, and can apply an electri cal tip bias. It is usable for contact, non-contact, force modulation, lateral force mode, Tapping mode, approach spectroscopy mode, nano indentation mode, lithography mo de, electrostatic force mode, among others.

Figure 2-5. Attach Probehand



NX10 Z scanner

The Z scanner consists of stacked piezoelectric material, which moves in respon se to applied voltage. The cantilever is attached to the Z scanner through the probe h and. This enables the tip to maintain constant feedback conditions (force or distance) as it is moved over a sample surface. The measurable dimensions are limited by the Z scanner range. The Z scanner on the standard head can move up to 15 μ m and the Long Travel Head's Z scanner can move up to 38 μ m.

WARNING!

Never disassemble the Z scanner on your own. Park Systems will not be responsible for any personal, physical damage or degraded performance that may result from unauthorized disassembly.

Figure 2-6. Z scanner Assembly



The NX10's Z scanner is separated from the X-Y scanner. This independence pr ovides the user with several operational advantages.

- The separated Z scanner is lighter. This allows for quicker response times, enabling the tip to follow the topography of a sample surface more quickly, ultimately leading to quicker measurements without sacrificing accuracy or precision. The faster response time also protects the tip, resulting in the ability to acquire clear images for an extended period of time.
- Since the tip wears out eventually, it is necessary to replace it after some amount of use. The NX10's Kinematic Mount makes tip exchanges routine and easy.
- 3. The beam and PSPD alignment process, necessary for signal acquisition in most SPM modes, becomes easy and convenient. Manageable control knobs on the NX10 head can be adjusted manually with the help of the control software (SmartScan[™] and the vision program), making location and movement of the beam easy and accurate.
- 4. Whenever it is necessary to remove the NX10 head from the main frame, it is Simple to do so. This procedure can be accomplished by unlocking the

dovetail thumb locks and sliding the NX10 head off the dovetail rail. Remounting the head is as easy as removing it.





AFM Beam Detection Array

AFMs collect a beam signal after it is reflected from the back side of a cantilever i n order to detect the probe's movement. The NX10 uses an SLD beam with a wavele ngth of 830nm.





The cantilever and the PSPD move together with the Z scanner while the SLD be am, a steering mirror and a fixed mirror are fixed relative to the scanner frame. The S LD beam, positioned in front of the Z scanner, is aimed at a fixed mirror that is situate d above the cantilever. The mirror reflects the SLD beam downward and onto the bac k surface of the cantilever. The SLD beam will always hit the same spot on the cantile ver's surface since the Z scanner only moves vertically. The steering mirror, located at the front of the Z scanner assembly, adjusts the reflection angle of the SLD beam tha t is reflected off the cantilever's surface. The steering mirror reflects the SLD beam on to the PSPD. The beam alignment knobs, which are located on the head, control the fi xed mirror angle and make it possible for beam to align onto the cantilever's surface a s shown in Figures 2-8 and 2-9. The PSPD alignment knobs in front of the head contr ols the steering mirror.

Figure 2-9. Beam & PSPD Alignment Knobs



2-1-2. XY Stage

The X-Y scanner is mounted on top of the XY stage. The motorized XY stage ca n be used to horizontally position the sample. The NX10 XY stage has a maximum ra nge of 20mm in both X and Y axes, with a resolution of 5µm. The XY stage is software controlled.

NX10 X-Y scanner

The NX10 X-Y scanner that moves the sample in the X-Y plane is a Body Guided Flexure scanner. The X-Y scanner is fabricated from a solid aluminum block. The desi red area is cut out from inside the aluminum block, and the lines indicated in Figure 2-10 are fabricated with a special technique called 'Wire Electric Discharge Machining' r esulting in a flexure hinge structure.



Figure 2-10. X-Y scanner

WARNING!

Never disassemble the X-Y scanner on your own. Park Systems will not be responsible for any personal, physical damage or reduced performance resulting from unauthorized disassembly.

An X-Y scanner with a flexure hinge structure has the advantage of highly orthog onal two-dimensional movement with minimal out-of-plane motion. Due to the Parallel Kinematics design, the X-Y scanner has low inertia and axis-independent performanc e. Hysteresis-correcting Servo Scan (described in Chapter 7) is accomplished by mea ns of an optical sensor in the flexure scanner.

There are two X-Y scanner sizes that may be used with the NX10: 50 μ m × 50 μ m, or 100 μ m × 100 μ m, or 10 μ m x 10 μ m depending on the desired maximum measureme nt range.

Sample Chuck

The sample is loaded on a sample chuck which is fixed on the XY scanner. A ma gnetic holder allows samples prepared on metal plates to be easily loaded onto the sc anner. Voltage Bias from -10V to 10V can be applied to samples through the sample c huck. The chuck can accommodate samples with dimensions of up to 50mm \times 50mm \times 20mm and a mass of up to 500g.

NX10 Vacuum Sample Chuck (Optional)

Figure 2-11. Vacuum Sample Chuck for NX10



Check the vibration noise by applying a sample Bias to the structure attached to the existing sample chamber. The vacuum sample chuck can be mounted on the NX10 sample chuck.

Bolt size: 4-M2.5X6

Installation

- 1. Tighten the 4 mm tube to the Vacuum Sample Chuck.
- 2. Connect the 4-6mm tube using a binocular fitting.
- 3. Connect the 6 mm tube to the System terminal of the Pressure Controller.
- 4. Connect the pump to the Pressure Controller's Pump terminal.
- 5. Connect power to the pump and power it on.

2-1-3. Focus Stage

The optical microscope is attached to the motorized Focus stage, which is control led via software. The Focus stage is used to vertically position the optical microscope so as to observe the cantilever or sample. The NX10 Focus stage has a maximum ra nge of 15mm.

NX10 Optical Microscope

The Optical microscope is used when positioning the SLD beam onto the cantilev er, and for locating regions of interest on the sample surface for measurement. Since the optical microscope's axis is parallel with the Z scanner's, it is possible to have a di rect on-axis view of the cantilever in conjunction with the sample area that will be sca nned. All of the components of the optical microscope - the objective lens, the frame, and CCD camera - are rigidly fixed on a single body. Since the entire assembly move s together for focusing and panning, the axis lining the sample and the CCD camera a re always fixed, and a high quality optical view is preserved. The NX10 provides two options for the objective lens' choice- 10X and 20X. Plea se refer to the table below for details.

Table	2-4.	Specification	of	Objective	Lens
-------	------	---------------	----	-----------	------

EL20 X (Enhanced long working distance objective lens)	UL10 X (Ultra long working distance objective lens)
NA 0.4	NA 0.23
WD 25 mm	WD 50.5 mm
Compatible for Long Travel Head	Compatible for Standard Head, Long Travel Head

The 10X objective lens yields about 500 times magnification and the optional 20X objective lens yields about 1000 times magnification.





2-2. NX10 Control Electronics

The NX10 main system is divided into three components, the NX10 main system, the Control Electronics, and the computer. The Control Electronics serves as a medi ator between the main system and the computer.



Figure 2-12. Control Electronics (Rear View)

In order to maintain fast, effective communication between the computer and the NX10 main system, an Ethernet connection is used. The DSP contained in the NX10 Control Electronics is the 9600MMACS.



Connector Label	Туре	Connection to	Purpose/ specification
Analog	1.27 mm pitch, 68 pin	Frame COMPUTERB	Analog signal I/O (+/-15V , PSPD, Detector, Piezo drive signal, Tip bias , Sample bias, Modulation) for AFM operation.
Motor	1.27 mm pitch, 50 pin	Frame COMPUTERB	Digital signal I/O (+5V, Z motor, Focus motor, Limit sensor, SPI) for XY, Z , Focus Stage control.
Ethernet	RJ45 Cat.5e	COMPUTER	Connect Electronics with COMPUTER for controlling NX 10 system by COMPUTER.
EMG	Circular 4pin		Power for XY, Z and focus stage motor. Motor can be stopped by disconnecting it in an urgent status. This function is disabled for research system.
HV	2.54 mm pitch 15 pin	Frame COMPUTERB	High Voltage Signal for Scanners

Table 2-5. Connector Information

NX Control Electronics supports access of important input and output (I/O) signal s such as VERTICAL and Tip bias to external instruments via BNC connections. The se signals are detailed in Table 2-6.

Connector Label	Purpose	Specification				
AUX 5 IN	Inputs connector for user-supplie					
AUX 4 IN	d signals. External signals are intro duced through these connectors ca n be viewed alongside SPM param					
AUX 3 IN	eters, each being assigned to a cha nnel selectable from the "Input Conf ig" menu. These auxiliary signals ca	DVV 20 KH2, +/- 10V				
AUX 2 IN	n also be captured as an image in XEP or SmartScan for analysis. Th e three signal paths are identical an					
AUX 1 IN	d independent.	BW 5 MHz, 50 Ω input impedance. +/- 5V				
AUX 1 OUT		BW 5 MHz, 50 Ω input impedance. +/- 2V				
AUX 2 OUT		BW 20 kHz, +/- 10V				
Ext. Tip Bias	Input connector used when the experimenter wants to apply bias	Input voltage range: -10V to ~ +10V If experimenter wants to apply				
Ext. Sample Bias	from the external source to the sample.	higher bias to the sample, user can use 'External High Voltage toolkit' Full Power Bandwidth: <100kHz				
TTL OUT	Decorned					
TTL IN	Reserved					
Counter 1	Input connector when the	LVTTL input compliant Minimum pulse width: 10 ns Max counting value: 2032				
Counter 2	signal from detector.	Time constant: 1 ms ~ 1 sec. (To be determined)				
A-B		Output range: -5V ~ +5 V Small Signal Bandwidth: 5 MHz Impedance: 50 Ω				
Alarm	Reserved					
Tip SYNC	Tip bias (sample bias, Z scanner modulation) frequency output.	- LVTTL compliant.				
MOD SYNC	Frequency output of NCM modulation					

Table 2-6. BNC Input/Output Signals

■ Image Sync.

NX10 provides Image Sync outputs (Frame, Line, Pixel) for your experiment. Ex ample Pixel, Line, and Frame signals for a 4×4 pixels image are shown in Figure 2-1 3. The forward (left-to-right) scan order is denoted by numbers, and the backwards (ri ght-to-left) scan order is denoted by letters.

Figure 2-13. Standard Scanning



13	14	15	16
р	0	n	m
9	10	11	12
1	k	j	i
5	6	7	8
h	g	f	е
1	2	3	4
d	С	b	а

<[Dis	spla	ay>																			
DISPLAY		1	2	3	4	a	b	c		1	5	6	7	8	e	f	g	h	9	10	11	12
]] <u>H</u>																				
	1							8 8	10	4	8_1			_								

(H: High Status, L: Low Status)

Table 2-7 explains the meaning of each sync signal.

Table 2-7. Image Sync Signals

Name	Purpose	Purpose Low				
FRAME	Indicates if the images is acquired	Acquiring the image	No activity			
LINE	Indicates the direction of the scanner movement	Trace (or forward) direction	Retrace (or backward) direction			
PIXEL	Indicates if the scanner status	Acquiring the pixel data (hence the scanner is stationary)	Moving to the next point			

2-2-1. Power/Fuse Change

Power

The power to the NX10 Control Electronics is not free voltage. The procedure for changing the input power voltage follows below:



Figure 2-14. Change Power



1. Remove power cord.

230V

- 2. Pry door open at socket.
- 3. Lift and swing door into socket.
- 4. Lift fuse holder out of housing.
- 5. Install one AG fuse or two metric fuses.

240V

- 6. Replace fuse holder into housing.
- 7. Swing and snap door back in place.
- Fuse

[Fuse Specification in: 230V/240V:2A or 100V/120V:4A]

You can change the fuse by following the procedure below:

- a) Removing Fuse Holder
- 1. Insert a pocket screwdriver at point "X" as shown.



2. Gently lift UP until the entire door lifts up at least 1/4".



3. Once lifted, the door will pivot on its hinges and expose the fuse holder.



4. When the fuse holder is installed in the single fuse position, apply the screwdri ver as shown and gently pry up. Insert screwdriver as shown - do not use fingers to pr y the unit loose.



When the fuse holder is installed in the dual fuse configuration, it will release as s oon as the door is opened.

b) Changing FuseEuropean Fusing Arrangement



North American Fusing Arrangement



Install fuses on one side only. Do not install both AG and Metric fuses at the sam e time.

2-3. Computer & Monitor

Programs related to controlling the system - performing measurement and image processing are installed in the computer. They are SmartScan[™], XEI and the Vision program. SmartScan[™] is for system operation, data acquisition and communication b etween control electronics and computer. XEI is for image processing and analysis. T he Vision program is to observe the cantilever/sample/beam/etc for system operation. See the software manual for further description of the software.

The computer is connected to the NX10 Control Electronics via Ethernet cable. T he two 23 inch LCD monitors each provide 1920×1080 pixels with 32 bit color. These monitors are digitally connected to the computer via SVGA DVI (Digital Video Interfac e) port. The specifications for the computer are listed below. (The computer and monit or specifications and configurations may be upgraded without notice.)

2-4. Acoustic and Vibration Isolation System

AFMs are instruments that are very sensitive to vibrations. Both vibrations from th e floor and acoustic noise of the surroundings have adverse effects on AFM measure ments. It is recommended that the NX10 system is placed in an Acoustic Enclosure to block acoustic noise from the surrounding environment, and supported by an Active Vibration Isolation System to block floor vibrations.

2-4-1. Acoustic Enclosure

The Acoustic Enclosure (AE) shields the AFM from acoustic and electromagnetic noise.

Standard Acoustic Enclosure:

Designed exclusively for the NX series system, the integrated acoustic enclosure and granite table isolate the NX system from external acoustic and light noise for an i mproved performance. The walls of the acoustic enclosure are 40 mm thick, consistin g of a 1.5mm stainless steel board, filled with soundproof material. The inner surfaces are covered with ESD coated micro fibers, which won't emit particulates. Acoustic en closure reduces typically over 10 dB of acoustic noise level, varying with frequency. T he Acoustic Enclosure also blocks EMI noise, and is specially coated to prevent electr ostatic discharge. -Dimensions: 700 × 800 × 1,300 mm (outer) -Mass: 300 kg

2-4-2. Vibration Isolation Systems

• Active Vibration Isolation Table:

An Active Vibration Isolation System (AVIS) uses an electromagnetic transducer t o isolate any vibrations generated by the building as well as the system. The AVIS pro vided by Park Systems can block the vibrations in the frequency range of 0.7Hz-1kHz. Vibrations above 1kHz will penetrate the AVIS.

WARNING!

The AVIS can be damaged by shock. Please consult with Park Systems when moving the system.

2-5. Specifications

Scanner	Decoupled XY and Z-scanner Single module flexure XY-scanner with closed-loop control Scan range of XY-scanner: 50 μm (Optional 100 μm), 10 μm Scan range of Z-scanner: 15 μm (Optional 38μm)
Stage	XY stage trave rangel: 20 mm × 20 mm, motorized precision movement Z stage travel range: 25 mm, motorized movement Sample size : 100 mm × 100 mm × 20 mm thickness Sample weight: up to 500 g
Head	Super Luminescent Diode (SLD) : 830 nm with low coherence
Optics	Direct on-axis vision of sample surface and cantilever Focus range: 15 mm, motorized and software controlled Magnification: 780× (optional 1000×) Field of view: 480 µm × 360 µm (for 10× obj. lens) CCD: 1 Mpixel Objective lens: 10× (0.23NA), 20× (0.4NA)
Electronics	Main Control: DSP(9600 MMACS), 100Mbps TCP/IP ADC: 18 Channels 4 high speed ADC channels (50 MSPS) 24 bit ADCs for X, Y and Z scanner position sensor DAC: 12 channels 2 high speed DAC channels (50 MSPS) 20 bit DACs for X, Y and Z scanner positioning Programmable VERTICAL and LFM gain control Integrated light source control 2 channels of 32 bit counter 3 channels of digital lock-in amplifier digital Q-control 20 embedded signal input/output ports 5 TTL outputs: EOF, EOL, EOP, Modulation, and AC bias Maximum 16 data channels Maximum data size : 4096 × 4096 pixels Power: 200 W
Software	SmartScan [™] . Dedicated system control and data acquisition software . Adjusts feedback gain, set point in real time . Step-and-Scan function for programmable imaging XEI . AFM data analysis software
Main body	Dimension: 250mm × 294mm × 450mm (W × D × H)

Chapter 3. Installation

The installation procedure and environmental specifications for the NX10 play a sig nificant role in the safe operation of the system. Since the durability, safety and overall p erformance of the NX10 depend on the environment and proper installation, close attent ion to the following installation environment and procedures recommended in this chapt er are necessary.

3-1. Environment

Facility Requirements	
Room Temperature (Stand By)	10 °C ~ 40 °C
Room Temperature (Operating)	18 °C ~ 24 °C
Humidity	30% to 80% (not condensing)
Floor Vibration Level	VCD (6.25µm/sec)
Acoustic Noise	Below 65 dB
Floor Space (mm)	2440 (w) × 920 (d)
Ceiling height (mm)	2000 or more
Operator Working Space (mm)	2440 (w) × 1200 (d)

Table 3-1. Facility Requirement

Temperature and Humidity

The location of installation should be clean and well ventilated. The maximum acce ptable relative humidity is 80% for temperatures up to 30 $^{\circ}$ C, and decreases linearly to 50% at 40 $^{\circ}$ C.

Vibration and Noise

The NX Series AFM should be installed on a leveled and hard table for efficient ope ration. Further improvements can be achieved by placing the system in an acoustically shielded room, basement, or in lower levels of buildings which are less susceptible to vi brations. It is recommended that the AFM be located near a wall or pillar.

As measurement dimensions decrease, the significance of noise from external vibr ations increases. Since vibrations can flow through cables, system cables should not be stretched taut. The AFM should not be located near fans or other sources of noise.

In addition to mechanical vibrations, AFM measurements can be influenced by acoustic and electromagnetic noise. Therefore, the AFM needs to be installed away from areas w here there is a vast amount of air flow or electromagnetic radiation.

Temperature variance can also contribute noise, and can cause most types of sam ples to expand or contract by a significant scale. The AFM should be installed away fro m HVAC systems or windows.

After determining a suitable location, the Acoustic Enclosure and Active Vibration Is olation System can be used to mitigate any remaining sources of noise.

The Vibration Criteria for NX10 should be below the line labeled "VC-D" on the crit eria plot in Figure 3-1. The vibration level will be improved with the Acoustic Enclosure a nd Active Isolation System. In this case, the Vibration Criteria should be below the dotte d line labeled "VC-C".



Figure 3-1. Vibration Criteria Graph

Electrical Requirements

-The NX10 requires an AC power supply.

- -Power Supply: 100/120 V or 230/240V, Single Phase, 50~60Hz
- -Consumption: 1000VA (max)
- -Ground Resistance: Recommended below 100ohms

Since the NX10 SPM is a highly sensitive equipment, it is ideal to use it with an Uni nterruptible Power Supply (UPS) installed to provide a stable power supply. It is also rec ommended to connect all the ground pins in the power lines to a ground source to reduc e noise.

System Layout

The following table displays system dimensions. The space requirement of the NX1 0 system installation is shown in Figure 3-2.

Dimensions and Weight	Width (mm)	Depth (mm)	Height (mm)
Main body	250	296	465
AFM Controller	450	480	190
Standard Acoustic Enclosure (outer)	700	830	1303
Computer	178	445	448
Monitor	330	585	595

Figure 3-2. Top View of Dimension NX10 with Clearance



3-2. Component List

NX10 SPM Main System

	Item	Qty	Remarks
	NX Standard Head	1 ea	
	XY Scanner	1 ea	
	Objective Lens	1 ea	
NX10	Camera	1 ea	
AFM System	Outer Skin	1 ea	
	Focus Stage	1 ea	
	Z Stage	1 ea	
	XY Stage	1 ea	
AFM Controller	NX10 AFM Control Electronics	1 ea	
	Computer Main Unit	1 ea	
	Computer Power Cable	1 ea	
	Mouse (USB Type)	1 ea	
	Mouse Pad	1 ea	
Computer	Keyboard (USB Type)	1 ea	
	Recovery Booting CD	1 ea	
	McAfee Anti Virus CD	1 ea	
	HDMI to DVI Cable	1 ea	
	Monitor	2 ea	
	Contact mode Cantilever	10 ea	Mounted on Chip carrier
NX10	Non-Contact mode Cantilever	10 ea	Mounted on Chip carrier
Accessory 1	Calibration grating for XYZ	1 ea	
	Sample disk	10 ea	
	Camera Cable	1 ea	
	Analog Cable	1 ea	
	Motor Cable (Digital)	1 ea	
	TCP/IP Cable	1 ea	
NX10 Accessory 2	HV Cable (SR Ver)	1 ea	
_	Electronics Power Cable	1 ea	
	Manual	3 ea	System, SmartScan, XEI
	Data Base & Software Install CD	1 ea	
	Check List	1 set	
NX10	Clip Type Chip Carrier	2 ea	
Accessory 3	Cantilever Exchanger for Clip Type Chip Carrier	1 ea	

	Item	Qty	Remarks
Tool Box	Glue for Mounting	1 ea	Super X
	Tweezer	1 Set	RR-SA & ESD
	Controller spare fuse	1 ea	
	SLD Detector Chip Carrier	1 ea	
	SLD Paper	1 ea	
	Screw Driver	1 set	
	Wrench Set (369-H7) and Hex Driver	1 set	Each 1 set
	Nipper	1 ea	
	Head Supporter	1 ea	
Multi Cord	Multi Cord	1 ea	
Wooden Box 1	NX Head Box	1 ea	
Wooden Box 2	XY Scanner Box	1 ea	
Acoustic Enclosure	Acoustic Enclosure	1 ea	Optional
	Ground Sticker	1 ea	
	Specifications Plate	1 ea	
AVI Table	Mini-450	1 ea	Optional
	Power Cable	1 ea	Optional

3-3. Uncrate

• Use a Phillips screwdriver to peel off the back of a blue vinyl top box and remove the screws that hold the panels in each side.



CAUTION: Two people are required to remove all panels of the crate. Improper lifting can cause muscle strain or back injury.

• After removing the panel discard cut the banding strap wrapped around the product.



- Remove the vinyl using a knife or scissors.
- Collect as much as possible the forks of the forklift is located on the bottom, then place the product on the floor.

3-4. Setup easy guide



Check the voltage on the socket, and make sure that voltage matches the **O** controller voltage inlet setting. Check if the installation location is well-grounded. Make sure the floor noise level is equal to or better than VC-D rating.



2

Set up the PC, monitors and AFM controller on a user-provided table, and then place the A/E^* about 10~20 cm from it. Due to the cable lengths between the controllers and the main AFM body, the total width of the footprint for this setup should be less than 3 meters.





Turn each of the four (4) wheel nuts with the 5mm Allen wrench in the counterclockwise direction to crank down the feet evenly onto the floor. Then adjust the wheels themselves until those are approximately 5mm off the floor.





O Level the A/E by adjusting the feet's height, by using

the 5mm wrench, while monitoring with the Leveling gauge. D Put the AVIT* inside the A/E and adjust the position of the mirror block,

as indicated in the figure, to have it reflect upwards the LCD screen of the AVIT.





Remove the bolts from the A/E back cover using ⁽²⁾ flat-head driver and ⁽³⁾ 6mm wrench. Set the AFM main body on the AVIT and adjust the center of mass located at the center of the AVIT as shown in the figure above.





Disassemble the cable holder, and then connect the cables to the main body. Place the cable holder back. Make sure that only a single cable is fitted into each of the holes within the cable holder. There shouldn't be any cables which overlap within a single hole within the holder.



7

Check the front display on AVIT Mini 450 through the mirror in front of it. Turn on Mini 450, Press the [][ACT] button and the LED on Mini 450 will blink on, the upper table will be floating while the inside motor runs. When the LED is off, the active vibration isolation will begin.





Set-up complete.



3-5. System Setup

3-5-1. Install Acoustic Enclosure

- Place the AE at the installation location. Note that there is a metal foot(1), nut
 (2), and wheel(3) assembly on each of the lower corners of the AE.
- Use a wrench to turn the nut(2) clockwise to lower each foot(1) until the wheels
 (3) are slightly raised off of the floor (approximately 5mm). Do not raise the whe els(3) more than necessary, as they may contribute noise if raised too high.
- 3. Use a spirit level to ensure that the platform is leveled.



Figure 3-3. Acoustic Enclosure Bottom

3-5-2. Remove AE Cover

To install the NX10 main system, you can either completely remove the AE cover, or only remove the AE back panel.

1. Remove Whole Cover

Remove the cover of the Acoustic Enclosure (AE). To do so, use a wrench to turn th e stopper counter-clockwise until it is removed. Carefully take off the AE cover by lift ing the handle on each side and placing it down on the ground.

<u>NOTE!</u>

Be very cautious with the cover; as the cover itself weighs 72kg.At least 3 able-bodied persons should aid in this process.



Figure 3-4. Acoustic Enclosure

2. Remove Back Cover

Unscrew the four bolts marked in Figure 3-5. Pull the handles to remove the back c over of the AE.



Figure 3-5. Acoustic Enclosure Rear

3-5-3. AVIS Setup (Optional)

1. Place the AVIS on the center of the stone tabletop. Install the AVIS according to installation manual provided by manufacturer of each AVIS.

3-5-4. Load NX10 main system

 Carefully, place the NX10 main system on the center of the AVIS. Make sure th at the AVIS is "locked" before placing the NX10 main system on the center top o f the AVIS. (Refer the manual provided by manufacturer of each AVIS to lock th e AVIS.)
Figure 3-6. NX10 placed on AVIS



3-5-5. Load Control Electronics & Computer/Monitor

 Load NX10 control electronics and monitors/keyboard/mouse/COMPUTER on t he desk.



Figure 3-7. Electronics, Monitor, Computer Desk Setup

3-5-6. Cabling

- 1. Required Cables:
 - (a)Camera cable
 - (b)Ethernet cable
 - (c)Power Cable
 - (d)Analog Cable
 - (e)Motor cable
 - (f)High Voltage cable

Figure 3-8. NX10 Main System Cables



- 2. Cabling NX10 main system (Figures 3-9 and 3-10)
- ① Connect the **Camera cable** between the illuminator connector (1) on the back of the NX10 main system and the LAN port of the computer.
- ② Connect the Motor cable between the 50 pin connector (a) on the back of the NX10 main system and the motor connector (A) on the rear panel of the NX10 control electronics.
- ③ Connect the analog cable between the 68 pin connector (b) on the back of the NX10 main system and the analog connector (B) on the rear panel of the NX10 control electronics.
- ④ Connect the High Voltage cable between the High Voltage connector (c) on the back of the NX10 main system and the Voltage connector (C) on the rear panel of the NX10 control electronics.
- S Connect the Ethernet cable between the Ethernet connector (2) on the rear panel of NX10 control electronics and the LAN port of the computer.
- 6 Connect the **power cable** (3) on the NX10 control electronics.



Figure 3-9. Cabling NX10 Main System

Figure 3-10. Cabling NX10 control electronics



Mounting Cables on Acoustic Enclosure

To reduce the vibration noises transferred to the system through the cables, the main body cables should be attached by the Cable Mount (outlined in orange) on the back side of the Acoustic Enclosure.

There should be some slack in the cables between the Cable Mount and the main body to prevent vibrations from entering the system through the cables. However, the cables should not be so loose as to touch the AVIS, in which case they can still conduct vibration into the system.

- 1. Remove the small back cover of the AE by unscrewing four bolts
- 2. Remove the cover of the Cable Mount by unscrewing four bolts.
- 3. Arrange the cables and put the cover back on and fix it using the screws.
- 4. In reverse order, attach the large/small back cover of AE back.





Acoustic Enclosure Cable Mount



Left: Too tight; Right: Too loose

3. Cabling Computer

Please refer to the manual supplied by the computer manufacturer.

3-5-7. Place Acoustic Enclosure Cover Back

1. Place the AE cover back on in reverse order.

<u>NOTE!</u>

Be very cautious with the cover; as the whole cover itself weighs 72kg.At least 3 able-bodied persons should aid in this process.

<u>NOTE!</u>

When carrying the whole cover over the NX-System, make sure to lift it up high enough to clear the top of the NX-System.

3-5-8. Unlock Optical Microscope

When moving the NX10 system from one place to another, it must be locked to the main body to prevent possible damage to the optical microscope. Therefore, after placing th e NX10, the optical microscope has to be unlocked.

Figure 3-11. Unlocking Optical Microscope



- ① Unscrew the bolt labeled (B) on the optical microscope's right lock block while supporting the optical microscope with one hand.
- ② Support and lift down the optical microscope as carefully as possible.
- ③ Loosen the screw labeled (C) slightly in order to move the optical microscope's lock block.
- ④ Place the hole on the lock block over the hole on the (A) and fasten the screw.
- 5 Fasten the screw on the (C) to fix the lock block.

3-5-9. Power On

1. Connect to Power Supply

Connect the NX10 control electronics, the computer and the monitor to a grounded power supply before connecting to power supply. Make sure that all the switches are tur ned off to prevent any damage to the equipment.

2. Power On

Turn on the power supply of all of the NX10 system components.

<u>NOTE!</u>

The NX10 Control Electronics must be turned on prior to the SmartScan[™] Software. Any other component can be switched on in any order.SmartScan[™]

3-5-10. Installation Checkup

1. Run SmartScan™ program

Click the SmartScan[™] icon on the main window screen or in the folder C:\Park Sys tems\SmartScan\Bin. The program will start and you can check to ensure that system i nitialization completes without any error messages. If there is a problem, check whether the power supply is on, and make sure all the components are arranged correctly as sh own in Figure 3-12 Components Setup.





- 2. Check Calibration of Scanners
- 1 Load the standard sample on the magnetic sample holder of the XY scanner.
- ② Take image and check if the dimensions of the standard sample measured from the obtained image correspond to the specification of the standard sample.
- 3. Zero Scan Test
- ① Load a flat sample, such as bare silicon wafer, to the magnetic sample holder of the XY scanner.
- ② Mount a Non-contact cantilever (NCHR) to the probe hand.
- ③ Set the head mode to 'Contact mode' and approach to the sample.
- ④ Set the XY/Z scanner range to 0.2 at part config.

Setup	Part Config	1	×ect
Scan Rate	Cantilever	NCHR	pt
Z Slope	XY Scanner	100µm	, di
Scan Area	Z Scanner	15µm	
>			
Pixels			
Size	2.000 µm 🌲	2.000 µm 🛟	
Offset	0.0000 µm 🗘	0.0000 µm 🤤	
Rotation	0.00 ° 🤤		
	1 + 7 +	>	

Figure 3-13. Part Config window

- ⑤ Set Scan rate to 2 Hz, Gain 0.5, LPF 0, 256×256pixels and take a sample image with Scan Size 0.
- 6 Open the obtained Height image on XEI and flatten using the following conditions.

 $[1^{st} \text{ order } 1\mu \text{m } x \ 1\mu \text{m } \text{fast scan}]$

[2nd Order line by line in both X and Y direction]

⑦ Check the RMS roughness of the processed image. The RMS roughness should be less than 0.5 Å for a properly installed NX10 system equipped with an AVIS and AE.

3-6. System Relocation

① Check the system (Section 3-3-10) before transferring the system.

WARNING!

If you have an AVIS system, it should be in "Lock" mode in order to protect from outside impacts that may occur during shipping or storage. Please refer to the manual provided by the manufacturer for more detailed information regarding the AVIS system.

② Reset the motorized stages (XY, Z and focus) by selecting the [Reset] & [R.Origin] buttons.



Figure 3-14. Reset the motorized Stages

- ③ Put the power of NX10 system [Control Electronics, Computer, Monitor] off.
- ④ Lock the optical microscope by following the Section 3-3-8 in reverse order.
- 5 Lock the AVIT before Turn off the power.
- 6 Disconnect the cables.
- 1 Lift the foot of the acoustic enclosure.
- 8 Relocate the acoustic enclosure to new installation site by pushing it using the wheel on the bottom.

WARNING!

Do not move the acoustic enclosure with the NX10 main system inside it. The system may be damaged if the enclosure encounters uneven flooring. Move the NX10 main system separately from the acoustic enclosure.

Figure 3-15. System Relocation



Set up the NX10 system after finishing the system relocation. Refer to Section 3-3.

Chapter 4. Cantilever Selection

4-1. Cantilever Characteristics

Generally speaking, the term 'cantilever' includes the silicon chip, a cantilever hang ing from the chip, and a tip hanging from the end of the cantilever. Figure 4-1 shows the overall view and the names of the parts of the cantilever used in the SPM (Scanning Pr obe Microscope).





The chip, the cantilever, and the tip are made from Silicon (Si) or Silicon Nitride (Si₃ N_4), and are manufactured using macro-machining techniques.

Because a cantilever has very small dimensions - 10µm width, 100µm length, and se veral µm thickness - it is very difficult to handle in the process of attaching to the SPM. T o make it easier to use, the SPM uses a relatively large chip, the size of several millimet ers.

Figure 4-2 is an SEM image of a cantilever manufactured this way.



Figure 4-2. SEM image of silicon cantilever

The cantilever is the part sensing the surface properties (the topographic distributio n, the physical solidity, electrical properties, magnetic properties, chemical properties, et c.) by detecting the degree of deflection due to the interaction with the sample surface, and is a determining factor for image resolution.

When viewed from the top, the structures of cantilevers are divided into two groups: those with a rectangular shape and those with a triangular shape. Each design has a di fferent force constant depending on the width, depth, thickness, and composition materi al. Among different materials, the Silicon Nitride cantilever is stronger than the Silicon c antilever, but it has some disadvantages:

When the thickness is more than $1\mu m$, contortion may occur.

The curvature at the end of the tip is large – on the order of tens of nanometers. It has a low aspect ratio.

Compared to this, the Silicon cantilever has a tip curvature of less than 10nm, and i s more commonly used. In non-contact mode, which has a high resonant frequency, the rectangular shaped cantilever with a bigger Q-factor, a cantilever with a high force cons tant, is used more than the V shape. The cantilever provided with the NX10 by default is a silicon, rectangular shaped cantilever for use in both contact and non-contact mode.

In addition, the upper surface of the cantilever (the opposite side of the tip) is coate d very thinly with a metal such as gold (Au) or aluminum (Al) to enhance the reflectivity. However, for EFM (Electrostatic Force Microscopy) or MFM (Magnetic Force Microscop y), when the whole cantilever and tip is coated to measure the electric or magnetic prop erties, there is no extra coating on the cantilever.

4-2. Cantilever Selection

There are several types of cantilevers varying in material, shape, softness (represe nted by the spring constant), intrinsic frequency, and Q-factor. The choice of a cantilever from among these is primarily determined by the measurement mode.

For contact mode, a "soft" cantilever which has a small spring constant (typically 0. 01 N/m ~ 3N/m) is chosen. The softer cantilever has a more sensitive response to the ti ny forces between atoms. The probe tip used in contact mode has a thickness of about 1 μ m to achieve a small spring constant. The smaller spring constant results in larger defl ections in response to small forces, and thus provides a very fine image of the surface s tructure.

Cantilevers used for non-contact mode are thicker (~ 4μ m), with a typical spring con stant of 40N/m and a high resonant frequency. In Non-Contact Mode, the AFM vibrates a cantilever near its resonant frequency, and measures the force gradient via the amplit ude and phase shift due to interaction between the probe and the sample. When an AF M is operating in the atmosphere, if the probe tip is situated on a moist or contaminated layer, it may often stick to the layer due to the surface tension of the tip. This happens m ore frequently when the spring constant of the cantilever is smaller. Because of the smal I spring constant, it is difficult to bring it back to the original position. Therefore we need a cantilever with a spring constant which can overcome the surface tension. The sharpe r the tip, the more stable operation can be expected because the surface area of the tip and the surface tension are reduced.

Selecting the proper cantilever depends partly on the morphology of a sample's sur face. For example, when the tip radius is bigger than the features of a sample, the tip sh ape will influence the resulting image, as shown in Figure 4-3(b). Therefore, a tip sharpe r than the smallest sample features should be selected in order to avoid these artifacts. Sharper tips, however, have shorter life times and are more expensive than general-pur pose cantilevers. The standard cantilevers have a tip radius of 10nm.





Measuring a sample twice before and after rotating it relative to the sample stage al

lows a user to determine if there are any tip-shape artifacts in images. If such artifacts a re present, one will see image features with the same orientation in both scans. Howeve r, if the original image is a true representation of the sample surface, then every feature within the images will appear rotated along with the sample.

4-3. Cantilever Mounting

Cantilever chips must be mounted on chip carriers before use. Park Systems provi des various types of chip carriers for different measurements. Both pre-mounted and un mounted cantilever chips are provided. If your cantilever chip is not mounted onto a chip carrier, you must do so with adhesive using glue type chip carriers or with clip type chip carriers. Once your cantilever chip is on a chip carrier, you can simply attach it to the pr obehand, where it will be held in place by magnets.

4-3-1. Glue Type Chip Carrier

The cantilever chip is attached onto the marked area on the glue type chip carrier (Figure 4-4) using glue. There are various glue type chip carriers:

Figure 4-4. Glue Type Chip Carrier



- Standard Chip Carrier
- Ceramic Chip Carrier for SThM
- Ceramic Chip Carrier for SCM
- Teflon Coated Chip Carrier for CP-AFM
- Teflon Coated Chip Carrier for EC-Cell

Required Components

The following items are required to load un-mounted cantilever chips in general.

- Glue type chip carrier
- Instant adhesive for metal

(Cyanoacrylate (superglue) adhesives provided with NX system are recommended)

- Un-mounted cantilever chips

How to Load Un-mounted Cantilever Chip

- ① Remove any dust from the chip carrier.
- ② Pour some glue onto any flat area. Use a small stick, such as a toothpick, to place a dab of the adhesive on the chip carrier. The two small points (or grooved lines) on the chip carrier should be used as guidance for aligning the cantilever chip.

Figure 4-5. Loading Cantilever Chip on Glue Type Chip Carrier



- ③ Place the cantilever chip on top of the adhesive using forceps and align the edge with the two small points (or grooved lines).
- ④ Gently press down on the chip for several seconds.

Figure 4-6. Cantilever Chip Positioned on Glue Type Chip Carrier



<u>NOTE!</u>

You should allow several hours for the adhesive to completely dry; otherwise, Non-Contact mode images may be affected.

<u>NOTE!</u>

Make sure that the cantilever chip is attached the right way up. If necessary, reattach the cantilever chip.



Figure 4-7. Correct Mounting of Cantilever Chip

4-3-2. Clip Type Chip Carrier

Using the clip type chip carrier, an unmounted cantilever chip can be easily stabiliz ed without glue. Figure 4-8 shows the structure of the clip type chip carrier.

- Chip Mount: Cantilever chip is placed here. (Size: 1.7mm × 2.55mm, 0.2mm thickness)
- Clip: Holds cantilever chip.
- Lift Hole: Meets with Cantilever Exchanger Pin. Pressing down this hole will open space between the Clip and Chip Mount area for mounting.
- Round Hole & Slot: These two Hole & Slot will be mounted on the probe hand.
 They will guide Clip Type Chip Carrier to be placed on the probe hand in consistent position.

Figure 4-8. Structure of Clip Type Chip Carrier



The chip type chip carrier is coated with chromium, is designed for various environ

ments such as air, liquid, wiring, and does not need electrically conductive glue to be co nnected between the cantilever and chip carrier electrically.

Required Components

The following items are required to load un-mounted cantilever chip to standard clip type chip carrier.

- Standard Clip Type Chip Carrier
- Cantilever Exchanger
- Un-mounted Cantilever Chips

1. Cantilever Exchanger

There is a round hole, visible when the upper base is uncovered. When you overlay the chip carrier hole above the pin located on the chip carrier mount part of the cantilev er exchanger and press the upper base of the cantilever exchanger, then the bottom par t of chip carrier will go down and the clip will be opened. Then, the un-mounted cantilev er chip can be easily placed.





There is an adjustment screw on the upper base of the cantilever exchanger to mo ve the upper base up or down. Turning the screw clockwise moves the upper base dow n and keeps the clip opened. Turning the screw counter-clockwise moves the upper bas e up and keeps the clip closed.

How to Load Un-mounted Cantilever Chip

- 1 Lift up the upper base of the cantilever exchanger.
- ② Place the clip type chip carrier on top of chip carrier mount. Make sure that the round hole on the bottom of the chip carrier is overlaid on the chip carrier mount pin.



Figure 4-10. Placing Clip Type Chip Carrier on Cantilever Exchanger

<u>NOTE!</u>

The cantilever may not be mounted correctly if the cantilever chip isn't sufficiently aligned on the Cantilever Exchanger.

③ Close the upper base of cantilever exchanger and turn the adjustment screw clockwise to open the chip carrier's clip.

Figure 4-11. Adjust Clip Position



- ④ Pick an un-mounted cantilever chip using tweezers and place it on chip loading place of the cantilever exchanger.
- $\ensuremath{\mathbb{5}}$ Slide the cantilever chip into end of the chip groove on the chip carrier.

Chip Groove

Figure 4-12. Mount Cantilever Chip



Make sure that the cantilever chip is placed the right way up. If necessary, reinsert the cantilever chip.





6 Turn the adjustment screw of the cantilever exchanger counter-clockwise to lift up the upper base.

4-3-3. Chip Carrier Mount

Mount the chip carrier with a cantilever chip to the probehand.





There are two holes in a chip carrier; a round hole and an elongated slot. When yo u overlay the two ruby nodules located on the end of the probe arm with these holes, th e cantilever chip will be attached into place by a magnet, and the position of the cantilev er will be firmly fixed in one position.

4-4. Cantilever DB

The cantilever DB stores specifications for each cantilever type. The SmartScan[™] software comes preloaded with database entries for cantilevers shipped with the system. The user can specify which cantilever is in use in SmartScan[™] through the Setup men u: [Line Scan Click>Setup>Cantilever]. If an entry doesn't exist for your cantilever, you can create one with the following steps:

[Step]:

Create Cantilever DB -> Input Cantilever Spec -> Calibrate VERTICAL Sensitivity

4-4-1. Create Cantilever DB

- 1. Turn off the head.
- Open the 'SmartScan[™] Part Selection' dialog by clicking [Line Scan Click>Setu p>Cantilever] on SmartScan[™].
- 3. Clicking the [Advanced] button will display the 'Create Part' panel. In this panel, select 'Part Type' as 'Cantilever'.
- 4. Write a name for your cantilever in the blank space on the left of the [Create] bu tton, and click the [Create] button. It creates a new cantilever DB using the curr ently selected cantilever DB and switches to the newly created cantilever DB.

innels		Select Cantilever		🍙 ×
		DT_NCHR	NCHR	remove
		LFMR	Resonance	330 KHz
		MFMR Multi75E_G	Force Const. Sensitivity	ant 42 N/m 9.887 V/µm
		NCHR	Length	105 µm
Part Config	🐚 ×	NCLR	Height	15µm
Cantilever	NCHR	NCSTAU NSC14_Co_Cr		
XY Scanner	100µm	NSC14_Cr_AU NSC14_SI3_N4		
Z Scanner	15µm	NSC14_TI_Pt		
(NSC15 NSC18_Co_Cr	Create New (Cantilever
2.000 µm 🗘 😑	2.000 µm 📰 🚦	Search		
0.0000 µm ‡ 0.00 ° ‡	0.0000 µm 🗘	Select		

Figure 4-15. Create Cantilever DB

<u>NOTE!</u>

Before you create the cantilever DB, it is recommended to select the cantilever type with a similar force constant since the cantilever DB is created by copying the previous selected one.

4-4-2. Input Cantilever Specification

1. Turn on the head and switch to 'Maintenance' mode by selecting [Mode>Mainte nance Mode].

<u>NOTE!</u>

The default password is set to 'probe' .

- 2. Go to 'Cantilever Calibration' by clicking [Mode>Calib Mode>Cantilever].
- Write the resonance frequency range (Minimum Frequency, Maximum frequenc y), the typical resonance frequency and the force constant. Refer to the cantilev er specification sheet provided by the cantilever manufacturer.
- 4. Save the input values by clicking the **Apply** button.



Figure 4-16. Input Cantilever Specification

4-4-3. Calibrate VERTICAL Sensitivity

VERTICAL sensitivity is the calibration factor between the deflection of the cantilev er and the movement of the reflected beam on the PSPD. In contact mode, this PSPD p osition is converted to a distance deflected by the cantilever using the VERTICAL sensit ivity calibration. That deflection is then converted to a force in Newtons using the spring constant of the cantilever stored in its DB file.

For Force (V), F=Sx

For Force (N), F=kx

(F: Force (N or V), k: Force constant (N/m), x: Deflection (m), S: VERTICAL Sensitivity (V/m))

VERTICAL sensitivity is obtained by taking a FD (Force vs. Z scanner displacemen t) curve. Before this curve can be taken accurately, however, one first needs to calibrate the AFM's Z scanner and the force constant of the cantilever

1. Taking an FD curve (in contact mode) on a bare Si wafer sample with your canti lever.

*FD Curve

- a) Approach your cantilever to the sample.
- b) Go to FD spectroscopy by selecting [Mode>Scan Mode>FD Spectroscopy] or the icon.
- c) Add a point on the scan image.
- d) Set the parameters on FD spectroscopy control window.
- e) Perform FD spectroscopy by clicking the [Acquire] button.
- f) Zoom in the region that has a linear slope by dragging the mouse after acquiring the FD curve and click the [Apply] button. Then, the Z scanner moving range (Min, Max) is automatically changed to one in the selected region.
- g) Perform FD spectroscopy again by clicking the [Acquire] button.

(For more information about FD Mode, Please refer to 10-10-1.)

2. Switch to 'Maintenance' mode by selecting [Mode>Maintenance Mode].

<u>NOTE!</u>

The default password is set to 'probe '.

- Go to 'Cantilever Calibration' by clicking [Mode>Calib Mode>Cantilever] or by cl icking the icon.
- 4. Select 'Sensitivity' in the 'Cantilever' tab.
- 5. Open the curve you obtained in FD spectroscopy. If it was the last FD curve obt ained with this system, it should already be open.
- 6. Select the linear area in this curve by clicking and dragging on the mouse.
- 7. Click the [Calculate] button to calculate VERTICAL sensitivity value.
- 8. Click the [Calibrate] button for VERTICAL Sensitivity to apply the value obtained in the previous step. Click the **Apply** button to make the calibration permanent.

Figure 4-17 shows a labeled image of SmartScan[™], showing some of the steps for VERTICAL sensitivity calibration.



Figure 4-17. VERTICAL Sensitivity Calibration

4-5. Cantilever Storage

When cantilevers are kept in ambient conditions with variable temperature and hum idity for long periods, their reflected beam intensity can decrease due to oxidation of the cantilever coating material. It is also possible that the end of the tip can become damag ed. For these reasons, it is recommended to store cantilevers in a desiccator

4-6. SLD Detector Chip Carrier

When the SLD beam is aligned to the Infrared Ray Detector, it is visible to the naked eye. The beam falls on the same location as the cantilever when using the Standard Chip Carrier, marked by the \uparrow arrow in the figure above, so it is easy to position the SLD beam onto the SLD detector.

Figure 4-18. (Left) Beam position when using SLD detector Chip Carrier, (Right) Beam position when using Standard Chip Carrier.



Attachment

There are two holes in the chip carrier, a round hole and an elongated slot. Overlay the two ruby nodules located on the probehand with these holes. The detector chip carrier will be attached into place by a magnet, and its position will be firmly fixed in this one position.

Figure 4.19. (Left) Standard Probehand, (Right) with the SLD detector chip carrier attached.



Usage

- 1. Attach the detector chip carrier on the probehand (4-19).
- Position the SLD beam on the location marked ↑ by adjusting the beam alignmen t knobs until the SLD beam becomes visible (4-20).



Figure 4-20. SLD beam on the Detector Chip Carrier

- 3. Re-attach the chip carrier and cantilever after removing the detector chip carrier.
- 4. Move the SLD beam upward or downward while turning the Y beam alignment kn ob (large knob on the left side of head), since the SLD beam has been located on the cantilever position in X axis but with an offset in Y axis, depending on th e cantilever type (Figure 4-18).

Note!

When using the SLD detector Chip Carrier, please turn off the illuminator.

Chapter 5. Operation Procedure

5-1. Basic Procedure

[Step] Following is procedure for Operation measurement:

Power On -> Load Sample -> Remove Head -> Load Cantilever -> Find Cantilever -> Align Beam on Cantilever -> Center Beam on PSPD -> Approach Tip to Sample

5-1-1. Power On

 Turn on the components of your NX10 system [Computer, Monitors and Control Electronics]

<u>NOTE!</u>

The Control Electronics must be turned on before the SmartScan[™] Program is started; otherwise, you will receive a initialization error message and will need to restart SmartScan[™].

 The NX10 is operated by the SmartScan[™] software. When you click the SmartSc an[™] icon in the desktop or in C:\Park Systems\SmartScan\bin of your computer, y ou can start SmartScan[™], the software program for controlling NX10.



Figure 5-1. SmartScan™ User Interface of NX10

Workspace

5-1-2. Load Sample

- 1. Mount sample onto the sample plate using tape or glue. If possible, the best method is to glue the sample on using hard-setting instant adhesives.
- Load sample on the magnetic sample holder. If the sample is large, unscrew the magnetic sample holder from the XY scanner and place sample directly on the Sample chuck at the XY Scanner.
- Locate sample underneath the probe hand using the XY stage control.
 See 'Motorized Stage Control' section for more information on stage control.

<u>NOTE!</u>

Before you use the XY stage pad, be sure to lift the tip off the sample by using the Z stage control pad.

WARNING!

When the Z scanner's arm and the sample are very close, a rapid movement of the Z scanner may cause the scanner's arm to collide with the sample. This may result in severe damage to the probe tip, the sample, and/or the scanner itself.

<u>NOTE!</u>

The cantilever depiction in the Z Stage pad is not the center bar. Treating it as the center bar will result in unintended movement of the Z Stage.

*XY Stage Control Window

For the NX10, you can see both XY and Z/F stage pads on the screen. If there is no "X-Y stage control window" on your computer screen, you can open this window by clicking the X-Y stage icon .



The XY stage pad is used to move the tip around the sample surface before you take an image. The XY stage can be moved in both the x and y directions, which moves the sample relative to the probe tip. The XY stage pad controls both the direction and the speed of the XY stage. Move the cursor with your mouse and click the cursor where you want to get images in the X-Y stage pad, then the X-Y stage will move in the opposite direction so that the NX head move to the defined location. The red point represents the position of the NX head, and you can see its movement by watching this point. This allows for convenient repositioning of the NX head around the sample surface. To increase the speed of movement, click the cursor further from the center cross on the XY stage pad.

*Z stage and Focus stage Control

The Motor Control window contains the Z Stage and Focus components. These control pads are used to lower or raise the Z Stage and Focus Stage.

Clicking above the center bar will raise that stage, and clicking below the center bar will lower it. The speed depends on how far away from the bar you click. The NX10 Focus stage's movement is synchronized with that of the Z stage. Therefore clicking Z stage pad will move the Z stage and the Focus stage together. However clicking Focus stage pad will move the focus stage only.



WARNING!

Be cautious when loading large modules such as EC Cell or ULC onto the NX sample chuck. They may collide with the motorized XY stage when it is moved through a large range, and the module and/or the NX system may be damaged.

5-1-3. Cantilever Mount

Remove Head from the NX10 Main System

- Confirm that the head has clearance. If it is too close to the sample, raise the Z st age. If the Focus stage is too close to the head, raise the Focus stage.
- Turn off the AFM beam switch and unlock the dovetail locks on the sides of the he ad. Disconnect the head from the connector of the NX10 main system. Then, slid e the head out to the right.



Figure 5-2. Removing NX10 Head

Load Cantilever onto Probehand

 Mount cantilever onto the probehand on the head. To ensure no damage to the ca ntilever occurs, it is recommended that the cantilever chip mount is held between your thumb and your index finger. The alignment is simple since two holes on the chip mount should fit directly over the two ruby balls on the magnetic tip holder on the probe hand.

Figure 5-3. Load Cantilever onto Probehand



2. The center of the camera on the monitor screen is the approximate position of the last user's cantilever, and therefore the beam. It is very important to NOT adjust the optical alignment knobs in order to make Section 6-5 much easier.

Attach Head to the NX10 Main System

5-1-4. Select Head Mode/Cantilever

- Attach the head to the NX10 main system in inverse order of Figure 5-2 Turn off the Line scan by clicking the Head mode box to select Head mode and th en clicking the Setup box in the SmartScan[™] software.
- 2. Select the desired Head mode in this SmartScan[™] Part Selection dialog. Then, turn on the Line scan.





5-1-5. Find Cantilever

- Move the focus stage down nearby the head using the focus motor control in the software. As you click upper or lower position from the center bar on the stage pad, the stage will move up or down. If you cannot move the focus stage, please move down the Z stage a little in maintenance mode (Mode->Maintenance mode) and try again.
- 2. Turn the X and Y optical alignment knobs to find the cantilever on the camera. Pay close attention to how much you turn in order to perform Step 5.
- 3. The cantilever is on the cantilever chip and the cantilever chip is mounted on the chip carrier. Therefore, you only need to focus on the cantilever.

*When you cannot find the cantilever

a) Move the head towards the sample, using the Z stage motor control in SmartScan[™], as close as possible by visually monitoring the cantilever-sample separation. If the head cannot move down after the error message "Laser intensity is too low" appears, change the mode to maintenance mode. [Mode ->Maintenance mode]

WARNING!

Be careful when controlling the Z stage in maintenance mode as the interlock f unction to protect the system against head crashes is deactivated. Crashing the ca ntilever into the sample may heavily damage the XY or Z scanners.

b) Focus on the sample surface using the Focus stage motor control in SmartScan[™].
 Find the cantilever chip shadow by the illuminator, using the X and Y optical alignment knobs.



Left: Sample Surface, Right: Cantilever Shadow on Sample Surface

c) Lift the Focus stage up, and you can see the cantilever chip substrate. Then, move the optical alignment knobs to position the cantilever on the center of the vision program.



Figure 5-5. Focus On Cantilever

5-1-6. Align Beam on Cantilever

The AFM obtains images of the sample surface by detecting the bend of the cantilever using the position of a reflected laser beam since these deflections are too small to detect directly. Align the laser beam on the backside of the cantilever.

Remember how much you turn the X and Y optical alignment knobs to find the cantile ver on the camera. Repeat the EXACT translation, but with the beam alignment knobs. De pending on how accurate your movements are, the beam should be near the edge of the c antilever or cantilever chip substrate.

- Focus on the Cantilever Chip Substrate: It is easier to find the beam by placing it o
 n the edge of the cantilever chip substrate with the Y beam alignment knob since it
 s area is large.
- Find Beam Spot: While turning the Y beam alignment (large knob on the left side o f head) CLOCKWISE, you should see the beam spot move UP on the cantilever. A bright spot (see Figure 5-6) appears when the beam hits the edge of the cantileve r chip substrate.
- Position Beam Spot on Front Half of Cantilever: With the beam spot on the edge o
 f cantilever substrate, turn the X beam alignment knob. You should see the beam
 spot move along the edge of the cantilever chip substrate.

<u>NOTE!</u>

If you don't observe ALL of the above, then the spot you see on the cantilever is not the direct laser beam.

Figure 5-6. Focus On Cantilever



*When Beam spot cannot be found: You can easily find the laser using the IR chip carrier.

5-1-7. Align Beam on PSPD

The reflected laser beam from the cantilever travels to the PSPD (position sensitive p hoto detector) where its position is tracked and this data is then fed into a feedback loop c ontrolling the vertical motion of the Z scanner. This feedback is active at all times when the AFM is powered on. This detection scheme allows cantilever movement smaller than an a tomic radius to be measured by the AFM.

Align the reflected laser beam from the cantilever to the PSPD by controlling the PSP D alignment knobs.,

- 1. Turn Y PSPD alignment knob to maximize INTENSITY.
- 2. Turn X PSPD alignment knob to maximize INTENSITY.

<u>NOTE!</u>

Align the PSPD to find the maximum INTENSITY signal. The easiest way to do this is to change only one alignment knob at a time (either the X or the Y) until a maximum is found, then move to the other knob and repeat the process. If you adjust both knobs at once, the process will be difficult.

CAUTION!

If the PSPD alignment knob is too tight or loose during adjustment, the Sus ball between the knob and mirror can fall out. The laser beam will not align to the PSPD when the knob is too tight or too loose.

3. Repeat steps 1 and 2 until beam intensity is maximized (2-3V when the backside of cantilever is metal-coated as general) on the PSPD.

<u>NOTE!</u>

Sometimes if the backside of the cantilever is rough or coated, INTENSITY can be smaller or bigger. In general, when the cantilever surface is not coated with metal, the INTENSITY value is closer to 1V because of the difference in surface reflectivity.

4. When INTENSITY is maximized, turning the X PSPD alignment knob (small right k nob on front of head) CLOCKWISE will move the red spot on the PSPD to the LEF T. Turning the Y PSPD alignment knob (small left knob on front of head) CLOCK WISE will move the red spot on the PSPD UP. By adjusting the knobs, position th e beam spot (red spot) on the center of the PSPD in SMARTSCAN[™] so that VER TICAL, LATERAL value is smaller than ±0.5V.

*When INTENSITY value on PSPD is too small:

Even if the VERTICAL value is within the acceptable range, if the INTENSITY value is too small, it may be difficult for the beam to approach the center of the PSPD and for the tip to approach the sample properly. Therefore, a proper INTENSITY value should be obtained before adjusting the VERTICAL. If the INTENSITY is too small, then the beam path depicted in the figure below is not optimized. In this case, by using the IR detector card, check if the beam is located in beam path. After adjusting the direct beam spot on the PSPD, proceed to the PSPD alignment procedure.



<u>NOTE!</u>

Turning the X or Y PSPD alignment knob can make INTENSITY (beam intensity) suddenly smaller. In this case, stop turning the X or Y PSPD alignment knob and turn it the opposite direction.

5-1-8. Approach Tip to Sample

1. Set Point Setting:

a) Contact Mode

- Set Point in Contact Mode: Reference force for Z feedback
 - As the cantilever and sample's surface get closer, the repulsive force between tip and sample grows bigger, the cantilever bends more and PSPD' s VERTICAL value changes. We can calculate force between the sample's surface and cantilever through changed VERTICAL value. For Z scanner feedback loop, the specific force is chosen, called the 'Set Point' in Contact mode.
- Contact Mode uses the repulsive force that actually makes physical contact between the tip and sample's surface. In other words, applying the Set Point more means that the tip pushes on the sample stronger.

b) Non-contact Mode

- Set Point in Non-contact Mode: Reference amplitude for Z feedback
 The cantilever vibrates after setting the drive frequency and drive amplitude
 to resonant frequency. As the cantilever and sample's surface get closer, the
 attractive force between tip and sample increases and the cantilever's
 vibration amplitude will decrease. For the Z scanner feedback loop,
 the specific amplitude is chosen, called the 'Set Point' in Non-contact mode.
- Drive amplitude is changed depending on the distance between the tip and sample's surface. Therefore, the 'Set Point' also is the distance between the probe tip and the sample surface.
- <u>Perform frequency sweep (NCM ASetup)</u>. Please make sure the selected <u>frequency is within the range of resonant frequency</u>. <u>The amplitude of the selected frequency (red cross)-drive amplitude is</u> <u>recommended to be set near 20nm</u>.
- You need to adjust the drive amplitude depending on your sample.
 For example, setting the drive amplitude over 20nm might be more effective when the sample is fragile and/or consists of strong adhesion force.

2. Check Desired Position on Sample:

a) Bring tip a few millimeters from the sample.

- Move head towards the sample using the Z-stage motor control in the software.
 Visually inspect the tip-sample separation. Stop when the tip is a few millimeters from sample.
- Focus on cantilever.

b) Bring tip 50-100 µm from sample.

- After the cantilever is focused, lower the focus stage until the sample is focuse d in order to check the sample surface.
- Lift the focus stage until the cantilever is focused then and lower the focus stage about 50~100µm from the cantilever.

- Move the Z stage slow down until the sample surface comes into focus, and the distance between the cantilever and the sample is approximately $50 \sim 100 \mu m$.

- Focus on the cantilever using the focus stage motor control in the software.



Figure 6-7. Focus on Sample/Cantilever Positon

WARNING!

If the cantilever and sample are in focus at the same time, the tip has crashed into the sample. The cantilever and sample should NOT be in focus at the same time.

3. Approach

- Click "Approach" (underneath the Z stage motor control). When the light to the right of the motor controls stops blinking, the tip's approach is complete.
- The upper half of Z scanner bar in the PSPD window will be green if

"Approach" is successful. Before approaching, it is recommended to set the scan size to 0 and Z servo gain to 1 in the scan control window.


CAUTION!

Don't perform a frequency sweep after approach.

CAUTION!

Don't move the XY position using XY stage after approach.

CAUTION!

Don't turn off the beam after approach.

5-1-9. Imaging

1. Channel Config Setting

- Select the desired input signals in Channel Configuration [Setup-> Channel Config].
- If the desired input signal isn't shown in the main panel of Channel Config, Clic k the [___] button to see hidden signals or use the channel search feature at t he bottom right portion of the window. The selected signals in the popup dial og will be displayed on the main panel of Channel Config.

Channel Config					<u> </u>	×	
Selected Channels		4 sele	cted		Available Channels		
Lateral (C-D / LFM)			~	reset	Vertical (A-B)		
Z Drive		μm	~	clear	Lateral (C-D / LF		
Z Height		μm	\checkmark	clear	Intensity		
Error Signal		nN		add	Force		
					NCM Amplitude		
					NCM Phase		
				Tip Bias			
					Sample Bias		
			preset	Lockin1 I			
					Lockin1 Q		
Details '7 Height'					Lockin2 I		
Low Pass Filter		0.0%			Lockin2 Q		
					Lockin3 I	•	
Flatten No	ne •				Lockin3 Q	•	
Plane Fit	Enabled						
Apply		ľ					

Figure 5-8. Channel Config

- **Z Drive**: Calculated Value from the driving voltage to the Z scanner in the feedback system. Considered as the height information of the sample surface since the Z scanner is in feedback to respond to the sample height.
- Error Signal: PSPD(VERTICAL)-SetPoint. The feedback loop works this signal to be 0. During the scan, this signal returns to 0 rapidly in good feedback but slowly in poor feedback. For such reason, through this signal, the feedback status can be confirmed, and it is recommended to monitor the Error Signal during the parameter setting.
- Height: Z scanner's actual movement from the sensor directly. Considered the height information of the sample surface since the Z scanner is in feedback to respond to the sample height.
- NCM Amplitude: Amplitude of cantilever vibration in NCM. This signal is maintained to be constant in the feedback loop. During the scan, it returns to the reference amplitude rapidly in good feedback but slowly in poor feedback. For such reasons, through this signal, the feedback status can be confirmed. It is recommended to monitor this NCM amplitude during the parameter setting in NCM and TAPPING MODE.
- **NCM Phase**: Phase of cantilever vibration in NCM and TAPPING MODE. This signal is sensitive for elasticity and viscosity of the surface. These properties can be displayed on phase imaging. It is recommended to acquire this signal in NCM.
- Lateral Force: value in PSPD containing the cantilever's twisting
- **Force**: Calculated force from value in PSPD. Refer to Section 4-4-3 for detail information.
- VERTICAL: value in PSPD containing cantilever deflection information

2. Parameter Setting

- Input a value in the Scan Size field found in the Scan Area tab. This will be the size of your image. The XY Scanner will begin moving back and forth using t his value. This movement may be visible on the vision program.
- Select a value in Scan Rate field. Keep in mind that the speed of the tip will be (2 * Scan Size) / (Scan Rate), and that too high speed will result in tip and s ample damage. Observe the input signal of Height and Z Drive Channels in Scan Traces Window.

- Adjust the value in Z Gain field found in Z Servo tab until the trace line is stable. If this is difficult, try lowering the Scan Rate.

An abridged list of the parameters in the Scan Control Windows follows.

For a complete list, see the SmartScan[™] Software Manual.



Figure 5-9. Proper Gain (top); Noise from Excessive Gain (bottom)

- Scan Traces auto scale Cursor → Hold Cu
- **Repeat**: If selected, the system will activate the text display to the right. Enter a number between 2 and 1000 to repeat the scan the specified number of times.
- Retract Z after Done: Checking the Retract Z after Done check box will lift the cantilever away from the sample 100µm using the Z stage control once the image has been acquired.
- Offset X, Y: Specifies the center of the scan area in a relative coordinate system with (0,0) being the center of the X-Y scanner.
- Rotation: Allows the direction of scanning to be changed within the range of 180°~+180°. Rotation can be set by adjusting the value in rotation field or using the icon buttons below.



- **Z Servo**: Select Z scanner feedback on/off. The Z Servo must be on for most SPM modes.:
- Z Servo Gain: Controls the sensitivity of the Z scanner feedback loop. If this
 value is too high, the Z scanner will oscillate, producing noise in the image or line
 scan. If it is too small, then the SPM probe will not track the sample surface
 properly.
- Set Point: In Contact mode, specifies the force that will be applied by the end of the tip to the sample surface when the system is in feedback. In Non-Contact mode, the set point represents the amplitude of tip oscillation.
- **Tip Bias**: Controls the voltage applied to the tip when EFM or C-AFM modes are used.

3. Acquire Image

- Once the trace line is stabilized, click the "Scan" button to start the measurement.

4. <u>Finish</u>

 After the imaging is complete, set the Scan Size to zero in order to avoid dama ge to the tip and sample and lift the Z stage. Then close the SmartScan[™] pr ogram and turn off NX10 control electronics.

5-2. Sample Loading

5-2-1. Lift NX10 Head

First, raise the head high enough so that you have no difficulties in loading the sample onto the X-Y scanner.

CAUTION!

If the head is not rasied high enough, the sample or the cantilever may be damaged.

5-2-2. Load Sample on XY scanner

Generally, the sample is fixed to a magnetic sample plate prior to imaging using glue or tape. The sample plate is then placed on the magnetic sample holder.



Figure 5-10. Magnetic Sample Holder

When the sample is larger than a sample plate, remove the sample chuck by turning it counter-clockwise. The sample can then be placed directly on the sample chuck. Resulting images may display drift, as the sample is not securely fixed.

Instant Adhesive

You can fix the sample on a sample disk using an adhesive. Hard-setting adhesives s uch as cyanoacrylate glues are recommended; otherwise, the sample may move significan tly during the imaging procedure. When the sample is glued on a metal sample plate using electro-conductive adhesives, such as silver paste, it will be grounded through the magnet ic sample holder but will take a longer time to dry.





Таре

You can fix the sample using adhesive tape. Using tape to fix the sample is convenien t but may result in sample drift during imaging. Using a single strip of double-sided tape wi Il reduce drift, but the adhesive layer on any tape will move significantly on a nano scale ov er time.





When the sample is a film, the user should be especially careful when mounting it bec ause unintended gaps between the tape and sample can allow the film to move during sca nning.





5-3. Operating Concept

5-3-1. Basic Operating Concept

The NX10 scanner is separated into an X-Y scanner and a Z scanner instead of the si ngle piezoelectric tube scanner used in most other SPMs. The X-Y scanner moves the sa mple in the horizontal direction for the range you want to image. The Z scanner moves the cantilever in the vertical direction to trace the morphology of the sample. These independe nt movements of the XY direction and the Z direction are combined to make a three-dimen sional image.

Two measurement types for XY scanner, 'Closed Loop' and 'Open Loop', are possible depending on the status of the XY Servo scan. 'Closed loop' refers to when the XY Servo scan is "ON" and 'Open loop' is when the XY Servo scan is "OFF".

XY Servo Cor	ıfig			🐚 ×
Mode	• on O	off 🔾 I	Hold	
Gain X		0.40 💲		
Cut Off X	24	0 Hz 🌲		
1				
Apply		* 5	0um, Resor	ance = 450 Hz

Figure 5-14. XY Servo scan is ON

In general, piezoelectric materials display nonlinear behavior in response to an applie d voltage. Therefore, the scanner, which is made of a piezoelectric material, displays nonli nearity and hysteresis (Refer to Chapter 1). When the scanner's range of motion increase s, nonlinearity and hysteresis can be calibrated by means of hardware corrections.

In the NX10 system, detectors are used to measure the actual movement of XY or Z s canners. This information is compared with the desired movement, and discrepancies are corrected for by modifying the voltage applied to the scanner. This ServoScan system effe ctively eliminates the nonlinearity of the piezoelectric actuators.

NX10 scanners, both X-Y and Z scanner, have a maximum range of movement. Figure 5-15 depicts the maximum XY scan range as a solid gray-shaded square. The area outside of this square cannot be observed. For example, if the scanner's maximum range i s 50 μ m, it is not possible to scan both areas **A** and **C** even though they have the same sca n size (15 μ m). Area **A** is impossible to scan because its offset (the black point) extends its range over the maximum range of the scanner. Area C, however, is possible to scan. Also, although B and D have the same size and the same offset, it is impossible to scan area B which extends over the maximum range due to its different angle of rotation. Whenever the user enters an "excessive range" like A and B, the scan range will be changed automatical ly to an observable area that falls within the scanner's maximum allowable range.



Figure 5-15. Scanner's observable area

The lateral resolution of an image acquired by AFM is calculated by dividing the scan size by the pixel size. If you measure a 10 μ m square image with 256 × 256 pixels, the later al resolution is 10 μ m/256 = 39.1 nm. This means the size of one data point in the 10 μ m square image is 39.1 nm. Even though you can increase an image's pixel count to get higher lateral resolution, it will take much longer to acquire an image. Another solution to get high er resolution data is to decrease the scan size. If you measure a 100 nm image with 256 × 256 pixels, you can get a lateral resolution of 3.91 Å per data point. Therefore, when you want to measure fine structure, it is desirable to reduce the scan size.

Also, the scanner's ability to make an elaborate motion is another factor that influence s the lateral resolution. The scanner expands or shrinks in proportion to an applied voltage. Hence, you can manage the scanner's motion more precisely by dividing the applied volta ge into smaller units in the DAC (digital-to-analog converter). The NX10 system uses a 20-bit DAC for controlling scan movement in X and in Y. A 16-bit DAC is used for determining scale so that the scanner's motion and position can be elaborately controlled. When an ap plied voltage that can make the scanner move 50 μ m is controlled using a simple 20-bit DA C, the lateral resolution is 50 μ m / 2²⁰ = 0.19 Å.

The resolution of the Z scanner can be adjusted by limiting the Z scanner's motion ran ge. The number entered in the text box labeled Z scanner range can be regarded as a pro portionality factor relating Z scanner's maximum movability. Basically, if the Z scanner Ran ge is 1.0, then the Z scanner in the standard head can move through a 15μ m range. Howev er, if the Z scanner Range is 0.5, then the Z scanner's maximum movable range would be reduced to 7.5μ m. This adjustment that effectively reduces the Z-scanner's maximum rang

e results in an increase in vertical resolution. To use the Z scanner Range feature effectivel y, you should consider two points: the z-scanner's available maximum range and the vertic al resolution. Before adjusting the Z scanner Range, one must first consider the overall hei ght variation of the sample surface. Of course, this height difference should not be greater than the Z scanner's maximum available range. For example, if a sample has 30µm height difference, it cannot be measured if the standard head is used..

XY Scan Method

The XY scanner movement is controlled using piezoelectric elements which expand or contract in length in response to an applied voltage. This allows for circuits containing sev eral DACs (digital-to-analog converter) and high-voltage amplifiers to electronically drive th e XY scanner in a raster pattern while imaging samples.

That is, the X scanner moves the sample first along a line from left to right and then re traces this line until it is back to its original position. Next, the Y scanner takes a single ste p along the orthogonal direction and the process repeats. In this way, the XY scanner can get two dimensional data by repeating the process many times. The user can define the a mount of data per line and the number of lines to be collected, corresponding to image pix el width and height, respectively AFM data is most commonly collected in square, n x n pix el scans. In this example, the X direction is known as the fast-scan direction and the Y is k nown as the slow-scan direction. (See Figure 5-16) The fast-scan direction can be selecte d by software arbitrarily within the XY-plane the slow-scan direction will always be orthogo nal.



Figure 5-16. Standrad Scanning

Z Scan Method

When the XY scanner is moving in the fast scan direction, the Z scanner is vertically moving to track the sample morphology. The AFM image is created by the digitized Z scan ner feedback signal which is collected at every X and Y position corresponding to a pixel a

s defined by the user's scan parameters. Point data is acquired and forms a line; this line is s a collection of consecutive points throughout the X axis, in form of digitalized data that d erives from Z scanner feedback signals. Next, these lines are consecutively acquired alon g the Y axis, thus creating an AFM image. The brightness of the AFM image indicates sam ple height information. Park SYSTEMS AFMs have strain gauge sensors on the Z scanner in order to accurately measure sample heights regardless of the non-linear characteristics inherent to all piezoelectric devices.

5-3-2. Image Data Type

Unlike other common file formats, the 'TIFF' files have tags. The 'TIFF' file format inclu des a header and many tagged fields. The tagged fields can describe dimensional informat ion such as the width and the height of the images so that the software that handles the 'TI FF' file can read these tagged fields and then extract information from them in order to gen erate images to display in the image viewer. Consequently, the 'TIFF' does not affect the o riginal image file and has superior compressibility as well as no resolution limits. These ad vantages make the TIFF format ideal for handling larger, capacious files.

The data files produced by conventional SPM instruments are not a common image fil e format. Thus, to see these acquired images in an Image viewer and the traditional Windo ws Explorer display, it is necessary to change the file format saved by individual SPM instr uments into the image file format by using image processing software.

If the collected data is saved as a common image file format, it may be quite convenie nt to view the images without any special software conversion process of the information fil e. However, this is difficult since conventional image file formats include only image data (R, G, B) and cannot save the large amount of sample data which is measured by the NX s ystems.

Considering these difficulties, the TIFF format is a more flexible means of storing SPM images. Therefore, in the NX system, the image data is saved as a 'TIFF' file format, in w hich a huge amount of data can be saved in the private-tagged area and the acquired ima ges from this data can be saved in the standard-tagged fields as an image file so that it can be viewed in the common image viewer.

When you see the NX system's acquired data in the common image viewer, you can i dentify this data as a familiar sample image, and you can process the images without trans forming original measurement data.

The part changed in the common Image viewer is the information that is saved in the standard-tagged fields of the data file. Therefore, the collected data saved in the private-ta

gged field will be secure from the transformation of the data in the image viewer, and also this data may be changed or processed in the XEI image processing program.

Data Export

Obtained images can be exported to Text or bitmap format to allow analysis by extern al software. Tiff files can be exported in the form of a text file or an image file (jpg, png, bm p, and emf) from the "Export" command in the context menu of the Image display panel in XEI image processing program. The raw tiff file consists of two parts, the scan data and th e image. When the tiff file is exported as a text file, the file will contain basic information ab out the tiff file and the data array of the scan data. On the other hand, when the tiff file is e xported as an image file, the exported image file only includes the image of the tiff file but not the scan data within it; the image will not include any dimensional information.



Figure 5-17. Data Export



On SmartScan

5-4. Maintenance

Maintenance procedures are required to maintain accuracy and safety of the SPM. Th ere are two kind of maintenance procedure. One is a daily check and the other is a periodi c test which is recommended to be performed after operating the SPM for a certain period of time.

Frequency checklist

Frequency	ltem	Action		
1 Day	Noise and Vibration	- Any abnormal noise generated or vibration near AFM main body.		
	Fixation	- Fixation of the bolts		
3 Month	Clean condition and broken parts	- Clean inside the AE.		
	Calibration	- Calibration of XY scanner		
	Cable connection	- Cable connection of all the cables.		
6 Month	XY, Z, Focus Stage	- Reset all the motorized stages to check the performance of stage motor and limit sensor.		
	Calibration	-Calibration of Z scanner		
	Any parts	- Check for any broken parts		
1 year	Cables	- Check the cabling conditions of the main cables that connects the electronics and SPM		

Table a. Checklist for freqency checkup

* For height or width analysis, in order to improve the accuracy of the measured data, It recommended to check the scanner (XY or Z) Calibration before measurement.

5-4-1. Calibration

Access the maintenance mode workspace by clicking the **Maintenance** tab or by clicking **SmartScan** ▼ -> **Maintenance**. Figure i-1 shows the maintenance mode workspace and labels for each view area (Vision View, Monitoring View, Setup View, and Sweep Result View). Each view area is described below.



Figure 5-18. The maintenance mode workspace

Setup View

Setup View displays setup parameters for sweep tests and scanner and cantilever calibration. Setup View is separated into two tabs: the Sweep Setup View and the Calibration Setup View.

Sweep Setup View

Use the controls here, you can perform a sweep of the desired driving channel and view various resulting signal traces. For more detail, refer to Chapter 12, Sweep Tests.

Calibration Setup View

Various instrument components, including the Z scanner, XY scanner, and cantilever can be calibrated from this tab. The sub-interface for each component can be toggled by clicking the desired radio button.

■ Calibration/Z Scanner

Sweep	Calibration
Part • Z • XY • Cantile	Z Scan Stroke / full = 8.696 µm Measured 0.000 µm Expected 0.000 µm Apply
 Offsets 	Z Detector Stroke / full = 17.464 μ Measured 0.000 μm 🗧
	Expected 0.000 µm 🗘 Apply
	Z Detector Correction factors
	CX1 0.000 ÷
	CX2 0.000 ÷
	CY1 0.000 ‡
	CY2 0.000 ÷ Apply
	Sweep Z

Figure 5-19. Z Scanner calibration setup

Z Calibration	Function
Parameters	
Z Scan Stroke	Z Scan Stroke is the Z scanner movement
	determined by the applied voltage bias to the Z
	piezo/scanner. The Measured value is the height
	derived from the Z scan calibration image. The
	Expected value is the height value reported for the
	known sample. After entering the Measured and
	Expected values, click Apply to adjust the
	calibration.
Z Detector Stroke	Z Detector Stroke is the Z scanner movement
	determined by the linearized sensor. The Measured
	value is the height derived from the Z height
	calibration image. The Expected value is the height
	value reported for the known sample. After entering
	the Measured and Expected values, click Apply to
	adjust the calibration.
Z Detector Correction	These are factory calibrated nonlinear correction
Factors	factors

Stroke Calibration

The Z scanner stroke can be calculated by imaging a grating sample that has a known height. Differences between the known value and the measured value can be adjusted through calibration tables.

For example, a grating sample of height 3µm×3µm is measured as 120nm. If the known height reported by the manufacturer is 100nm +/-7nm, the measured value is off by 13-20nm. Calibration of the scanner stroke can correct the measurement discrepancy.

The Z stroke is calculated two ways. The first way to determine the stroke is using the voltage applied to the Z piezo. The second way determines the stroke using a sensor. Each signal must be calibrated separately using different channels.

Non-Linear Correction Factors

The Z scanner calibration provides software correction for non-linear Z scanner movement. For example, if you can see a non-linear image like the one below, you can correct it by calibrating **CX**, **CX2**, **CY**, and **CY2**.

The X direction and Y direction of this surface are expressed as $AX^2+BX+CY^2+DY$, where $AX^2=CX2$, BX=CX, $CY^2=CY2$ and DY=CY. When you obtain a 1st order slope from the Z height as below, you can enter +0.1 into CX since the equation is $Y=AX^2+BX+C$, where A=0, B=10µm/100µm=0.1, and C=0. Please note that currently the software does not calculate the values of X, Y, X2 and Y2. You will need to calculate these values manually.



X direction scan size 100um

To calibrate the Z scanner, image a standard sample with a known step height and enter the measured and known (expected) heights into the calibration setup interface. A summary of the parameters for Z scanner calibration can be found in the table below. For more information about scanner calibration, please refer to the NX User's Manual.

Sweep Z

Sweep Z

Clicking this button sweeps the Z scanner in full bias range and displays the result in the Sweep Result panel on the right. With this, you can check the full stroke of the Z drive/detector. When the button is active, the system will continuously sweep the scanner range, and **Cancel** will appear in the button. Clicking **Cancel** will stop the sweep.

Calibration/XY Scanner

Figure 11-3-2 below shows the XY scanner calibration setup. XY calibration values include **Scan Stroke**, **Detector Stroke**, and **Detector Offset**. Each can be adjusted independently for the X and Y directions. When calibrating the scanner, the X scan values are used when then XY servo is off (open loop), while the X detector values are used when the XY servo is on (closed loop). Please see the NX manual for more detailed information about XY scanner calibration.



Figure 5-20. XY scanner calibration setup

To change XY scanner calibration values:

- 1. Click the appropriate display button next to the parameters to change.
- A dialog box will open. Enter the measured and expected values for the desired par ameter.
- 3. Click Apply. The software will calibrate the scanner automatically.

Figure 5-21 gives an example of how to change the X Scan Stroke value.



Figure 5-21. XY scanner calibration example

Stroke Calibration

Stroke calibration changes the movement in the X or Y direction. Scan signals are used for calibration of open loop scanning (XY servo off). Detector signals are used for calibration of the detector used for closed loop scanning (XY servo on). It is verified by checking the measured scanner movement against a known structure. For example, if a known 3µm×3µm grating sample is measured, the width of three gratings is known to be 3µm×3 gratings=9µm. If the actual measured value is 9.8µm, then the X or Y scanner needs calibration. In this example, depending on the direction of measurement, you would enter 9µm and 9.8µm in the **Expected** and **Measured** fields for the X or Y row.

Offset Detector

The offfset detector is used to center scanner movement within the stroke range. For the detectors' offset calibration, you should enter the **Offset**

value. This value describes how the detector shifts from the origin in the X and Y directions. You can estimate the **Offset** values by performing a sweep test of the X and Y scan and monitoring the non-zero X and Y coordinates of the origin in the the **Oscilloscope** screen. Then the X and Y detectors can be calibrated by entering the **Offset** value and then clicking **Apply**. For more information, please refer to the NX User's Manual.

Panel	Function
Measured X(Y) Scan/Detector	Input the measured XY scan/detector's stroke length.
Expected X(Y) Scan Detector	Input the known XY scan/detector's stroke length.
Offset of X(Y) Detector	Input how the detector shifts from the origin.

*XY Scan: XY movement in open loop (XY servo off) *XY Detector: XY movement in closed loop (XY servo on)

Sweep X

```
Sweep X
```

Click this button to sweep the X scanner in full bias range and display the result in the Sweep Result panel on the right. With this, you can check the full stroke of the X scan and X detector. When the button is active, the system will continuously sweep the scanner range, and **Cancel** will appear in the button. Clicking **Cancel** will stop the sweep.

Sweep Y

```
Sweep Y
```

Click this button to sweep the Y scanner in full bias range and display the result in the Sweep Result panel on the right. With this, you can check the full stroke of the Y scan and Y detector. When the button is active, the system will continuously sweep the scanner range, and **Cancel** will appear in the button. Clicking **Cancel** will stop the sweep.

XY Servo Check box



Checking this box turns on the XY servo. Unchecking this box turns off the XY servo.

Config



Clicking **config** opens the XY Servo Configuration dialog. Please see Section 8-4-11 for more information about XY servo configuration.

■ Calibration/Cantilever

Different models of cantilevers are produced with varying force constants and resonance

frequencies, resulting in differing values in various performance metrics. Because these values cannot always be obtained nondestructively, SmartScan maintains a database of known cantilever properties. This database is pre-populated with several common cantilevers. If you choose a different cantilever, you must perform a cantilever calibration and create a database entry for it. Cantilever calibration is divided four main sections: resonance frequency range, cantilever constants, A-B sensitivity, force constant, and NCM amplitude gain.

Sweep Cali	bration	
Part O Z	Cantilever 'NCHR'	Reload All
 XY Cantilever Offsets 	Frequency Min Max	330 kHz ‡ 200 kHz ‡ 400 kHz ‡ Apply
	Constants Tip Angle Tip Height Length Sensitivity Force Slope orce Constant cm Amp Gain	0.000 deg \$ 15.000 um \$ 125.000 um \$ Apply 59.988 V / µm 0.000 mV / µm 42.000 N / m 0.500

Figure 5-22. Cantilever calibration setup

Cantilever Resonant Frequency

When performing an NCM sweep, the frequency is varied from the known minimum to the known maximum resonance range for the current cantilever type. Enter the minimum, maximum, and typical resonant frequencies for your cantilever. These values are usually provided by the cantilever manufacturer. Click **Apply** to finish calibration.

Cantilever Constants

Descriptions for cantilever constants are provided by the manufacturer and defined as follows:

Tip Angle	This is the angle between the cantilever arm
	and the tip.
Tip Height	This is the height of the tip, defined as the length from the end of the tip to the center of
	the cantilever beam. This value is provided by
	the cantilever manufacturer.
Cantilever	This is the length of the cantilever. This value
Length	is provided by the cantilever manufacturer.

To change values, update values in the appropriate fields and click Apply.

A-B sensitivity calibration

As the cantilever moves across a sample surface and deflects upwards or downwards, the A-B signal on the PSPD is changed because the beam is reflected off the top of the cantilever. The A-B sensitivity value determines how much the A-B signal changes in respect to the cantilever deflection (A-B sensitivity=A-B/height).

To input this value automatically using acquired data, click the **Data** button. A dialog box will appear. Select a file containing an FD curve obtained in contact mode. Select the linear region and click **Apply**. The A-B Sensitivity Cantilever Calibration dialog is shown in Figure i-6.

Force Constant

Input the typical force constant for the cantilever. You can find this value in the cantilever manufacturer's specifications. Update the field for the force constant and click **Apply** to save the force constant to the cantilever file listed at the top of the view area. [Thermal Tune]

NCM Amp Gain

The A-B(AC) signal on the PSPD is amplified by a lock-in circuit. This electronics gain is called **NCM Amp Gain** (V/V). The value for **NCM Amp Gain** can be entered into the text field. Click **Apply** to save the values into the cantilever calibration.

To determine the gain automatically, click the **Data** button, which opens the NCM Amplitude Calibration Dialog window.

Sweep Result

The Sweep Result workspace displays up to eight signal channels resulting from sweeping the driving signal. Figure i-6 shows the Sweep Result workspace.

Figure 5-23. Sweep Result workspace



The horizontal axis of the all the graphs displayed can be set by clicking the **Horizontal Axis** button and choosing the desired parameter from the drop-down menu. The name of the channel used for the horizontal axis will be displayed in the button.

Figure 5-24. Horizontal Axis button with Driving Channel selected



Options buttons for the Sweep Result graphs are as follows:



Displays cursors on all sweep result graphs



Auto scales the graph to display the enter curve

亖

Allows for graphs to be rearranged by clicking and dragging graphs to the desired location. Re-click the button once rearrangement is complete.



Moves to the previous or next page of graphs in the

sweep results.

For more information about the Sweep Result workspace and Graphs View.

Chapter 6. AFM in Contact Mode

6-1. Principle of Contact Mode AFM

The AFM (Atomic Force Microscope) is an instrument that is used to study the surf ace structure of a sample by measuring the force between atoms.

At the lower end of the Z scanner, there is a cantilever of very tiny dimensions: 100 μ m long, 10 μ m wide and 1 μ m thick, which is manufactured by means of micro-machining techniques. At the free end of the cantilever, there is a very sharp cone-shaped or pyra mid-shaped tip. As the distance between the atoms at this tip and the atoms on the surf ace of the sample becomes shorter, these two sets of atoms will interact with each other. As shown in Figure 6-1, when the distance between the tip and the surface atoms becomes very short, the interaction force is repulsive due to electrostatic repulsion, and whe n the distance gets relatively longer, the interatomic force becomes attractive due to the long-range van der Waals force.

This interatomic force between atoms can bend or deflect the cantilever, and the a mount of the deflection will cause a change in the reflection angle of the laser beam that is bounced off the upper surface of the cantilever. This change in laser path will in turn be detected by the PSPD (Position Sensitive Photo Detector), thus enabling the comput er to generate a map of the surface topography.



Figure 6-1. Relation between the force and the distance between atoms

In contact mode AFM the probe makes "soft contact" with the sample surface, an d the study of the sample's topography is then conducted by utilizing the repulsive force that is exerted vertically between the sample and the probe tip. Even though the interat omic repulsive force in this case is very small, on the order of 1~10 nN, the spring const ant of the cantilever is also sufficiently small (less than 1 N/m), thus allowing the cantile ver to react very sensitively to very minute forces. The SPM is able to detect even the sl ightest amount of a cantilever's deflection as it moves across a sample surface. Therefor re, when the cantilever scans a convex area (\square) of a sample, it will deflect upward, and when it scans a concave area (\square), it will deflect downward. This probe deflection will be used as a feedback loop input that is sent to an actuator (z-piezo). In order to produce an image of the surface topography, the z-piezo will maintain the same cantilever deflect to by keeping a constant distance between the probe and the sample – if the cantilever r tip reaches a lower area, the Z actuator will move the cantilever down by that distance, or back up if the cantilever's tip begins rising.

6-2. Contact mode setup

To use contact mode AFM, select the appropriate Head mode as follows:

- Turn off the beam by unclicking the beam control check box at the bottom left p ortion of the Vision View
- Once the beam is off, click the Head Mode tab at the top of parameters view an d select Contact.
- 3. Turn on the beam by clicking the beam control check box

6-3. Cantilever Selection

Selecting the appropriate probe is a critical aspect of using AFM. Choosing a probe means determining the combination of a tip, which interacts with sample surface atoms, and a cantilever, which deflects depending on the interatomic forces and quantifies the deflection. Generally, the upper surface of a cantilever is coated with a metal such as go Id (Au) or aluminum (AI). This coating, which enhances the surfaces reflectivity, has a thi ckness of about 1000 Å. There are several types of cantilevers that vary in material, sha pe, softness (represented by the spring constant), intrinsic frequency, and Q-factor. The type of cantilever selected is primarily determined by the measurement mode. As menti oned in Chapter 4, a "soft" cantilever is used for contact mode AFM. Typically, such cant ilevers are made of silicon and have a spring constant less than 1~3 N/m. With such a I ow spring constant, the contact mode cantilever is sensitive to extremely small forces, a nd it will bend more significantly than a cantilever with a higher spring constant when ex posed to an equal force. This allows the AFM to measure even extremely tiny structures.

Figure 6-2 shows the SEM image of a cantilever commonly used for contact mode, the PPP-CONTSCR series. To improve the beam reflectivity, the upper surface of the ca ntilever (the opposite side of the tip) is coated with aluminum.



Figure 6-2. SEM image of the shorter cantilevers (A, B, C) from a chip of the PPP-CONTSCR series

Figure 6-3 shows the detailed standardized gauge of the PPP-CONTSCR series ch ip. Altogether, this chip contains three cantilevers, all with different spring constants.. If t he unmounted cantilevers are purchased separately, you may choose from the set of ca ntilevers A,B,C.



Figure 6-3. Silicon chip of the NSC36 series has 3 rectangular cantilevers.

Table 6-1 shows the specification for the three cantilevers in the NSC36 series.

Cantilever Type	А		В			С			
	Min	Typica	Max	Min	Typica	Max	Min	Typica	Max
Length, I±5, µm		110			90			130	
width, w±3, µm		35			35			35	
Thickness, µm	0.7	1	1.3	0.7	1	1.3	0.7	1	1.3
Resonant frequency, kHz	65	105	150	95	155	230	50	75	105
Force constant, N/m	0.3	0.95	2.5	0.5	1.75	5	0.2	0.6	1.5

Table 6-1. Specifications of NSC36 Series Cantilevers

6-4. Measurement Procedure

The measurement procedure hereafter is the same as in Chapter 6. Please review Chapter 6.

Chapter 7. Lateral Force Microscopy (LFM)

7-1. Principle of Lateral Force Microscopy (LFM)

The principle of Lateral Force Microscopy (LFM) is very similar to that of Contact mod e AFM. Whereas in contact mode we measure the deflection of the cantilever in the vertica I direction to gather sample surface information, we measure the deflection of the cantileve r in the horizontal direction in LFM. The lateral deflection of the cantilever is a result of the force applied to the cantilever when it moves horizontally across the sample surface, and t he magnitude of this deflection is determined by the frictional coefficient, the topography of the sample surface, the direction of the cantilever movement, and the cantilever's lateral s pring constant. Lateral Force Microscopy is very useful for studying a sample whose surfa ce consists of inhomogeneous compounds. It is also used to enhance contrast at the edge of an abruptly changing slope of a sample surface, or at a boundary between different co mpounds.

Since the LFM measures the cantilever movement in the horizontal direction as well a s the vertical one to quantitatively indicate the surface friction between the probe tip and th e sample, it uses a PSPD (position sensitive photo detector) that consists of four domains (quad-cell), as shown in Figure 9-1.





Generally, in AFM, to measure the topography of a sample surface, the "A-B" signal is

used. This signal is related to the difference between the upper cells (a+c) and the lower c ells (b+d) of the PSPD.

Topographic information = A-B = (a+c)-(b+d)

The LFM("A-B") signal, which is related to the change in the surface friction on a sam ple surface, measures the deflection of the cantilever in the horizontal direction and can be represented as the difference in the signals recorded in the right cells (Intensity) and the I eft cells (c+d).

Frictional information = LATERAL = (Intensity) - (c+d)



Figure 7-2 (a) shows a surface structure with a centrally located step with low, smooth

areas on either side. The flat part on the left contains a domain with a relatively high frictio nal coefficient. Profile b indicates the cantilever's deflection as it encounters topographic f eatures as well as different frictional coefficients as it scans from left to right. Profile c is a n AFM image of the surface topography and structure; it is represented by the change in th e vertical deflection of the cantilever which does not include the horizontal deflection. Profi le d and Profile e show the LFM signal which indicates the horizontal deflection of the can tilever. When scanning left-to-right, the surface structure of a sudden peak will instantaneo usly twist the cantilever to the right. This results in a lateral force signal with a convex sha pe as seen in Figure 7-2 (d) (3). The opposite occurs when the probe encounters a sudden downward step as depicted at location 4. The region between 1 and 2 indicates an are a on the sample surface where there is a material with a higher surface frictional coefficien t compared to the surrounding area. There are no distinguishable surface features that will allow the user to differentiate this region utilizing the height signal. Even though the topogr aphical information is the same between ① and ②, there will be a conspicuous difference noticeable in the LFM signal. When the cantilever scans this area from left to right, an incr ease in relative friction will cause it to tilt to the right, thus producing an increase in the LF M signal.

Figure 7-2 (e) shows the LFM signal when the scan direction is reversed. If the cantile ver scans direction as indicated by the arrow, there will be no change in the LFM signal at region ③ and ④ which are related to the topographic features of the sample surface. How ever, when the scan direction is reversed, the cantilever will now tilt to the left in the area w here the frictional coefficient between ① and ② is larger, yielding a decrease in the LFM si gnal in this area.

Considering the simple comparison described above, the LFM result contains the surf ace frictional information as well as the surface topographical information.

Hence, when you analyze the result of the LFM measurement, it is necessary to distin guish the information due to difference in the frictional coefficient from the information due to the change in the sample surface topography by taking the AFM image into account.

7-2. Conversion to LFM

As mentioned above, since lateral force mode is an extension of contact mode, the H ead mode will be set to "Contact mode".

- 1. Turn off the beam by unclicking the beam control check box at the bottom left porti on of the Vision View.
- 2. Once the beam is off, click the Head Mode tab at the top of parameters view and s elect **Contact**.
- 3. Turn on the beam by clicking the beam control check box.

7-3. Cantilever Selection

The Lateral Force Microscope (LFM) measures the horizontal deflection of the cantile ver under the same conditions as contact AFM. Therefore, LFM uses the same type of can tilever.

7-4. Measurement Procedure

You can obtain an LFM image and a topographic image simultaneously when you me asure in contact mode. If you press the () button, the Config window will appear as show n in Figure 7-3 below. You can take an LFM image if you selected 'Lateral Force' option in t his "Input Configuration" box. Also, LPF and Flattening can be chosen based on the sampl e. It is recommended to consult the SmartScan[™] software manual for instructions on how to set them.

		Channel Config			🐚 ×
		Selected Channels	3 selected		Available Channels
		■ Lateral (C-D / LFM) ■ Z Drive ■ Z Height	V ▼ µm ▼ µm ▼	reset clear	 Vertical (A-B) Lateral (C-D / LF Intensity
Channels				add oreset	 Force NCM Amplitude NCM Phase Tip Bias Sample Bias Lockin1 I Lockin1 Q
• 2 Height • PinPoint Ba	μm ~'	Details 'Lateral (C-D / LFM)' Low Pass Filter Flatten None - Plane Fit Enabled	0 %		Lockin2 I Lockin2 Q Lockin3 I Lockin3 Q

Figure 7-3. Setup for LFM mode

The procedure to measure in 'Lateral Force' mode is the same as that in contact mode.

Chapter 8. AFM in Non-Contact Mode

8-1. Principle of Non-contact Mode AFM

There are two major forces, the static electric repulsive force and attractive force, exis ting between atoms a short distance apart: The static electric repulsive forces (F_{ion}) between n ion cores and the static electric attractive forces (F_{el}) between valence electrons and ion cores. When the distance between the atoms at the end of the probe tip and the atoms on the sample surface become much shorter, the repulsive forces between them become do minant, and the force change due to the distance change becomes greater and greater. The erefore, contact AFM measures surface topography by utilizing the system's sensitive resp onse to the Repulsive Coulomb Interactions that exist between the ion cores when the distance between the probe tip and the sample surface atoms is very small. However, as sho wn in Figure 8-1, when the distance between the probe tip and the sample sufface due to the valence electric dipoles due to the valence electrons in the other atoms. The force induced by the dipole-dipole interaction is the van der Waals Force. Non-contact AFM (NC-AFM) measures surface topograph y by utilizing this attractive atomic force in relatively larger distance between the tip and a s ample surface.



Figure 8-1. Concept diagram of contact mode and non-contact mode

Figure 8-1 compares the movement of the probe tip relative to the sample surface for i mages being acquired between in contact AFM and non-contact AFM. Contact AFM uses t he "physical contact" between the probe tip and the sample surface, whereas non-contact AFM does not require this contact with the sample. In Non-Contact mode, the force betwe en the tip and the sample is very weak so that there is no unexpected change in the sample e during the measurement. Therefore, Non-Contact AFM is very useful when a biological s ample or another very soft sample is being measured; the tip will also have an extended lif etime because it is not abraded during the scanning process. On the other hand, the force between the tip and the sample in the non-contact regime is very low, and it is not possible to measure the deflection of the cantilever directly. So, Non-Contact AFM detects the chan ges in the phase or the vibration amplitude of the cantilever that are induced by the attracti ve force between the probe tip and the sample while the cantilever is mechanically oscillat ed near its resonant frequency.

A cantilever used in Non-Contact AFM typically has a resonant frequency between 10 0 kHz and 400 kHz with a vibration amplitude of a few nanometers. Because of the attractive force between the probe tip and the surface atoms, the cantilever's vibration at its reson ant frequency near the sample surface experiences a shift in the spring constant from its in trinsic spring constant (k_o). This is called the effective spring constant (k_{eff}), and the following equation holds:

$$k_{eff} = k_o - F' \quad (1)$$

When the attractive force is applied, k_{eff} becomes smaller than k_0 since the force gradi ent F' (= ∂ F/ ∂) is positive. Accordingly, the stronger the interaction between the surface and the tip (in other words, the closer the tip is brought to the surface), the smaller the effectiv e spring constant becomes. This alternating current method (AC detection) creates a more sensitive responds to the force gradient as opposed to the force itself. Thus, it is also appl ied in such techniques as MFM (Magnetic Force Microscopy and Tapping mode).

A bimorph is used to mechanically vibrate the cantilever. When the bimorph's drive fre quency reaches the vicinity of the cantilever's natural/intrinsic vibration frequency (f_0), reso nance will take place, and the vibration that is transferred to the cantilever becomes very I arge. This intrinsic frequency can be detected by measuring and recording the amplitude o f the cantilever vibration while scanning the drive frequency of the voltage being applied to the bimorph. Figure 8-2 displays the relationship between the cantilever's amplitude and th e vibration frequency. From this output, we can determine the cantilever's intrinsic frequency cy.





On the other hand, the spring constant affects the resonant frequency (f_0) of the cantil ever, and the relation between the spring constant (k_0) in free space and the resonant freq uency (f_0) is as in Equation (2).

$$f_0 = \sqrt{\frac{k_0}{m}} \quad (2)$$

As in Equation (1), since k_{eff} becomes smaller than k_0 due to the attractive force, f_{eff} to o becomes smaller than f_0 as shown in Figure 8-3 (a). If you vibrate the cantilever at the fr equency f_1 (a little larger than f_0), where a steep slope is observed in the graph representin

g free space frequency vs. amplitude, the amplitude change (ΔA) at f₁ becomes very large even with a small change of intrinsic frequency caused by atomic attractions. Therefore, th e amplitude change measured in f₁ reflects the distance change (Δd) between the probe ti p and the surface atoms.

If the change in the intrinsic frequency, resulting from the interaction between the surf ace atoms and the probe or the amplitude change (ΔA) at a given frequency (f₁), can be m easured, the non-contact mode feedback loop will then compensate for the distance chang e between the tip and the sample surface as shown in Figure 8-3 (b). By maintaining const ant cantilever's amplitude (A₀) and distance (d₀), non-contact mode can measure the topo graphy of the sample surface by using the feedback mechanism to control the Z scanner movement following the measurement of the force gradient represented in Equation (1).

Figure 8-3. (a) Resonant frequency shift (b) Amplitude vs Z-feedback



8-2. Non-contact mode setup

The non-contact mode setup can be done easily by selecting NC-AFM as the Head m ode, similar to the setup for contact mode explained in Section 2 of Chapter 8.

- 1. Turn off the beam switch by unclicking the beam control check box on the bottom left portion of the Vision View.
- Once the beam is off, click the Head Mode tab at the top of parameters view and select NCM.
 - 3. Turn on the beam by clicking the beam control check box.

8-3. Resonant Frequency setup

Once the Head mode is selected as NC-AFM, turn on the beam by clicking the beam control check box. The system will then automatically find the resonant frequency. Instead of turning the beam on and off, you can also access the Frequency Sweep dialog by clicking the (



Figure 8-4. Resonant Frequency setup in Non-Contact Mode

When the "NCM Frequency Sweep window opens, you can manually select the reson ant frequency as follows.

- 1. If the 'Refresh' button or 'Zoom Out' button is clicked, one unit on the X-axis repre sents 5 kHz as shown above.
- 2. Select the resonant frequency as follows: First, press the 'Refresh' button and the n the graph of frequency vs amplitude will appear. Press the 'Refresh' button again while adjusting the drive % to make the strongest peak fall within 20nm in the Y-a xis(You can check the Y-axis unit on the upper left corner of graph. It is adjustable using the mouse wheel). After adjusting the height of the peak, press the (Zoom) "I n" button until the X-axis unit is 1kHz/div
- 3. After positioning the mouse pointer on the slope just to the right hand side of the st rongest peak as shown in Figure 8-4, click on it with the left mouse button and a '+' sign will appear. The location of the '+' sign corresponds to the selected frequen cy f1 at which the cantilever will vibrate in non-contact mode. After positioning the mouse pointer on the red horizontal line, move this red line up and down while hol ding the left mouse button; this will allow you to change the set point value. In gen eral, make the set point just higher than half of the peak height, and press the "O K" button once to enter the selection.

The value of the drive amplitude(%) and set point can also be changed in the "Sca n Control" window.

8-4. Cantilever selection

The non-contact mode cantilever has a relatively large frequency since the non-conta ct mode uses the vibrating cantilever method which enables to measure the force gradient by the amplitude and phase change due to the interaction between the probe and a sampl e surface. Figure 8-5 shown below is a SEM image of a typical non-contact mode cantilev er, the PPP-NCHR series. The upper surface of the cantilever (the opposite side of the tip) is coated with aluminum (AI) to enhance the beam reflectivity.


Figure 8-5. SEM image of ULTRASHARP silicon cantilever (the PPP-NCHR series)

Figure 8-6 shows the standard dimensions of the NCHR series chip. The thickness of the chip is 0.4 mm, and a rectangular shaped cantilever is at the end of the chip. Table 8-1 lists the specifications for this cantilever. The non-contact mode cantilever has a thickness of about 4μ m, and the spring constant is very large (42N/m) relative to that of a contact mode cantilever.





Table 8-1. NCHR series Cantilever Specifications

Cantilever Type	Cantilever Length, I ± 5, μm	Cantilever Width, w ± 3, µm	Cantilever Thickness, µm		Resonant Frequency, kHz		Force Constant, N/m				
			min	typical	max	min	typical	max	min	typical	max
А	125	30	3.0	4.0	5.0	204	330	497	10	42	130

8-5. Measurement Procedure

The measurement procedure hereafter is the same as in Chapter 6.

Please refer to Chapter 6.

Chapter 9. Tapping mode

9-1. Principle of Tapping mode

Tapping mode is very similar to Non-contact mode AFM in many ways suc h as the applied force and the measurement principle. Tapping mode is a hybri d of the two most fundamental measurement methods, represented by contact mode and non-contact mode. In Tapping mode, the cantilever vibrates in free-s pace in the vicinity of the resonant frequency like in non-contact mode. At the s ame time, since the vibrating cantilever gets very close to the sample surface, i t taps the surface repeatedly, and the tip "contacts" the sample surface as it do es in contact mode. If you measure the amplitude of vibration of the cantilever used in Tapping mode while changing the frequency, as shown in Figure 9-1, th ere appears a special frequency where the amplitude resonates and amplifies greatly. This is called the intrinsic frequency (f_0).





Tapping mode uses the non-contact mode feedback circuit with keeping th e vibrating frequency (f1) a little bit lower than the resonant frequency while os cillating in free-space. Then, as the tip is lowered, the real spring constant redu ces due to the attractive van der Waals force which increase as the tip approac hes the sample surface, as shown in Figure 9-2 (a). Therefore the resonant fre quency changes to effective frequency(feff) in non-contact regime and the amp litude at the frequency f1 increases by ΔA . Since the amplitude increases by Δ A, the non-contact mode feedback circuit decreases the distance between the t ip and the sample surface by Δd , indicated in the graph of vibration amplitude v s tip-sample distance and z-feedback as shown in Figure 9-2 (b). Therefore, th e vibrating cantilever, which is oscillating above the sample, approaches the sa mple almost in contact or in collision with the surface. This method, keeping int ermittent contact between the sample surface and the vibrating cantilever is cal led Tapping mode.

Similar to the initial approach of making contact with the sample, while sca nning, a larger amplitude reduces the distance between the tip and sample, an d a smaller amplitude increases the distance depending on the surface roughn ess to determine the surface topology.



Figure 9-2. (a) Resonant frequency shift (b) Amplitude vs. Z-feedback



For certain samples, Tapping mode yields better measurements than cont act mode or non-contact mode AFM. Tapping mode has an advantage over co ntact mode in the sense that it will damage the sample less since there is no fri ctional force as the cantilever "skips" across the sample surface instead of "dra gging" across it. Since the amplitude of oscillation is so large, there is a much better chance that the probe will not be caught by the meniscus forces of moist ure condensed on the sample surface, as there is with NC-AFM.

9-2. Conversion to Tapping mode

In Tapping mode, the Head mode will be set to NCM just as in non-contact mode. However, the "Intermittent" checkbox in NCM Frequency Sweep Windo w must be selected.



Figure 9-3. Conversion to Tapping mode

9-3. Resonant Frequency setup

As explained in section 1, Tapping mode uses non-contact mode feedback, but, as opposed to non-contact mode, the driving frequency should be selecte d at the left part of the peak in the graph. The other conditions are the same as the non-contact mode.



Figure 9-4. Resonant frequency setup in Tapping mode

9-4. Cantilever Selection

Since Tapping mode uses the same method as non-contact AFM, which is to vibrate the cantilever when measuring the sample surface, the same type of cantilevers are used in Tapping mode as in non-contact mode unless the user prefers a different type of cantilever for a specific purpose. See the Cantilever Selection section in the Non-Contact AFM chapter.

9-5. Measurement Procedure

The method of measurement of Tapping mode is the same as that of noncontact mode. The absolute value of the Set Point also means the distance bet ween the probe tip and the sample surface, just as in non-contact mode, but th e value is much smaller. The vibrating probe tip moves as if it is pecking the sa mple surface using the same feedback circuit. Determining the set point plays a very important role in obtaining the best image.

Chapter 10. Approach Spectroscopy

Activating spectroscopy control mode changes the workspace as shown below. To get to the Spectroscopy Control View, click the **Manual** tab, then choose the **Spectroscopy** button in the Spectroscopy Parameters View. The workspace will change to the Spectroscopy Control workspace, as shown in Figure 10-1.





Spectroscopy control mode allows users to monitor signals at one point as a paramter change is made. The following spectroscopy modes are available:

- FD: Approach spectroscopy (generally referred to as force-distance spectroscopy)
- IV: Voltage spectroscopy (generally referred to as current-voltage spectroscopy); ac tivated only in current atomic force microscopy (CP-AFM)

- Ind.: Nano-indentation
- AD: Amplitude spectroscopy (generally referred to as amplitude-distance spectrosc opy)
- **TA**: Temperature analysis spectroscopy (used with scanning thermal microscopy)

The Manual tab of the Spectroscopy Control workspace includes.

Vision View

The Vision View displays the optical view from the digital camera. The camera can be focused on the cantilever or sample. Focus on the cantilever to align the beam onto the cantilever. Focus on the sample to locate the general area for imaging.



Figure 10-2. Vision View and expanded Vision View

The Vision View can also be used to control a) light strength, b) turning the beam on/off, c) the XY stage, and d) the Z/focus stage. Clicking **Expand** (view to allow the user to easily see the optical image.

Monitor View

The Monitor View displays useful information during measurement. The Monitor View has three tabs: **Head**, **Channels**.



Figure 10-3. Monitor View

Head Tab

As shown in Figure 10-3, the **Head** tab contains a visual representation of the quad-cell PSPD (position-sensitive photo detector) and three related scale bars (**Vertical**, **Lateral**, **Intensity**) with the value in voltage units, as displayed above. The **Head** tab also contains a servo error bar and a Z scanner bar, which display the status of the feedback loop and the Z scanner in real time.

Panel	Function
Quad Cell-PSPD	Shows the position of the reflected laser beam on the PSPD so that you can monitor the deflection of the cantilever.
Vertical	Monitors the vertical PSPD signal, such as cantilever deflection, amplitude of cantilever vibration, or tunneling current, depending on your experimental setup.
Lateral	Monitors the lateral signal, which is related to the change in the surface friction on a sample surface.
Intensity	Monitors the intensity of the reflected laser beam on the PSPD.
Servo Error Bar	Graphically displays the value of the servo error signal from the PSPD relative to the set point value. The value of the servo error signal is represented by the aqua-green bar.
Z Scan Bar	Graphically displays the Z extension of the piezoelectric scanner within its total range. The value of the Z extension is represented by an orange bar. The working range of the Z scanner is represented by this bar during each scan line.

Quad-cell PSPD and Scale Bars

The quad-cell PSPD can detect vertical as well as lateral deflection of the laser beam of the cantilever. The quad-cell PSPD has four cells as shown in Figure 10-4. You can get information about both surface height (AFM) and surface friction (LATERAL) during scanning by monitoring laser deflection.

The vertical deflection of the cantilever is measured as the difference between the upper cells (A=a+c) and the lower cells (B=b+d) of the quad-cell PSPD and provides the information about the sample's topography.

A-B signal=Topographic information=(a+c)-(b+d)

The lateral deflection of the cantilever is measured as the difference between the left cells (D=c+d) and the right cells (C=a+b) of the quad-cell PSPD and provides frictional information.

Lateral signal=C-D signal=frictional information=(a+b)-(c+d)

Figure 10-4. Quad-cell PSPD



In order to perform an approach and take an image, in general you should set the value of the A-B signal smaller than $\pm 0.3V$ (the red point should be positioned at the center (crosshair) of the quad-cell PSPD display) and the value of the A+B signal (in other words, the laser total intensity) greater than 2V. You can adjust the A-B signal and the intensity signal mechanically using the PSPD adjustment screws on the head (please refer to the User's Manual for more information).

Servo Error Bar

The servo error bar graphically shows the values of the servo error signal from the PSPD relative to the set point value, the reference signal for the feedback loop.

During a scan, deflection of the cantilever changes as the tip responds to surface topography. The Z feedback loop works to keep this deflection constant during a scan by adjusting the Z position of the scanner. The deflection sensor monitors the amount of cantilever bending and sends a deflection signal to the feedback electronics. There, the deflection signal is compared to a reference signal (deflection at the set point) and an servo error signal is generated. This servo error signal is used to generate a feedback signal, which is sent to the Z scanner so that it causes the scanner to extend or retract. This feedback signal can also be used to generate an image of the sample surface.

Figure 10-5 shows the servo error bar. The aqua-green portion represents the value of the servo error signal, and the position at 0V represents the set point value. The feedback loop is optimized when the servo error signal bar matches the set point value.

Z Scanner Bar

The Z scanner bar monitors the extension or retraction of the Z scanner in response to feedback voltage. The orange portion of the Z scanner bar represents the extension of the piezoelectric Z scanner within its total allowable range of motion. The upper end of the Z scanner bar represents the scanner's position when it is fully retracted. The lower end of the Z scanner bar represents the scanner's position when it is fully extended.

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Initially, the Z scanner bar is gray, indicating that the Z scanner is fully retracted. After you enter a set point value, the Z scanner bar fills with orange color to the lower end of the Z scanner bar. This means that the Z scanner bar is fully extended and is ready to approach the sample surface.

Once tip approach is complete, you will see that half of the Z scanner bar is filled with orange. During scanning, when the probe tip encounters peaks on the surface, the Z scanner retracts (the orange bar moves toward the upper end of the Z scanner bar). When the tip encounters valleys on the sample surface, the Z scanner extends (the orange bar moves toward the lower end of the Z scanner bar).

The Z scanner bar always represents the Z scanner's maximum range of motion. Thus, depending on the Z scanner range, its relative motion is scaled differently. When the Z scanner is moving in a small range, the change of the Z scanner bar is relative to that small range, rather than the whole range.

The center of Z scanner bar is 0. As the Z scanner moves up from the center, it retracts and moves in the positive direction (+). As the Z scanner moves down from the center, it extends and moves in the negative direction (-). The value on the top of the Z scanner bar indicates the current Z scanner position.





Channels Tab

In the **Channels** tab, you can select several input signals (up to six) and monitor them through digital panels in real time.

Head Channels				
Vertical (A-B)	+0.35 V	Intensity	+3.13 V	Edit
				Clear
NCM Amplitude	0.0	NCM Phase	+99.77 °	Ciedi
Sample Bias	0.0	Z Scan	-5.09 µm	

Figure 10-6. Channels tab

Edit

Edit opens the Channel List dialog to select signals to be monitored through the digital panel. Check the box on the right side of the the desired signal, and then click **OK** in the Channel List dialog to display the signals in the **Channels** tab.

Clear

Clicking Clear removes all signals displayed in the Channels tab.

The Spectroscopy Workspace is further separated into three main areas: the Spectroscopy Positions View, the Spectroscopy Data List, and the Setup View, as shown in Figure 10-7.

Figure 10-7. Spectroscopy control workspace



10-1. Spectroscopy Parameters View

The Spectroscopy Parameters View sets the parameters used to take the point measurement. The parameter interface changes depend on the type of spectroscopy measurement being taken. The various spectroscopy parameter setup interfaces are shown in Figure 10-8.



Figure 10-8. Spectroscopy parameter View

The general procedure for spectroscopy measurement is as follows:

- 1. Obtain an SPM image of the sample to identify regions of interest for spectroscopy curves using Scan Control or Auto mode.
- 2. Change to Spectroscopy Control.
- 3. Select points to identify for spectroscopy measurement.
- 4. Select one desired spectroscopy mode and parameters for one-point spectroscopy.
- 5. Select the parameters related to moving between points.
- 6. Acquire the scan.
- FD: approach spectroscopy or force-distance spectroscopy

FD spectroscopy mode supports the acquisition of force vs. distance curves, whic h are useful for the investigation of a sample's mechanical properties. The FD curv e is a plot of the force between the tip and the sample as a function of the extensio n of the Z scanner.

• IV: voltage spectroscopy or current-voltage spectroscopy

Used only in current atomic force microscopy (CP-AFM) mode, IV spectroscopy mode supports the acquisition of a current (I) vs. voltage (V) curve to investigate electrical properties of a sample surface. An IV curve is a plot of the current as a function of the tip bias voltage that is applied to the sample.

• Ind: Nano-Indentation

Nano-indentation enables the users to perform indentation tests to measure material properties, such as nanoscale hardness and elasticity. A single indentation cycle consists of loading, holding, and unloading processes. Nano-indentation has two sub-modes: set point mode and Z scanner mode. Each sub-mode uses different parameters to control the indentation cycle. In set point mode, the force (load) between the tip and sample is varied as a linear function of time while the corresponding position of the Z scanner is measured. In Z scanner mode, the Z scanner position is varied as a linear function of time while the corresponding load applied to the tip is measured.

• AD: amplitude distance spectroscopy

Amplitude spectroscopy, or amplitude-distance spectroscopy, allows users to acq uire NCM amplitude and NCM phase information as a function of distance from th e surface. This technique can be used to study tip-sample interaction.

• TA: Thermal analysis spectroscopy

Thermal analysis spectroscopy is used only with SThM for SThM probe temperatu re calibration.

*For detailed information of these modes, refer to the User's Manual or the Mode Manual.

Start begins the spectroscopy data curve at the current XY position. To take spectroscopy curves of the entire point list or point grid, use the **Start** button found in the Spectrocopy Positions area

10-1-1. Options

Clicking **Options** opens the Spectroscopy Options dialog. The Spectroscopy Options dialog allows you to control how the Z scanner behaves while the XY scanner is moving in spectroscopy modes.

Spectroscopy Options	🐚 🗙
XY Speed	1.000 µm/sec ≑
Z Control while XY in Mo	otion
Z Servo On	Custom Set Point
	2,548.73 nN ≑
🔿 Lift	
	1.000 µm 🗘
	100.0 ms ≑
	0.0 ms ≑
	Jok X Cancel

Figure 10-9. Spectroscopy Options dialog

• XY Speed

This field determines how fast the XY scanner moves to relocate the sample relative to the tip. When in **Z Servo On** mode, a high XY speed may be too fast for the Z servo to prevent the tip from being damaged.

• Z Control while XY in Motion

When moving between two measurement points, the cantilever may crash into variations in the sample surface, which may damage both the sample and cantilever. You can select between two different methods to keep the cantilever from crashing into the surface: **Z Servo On** and **Z Servo Off** with **Lift** options.

• Z Servo On

When **Use Z Servo** is checked, the Z servo may be kept on during movement so that the Z scanner follows surface variations and keeps the cantilever from crashing. **Z Servo On** utilizes this concept.

Use Custom Set Point

When in **Z Servo On** mode, the Z servo maintains a certain set point. This option determines whether this is specified separately in the **Set Point** field or is the same as the current imaging set point. When this option is checked, the **Set Point** field is activated.

Set Point

Applicable when **Use Custom Set Point** is checked, the **Set Point** field allows you to specify the set point to maintain while moving between measurement points. By default, this value will be the same as the imaging value, but you can select a different one.

• Lift

When the **Lift** radio button is selected, the Z servo is off and lift options such as **Set Point**, **Lift Height**, and **Settling Time** are activated. When the Z servo is set to be off during the motion between points, the Z scanner raises by a set distance. When the cantilever reaches the new location, a new approach is performed, and measurements resume.

• Height

When in **Lift** mode, the Z scanner is raised by the value shown in this field while the cantilever moves between points. A higher value will be safer, as the cantilever is less likely to crash into sample variations, but will result in a longer reapproach time. A low value reduces reapproach time, but may be insufficient to prevent collision.

• Settling Time

After the tip is relocated, the Z servo must be turned on. **Settling Time** allows the user to define how much time is given for the Z servo to activate.

10-1-2. Presets

Presets opens the Presets window to save spectroscopy parameters so that they can be recalled at a later time.

10-1-3. Calibration

The **Calibration** button displays a pop-up menu of calibration features. The menu is displayed in Figure 10-10. The **NCM Amplitude Calibration** option is only active when a non-contact-based measurement mode is selected (NCM, tapping, EFM, or MFM).

Figure 10-10. Spectroscopy calibration menu



Calibration Sensitivity

This option opens the Sensitivity Calibration window. The Cantilever Sensivity Calibration window is shown in Figure 10-10. To calculate the sensitivity of an FD curve:

- 1. Choose the data. Click **recent data** to use the data recently taken. Click **file** to sav e an FD curve taken previously.
- 2. Choose where the sensitivity will be calculated with the forward (approach curve) or backward data (retract curve).
- 3. Adjust the dotted lines of the calculation window by clicking and dragging on the ed ges of vertical rectange. As the area is adjusted, the green fitting curve will display the calculated sensitivity. For best results, the green line should follow the linear p ortion of the FD curve.
- 4. The Sensitvity to be applied display box will update the calulated sensitivity.
- 5. Click **Apply** to update the sensitivity of the cantilever file.



Figure 10-11. Cantilever Sensitivity Calibration window

Cantilever Spring Constant

This option opens the Cantilever Spring Constant Calibration window. The Cantilever Spring Constant Calibration window is shown in Figure 10-12. To calculate the spring constant of a cantilever:

- Change the cantilever file to the desired calibration file. This will load cantilever con stants used to calculate the spring constant. For information on changing the cantil ever file.
- 2. Install and align the laser on the cantilever.
- 3. Click Acquire to generate the power spectrum density of the thermal tune data. Clic king the time domain check box in the upper right corner will display the time dom ain of the spectrum in the upper right corner.
- Click Calculate to calculate the spring constant, which will be shown in the box belo w.
- 5. Click **Save** to save the new spring constant to the cantilever file. The exact value sa ved to the cantilever file can be adjusted by changing the value in the Spring Cons tant field.

Thermal Tune				n ×
NCHR, K = 42.0 N/m				✓ time domain copy
Acquire	2.0e+02 • PSD • Fitting			
Carculate				
Spring Constant 10.000 N/m 🗘				
Save				
142N				
- Caracity	1.00+02-			
a Dectra	1.06702			
Sower S				
Constants				
Angle Offset 0.0 ° Height 15.0 µm				
Length 105.0 µm Sansthilty 98.9 V/vm				
Schadwicy 50.5 v/pm				
Plot is auto scaled		Frequer	ncy (Hz)	

Figure 10-12. Cantilever Spring Constant Calibration window

■ Force Slope Correction

This option opens the Force Slope Correction window. The Force Slope Correction window is shown in Figure 10-13. The Force Slope Correction feature corrects the baseline of the FD curve. When the FD baseline is sloped, the curve can be corrected using the slope correction. Clicking **Apply** will correct the slope of the data by the value shown in the **Deflection Slope Correction** field.

- 1. Measure the force/distance curve without approach.
- 2. Calculate the slope by dragging the sides of the plot bar. The slope is automatically calculated when dragging the bar.
- 3. Enter the slope calibration in the text box, and then click Apply.
- 4. The value is uploaded to the database (V/μm) and saved in the DSP. The correction is applied when the **Enable** check box is chosen in FD Measurement Parameters.



Figure 10-13. Force Slope Calibration window

NCM Amplitude

This option opens the NCM Amplitude Calibration window. To activate the **NCM Amplitude** menu selection, SmartScan must be set to a non-contact-based scanning mode and **AD Spectroscopy** must be selected. The NCM Amplitude Calibration window is shown in Figure 10-14.



Figure 10-14. NCM Amplitude Calibration window

Z Servo



Clicking this button opens the Z Servo Configuration dialog.

XY Servo



Clicking this button opens XY Servo Configuration dialog.

10-2. Spectroscopy Positions View

You can select the points from the Spectroscopy Positions View to indicate where you want to obtain spectroscopy data within the scan area.



Figure 10-15. Spectroscopy Positions

10-2-1. Reference and Point List

The Reference window displays the last image acquired. This image can be used as a reference to determine the desired points for measurement.

Adding a point

Measurement points can be added by left-clicking on the refrence at the desired location. This will create a single numbered point on the reference. Subsequent clicks will add points to the reference. XY coordinates of the added points will be displayed on the Point List. Additional points can also be added by clicking on the + button next to the Point List. New points can be inserted between two existing measuring points by clicking on the line between the two points in the reference.

Moving a point

Measurement points can be moved by clicking and dragging the numbered point on the reference or by changing the coordinate values in the Point List.

Figure 10-16. Moving a spectroscopy point. Left: original position and movement direction; right: final position after move.



Deleting a point

Measurement points can be deleted by highlighting the point on the Point List and clicking the – button.

■ Changing Measurement Order

The spectroscopy will be measured in ascending order starting from 1. To change the order, click the point on the Point List to hightlight the point and use the **up** or **dn** button to move the point up or down on the Point List.

Moving All Spectroscopy Points

To move the entire Point List as a whole, hover above the border surrounding the points. Once the cursor changes to the **Move** tool, click on the border and drag the spectroscopy points box to the new location. Figure 10-17. Moving all spectroscopy points. Left: original position and movement direction; right: final position after move.



10-2-2. Point Grid

After selecting Point Grid Spectroscopy Positions View, a white grid with pink dots will appear in Reference area. The white box indicates the grid size. Depending on the selected grid pixels, the box is divided. The measurement points will be automatically selected on all the center points of each divided small box. When you click **Start**, the spectroscopy measurements will be acquired, starting from the left bottom corner, using the parameters in parameters control panel. For example, when the grid size is 4x4, 16 measurements are acquired following the order below.

Grid points, size, and offset can be changed under the **Point Grid** tab. Size and offset can be changed visually in the Reference View. The size of the grid box can be changed by clicking and dragging any of the pink dots on the grid box. The offset can be adjusted by clicking and dragging the entire grid box once the hand cursor is visible.



Figure 10-18. Point grid setup and grid box

10-2-3. Single Shot at Current xy

When the **Single shot** box is checked, a single spectroscopy curve will be performed at the current tip (XY) position when **Start** button is clicked. Point List and Point Grid information is ignored.

10-2-4. Repeat

This option repeats the spectroscopy curves the number of times indicated. Values between 2 and 100 can be entered.

10-2-5. Start

Spectroscopy measurements will be acquired in numerical order using the parameters in the parameters control panel. This procedure produces a) a single spectroscopy curve (Single shot), b) a collection of curves (Point List), or c) a 2-D grid of curves (Point Grid).



Figure 10-19. Point List setup (Left) and Point Grid setup (Right)

10-3. Data View

The **Data View** displays the acquired spectroscopy data. Figure 10-20 shows the Data View. For the FD spectroscopy curve, analysis results are listed to the right of the plot.

10-3-1. Axis Menu

Choose the **Compare** tab to compare data from different point curves.



Figure 10-20. Data View Axis menu

10-3-2. Cursor





Figure 10-22. Single cursor example



10-3-3. Copy Menu

Figure 10-23. Copy menu pop-up box

Data View Compare				
📄 Force (μN)	Cursor Point 1 of 1		py as Image	
2.0		Forward Cop Backward		0.00 µN 🛟
			- Maximum Load	1.979 μN

10-3-4. Compare Tab

Choose the **Compare** tab to compare data from different point curves taken within the same Point List or Point Grid run.

10-4. Spectroscopy Data List View

As shown in Figure below, the Spectroscopy Data List View is an oscilloscope window that can be used to display selected input and output signals immediately after spectroscopy measurement. Following spectroscopy measurement, the signals will be updated immediately. To check the results already obtained during the measurement, click the desired point.

The Spectroscopy Data List View can display up to three graphs on the same screen.

10-4-1. Rescale

This control rescales all plots simultaneously so that curve data fits on the oscilloscope screen. Double-clicking on each plot automatically rescales the display. The scale can also be controlled manually using the mouse wheel. The vertical axis on the screen may be rescaled accordingly.

10-4-2. Relocate



Relocate each plot by clicking and dragging it after clicking this icon.

10-4-3. Axis Menu

This control is used to set the X and Y axis of spectroscopy data. The **Axis** menu is located on the left side of the plot. Clicking this button opens a drop-down menu to choose the signals displayed for X and Y. The drop-down menu for the Spectroscopy Data List is shown in Figure 10-24.

Highlight the desired signals to be displayed in X and Y. If multiple points are taken during a measurement cycle, specific points can be displayed by changing the point number in the **Point** field at the bottom of the menu.

Y	x	NCM Amplitude (pm)		Cursor Point 1 of 1 📃
NCM Amplitude	NCM Amplitude			• Forward
NCM Phase	NCM Phase			• Backwaru
Z Scan	Z Scan			
Z Height	Z Height			
Time Stamp	Time Stamp			
Point 1 ÷	(total : 1)			
		8,515,680 8,51	5,685 8,51 Z Height (pm)	5,690 8,515,695

Figure 10-24. Spectroscopy data axis

10-4-4. Copy Menu (Right)

Clicking this opens the **Copy** menu. The **Copy** menu is located on the right side of the Spectroscopy plot. The copy choices are **Copy to Image** or **Copy to Text**.

10-5. FD Spectroscopy

Force spectroscopy measures the force (deflection of the the cantilever) as the tip is brought toward and away from the surface. The FD parameters in the Spectoscopy Parameter View can be found in Table 10-25.

FD IV	Ind AD PCM	J TA	
Z Position	Auto Offset		Start
Highest	1.00 μm 📫		Options
Lowest	1.00 µm 🗘		Presets
Offset	1.00 µm 🗘		
Speed / Down	1.00 µm/s 🗘	Split	
Speed / Up	1.00 µm/s 🗘		
Force Corr.	🗌 Enable		
Force Limit	1,000.000 V 🔹	Use	
Hold Time	0.000 sec 🗘		
Acquisition	Channels	512	Calibration
			= Z Servo
Est. time (per	point) 00:00:00		= XY Servo

Figure 10-25. FD spectroscopy parameters

Controls	Function
Z/Highest	Z scanner retracts (away from the sample) to the max value distance from the offset position. Input range is $[(-1/2 \sim 1/2) \times Z \text{ scanner's maximum movement range]}$ when Z Offset turns on and $[(-1 \sim +1) \times Z \text{ scanner maximum movement range]}$ when Z Offset turns off.
Z/Lowest	Z scanner extends (towards the sample) until it reaches the min value distance from the offset position. Input range is [(- 1/2~1/2) x scanner's maximum movement range] when Z Offset turns on and [(-1~+1) x scanner maximum movement range] when Z Offset turns off.
Z/Offset	When Auto Offset is unchecked, the Offset box is shown. This is the starting value of the Z scanner for an FD curve (see Auto Offset, below).
Auto Offset	When Auto Offset is checked, the offset position is determined by the set point value in the Scan Control window.
Speed	Speed of Z scanner extension and retraction
Split	When the Split box is checked, the speed of the retraction (up) and extension (down) of the scanner can be controlled independently.
Down Speed	Speed of Z scanner extension
Up Speed	Speed of Z scanner retraction
Force Corr	The force correction applied to the FD curve
Force Limit	When the force applied to the cantilever exceeds the force limit, the Z scanner does not extend any farther. To protect the tip, lower the force limit.
Use	When Use is checked, the allowable maximum force (force limit Force Corr) is determined by the value in the text box to the left.
Channels	Opens the Channel Config dialog. The number of channels acquired during the FD curve measurement is displayed on the channel acquisition button.
Pixels	Opens the Pixel drop-down menu, where you can select the number of pixels in each approach or retract curve. The selected pixel number is displayed on the pixel acquisition button.

Table 10-1. Controls in FD spectroscopy mode

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When setting the FD input range, the **Min** value must be smaller than **Max** value. If not, the system will ask you to swap the values.

- Auto Offset
 - Auto Offset On : Z scanner moves from the highest to the lowest position using the current Z scanner position (by the Z servo) as the reference posi tion. For example, in the figure below, the current Z scanner position to m aintain the set point value is 0.04µm. This value will be used as the 0 valu e, or starting point, and the Z scanner will travel from +1.04µm to -0.96µm.



2) Auto Offset Off: Z scanner moves from the highest to the lowest position using the value in the Offset field as the reference position or starting posi tion. For example, in the figure below, the offset value is 1µm. This value will be used as the 0 value, or starting point, and the Z scanner will travel f rom 2µm to 0µm.



10-6. IV Spectroscopy

Table 10-2, shown below, lists each control in the Spectroscopy control window with a brief description of its function.

Figure 10-26. IV Spectroscopy parameters

FD IV	Ind AD PCM	J TA	
Sample Bias	Reverse		= Start
Highest	1.000 V 🗘		Options
Lowest	-1.000 V 📫		Presets
Start	0.000 V 📫		
End	0.000 V 📫		
Period	1.000 sec 🗘		
Hold Time	0.000 sec 🔹		
Current Limit	10.000 nA 🔶	🗌 Use	
Acquisition	Channels	512	Amplifier
	🗌 Turn Off AFM Bea	m	Calibration
			= Z Servo
Est. time (per po	int) 00:00:00		= XY Servo

	Function			
Controls				
Bias/Highest	Highest sample bias value in acquiring an IV curve			
Bias/Lowest	Lowest sample bias value in acquiring an IV curve			
Reverse	When Reverse is selected, the sample bias is applied in this order: Start->Lowest->Highest->End.			
Bias/Start	Start sample bias in an IV curve			
Bias/End	End sample bias in an IV curve			
Period	Time elapsed while changing the sample bias to acquire an IV curve			
Hold Time	Amount of time that the voltage is held at the start voltage value per cycle.			
Current Limit	Maximum current limit. If the current reaches this limit, the sample bias will no longer be applied.			
Use	When Use is checked, the allowable maximum current (current limit) is determined by the value in the text box to the left.			
Channels	Opens the Channel Config dialog. The number of channels acquired during the IV curve measurement is displayed on the channel acquisition button.			
Pixels	Opens the Pixel drop-down menu, where you can select the number of pixels in each approach or retract curve. The selected pixel number is displayed on the pixel acquisition button.			
Turn Off AFM Beam	The laser beam is turned off in IV spectroscopy if this option is checked. It may be useful if the laser beam affects the sample.			

Table 10-2. Controls in IV spectroscopy mode

10-7. Indenter

Nano-Indentation has two sub-modes: Z scanner mode and force mode. Each sub-mode uses different parameters to control the indentation cycle. In force mode, the force (load) between the tip and sample is varied as a linear function of time while the corresponding position of the Z scanner is measured. In Z scanner mode, the Z scanner position is varied as a linear function of time while the corresponding load applied to the tip is measured. Select set point mode by clicking the **Force** radio button. Click the Z radio button to select Z scanner mode.



Figure 10-27. Indenter control window

Parameters related to controlling the measurement process for nano-indentation can be changed from the **Indenter** tab.

Controls	Function
Z or Force	When the Z radio button is checked, nano-indentation is performed in Z scanner mode. When the Force radio button is checked, nano-indentation is performed in set point mode.
Z Scanner Mode	
Z Depth	The Z scanner extends (toward the sample) until it reaches the load depth value distance. The distance is calculated from the offset position. It is activated in Z scanner mode.
Force Limit	Once at the force limit, the Z scanner does not extend any further. To protect the tip, lower the force limit.
Use	When Use is checked, the force is applied to the cantilever until it reaches the force limit.
Force Min	Minimum force value applied to the tip in set point mode
Force Max	Maximum force applied to the tip in set point mode
Speed	Speed of Z scanner extension/retraction
Split	When Split is checked, speed of scanner retraction (up) and extension (down) can be controlled independently.
Speed/Load	Speed of Z scanner extension
Speed/Unload	Speed of Z scanner retraction
Hold time	Amount of the time the indenter is held at the load depth position before it is lifted
Channels	Opens Channel Config dialog. The number of channels acquired during the indentation curve measurement is displayed on the channel acquisition button.
Pixels	Opens the Pixel drop-down menu, where you can select the number of pixels in each approach or retract curve. The selected pixel number is displayed on the pixel acquisition button.
Move Y position along with Z	When this option is checked, Y scanner movement compensation will be used to adjust the Y movement relative to the Z movement in the indentation process.
Load/Unload Ratio	Ratio between movement of Z scanner and Y scanner. This is activated when the Move Y position along with z box is checked. Positive values for the Load/Unload Ratio refer to the case where the movements of the Z scanner and Y scanner are the same. Movement of the Z scanner is always in the negative direction (extension) for Load and positive direction for Unload . Y scanner movement in a positive direction is referred to as "bottom to top," while Z scanner movement in a positive direction is referred to as the "retraction in Z." The allowable input range is -100,0~ +100.0%.

Table 10-3. Controls in nano-indentation mode

10-7-1. Moving the Y Scanner During Indentation

Normally, the cantilever will not be able to approach the sample top-down with zero degree cantilever tilt because an AFM cantilever has certain degree of tilt. Furthermore, it

is possible for the tip to slip laterally as the indenter tip pushes down the sample. To prevent this problem, the Y scanner can be moved or adjusted to compensate for Z scanner extension. This feature is activated when the **Y Travel Ratio** button is selected.



Figure 10-28. Scanner movement during indentation

Related options can be activated as shown in figure below.

Figure 10-29. Load/Unload ratio control

✓ Move y position along with z	
Load Ratio	20.0 % 🗘
Unload Ratio	20.0 % 🗘

In Figure 10-27, the values displayed in the Load Ratio and Unload Ratio specify the ratio between the movement of the Z scanner and the Y scanner. Inserting positive values will cause the Y scanner and Z scanner to adjust in the same direction. Inserting negative values will cause the Y scanner and Z scanner to adjust in opposite directions.

In Figure 9-7-4, when the Y scanner adjusts in the +Y direction, it adjusts in the positive direction, and vice versa. If the Z scanner shrinks, it will adjust in the positive direction, and vice versa.





For example, when entering a loading value of 100%, as the Z scanner adjusts in the -Z direction with a certain distance, the Y scanner will adjust in the -Y direction with the same distance. When entering a loading value of -100%, as the Z scanner adjusts in the -Z direction with a certain distance, the Y scanner will adjust in the +Y direction with the same distance. For example, to improve angle of the tip (13°), it is recommended to enter a **Loading/Unloading** value of -Tan(13°) x 100 ~ -23%.
10-8. AD Spectroscopy

In AD spectroscopy, the cantilever is oscillated at a constant frequency and amplitude. As the scanner is extended and the cantilever approaches the surface, the amplitude begins to decrease. This change in amplitude can be measured as a function of distance to the surface using AD spectroscopy. Controls for AD spectroscopy can be set so that the cantilever moves a specified distance or stops at set amplitude (amplitude limit).



Figure 10-31. AD spectroscopy control window

Controls	Function
Z/Highest	The Z scanner retracts (away from the sample); the highest value that the Z scanner will be lifted away from the surface
Z/Lowest Z	The Z scanner extends (toward the sample); the lowest value that the scan will extend toward the surface
Auto Offset	Uses the relative Z position
Speed	Speed of Z scanner extension/retraction
Split	When the Split box is checked, the speed of the retraction (up) and extension (down) of the scanner can be controlled independently.
Down Speed	Speed of Z scanner extension
Up Speed	Speed of Z scanner retraction
Amp Limit	Amplitude at which the cantilever will stop
Use	When Use is checked, the system will use the amplitude limit
Channels	Opens the Channel Config dialog. The number of channels acquired during the AD curve measurement is displayed on the channel acquisition button.
Pixels	Opens the Pixel drop-down menu, where you can select the number of pixels in each approach or retract curve. The selected pixel number is displayed on the pixel acquisition button.
Start	Initiates an AD curve at the current probe location
Options	Opens the Options window
Presets	Opens the Presets window
Calibration	Opens the Calibration popup menu
Z Servo	Opens the Z Servo window
XY Servo	Opens the XY Servo window

Table 10-4. Controls in AD spectroscopy mode

10-9. TA Spectroscopy

Thermal analysis (TA) spectroscopy mode is used to control current through the nanothermal probe in SmartScan. When current is transmitted to the nano thermal probe, melting can occur (heating the probe tip), and change the probe's deflection. The sample temperature measured by the probe can be calibrated in this way. TA spectroscopy mode requires SThM hardware options. Figure 10-32 shows the TA spectroscopy mode interface.



Figure 10-32. Thermal analysis spectroscopy control window

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Controls	Function
Probe Current	
Start	Starting current of the temperature ramp
	(Min 0.01mA – Max 2.5mA)
End	Ending current of the temperature ramp
	(Min 0.01mA – Max 2.5mA)
Base	Base probe current before and after acquiring data
<u>Timing</u>	
Speed	Speed of probe's current sweep
Hold time	Amount of time that the current is held at the Start Probe Current
Channel Config	The Channel Config window allows choosing data channels to
	monitor and record while scanning.
Data Count	Number of points for acquiring data. The number of points can be
	changed by clicking the indicator button and choosing the points
	desired.
Start	Initiates a TA curve at the current probe location
Options	Opens the Options window
Presets	Opens the Presets window
Calibration	Opens the Calibration pop-up menu. In this mode, you can adjust
	the SThM parameters by selecting SThM reference
SThM Reference	
Reference:	Standard sample's melting temperature for calibration
Temperature	
Reference:	SThM error (V) value when the standard sample starts to melt
SThM Error	
Reference: Current	Probe current (mA) value when the standard sample starts to melt
Offset:	Temperature inside the acoustic enclosure
Temperature	
Offset: SThM Error	SThM error (V) value when the probe current is 0mA
Z Servo	Opens the \angle Servo window. This function can also be use in
	adjusting the Set point value of the nanothermal probe.
XY Servo	Opens the XY Servo window

Table 10-5. Controls in TA spectroscopy mode

Setup NCM	1 = Scan	 Spectroscopy
F/d I/V Ind	A/d TA	SThM Reference Calibration
Current / Star Current / End Current / Base Speed Hold Time	0.010 mA ÷ 0.500 mA ÷ 0.010 mA ÷ 0.100 mA/s ÷ 0 ms ÷	Temperature 25.0 °C ÷ SThM Error 0.0 mV ÷ Reference Temperature Tomperature 55.0 °C ÷ SThM Error 500.0 mV ÷ Current 1.000 mA ÷
Acquisition	5 Channels 512	2 Apply
Est. time (per po	oint) 00:00:09	Calibration Z Servo XY Servo

Figure 10-33. SThM Reference Calibration

Figure 10-34. Z Servo Config



10-10. General Procedure for Spectroscopy measurement

10-10-1. FD spectroscopy

Cantilever Selection

In selecting the appropriate cantilever for FD spectroscopy, force constant must be closely considered. It is recommended to use cantilever with low force constant (E.g. NCSTR, FMR, NSC36) to avoid sample damage. The hardness and softness of cantilever depends on force constant value, wherein, the higher the force constant, the harder is the cantilever. Selecting the cantilever type in the Cantilever database must be done whenever the cantilever type has been changed. The parameters of the cantilever in used, should matched with the parameters of the selected cantilever. The procedure on how to select cantilever type is shown below.

 Turn OFF the Line Scan by clicking the *Line Scan* button on control panel. The yellow light on the Line Scan button denotes that it is ON.

Channels		File Palette Option		Controls
Z Drive	μm		confia	Line Scan
Z Height			0226/7070	Lockin Setup
		select	explorer	
				🗕 Lift 100 μm 🔳
				📃 Approach 🧮

Figure 10-35. Turn OFF the Line Scan

2) Open Part Config window by clicking the Setup tab on control panel. Open the Cantilever Selection Window by clicking the Cantilever type button. The Select Cantilever Window shows the list of common cantilevers offered by Park Systems. If the cantilever type is not on the list, create a new cantilever list by clicking the Create New Cantilever button (Refer to Section 4-1-1. Probe Setup in the attached SmartScan manual).

3)Click **Select** button to activate the selected cantilever type.

			Select Cantilever		🗋 × 🗋
			FMR	NCHR	remove
			LFMR MFMR	Resona Force C	ince 330 KHz Constant 42 N/m
Setup	Contact	- Scan =	Multi75E_G	Sensitiv	rity 5.999 V/μm
			NCH	Length	125 µm
Scan Pate	Part Config	n x	NCHR	Height	15 µm
Scall Rate	Fare Cornig		NCL		
Z Slope			NCLR		
X	Cantilever	NCHR	NCSTAU		
Scan Area	W Scanner	E0up 20.94	NSC14		
		50µm, 20 %	NSC19		
	Z Scanner	15µm, 20 %	NSC36 A		
Pixels			Necac n	Create	New Cantilever
- more			search		
Size			Colact		
Offset					

Figure 10-36. Select Cantilever

General Procedure for FD Spectroscopy measurement

- Replace current cantilever in the system with low force constant cantilever and mount sample.
- Select the type of cantilever in Cantilever Database (Refer to Section 1: Cantilever Selection of this procedure for more information).
- 3) Switch to Contact Mode by clicking the Head Mode button in control panel. Figure 10-37. Select Contact mode



- 4) Approach the tip towards the sample
- Acquire image of the sample to identify regions of interest for FD curve acquisition. This process can be skipped and instead, a random point on the sample can be selected.

 Switch to Spectroscopy Control by clicking *Spectroscopy* button in control panel.

Figure 10-38. Select Spectroscopy mode



- 7) Select FD spectroscopy by clicking the *FD* button in the spectroscopy view.
- 8) Open Spectroscopy Options window by clicking the *Options* button in the Setup menu. Set the parameters to prevent the tip from crashing the into the sample surface as it is being moved to a new measurement location. (Refer to Section 9-1-2 in the attached SmartScan manual).

Figure 10-39. Set the parameters for FD Spectroscopy Options



9) Select points at which to take FD measurements on the reference image.

There are three ways to do this.

 a) First is by clicking the *Start* button, the tip will approach the sample and perform FD measurement at the current location.



Figure 10-40. Clicking the FD Start button

b) Next, is to add points to a list. By clicking the location on the reference image, a point will be added to the list. Points can also be added directly by entering values into the Points List, which is accessible by selecting the *Edit Points* item in the context





Lastly, is to use Map, which designates evenly spaced points on matrix that is

overlaid on the sample surface. (Refer to Section 9-2 Spectroscopy Positions View in the attached SmartScan manual for more information)



Figure 10-42. How to use map

10) Set the parameters in the FD spectroscopy window to obtain optimum data measurement. (*Refer to Section 9-6 in the attached SmartScan manual*

Figure 10-43. Set the Parameters in the FD spectroscopy



 Acquire FD Spectroscopy data by clicking the *Start* button. If the points of interest are designated using the Point list or Map, click the *Start* button found in the Spectroscopy Control Workspace.



Figure 10-44. Acquire FD spectroscopy data

 Once all of the measurement are complete, perform a curve analysis in the XEI software by right-clicking on the file in the buffer window and select "Select to XEI".

If the user needs to perform an absolute measurement in the sample, the Cantilever Sensitivity and Cantilever Spring Constant must be calibrated prior measuring the actual sample. Calibration is done by following the procedures below:

1. Replace current cantilever in the system with low force constant cantilever and mount a hard sample for calibration.

The sample used in calibration must be different with the actual sample to be measured, this is, to avoid sample damage. It's recommended to use hard sample (E.g. Silicon) during calibration to get accurate measurement for actual sample with soft and hard material.

- 2. Perform procedure 2 to 12 of the General procedure of FD Spectroscopy measurement.
- 3. Open the Calibration features by clicking the *Calibration* button.



Figure 10-45. Open the Calibration features

4. Perform Cantilever Sensitivity Calibration. (Refer to Section 9-1-4

Calibration in the attached SmartScan manual)



Figure 10-46. Cantilever Sensitivity Calibration

5. Perform Cantilever Spring Constant Calibration. (Refer to Section 9-1-4

Calibration in the attached SmartScan manual)





10-10-2. IV spectroscopy

• Cantilever Selection

The type of cantilever in IV spectroscopy must be coated by conductive material.

This is to measure the current between the tip and the sample. It is also important to c onsider the type of sample and its application, since the cantilever tip is in contact with the sample during measurement. It is recommended to use Conductive Diamond Coa ted (CDC) type of cantilever for sample with high conductivity. This is because the coat ing on the cantilever will be easily taken off by the high current flown between the canti lever and the sample. On the other hand, CDC cantilever is not recommended for soft sample, since CDC has hard tip that can damage the sample surface, which can result to inaccurate height and unstable current measurement.

For IV spectroscopy, conductive cantilever chip should be mounted on the chip carrier with wire. Cantilever Chip-Wire-Metal of chip carrier should be electronically co nnected by an electro-conductive adhesive such as silver paste. During operation, the wire in the chip carrier is connected into the AFM head.

Selecting the cantilever type in the Cantilever database must be done whenever the c antilever type has been changed. The parameters of the cantilever in used, should ma tched with the parameters of the selected cantilever. The procedure on how to select c antilever type is shown below.

> Turn OFF the Line Scan by clicking the *Line Scan* button on control panel. The yellow light on the Line Scan button denotes that it is ON.

Figure 10-48. Turn OFF the Line Scan



2)Open Part Config window by clicking the Setup tab on control panel and open the Cantilever Selection Window by clicking the cantilever typ e button. The Select Cantilever Window shows the list of common cantile vers offered by Park Systems. If the cantilever type is not on the list, crea te a new cantilever list by clicking the Create New Cantilever button.



Figure 10-49. Select Cantilever

3) Click Select button to activate the selected cantilever type.

General Procedure for IV Spectroscopy measurement

- 1) Replace current cantilever in the system with the appropriate cantilever for the sample and application.
- 2) Setup the Variable Current amplifier hardware. There are two types of Variable Current Amplifier that can be used, External and Internal Variable amplifier and each have different hardware setup. (*Refer to the CP-AFM manual for mo re information*)
- Select the type of cantilever in Cantilever Database (Refer to Cantilever Selection procedure for more information).

Figure 10-50. Select CP-AFM mode



- 4) Switch to CP-AFM Mode by clicking the Head Mode button in control panel.
- 5) Select the type of amplifier and adjust the parameters in the Current Amplifier window. (*Refer to Section 5-7 Current Amplifier in the attached SmartScan manual*)



For External Current Amplifier, the gain setting on the Current Amplifier window should match the gain setting on the hardware in order to display the proper current units. The gain should be adjusted manually by turning the Remote knob depending on the desired value. The current offset should also be adjusted using screw driver.

- 6) Approach the tip towards the sample
- Acquire image of the sample to identify regions of interest for IV curve acquisition. This process can be skipped and instead, a random point on the sample can be selected instead.
- Switch to Spectroscopy Control by clicking *Spectroscopy* button in control panel.



Figure 10-52. Clicking Spectroscopy button with setting to IV Convertor gain

- Switch to IV spectroscopy mode by clicking the *IV* button in the spectroscopy view.
- 10) Open Spectroscopy Options window by clicking the Options button in the Setup menu. Set the parameters to prevent the tip from crashing into the sample surface as it is being moved to a new measurement location. (Refer to Section 9-1-2 in the attached SmartScan manual).



Figure 10-53. Set the parameters for IV Spectroscopy Options

11) Select points at which to take IV measurements on the reference image.

There are three ways to do this.

a. First is by Clicking the *Start* button, the tip will approach the sample and perform FD measurement at the current location.

FD IV	Ind AD PCM	J TA	
Sample Bias	Reverse		= Start
Highest	2.000 V 🗘	L	Options
Lowest	-3.000 V 🗘		Presets
Start	0.000 V 🗘		
End	0.000 V 🗘		
Period	1.000 sec 🗘		
Hold Time	0.000 sec 🗘		
Current Limit	10.000 nA 🗘	🖌 Use	
Acquisition	3 Channels	512	Amplifier
	Turn Off AFM Bea	im	Calibration
			= Z Servo
Est, time (per po	int) 00:00:01		= XY Servo

Figure 10-54. Clicking the IV Start button

b. Next, is to add points to a list. By clicking the location on the reference image, a point will be added to the list. Points can also be added directly by entering values into the Points List, which is accessible by selecting the *Edit Points* item in the context menu



Figure 10-55. Add points to a list

 c. Lastly, is to use Map, which designates evenly spaced points on matrix that is overlaid on the sample surface. (*Refer to Section 9-2 Spectroscopy Positions View in the attached SmartScan manual for more information*)



Figure 10-56. How to use map

12) Set the parameters in the IV spectroscopy window to obtain optimum data measurement. (*Refer to Section 9-6 in the attached SmartScan manual*)
 Figure 10-57. Set the Parameters in the IV spectroscopy

FD IV	Ind AD PCM	J TA	
Sample Bias	Reverse		= Start
Highest	2.000 V 🗘		Options
Lowest	-3.000 V 🗘		Presets
Start	0.000 V 🗘		
End	0.000 V 🗘		
Period	1.000 sec 🗘		
Hold Time	0.000 sec 🗘		
Current Limit	10.000 nA 🗘	🖌 Use	
Acquisition	3 Channels	512	Amplifier
	Turn Off AFM Bea	am	Calibration
			= Z Servo
			= XY Servo

13) Acquire IV Spectroscopy data by clicking the Start button. If the points of interest are designated using the Point list or Map, click the Start button found in the Spectroscopy Control Workspace.



Figure 10-58. Acquire IV spectroscopy data

14) Once all of the measurement are complete, perform a curve analysis in the XEI software by right-clicking on the file in the buffer window and select "Select to XEI".

10-10-3. Indentation spectroscopy

Cantilever Selection

The cantilever used for Indentation spectroscopy is a Diamond Tip cantilever. In this type of spectroscopy, the cantilever is pressed down to the sample with excessive force to indent and measure its mechanical property. The depth and the area of the indent are correlated with the hardness. The force constant of a cantilever, the shape of the tip, and other mechanical properties such as tip glue determine the total force exerted onto a sample. Therefore, hard material such as diamond with sharp edge is more effective in high resolution and sensitivity indentation.

Selecting the cantilever type in the Cantilever database must be done whenever the cantilever type has been changed. The parameters of the cantilever in used, should matched with the parameters of the selected cantilever. The procedure on how to select cantilever type is shown below.

 Turn OFF the Line Scan by clicking the *Line Scan* button on control panel. The yellow light on the Line Scan button denotes that it is ON.



Figure 10-59. Turn OFF the Line Scan

2) Open Part Config window by clicking the Setup tab on control panel and Open the Cantilever Selection Window by clicking the cantilever type button. The Select CantileverWindow shows the list of common cantilevers offered by Park Systems. If the cantilever type is not on the list, create a new cantilever list by clicking the Create New Cantilever button (Refer to Section 4-1-1. Probe Setup in the attached SmartScan manual).



Figure 10-60. Select Cantilever

3) Click **Select** button to activate the selected cantilever type.

General Procedure for Indentation Spectroscopy measurement

- 1) Once all of the measurement are complete, perform a curve analysis by rightclicking on the file in the buffer window and select "Select to XEI".
- Select the type of cantilever in Cantilever Database (Refer to Cantilever Selection procedure for more information).
- 3) Switch to *Contact Mode* by clicking the Head Mode button in control panel.

Figure 10-61. Select Contact mode



4) Approach the tip to the sample.

- Acquire image of the sample to identify regions of interest for Indentation spectroscopy. This process can be skipped and instead, a random point on the sample can be selected instead.
- 6) Switch to Spectroscopy Control by clicking Spectroscopy button in control panel.



Figure 10-62. Clicking Spectroscopy button

- Switch to Indentation spectroscopy mode by clicking the *Ind* button in the spectroscopy view.
- 8) Open Spectroscopy Options window by clicking the *Options* button in the Setup menu. Set the parameters to prevent the tip from crashing the into the sample surface as it is being moved to a new measurement location. (Refer to Section 9-1-2 in the attached SmartScan manual).



Figure 10-63. Set the parameters for Indentation Spectroscopy Options

 Select points for Indentation measurements on the reference image. There are three ways to do this.

a. First is by clicking the **Start** button, the tip will approach the sample and perform FD measurement at the current location.



Figure 10-64. Clicking the Indentation Start button

b. Next, is to add points to a list. By clicking the location on the reference image, a point will be added to the list. Points can also be added directly by entering values into the Points List, which is accessible by selecting the Edit Points item in the context menu



Figure 10-65. Add points to a list

c. Lastly, is to use Map, which designates evenly spaced points on matrix that is overlaid on the sample surface. (Refer to Section 9-2 Spectroscopy Positions View in the attached SmartScan manual for more information)



Figure 10-66. How to use map

10) Set the parameters in the Indenter tab to obtain optimum data measurement. Indentation has two sub-modes: Z scanner mode and force mode. Each sub-mode uses different parameters to control the indentation cycle. (Refer to Section 9-7 in the attached SmartScan manual)



Figure 10-67. Set the Parameters in the Indentation spectroscopy

11) Acquire Indentation Spectroscopy data by clicking the Start button. If the points of interest are selected using the Point list or Map, click the *Start* button found in the Spectroscopy Control Workspace.



Figure 10-68. Acquire Indentation spectroscopy data

12) Once all of the measurement are complete, perform a curve analysis in the XEI software by right-clicking on the file in the buffer window and select"Select to XEI".

10-10-4. AD spectroscopy

Cantilever Selection

In selecting the appropriate cantilever for AD spectroscopy, force constant must be closely considered. It is recommended to use cantilever with high for ce constant (E.g. NCHR) to measure accurate amplitude during operation. AD spectroscopy allows users to acquire NCM amplitude and NCM phase information as a function of distance from surface to study tip-sample interaction. During NCM, the cantilever vibrates and the changes in the amplitude reflect the changes in distance between the tip and the surface. The hardness and softness of cantilever depends on force constant value, wherein, the higher the force constant, the harder is the cantilever. Therefore, using a cantilever with low force constant in NCM will result to excessive vibration and inaccuracy of amplitude measurement.

whenever the cantilever type has been changed. The parameters of the cantilever in used, should matched with the parameters of the selected cantilever. The procedure on how to select cantilever type is shown below.

Turn OFF the Line Scan by clicking the *Line Scan* button on control panel.
 The yellow light on the Line Scan button denotes that it is ON.

Figure 10-69. Turn OFF the Line Scan

2) Open Part Config window by clicking the Setup tab on control panel and Open the Cantilever Selection Window by clicking the cantilever type button. The Select CantileverWindow shows the list of common cantilevers offered by Park Systems. If the cantilever type is not on the list, create a new cantilever list by clicking the Create New Cantilever button. (Refer to Section 4-1-1. Probe Setup in the attached SmartScan manual)

 Select Cantlever
 Image: Select Cantlever

 FMR
 NCHR

 General
 Resonance

 LEMR
 Resonance

 MMR
 Force Constant

 MMR
 Sensitivity

 Sensitivity
 5.999 V/µm

 Length
 125 µm

 NCH
 Length

 NCH
 NCH

 NCR
 NCR

 NCR
 NCR

 NCR
 NCR

 NCR
 NCSTAU

 NSC14
 NSC18

 NSC36_A
 Create New Cantlever

 search
 Select

Figure 10-70. Select Cantilever

3) Click Select button to activate the selected cantilever type.

General Procedure for AD Spectroscopy measurement

- 1) Replace current cantilever in the system with low force constant cantilever.
- Select the type of cantilever in Cantilever Database (Refer to Section 1: Cantilever Selection of this procedure for more information).
- 3) Switch to NCM Mode by clicking the *Head Mode* button in control panel.

Figure 10-71. Select NCM mode



4) Approach the tip towards the sample

- 5) Acquire image of the sample to identify regions of interest for AD curve acquisition. This process can be skipped and instead, a random point on the sample can be selected.
- Switch to Spectroscopy Control by clicking *Spectroscopy* button in control panel.



Figure 10-72. Clicking Spectroscopy button

7) Select AD spectroscopy by clicking the **AD** button in the spectroscopy view.

 Open Spectroscopy Options window by clicking the *Options* button in the Setup menu. Set the parameters to prevent the tip from crashing the into the sample surface as it is being moved to a new measurement location. (Refer to Section 9-1-2 in the attached SmartScan manual).



Figure 10-73. Set the parameters for AD Spectroscopy Options

9) Select points at which to take AD measurements on the reference image.

There are three ways to do this.

a. First is by clicking the *Start* button, the tip will approach the sample and perform AD measurement at the current location.

		= Scan	Spectroscopy
FD IV	Ind AD TA		
Z Position	Auto Offset		- Start
Highest	1.0000 µm 🗘		Options
Lowest	-1.0000 µm 🤿		Presets
Speed / Down	0.10 µm/s 🗸	Split 🖌	
Speed / Up	0.10 µm/s 🗘		
Amp. Limit	5.00 nm 🗘	✓ Use	
Acquisition	4 Channels	512	
			Calibration
			Z Servo
			= XY Servo

Figure 10-74. Clicking the AD Start button

Next, is to add points to a list. By clicking the location on the reference image, a point will be added to the list. Points can also be added directly by entering values into the Points List, which is accessible by selecting the *Edit Points* item in the context menu



Figure 10-75. Add points to a list

c. Lastly, is to use Map, which designates evenly spaced points on matrix that is overlaid on the sample surface. (Refer to Section 9-2 Spectroscopy Positions View in the attached SmartScan manual for more information)



Figure 10-76. How to use map

 Set the parameters in the AD spectroscopy window to obtain optimum data measurement. (Refer to Section 9-8 in the attached SmartScan manual)



Figure 10-77. Set the Parameters in the AD spectroscopy

Acquire AD Spectroscopy data by clicking the *Start* button. If the points of interest are designated using the Point list or Map, click the Start button found in the Spectroscopy Control Workspace.





11) Once all of the measurement are complete, perform a curve analysis by right-clicking on the file in the buffer window and select*"Select to XEI".*

10-10-5. TA spectroscopy

Cantilever Selection

The type of cantilever used in TA spectroscopy measurement is Thermal Probe with a resistive element. Thermal probe serves as a resistance thermometer (or a heater in CCM mode) and at the same time as Contact AFM probe. As a resistance thermometer in TCM, its temperature changes as the tip scans the surface according to the surface temperature. Temperature change of the resistive element leads to change of its resistance. By running a constant current through the tip and measuring the resistance, the temperature of a very small region can be measured. As a resistive heater in CCM, Sufficient energy is applied to the probe tip to keep it at a set temperature via a feedback loop. The feedback loop senses the error signal and adjusts the Probe Current to cancel out the Error signal. The Probe Current, via feedback loop, increases or decreases the energy supplied to the tip in order to maintain a constant temperature and therefore a constant resistance.

Selecting the cantilever type in the Cantilever database must be done whenever the cantilever type has been changed. The parameters of the cantilever in used, should matched with the parameters of the selected cantilever. The procedure on how to select cantilever type is shown below.

 Turn OFF the Line Scan by clicking the *Line Scan* button on control panel. The yellow light on the Line Scan button denotes that it is ON.



Figure 10-79. Turn OFF the Line Scan

2) Open Part Config window by clicking the Setup tab on control panel and Open the Cantilever Selection Window by clicking the cantilever type button. The Select CantileverWindow shows the list of common cantilevers offered by Park Systems. If the cantilever type is not on the list, create a new cantilever list by clicking the Create New Cantilever button (Refer to Section 4-1-1. Probe Setup in the attached SmartScan manual).

		4	Select Cantilever		n ×
		- i	FMR	NCHR	remove
		- 1			
			LFMR		330 KHz
		- /	MFMR	Force Constant	42 N/m
		- <i>i</i>	Multi75E_G	Sensitivity	5.999 V/µm
		i i	NCH	Length	125 µm
Dart Config	1	_	NCHR	Height	15 µm
Part Coning		<u>^ </u>	NCL		
		1	NCLR		
Cantilever	NCHR		NCSTAu		
			NSC14		
XY Scanner	50µm, 20 %		NSC15		
			NSC18		
Z Scanner	15µm, 20 %		NSC36_A	Croate New Cap	tilovor
				Create New Carr	ulevei
		N I			
			Select		

Figure 10-80. Select Cantilever

3) Click **Select** button to activate the selected cantilever type.

General Procedure for TA Spectroscopy measurement

- 1) Setup SThM toolkits and replace current cantilever using Thermal Probe with a resistive element.
- Select the type of cantilever in Cantilever Database (Refer to Cantilever Selection procedure for more information).
- 3) Switch to SThM Mode by clicking the Head Mode button in control panel.

Figure 10-81. Select SThM mode



Load SThM standard sample with known melting point temperature.

4) Sweep the Probe Current signal and adjust the variable resistor located in the HEM with a flat head screw driver to establish same STHM Error value between 0mA and 0.1mA position. (Refer to SThM manual for more information)

Figure 10-82. Adjust the Variable resistor



Approach the tip to the sample.

- 5) Acquire image of the sample to identify regions of interest for Indentation spectroscopy. This process can be skipped and instead, a random point on the sample can be selected.
- 6) Switch to Spectroscopy Control by clicking Spectroscopy button in control panel.



Figure 10-83. Clicking Spectroscopy button

7) Switch to TA spectroscopy mode by clicking the **TA** button in the spectroscopy view.

8) Open Spectroscopy Options window by clicking the *Options* button in the Setup menu. Set the parameters to prevent the tip from crashing into the sample surface as it is being moved to a new measurement location. (Refer to Section 9-1-2 in the attached SmartScan manual).



Figure 10-84. Set the parameters for TA Spectroscopy Options

Select points at which to take Indentation measurements on the reference image There are three ways to do this.

a. First is by clicking the **Start** button, the tip will approach the sample and perform FD measurement at the current location.

Figure 10-85. Clicking the TA Start button



b. Second is to add points to a list. Clicking on a location in the reference image, will add a point to the list. Points can also be added directly by entering values into the Points List, which is accessible by selecting the Edit Points item in the context menu



Figure 10-86. Add points to a list

c. Third is to use Map, which designates evenly spaced points on matrix that is overlaid on the sample surface.



Figure 10-87. How to use map

(Refer to Section 9-2 Spectroscopy Positions View in the attached SmartScan manual for more information)
9) Set the parameters in the TA tab to obtain optimum data measurement. (Refer to

Section 9-9 in the attached SmartScan manual)

		= Scan	Spectroscopy
FD IV	Ind AD	J TA	
Probe Current Start End Base Speed Hold Time Acquisition	0.010 mA 0.500 mA 0.010 mA 0.100 mA/s 0 ms 5 Channels	512	Start Options Presets
Est. time (per p	oint) 00:00:09		Calibration Z Servo XY Servo

Figure 10-88. Set the Parameters in the TA spectroscopy

 Acquire TA Spectroscopy data by clicking the *Start* button. If the points of interest are designated using the Point list or Map, click the *Start* button found in the Spectroscopy Control Workspace.



Figure 10-89. Acquire TA spectroscopy data

Once all of the measurement are complete, perform analysis by right-clicking on the file in the buffer window and select "*Select to XEI*'.

(Refer to XEI manual for more information)

Place a cursor pairs where A-B value changes dramatically on A-B vs Probe Current curve.



Figure 10-90. A-B vs Probe Current

Change Y axis as SThM Error on SThM Error vs Probe Current curve, then acquire X axis value (Probe Current value), and Y axis (SThM Error value).

Figure 10-91. SThM Error vs Probe Current



 Open SThM reference calibration window as shown below and input the calibration value. Click the *Apply* button to start calibration.



Figure 10-92. SThM reference calibration

12) Check the melting point with A-B vs SThM Temperature curve in XEI, after the temperature of nano thermal probe decreases. For better calibration, repeat procedure 14 to 16 more than three times. In repeating the measurement, the user must select another location since the surface condition of the previous location has melted during calibration. Figure below shows A-B vs SThM Temperature curve after thermal calibration using standard sample with melting temperature at 55°C.



Figure 10-93. A-B vs SThM Temperature

Chapter 11. Q Control Mode

The Q Control Mode enables the control of the quality factor of the cantilever while operating in Non Contact Mode and Tapping mode. The quality factor of the cantilever is inversely proportional to the viscous drag (damping) acting on the cantilever. Like the spring constant, the quality factor is also an important parameter of the cantilever. However, quality factor depends not only on the cantilever but also on the environment in which the cantilever oscillates. For example, in liquid conditions, the resonance frequency peak of the cantilever is dampened and broadened, and the quality factor may decrease compared to the quality factor in air.

Basically, the Q control is an appropriately tuned feedback system where the sensor signal (output of the cantilever) is fed back to the excitation signal (input of the cantilever). Since the feedback can be either positive or negative, we can realize Q enhancing and Q reducing operation of the Q control mode. In Q enhancing mode, the differential tip-sample force will be bigger than without Q control and the sensitivity will be better and the image quality may be better in some cases but the tip response time (settling time) will be longer which may limit the scan speed.

11-1. Principle of Q Control Mode

In Non Contact Mode and Tapping mode, the cantilever is modulated with constant amplitude at the resonant frequency using a function generator. This cantilever modulation is detected by PSPD and is divided into a phase signal and amplitude signal through the Lock in Amplifier. The amplitude signal is used for feedback to get the sample topography. Figure 11-1 below shows a schematic diagram of Non Contact mode in NX AFM System.



Figure 11-1. Schematic diagram of Non Contact Mode

When the cantilever is vibrated at the resonance frequency, a +90 degree phase difference between the driving signal (green) and output signal (yellow, VERTICAL (AC) s ignal occurs. Please see Figure 11-2.





At this point, the phase changed is as shown in Figure 11-3. F_0 is the resonance f requency in the graph.



Q Control Mode can make the output signal shift -90 degrees, adding to the driving signal. Then, the driving signal of the cantilever modulation will be amplified. This calls the "Phase Shifter". The "Phase shifter" makes the phase shift through a time delay. In the NX system, input signal for cantilever modulation is a Sine function and output signal through VERTICAL signal is a Cosine function. Therefore, if the output signal is shifted to -90 degree and added to the input signal, the cantilever modulation is amplified

about 10³ in air. Figure 11-4 shows the cantilever modulation signal according to the phase shift. (a) Output signal is amplified when the output signal is shifted to -90 degree in phase and (b) Output signal is decreased when the output signal is shifted to 90 degree in phase.



Figure 11-4. Output signal after phase shift

(a) After increasing Q factor (-90 degree phase shift)



(B) After decreasing Q factor (90 degree phase shift)

After passing through the phase shifter, the signal can be amplified again when given an appropriate gain. This additional feedback circuit is called a "Gain Amplifier". Figure 11-5 shows a schematic diagram of Non Contact mode with Q Control. The output signal, the AC signal from PSPD, passes through the Phase Shifter and Gain Amplifier and is then sent to the Adder. Finally, it is added to the driving signal for cantilever motion.

Figure 11-5. Schematic diagram in Non Contact Mode with Q Control



When the external driving force is applied in Non-Contact mode, the simple harmonic oscillation is expressed by the following equation.

$$m\ddot{x}(t) + \gamma \dot{x}(t) + kx(t) = F_0 e^{i\omega t}$$

m, γ , *k*, and $F_0 e^{i\omega t}$ he cantilever mass, damping constant, spring constant of t he cantilever, and driving signal from NX-electronics respectively.

When the Q control mode is enabled, the equation is as follows:

$$m\ddot{x}(t) + \gamma \dot{x}(t) + kx(t) = F_0 e^{i\omega t} + G e^{i\pi/2} x(t)$$

G is the Q Control Gain and X(t) is the cantilever's motion when time changes. i: $Ge^{t\pi/2}x(t)$ ontrol term. If the driving signal is added to the cantilever modulation, , the equation of the cantilever motion is changed as shown.

$$m\ddot{x}(t) + \gamma_{\text{eff}}\dot{x}(t) + kx(t) = F_0 e^{i\omega t}$$

$$\gamma_{\rm eff} = \gamma - \frac{G}{\omega} \qquad Q_{\rm eff} = \frac{m\omega}{\gamma_{\rm eff}}$$

Therefore, the effective damping is changed by Q control gain, and the effective quality factor is changed.

However, the resonant frequency is shifted because of electronics, drive frequency, cantilever and so on. Therefore, when the phase is set to $-90^{\circ} + \alpha$, the resonance frequency does not change during Q control. In other words, the input value of the phase shifter is (+or-90^{\circ} + α). The process to find the α is called the "Initial Phase Calibration" in the SmartScanTM program.

Figure 11-6 shows the modulation amplitude change while increasing the Q control gain (Left: before initial phase calibration, Right:after initial phase calibration). When the gain is increased after initial phase calibration, the modulation amplitude is increased without a resonance frequency shift.

Figure 11-6. Modulation Amplitude Change according to gain (Left: Before Initial Calibration, Right: After Initial Calibration)



11-2. Q Control User Interface

Clicking the 'To Q Control" button on the Frequency Sweep Window will change the button to the 'To Control' button and change the UI of the Frequency Sweep Window. See Figure 11-7. In this window, you can control the Q value easily.



Figure 11-7. Frequency Sweep Window with Q Control

Q Amplify

This absolute Q Amplify value applies to the gain amplifier of Q control.

Q Amplify	Gain	Phase
0	0 (Deactivate Q Control)	-
0~1	IQ amplify input Valuel	Initial Phase-90
-1~0	IQ amplify Input Valuel	Initial Phase+90

The negative input changes the Q value in the direction of decreasing Q. The

positive input changes the Q value in the direction to increase Q. When you input '0' on the text field on Q Amplify, the Q control mode is deactivated. Figure 11-7 shows that the Q value changes when Q amplify is set to -0.05(Left) and 0.05(Right) after the initial phase calibration is done. You can input -1 to 1 in this text field.





Initial Phase

In principle, resonant frequency is unchanged when the phase is shifted -90° degrees in the NX System. However, in fact, the resonant frequency is shifted because of electronics, drive frequency, cantilever and so on. Therefore, when the phase is set to -90° + α , the resonance frequency is not changed during Q control. This ' α ' is called the initial phase. In other words, the input value of the phase shifter is (+or-90°+initial phase). Clicking the 'Auto Calib.' button automatically changes the phase little by little to find the initial phase without a resonance frequency shift. This process is called "Initial Calibration". Figure 11-9 shows the modulation amplitude change by increasing the gain. Left is before initial phase calibration and Right is after initial phase calibration. When gain is increased, the modulation amplitude is increased without resonance frequency shi ft.





Deactivate

Q control mode is deactivated when the 'Deactivate' button is clicked. It means th at the Q amplify value becomes 0. Figure 11-10 is shown when the 'Deactivate' button is clicked.





11-3. Q Control Procedure

- 1. Change to the maintenance mode by clicking [Mode->Maintenance Mode]
- 2. Open the Frequency Sweep Window.
- 3. Click 'To Q Control" to change the UI of Frequency Sweep Window.
- 4. Input the adequate value to the Q Amplify field. It is recommended to try to set it to "0.05" first. The input range is -1 to 1.
- 5. Click 'Auto Calib.' to find the initial phase and wait until the calibration is done. If yo u hear a noise from the system, stop and go back to step 4 to decrease the Q am plify value.
- 6. Input the desired Q amplify value.

Chapter 12. Magnetic Force Microscopy (MFM)

This document is an operating manual for Magnetic Force Microscopy, one of the many application modes for the NX series SPM from Park Systems. MFM is a technique used to map magnetic properties of a sample surface by measuring the magnetic force between the magnetized tip and magnetic surface. MFM images contain information about magnetic properties such as distribution of magnetic domain.

This manual assumes that you are familiar with the NX series SPM and SmartScan[™] Data Acquisition program. If not please refer to your user's manual for the NX series SPM and software manual for the SmartScan[™] software.

12-1. Principle of Magnetic Force Microscopy

Almost every surface property measured by SPM is acquired by the following process.

MFM measurements follow the same procedure. For MFM, the surface properties wo uld be magnetic properties and the interaction force will be the magnetic force between the magnetized tip and magnetic sample.

However, in addition to the magnetic forces, Van der Waals forces always exist betwe en the tip and sample. These Van der Waals forces varies according to the tip-sample dist ance and therefore are used to measure the surface height.

Hence, in MFM, the signal contains both information of surface height (called 'Height' signal) and surface magnetic property (called 'MFM signal') generated by Van der Waals a nd magnetic forces, respectively. The key to successful MFM imaging lies in separating th e signal which contains magnetic information from the entire obtained signal.



Figure 12-1. Process of the SPM imaging

While the Van der Waals forces present in height imaging are inversely proportional to distance to the power of 7, magnetic signals are long range forces inversely proportional to the second power of distance. This is why NX SPM MFM mode scans a sample twice, in o rder to separate the signals. Height is measured within a range where Van der Waals force s are dominant, and then the tip is moved further away from the sample in order to measure e the effects of magnetic forces.

In the MFM mode of the NX SPM, sample is scanned twice to separate the signal. In first scan the tip scans the surface as in NC-AFM and the surface height of the sample is o btained. In the second scan, the tip-sample distance is increased and the tip is scanned al ong the surface height line obtained from the first scan as shown in Figure 12-2.

The surface height line is the line of the constant tip sample distance, which equals th e line of the constant Van der Waals force. So, when the tip follows the surface height line in the second scan of 'MFM mode', the Van der Waals forces acting on the tip are kept co nstant. Thus, the only change in force affecting the signal is the change of the magnetic fo rce. So, from the second scan, a surface height free signal can be obtained from which th e MFM image is obtained.





Figure 12-3 Obtained signals in MFM mode



12-2. Components

Required components for the MFM imaging are shown in the Figure 12-4

Figure 12-4. Required Components



- 1. Magnetizer
- 2. Magnetizer Clip
- 3. Standard Sample (a piece of hard disk)
- 4. Non magnetic sample holder
- 5. MFM Cantilevers (coated with magnetic materials such as Co-Cr, FeCoNi).

If you are planning to apply external magnetic field as you take image, magnetic field generator is also required. Please see section 'Magnetic Field Generator' for detailed infor mation about magnetic field generator.

12-3. Setup.

A. Magnetizing the tip

- 1. Hold the chip carrier with tweezers. Place the chip carrier on the magnetizer clip with the chip facing down into rectangular cutout. See Figure 12-5 (a).
- 2. Slide in the cover of the magnetizer clip with the groove facing up and to the edge of the magnetizer clip as shown in Figure 12-5 (a). Attach the cantilever chip carrier to the magnetizer clip, taking care to ensure that the chip faces downward, then fasten the magnetizer clip. When the cover is closed, the cantilever chip should be visible through the circular hold of the cover as show in Figure 12-5 (c).



Figure 12-5. Magnetizing the MFM tip

- 3. Insert the magnetizer clip in the magnetizer for 5 ~ 10 seconds as shown in Figure
 4 (d). Insert the magnetic chip according to desired tip magnetization direction, following N and S pole indicators as labeled on the magnetizer.
- 4. Take magnetizer clip out and remove the cover.
- 5. Remove the chip carrier from the magnetizer clip with tweezers. Hold the clip in the air to prevent the damage of the cantilever by touching the floor as you remove the chip carrier from the clip. It is also possible to obtain MFM signals without going through the tip magnetization procedure if you are using a magnetically coated tip, but the magnetizer is still useful to control the magnetization direction of the tip.

Caution!

The cantilever is very small and fragile. It can be broken easily even by a small force such as touching the edge of a clip. Thus, during the whole tip magnetizing process, be careful not to damage the cantilever. We recommend using a cantilever purchased within the past 3 months, as MFM cantilevers may no longer be usable in obtaining MFM signals despite magnetization after 3 months depending on storage conditions.

B. Preparing the Sample

 The magnetic sample holder cannot be used for MFM because its magnetic field affects the MFM image. Remove the magnetic sample holder by unscrewing it from the sample chuck, and replace it by screwing the non magnetic sample holder into the sample chuck as shown in the figure below.

Figure 12-6. Exchanging the Sample Holder



- 2. Mount the sample on the non magnetic sample holder by using the holder clips.
 - I. Loose the screws holding clips.
 - II. Place the clips on the sample.
 - III. Tighten the screws to fix the sample well.





If your sample is large enough not to be mounted using the holder clips. Attach the sample to the sample disk using adhesive.

12-4. Operation

- 1. First, prepare the tip and sample properly as described in the 'Setting Up' section.
- 2. Mount the cantilever onto the head and install the head to the system.
- 3. Align the laser beam on the cantilever. For detailed procedures, refer to the NX user's manual.
- 4. 1) The 'Line Scan Off' button in the toolbar. Select 2) the "MFM" head mode and choose your 3) cantilever type, 'MFMR'.



Figure 12-8. Selecting the Head Mode and cantilever type

5. After selecting the MFM mode, 1) the 'Line Scan on' On/Off button in the toolbar.

2) 'NCM Sweep' window, which is similar to the ones encountered in NC-AFM will appear. Set the cantilever's resonant frequency, set point, and drive % as it is usually done in NC-AFM.



Figure 12-9. Select the NCM Sweep

6. Figure 12-9 shows the parameter view. There are many scan control parameters, however this manual will introduce only those required for MFM mode. Please refer to the SmartScan[™] manual for a description of all other scan parameters in the parameter view



Figure 12-10. Scan Control window of MFM mode

• Tip bias

When imaging in the MFM mode, tip automatically scans the sample surface twice.

First scan is done for height image and second scan is done following the surface height line for MFM image. "Tip bias" is a voltage applied to the tip when the system performs the first scan.

• MFM tip bias

The MFM tip bias is a voltage applied to the tip when the system performs the second scan of the MFM mode to generate the MFM image. For some samples, according to the materials which it is made of, applying the MFM bias has effect of improving image quality.

7. Select "Z Height", "Error Signal", "MFM Amplitude", and "MFM Phase" from the Input configuration window. It is possible to easily adjust settings for recommended channels by selecting MFM from presets located at the bottom of Channel Config.

Channel Config				🐚 🗙
Selected Channels	3 se	lected		Available Channels
Z Height	μm		reset	Aux1 Out
MFM Amplitude		~	clear	Aux2 Out
MFM Phase		~	ədd	Aux1 Scale Out
			duu	HEM1 In
				HEM2 In
				HEM3 In
				HEM Out
				MFM Amplitude
				MFM Phase
			preset	SCM MFM Phase
				PinPoint Baseline
Details 'Z Height'				Stiffness Approach
Low Pass Filter				Stiffness Retract
Elatten None -			n Adharina	
			Snow All	
Plane Fit Enabled				
Apply				

Figure 12-11. Selecting the Input Signal

8. Select "Height", "MFM Amplitude" and "MFM Phase" at the signal name list of each trace control window. Add trace control windows using the trace control icon if necessary.





9. Approach the tip to the sample as it is done in NC-AFM. For detailed procedure, refer to the NX user's manual.

- After the approach is made, change the scan control parameters (Scan size, Slope, Scan rate, Z servo gain, Set point) to obtain an optimal surface height trace.
- After getting a height image, if it is unchecked, check the "Tip-sample dist" check box. Specify the tip-sample distance by entering the value in the Tip-sample distance text field. Now the system will automatically

perform the first scan to get the surface height and then the second scan will travel along the surface height line while maintaining the tip sample distance.

iii. While observing the trace line of MFM Amplitude and MFM Phase, change the "Tip-sample distance" and other scan parameters for the optimal MFM image. Adjust Tip-sample distance settings to minimize atomic force effects and find maximal MFM signal strength.

12-5. Practice

Park Systems offers a standard MFM test sample with MFM mode. Users can test their MFM and practice imaging skill by obtaining the MFM image of the standard sample and comparing it with the expected image. This section presents a typical image of a standard sample and expected result.

Standard test sample

The standard sample shown in Figure. 12-7 is a piece of hard disk glued on a sample disk. Its surface is flat a bit whereas in the same region, magnetic domains with each domain representing a single data bit are engraved. Direction of the trench and magnetic domain lines are perpendicular. The actually obtained surface height and magnetic domain of the hard disk is shown in Figure 12-12.





12-6. Advanced Application

Notes on MFM Imaging

Adjust scan parameters to obtain good Height image

A bad Height image indicates that the distance between the sample and the tip is not constant, and MFM signals obtained when this is the case cannot be considered reasonable data.

• Cantilever resonance frequency

Because MFM mode uses the resonance frequency of the selected cantilever in NCM scanning, selecting a peak outside of the cantilever's resonance frequency range can result in failure to obtain an MFM signal. If there are multiple peaks within the resonance frequency range, scan for each peak until one yields a clear signal.



Figure 12-14. PPP-MFMR NCM Frequency Sweep data

For example, Figure12-14 shows NCM Frequency Sweep results using a PPP-LM-MFMR cantilever. A high amplitude peak appears both inside the resonance frequency range and at above 350 kHz. Here, the peak within the resonance frequency range should be selected.



• MFM Signal Interference with Height signal

Figure 12-15. Example of MFM signal interference in Height image



Figure 12-16. Sample from Figure2, improved scan result

As illustrated in Figure 12-15, the MFM signal can interfere with the Height signal when a sample has a strong magnetic force. One way to eliminate this effect is to adjust the set point to bring tip and sample close enough for the Van der Waals force to overcome the magnetic force. Note, adjusting the set point so low that tip and sample come into contact can result in damage to either or both.

Another way to cope with a sample with a strong magnetic force is to switch out the cantilever type to match. For example, if the PPP-MFMR is producing MFM images such as that in Figure 12-15, changing the cantilever to the PPP-LM-MFMR can eliminate the effect as in Figure 12-16.

• Height signal interference with MFM signal



Figure 12-17. Example of Height signal interference in MFM image



Figure 12-18. Sample from Figure4, improved scan result

When obtaining MFM signals, the tip-sample distance is widened in order to separate magnetic and Van der Waals forces while scanning - when the two forces are not completely separated, the Height image distorts the MFM signal (as illustrated in Figure 12-17). In this instance, the tip-sample distance must be widened enough that the MFM signal is unaffected by Van der Waals forces. Completely separating the two forces results in an undistorted MFM Phase image (as in Figure 12-18).



Figure 12-19. Sample with weak magnetic force

The tip-sample distance must be increased in order to prevent Height signal interference with MFM imaging (as illustrated in Figure 12-17), but there are instances when a sample's magnetic force is too weak to detect at that range (as illustrated in Figure 12-19). In this case, it is advisable to switch the cantilever to one sensitive to magnetic force. For example, if the PPP-MFMR yields blurry MFM images (as in Figure 12-19), changing the cantilever to the PPP-LC-MFMR would yield clear MFM images (as in Figure 12-18).

12-7. Magnetic Field Generator (Optional)

The Magnetic Field Generator is an additional device for MFM used to apply an extern al magnetic field to the sample. When powered, a magnetic field is generated between th e ends of the probe arm.

• Maximum Field Intensity using Park MFG: 300 gauss

A. Required Components

Your Magnetic Field Generator toolkit should include the following components sh own in Figure 12-20. If any of these items are missing or damaged, please notify your retailer.



Figure 12-20. Magnetic Field Generator Tool kit

- 1. Power supply
- 2. Magnetic Field Generator Power Cable
- 3. HEM Jig with screw (2ea)
- 4. Magnetic Field Generator Main body

B. Equipping

The Magnetic Field Generator is attached to the NX Head. Figure 12-21 shows th e Hem jig is attached to the NX Head (a) and screws are screwed to the Hem jig (b) a nd the Magnetic Field Generator Main body is attached to the Hem jig (c) and Hem jig is screwed to the Magnetic Field Generator.



Figure 12-21. Magnetic Field Generator attached to the NX Head

Figure 12-22 shows the Magnetic Field Generator power cable is connected to th e Magnetic Field Generator (a) and Power cable (Black to -, Red to +) is connected to the Power supply (b) and Complete to Set up the Magnetic Field Generator after turn o n the Power supply (c).



Figure 12-22. Set up the Magnetic Field Generator Power

C. Adjusting the field

You can position the bracket and probe arms of the magnetic field generator to ad just the direction of the applied field. The bracket position can be adjusted back and fo rth as shown in the Figure 12-23(a) by loosening the two M1.5 fastening screws. The probe arms can be moved up and down as shown in Figure 12-23(b) by used to attach the probe arm to the bracket.



Figure 12-23. Adjusting the magnetic Field Direction

D. Applying the field

Supply DC voltage (-10V to 10V) to the generator. This produces a field of approx imately 300~500G in the magnetic field generator. Check the MFG datasheet included in each MFG for detail information.

Blow graph (Figure 12-24) shows the change in magnetic field supplying DC volta ge twice from $0V \rightarrow 10V \rightarrow 0V \rightarrow -10V \rightarrow 0V$. According to voltage supplied, magnetic field is generated nonlinearly. First supply voltage (red \rightarrow orange \rightarrow blue) creates resid ual magnetism and after supplying voltage secondly (blue \rightarrow green \rightarrow purple), magneti sm saturates. Due to this, magnetic field increase or decrease nonlinearly depending on voltage supplied.



Figure 12-24. Changes in Magnetic Field due to DC voltage change

Chapter 13. Force Modulation Microscopy (FMM)

This document is an operating manual for FMM (Force Modulation Microscopy) mode for Park Systems' NX series SPM. FMM is used for investigating samples' mechanical properties.

During FMM measurements, the system scans the sample in contact mode while oscillating the tip. The resulting movement of the cantilever is analyzed to obtain FMM and topographic images. FMM images can provide information related to the mechanical properties of a sample, such as elasticity, adhesion force, and friction. FMM images can also distinguish variations in the composition of a sample.

This manual describes the theory behind FMM imaging and the FMM imaging procedure. This manual assumes that you are familiar with your NX SPM system and the SmartScan[™] software.

13-1. Principle of Force Modulation Microscopy

FMM operates in contact atomic force microscopy (C-AFM) mode and is used to detect variations in the mechanical properties of a surface, such as surface elasticity, adhesion, and friction.

During FMM measurements, the tip is scanned in contact with the sample surface. At the same time, the tip is oscillated in the vertical direction by a bimorph piezo at the end of probe arm. As a result, deflection of the cantilever continuously oscillates as the tip scans the surface, generating an oscillating signal at the PSPD. This oscillating deflection signal is separated into two parts, DC deflection signal and AC deflection signal.



Figure 13-1. Oscillating deflection of the cantilever

The DC deflection signal represents average deflection of the tip, which depends on the force exerted on the sample. The Z feedback loop maintains the DC deflection signal and generates a topographic image.

The AC deflection signal is analyzed in terms of FMM amplitude and FMM phase. Both signals are sensitive to elastic properties of the sample surface and are used to generate FMM images.

FMM amplitude is amplitude of tip oscillation. When the tip is oscillated in contact with the sample surface, hard sample surface reflects the oscillation, resulting in a large FMM amplitude signal. On the other hand, a soft surface will absorb the oscillation, resulting small FMM amplitude signal.

FMM phase is the phase difference between the driving signal that oscillates the bimorph and resulting AC deflection signal. Often, FMM Phase is more sensitive to the elastic properties of the surface than FMM Amplitude. Hence, FMM phase imaging provides an additional contrast mechanism within a region of homogeneous hardness.



Figure 13-2. FMM Amplitude and FMM Phase Signal

13-2. Operation

No additional NX hardware components are required for FMM imaging. Only the additional SmartScan[™] software module for FMM needs to be installed to support the FMM mode. Setting up your NX SPM for FMM is same as that of the standard Contact AFM. Please refer to NX user's manual for detailed instructions about setting up the NX SPM for standard Contact AFM.

1. Mount the sample and tip and align laser on the cantilever as in C-AFM.

2. Turn off the line scan (💻 Line Scan	-> = U	ne Scan	and Head mode by
changing the order of	the FMM and	then proceed	Setup-Select	Cantilever-NSC14-
Select.				



Figure 13-3. Changing the Head mode and select the cantilever

3. Open Input Config by selecting 'Channel Config' from the Setup menu. Select Z Drive, Z Height, FMM Amplitude, and FMM Phase input signals.

Figure 13-4. Select the Channel Config

	Channel Config			🌇 🗙
	Selected Channels	4 selec	ted	Available Channels
	Z Drive		✓ reset	HEM1 In
	Z Height		✓ dear	HEM2 In
	FMM Amplitude			HEM3 In
	FMM Phase			HEM Out
				SCM Quad
Click				FMM Amplitude
CIICK				FMM Phase
				PinPoint Bar FMM Phase
Chappede 🗔 🗔	0.1.4			Stiffness Approach
Channels 🗧 🗾	Palet		preset	Stiffness Retract
				Adhesion
Dat	a File Lo Detais 'Z Height'			Modulus
	Low Pass Filter	— 0 %		
File	Name Batter			
The	INathe nation			Show All
92	Plane FitEnabled			
Basi	e Dir			

4. There are many scan control parameters in the Scan Control Window; this manual only discusses those parameters particular to FMM. For descriptions of the standard scan parameters, please refer to the SmartScan[™] manual.

Set point

The Set Point value for FMM is a force value. This represents the force that the cantilever is pressing down on the sample surface with. This value is maintained during the imaging. A higher value indicates a stronger force with which the cantilever presses on the sample. Too high a value will result in tip and sample damage, and too low a value may result in a weak FMM signal.

Setup	FMM	Scan S
Scan Rate	1.00 Hz 🗘	
Z Slope	0.00 ° 🗘 🛛 auto	
Scan Area	Z Servo Bias	
○ Off		
• On		
SetPoint	500.100 nN 🗘	🗌 normalize
Z Gain	1.000 🗘	advanced

Figure 13-5. Adjust to Set point

Drive

In FMM, the tip is oscillated in the Z direction. The amplitude of this oscillation is controlled by the drive. 100% indicates the maximum amplitude that the system is capable of applying. As with the Set Point, too high a value may result in damage, and too low a value may result in a weak signal.



Figure 13-6. Adjust to Drive

5. Set the Drive % and Scan Size to 0.



- 6. Approach the tip to the sample as with C-AFM.
- 7. Click the "Frequency sweep and set up" NCM Sweep icon. The Frequency Sweep dialog will appear.
- 8. Input a Drive % value, and click the Refresh button. A resonance curve will automatically be selected. If this curve is not satisfactory, you can zoom in and out, and select different resonance peaks.



Figure 13-7. NCM Frequency sweep

9. Modify the Drive % and selected frequency until satisfactory values are obtained.

<u>Note</u>

The drive % and drive frequency are the parameters that have greatest effect on the FMM signal. Selecting proper value of drive and drive frequency is the key to obtaining good FMM image. Normally, increasing the drive value gives higher contrast image. But if the drive value is increased too much, there can be a damage to sample surface and tip. The driving frequency must be chosen outside a resonance of the system, but high enough to generate a strong dynamic load that can sufficiently indent the sample.

10. Select "FMM Amplitude" and "FMM Phase" in 1) the trace control windows. Add trace control windows using 2) the page button. if necessary.



Figure 13-8. The trace control windows

11. Input a scan size suitable for an image scan. Change the scan control parameters (scan size, scan rate, Z servo gain, setpoint, and drive) to obtain an optimal signal trace. Click the 'image' button to get the FMM image.

Some Imaging tips

Contrast of the FMM signal decreases if the Z servo gain is increased. Contrast of the FMM signal increases if the set point is increased. Increase drive if FMM amplitude of the FMM signal is too weak. Rubber and vinyl samples are ideal for practicing obtaining FMM images.
13-3. Advanced Application

Notes on MFM Imaging

Adjust Setpoint

Adjust setpoint within a range yielding clear Height signals. Determine cantilever k value and choose setpoint according to the sample in use, as FMM is based on contact mode and the tip may damage the sample surface depending on setpoint settings.

• Frequency Sweep for FMM

After designating setpoint, set scan size to 0 and carry out frequency sweep. Generally, when tip and sample come into contact, peak will shift to less than cantilever's resonance, and multi peak arises.

Refer to the spec. sheet provided along with the cantilever to find the cantilever's resonant frequency, select a smaller frequency, and proceed with FMM measurement. If you wish to verify the cantilever's actual resonant frequency, perform NCM frequency sweep in non-contact mode in order to retrieve the cantilever's actual resonant frequency. Upon FMM image measurement, if resonant frequency settings have been chosen well, images can be observed from FMM amplitude and FMM phase, and if resonant frequency settings have not been chosen well, a signal will not be observed in FMM amplitude and FMM phase.

Monitor measured FMM amplitude and phase while selecting frequencies one at a time and carrying out image measurement tests, in order to find the frequency yielding the clearest image. (trial and error method)

[Example frequency change pre/post]

-Frequency sweep display in Non contact mode (cantilever: PPP-FMR).

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-Frequency sweep display in FMM mode : Peak alters after Tip-sample contact



One of the Peaks indicated in red in the forward area is the peak yielding a clear FMM signal, try selecting these one by one and select the frequency yielding a clear FMM signal.



• FMM signal according to selected resonant frequency [FMM phase line profile example].

Example of bad Frequency choice (e.g. 55Hz)Example of good Frequency choice (e.g. 38Hz)

Closely monitor cantilever resonant frequency and FMM phase to choose frequency. Note, take care as FMM signal yield may be absent depending on frequency selection. Depending on the selected frequency, only one signal may be yielded between FMM amplitude and FMM phase. Find the frequency where FMM amplitude and FMM phase are both clearly received.

If you have checked all frequencies below cantilever spec. and are still unable to carry out image measurement, find harmonics larger than resonant peak and attempt measurement.

• FMM signal by amplitude in Frequency sweep

[Example FMM amplitude line profile].



From the Frequency sweep window, if the selected amplitude is small, FMM signal will be observed but it will be small. In contrast, if the selected amplitude is too large, there will be observable difficulties in differentiating sample surface. As Amplitude size has varying degrees of influence depending on sample and setpoint selection, monitor FMM amplitude and FMM phase closely and change it to fit the sample and situation.

Chapter 14. Electrostatic Force Microscopy (EFM)

This document is an operating manual for Electrostatic Force Microscopy, one of the many application modes for the NX series SPM from Park Systems. EFM is a technique used to map electric properties on a sample surface by measuring the electrostatic force between the surface and a biased AFM cantilever. EFM images contain information about electric properties such as the surface potential and charge distribution of a sample surface.

EFM is largely distinguished as two different modes by the method which the surface morphology information is obtained. These are EFM mode and Dynamic-Contact EFM mode. In addition, EFM mode supports Kelvin Probe Force Microscopy(KPFM). In this ma nual, basic principles, sample preparation, and EFM image taking for each mode is expl ained.

This manual assumes that you have experience taking ordinary AFM images in both contact and non-contact mode with the NX series SPM and the SmartScan[™] Data Acquisition program. If not, please refer to your user's manual for the NX series SPM and SmartScan[™] software.

• EFM Applications: Localized charge distribution on the insulator layer, Ferroelectric domain, Local surface potential distribution, Variations in surface work function and so on.

14-1. Principle of Electrostatic Force Microscopy

Surface electrical property measured by EFM is acquired by the following process.



Figure 14-1. Process of the EFM imaging

For EFM, the sample surface properties would be electrical properties and the interact ion force will be the electrostatic force between the biased tip and sample.

However, in addition to the electrostatic force, the Van der Waals forces between the tip and the sample surface are always present. The magnitude of these Van der Waals for ces change according to the tip-sample distance, and are therefore used to measure the surface topography.

Hence, the obtained signal contains both information of surface topography ('Height signal' in SmartScan[™]) and information of surface electrical property ('EFM signal' in SmartScan[™]) generated by the Van der Waals and electrostatic forces, respectively. The key to successful EFM imaging lies in the separation of the EFM signal from the entire signal. To separate the EFM signal, Park Systems EFM uses the Lock-in Amplifier imbedded internally in NX electronics.

In EFM, a Lock-in Amplifier is used for two purposes. One purpose is to apply AC bias of frequency ω , in addition to the DC bias applied by the NX controller, to the tip. The other purpose is to separate the frequency ω component from a whole output signal.

In the EFM, the voltage between the tip and the sample can be expressed by the following equation:

$$V(t) = V_{dc} - V_s + V_{ac} \sin(\omega t)$$
 (1)

Where V_{dc} is the DC offset potential, V_s is the surface potential on the sample and V_{ac} and ω is the amplitude and frequency of the applied AC voltage signal, respectively.

Equation 1 is appropriate if the geometry of the tip and sample can be approximate using two parallel plates. Other geometries can be assumed as well. Equation 2 can be used to derive an expression for the electrostatic force between the tip and the sample: (Again, parallel-plate geometry is assumed.)

$$F = q \times E = q \times V/d = C \times V^2/d$$
 (2)

$$F(t) = (C/d) \times V(t)^2$$

$$= (C/d) \times [(V_{dc} - V_s)^2 + \frac{1}{2}V_{ac}^2]$$
 (a)

$$+ 2 \times (C/d) \times (V_{dc} - V_s) \times V_{ac} \sin(\omega t)$$
 (b)

$$- \frac{1}{2}(C/d) \times V_{ac}^2 \cos(2\omega t)$$
 (c)

Here, F is electrostatic force applied to the tip, q is charge, E is electric field, V is electric potential, C is capacitance, and d is tip to sample spacing. Note that since both AC and DC bias are applied between the tip and the sample, three terms arise in the expression for the force between the tip and the sample. These terms can be referred to a s the DC term (a), the ω term (b), and the 2ω term (c), respectively.

The ω and 2ω term contains the electrostatic properties and the capacitive properties of the sample, respectively. Lock-in Amplifier can separate the certain frequency term from the signal. However, change of the signal with a frequency of 2ω is too small to detect and it is hard to detect the capacitive properties of the sample through EFM, and the part of the signal with a frequency of ω is only read through internal Lock-in Amplifier.



Figure 14-2. Diagram of 1) EFM, 2) EFM-DC (PFM)

Images can be generated from any of the above-mentioned signals. Analysis of an image involves understanding the contributions to the signal used to generate the image.

1. EFM

EFM is a mode that operates in Non-Contact mode. In the EFM, tip scans over the surface while oscillating in frequency to obtain the non contact AFM topography image. At the same time, AC bias of frequency ω is applied to the tip via Lock in Amplifier 2. This results the force between the AC biased tip and charged surface. Using the Lock in Amplifier, signal resulting from the tip's motion by the force can be decomposed and analyzed into DC part and frequency ω part. ω part of the signal contains information of surface charge, . The frequency ω is chosen to be smaller(14~17kHz recommended) enough than the cantilever oscillation frequency(70~330kHz), so that the two signals do not interfere each other.

2. EFM-DC (Dynamic Contact EFM)

EFM-DC is a mode that operates in Contact mode. EFM-DC uses same method as EFM but is operated in contact mode to give the more improved spatial resolution and clear detection. Figure 14-3. makes the comparison of surface height and surface charge image of TGS single crystal by EFM-DC (upper) and conventional EFM (lower). Image taken by conventional EFM shows strong coupling of the topography to the image while the image taken.

Basic technique of PFM is based on EFM-DC. When the sample is piezo, ferroelectric materials, EFM-DC is equivalent to PFM.





3. PFM (Piezoelectric Force Microscopy)

PFM is to obtain characteristics of material in piezoelectric property. Materials which contain ferro-electricity can be determined the distribution of intrinsic remnant polarization by scanning 2D image. Also, size and direction of polarization can be estimated at one point by spectroscopy. In addition, **Reverse Piezoelectric Effect** (the internal generation of a mechanical force resulting from an applied electrical field) of piezoelectric materials can be measured. To observe a response of reverse piezoelectric effect more effectively, we recommend AC bias method by internal lock-in2.

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In PFM operation, a conductive AFM tip is brought into contact with the surface of the studied ferroelectric or piezoelectric materials, and a pre-set voltage is applied between the sample surface and the AFM tip, establishing an external electric field within the sample. Due to the electrostriction, or "inversed piezoelectric" effects of such ferroelectric or piezoelectric materials, the sample would locally expand or contract according to the electric field. For example, if the initial polarization of the electrical domain of the measured sample is perpendicular to the sample surface, and parallel to the applied electric field, the domains would experience a vertical expansion. Since the AFM tip is in contact with the sample surface, such domain expansion would bend the AFM cantilever upwards, and result in an increased deflection compared to the status before applying the electric field, the domain would contract and in turn result in a decreased cantilever deflection (Figure 1). The amount of cantilever deflection change, in such situation, is directly related to the amount of expansion or contraction of the sample electric domains, and hence proportional to the applied electric field.





Ferroelectric domain with polarization **P**



Electric field, \vec{E} , parallel to \vec{P} , applied to ferroelectric domain



Ferroelectric domain expands and bends the AFM cantilever up, more than the initial deflection



Ferroelectric domain with polarization **P**

Electric field, **Ē**, anti-parallel to **P**, applied to ferroelectric domain

Ferroelectric domain contracts, and reduces upaward bending of AFM probe

If the applied voltage contains a small AC component, the inversed piezoelectric response from the sample would result in sample surface oscillation in the same frequency as the applied AC voltage. In the case that the sample is an ideal piezoelectric crystal, its polarization \vec{P} would be related to applied mechanical stress \vec{s} by the following equation:

$$P_i = d_{ijk}\sigma_{jk}$$

in which d_{ijk} is the rank-3 peizoelectric tensor of the material. For such materials with tetragonal crystal structures, this piezoelectric tensor can be reduced to the following form:

 $\begin{pmatrix} 0 & 0 & 0 & d_{15} \\ 0 & 0 & 0 & d_{15} & 0 \\ d_{31}d_{31}d_{33} & 0 & 0 & 0 \end{pmatrix}$

in which case, under the applied AC modulation voltage $V = V_0 cos(\omega t)$, sample surface vibration would take the form $\Delta Z = \Delta Z_0 cos(\omega t + \varphi)$, with the vibration amplitude $\Delta Z_0 = d_{33}V_0$, and phase $\varphi = 0$ if the sample domain polarization is oriented parallel to the applied electric field, and out of $\varphi = 180^\circ$ if it is oriented anti-parallel to the applied electric field (Figure 2). Such oscillation would be directly reflected in the amplitude and phase signal of the AFM probe contacting the surface, and can be read out using a lock-in amplifier.



In typical PFM imaging, the applied AC voltage is set to be much lower than the coercive bias for sample domain switching, to avoid alternation of the local domain structure of the studied sample. If such criterion is met, the phase contrast generated in PFM imaging would reflect the domain polarity in different sample locations, while from the magnitude of the amplitude signal local piezocoefficient of the sample can be extracted, as discussed in the former paragraph.

For more complicated sample domain orientation containing not only components perpendicular to the surface in contact with the AFM tip, but also components along different directions within the surface plane, vector PFM with one vertical and two lateral channels can provide more complete information. For example, to obtain the d_{15} component of the piezoelectric tensor in tetragonal piezoelectric crystals, we need to measure lateral components of AFM tip vibration proportional to the in-plane sample

surface displacement (Figure 4), which would take the form would take the form $\Delta L = \Delta L_0 cos(\omega t + \varphi)$, with the vibration amplitude $\Delta L_0 = d_{15}V_0$ Notice if a DC bias is applied between the tip and the sample in conjunction with the AC voltage, both the inplane and out-of-plane electromechanical response of the sample are also functions of this applied DC voltage.



In most of the real cases, the studied sample contains random-oriented polycrystalline grain structure, often with non-zero lateral components in its piezoelectric tensor. In this case, the detected vertical PFM signal is no longer only proportional to d_{33} , but also dependent on the d_{31} and d_{15} components. E.g., the vertical PFM amplitude would no longer be $\Delta Z_0 = d_{33}V_0$; instead, it would take the form

$\Delta Z_0 = d_{zz}V_0 = \left[(d_{31} + d_{15})sin^2\theta cos\theta + d_{33}cos^3\theta \right]V_0$

in which θ is part of the local orientation map (θ, ϕ, ψ) between the lab coordinate system and the crystal coordinate system of the sample. Nevertheless, if both the vertical and two lateral components of PFM signal are obtained on the sample location, either the intrinsic

sample piezoelectric constants d_{ij} or the local orientation map (θ, ϕ, ψ) can be extracted

from such data. In a word, 3D PFM has opened the possibility of a complete 3D reconstruction of the polarization vector of the studied sample at nanometer scale.

4. Scanning Kelvin Probe Microscopy (KPFM)

As presented in previous section, the ω signal from Lock-in Amplifier can be expressed as following equation.

$2 \times (C/d) \times (V_{dc}$ - $V_s) \times V_{ac} \sin \left(\omega t \right)$

From the same configuration of the EFM, this ω signal is related with the electrostatic properties of the sample. The ω signal goes to zero when $V_{dc} = V_s$ in the equation, or the DC bias applied to the cantilever is equal to the surface potential. Using the way, NX series KPFM measures the surface potential. The DC bias applied to the cantilever is controlled and acquired so that the ω signal from Lock-in Amplifier is maintained to zero during the imaging. In other words, by reading the ω signal from the Lock-in Amplifier and feeding back the signal to V_{dc} , the surface potential map is acquired from the feedback signal, V_{dc} .

Figure 14-4 shows the 2D image of the potential distribution of Nanowire Bundle between electrodes.



Figure 14-4. (Left)Surface Height, (Right)Surface Potential

14-2. Setup

1. Devices

Set up your NX system as you would for the ordinary Contact/Non Contact AFM. F or detailed instructions, refer to your NX user's manual.

Caution!

You must set 0V for AC Amplitude before tip approaches to sample. After approach, set the AC Amplitude to 0~3V range. Otherwise, tip can be damaged during approach process.

Note!

To obtain the highest quality EFM images, be sure to familiarize yourself with the operation of the Lock-in Amplifier you are using.

2. Preparing the Tip and Sample

Sample

- 1. Attach the sample to the sample disk by using an electro-conductive adhesive such as silver paste.
- 2. Mount the sample disk on the magnetic sample holder.



Figure 14-5. Sample Preparation

<u>Tips!</u>

Sample bias will be applied through the sample holder and can be controlled by changing the 'Sample bias' value in the SMARTSCAN software. Setting the sample bias value to zero will have same effect as grounding the sample. But if needed, connect the ground wire or the proper external voltage line to the sample. If you have connected the ground wire or external voltage line to the sample, connect the other end of the wire or line to the proper grounding or voltage source, respectively. Any conducting part on the SPM body or the acoustic enclosure bolts can be used as grounding.

Cantilever Tip

You must use conductive tip for the EFM measurement. If you use the metal chip carrier to hold cantilever tip, please follow the steps below.

- 1. Attach the cantilever chip to the chip carrier using adhesive.
- Connect them electrically by an electro-conductive adhesive such as silver paste.
 You can check if they are electrically connected with multi-meter.

Figure 14-6. Cantilever Preparation



14-3. Software UI

The EFM modes can be classified by the method to acquire the sample height and result some difference in software UI. Please note that to improve the product, software UI can be changed without notice.

1. Scan Control Window UI

Figure 14-7 shows the Scan control window for the EFM mode in Non-contact mode base(Left) and in Contact mode base(Right). There are many scan control parameters, but in this section, only the parameters which are introduced in EFM modes are explained. Please refer to the SmartScan[™] manual for all other scanning parameters.

Figure 14-7. Scan Control Window (Left: EFM, Center: EFM-DC, Right: PFM)

Setup	EFM	Setup	EFM-DC	Setup	PFM
Scan Rate	0.30 Hz 🛟	Scan Rate	0.30 Hz 🗘	Scan Rate	1.00 Hz 🛟
Z Slope	0.00 ° 🗘	Z Slope	0.00 ° 🜲	Z Slope	0.00 ° 📫

Tip Bias Servo

Checking the Tip Bias Servo enables feedback control of the potential difference between the tip and the sample by adjusting the tip bias. When the tip bias servo is on, the system adjusts the tip bias to minimize the difference in potential between tip and sample. **Please check this option only in KPFM.** Checking Tip bias servo, Gain is activated to can changing the tip bias servo gain. Tip bias is a voltage applied to the tip. In two pass scan option, this tip bias value is to apply the tip bias only in first scan. Since the effect of electrostatic force needs to be minimized in first scan, tip bias is generally set to zero.





■ Tip Bias Servo Gain

The Tip Bias Servo Gain controls the sensitivity of KPFM feedback control. As this value is higher, the KPFM feedback will be better and the trace/retrace signal of Potential will be matched better. However, if this value is too high, the noise can be shown at Potential signal on the trace window such as Z servo gain parameter in NCM. Tip bias servo gain is recommended to set less than '1' as usual.

■ EFM tip bias

Setup	EFM	- Scan =	S
Scan Rate Z Slope	0.30 Hz ÷	auto	
Scan Area	Z Servo	Bias Lift	
Sample Bias	0	.000 V 🗘	
Tip Bias	0	.000 V ‡	

Figure 14-9. Tip Bias

EFM tip bias is a voltage applied to the tip when the system performs the second scan to get the EFM image. It is activated when Tip-sample distance is checked. EFM tip bias is a voltage applied to the tip when the system performs the second scan of the EFM mode to get the EFM image.

3. Signals

-	EFM	Signals
---	-----	---------

Height	Sample surface morphology
EFM Amplitude	Magnitude of electric force from potential difference. KPFM feedback system works to keep the (EFM Amplitude+EFM phase) signal zero.
EFM Phase	Polarity of electric force from potential difference. KPFM feedback system works to keep the (EFM Amplitude+EFM phase) signal zero as default. If you select 'Phase only' option, the EFM Phase will be only considered for KPFM feedback loop.
KPFM Potential	KPFM Potential on the sample surface.

-PFM Signals

Z Height	Sample surface morphology
PFM Amplitude	Magnitude of electric force from potential difference. KPFM feedback system works to keep the (PFM Amplitude+PFM phase) signal zero.
PFM Phase	Polarity of electric force from potential difference. KPFM feedback system works to keep the (PFM Amplitude+PFM phase) signal zero as default. If you select 'Phase only' option, the PFM Phase will be only considered for KPFM feedback loop.
PFM Quad	Quadrature Signal of Lock in 2

3. Lock-in Setup Window UI

Figure 14-10 shows the Lock-in Setup Window, displayed when [View->Lock-in Setup Window] on the Menus is selected.



Figure 14-10. Lock-in Window

3-1. Lockin Config

There is an independent lock-in channel for each lock-in(1/2/3/4)

- 1st: For NCM Feedback
- 2nd: For Optional Mode Feedback or Monitoring
- 3rd, 4th: Reserved For Later Use

Input Channel

Choose the Input Channel. Currently the A-B(AC), LFM (AC), HEM1, HEM2, HEM3, AUX1, AUX2, AUX3 and None are available. However, the operation mode may select the source for Lock-in automatically.

- Source for Lock-in 1: 'A-B' and 'None' automatically selected in EFM and SCM and EFM-DC, respectively.
- Source for Lock-in 2: 'A-B' and 'HEM3' automatically selected in EFM modes and SCM, respectively.

Phase

When modulating the signal, its phase becomes the reference phase value as a default. This reference phase is shifted so as to choose your preferred phase value by controlling this [Phase] value. The Phase can be controlled from -180 degrees to 180 degrees in 0.01 resolution and one should monitor the Output phase signal (SCM phase in SCM mode and EFM phase in EFM mode) value to receive the optimal value.

Time Constant

Set time constant on internal Lockin post filter which is a kind of 'low pass filter'. Therefore, the filter order is set to 'None', and the time constant value will not contribute the signal since the low pass filter is not applied. It is recommended to set approximately 1~3msec for the time constant to acquire the available signal with 0.2~1Hz. As the time constant value increases, the output signal is smoother. If it increases too much the signal may become blurry. To avoid this, be monitoring/setting the output signal at all times.

Sensitivity

The output signal coming from interaction between the tip and sample is amplified, divided by same amplified amount to avoid the underflow of measuring signal, and finally displayed on the trace window in SmartScan. As this amplified amount called as 'Output Gain' increases, the maximum detectable bias decreases since the detectable bias in NX series is fixed(-10~10V). To know the maximum detectable bias directly and the amplified amount, it suggests to adjust the 'Sensitivity' which is 10V/'Output Gain'. For example, one adjusts 'Sensitivity' from 0.1V to 10V, the maximum detectable bias changes from 0.1V to 10V but the Output Gain decreases from 100 to 1. Please note that 'Sensitivity' is set too low, the detected signal can become too noisy.

Guideline for Sensitivity Setting			
Raw Signal Level 100uV ~ 1mV 1mV ~ 10mV Higher than10mV			
Sensitivity	0.1~1V	1 ~ 10V	10V

Filter Order

Set the filter order(in other words, rate of 'frequency roll off') on internal Lockin post filter which is a kind of 'low pass filter'. Currently, one among 'None', '1' and '2' is selectable. When 'None' is selected, the low pass filter is not applied. As increasing the filter order steeper attenuates higher frequencies than cut off frequency, and can cause the output signal smoother.

3-2. Modulator Config

There is an independent Modulator channel for each Modulator (1/2)

- 1st: For NCM Feedback
- 2nd: For Optional Mode Feedback or Monitoring

Target

Choose the desired channel for modulation. The selectable channels depend on the lock-in channel and the measurement head mode may select the Target automatically.

- Target Control Source for Lock-in 1: Selectable between 'NCM Modulator' or 'Off'. 'Off' is automatically selected in contact based modes such as SCM, EFM-DC.
- Target Source for Lock-in 2: Selectable various control sources such as 'Tip Bias', 'Sample Bias', 'Z Scan' and 'Off'. The Tip Bias is automatically selected in EFM modes. The Sample Bias in SCM and lastly the Z Scanner (SCAN) in the SICM (AC).

Frequency

Frequency for modulation and source to Lockin amplifier is selectable. In case of EFM, in order to differentiate the NCM, you must select the frequency lower than one in NCM modulation (generally, (100-300 kHz) used for NCM modulation). Also, the SCM uses the radio frequency(MHz) for sensing the capacitance.

Therefore, around 17 kHz is generally used for EFM and SCM to avoid any disturbance.

Drive

Modulation Drive is selectable. In case of electrical properties measurement, if the Modulation Drive value increases too much, an electric field may form between tip and sample and may affect the sample surface potential. Thus, it is recommended to select lower than 2-3V.

*Modulation Drive and Modulation&Lock-in Frequency are also selectable through the Lock-in1(NCM)/Lock-in 2(EFM, SCM, SICM(AC) Frequency Sweep window in Setup menu.

Guideline for Sensitivity Setting			
SCM/EFM Amplitude 100uV ~ Level 1mV 1mV ~ 10mV Higher than10mV			
Sensitivity	0.1~1V	1 ~ 10V	10V

14-4. Operation

1. EFM Measurement

- Base mode: Non-contact mode
- Default setup for lock-in 1st, lock-in 2nd

Figure 14-11. Lock-in Setup in EFM

Lockin / Modulator	Setup			👼 🖍 ×
Lockin Config			Modulator Co	nfig
🗖 1st 🗖 2nd	■ 3rd = 4th		■ 1st 🛛 = 2	nd
Input Channel	 None Vertical (A-B) Lateral (C-D / L HEM3 In Aux1 In 		Target	 None Tip Blas Sample Blas Z Scan
Phase Time Constant Sensitivity Filter Order	2nd Order	0.00 ° ‡ 1.0 ms ‡ 0.10 V ‡	Frequency Drive	17.000 KHz 🗘 0.001 V 🗘
			- 3-	🗶 Close

*Note that to improve the product, software UI can be changed without notice.

① Head Mode: Select as 'EFM'.

Figure 14-12. Head Mode Setup



② Channel Config:

Choose signals to be imaged. Recommend to set 'Z Height', 'EFM Amplitude', 'EFM Phase'.

③ Trace Window:

Choose signals to be monitored in real time. Recommended to set 'Height', 'EFM Amplitude', 'EFM Phase'.

④ NCM parameter setting:

As generally done in NCM. Click 'Approach' button to approach the tip to the sample surface. If the approach is successful, the upper half of the Z scanner bar will turn green and the green light at the Stage Control Window will stop blinking. Control the scan parameters and obtain optimized height line profile. Before you begin Lock in setup To get the 'Heght' to complete the Parameter Setting. *Note after checking cantilever specification, choose optimal drive frequency, Amplitude. For specific description, go to Non-contact mode page in User's manual.

5 Lock-in setting for EFM Measurement:

When you select a mode, the EFM Lockin 2 is activated. After that the Input channel is automatically vertical, Target is fixed at Tip Bias. In order for optimized EFM Amplitude/Phase to appear, control the parameters such as Time Constant, Sensitivity and so on.

(For more information about Parameter setting, please refer to 14-6.)

Caution!

Before approach, please make sure that the modulator amplitude is set to zero so as to avoid any damage to the tip.

2. EFM-DC Measurement

- Base mode: Contact mode
- Default Set Up For Lock-in 1, Lock-in 2

Figure 14-13. Lock-in Setup in EFM-DC

Lockin / Modulator	Setup			🗰 🖬 🗙
Lockin Config		Modulat	tor Config	
🗖 1st 🗖 2nd	■ 3rd = 4th	= 1st	a 2nd	
Input Channel	 None Vertical (A-B) Lateral (C-D / LFM) HEM3 In Aux1 In 	Targe	et None Tip Blas Sample Blas Z Scan	
Phase Time Constant Sensitivity Filter Order	0.00 1.0 m 0.10 V 2nd Order	Freque Drive	iency 1	7.000 KHz 🛟 0.001 V 🗘
				X Close

*Note that to improve the product, software UI can be changed without notice.

① Part Configuration:

Head Mode-EFM-DC, Cantilever-loaded cantilever

Figure 14-14. Head Mode Setup

Setup	EFM-DC	
Scan Rate	0.30 Hz 🗘	
Z Slope	0.00 ° 🗘	auto

Note that when installed cantilever is not there, choose a similar spec cantilever and create a cantilever DB. To do so, please refer to the cantilever page in User's manual.

2 Channel Config:

Choose signals to be imaged. Recommend to set 'Z Height', 'EFM Amplitude', 'EFM Phase'.

③ Trace Window:

Choose signals to be monitored in real time. Recommended to set 'Height', 'EFM Amplitude', 'EFM Phase'. Before you begin Lock in setup To get the 'Heght' to complete the Parameter Setting.

④ Parameter setting:

As generally done in Contact. Click 'Approach' button to approach the tip to the sample surface. If the approach is successful, the upper half of the Z scanner bar will turn green and the green light at the Stage Control Window will stop blinking. Control the scan parameters and obtain optimized height line profile. Before you begin Lock in setup To get the 'Heght' to complete the Parameter Setting.

(5) Lock-in setting for EFM Measurement: When you select a mode, the EFM Lockin 2 is activated. After that the Input channel is automatically vertical, Target is fixed at Tip Bias. In order for optimized EFM Amplitude/Phase to appear, control the parameters such as Time Constant, Sensitivity and so on. (For more information about Parameter setting , please refer to 14-6.)

Caution!

If the set point is increased too much, force exerted on the tip will becomes too high. This will result in tip breaking or the damage to the sample surface. Also if the set point is increased too much, the tip can come to ohmic contact with sample surface resulting in shorts and excessive current between tip and sample even thgough sample is an insulator since it may have the defect region.

Caution!

Before approach, please make sure that the modulator2 amplitude is set to zero so as to avoid any damage to the tip.

3. PFM Measurement

- Base mode: Contact mode
- Default Set Up For Lock-in 2nd, Lock-in 2nd (Vertical PFM)

Lockin / Modulator	Setup		📪 🐚 🗙
Lockin Config	· · · · · · · · · · · · · · · · · · ·	Modulator Config	1
■ 1st 🛛 ■ 2nd	¤ 3rd ¤ 4th	■1st ■ 2nd	
Input Channel	 None Vertical (A-B) Lateral (C-D / LFM) HEM3 In Aux1 In 	Target 🖷	None Tip Blas Sample Blas Z Scan
		Frequency	17.000 KHz 🛟
Phase Time Constant	0.00 ° 🗧	Drive	0.001 V ≑
Sensitivity	0.10 V ÷		
Filter Order	2nd Order +		
			X Close

Figure 14-15. Lock-in Setup for Vertical PFM

• Default Set Up For Lock-in 3rd, Lock-in 2nd (Lateral PFM)

Lockin / Modulato	r Setup		📪 🗅 🗙
Lockin Config		Modulator Co	onfig
■ 1st ■ 2nd	■ 3rd 4th	= 1st = 2	nd
Input Channel	 None Vertical (A-B) Lateral (C-D / LFM) HEM3 In Aux1 In 	Target	 None Tip Bias Sample Bias Z Scan
Phase	0.00 ° 茾	Frequency	17.000 KHz 🗘
Time Constant	1.0 ms 🗘	Drive	0.001 V 📫
Sensitivity	10.00 V 🗘		
Filter Order	2nd Order 🔹		
			× Close

Figure 14-16. Lock-in Setup for Vertical PFM

*Note that to improve the product, software UI can be changed without notice.



④ Part Configuration: Head Mode-PFM, Cantilever-loaded cantilever



Note that when installed cantilever is not there, choose a similar spec cantilever and create a cantilever DB. To do so, please refer to the cantilever page in User's manual.

- (5) Channel Config: Choose signals to be imaged. Recommend to set 'Z Height', 'P FM Amplitude', 'PFM Phase', 'PFM Quad'. (Vertical PFM) Choose signals to be imaged. Recommend to set 'Z Height', 'PFM Amplitude', 'P FM Phase', 'PFM Quad'. (Lateral PFM)
- 6 Trace Window: Choose signals to be monitored in real time. Recommended to set 'Z Height', 'PFM Amplitude', 'PFM Phase'.Before you begin Lock in setup To get the 'Z Height' to complete the Parameter Setting.
- 6 Parameter setting: As generally done in Contact. Click 'Approach' button to approach the tip to the sample surface. If the approach is successful, the upper half of the Z scanner bar will turn green and the green light at the Stage Control Window will stop blinking. Control the scan parameters and obtain optimized height line profile. Before you begin Lock in setup To get the 'Heght' to complete the Parameter Setting.

⑦ Lock-in setting for EFM Measurement:

When you select a mode, the PFM Lockin 2 is activated. After that the Input channel is automatically vertical, Target is fixed at Tip Bias. In order for optimized PFM Amplitude/Phase to appear, control the parameters such as Time Constant, Sensitivity and so on.

(For more information about Parameter setting, please refer to 14-6.)

Caution!

If the set point is increased too much, force exerted on the tip will becomes too high. This will result in tip breaking or the damage to the sample surface. Also if the set point is increased too much, the tip can come to ohmic contact with sample surface resulting in shorts and excessive current between tip and sample even thgough sample is an insulator since it may have the defect region.

4. KPFM(Scanning Kelvin Probe Microscope) Measurement

KPFM measurement procedure is same as for the EFM or EFM-DC. The tip DC voltage is controlled by feedback to keep the potential difference between the tip and the sample at zero. Then, value of the absolute surface potential can be obtained by using the KPFM.

- ① Follow steps 1 to 9 of EFM or EFM-DC.
- ② Input channel & Trace Window: Add "Potential" signal.
- ③ Scan Control Window: Change the scan parameters as follows

- Scan size: 0

- Sample bias: 0

- ④ Title Bar&Trace Mode: Perform the Potential Sweep.
 - i. Click the "Trace Mode(maintenace on the tap.)" on the toolbar, and select "Potential(sample bias)" at the driving source combo box.

Warning!

Make sure that Potential is selected for driving source. If you select other parameters, NX system and/or the sample can be severely damaged.

ii. Trace Mode (mauntenace): When the XY Scanner is changed off, then "Sweep(Start)" button is enabled.





iii. Trace Mode&Lock-in Setup Window:Click the "Sweep" button.

Then, DC bias to the sample will be applied from -10V to 10V. The EFM phase curve versus potential is plotted. Adjust the "Phase" of the output signal on the Lock-In Amplifier control panel to center the curve in the window.



Figure 14-19. Centering the Trace Curve

EFM phase is used for KPFM feedback. This value includes polarity of sample and Y offset from system in fact. Therefore, we need to set the signal above by adjusting phase shift on the Lock-in Amplifier in order to distinguish the polarity of the sample potential.

- iv. Click the "Stop" button, and return to the 'Scan Mode Control' by clicking the "Scan Mode" button in the main toolbar.
- (6) Scan Control Window: Set the scan parameters to scan the sample properly and confirm if the signals are acquiring correctly.
- ⑦ Check the "Tip Bias Servo" button



Figure 14-20. Tip Bias Servo

Then, tip bias will be applied to make ω component from Lock-in Amplifier zero.

*Use Phase Only in Tip Bias Servo

Both EFM Amplitude and EFM Phase $[R_0sine_0-R_1sine_1, (R_0: Modulator2 amplitude from Lock-in Amplifier setting, <math>e_0:Modulator2$ phase from Lock-in Amplifier setting, $A_1:Current$ EFM amplitude, e1:Current EFM Phase) will be used for KPFM tip bias feedback. Checking this button make EFM Phase used only in KPFM feedback loop system. Therefore, tip bias is applied to make EFM phase difference $[sine_0-sine_1]$ zero this option is located in [Manual->bias].



Figure 14-21. Use Phase Only in Tip Bias Servo

- ⑧ Trace Window: Monitor the "Potential" signal. The potential curve is a representation of the surface voltage profile.
- ⑨ Adjust the Lock-in Amplifier parameters (Time constant, Sensitivity and etc.) and the Tip bias servo gain on SmartScan[™] to optimize the Potential signal.

14-5. Practice

Park Systems offers the EFM test sample with EFM mode. You can test your EFM and imaging skills by obtaining an EFM image of the test sample and comparing it with the expected image. This section describes the test sample and expected results.

1. EFM

A. Test Sample

Figure 14-22. shows the standard test sample and its magnified view. The test sample consists of two comb shaped Ti/Au electrodes with each tooth of one elec trode lying

between teeth of the other. One electrode is electrically connected to the metal plate, the

sample holder, and thus can be biased through NX electronics. The other electrode is connected to the wire slot. If the wire is connected between this slot and the ground, it will be grounded.





Sample bias will be applied through the sample holder and can be controlled by changing the 'Sample bias' value in the SmartScan[™] software.

For grounding, any conducting part on the SPM body or the acoustic enclosure bolts can be used.

B. Obtaining an EFM Image of the Test sample

1. Test Sample Installation:

a. Mount the test sample on the sample holder.

b. Connect the ground wire between ground slot and the grounding.

2. Head Mode: EFM, KPFM

3. Recommended Scan Parameters:

- a. Scan size: 20 µm
- b. Tip bias: 0 Volts
- c. Sample bias: ± 1V
- d. Frequency: 17kHz
- e. Drive: 1V
- f. Phase: 40 Degree
- g. Time Constant: 2ms
- h. Filter: 2nd order
- i. Sensitivity: 1V

4. Results:

Figure 14-23 shows the expected EFM and surface height signals and Figure 14-24 shows the actual EFM and height image of the test sample. Since all the neighboring teeth of the comb shaped electrode are the same height, the surface height signal is in the shape of a square wave. However, since the neighboring electrodes differ in potential, every other height peak is missing in the EFM image.



Figure 14-23. Expected results of the test sample



Figure 14-24. Actual Height and EFM image of the test sample



Sample bias = -1V







[Height line profile]







Sample bias = 1V



[EFM Phase Image]











2. PFM (EFM-DC)

A. Test Sample

PFM (EFM-DC) provides ferroelectric piezo material for the test sample. The thickness of piezo material(Zr/Ti composition) is approximately 150nm and the substrate is Ti/Pt on SiO_2/Si wafer. The test sample Figure below shows the test sample electrically connected to the metal plate.



Figure 14-25. EFM-DC Test Sample

B. Obtaining an PFM Image of the Test sample

1. Test Sample Installation:

Mount the test sample on the sample holder.

2. Head Mode: PFM mode

3. Recommended Scan Parameters:

- a. Scan size: 1 µm
- b. Tip bias: 0 Volts
- c. Sample bias: 0 Volts
- d. Frequency: 17kHz
- e. Drive: 1V
- f. Phase: 40 Degree
- g. Time Constant: 2ms
- h. Filter: 2nd order
- i. Sensitivity: 5V

4. Results:

This piezo material has the ferroelectric property and possess a spontaneous electric polarization. Therefore, the sample affects the electric field between tip and sample and we can get the PFM image below.



Figure 14-26. PFM Image of Test Sample

C. Obtaining an PFM Image of the Test sample after Domain Switching

This electric polarization of this piezo matrial can be reversed by external electric field. We can confirm it through PFM after applying the +/- voltage to the tip.

1. Test Sample Installation:

Mount the test sample on the sample holder.

- 2. Head Mode: EFM-DC mode or PFM mode
- 3. Recommended Measurement Procedure:



a. Scan the sample with Scan size: 8um, Tip Bias: -10V
b. Scan the sample with Scan size: 5um, Tip Bias: 10V
c. Acquire the EFM image with Scan size: 10um, Tip Bias: 0 V

Scan XY offset of (a, b) should be same.

4. Results:

Scanned area of 8 µm has electric domain by -10 V tip bias. After that, tip charged +10V scanned 5um scan area with same offset. Therefore, the electric polarization of 5um scanned area is reversed. Therefore, PFM phase should have opposite direction each other. We can confirm it with PFM.





3. KPFM

A. Test Sample

The HOPG(Highly Oriented Pirolytical Graphite), a material that consists of many atomic layers of carbon. is provided as the KPFM test sample. It is used a lot of applications by its atomic flatness, cleanness and conductivity. The parallelism of atomic layer is characterized by "mosaic spread angle" and the HOPG provided in KPFM has 0.7 degree high quality in it and 10 x 10 mm² in size, 1.2 mm(\pm 0.2) in thickness. Figure 24 shows the provided sample and its magnified view. This test sample electrically connected to the metal plate by electro-conductive adhesive. The metal plate is attached onto the sample holder, and thus the sample can be biased through NX electronics.



Figure 14-28. HOPG

B. Obtaining an KPFM Image of the Test sample

5. Test Sample Installation:

a. Mount the HOPG test sample on the sample holder.

6. Head Mode: EFM

7. Recommended Scan Parameters:

- a. Tip bias: 0 Volts
- b. Sample bias: Change the 'Sample Bias' during scanning.
- c. Frequency: 17kHz
- d. Drive: 0.5V
- e. Phase: 40 Degree
- f. Time Constant: 2ms
- g. Filter: 2nd order
- h. Sensitivity: 0.1V

8. Results:

The potential value according to the applied sample bias will be observed by KPFM. Figure 25 shows Potential image by KPFM and EFM image by EFM during scanning the HOPG sample(scan size: $4\mu m \times 5\mu m$) and applying the sample bias as $(0V \rightarrow +1V \rightarrow +2V \rightarrow +3V \rightarrow +4V \rightarrow +5V \rightarrow 0V \rightarrow -1V \rightarrow -2V \rightarrow -3V \rightarrow -4V \rightarrow -5V \rightarrow 0V)$. As you see in each line profile, unlike EFM amplitude, the Potential value was measured equally with the applied sample bias. In other words, the KPFM can distinguish the real potential difference.



Figure 14-29. Expected results of the test sample
14-6. Advanced Application

Notes on EFM Imaging

• Scan parameter setting (Height)

A bad Height image indicates that the distance between the sample and the tip is not constant – EFM signals obtained when this is the case cannot be considered reasonable data.

In order to make EFM imaging exact, scan parameters (scan rate, set point, z-servo gain) must be adjusted so the distance between tip and sample is always constant and yields Height Image of good quality. A bad Height Image indicates that the sample-tip distance is not being maintained at a constant, and EFM data obtained when this is the case cannot be considered reasonable data.

• Lock-In Amplifier Parameter Setting (EFM Image)

In order to obtain EFM data, Lock-In Amplifier Parameters must be adjusted appropriately for each situation. There are 6 Lock-In Amplifier Parameters: Frequency, Drive voltage, Phase, Time constant, Sensitivity, Filter order.

- Frequency; Use of 17kHz is effective for obtaining good results.
- Drive voltage; The larger the Drive voltage, the higher the magnitude of response to surface potential, and when this value is too large it can act as DC bias on the sample, so we recommend usage with this value within 3V.
- Phase; The EFM phase can determine whether the surface potential value of the sample is + or –. This parameter modulates the offset of the EFM Phase signal.
- Time constant; The Time constant is used in reading the averaged data

received for the duration of time set as the Time constant in Lock-In Amplifier. This parameter is highly relevant to scan rate. For example, if scan rate is 0.5 Hz and pixel count is 512 then pixel-to-pixel travel time is 2ms, but if the Time constant is 4ms then the data for 2 pixels are averaged into 1 EFM data point input and resulting images may be blurry. Or, if the time constant is too short, resulting images may be blurry.

 Sensitivity; This parameter determines EFM Amplitude signal detection range.
You can set this parameter in accordance with the below table while monitoring the EFM Amplitude signal value.

Guideline for Sensitivity Setting					
EFM Amplitude	<10mV	<100mV	<1V	>1V	
Sensitivity	0.01V	0.1V	1V	10V	

- **Filter order**; This is a Low pass filter function and we recommend use of 2nd order.

When the above Lock-In Amplifier Parameter Settings are unsuitable, Height image output may be inaccurate and EFM image output may be noisy or blurry. This is why EFM data must be obtained after setting Lock-in Amplifier parameters to suitable values using line profiles as guide.

Examples

1. Influence of Drive voltage on Height

Drive voltage may be set to a higher value in order to increase the tip's reaction sensitivity to surface potential. However, setting the Drive voltage to a large value increases the surface potential of the sample and this surface potential pushes the tip away from the sample, resulting in inaccurate imaging of surface morphology. Therefore, Drive voltage increases must be to within a range which does not cause height signal distortion.

Figure 14-30. Z Height line profiles according to Drive voltage; Drive voltage = a) 5V, b) 1V



2. Influence of Time Constant on EFM Image

As aforementioned, the Time constant is closely related to the scan rate, and must be adjusted appropriately according to scan rate. Examine EFM signal' s Line profiles for forward/backward consistency, noisiness, or blurriness and adjust the Time constant accordingly prior to imaging.

Figure 14-31. EFM line profiles according to Time constant; Time constant = a) 1ms, b) 3ms, c) 5ms



Figure 14-32. EFM Amplitude Image according to Time constant; Time constant = a) 10ms, b) 5ms, c) 1ms



Figure 14-33. Line profiles of Figure 3 EFM Amplitude Image



As seen above in Figure 14-31, Figure 14-32, and Figure 14-33, if the Time constant is long then image and line profile become blurry, while if the Time constant is short then image and line profile become noisy.

• EFM-DC

As EFM-DC is distinct only in that standard EFM operation is based on Contact mode, with identical imaging procedures for all other measurements, there should be no difficulties in obtaining images through adjusting the parameter setting as detailed afore in 1, 2. As domain direction may be known through the EFM Phase, EFM-DC is mainly used to obtain Phase image.

Examples

Influence of EFM Image on Time constant

Longer Time constant produces blurry images while shorter Time constant produces noisy images, as with EFM.

Figure 14-33. EFM Phase Image according to Time constant; Time constant = a) 3ms, b) 1ms, c) 0.5ms



• PE curve

PE curve is obtained as a good method for examining Piezoelectric properties. To observe the characteristics of Ferroelectric good way is to obtain a PE curve. In order to obtain a good result of PE Curve should be setting as well as to the Time constant of sample bias sweep period, Lock-in setting.

Examples

PE Curve according to Sample Bias Period.

Sample bias sweep period is not enough time is given short sample of this domain is difficult to return to observe the hysteresis.

Figure 14-34. PFM Phase Amplitude according to Sample Bias sweep period; a) 1s, b) 5s, c) 10s



Figure 14-35. PFM Phase according to Sample bias sweep period; a) 1s, b) 5s, c) 10s



PE Curve according to Time constant.

If the time constant in the Lock-in setting is set faster PE curve is noisy, and if the time constant is set to slow PE curve emerges blurry.





Figure 14-37. PFM Phase according to Time constant; Time co a) 1ms, b) 5ms,

c) 10ms



Notes on KPFM Imaging

• Adjust scan parameters to obtain good Topography results.

Bad Topography results indicate that the distance between the sample and the tip is not constant - KPFM signals obtained when this is the case cannot be considered reasonable data.

• If KPFM Potential Image is blurry or noisy.

Example

· KPFM Potential Images depending on Tip bias servo gain settings



Figure 14-38; a) Tip bias servo gain=0.1, b) Tip bias servo gain=0.3, c) Tip bias servo gain=0.5



Figure 14-39. Line profiles of KPFM Potential Images

2): Figure 14-39-a, 1): Figure 14-39-b, 3): Figure 14-39-c

When taking KPFM scans, the KPFM Potential image obtained may be blurry as shown in Figure 14-38-a, or noisy as shown in Figure 14-38-c. In this case, adjusting the Tip bias servo gain can improve the quality of the KPFM Potential image.

Figure 14-38-a shows an image which is blurry due to a small Tip bias servo gain value,

while Figure 14-38-c shows an image which is noisy due to a large Tip bias servo gain value. Adjusting the Tip bias servo gain value can yield results such as Figure 14-38-b. In Figure 14-39, the line profiles of each KPFM Potential image in Figure 14-39 show noisier results with larger Tip bias servo gains and blurrier results with smaller Tip bias servo gains. It is important to run line scans to look at line profiles and set the right Tip bias servo gain prior to obtaining KPFM Potential images.

Tip bias servo gain setting

• KPFM Potential line profiles by Tip bias servo gain setting.



Figure 14-40-a. Tip bias servo gain=0.1



Figure 14-40-b. Tip bias servo gain=0.05





Figure 14-40-a displays a noisy line profile resulting from a large Tip bias servo gain value and Figure 14-40-c displays a blurry line profile resulting from a small Tip bias servo gain value. Adjust the Tip bias servo gain value to minimize line profile noise and synchronize forward/backward lines.

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