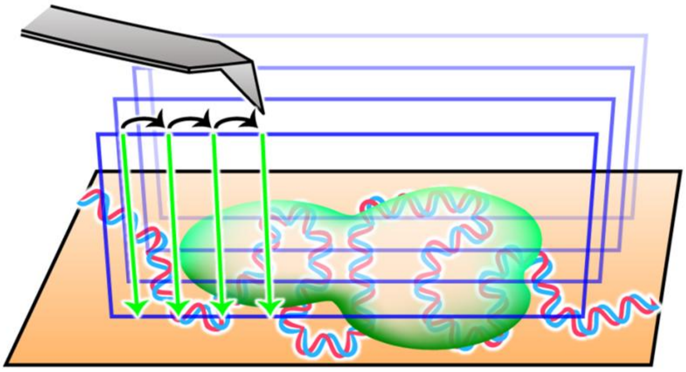
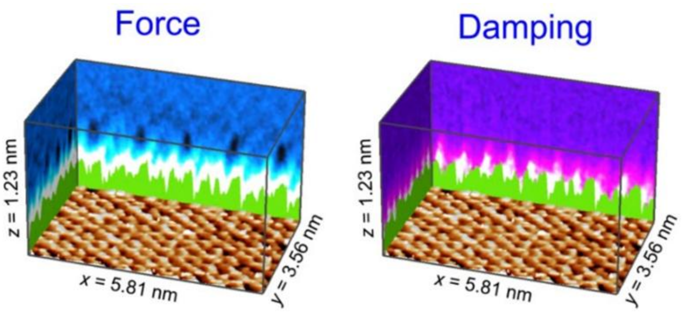
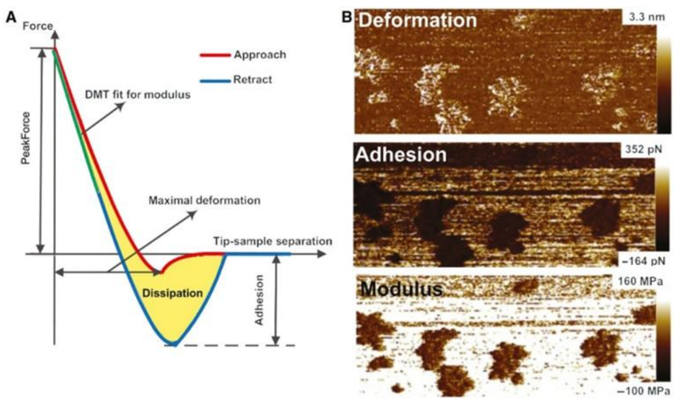
Quick Guide for 3D Force Mapping

Basic principle of 3D force mapping





K. Umeda et al., Phys. Rev. Lett., 122, 116001 (2019).



K. Xu et al., Nanotechnol. Rev. 7 605–621 (2018)

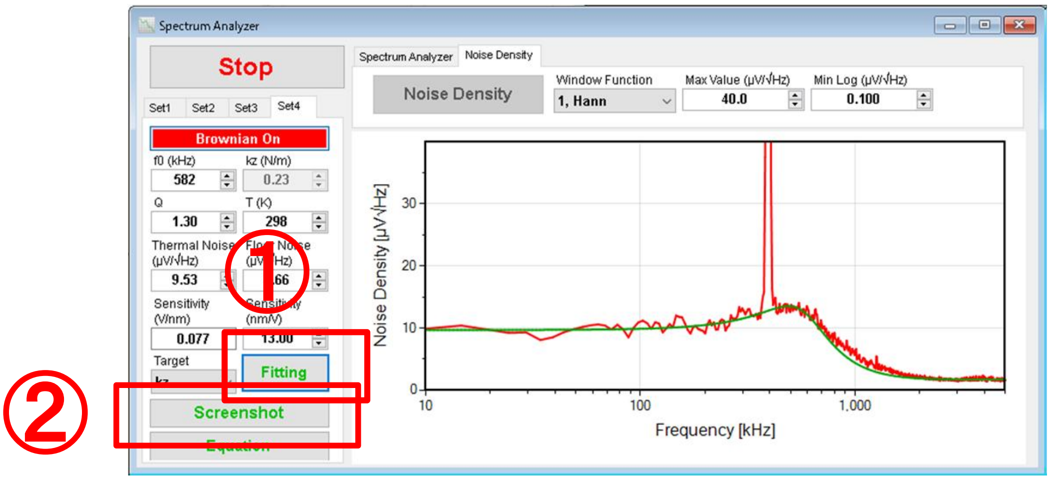
When the tip is brought closer to the sample, the tip receives force from the sample surface, which changes the deflection of the cantilever. The measurement of the deflection signal with respect to the tip-sample distance is called the force curve. A measurement that obtains 3D volumetric data by repeating this force curve measurement on each pixel on the surface is called a 3D force map (other names include force volume, peak force tapping, etc.).

A major advantage of the 3D force map is that, unlike the normal topographic measurement, it is possible to visualize a three-dimensional force field. Moreover, it is possible to quantitatively obtain information on surface physical properties at the nanoscale. In contrast, the disadvantage is that the frame rate is lower than that of normal topographic imaging.

Operation before 3D force map experiments

1. Brownian Fitting

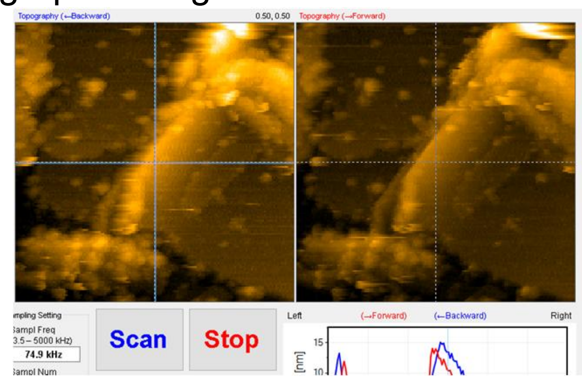
For the analysis of force curve data, the spring constant (kz) of the cantilever is needed to be obtained by fitting the spectrum onto the theoretical equation. For the fitting, the Sensitivity(V/nm) of displacement detection is needed. But tentatively, a typical value should be input to the Sensitivity box, and in the analysis after experiments, it will be corrected by the value exactly estimated. By pressing the Screenshot button, the program recognizes the fitting was completed. If the scan was tried without pressing the Screenshot button, a confirmation error message box appears, so press the Screenshot button for sure.



2. Topographic Imaging

Slope correction should be done before the force experiments.

If the height variation is larger than the Z range of force mapping, the tip inevitably crashed into the surface. To avoid this, software checks whether the Z range is safe or not before 3D force map experiment based on the topographic image data.

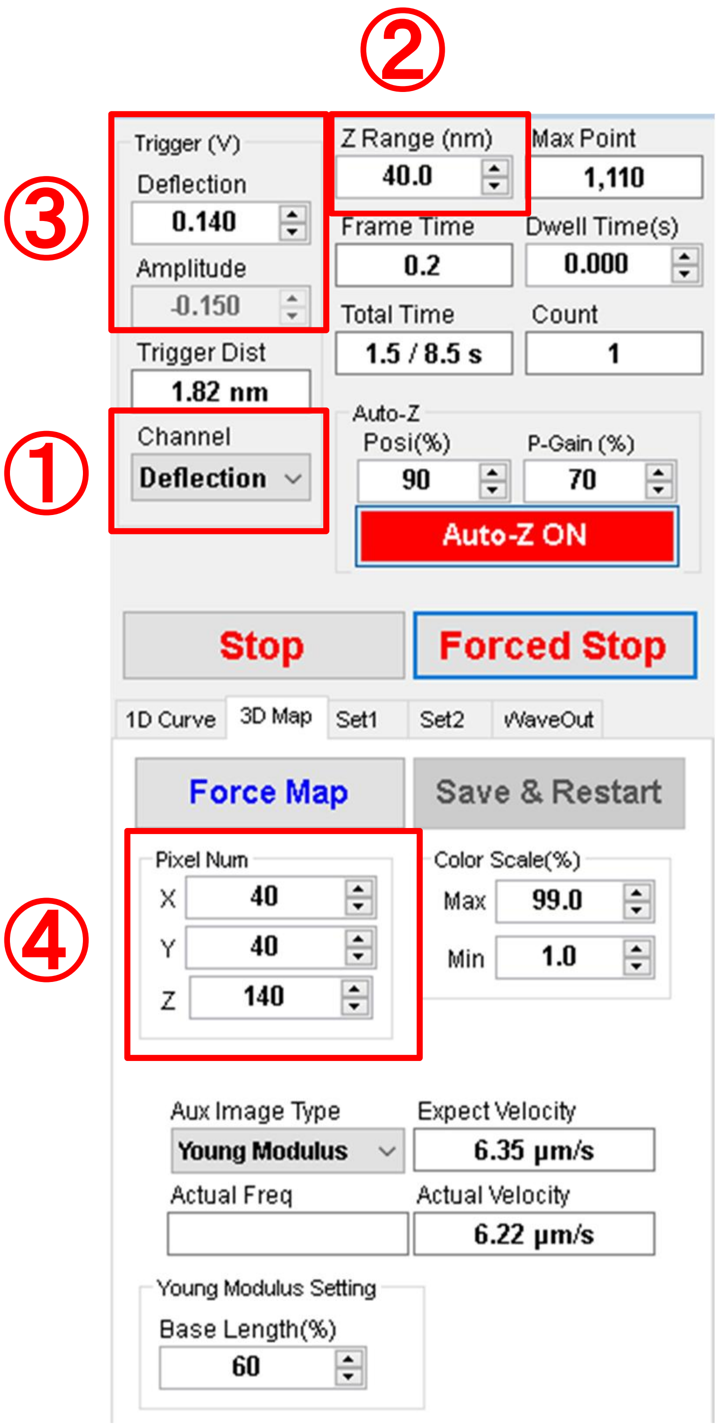


Setting for Controller

Front panel of Tsuji feedback circuit.

Confirm that the “Z Modulation” switch is flipped on before the measurement. Just in case that it is flipped off, flip it on. Also check that a BNC cable is properly wired between the connector “Z modulation in” on the back panel of feedback circuit and 4CH of AD3.

Parameter Setting before 3D force mapping



Trigger Channel:

Check that Trigger Channel is set to Deflection signal for sure.

The software reload the previous setting of Trigger Channel.

Z Range:

Set scan range along Z axis.

Trigger (V):

Predetermined voltage at which the approach is automatically stopped and returned to far from the surface.

With increasing Trigger, the force applied to a sample becomes larger.

When the Reference in Trigger Setting is set to Far Value, the signal relative to the signal at the far end is used. For Absolute setting, the absolute value of the signal is used.

To retrieve Young's modulus, the tip must be sufficiently indented into the surface.

Pixel Num:

Set pixel numbers in the XYZ axes.

With increasing the pixel number, the spatial resolution becomes higher but the measurement time becomes longer.

If you want to do quantitative Young's modulus measurement, increase the Z pixel sufficiently.

Aux Image Type:

Specify the data type on 2CH image. Notice that only qualitative analysis is available and post analysis using 3D Force Map Viewer is required for quantitative evaluation.

Young Modulus Base Length (%):

When the Young's modulus fitting is abnormal, adjust this setting.

Operation before starting 3D force mapping

Machine with switching circuit installed

(Trigger Deflection) :

There is no hardware change, so just press the “Force Map” button to start the 3D force mapping.

(Trigger Amplitude) :

In the setting for deflection, the excitation signal is automatically stopped. So to avoid this, please bypass the excitation cable through the switching circuit.

Machine without switching circuit

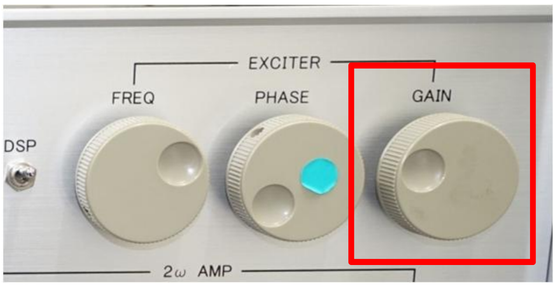
(Trigger Deflection) :

①Switch from the Run to Hold mode on the front panel of feedback circuit.

②Set the GAIN of Fourier amplitude detector to zero.

After the 3D force map, to return to the topographic imaging, restore the above setting in the order of ①→②.

①

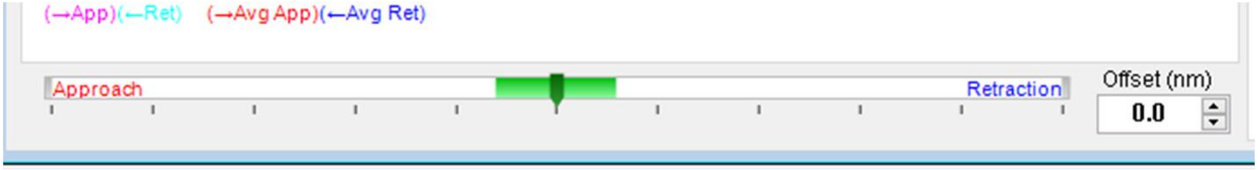


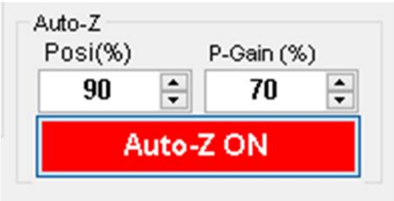
②

(Trigger Amplitude) :

There is no hardware change, so just press the “Force Map” button to start the 3D force mapping.

Operation during 3D force mapping



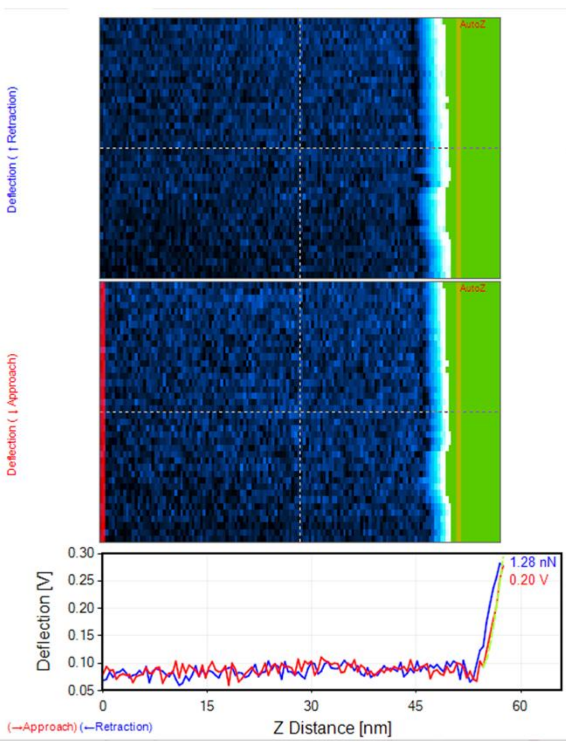


Offset (nm):

Middle green bar indicates the scanning range. When the surface position is shifted by thermal drift, adjust the slider to correct the Z position.

Auto-Z:

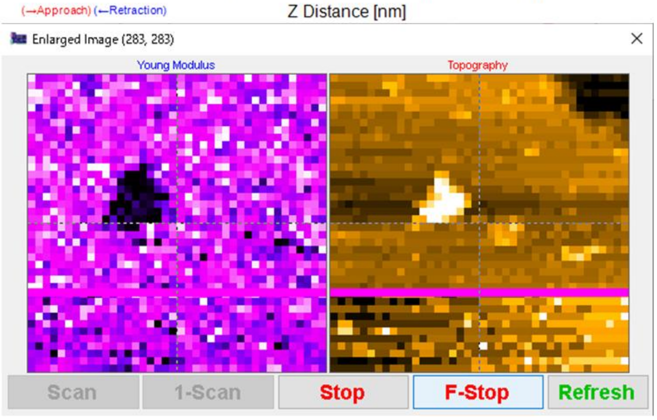
When Auto-Z is activated, the Z position is automatically adjusted by software. P-Gain sets the speed of the adjustment.



When the scanning range reaches the end of the offset range, by holding the mouse middle button, stepping motor can be automatically moved in the same way as the topographic imaging.

An extracted data set at the center of the image is displayed in the 1D profile, where the red and blue curves indicates the approach and retraction force curves. The green curve indicate the fitted Hertz equation for Young's modulus.

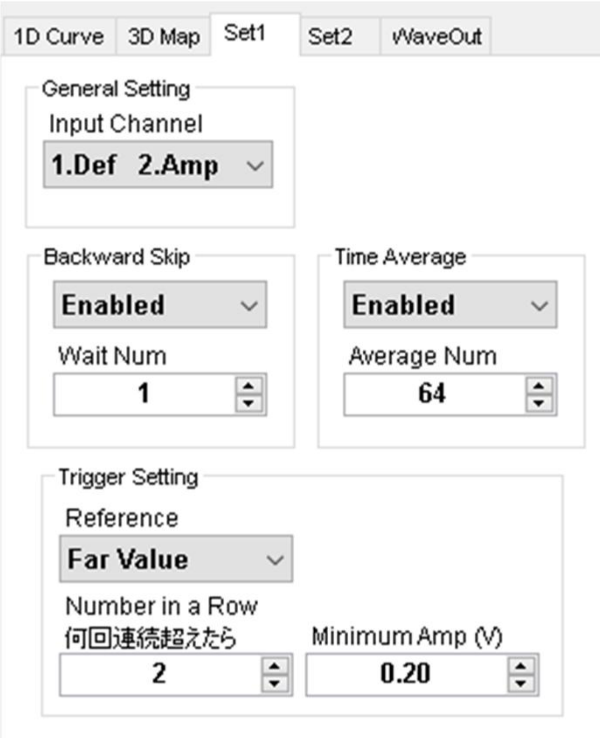
The blue text on the top right corner indicate the maximum tip-sample interaction force. Please adjust the trigger while checking the force is appropriate.



The right image displays reconstructed topographic image, and the left image displays the data that is set in Aux Image Type.

By pressing the right click button, the center of the image can be adjusted in the same way as the topographic imaging.

Other Setting



Input Channel:

This setting is changed only when the phase signal is acquired with amplitude signal. When the deflection is used as trigger, this setting is no use.

Backward Skip:

By skipping the backward movement, the acquisition time can be reduced by half. The retraction data is required for the analysis of pull-off and energy dissipation but it is not necessary for that of Young's modulus. When the adhesion force is very strong, the deflection signal is depressed at the far end of the force curves. In this case, please increase the Wait Num to increase the wait time at the far end of the force curve or disable the backward skip option.

Time Average :

This function is for sampling the signal several times and calculating the average data in each height pixel. This is useful for reducing the damage to a sample but increases the acquisition time. This function is used only when the amplitude signal is used as trigger.

Approach Retraction Velocity:

To increase the approach/retract speed, decrease the Pixel Num Z value. Conversely, to reduce the speed, increase Pixel Num Z. If you wish to acquire data at a lower speed but increasing the pixel number becomes impractical—or if you want to lower the speed without changing the number of pixels—please enable Time Average and adjust the Average Num parameter accordingly. The sampling frequency during force curve acquisition is approximately 30 kHz, which defines the upper limit of the achievable speed. If triggering is not required, technically it is possible to acquire data at even higher speeds.

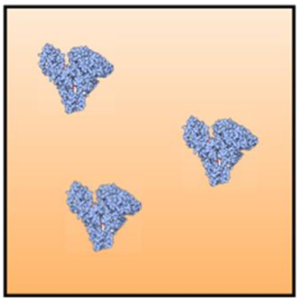
Operation after 3D force mapping

1. Calibration of deflection Sensitivity

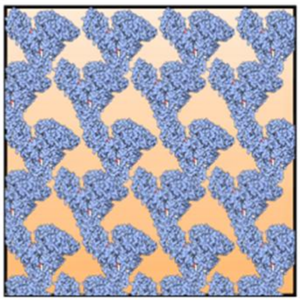
The deflection sensitivity varies ±20% depending on the position of focused laser beam and objective lens. So the sensitivity is better to be measured for each experiment for quantitative measurement.

To retrieve the sensitivity, the force curve or 3D force map should be acquired with high trigger. Therefore, this should be done after experiments to avoid the risk that the tip is damaged.

When a hard substrate, e.g., mica, is exposed to the surface, the sensitivity can be obtained from a 3D force map image, so additional experiment is not required.



When a whole surface is covered by soft molecules, after cleaving a mica again, the force curve or map should be acquired using the same cantilever to calculate the sensitivity. The cantilevers in the same batch have a similar sensitivity, and a calibration only for one cantilever may be enough if quantitative analysis is not required.



Quick Guide for 1D Force Curve

Advantage of 1D force curve mode

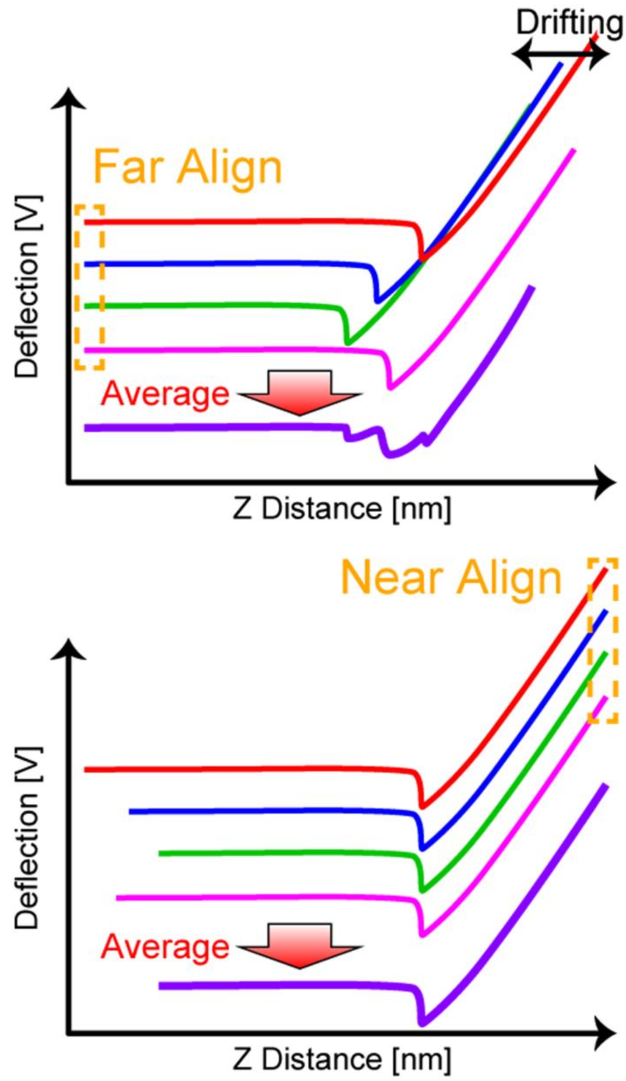
In most of the cases, 3D force mapping is useful, but 1D force curve mode has advantage over 3D force mapping mode as below.

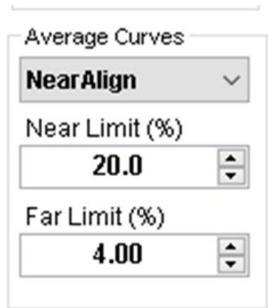
In 3D force mapping, Auto-Z corrects the drifting in each ZX map, and hence Z pixel number is hard to be increased. In 1D force curve, the drifting is corrected in each force curve.

For 3D mapping, offline 3D viewer is required for detail analysis such as background subtract and slope measurement while it can be done online for 1D force curve.

So if site-specific measurement is not prerequisite, 1D force curve has much advantage.

Average Curves





For quantitative analysis, several force curves should be averaged; however, the drifting along Z direction is problematic.

There are two averaging modes as below

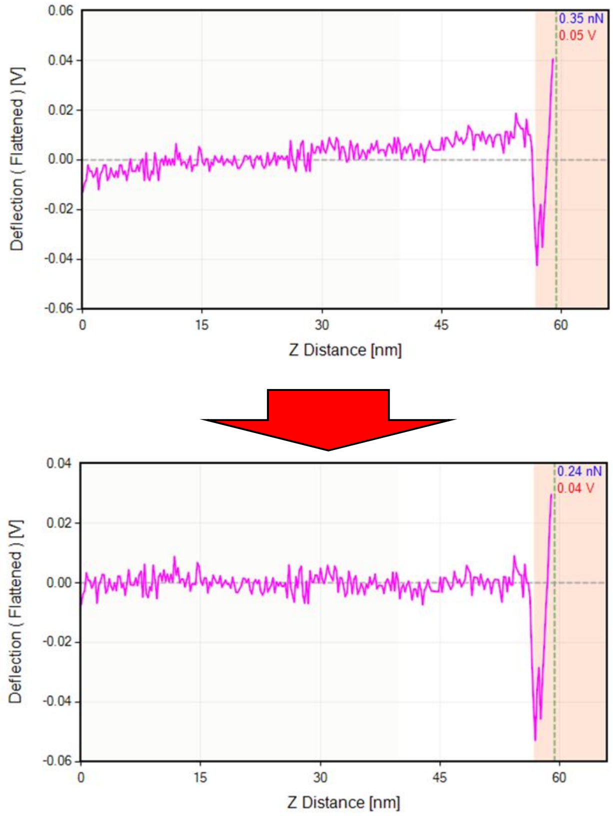
Far Align :

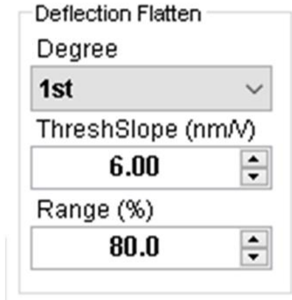
Force curves acquires are simply averaged. It is difficult to appropriate results when the Z drifting is large because the surface position is fluctuated.

Near Align :

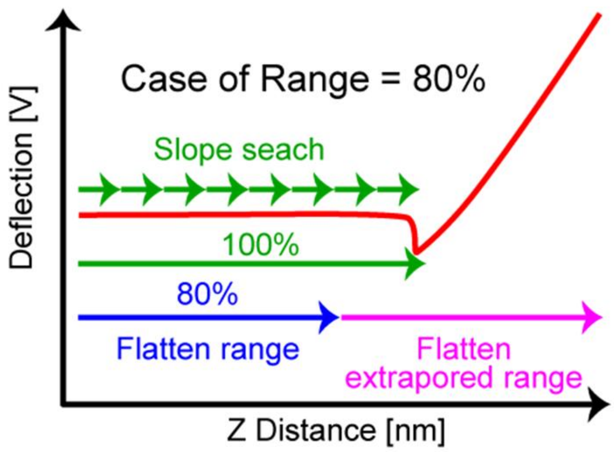
After aligning the near end of the force curves, they are averaged. This mode is effective even when the severe drifting condition. In most of the cases, Near Align mode should be used.

Deflection Flatten





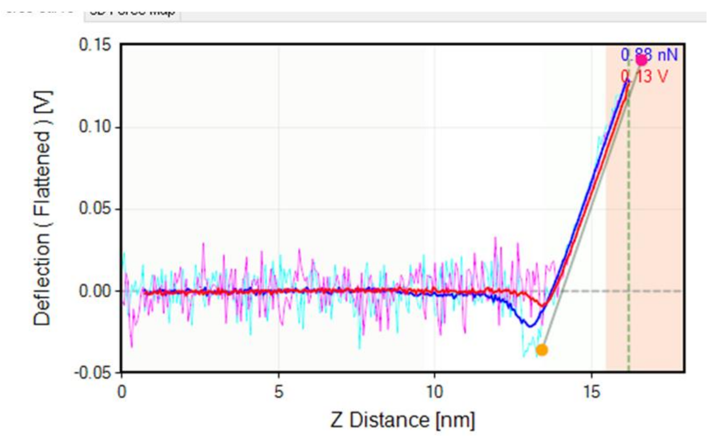
The deflection signal in the force curve has often distance dependency even far from the due to the interferes of the laser beam. This unwanted fluctuation is needed to be corrected prior to the slope measurement. Therefore, a polynomial fitting subtraction should be processed to the region where no tip-sample interaction occurs follows the algorithm below.

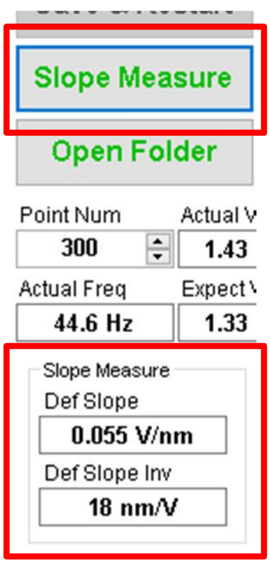


First, to detect the distance where the tip first contacts with the surface, program searches the position where the slope exceeds ThreshSlope. Then, polynomial fitting is processed to the region between the far end to reduced range specified by Range(%). And the subtraction is performed to whole curve by extrapolating the polynomial to the surface region.

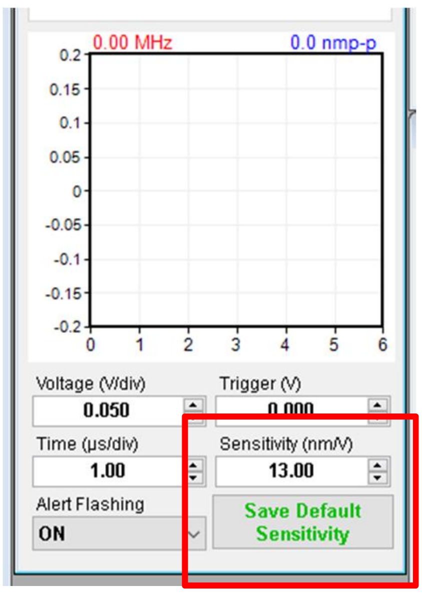
Slope Measure

For the quantitative analysis of 3D force map data, accurate value of sensitivity is required. Sensitivity is calculated by dividing the Z piezo displacement by the cantilever deflection signal in the unit of voltage. Notice that the piezo constant of Z piezo is calibrated accurately beforehand.





For the estimation of Sensitivity, press the Slope Measure button. Then, draw the start and end point by pressing the left and right click button, respectively. By fitting the slope to the force curve, slope which reflects the sensitivity is displayed in the Slope Measure groupbox.



Sensitivity measured can be saved as a default value by inputting the value to the Sensitivity numeric box and pressing the Save Default Sensitivity button on the Z piezo window.

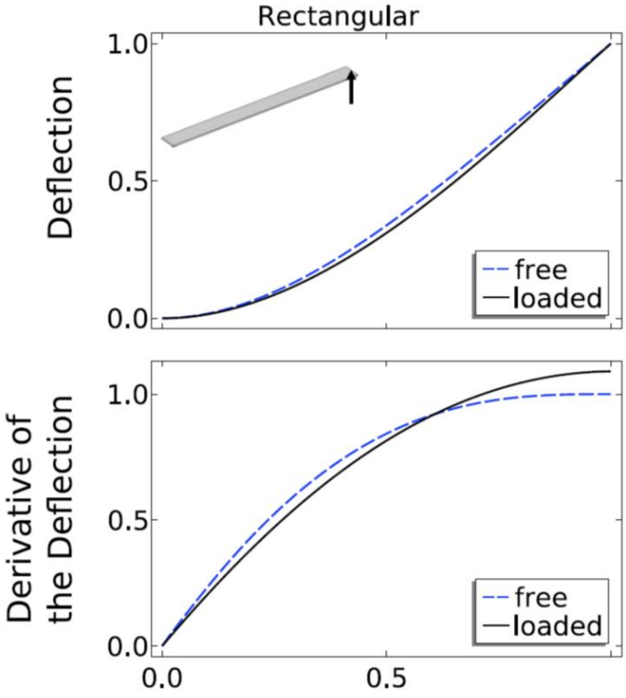
Theory of Force Curve Measurement

Sensitivity correction formula

In high-speed AFM, optical beam deflection (OBD) method is used to detect cantilever displacement. This method does not directly measure the displacement (z) of the cantilever itself, but the differential (dz/dx) (also called the angle) of the cantilever displacement. Since dz/dx is proportional to z, z can be measured from InvOLS (Inverse of optical sensitivity) for the same vibration mode. On the other hand, since the vibration modes are different between static mode and dynamic mode, the proportional coefficient of dz/dx and z and InvOLS are different. In static mode, force is applied only to the tip of the cantilever, causing the cantilever to displace steeply. On the other hand, in dynamic mode or thermal vibration, force is applied to the entire cantilever, causing the cantilever to be displaced smoothly. In other words, in dynamic mode, only the displacement due to the first or second resonance can be detected due to the influence of the Q value, whereas in DC displacement there is no influence of the Q value, so the displacement of all resonance modes is taken into account. The conversion coefficient χ of InvOLS in static mode and dynamic mode is expressed by the following equation.

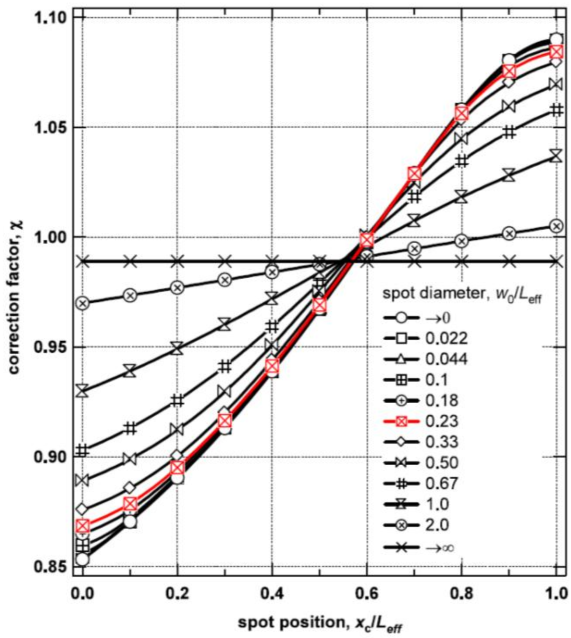
$$\chi = \frac{\mathrm{InvOLS_{free}}\,[\mathrm{m/V}]}{\mathrm{InvOLS_{end}}\,[\mathrm{m/V}]} = \frac{\dfrac{\mathrm{d}}{\mathrm{d}x}\left(z_{\mathrm{loaded}}\right)}{\dfrac{\mathrm{d}}{\mathrm{d}x}\left(z_{\mathrm{free}}\right)}$$

$$\approx 1.090 \;\; \text{(for rectangular cantilever)}$$



Rev. Sci. Instrum. 92, 045001 (2021).

However, the correction coefficient varies depending on the laser position on the cantilever and the size of the laser spot. If the spot is infinitely small and it hits the tip of the cantilever, it will be 1.09. However, in high-speed AFM system, the spot size is about the same as the cantilever size, and in order to maximize the Sum value, it will be placed near the center of the cantilever. Therefore, the correction coefficient is considered to be 1 or less than 1, and there is no need to consider correction as will be described later. Commercially available AFM devices such as Bruker use large cantilevers, so the laser spot is small relative to the cantilever size, so this correction effect is taken into account.



Nanotechnology 15 (2004) 1344–1350

- Rev. Sci. Instrum. **85**, 113702 (2014)
- J. Res. Natl. Inst. Stand. Technol. **116**, 703-727 (2011)
- Rev. Sci. Instrum. **78**, 093705 2007
- Front. Phys. 8:301(2020)

Spring constant correction formula (oscillation mode)

Since the vibration modes are different in static mode and dynamic mode, the spring constants are also different. In the dynamic mode, the displacement corresponds to the first-order resonance mode, whereas in the static mode, the higher-order resonance mode can also be displaced at the same time, so the entire cantilever can be displaced. Therefore, the spring constant is estimated to be slightly smaller in the static mode.

$$\beta = \frac{k_c}{k_1}$$
$$\approx 0.971 \ \text{(for rectangular cantilever)}$$

- J. Res. Natl. Inst. Stand. Technol. **116**, 703-727 (2011)
- Rev. Sci. Instrum. **85**, 113702 (2014)
- Rev. Sci. Instrum. **80**, 035110 2009
- Nanotechnology **6** (1), 1-7 (1995)

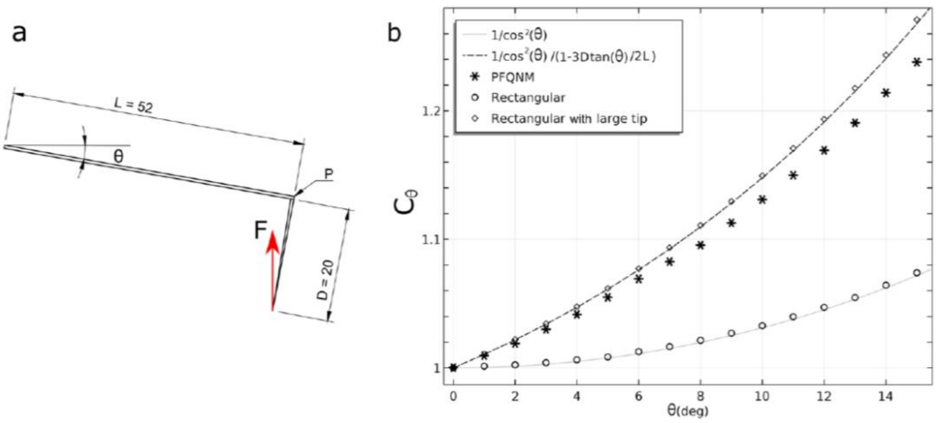
Spring constant correction formula (tilt angle)

Furthermore, in the OBD method, the cantilever is installed at an angle of about 12 degrees, so the tip-sample force is applied not perpendicularly to the cantilever surface, but slightly obliquely. Therefore, if the user wants to use the spring constant calculated using a mechanics of materials formula from the dimensions of the cantilever measured with an electron microscope for actual AFM measurements, it should be increased by about 5%. Further, correction is also required when a cantilever whose spring constant has been calibrated in one device is to be used in another device with a different tilt angle. However, in most measurements, the same device is used and the spring constant determined from Brownian fitting is used, so there is no need to consider this conversion.

$$k_\theta = C_\theta k_c$$

$$C_\theta = \left[\cos^2\theta\left(1 - \frac{3D}{2L}\tan\theta\right)\right]^{-1}$$
$$\approx 1.05 \ \text{(for rectangular cantilever)}$$

where D is the tip height and L is the cantilever's length.



Rev. Sci. Instrum. 92, 045001 (2021).

Correction in high-speed AFM force curve measurements

- In normal force curve measurement, InvOLS is first determined from the slope of the force curve.
- Based on $\text{InvOLS}_{\text{end}}$ using this static mode, perform Brownian fitting and calibrate the spring constant.
- Then, a force curve measurement is performed using the spring constant calibrated according to this dynamic mode.

Therefore, in force curve measurement, the obtained InvOLS can be used as is, but the obtained spring constant needs to take into account the correction coefficient. The correction coefficient of the spring constant, which also takes into consideration the correction coefficient of sensitivity, is expressed by the following equation.

$$
\begin{aligned}
k_c &= \beta k_1 \\
&= \beta \frac{k_B T}{\text{InvOLS}_{\text{free}}^2 \langle V^2 \rangle} \\
&= \frac{\beta}{\chi^2} \frac{k_B T}{\text{InvOLS}_{\text{end}}^2 \langle V^2 \rangle} \\
&\approx 0.8175 \frac{k_B T}{\text{InvOLS}_{\text{end}}^2 \langle V^2 \rangle} \quad \text{(for rectangular cantilever)}
\end{aligned}
$$

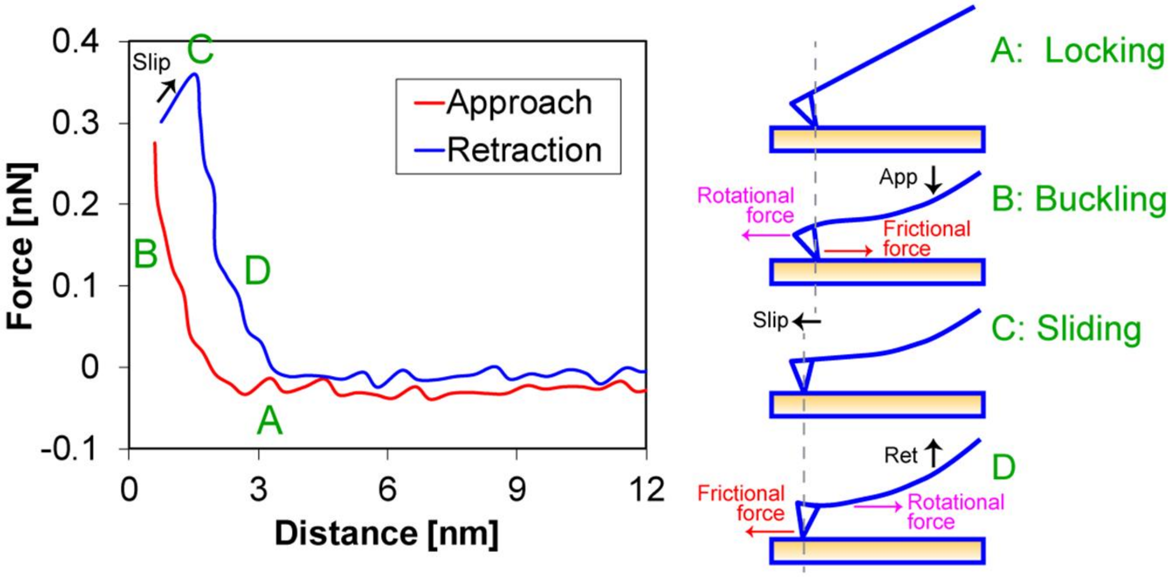
This means that if a cantilever with 0.1 N/m is used, the spring constant is estimated to be 0.122 N/m.

However, as mentioned above, in high-speed AFM, the laser spot size and cantilever size are almost the same, so χ is considered to be 1 or less than 1, and the correction coefficient for the spring constant alone is not that large. Therefore, the overall correction coefficient is considered to be almost negligible. Therefore, the UMEX software does not take this correction coefficient into account.

- Rev. Sci. Instrum. **78**, 063701 2007
- J. Res. Natl. Inst. Stand. Technol. **116**, 703-727 (2011)

Force curve hysteresis principle

When force curve measurements are performed on hard surfaces such as mica, hysteresis may occur where the retraction curve is shifted upwards compared to the approach curve, as shown in the figure below. This is because the cantilever is installed at an angle of about 12 degrees, so the in-plane force acting on the probe balances the frictional force acting between the tip of the probe and the surface. This results in the rotation of the probe. Since the OBD method detects the displacement angle, not the displacement itself, of the probe, the rotation of the probe is artificially detected as displacement.



B. The cantilever is displaced by contact with the sample, and as it approaches the horizontal direction, the tip is pushed to the left direction. However, during the approach, the tip of the probe comes into contact with the sample surface and cannot freely move due to frictional force, so the tip of the cantilever bends downward in a bowing shape (in other words, The displacement of the cantilever becomes S-shaped). Therefore, the estimated displacement will be smaller than the actual displacement.

C. After the tip is indent into the sample surface, the approach is stopped, then the frictional force disappears, so the tip slips and becomes in a state where no force is applied.

D. During retraction, the probe is pushed to the right direction, but it cannot move due to frictional force, so the probe rotates in the opposite direction to the approach. Therefore, the retract curve becomes similar to the approach curve but is shifted in the far direction.

According to the literature, piezo hysteresis and creep also affect hysteresis, but the effect of frictional force is greater.
If optical interferometry is used instead of the optical lever method, this hysteresis does not appear.

- **Langmuir 9**, 3310 (1993).
- **Rev. Sci. Instrum. 76**, 053706 (2005).
- **J. Phys. Conf. Ser. 61**, 805 (2007).
- **J. Appl. Phys. 107**, 044305 (2010).
- **J. Appl. Phys. 135**, 035104 (2024).
- https://www.jvss.jp/chapter/kanto/files/2024_kenbi/Seminar2024_04.fld/Oxford.pdf