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Application Of Atomic Force Microscopy For The Study Of Friction Properties Of Surfaces

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ABSTRACT

In this paper, a brief description of lateral force microscopy and the most important aspects, which must be considered in LFM experiments and analysis, are presented. Material-induced and topography-induced friction and the way of recognizing them, the procedure for measuring of lateral force and the different methods for calibrating cantilever force constant are some aspects, which have been given.

KEYWORDS

Friction force;
Lateral force;
Atomic force microscopy;
Cantilever forceconstant;
Calibration.

INTRODUCTION

The Atomic Force Microscope (AFM) is a scanning probe microscope where an image is obtained based on the interaction between a sample and tip. In AFM a sharp tip is mounted at the end of a spring cantilever of known spring constant.

AFM head employs an optical detection system in which a laser beam is focused onto the backside of a reflective cantilever and is reflected from the cantilever onto a position sensitive photo detector (figure.1).

As the tip scans the surface of the sample, variation in the height of the surface is easily measured as flexing of the cantilever, then variation in the photodiode signal. This gives a 3-D profile map of surface topography[1].

There are feedback mechanisms that enable the piezo-electric scanners to maintain the tip at a constant force (to obtain height information), or height (to obtain force information) above the sample surface .

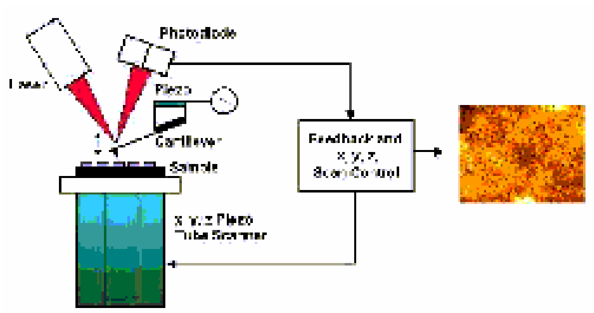

Figure 1: A schematic of AFM

AFM can be used not only for imaging the surfaces in atomic resolution but also for measuring the forces at nano-newton scale. The force between the tip and the sample surface is very small, usually less than 10^{-9} N. The detection system does not measure force directly. It senses the deflection of the micro cantilever.

The atomic force microscope is being used to solve processing problems in a wide range of technologies affecting electronic, telecommunication, bio logical, chemical, automotive, aerospace, and energy industries .

The AFM is applied to study of phenomena such as abrasion, adhesion, cleaning, corrosion, etching, friction, lubrication, plating, and polishing[2].

Since the invention of the Atomic Force Microscope (AFM) a great deal of attention has been focused on using AFM techniques to measure nanometer-scale frictional properties.

The AFM can provide information on the atomic-level frictional properties of surface, but reproducible quantitative measurements are difficult to obtain [1,2].

Studying of friction properties are specially important in some application fields such as Nano Electro Mechanical Systems (NEMS), lubricants and the mechanical properties of polymers and coating.

Lateral Force Microscope

Lateral force microscopy (LFM) is a scanning probe microscopy (SPM) technique that identifies and maps relative differences in surface frictional characteristics. Applied with contact mode atomic force microscopy (AFM), LFM is particularly useful for differentiating components of heterogeneous surfaces and identifying surface compositional differences where materials with relatively flat topography have differing frictional characteristics. It should be noted, however, that these differences can be obscured by rough topography or by contamination on the sample surface[3].

Lateral force-records measurements of friction; the degree of tortion of the cantilever is proportional to the surface friction caused by the lateral force exerted on the probe;measured by the left and right deflection of the laser.

During scanning in contact mode the cantilever bends not only along normally to the surface but also the cantilever torsional (lateral) deformation occurs. LFM measures torsional deformation of the cantilever during scanning in contact mode (Figure.2).

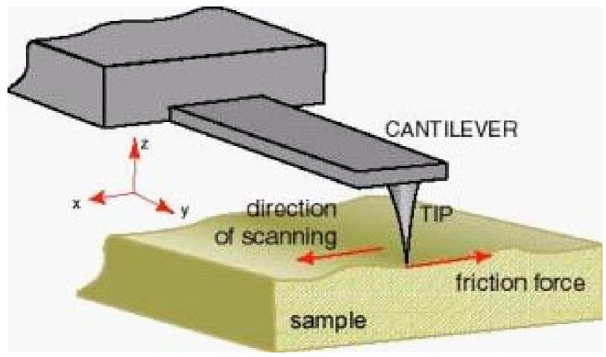

Figure 2: Deformation of cantilever in LFM

The LFM image and topography can be obtained simultaneously. The lateral deformation depends on a frictional(lateral) force acting on tip.

The cantilever deflections are registered by optical system of microscope[4-5].

The photodetector measures the difference in light intensities between the upper and lower photodetectors, and then converts voltage. Feedback from the photodiode difference signal, through software control from the computer, enables the tip to maintain either a constant force or constant height above the sample. In the constant force mode the piezoelectric transducer monitors real time height deviation. In the constant height mode the deflection force on the sample is recorded[2].

The measurements of cantilever torsion are carried out with constant force condition, i.e. with constant vertical deflection of a cantilever. Therefore, Full Paper

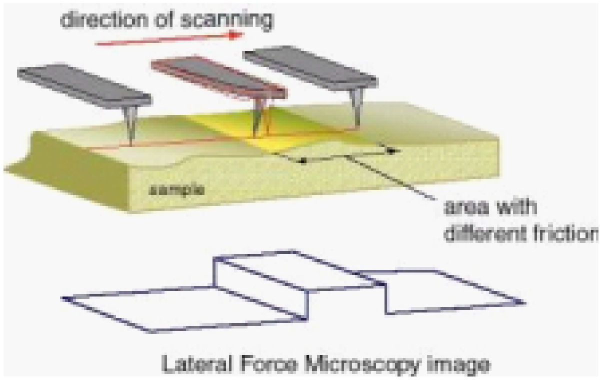

Figure 3: LFM image of surface with different friction

it is possible to distinguish the areas with different friction (Figure.3) in other words the LFM is sensitive to chemical composition or structure of the surface [4-7].

If the sample surface is rough then such interpretation of the LFM image is difficult because lateral deflection is caused also by topography. The direction of scanning in LFM mode should be perpendicular to cantilever axis. This direction is x-axis for some microscopes[5].

AFM and LFM detect the position of the cantilever by means of a four quadrant spot detector. A laser beam is reflected from the top of the cantilever. As the cantilever bends up or down, the reflected beam will be deflected up or down. Likewise, frictional drag forces will cause the cantilever to twist. This in turn will deflect the laser beam left or right, depending on scan direction. A schematic of such a detector is shown in figure 4.

Spot detector consists of a single p-i-n diode solid state detector that is divided into four quadrants .the vertical position of the laser spot is detected by adding the signals from UL+UR, and DL+DR, and these two sums are then subtracted. This difference is proportional to the vertical deflection of the laser beam. The lateral force is measured by adding the signals from the two left detectors and two right detectors, and taking the differences, i.e., (UL +DL)-(UR+DR).

If the detector is rotated as shown on the right side of figure 4, then lateral motion of the laser spot will cause the vertical signal to change. The rotation of the detector and the lateral motion of the laser spot have been exaggerated for clarity .In this way, frictional forces are sensed by the AFM as changes in height. In other words, lateral and vertical force signals are coupled[8].

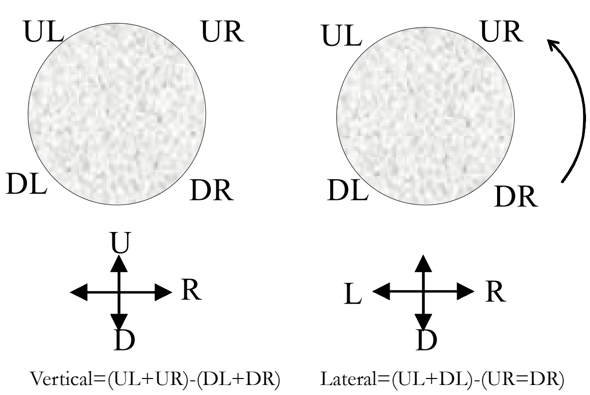

Figure 4: A schematic of quadrapole detector

AFM Resolution

The concept of resolution in AFM is different from

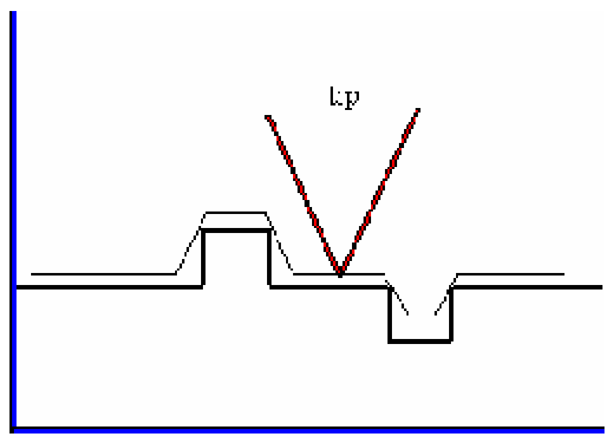

Figure 5 : Lateral dimensions(μm)

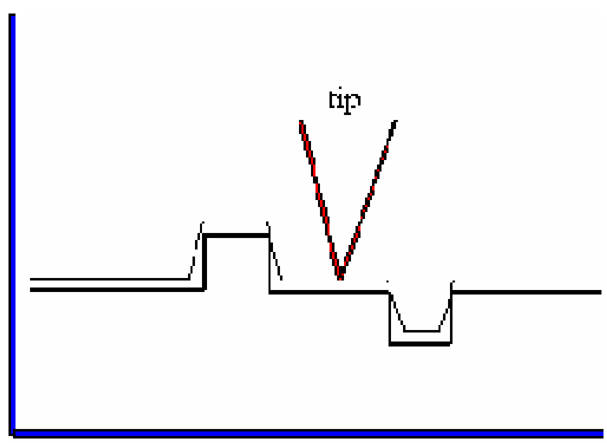

Figure 6: Lateral dimensions(μm)

radiation based microscopies because AFM imaging is a three dimensional imaging technique. The ability to distinguish two separate points on an image is the standard by which lateral resolution is usually defined. There is clearly an important distinction between images resolved by wave optics and scanning probe techniques. The former is limited by diffraction, and later primarily by apical probe geometry and sample geometry.

Indeed, many authors have seen that it is the radius of curvature that significantly influences the resolving ability of the AFM. Images of samples made by the sharper tip have shown dramatic improvements in resolution widths (Figure 5 and 6). Even greater improvements in resolution have been attained with Tapping mode but contact imaging still is capable of high resolution imaging[2].

Noise in the AFM probe

The noise in an AFM probe limits the resolution of the obtained AFM image. The noise sources primarily consist of thermal vibrational noise, Johnson noise and 1/f noise. An expression for the total noise neglecting the 1/f noise contribution can be expressed as:

$$< Z^2_{total} >= \frac{1}{3}\frac{k_B T l \Delta f}{(1-\frac{1}{2}\frac{\lambda}{l})^2}\left(\frac{1}{k\lambda f_{ref}}+\frac{256Rl^3}{3U^2k^2t^2}\right)$$

Where T is the temperature, k the spring constant, f_{res} the resonant frequency, R the resistance, U the supply voltage, Δf the bandwidth and k_B the Boltzman constant.The first part of the parentheses corresponds to the thermal vibrational noise and the second part corresponds to the Johnson noise. For the cantilever dimension given above and T = 300K, R=6kΩ, U=2 V, K=50 and Δf=10 Hz the total equivalent deflection noise can be calculated to z_{total} =0.3 Å. This result implies that atomic resolution should be obtainable with the probe. The electrical noise in the Wheatstone bridge has been measured and the noise floor is in good agreement with the calculated noise limit[9].

Sensitivity of the photo detector

For determination of sensitivity of photo detector, we must to know about force curve measurements. Atomic Force Microscope can record the amount of force felt by the cantilever as the probe tip is brought close to – and even indented into a sample surface and then pulled away. This technique can be used to measure the long range attractive or repulsive forces between the probe tip and the sample surface(Figure 7).

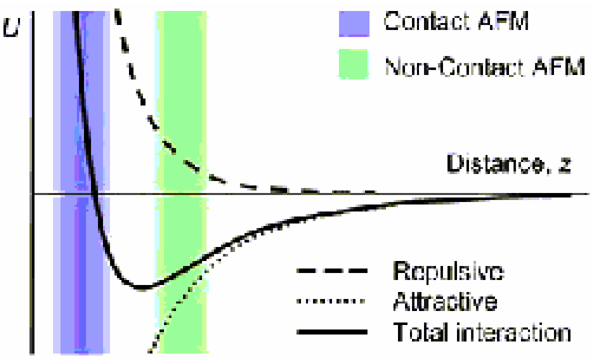

Figure 7: Force curve in AFM

As the atoms of the tip and sample are brought together they initially weakly attract each other. This attractive force increases until the interacting atoms are so close that their electron clouds begin to repel one another electrostatically. This electrostatic repulsion continues to weaken the attractive force as the separation distance decreases. The attractive force goes to zero (i.e., approaches the limit of zero) when the distance comes within the length of a chemical bond (a few Angstroms). Once the total Van der Waals forces (repulsive forces) are positive, the atoms are in contact[2,10].

In the AFM the sensitivity of the photo detector S_z [nm/V] has to be determined by measuring force vs. distance curves on reasonably hard surface (e.g. Al_2O_3) where elastic deformations can be neglected and the movement of the z-piezo Z_s equals the deflection of the cantilever Z_t. It is found that the laser focus should be well defined in order to position the laser beam above the probing tip and to achieve accurate calibrations[11].

Friction force calculation

In friction force measurements, at first we obtain the rough lateral signal (nA or V) by lateral force microscopy. Then the true friction signal should be obtained by eliminating the topography induced friction signal. The latter friction signal(nA/V) is converted to friction signal in nanometer by using cantilever sensitivity. Finally, friction signal (nm) will convert to friction force signal in nN according to Hooke's law and by using cantilever spring constant.

Eliminating the topography–induced friction signal from obtained friction signal

During friction measurements, the friction signal (nA or V) from both the forward and backward scans are needed in order to understand the origins of the observed friction forces. It is well Known that when an AFM tip is scanned across a sample surface the measured friction force (or lateral force) are generated by both material effects as well as topography-induced effects.

However, friction studies in the past, while concentrating on material- induced effects, often present users with conflicting and confusing interpretations of topography-induced friction forces.

It has been reported that topography-induced effects are independent of scanning direction and are hence eliminated when subtracting the friction data of the back-ward scan from that of the forward scan, leaving only material-induced effects. Other studies have attributed correlation between surface topography and friction forces in scanning probe microscopy to variation of van der Waals forces between high and low points on a surface, to influence of local slope of the sample (ratchet mechanism), and to torsion of the cantilever generated by reaction forces and friction forces at locations involving significant surface height change. These effects do explain the variations of friction as a function of topography but appear to be independent of the scan direction and agree with the previous suggestion that the subtraction process will eliminate the topography-induced effects.

The sign of the friction signal is reversed for the Retrace (backward or right to left) scan compared to that of the Trace (forward or left to right) scan.

This is of course due to the reversal of the torque applied to the end of the tip when the scanning direction is reversed. As a consequence when raw friction data are presented, peaks in two-dimensional (2D) friction profiles correspond to high friction for Trace data and low friction for Retrace. This also means that for gray-scale images, lighter regions in the Trace friction image correspond to higher value of friction force while in the Retrace image, lighter regions correspond to lower friction force.

However the friction data show large variations at the edges of the pit where the topography changes sharply .In addition; the friction data obtained with the dimension show a large tilt. This is due to cross talk between the vertical deflection signal and the horizontal deflection signal that arises from misalignment between the trajectory of the reflected of the reflected laser beam on the photo detector and the photo detector axis. Looking at the subtracted friction data (T-R), two points are clear: First, the subtraction process does not remove the topography – induced effects associated with the pit edges. Second, the effect of detector cross- talk is effectively removed by the subtraction process.

On the other hand, the change in friction force due to the material effect in Trace and Retrace will be in opposite directions (upwards or downwards). However, the changes in friction due to topography in Trace and Retrace will be toward the same direction. This is one difference between material induced effects and topography- induced effects on the friction forces. The magnitudes of the friction change due to material effects will be the same in Trace and Retrace but the magnitudes of the topography- Induced friction forces at a given location will be different[12]. It is obvious that the friction signal obtained by subtraction of R from T, is two times of the real friction signal and so must be divided by two(T-R/2).

Cantilever spring constant calibration

For quantitative force measurement, the spring constant of the cantilever must be calibrated, so that the nanometers deflection of the cantilever can be converted into actual force values. There are various different ways of calibrating constants of cantilevers, depending on the equipment that is available, and only the three most common methods are described here. Each method is subject to some limitations, and if the force measurement is particularly important , the spring constant should be calibrated using more than one method to verify the results[13].

Calculation from cantilever geometry

When cantilevers are purchased, they are usually supplied with a data sheet, which includes the spring constant. The values are generally given as a range, and may have been calculated from the average cantilever geometry, rather than having been experimentally measured for each cantilever. It is possible to calculate the spring constant, if the exact shape of the cantilever, and the young's modulus of the material is known.

A problem with this method is that the spring constant depends on the thickness of the cantilever cubed. This value is also the smallest dimension of the cantilever. Therefore the spring constant is extremely sensitive to slight differences in cantilever thickness between batches of cantilever. The quoted spring constant values from manufacturers are useful for choosing a cantilever of a particular application, but are usually not reliable for quantitative force measurements[13].

Measurement using a reference cantilever

When a reference cantilever with a known spring constant is available, other cantilever spring constants can be calculated by comparing the deflection of the two cantilevers when they are pushed together[13].

Measurement using the thermal noise method

Fluctuations in the environment (in air or fluid) constantly provide small force impulses, as can be seen for example in the diffusion of small particles (Brownian motion). Soft cantilevers are susceptible to thermal fluctuations, and the AFM can be used to measure and analyze the movements. The thermal noise spectrum is a plot of the cantilever fluctuations as a function of frequency; on average the greatest amplitude will be seen around the cantilever resonance frequency.

The amplitude of the fluctuations for a given temperature depends only on the spring constant of the cantilever[13].

Lateral spring constant calculation

When we use of cantilevers, each cantilever has to be characterized. One way is to use an electron microscope and to determine all the relevant parameters, such as: Tip radius R; height of tip h ; width, thickness and length of cantilever (ω,t,l) and position of tip on the cantilever. In addition, elastic constants are needed: Youngs modulus E, shear modulus G. Having determined all these parameters, the normal spring constant c_B and the torsion spring constant c_t for a rectangular cantilever are given by:

$$c_B = \frac{E.\omega.t^3}{4.l^3}$$

$$c_t = \frac{G.\omega.t^3}{3.h^2.l}$$

Where E is the Youngs modulus and G=E/(2(1+ν)) for commercially available silicon cantilevers, the elastic properties are well-defined and the first resonance frequency in normal direction $f_1 = \omega_1/2p$ can be used to determine the thickness of the cantilever more accurately:

$$t = \frac{2.\sqrt{12}\pi}{1.875104^2}\sqrt{\frac{\rho}{E}}f_1.l^2 = 7.23 * 10^{-4} s/m.f_1.l^2$$

Where ρ is the density of the cantilever (for silicon cantilevers : ρ=2.33 . 10^3 kg/m^3 , E=1.69 * 10^{11} N/m^2).

Thus, the procedure is more simplified: The lateral dimensions of width and length (**w** ,l) can be determined with an optical microscope. Usually, these dimensions are quite reproducible by current microfabrication procedures. The height of the tip h can very a few microns and should be checked with an optical or electron microscope[10].

Generally for a rectangular cantilever made of <110> Si and having length l, thickness t and tip height h the ratio of the torsional and normal spring constants is given by:

$$C_t / C_B = 0.394^* l^2 / (h + t/2)^2$$

Maximizing the lateral sensitivity in this case could mean minimizing the ratio C_t/C_B, or l/(h+t/2). In terms of the ratio of the torsional and deflection angles when the tip is subject to equal normal and lateral forces:

$$\theta_t / \theta_B = 1.69^* (h + t/2) l$$

so the highest angular ratio, i.e. the highest tortional sensitivity, again corresponds to the min of

$$l/(h + t/2)$$

For a given normal spring constant, this generally leads to the choice of cantilevers that are shorter

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and thinner than conventional contact mode cantilevers.

Triangular cantilevers are more difficult to be accurately calibrated, both the thickness of the cantilever, the length of the tip and the position of the tip on the cantilever can lead to large deviations of the spring constants from manufacturer data.

Thus it becomes necessary to characterize each cantilever with electron microscopy. Using this procedure and the formulae given by Neumeister et al. a reasonable accuracy of 10% could be achieved[14].

Conversion of the cantilever deflection from nA/v to nm

The deflection of the cantilever spring is directly proportional to the tip-sample interaction force.

The normal force between tip and sample was estimated from cantilever deflection (nA) curve plotted against Z-displacement of the cantilever and converting this curve to force–Distance curve. The conversion factor for converting nA to nm was obtained from the slop of the linear portion of the deflection–distance curve. There was also one conversion needed for the x-axis values. The change in piezo height, which has been used for the distance between the tip and the sample, was corrected for the deflection of the cantilever by subtracting the cantilever deflection from the piezo height.

on the other hand, there are two measurements required to convert the photo detector signal into a quantitative value of force.

The first stage is to calibrate the distance that the cantilever actually deflects for a certain measured change in photo detector voltage. This value depends on type of cantilever but also on the optical path of the AFM detection laser, and will be slightly different each time the cantilever is mounted in the instrument. Once the deflection of the cantilever is know as a distance, X, the spring constant k, is needed to convert this value into a force F, using the well-known Hooke's law:

$F=kx$

A force curve between a plain cantilever tip and a bare hard substrate is used to determine the sensitivity of the experimental setup. This is a measurement of the deflection of the tip in nanometers for a given movement of the detection laser on the photo detector.

The repulsive contact region, where the deflection rises steeply upwards is linear for a hard surface and tip (figure 6). Therefore it can easily be used for determining the factor for converting nA/volts into nanometers[15-18].

In figure 8, force curve draw by using contact cantilever(CSG01) with spring constant about 0.03N/m on mica surface. It means that when cantilever deviate 100 nm, the applied force to the probe is equal to 3nN. So by using DFL (Z) curve we can see dependence between nA and nm . In the figure 8 we see 100 nm deviation is equal to 1.5 nA. However if we consider F=0 when probe is far away from the surface (when DFL=-2.6) ,we must eliminate the offset and we obtain real DFL.In this case, DFL is zero because –2.6-(-2.6)=0

As it was mentioned before, the friction signal (nm) is converted to force value (nN) according to Hooke's formula by using the lateral spring constant.

In this case ,we obtain the force value by this formula:

F=[DFL–(-2.6nA)*100nm/1.5nA]*0.03] nN/nm. For example when DFL=4 then F=13.2nN.

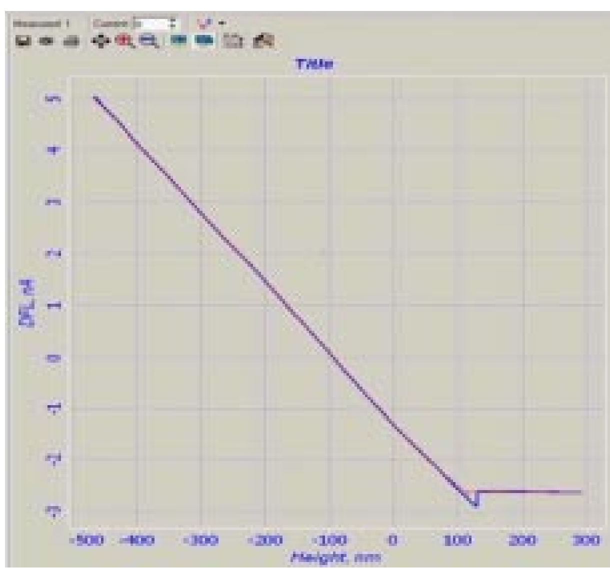

Figure 8: force curve obtain by CSG01 cantilever in contact mode

SUMMARY

This paper focuses on using AFM/FFM for study of friction properties of the surfaces.

In order to perform quantitative frictional force microscopy with the scanning probe microscope, it is important to perform these steps:

- Measuring the lateral signal by lateral force microscopy,
-Elimination of topography induced friction signal and obtain the true friction signal,
-Converting friction signal (nA/V) to nm by using cantilever sensitivity,
- Converting friction signal (nm) to friction force (nN) according to Hooke's law and by using cantilever spring constant.

Passing these steps is the starting point for any more complex analysis.

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