Eliminating Lateral Forces During AFM Indentation

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Abstract. We have implemented a technique to eliminate the in-plane lateral tip motion for AFM experiments that involve a cantilever deflection change due to bending. Because of the geometry limitation, there is an undesired lateral tip position change associated with the bending of the cantilever. Previous approaches for solving this problem used X-piezo motion to compensate this tip lateral motion based on the distance change between the tip and sample, or the Z-piezo travel. This method partially works based on the assumption that the cantilever deflection change is always proportional to the Z-travel distance, which is not true in many cases. The key factor in our approach is to apply X-piezo motion based on the cantilever deflection change. This is a direct and accurate method that will also take into account of the probe sample interactions. We will demonstrate the application results of this method with nanoindentation on polymer samples, as well as imaging on heterogeneous materials. The various kinds of calibration methods will be also discussed.

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1. Introduction

Atomic Force Microscopy has been used extensively to perform mechanical property measurements of a variety of materials. In the conventional measurements, an AFM probe tip or sample is moved with a vertical (Z) actuator material to indent the tip into the surface of the material. During the indentation process, the deflection of the cantilever is measured as a function of the Z-actuator position. Knowing the cantilever spring constant, this measurement is converted into a force versus distance curve. Various models have been used then to extract mechanical properties from the force versus distance curve.

A key issue with AFM-based indenting, however, is the parasitic lateral motion of the tip during this indentation process, as shown in figure 1 at right. Consider, for example, a cantilever that is bent by an angle \( \alpha \) and a resulting vertical tip displacement of \( \Delta h \). If the tip is free to move laterally, the bending will result in a translation of the base of the tip (shown below as \( \Delta x_1 \)) and a tip rotation through angle \( \alpha \) that results in addition tip motion (\( \Delta x_2 \)).

Figure 1. AFM tip motion during
From geometric analysis, we obtain for small bend angle $\alpha$:

$$\Delta x = \Delta h \tan \theta + h_i \alpha \cos \theta;$$  \hspace{1cm} (1)

where $\theta$ is the cantilever mounting angle and $h_i$ is the tip height. Then from Euler-Bernoulli cantilever bending theory, the cantilever end angle is given by:

$$\alpha = \frac{3}{2} \frac{\Delta h}{L \cos \theta};$$  \hspace{1cm} (2)

where $L$ is the cantilever length. Substituting (2) into (1) gives:

$$\Delta x = \Delta h \left( \tan \theta + \frac{3}{2} \frac{h_i}{L} \right);$$  \hspace{1cm} (3)

Where $\theta$ is the cantilever mounting angle, $h_i$ is the tip height and $L$ is the cantilever length. The first term corresponds to the tip translation $\Delta x_1$, and the second term corresponds to the tip rotation resulting in $\Delta x_2$. For example, a common AFM cantilever with $\theta = 12^\circ$, $h_i = 12 \mu m$, and $L = 225 \mu m$ results in the relationship $\Delta x \approx 0.29 \Delta h$.

Thus a standard AFM indenting experiment may result in almost one-third of the vertical actuator motion translated into unwanted lateral motion. There have been previous efforts to compensate for this motion by applying a fraction of the Z-actuator voltage to the X-actuator voltage. This compensation scheme was originally conceived by Hoh[1] and has been available for many years in Veeco’s NanoScope®AFM force curve software (under the name “X-rotate”) and recently described in the literature[2]. This compensation scheme works very well in the case that there is a consistent relationship between the Z-actuator motion $\Delta z$ and the cantilever deflection $\Delta h$, i.e. $\Delta z = b \Delta h$, where $b$ is a constant. While this relationship holds for uniform samples, it does not apply when for heterogeneous samples with varying mechanical properties. For example, consider the extreme cases of a soft sample where the cantilever deflection $\Delta h$ is zero versus the case of a hard sample where $\Delta z = \Delta h$. It is obviously not possible to select a single constant $b$ that compensates for the lateral tip motion for a sample of mixed hard and soft phases. As a result it is desirable to implement a compensation scheme that is independent of the sample properties.

2. Deflection Lateral Correction (DLC)

To provide a robust method to perform mechanical measurements on heterogeneous samples, we have implemented a compensation scheme (also suggested by Hoh[1]) based on the actual bend of the cantilever, not the motion of the Z-actuator. Combining equations (2) and (3) give the following:

$$\Delta x = \alpha \left( \frac{2}{3} L \sin \theta + h_i \cos \theta \right) = \beta \alpha$$  \hspace{1cm} (4)

This equation shows exactly how much motion $\Delta x$ is required to compensate for any given measured cantilever bend $\alpha$. For the cantilever described above, the compensation factor $\beta$ is approximately 43 nm/mrad.

3. Deflection Lateral Correction (DLC) Implementation

The implementation of DLC is very straightforward, as shown in figure 2. The cantilever deflection $\alpha_m$ is measured by the AFM head. The deflection signal is multiplied by a gain and then added to the X-piezo drive signal. The DLC gain $\beta$ is given approximately by the expression in equation (4) above.
In practice, we determine the appropriate gain experimentally. An AFM tip is engaged in contact mode on a sample with a clearly visible feature. Then the setpoint is changed to change the deflection of the cantilever. With no compensation enabled, the features in the image shift laterally as the setpoint is changed, as shown in figure 3a. When the DLC gain is appropriately adjusted no lateral shift is visible as shown in figure 3b when the setpoint is changed. The setpoint change shown in Figure 3 is chosen to make the effect clear. Normally we use setpoint changes of less than 0.5 V to minimize the tip sample force during this calibration step. It is also possible to set the DLC gain by performing test indents and adjusting the parameter to make the gain as symmetric as possible, as shown in Figure 4 below.

Figure 4 also shows the difference in slope in a measured force curve with DLC on and off. Notice that without DLC enabled, the slope of the force curve is underestimated from the correct value. Figure 5 shows the resulting contact stiffness measured with and without DLC enabled. With DLC enabled the resulting contact stiffness is higher because parasitic bending of the cantilever due to lateral forces is suppressed. In our current implementation there is a somewhat higher variability of the contact stiffness with DLC enabled, however.
4. Impact on deflection measurements

To understand the potential need for DLC in a given experiment, we will next explore the impact of unwanted lateral motion on cantilever deflection measurements. If an AFM tip penetrates into a material in an indentation experiment, as shown in figure 6, the tip is constrained such that it cannot move the full amount \( \Delta x \) from equation (3). Instead, the sample’s lateral stiffness will exert a lateral force \( F_L \) on the tip that will cause a parasitic bend \( \Delta \alpha \) in the cantilever end angle. The force on the tip depends on the relationship of the cantilever’s transverse stiffness \( k_L \) versus the sample’s lateral contact stiffness \( S_L \). Specifically,

\[
F_L = \left( \frac{S_L k_L}{S_L + k_L} \right) \Delta x \quad (5)
\]

Applied to the tip, this force \( F_L \) generates a torque that bends the cantilever as shown in Figure 6. If uncompensated, the motion \( \Delta \alpha \) will give erroneous results for the vertical force and vertical position of the cantilever. We can estimate the amount of the error.

In the presence of a lateral force, the cantilever bend angle is given by:

\[
\alpha = \frac{1}{k_v L} \left( \frac{3}{2} \frac{F_N}{k_v} - \frac{3h_v}{L} \frac{F_T}{k_v} \right) \quad (6)
\]

where \( F_N \) and \( F_T \) are the normal and transverse forces on the cantilever tip. The deflection error \( \delta \alpha \) is due to an uncompensated lateral force \( F_L \) and is then given by:

\[
\delta \alpha = -\frac{F_L}{k_v L} \left( \frac{3h_v}{L} \cos \theta + \frac{3}{2} \sin \theta \right) \quad (7)
\]

The first term is due to the torque created by the lateral force about the tip and the second term is due to the component of the lateral force in the direction normal to the cantilever. The deflection of the cantilever due only to vertical forces is:

\[
\alpha = \frac{F_T}{k_v L} \left( \frac{3h_v}{L} \sin \theta + \frac{3}{2} \cos \theta \right) \quad (8)
\]

The relative deflection error is given by:
Neglecting the first term in the denominator and combining equations (3), (7) and (9), we get:

$$\frac{\delta \alpha}{\alpha} = \frac{F_L}{F_y} \left( \frac{3h}{L} \cos \theta + \frac{3}{2} \sin \theta \right) \left( -\frac{3h}{L} \sin \theta + \frac{3}{2} \cos \theta \right)$$

(9)

By analysis of cantilever stiffness tensor [3] and appropriate coordinate rotations, we get:

$$\frac{\delta \alpha}{\alpha} = \frac{k_L}{k_y} \left( \frac{S_L}{S_L + k_L} \right) \left( \sin \theta + \frac{3h}{2L} \cos \theta \right) \left( \sin \theta + \frac{2h}{L} \cos \theta \right)$$

(10)

For the 225 µm cantilever described above, $k_L \sim 12 k_y$, Figure 7 shows representative magnitudes of the deflection error expected for a range of cantilever spring constants and sample elastic moduli. (Note this error is always negative, i.e. an apparent reduction in the vertical deflection of the cantilever and a reduction in the slope of the force curve.) The lateral contact stiffness $S_L$ was estimated by $8Ga$, where $G$ is the shear modulus and $a$ is the contact radius[4] and $G = E/(2(1+\nu))$, where the Poisson’s ratio $\nu$ was chosen to be 0.2.

**Conclusions**

We have implemented a simple method to counteract lateral forces otherwise present in AFM-based indentations experiments. If uncompensated, these lateral forces can create apparent deflections of greater than 100% of the deflection caused by the vertical indenting force. Eliminating these lateral forces by deflection lateral correction allows increases the accuracy of AFM measurements of material stiffness, especially for heterogeneous samples.

**References**

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