Near Contact Mode AFM: Overcoming Surface Fluid Layer In Air And Achieve Ultra-High Resolution

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Introduction

A major goal of Atomic Force Microscopy (AFM) is to achieve nanometer resolution on surface topography. Vibrating cantilever mode (VCM) is an important configuration of an AFM instrument. It was proposed in the first AFM paper[1].

VCM in ultra-high vacuum (UHV) results in true AFM atomic resolution, which reveals atomic scale surface defects such as a single missing atom in a lattice. However, the VCM operation in air has many difficulties due to the surface contamination on the sample and the AFM tip. The most popular operation modes of the VCM are the non-contact mode[2] and the Tapping mode[3]. Both of these have limited lateral resolution in air.

To optimize the lateral resolution of the VCM in air, we studied the surface contamination layer. With the new understanding of the surface condition in air, we proposed a new VCM operation mode, which is called near-contact mode[4]. In this mode, the tip-sample spacing is minimized and the tip sharpness preserved, and nanometer lateral resolution can be routinely achieved in air.

The Problems

In ambient air, the tip and sample are usually covered by a layer of surface contamination, which has liquid-like properties. When the tip approaches the sample, there is a moment when the two contamination layers are in contact. At this moment a meniscus forms between the tip and the sample, and a strong capillary attraction is abruptly applied to the AFM tip. The magnitude of the attraction is usually in the order of 100 nano-newtons. This can cause the tip to accelerate toward the sample and result in the tip being captured by the sample surface. In an AFM force-distance curve, this phenomena can be seen as jump-to-contact and adhesion.

When the tip is captured, the cantilever vibration stops and the VCM operation will fail. The other problem associated with this process is the hard physical contact between the tip and the sample when the tip is pulled to the sample. Various experiments have shown that such contact can result in severe tip and sample damage.

The problem of tip damage was acknowledged in the early days of AFM. In non-contact mode (the earliest VCM operation), to prevent tip-sample contact the tip is maintained at several nanometers away from the sample with a vibration amplitude of a few nanometers. Although this can prevent the tip-being-captured problem, the lateral resolution is limited by the relatively large tip sample-spacing.

Later, another method (Tapping mode) was proposed to solve the tip-being-captured problem. This mode vibrates the cantilever at a much larger amplitude, usually 100 nanometers. The tip contacts the sample periodically. Although this can overcome the capillary force that captures the tip, the large amount of energy dissipated at the tip-sample contact point can cause significant tip/sample damage, which also limits the lateral resolution.

In ambient air, both non-contact and periodic-contact have their limitations in achieving nanometer lateral resolution. A new method is needed for optimizing the AFM lateral resolution in air. Since small tip-sample spacing and small tip radius is crucial for obtaining ultra high lateral resolution for AFM, our goal is to find a method that can minimize the tip-sample spacing while maintaining the sharpness of the tip.

The Analysis

To find a better solution, we should look at the cause of the problem more closely. Figure 1 shows a microscopic model of the AFM tip and sample in ambient air[5]. Usually, the surface contamination layer can be considered as having a structure which consists of two different layers. The first is a molecular layer tightly bonded to the surface of a tip or a sample. The second is a liquid-like layer on top of the molecular layer.

Based on this model, the tip sample relationship can be classified into four different positions (Figure 1). "A" is the true non-contact position and "B" is the contamination contact position. In this case, a meniscus is formed between the tip and the sample, and liquid fills the space between the tip and sample. "C" is the near-contact position. In this case the two tightly-bonded molecular layers come into contact. They serve as a buffer between the tip and sample. This is the optimal imaging position because the tip-sample gap is minimized while the sharpness of the tip is protected by the molecular buffer layer. "D" is the physical contact position. In this case, the molecular layers are destroyed and the interaction among all the atoms in the contact region are as strong as in the bulk. Atom transfer among the tip, sample and contamination layers can occur.

Usually, the large capillary force will result in jump-to-contact, which makes the tip go from position B directly to position D. To bring the tip into the near-contact position, we must first prevent jump-to-contact. The simplest way is to use a cantilever with large spring constant. Experiments have shown that the jump-to-contact does not occur for the cantilevers with the largest spring constants.

In order to practically operate an AFM in near-contact mode, we need a method to indicate that the tip is in the near-contact position. Feedback Fluctuation Analysis (FFA) can be used for that. When an AFM is in feedback, there is a certain amount of fluctuation in the feedback signal. By monitoring the amplitude of the fluctuation versus the feedback set-point, the near-contact position can be located. Figure 2 shows a typical FFA curve. The large feedback fluctuation in the middle of the curve indicates the position B, in which the liquid between the tip and the sample causes the feedback to be unstable. The abrupt drop in the fluctuation amplitude in the tip-approach direction indicates the near-contact position.

To maintain the tip in the near-contact position and maintain its sharpness, we also need to preserve the molecular buffer layer. This requires the vibration amplitude to be small. This is to ensure that the energy dissipated in the tip-sample interaction region is not so large as to damage the molecular buffer layer.

TABLE 1: Comparison of three VCM operation modes

VCM Operation Modes		Non-contact	Periodic-contact	Near-contact
Operation	Amplitude	< 10 nm	> 20 nm	< 10 nm
	Feedback Set Point	50% amplitude reduction, or change of phase	10 % amplitude reduction	75 % change in phase signal
	Feedback Control PID Parameters	Small	Not sensitive	Large
	Spring Constant of Cantilever	Medium to High	Medium to High	High only
	Detection Method	Amplitude/Phase	Amplitude/Phase	Prefer Phase
Working Mechanism	Relation to the contamination layer	At the top of the layer	In and out of the layer	Staying in the layer, near the bottom
	Tip-sample interaction force	Attractive force, negative force gradient	Repulsive force, positive force gradient	Attractive force, positive force gradient
Performance	Tip damage	No	Yes	No
	Spatial Resolution	Low	High	Very High
	Uncertainty in Z	Large	Large	Small
	Feedback stability	Not stable	Stable	Stable

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The Solution

To optimize the lateral resolution of an AFM in air, the tip should be in the near-contact position. We call this new operation mode *Near-contact* mode. To run in near-contact mode, a stiff cantilever is needed to prevent jump-to-contact. A cantilever with a spring constant of $k = 50$ N/m is suitable for most samples. To preserve the molecular buffer layer, a small cantilever vibration amplitude should be used. We only need a vibration amplitude that is large enough to provide significant signal-to-noise ratio – an amplitude of a few nanometers is usually appropriate. Next, we need to bring the tip to the near-contact position to minimize the tip-sample spacing. This can be assured by monitoring the FFA curve. In a normal operation, a 75% reduction of the free vibration amplitude is usually a good set point for the near-contact mode. Finally, let us not forget to use a sharp tip for high lateral resolution AFM imaging.

Figure 3 shows the typical images of three different VCM operation modes on the same sample. The sample used was a polished alumina-silicate glass sample. This is a very good sample for testing lateral resolution. It has two kinds of surface features: the larger "cigar" sharp features on the order of 100 nm, and the smaller round features on the order of a nanometer. Both the images and line profiles show that the near-contact mode has the best lateral resolution. In the non-contact image, the ripples are probably due to the instability of the surface contamination. In the periodic-contact image, the loss of lateral resolution is due to the tip damage by the periodic contact.

Table 1 lists the detailed comparisons of the three VCM modes. With a sharp tip (tip radius <5 nm), the difference between the VCM operation modes can be clearly shown for images of 1 μm or smaller. Since the largest number of pixels in an AFM image is usually 1024 x 1024, for image sizes of 10 μm or larger, the pixel resolution is 10 nm. Therefore, we need smaller image sizes to compare their performance.

Summary

By analyzing the structure of the surface contamination layer, a new method of VCM operation in ambient air, near-contact mode, is developed. It uses the surface molecular layer as a buffer to minimize the tip-sample spacing and maintain the sharpness of the tip. In the near-contact mode, the feedback is stable and the lateral resolution is optimized. The operation of the near-contact mode can be summarized in a "Four-S Rule": stiff cantilever, small vibration amplitude, short tip-sample spacing, and sharp tip.

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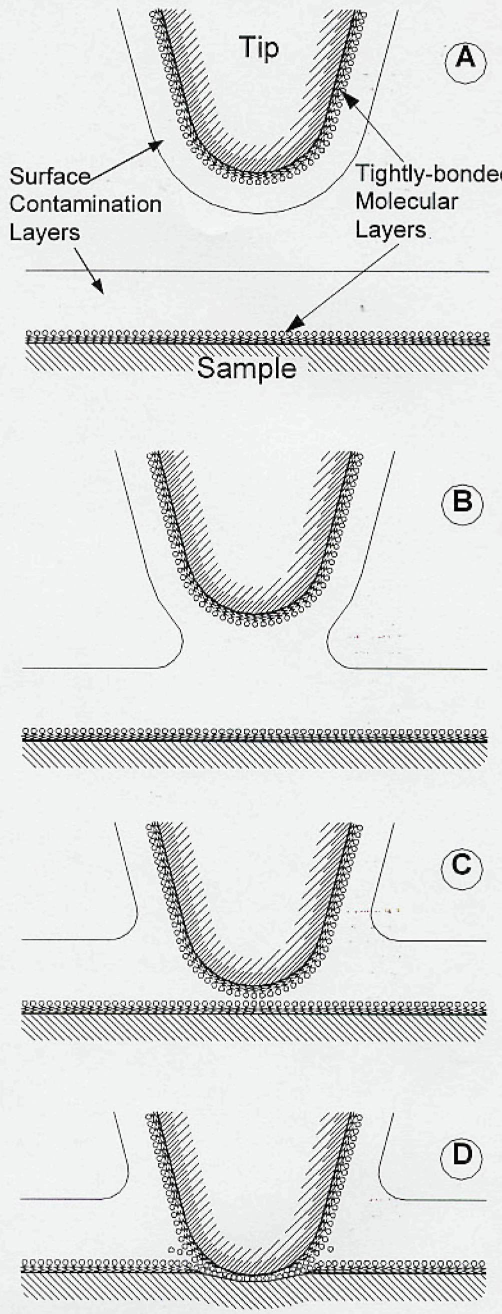

Figure 1. Molecular model of tip-approach process in air.

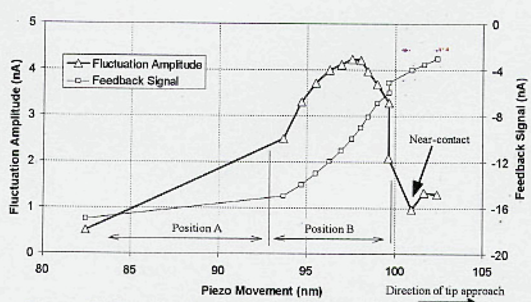

Figure 2: The feedback fluctuation analysis (FFA) shows the transition from true non-contact position to near-contact position. Three AFM tip positions (see Figure 1), A, B, and C (near-contact) can be easily identified from the curve of fluctuation amplitude.

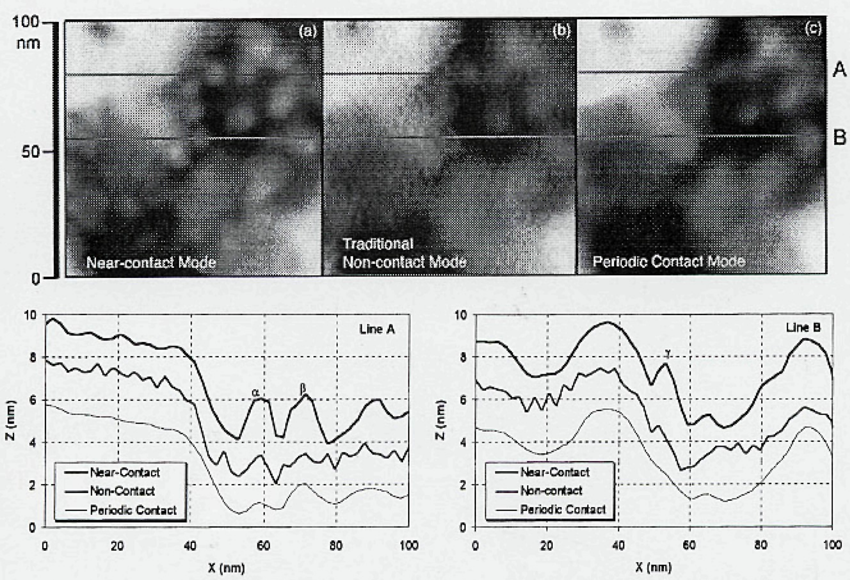

Figure 3: Topographic images and line-profiles of a polished alumina-silicate glass surface with three distinct VCM operation modes. The image size is 100 nm x 100 nm.