Frequency-modulation atomic force microscopy at high cantilever resonance frequencies using the heterodyne optical beam deflection method

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We have developed a frequency-modulation atomic force microscope (FM-AFM) with a wideband cantilever deflection sensor using the heterodyne optical beam deflection method. The method enhances the bandwidth of the deflection measurement up to the maximum frequency for the laser power modulation, which can be as high as gigahertz order. The phase and frequency of the cantilever vibration at 5.24 MHz are detected with a deflection noise density of 100 $\text{fm}/\sqrt{\text{Hz}}$. FM-AFM imaging is performed on a Au(111) surface with a high-frequency cantilever. © *2005 American Institute of Physics.* [DOI: 10.1063/1.2149004]

Dynamic force microscopy (DFM)[1] allows us to investigate subnanometer-scale structures and properties of a wide range of materials. Amplitude modulation atomic force microscopy (AM-AFM)[1] has been intensively used for high-resolution imaging of polymers and biological materials in air and liquids. Molecular resolution of AM-AFM has been demonstrated in the imaging of proteins[2,3] and polymer single crystals.[4] On the other hand, frequency-modulation AFM (FM-AFM)[5] has been mainly used for high-resolution imaging in ultrahigh vacuum environments. The true atomic and molecular resolutions of FM-AFM have been demonstrated on various surfaces including insulators[6,7] and conductive materials.[8,9] Furthermore, recent progress in FM-AFM has made it possible to obtain subnanometer-scale resolution even in air[10,11] and liquids[11–13] by using a small oscillation amplitude (typically less than 1 nm). The applications of DFM include not only structural imaging but also measurements of electrical,[14,15] magnetic,[16] and mechanical[17,18] surface properties at a subnanometer-scale resolution.

The basic principles of AM- and FM-AFMs have predicted that the use of a cantilever with a high resonance frequency (f_0) should enhance the time response and force sensitivity in DFM-based techniques.[1,5] Recent studies have also suggested that higher harmonics of the fundamental resonance and higher-order flexural modes are useful for high-resolution imaging[19,20] and surface property measurements.[21–23] In spite of those advantages of the high-frequency DFM, the resonance frequencies used in DFM have been limited to less than about 1 MHz due to the insufficient bandwidth of the cantilever deflection sensors. Recently we have proposed a method to overcome the frequency limitation in DFM, which is referred to as the heterodyne optical beam deflection (HOBD) method.[24] The method makes it possible to detect high-frequency cantilever vibrations with a bandwidth limited only by the maximum laser power modulation frequency, which can be as high as gigahertz order.[25] In the previous study, we performed AM-AFM imaging at a cantilever resonance frequency of about 7 MHz using the HOBD method.[24] The improved time response owing to the high cantilever resonance was experimentally demonstrated. However, the HOBD method has not been applied to FM-AFM techniques.

It is important to extend the application range of the HOBD method to FM-AFM techniques because of the applicability of FM-AFM to vacuum environments. The high Q factor of a cantilever resonance in vacuum provides a high force sensitivity in both AM- and FM-AFMs. However, the high Q factor deteriorates the time response of the vibration amplitude. This makes it difficult to use AM-AFM in vacuum with a realistic time response. The applicability of FM-AFM to the vacuum environment enables accurate measurements of physical and chemical surface properties on atomically clean surfaces.[14]

In this study, we have developed an FM-AFM with a wideband cantilever deflection sensor using the HOBD method. In FM-AFM, the deflection signal is used not only for the frequency shift detection but also for producing the cantilever excitation signal. Thus, the implementation of the HOBD method in FM-AFM requires modifications in the

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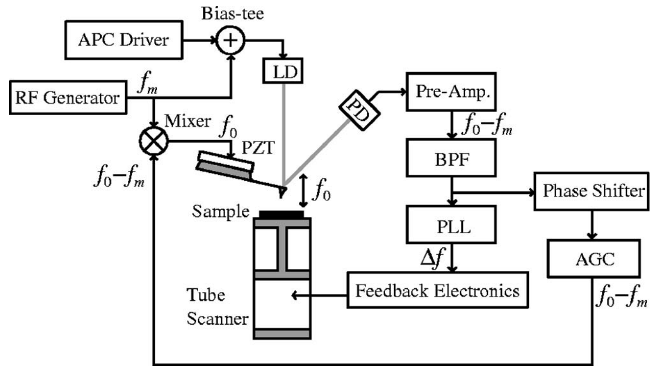

FIG. 1. Experimental setup for FM-AFM using the HOBD method. The phase and frequency of the cantilever vibration at f_0 are detected from the beat signal with a frequency f_0-f_m. The phase signal is used for the cantilever excitation in a self-oscillation circuit while the frequency shift signal (Δf) is used for the tip-sample distance regulation.

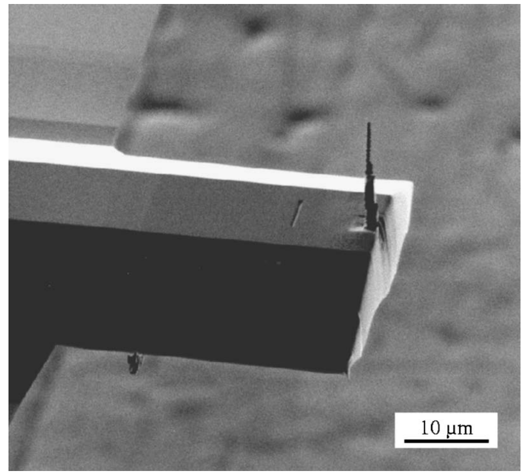

FIG. 2. Scanning ion microscopy image of a cantilever fabricated by cutting a commercially available Si cantilever using an FIB. A carbon tip was deposited at the end of the cantilever using the deposition mode of the FIB. The length, width, and thickness of the cantilever were typically 35 μm, 38 μm, and 7 μm, respectively. The resonance frequency was about 5.4 MHz. The spring constant estimated from the cantilever geometry was about 13 000 N/m.

self-excitation circuit as well as in the cantilever deflection sensor. This is, therefore, not necessarily straightforward from the AM-AFM setup using the HOBD method shown in the previous work.[24] In this article, we present a simple solution that enables stable self-oscillation using the HOBD method. The sensitivity of the deflection measurements by the HOBD method is quantitatively discussed. FM-AFM imaging with a high-frequency cantilever is performed on a Au(111) surface.

Figure 1 shows an experimental setup for FM-AFM using the HOBD method. The setup for the HOBD method is basically the same as those used for conventional optical beam deflection sensors. The only difference is that the laser power used in the HOBD method is modulated at a frequency (f_m) close to the cantilever fundamental resonance. The modulation signal is generated with a radiofrequency (rf) generator and fed into a bias-tee inserted between an automatic power control (APC) driver and a laser diode. In this way, the photocurrent induced by the bounced laser beam changes in proportion to both cantilever deflection and the laser power variation. Thus, the output signal of the preamplifier contains a beat signal with a frequency of f_0-f_m. If the frequency f_0-f_m is less than the bandwidth of the photodetector (B_{PD}), the amplitude, frequency, and phase of the cantilever vibration can be measured by detecting the beat component even when f_0 is much higher than B_{PD}.

A bandpass filter (BPF) is used for eliminating the unwanted frequency components contained in the deflection signal. Although the resonance frequencies of the cantilevers vary from one to another, we can use a fixed frequency BPF. This is because we can deliberately choose the beat frequency ($f_b=f_0-f_m$) by changing f_m. The Q factor of the BPF should be much smaller than that of the cantilever resonance so as not to affect its frequency-phase characteristics.

In FM-AFM, the frequency of the beat signal is detected with a frequency shift detector typically using a phase-locked loop (PLL) circuit. The detected frequency signal is used for the tip-sample distance regulation. The deflection signal is also fed into a phase shifter and an automatic gain controller (AGC), where the phase and the amplitude of the signal is adjusted to make the cantilever oscillate at its resonance with a constant amplitude (A). Then, the signal is mixed with the rf signal, producing two frequency components of $f_m+f_b(=f_0)$ and f_m-f_b. The former is used for the cantilever excitation while the latter is a spurious component that must be eliminated. The cantilever works as a mechanical BPF with a high Q factor for eliminating the spurious component. Thus, a variable frequency BPF is not required here.

A significant advantage of the HOBD method is its easy experimental setup. The setup requires only a few rf components: an rf generator, a bias-tee, and a frequency mixer. Since other components work in a low-frequency range, we can use those for conventional FM-AFMs without any modifications. The bandwidth of the HOBD method is not limited by B_{PD} but by the maximum frequency for laser power modulation, which can be as high as gigahertz order.[25]

The AFM used in this experiment was developed by modifying a commercially available AFM (JEOL: JSPM-4200).[11,24] An APC driver (ThorLabs: IP-500) and a bias-tee (Mini-Circuits: PBTC-1GW) were used for driving a laser diode (Hitachi: HL6312G). The rf signal for laser power modulation was obtained from an rf generator (Tektronics: AFG320). The degree of the laser power modulation was approximately 80%. The cantilevers used in this experiment were fabricated by cutting commercially available Si cantilevers (Nanosensors: NCL) using a focused ion beam (FIB). Figure 2 shows an example of the fabricated cantilevers. The length of the cantilever was about 35 μm. The resonance frequency was about 5.4 MHz. A carbon tip was deposited at the end of the cantilever using the deposition mode of the FIB. The Q factor was about 500 in air and about 2000 in vacuum.

Figure 3 shows a wave form (single sweep) and a fast Fourier transform (FFT) spectrum of the deflection signal measured with the high-frequency cantilever oscillated with a self-excitation circuit shown in Fig. 1. The frequencies f_0 and f_m were 5.434 and 5 MHz so that the beat frequency f_b was about 434 kHz in this case. The spectra show that the beat signal is detected with a sufficient signal-to-noise ratio. The voltage noise density of the deflection signal measured

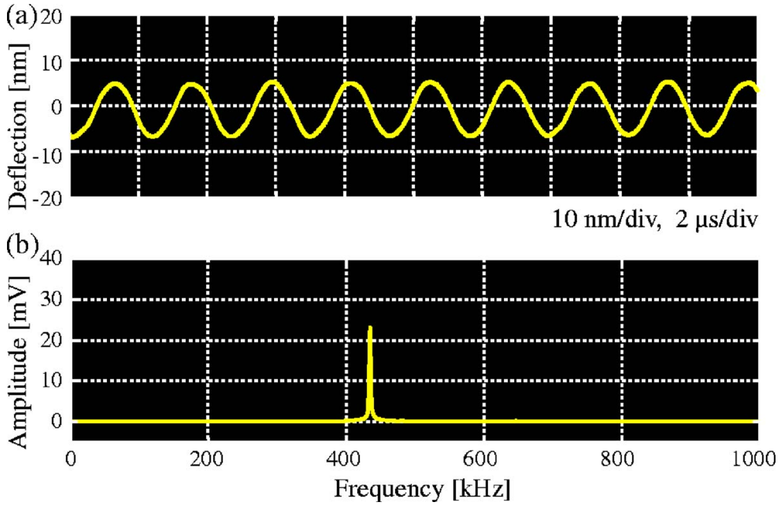

FIG. 3. (Color online) (a) A wave form (single sweep) and (b) an FFT spectrum of the deflection signal measured with a high-frequency cantilever oscillated with a self-excitation circuit shown in Fig. 1 (A=5 nm, f_0 =5.434 MHz, f_m=5 MHz, f_b=434 kHz). The deflection noise density measured with a spectrum analyzer was about 100 fm/$\sqrt{\text{Hz}}$. The measurement was performed in vacuum (vacuum pressure: about 10^{-4} Pa) at room temperature.

with a spectrum analyzer was about 860 nV/$\sqrt{\text{Hz}}$ while the deflection sensitivity was approximately 8 mV/nm. Thus, the deflection noise density was about 100 fm/$\sqrt{\text{Hz}}$, which is better than the typical values for commercially available AFMs (100–1000 fm/$\sqrt{\text{Hz}}$). The self-excitation circuit can be regarded as a phase feedback circuit. Accordingly, the stable self-oscillation of the cantilever shows the fact that the phase of the high-frequency cantilever vibration is detected by the HOBD method with a sufficient signal-to-noise ratio.

Figure 4 shows an FM-AFM image of a Au(111) surface taken in vacuum at room temperature using the HOBD method. The image shows the characteristic granular structure of the Au(111) surface. The tip-sample distance regulation was performed in a constant frequency shift mode with a positive frequency shift (Δf) set point. The stable tip-sample distance regulation demonstrates the applicability of the HOBD method to the frequency detection of high-frequency cantilever vibration.

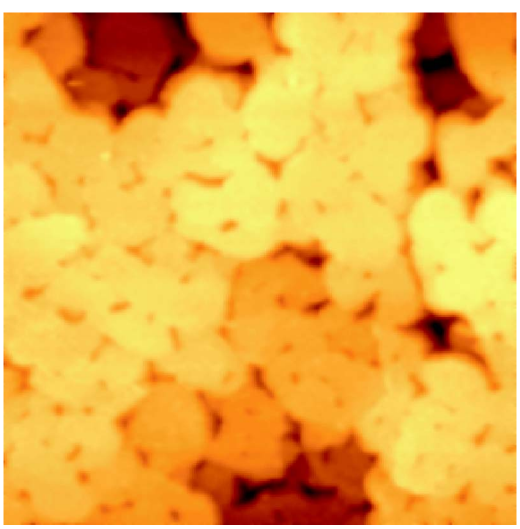

FIG. 4. (Color online) FM-AFM image of a Au(111) surface taken in vacuum (vacuum pressure: about 10^{-4} Pa) at room temperature using the HOBD method (4 μm$\times$4 μm, A=0.5 nm, Δf=+70 Hz, f_0=5.24 MHz, f_m=5 MHz, f_b=240 kHz, Q=1920). The tip-sample distance regulation was performed in constant frequency shift mode with a positive frequency shift set point.

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