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Optimization of Force Sensitivity in Q-Controlled Amplitude-Modulation Atomic Force Microscopy

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Control of noise in Q-controlled amplitude-modulation atomic force microscopy

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We present the controlled of noise in Q-controlled amplitude-modulation atomic force microscopy based on quartz tuning fork. It was found that the noise on phase is the same as the noise on amplitude divided by oscillation amplitude in AM-AFM. We found that Q-control does not change the signal-to-noise ratio. Nevertheless, the minimum detectable force gradient was found to be inversely proportional to the effective quality factor with large bandwidths in Q-controlled AM-AFM. This work provides that Q-control in AM-AFM is a useful technique for enhancement of the force sensitivity or for improvement of the scanning speed.

Since the invention of atomic force microscope (AFM),[1] it has been used in diverse research fields of physics, chemistry, biology and engineering. In particular, it has been introduced to study subatomic features of individual adatoms[2] or to measure the charge state of an adatom,[3] which requires high measurement sensitivity characterized by the minimum detectable force gradient.[4] In addition, for biological samples, increase of the scan speed of AFM is important for study of the dynamic behavior of biomolecules.[5–7] However, the signal can only be obtained at a finite accuracy and for a finite acquisition time due to the presence of noise. Therefore, the measurement noise is a critical factor that determines both the minimum detectable force gradient and the scan speed in AFM.

To determine the noise in AFM, the thermal noise spectra of oscillation amplitude has been usually measured in both amplitude modulation (AM)-AFM and frequency modulation (FM)-AFM. Recently, it was pointed out that the evolution of phase fluctuation to the frequency fluctuation is important in FM-AFM.[8] However, little attention has been paid on phase fluctuation or the fluctuation of force gradient in AM-AFM.

Q-control has been employed to increase Q for enhancement of force sensitivity at low-Q environment (e.g., in liquid). In contrast, the shorter relaxation time is required to image the solid surface faster in AM-AFM, low Q is necessary for force sensors which has high Q such as quartz tuning fork.[9] Because of these reasons, not only increasing Q but also reducing Q are required in AM-AFM. Meanwhile, many researchers have debated the effect of Q-control on the noise. It has been claimed that higher effective Q-factor confers little advantage in signal-to-noise ratio because the thermal noise is also amplified by Q-control in AM-AFM.[10] On the other hand, Kobayashi *et al.* demonstrated that the force sensitivity can be increased with Q-control in phase-modulation (PM)-AFM.[11,12] In PM-AFM, the force sensitivity was found to be proportional to $Q^{-1/2}$ for high Q. However, no experimental demonstration of noise control using Q-control has been performed in AM-AFM. Besides, how the Q-control affects the noise in AM-AFM has not also been clearly understood.

In this article, we investigate that the dependence of effective Q-factor on the noise of oscillation amplitude, phase and force gradient in AM-AFM. We show that the standard deviation of the phase fluctuation is the same as that of amplitude fluctuation divided by oscillation amplitude, which validates the method for quantification of noise. Based on the method, it is exhibited that the signal-to-noise ratio does not change by Q-control explicitly. Nevertheless, we demonstrate that the minimum detectable force gradient is controllable by using Q-control, and is shown to be proportional to Q^{-1} with large bandwidths.

Recently, the interaction stiffness has been frequently employed for quantitative description of tip-sample interaction force.[13–16] If the oscillation amplitude is small compared to the characteristic length of interaction, the interaction stiffness k_{int} in AM-AFM is given by[17–19]

$$k_{\text{int}} = k_0 \left[\frac{f}{Qf_0} \frac{A_0}{A} \sin\theta + \left(1 - \frac{f^2}{f_0^2}\right) \left(\frac{A_0}{A}\cos\theta - 1\right) \right], \tag{1}$$

where k_0 and Q are the spring constant and the quality factor of the force sensor, respectively, and A_0 is the free oscillation amplitude. A and θ are measured oscillation amplitude and phase difference, respectively, in the presence of external force at the driving frequency f.

The experiments were performed with our home-built AM-AFM that employs a quartz tuning fork (QTF)[20] as the force sensor in ambient conditions at temperature $T = 297.9 \pm 0.5$ K. It was determined experimentally that the effective stiffness of the QTF was $k_0 = 3820$ N/m and the piezoelectric coupling constant $\alpha = 5.99$ μC/m.[19] The QTF was driven by the resonance frequency, $f_0 = 32.76$ kHz. To drive the QTF, a function generator (33120A, Agilent Technologies) was equipped with a 1/1000 voltage divider, the resulting current due to displacement was converted and amplified into volt-

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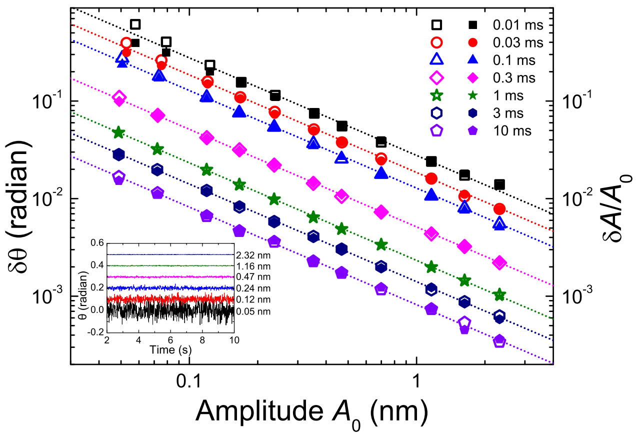

FIG. 1. Log-log plots of standard deviation (SD) of the phase, $\delta\theta$, (open points) and SD of amplitude divided by the oscillation amplitude, $\delta A/A_0$ (filled points) as a function of rms amplitude A_0 are depicted for several time constants τ of lock-in amplifier. The linear fit curves for SD of the phase exhibits the slope of -1.00. The inset shows the raw data of the fluctuation of phase in time domain with several values of A_0 for $\tau = 1$ ms, and the successive curves are presented with the offset just for clear eye guide.

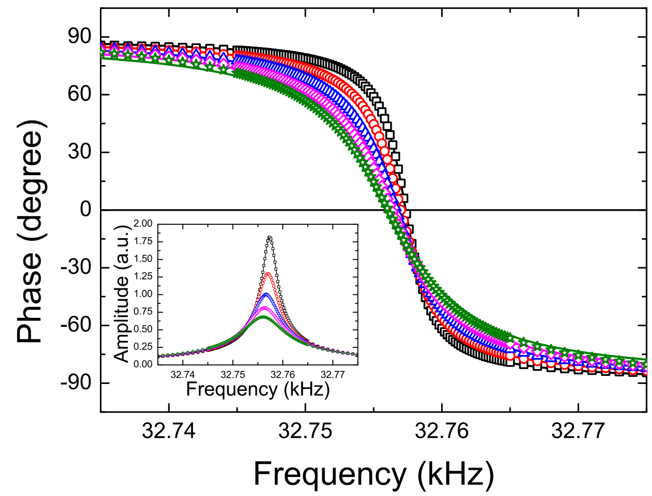

FIG. 2. The measured phases (open points) and their fits (solid lines) for several effective quality factors are represented as a function of driving frequency. Squares, circles, triangles, diamonds and stars correspond to the effective quality factor Q_{eff} of 11500, 8050, 6070, 4820, and 3990, respectively. It clearly shows that the Q-control changes the slope of phase-frequency curve near the resonance frequency. The inset shows the amplitude which were obtained by simultaneous measurements with the phase. Here the peak amplitude of the original resonance curve without Q-control ($Q = 6070$) was set to unity.

age by a preamplifier, and a lock-in amplifier (SR830, Standard Research Systems) decomposed the output into amplitude and phase, which are recorded by a computer. The signal passed through the preamplifier was fed back to the driving signal to the QTF via our home-made feedback circuit to control the quality factor.[9]

The inset of Fig. 1 shows the measured phase as a function of time for several oscillation amplitudes. It clearly shows that the larger oscillation amplitude, the smaller fluctuation of the phase. To approach the fluctuation quantitatively, we take the standard deviation (SD) of the fluctuation of the phase and amplitude without the transient signal.[21] Figure 1 presents $\delta\theta$ (SD of phase) and $\delta A/A_0$ (SD of amplitude divided by the oscillation amplitude) as a function of A_0 for various bandwidths B which were was controlled by adjusting the time constant of the lock-in amplifier.

It was observed that, first of all, $\delta A/A_0$ were inversely proportional to the oscillation amplitude A_0, which indicates that the noise on amplitude is constant as the oscillation amplitude changes. In addition, the slope of the plot of $\delta\theta$ versus B was found to be 0.541 ± 0.029 (not shown here), close to 1/2, suggesting that the noise density is constant. Besides, $\delta\theta$ was revealed to be the same as $\delta A/A_0$, which has good agreement with the result in PM-AFM,[11] and which also implies that $\delta\theta$ denotes an inverse of signal-to-noise ratio. From these results, we consider that the standard deviation of phase or amplitude is sufficient to be a measure of noise.

We now consider the response of QTF under Q-control. Figure 2 depicts the phase and the amplitude measured as a function of driving frequency f. The effective quality factor, Q_{eff} was enhanced or reduced with respect to the quality factor without Q-control, $Q = 6070$, by controlling the gain and of the feedback circuit. It was found that the peak amplitude grows as Q_{eff} increases in the inset of Fig. 2, which is consistent with the literature.

We had a close look at the phase curve affected by Q-control. A slight shift of the resonance frequency was observed as shown in Fig. 2, which is due to parasitic capacitance of electrically-driven QTF.[9] In addition, it was found that as Q_{eff} gets larger, the slope of the phase-frequency graph gets steeper near the resonance frequency. This suggests smaller frequency fluctuation for larger Q_{eff} under the same phase fluctuation. In other words, the slope of the phase-frequency graph at the resonance frequency, which is given by

$$\left|\frac{\Delta\theta}{\Delta f}\right| = \frac{2Q_{\mathrm{eff}}}{f_0} = \frac{1}{f_{\mathrm{c}}}, \tag{2}$$

is proportional to the effective quality factor, Q_{eff}, and roughly constant within $f_0 \pm f_{\mathrm{c}}$ where f_{c} is called the cut-off frequency.[8] It is worth emphasizing that this change of the slope is important in the evolution of the phase fluctuation $\delta\theta$ to the frequency fluctuation δf, i.e.,

$$\delta f = \left|\frac{\Delta f}{\Delta\theta}\right| \delta\theta = \left(\frac{2Q_{\mathrm{eff}}}{f_0}\right) \delta\theta\ , \tag{3}$$

and to the fluctuation of force gradient as discussed below.

We now consider the influence of Q-control on the phase fluctuation follwed by that on the fluctuation of

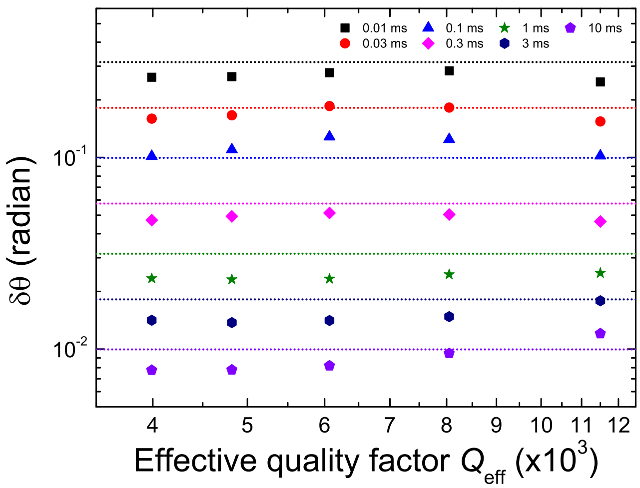

FIG. 3. The noise on phase, $\delta\theta$, as a function of the effective quality factor, $Q_{\rm eff}$, for various bandwidths is depicted when the amplitude is $A_0 = 0.1$ nm (rms). The dashed line of each bandwidth is the theoretical value obtained from Eq. (7). The noise on phase, an inverse of signal-to-noise ratio, does not change by Q-control.

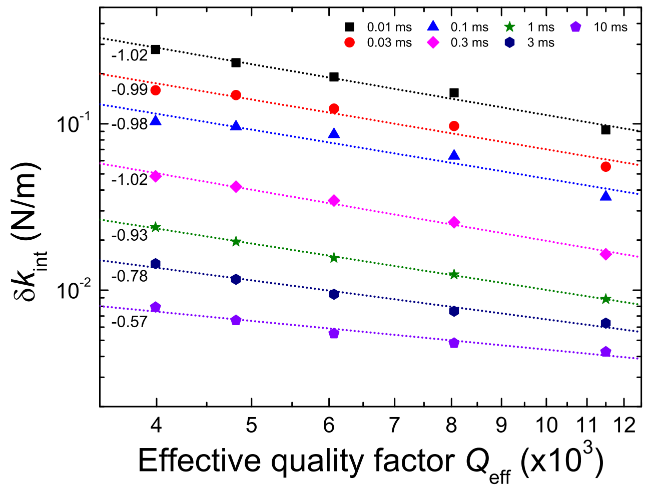

FIG. 4. Log-log plots of the noise of interaction stiffness at the rms oscillation amplitude of 0.1 nm versus the effective quality factor $Q_{\rm eff}$ for several time constants are presented. Each dashed line denotes the linear fit, and the value shown at the left-end of the line represents its slope.

force gradient. Figure 3 shows that the measured noise on phase, $\delta\theta$ versus the effective quality factor, $Q_{\rm eff}$, for various bandwidths when the oscillation amplitude was $A_0 = 0.1$ nm. It was found that $\delta\theta$ is almost constant as $Q_{\rm eff}$ changes, indicating the noise on phase, $\delta\theta$, an inverse of signal-to-noise ratio, does not change by Q-control. As pointed out by Ashby,[10] it implies that Q-control amplifies the noise as well as the signal when $Q_{\rm eff}$ is increased. In addition, it was observed that the phase noise is increased for large $Q_{\rm eff}$ and small bandwidths (long time constants), suggesting the signal which decreases due to small bandwidths comparable to the cutoff frequency $f_{\rm c}$. For example, the half of bandwidth $B/2 = 3.9$ Hz for $\tau = 10$ ms is comparable to $f_{\rm c} = 2.70$ Hz for $Q_{\rm eff} = 11500$. The results of phase fluctuation show that Q-control has no advantage in signal-to-noise ratio in AM-AFM, which has good agreement with a previous study.[10]

To compare the experimental results to the theoretical value quantatitively, the thermal noise is usually considered.[10] The magnitude of random driving force is given by[8]

$$F_{\rm th} = \sqrt{\frac{2k_0 k_{\rm B} T}{\pi f_0 Q}}\ , \tag{4}$$

where $k_{\rm B}$ is the Boltzmann constant. In addition, the magnitude of the transfer function $|G(f)|$ is given by

$$|G(f)| = \frac{1}{k_0}\frac{1}{\left[\left(1-f^2/f_0^2\right)^2 + \left(f/f_0 Q\right)^2\right]^{1/2}}\ . \tag{5}$$

which leads to $|G(f)| = Q/k_0$ when the force sensor is driven at the resonance frequency. The thermal displacement noise density $n_{\rm th} = |G(f)|\, F_{\rm th}$ is then given by

$$n_{\rm th} = \sqrt{\frac{2k_{\rm B} T Q}{\pi f_0 k_0}}\ . \tag{6}$$

Then the thermal fluctuation on phase, $\theta_{\rm th}$, is then given by

$$\delta\theta_{\rm th} = \frac{\delta A_{\rm th}}{A_0} = \sqrt{\frac{2k_{\rm B} T Q B}{\pi f_0 k_0 A_0^2}}\ . \tag{7}$$

The thermal noise on phase calculated using Eq. (7) is also represented in Fig. 3. It implies that thermal noise is dominant in this experiment, and that the effective quality factor $Q_{\rm eff}$ does not employed instead of Q in Eq. (7).

Now we take a look how Q-control affects the interaction stiffness. Figure 4 shows the noise on interaction stiffness (also represents minimum detectable force gradient), $\delta k_{\rm int}$, in Q-controlled system for various bandwidths when the oscillation amplitude was 0.1 nm. The interaction stiffness, $k_{\rm int}$ was obtained by using Eq. (1) in terms of the measured amplitude A and phase θ. It is worth emphasizing that $Q_{\rm eff}$ should be introduced instead of Q in Eq. (1) because the interaction stiffness is obtained from the frequency shift due to interacting forces.

Interestingly, it was found that large Q reduces $\delta k_{\rm int}$, which clearly shows the improved force sensitivity in AFM with the increase of Q. In particular, $\delta k_{\rm int}$ was observed to be proportional to $Q_{\rm eff}^{-1}$ with large bandwidths. This is not an expected result because the minimum detectable force gradient due to thermal noise is given by[4]

$$\delta k_{\rm int,th} = \sqrt{\frac{2k_0 k_{\rm B} T B}{\pi f_0 Q A_0^2}}\ . \tag{8}$$

which is proportional to $Q^{-1/2}$.

To resolve this discrepancy, the relation between $\delta k_{\rm int}$ and $\delta\theta$ is required to be found. For the first step, the frequency shift Δf due to a small interaction stiffness $k_{\rm int}$ is given by[16]

$$\Delta f = f_0 \left(\frac{k_{\rm int}}{2k_0}\right) . \tag{9}$$

Combining Eq. (9) with Eq. (3), the noise on interaction stiffness, $\delta k_{\rm int}$, is given by

$$\delta k_{\rm int} = \left(\frac{2k_0}{f_0}\right)\delta f = \left(\frac{k_0}{Q_{\rm eff}}\right)\delta\theta . \tag{10}$$

Equation (10) indicates that the noise on interaction stiffness, or minimum detectable force gradient is inversely proportional to $Q_{\rm eff}$ under the same phase fluctuation $\delta\theta$. Then the relation the noise on interaction stiffness with Q-control $\delta k_{\rm int}$ and without Q-control $\delta k_{\rm int}^{(0)}$ is given by

$$\delta k_{\rm int} = \left(\frac{Q}{Q_{\rm eff}}\right)\delta k_{\rm int}^{(0)} . \tag{11}$$

The result shown in Fig. 4 is consistent with Eq. (11), which clearly shows that the minimum detectable force gradient (equal to $\delta k_{\rm int}$) and the minimum detectable interaction force δF are inversely proportional to $Q_{\rm eff}$ with sufficiently large bandwidths. Note that when the phase fluctuation $\delta\theta$, or the deflection δA is constant, Eq. (11) holds no matter what kind of noise works.

In spite of the control of the force sensitivity, there is a trade-off between the minimum detectable force gradient and the relaxation time of the force sensor in AM-AFM. The relaxation time, which is the time constant of a change until the signal at a state reaches another steady state, is given by $\tau_{\rm sensor} = Q_{\rm eff}/(2\pi f_0)$,[9] which is proportional to $Q_{\rm eff}$. It implies that when $Q_{\rm eff}$ is adjusted to κQ, $\delta k_{\rm int}$ and $\tau_{\rm sensor}$ becomes $1/\kappa$ and κ times as much as their original values without Q-control. Therefore, the effective quality factor $Q_{\rm eff}$ can be properly selected using Q-control depending on the specific purpose such as the increased sensitivity or the increased measurement speed in AM-AFM.

Comparing these results to the result obtained in PM-AFM, δF is proportional to $Q_{\rm eff}^{-1/2}$ with large bandwidths in PM-AFM,[11,12] which is inconsistent with our result in AM-AFM. It is because the noise on amplitude (the deflection noise) δA (or $\delta\theta$) is proportional to $Q_{\rm eff}^{1/2}$ in PM-AFM, whereas $\delta\theta$ is independent of $Q_{\rm eff}$ in AM-AFM. Therefore, the enhancement or reduction of force sensitivity both in AM-AFM and in PM-AFM results from the variation of the slope in phase-frequency plot (see Fig. 2). In addition, the $1/Q_{\rm eff}$-dependence of $\delta k_{\rm int}$ in Q-controlled AM-AFM is similar to the oscillator noise in FM-AFM,[8,16] because the noise on frequency due to the oscillator noise, $\delta f_{\rm osc}$, is proportional to the frequency derivative of the phase shift, $\Delta f/\Delta\theta$.[8]

We have demonstrated that the minimum detectable force gradient is adjustable by Q-control using QTF-based AM-AFM. It has been found that the noise on phase is the same as the noise on amplitude divided by the oscillation amplitude, which indicates the standard deviation of phase or amplitude is a measure of noise. We have shown that the signal-to-noise ratio does not change under Q-control. Nevertheless, the minimum detectable force gradient is inversely proportional to the effective quality factor with sufficiently large bandwidths. Therefore, Q-control is expected to enhance the force sensitivity or fast the scanning speed in AM-AFM.

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[20] Epson C-004R purchased from Digikey Corporation.
[21] The data analysis starts after the initial two seconds during which the signal reaches the steady state.