Novel Method for High Speed Force Curve Measurement considering Cantilever Dynamics for Atomic Force Microscopy

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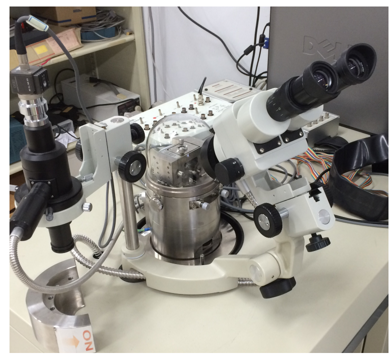
***Abstract*—Atomic Force Microscopy (AFM) is a scanning probe microscope with nanoscale resolution as well as an indispensable device for nanotechnology. Since AFM probe physically touches the sample surface, expectations for sample dynamics measurement are raising. One common measurement mode is force curve measurement. In the force curve measurement, atomic force is detected by the spring constant of the cantilever. In high speed measurement, however, the cantilever oscillates with its resonance frequency and cannot detect the atomic force. This paper proposes novel methods to identify cantilever dynamics and to measure the force curve in high speed using atomic force observer (AFO) and the effectiveness of the proposed measurement method was demonstrated by simulations and experiments. Furthermore, the robustness of the AFO against modeling error is discussed.**

I. Introduction

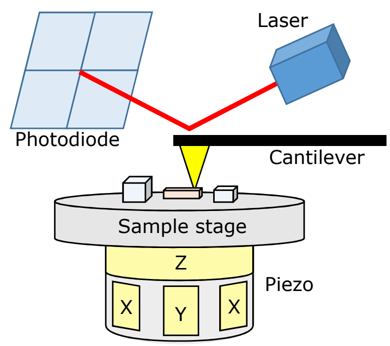
The Atomic Force Microscopy (AFM) is a scanning probe microscope with nanoscale resolution. Since AFM, unlike Scanning Tunneling Microscope (STM), can measure all types of sample material, it plays a crucial role in nanotechnology, molecular biology and material science. Fig. 1(a) shows the AFM of the authors' research group.

Fig. 1(b) shows the structure of AFM. AFM is composed by a piezo actuator which handles the stage and a nano probe with a metal stick called cantilever. When the probe tip touches the sample surface, the atomic force between them causes cantilever deflection. By detecting cantilever deflection with laser beams and photodiodes, the surface topology can be measured with very high resolution.

There are two common types of AFM [1]: one is a contact-mode AFM and the other is a dynamic-mode AFM. In the contact-mode AFM, the probe tip keeps contact with the sample and detects surface topology by the input voltage on the piezo stage. In the dynamic-mode AFM, resonance frequency shift or amplitude variation of the cantilever are used. Since AFM probe can hold physical contact with the sample, expectations for sample dynamics measurement are raising [2]-[3]. The authors' research group proposed simultaneous estimation of surface topology and elasticity using contact-mode AFM [4]. Dynamics measurements are especially important in biomaterials, where there are many unknown behaviors.



(a) Experimental setup (JSPM-5200).



(b) Structure of AFM.

Fig. 1. Atomic force microscope.

Force curve measurement is known as the typical measurement of sample dynamics for AFM. Force curve is the plot of atomic force against piezo displacement. Conventionally, the atomic force is estimated by the deflection and the spring constant of cantilever. Because soft cantilevers are more sensitive to atomic forces, soft cantilevers are used for force curve measurements. However, high speed measurement of the true atomic force is difficult because the soft cantilever has low resonance frequency and it leads to large cantilever vibration in high speed. One solution of this problem is using a stiffer cantilever, but in that case the cantilever is less sensitive to the atomic force and this is not desirable for force

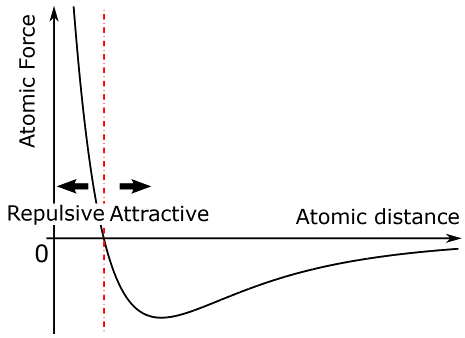
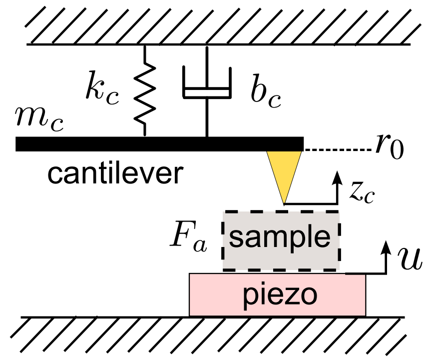
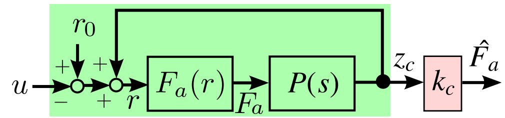


Fig. 2. Force / distance relationship.



(a) Dynamic model of AFM.



(b) Block diagram of AFM.

Fig. 3. Modeling of AFM

measurement. In this paper, a high speed force measurement method based on atomic force observer (AFO) [5] is proposed. AFO is proposed by the authors' research group, but since there were no good applications and proper ways to identify the dynamics of cantilever, AFO has never been used [6]. Thus, this paper proposes a novel model identification method using transient response of the cantilever for high speed force curve measurement.

This paper discusses how to realize the high speed force curve measurement. In section 2, atomic force and AFM are physically modeled. Section 3 describes the conventional method of force curve measurement. Section 4 proposes the method to identify the cantilever dynamics and high speed force curve measurement using AFO. In section 5, the performance of the proposed method is demonstrated in simulations and experiments. Section 6 discusses the robustness of the AFO.

II. Modeling

A. Modeling of the atomic force

The atomic force is the interactions between atoms, such as electrostatic forces and bounding forces. Atomic forces have two different behaviors dependent on atomic distance. When two atoms are distant, there are almost no interactions between them. When they get closer, they exert attractive forces with each other, and when the distance is bigger than the lattice constant, Van der Waals forces are dominant. On the other hand, inside lattice constant, strong repulsive forces are dominant on account of Pauli Exclusion Principle. Contact-mode AFM is operated in the latter case, while dynamic-mode AFM can be applied in both cases. Fig. 2 shows the relationship between the atomic force and distance based on Lennard-Jones model [7]. Lennard-Jones model is an approximation of atomic force F_a and can be expressed as a function of atomic distance r.

$$F_a(r) = 24\varepsilon\left(2\frac{\sigma^{12}}{r^{13}} - \frac{\sigma^6}{r^7}\right), \tag{1}$$

where ε is the binding energy and σ is the lattice constant.

B. Modeling of the AFM

Fig. 3(a) shows the physical model of AFM [8] and Fig. 3(b) shows its block diagram. In the Fig. 3(a), r, r_0, z_c, $F_a(r)$ are the distance between probe tip and sample, the initial value of r, the deflection of cantilever, and the function of r that regulates atomic force F_a respectively. The block $P(s)$ is the transfer function of cantilever from atomic force to deflection; its nominal model $P_n(s)$ is given as

$$P_n = \frac{z_c}{F_a} = \frac{1}{m_c s^2 + b_c s + k_c}. \tag{2}$$

Here, m_c, b_c, k_c are mass, viscosity, and elasticity of the cantilever respectively. In this paper, it is assumed that the piezo actuator is much stiffer than the sample and the cantilever and that the displacement of the piezo is proportional to the input voltage.

III. Conventional force curve measurement

A. Method of force curve measurement

Force curve measurement is a method to measure the atomic force with AFM. Force curve measurement is performed as follows.

- STEP 1 Elevate the stage until the probe touches the sample. Then, keep contact with them by feedback control.
- STEP 2 Inactivate the feedback control, and set the distance reference value.
- STEP 3 Approach the probe by elevating the stage, and push into the sample.
- STEP 4 Retract the probe by lowering the stage.

Atomic force is detected by multiplying the spring constant by the deflection of the cantilever. The force curve is the plot of atomic force against piezo displacement in STEP 3 and STEP 4. Fig. 4 is an example of force curve. As explained in section II, the repulsive force and the attractive force can be detected and, when the probe retracts from the sample, adhesive force acts on the probe. Therefore, there is a hysteresis behavior when retracting like Fig. 4.

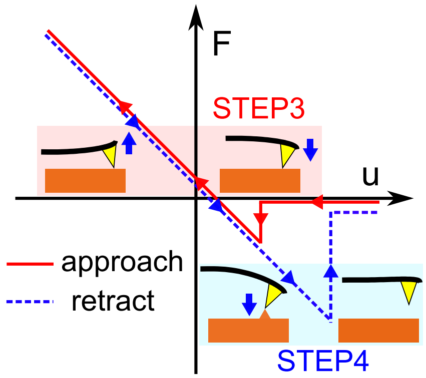
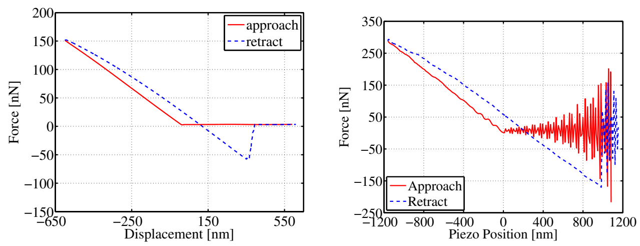


Fig. 4. Conceptual diagram of force curve and response of cantilever.



(a) Low speed measurement

(b) High speed measurement

Fig. 5. Force curve (experiment).

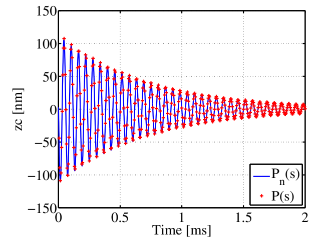
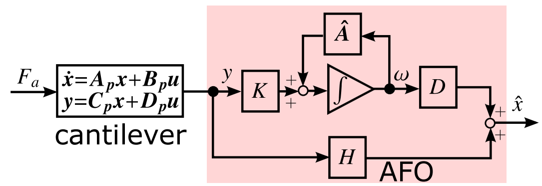
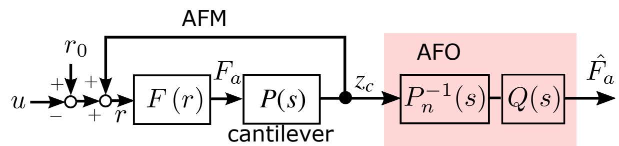


Fig. 6. System identification of AFM cantilever (experiment).



(a) Atomic force observer.



(b) Block diagram equivalent to atomic force observer.

Fig. 7. Design of atomic force observer.

B. *Problem of conventional method*

The atomic force is conventionally detected by the spring constant and the deflection of the cantilever. In low speed measurement, the conventional method can measure the atomic force correctly. In high speed measurement, however, the cantilever oscillates with resonance frequency, which makes a correct force measurement difficult.

Fig. 5 (a), (b) are the force curves of low speed measurement and high speed measurement, respectively. In slow speed measurement, the atomic force can be correctly measured and the force curve can be obtained. However, in high speed measurement, force curve vibrates because the conventional method cannot separate the cantilever oscillation from the deflection caused by atomic force.

IV. Proposal of high speed measurement of force curve using atomic force observer

In this section a novel high speed measurement method of force curve based on atomic force observer (AFO) is proposed. It is difficult to identify dynamics of cantilevers because input force contains sample dynamics. Thus, AFO have not been used to measure atomic force. This paper proposes a system identification method using impulse response and it becomes possible to design AFO.

A. *Proposal of dynamics identification for AFM cantilever*

As shown in Fig. 3(a), it is difficult to separate the dynamics of AFM cantilever from the atomic force between the probe and the sample. Frequency response from piezo position to cantilever deflection is used for system identification of AFM. However, because the atomic force between sample surface and probe tip changes nonlinearly according to their distance, it is extremely difficult to identify the cantilever model by frequency response. Thus, this paper proposes a novel method of system identification for AFM cantilever. When the probe and the sample are pulled apart, impulse force is impressed due to the adhesive force. The probe and the sample keep enough distance so that they do not interact with each other. Therefore, the cantilever dynamics without the sample dynamics can be obtained.

In Fig. 6, transient response of cantilever is shown. Generally, time response of damped vibration is given as (3). α is an initial phase angle.

$$z_c(t) = Ce^{-\zeta\omega_0 t}\cos(\omega_0\sqrt{1-\zeta^2}t-\alpha). \tag{3}$$

ζ and ω_0 can be given as (4) by using m_c, b_c, and k_c.

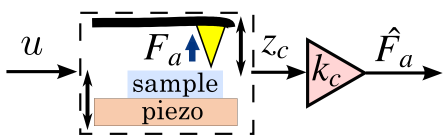
$$\omega_0 = \sqrt{\frac{k_c}{m_c}},\ \zeta = \frac{b_c}{2m_c\omega_0}. \tag{4}$$

$P_n(s)$ is derived from curve fitting. By fitting (3) to measurement result, the dynamics of the cantilever can be detected and expressed as Table I.

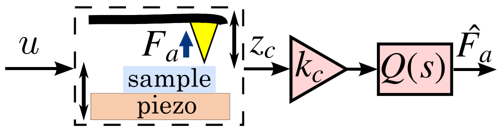
B. *Design of atomic force observer*

In this section, atomic force observer is designed with the parameters identified in the previous section. The motion equation of cantilever is

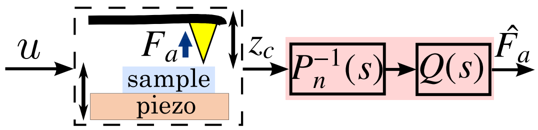
$$m_c\ddot{z}_c + b_c\dot{z}_c + k_cz_c = F_a. \tag{5}$$



(a) Conventional method1.



(b) Conventional method2.



(c) Proposed method.

Fig. 8. Structure of the conventional methods and the proposed method.

From (5), state equation of extended system can be expressed as (6) and its state variables are z_c, $\dot{z}_c$, and F_a.

$$\dot{\boldsymbol{x}} = \boldsymbol{A}\boldsymbol{x},\ \boldsymbol{A} = \begin{bmatrix} 0 & 1 & 0 \\ -\frac{k_c}{m_c} & -\frac{b_c}{m_c} & \frac{1}{m_c} \\ 0 & 0 & 0 \end{bmatrix}. \tag{6}$$

Its output equation is

$$\boldsymbol{y} = \boldsymbol{C}\boldsymbol{x},\ \boldsymbol{C} = \begin{bmatrix} 1 & 0 & 0 \end{bmatrix}. \tag{7}$$

AFO is an observer estimating F_a. Fig. 7(a) shows the state space expression of AFO. H and D are coefficient matrix of the observer and K is the observer gain.

A minimal order observer can be designed by removing z_c and $\dot{z}_c$. Fig. 7(b) is the minimal order expression of AFO derived from (6) and (7). $P_n^{-1}(s)$ is the inverse transfer function of nominal plant, $Q(s)$ is the second order low pass filter to make proper inverse model and ω_c is the cut-off frequency of $Q(s)$. Observer gain is reflected in ω_c.

$$P_n^{-1}(s) = m_c s^2 + b_c s + k_c, \tag{8}$$

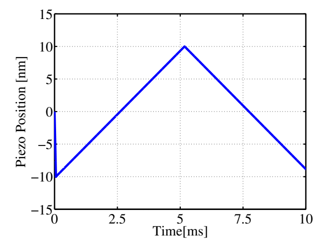
$$Q(s) = \frac{\omega_c^2}{(s + \omega_c)^2}. \tag{9}$$

Fig. 8(a) shows the structure of the conventional method in which only spring constant is used. Fig. 8(c) shows the proposed method using AFO. The proposed method uses spring constant, mass and viscosity.

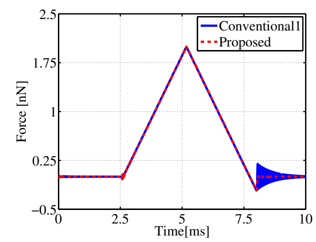
The proposed method aims to measure the real atomic force regardless of the cantilever oscillation. This means that the force curve oscillation can be suppressed in the experimental result. However, low pass filter $Q(s)$ also can decrease the oscillations in force curve. Therefore, the effect of $Q(s)$ should be separated. For comparison, a model using only $Q(s)$ and spring constant k_c is prepared. This model will be called "conventional method 2" in the latter part of this paper. The structure of the conventional method 2 is shown in Fig. 8(b).

TABLE I
CONDITIONS OF THE SIMULATIONS.

	value	unit
sampling frequency	200	kHz
pole of LPF	40	kHz
measurement range	±10	nm
σ	1.8×10^{-10}	m
ε	1.6×10^{-20}	J
r_0	1.6×10^{-10}	m
m_c	18.06	ng
b_c	160	pN·s/m
k_c	0.2	N/m



(a) Displacement of the stage



(b) Measurement of atomic force

Fig. 9. Results of the simulations.

TABLE II
CONDITIONS OF THE EXPERIMENTS.

	value	unit
sampling frequency	200	kHz
pole of LPF	40	kHz
measurement range	±1000	nm
sample	Si	
m_c	18.06	ng
b_c	160	pN·s/m
k_c	0.2	N/m

V. SIMULATIONS AND EXPERIMENTS

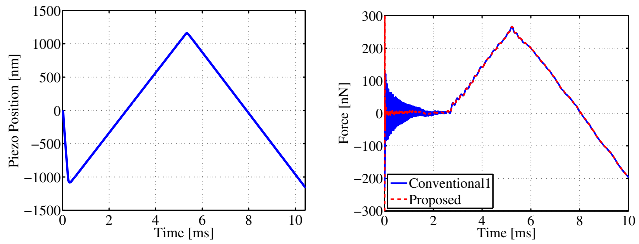
A. Simulations of high speed force curve measurement

Using the cantilever dynamics in the section IV-A, the approach and retraction of the probe to the sample is simulated. Table I shows the simulation parameters. Lennard Jones model is used to describe the atomic force and it is assumed that probe tip is made of SiO_2 and the sample is Cu. Fig. 9(a) plots the displacement of the piezo actuator. The motion of the piezo actuator follows STEP 2-STEP 4 in section III-A.

Fig. 9(b) shows the results of the simulation. Cantilever oscillates at 7.5 milliseconds sec due to adhesive forces. The conventional method is not able to measure the atomic force correctly. On the other hand, the proposed method is not affected by the cantilever oscillation and can estimate the atomic force correctly.

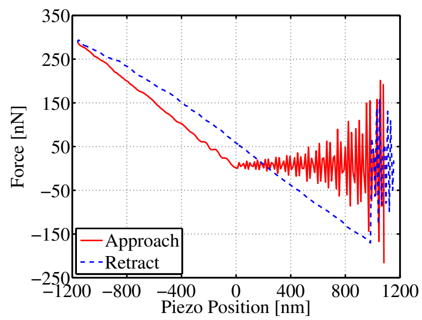
B. Experiment of high speed force curve measurement

The superiority of the proposed method to the conventional method is verified by the experiment. JSPM-5200 is used as the experimental device. The sample is made of copper and cantilever is CONTR, manufactured by NANO PROBE. The experimental conditions are shown on Table II. Initial distance r_0 cannot be decided like in simulations because it is difficult

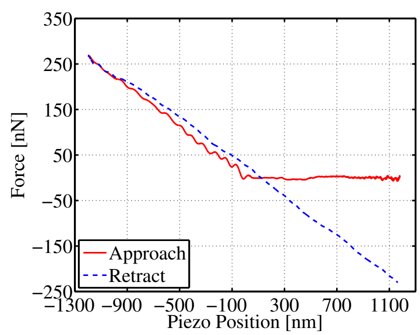


(a) Displacement of the stage (b) Result of the experiment

Fig. 10. High speed force curve measurement ($v_{high} = v_{normal} \times 20 = 240$ nm/ms).



(a) Conventional1



(b) Proposed method

Fig. 11. The force curve with high speed measurement.

to measure the distance between probe tip and sample. Thus, initial point is decided by the cantilever deflection instead of using r_0. Fig. 10(a) show the displacement of the piezo actuator. The velocity of the piezo actuatour is $v_{normal} = 12$ nm/ms in normal speed measurement and $v_{high} = 240$ nm/ms in high speed measurement.

Fig. 10 shows the high speed measurement of atomic force. Besides, Fig. 11 is the force curve of the proposal and conventional methods. In high speed measurement, the conventional method cannot measure the atomic force due to cantilever oscillation. On the other hand, proposed method can measure the atomic force despite of cantilever oscillation.

Fig. 12 shows the comparison of conventional method2 and the proposed method. Because the cut off frequency of Q is much larger than cantilever resonance, $Q(s)$ does not suppress the oscillation, while the proposed method does. Therefore, the fact that not $Q(s)$ but $P_n^{-1}(s)$ is a critical element for AFO

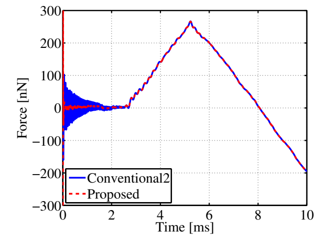


Fig. 12. Effect of the law pass filter

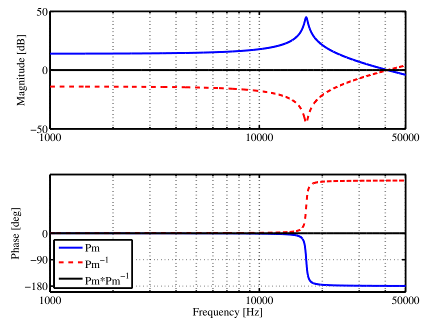


Fig. 13. Frequency response of AFO.

is proved.

The experimental result is much different from simulation in atomic force. Since Lennard Jones model is an approximation, a simulation using Lennard-Jones potential is not always true to the real atomic force. Lennard Jones model is assuming the case with two atoms. In this case, atomic force is much larger than that of the simulation [7].

VI. Robustness of AFO

Fig. 13 shows the Bode diagram of a cantilever, its nominal inverse model, and their product. When the nominal viscosity and the spring constant of the inverse model have no modeling error, F_a corresponds to $\hat{F}_a$.

In this section, the robustness of AFO against the modeling errors is discussed.

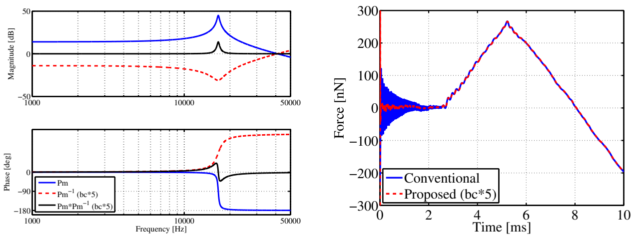
A. Robustness against viscosity errors

Fig. 14(a) shows the frequency response of a cantilever and its inverse model. When nominal viscosity is estimated 5 times larger than the real viscosity, the resonance peak become smaller as shown by red line. Therefore, cantilever oscillation can be reflected on estimated force $\hat{F}_a$. Fig. 14(b) is the experimental result of this case. Even though the nominal viscosity is very different from real viscosity, the force oscillation is considerably suppressed.

Fig. 15(a) shows the simulation result and Fig. 15(b) shows an enlargement of a portion of Fig. 15(a). Since the b_c error is 5 times larger, the oscillation amplitude becomes 1.5 times larger when there are no errors.

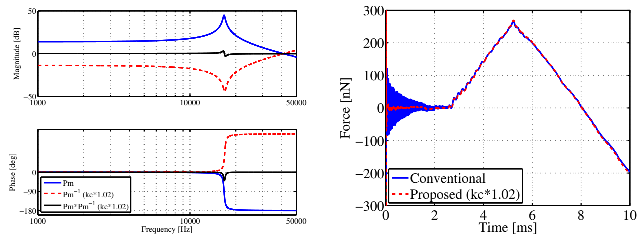
B. Robustness against spring constant errors

Fig. 16(a) shows the frequency response of the cantilever and its inverse model with a 2% k_c errors. Since the frequency



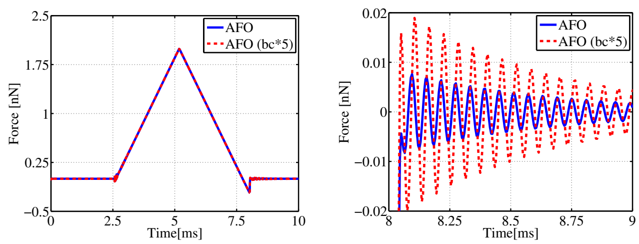
(a) Frequency response (b_c*5). (b) Time response (b_c*5).

Fig. 14. Robustness of the AFO in the experiment (b_c is 5 times larger).



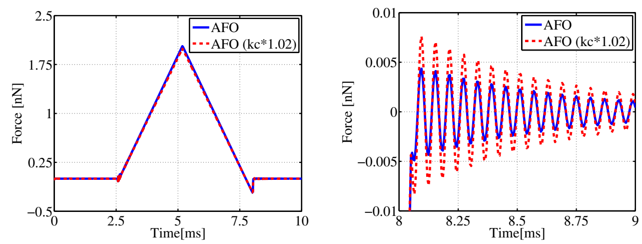
(a) Frequency response (k_c*1.02). (b) Time response (k_c*1.02).

Fig. 16. Robustness of the AFO in the experiment (k_c*1.02).



(a) Time response (b_c*5). (b) Time response (b_c*5).

Fig. 15. Robustness of the AFO in the simulation (b_c is 5 times larger).



(a) Time response (k_c*1.02). (b) Time response (k_c*1.02).

Fig. 17. Robustness of the AFO in the simulation (k_c*1.02).

response of the cantilever has a steep peak, unlike the case of the viscosity errors, $\hat{F}_a$ is sensitive to k_c errors. k_c error leads to resonance frequency shift of the inverse cantilever model.

Fig. 16(b) is the experimental time response of the cantilever in the case k_c is 2% larger. The force curve oscillation can be suppressed substantially despite of the k_c error. However, the estimated repulsive force is a bit different from the real one. When the maximum repulsive force works on the probe tip, the estimated force error is 1%.

Fig. 17(a) shows the simulation result and Fig. 17(b) shows the larger one. With a large k_c errors, not only oscillation suppression but also measurement result in the repulsive region becomes a problem. However, in the system identification, 0.5 to 1% k_c error can be expected. Therefore, 2% is a sufficient margin to identify the cantilever system.

VII. Conclusion

In force measurement with AFM, there is a trade-off between bandwidth of the cantilever and sensitivity, which makes high speed force curve measurement difficult. This paper proposed a method to identify cantilever dynamics and a high speed force curve measurement based on atomic force observer. Then, the robustness of an AFO was verified. The proposed method can measure the correct atomic force even when the cantilever is vibrating.

Not only high speed force measurement itself is important, but it has various applications such as nano-manipulation and elasticity estimation. AFO can observe the atomic force in spite of cantilever oscillation but cannot suppress the oscillation itself. Therefore, when the distance between probe tip and sample changes, the force curve vibrates. To solve this problem, vibration suppression control for the cantilever will be studied in the future.

References

[1] T. Shiraishi and H. Fujimoto, "Proposal of Surface Topography Observer considering Z-scanner for High-speed AFM," *American Control Conference, 2010*, pp. 2754–2759, 2010.

[2] M. a. Lantz, H. J. Hug, R. Hoffmann, P. J. van Schendel, P. Kappenberger, S. Martin, A. Baratoff, and H. J. Güntherodt, "Quantitative measurement of short-range chemical bonding forces." *Science (New York, N.Y.)*, vol. 291, no. 5513, pp. 2580–3, Mar. 2001.

[3] C. D. Onal, S. Member, M. Sitti, and S. Member, "Teleoperated 3-D Force Feedback From the Nanoscale With an Atomic Force Microscope," *IEEE TRANSACTIONS ON NANOTECHNOLOGY*, vol. 9, no. 1, pp. 46–54, 2010.

[4] W. S and F. H, "Proposal for Simultaneous Estimation of Sample Surface Topography and Elasticity Utilizing Contact-Mode AFM," *IEEJ Transactions on Industry Applications*, vol. 134, no. 12, pp. 982–988, 2014.

[5] T. Emmei, H. Fujimoto, and Y. Hori, "Proposal of High Speed Force Curve Measurement Method Considering Cantilever Dynamics for Atomic Force Microscope," *IEEJ InternationalWorkshop on Sensing, Actuation, and Motion Control Proposal*, pp. 2–7, 2015.

[6] K. Aoki and H. Fujimoto, "Novel Nano-scale Servo Technique on Atomic Force Microscope," *in Proc. IEEJ Industory Apprications Society Conf*, pp. 127–132, 2006.

[7] Daniel Y. Abramovitch, Sean B. Andersson, Lucy Y. Pao and G. Schitter, "A Tutorial on the Mechanisms, Dynamics, and Control of Atomic Force Microscopes," pp. 3488–3502, 2007.

[8] D. J. C. A. Sebastian', M. V. Salapaka and J. P. Cleveland, "Harmonic analysis based modeling of tapping-mode AFM," *Proceedings of the 1999 American Control Conference*, pp. 232–236, 1999.