

# Proposal of High Speed Force Curve Measurement Method Considering Cantilever Dynamics for Atomic Force Microscope

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Atomic Force Microscope (AFM) is a scanning probe microscope with nano-scale resolution as well as an indispensable device for nanotechnology. Since AFM probe physically touches the sample surface, expectations for sample dynamics measurement is raising. One of the most common measurements is force curve measurement. Conventionally, atomic force is detected by spring constant of the cantilever. In high speed measurement, however, the cantilever oscillates with its resonance frequency and could not detect the atomic force. In this paper, a novel method to identify cantilever dynamics and high speed force curve measurement using atomic force observer (AFO) is proposed, and its superiority to the conventional method was demonstrated by simulations and experiments.

**Keywords:** Atomic force microscope (AFM), Atomic force observer, Force measurement, Force curve

## 1. Introduction

The Atomic Force Microscope 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 shows the AFM of the authors' research group.

Fig. 2 shows the structure of AFM. AFM is composed of a piezo actuator which handles the stage and a nanoprobe 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, surface topology can be measured with very high resolution.

There are two common types of AFM<sup>(1)</sup>: 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 is used. Because AFM probe can hold physical contact with the sample, expectations for sample dynamics measurement are raising<sup>(2)-(5)</sup>. The authors' research group proposed simultaneous estimation of surface topology and elasticity using contact-mode AFM<sup>(6)(7)</sup>. Dynamics measurements are especially important in biomaterials, where there are a lot of unknown behavior.

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 deflection and the spring constant of cantilever. However, in high speed measurement,

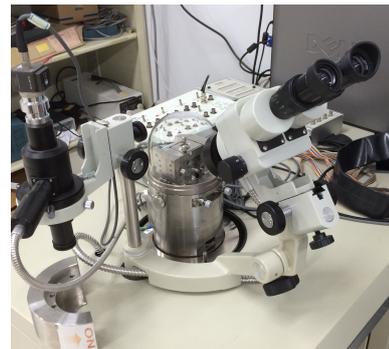


Fig. 1. Appearance of AFM (JSPM-5200).

cantilever vibration is bigger and measuring the true force is more difficult. 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 measurement. In this paper, high speed force measurement method based on atomic force observer (AFO)<sup>(8)</sup> 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. 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 in simulations and experiments. Finally, section 6 provides a summary and conclusion.

## 2. Modeling

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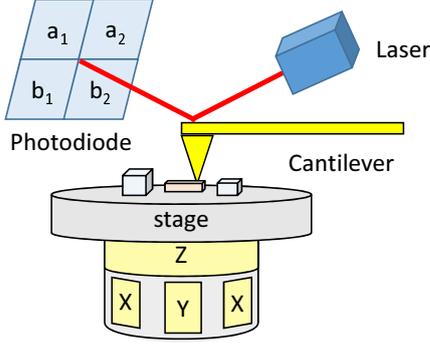


Fig. 2. Structure of AFM.

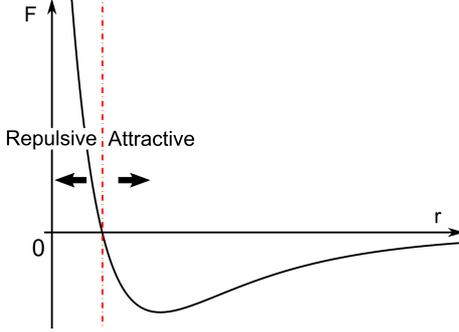


Fig. 3. Force distance relation.

**2.1 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. But when they are closer, they exert attractive forces with each other. 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. 3 shows the relationship between the atomic force and distance based on Lennard-Jones model<sup>(9)</sup>. Lennard-Jones model is an approximation of atomic force  $F_a$  and can be expressed as a function of atomic distance  $r$ .

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

Where  $\varepsilon$  is the binding energy and  $\sigma$  is the lattice constant.

**2.2 Modeling of the AFM** Fig. 4 shows the physical model of AFM<sup>(10)</sup> and Fig. 5 shows its block diagram. Here,  $r$ ,  $r_0$ ,  $z_c$ ,  $F_a(r)$  are respectively 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  $F_a$ . Then,  $P(s)$  is the transfer function of cantilever from atomic force to deflection; its nominal model  $P_n(s)$  is given as

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

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

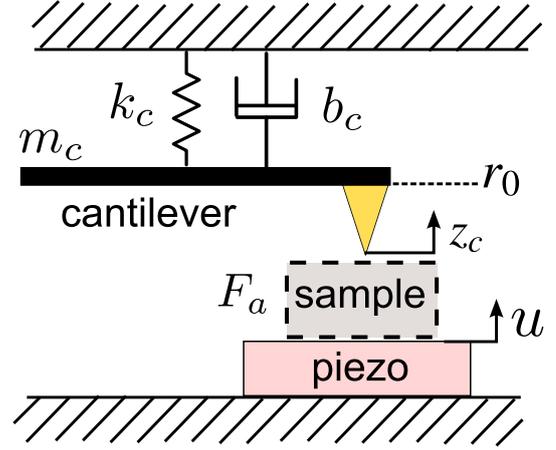


Fig. 4. Model of AFM.

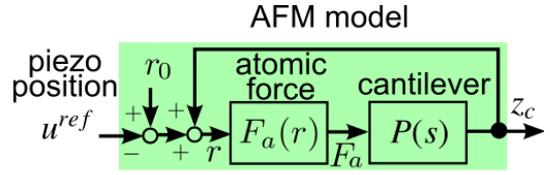


Fig. 5. Block diagram of AFM.

### 3. Conventional force curve measurement

**3.1 Method of force curve measurement** Force curve measurement is known as the method to measure the atomic force with AFM. Force curve measurement is implemented by following process.

- 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. Force curve is the plot of atomic force against piezo displacement in STEP 3 and STEP 4.

Fig. 6 is an example of force curve. As explained in section 2, 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 when retracting like Fig. 6.

**3.2 Problem of conventional method** The atomic force is conventionally detected by 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 difficult a correct force measurement.

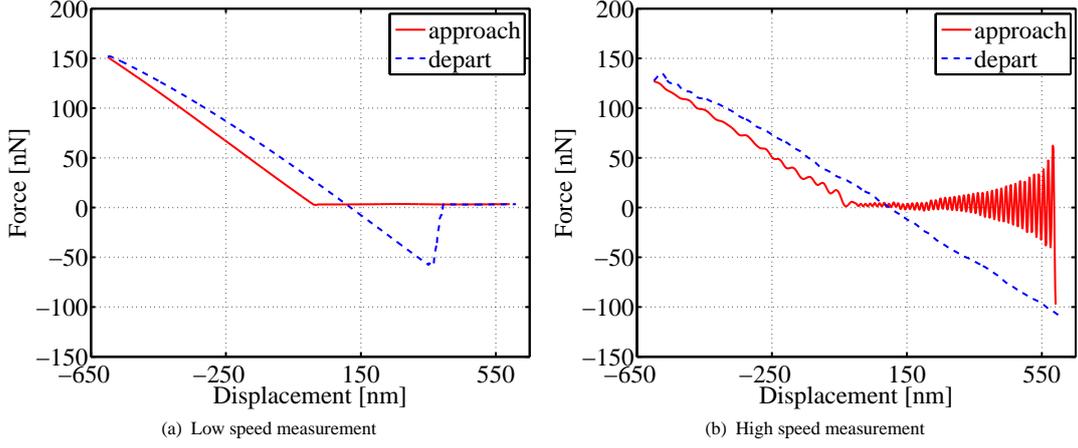


Fig. 7. Force curve (experiment).

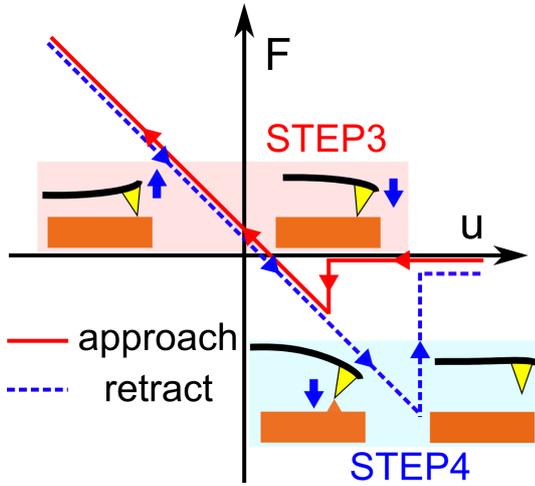


Fig. 6. Force curve and response of cantilever.

Fig. 7 (a), (b) are the force curves of low speed measurement and high speed measurement. In slow speed measurement, the atomic force can be correctly measured and the force curve is similar to Fig. 6. However, in high speed measurement, force curve vibrates because the conventional method cannot separate cantilever oscillation from the deflection caused by atomic force.

#### 4. 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. In this paper, a system identification using impulse response is proposed and this can make the design of AFO possible.

**4.1 Proposal of dynamics identification for AFM cantilever** As shown in Fig. 4, it is difficult to separate the dynamics of AFM cantilever from the atomic force between the probe and the sample, that is frequency response from piezo position to cantilever deflection for system identification. However, the atomic force between sample surface and probe tip changes nonlinearly according to their distance and

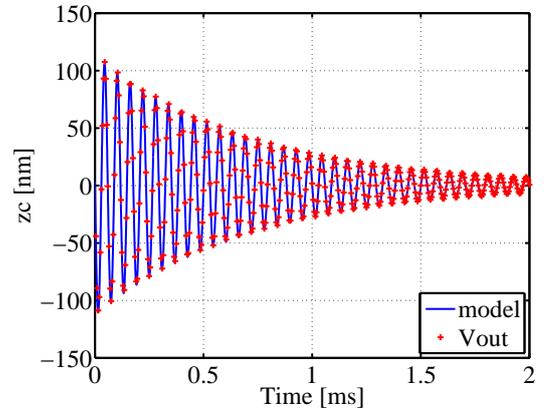


Fig. 8. System identification for AFM cantilever (experiment).

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 probe and sample are pulled apart, impulse force is impressed due to adhesive force. The probe and the sample keep enough distance so that they do not interact with each other. Therefore, cantilever dynamics without sample dynamics can be obtained.

In Fig. 8, transient response of cantilever is shown. Generally, time response of damped vibration is given as (3).

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

$\zeta$  and  $\omega_0$  can be given as (4) and (5) by using  $m_c$ ,  $b_c$ , and  $k_c$ .

$$\omega_0 = \sqrt{\frac{k_c}{m_c}}, \quad (4)$$

$$\zeta = \frac{b_c}{2m_c \omega_0}. \quad (5)$$

$P_n(s)$  identified from fitting is given as (6)

$$P_n(s) = \frac{1}{1.8 \times 10^{-11} s^2 + 4.9 \times 10^{-8} s + 0.20}. \quad (6)$$

Then, by fitting (3) to measurement result, the dynamics of the cantilever can be detected.

**4.2 Design of atomic force observer** In this section,

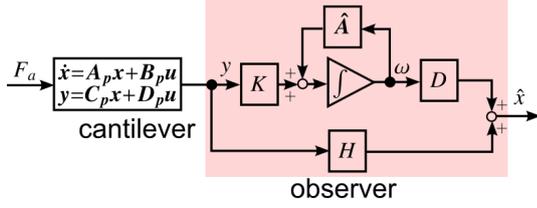


Fig. 9. Atomic force observer.

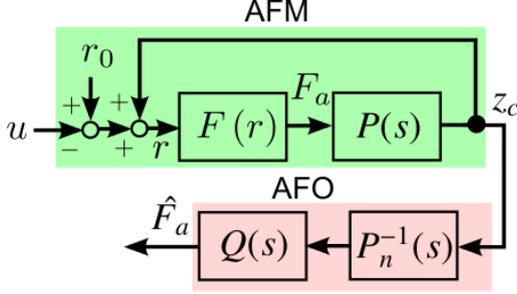


Fig. 10. Block diagram equivalent to atomic force observer.

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

$$m_c \ddot{z}_c + b_c \dot{z}_c + k_c z_c = F_a. \quad (7)$$

From (7), state equation of extended system can be expressed as (8) and its state variables are  $z_c$ ,  $\dot{z}_c$ , and  $F_a$ .

$$\dot{x} = Ax, \quad (8)$$

$$A = \begin{bmatrix} 0 & 1 & 0 \\ -\frac{k_c}{m_c} & -\frac{b_c}{m_c} & \frac{1}{m_c} \\ 0 & 0 & 0 \end{bmatrix}. \quad (9)$$

Its output equation is

$$y = Cx, \quad (10)$$

$$C = [1 \quad 0 \quad 0]. \quad (11)$$

Thus, minimal order observer can be designed taking  $z_c$  as output. Fig. 9 shows this minimal order observer and Fig. 10 can be derived from (8)-(11).  $P_n^{-1}(s)$  is the inverse transfer function of nominal plant,  $Q(s)$  is the second order low pass filter to make proper inverse model and  $\omega_c$  is the cut off frequency of  $Q(s)$ .

$$P_n^{-1}(s) = m_c s^2 + b_c s + k_c, \quad (12)$$

$$Q(s) = \frac{\omega_c^2}{(s + \omega_c)^2}. \quad (13)$$

Fig. 11 is the structure of the conventional method and Fig. 12 is that of proposal method.

## 5. Simulations and experiments

**5.1 Simulations of high speed force curve measurement** Using the cantilever dynamics in the section 4.1,

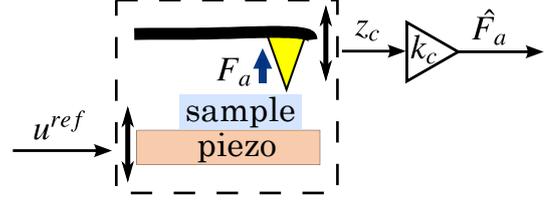


Fig. 11. The structure of the conventional method.

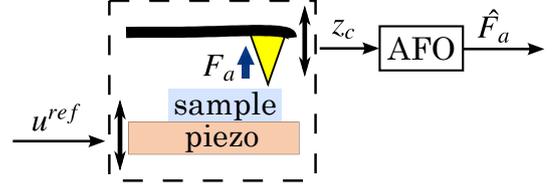


Fig. 12. The structure of the proposal method.

Table 1. Conditions of the simulations.

	value	unit
sampling frequency	40	kHz
pole of LPF	20	kHz
measurement range	$\pm 5.0$	nm
$\sigma$	$1.8 \times 10^{-10}$	m
$\varepsilon$	$1.6 \times 10^{-20}$	J
$r_0$	$1.6 \times 10^{-10}$	m

the approach and retraction of the probe to the sample is simulated. Table 1 shows the simulation parameters. Lennard Jones model is used to describe the atomic force and it is assumed that probe tip is made of  $\text{SiO}_2$  and the sample is Cu. Fig. 13(a) plots the displacement of the piezo actuator. The motion of the piezo actuator follows STEP 2-STEP 4 in section 3.1.

Fig. 13(b) shows the results of the simulation. Cantilever oscillates at 0 sec and 0.18 sec due to adhesive forces. Conventional method is not able to measure correctly the atomic force. On the other hand, proposed method is not affected by the cantilever oscillation and can estimate the atomic force correctly.

**5.2 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 experiment conditions are the same as those of simulation, except for the initial distance. This is because it is difficult to measure the distance between probe tip and sample. Thus, initial point is decided by the cantilever deflection instead of using  $r_0$ . Fig. 14(a) and Fig. 15(a) show the displacement of the piezo actuator. The velocity of the piezo actuator is  $v_{normal} = 1.2$  nm/ms in normal speed measurement and  $v_{high} = 240$  nm/ms in high speed measurement.

Fig. 14 shows the normal speed measurement and Fig. 15 shows the high speed measurement of atomic force. Besides, Fig. 16 is the force curve of them. 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.

The experimental result widely differs from simulation in atomic force. Since the probe and the sample are composed

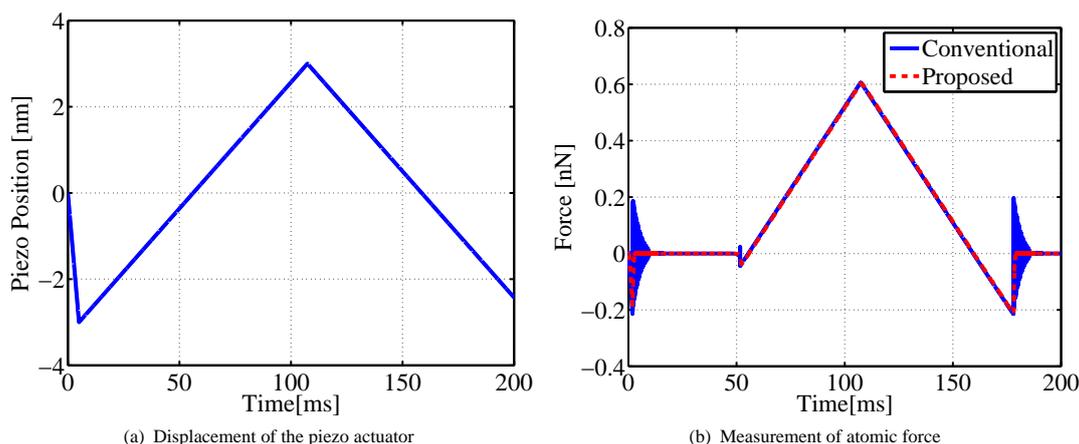


Fig. 13. Results of the simulations.

of many atoms, 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<sup>(11)</sup>.

## 6. 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. The proposed method can measure the correct atomic force even when the cantilever is vibrating. Not just 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. Therefore, the distance between probe tip and sample changes, makes the force curve vibrate. To solve this problem, in the future vibration suppression control for the cantilever will be studied.

## References

- (1) T. Shiraishi, H. Fujimoto, "Proposal of surface topology observer considering Z-scanner for high speed AFM," in Proc. IEEE Industry Applications Society Conf., pp. 37–42, 2009, (in Japanese).
- (2) P. Agarwal, M. V. Salapaka, "Real time estimation of equivalent cantilever parameters in tapping mode atomic force microscopy," *Applied Physics Letters*, 95, 083113 2009.
- (3) M. Lantz, H. Hug, R. Hoffman, "Quantitative measurement of short-range chemical bonding forces," *Science*, Vol. 291, pp. 2580–2583 2001.
- (4) C. Onal, M. Sitti, "A Scaled Bilateral Control System for Experimental One-dimensional Teleoperated Nanomanipulation," *The International Journal of Robotics Research*, pp. 484–497, 2009.
- (5) C. D. Onal, M. Sitti, "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.
- (6) S. Watanabe, H. Fujimoto, "Estimation of Sample Elasticity Utilizing Previous Line Surface Topography by AFM," in Proc. IEEE Industry Applications Society Conf., pp. 6460–6465, 2012 (in Japanese).
- (7) S. Watanabe, H. Fujimoto, "Proposal for Simultaneous Estimation of Sample Surface Topography and Elasticity Utilizing Contact-Mode AFM," *IEEE Transactions on Industry Applications*, Vol. 134, No. 12, pp. 982–988 2014 (in Japanese).
- (8) K. Aoki, H. Fujimoto, "Novel nano-scale servo technique on atomic force microscope," in Proc. IEEE Industry Applications Society Conf., pp. 127–132,

2006 (in Japanese).

- (9) J. Israelachvili, "Intermolecular & surface Forces," Academic Press, Third Edition, 2011.
- (10) A. Sebastian, M. V. Salapaka, D.J.Chen, J. P. Cleveland, "Harmonic analysis based modeling of tapping-mode AFM," *Proceedings of the American Control Conference*, pp. 232–236, 1999.
- (11) J. B. Pethica, W. C. Oliver, "Tip Surface Interactions in STM and AFM," *Physica Scripta*. Vol. 19, pp. 61–66 1987.

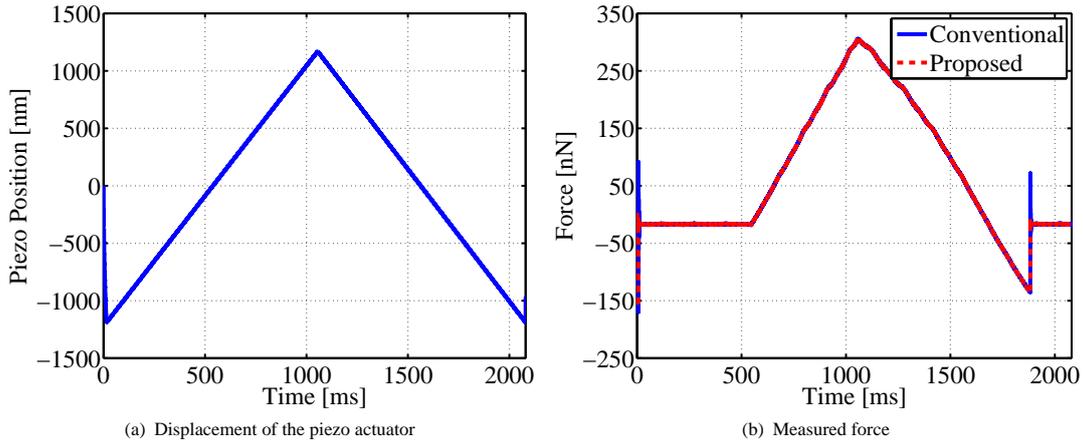


Fig. 14. Normal speed force curve measurement ( $v_{normal} = 1.2$  nm/ms).

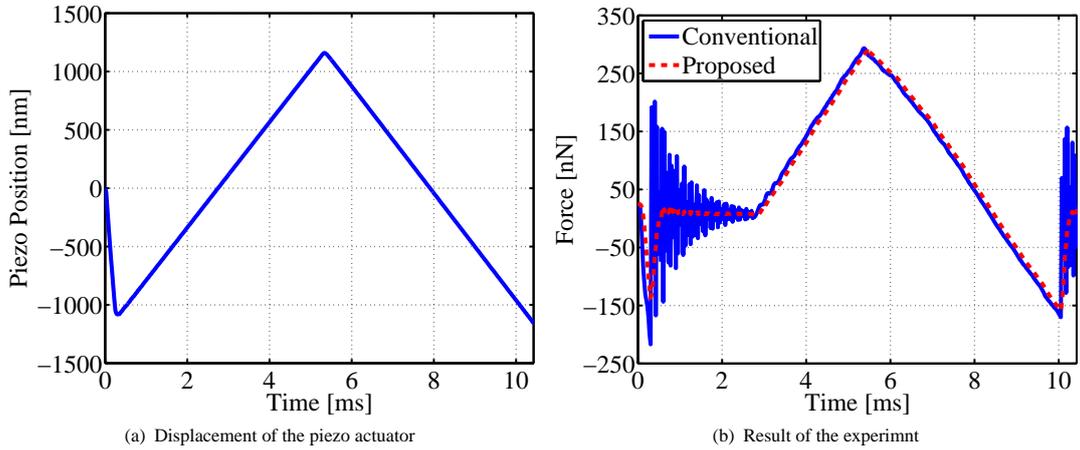


Fig. 15. High speed force curve measurement ( $v_{high} = v_{normal} \times 200 = 240$  nm/ms).

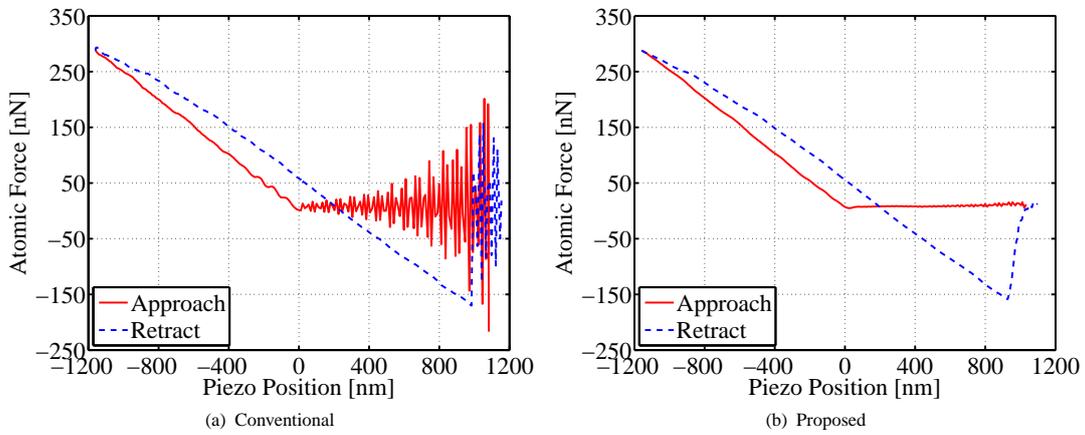


Fig. 16. The force curve with high speed measurement.